Tunisia Chemistry Conference

TCC 2016

Organized by the

Tunisian Chemical Society

Société Chimique de Tunisie

19-22 December 2016

Bel Azur Hotel, Hammamet - Tunisia

Lectures and Communications Abstracts
List of Participants

Tunisian Chemical Society - Short Program of TCC 2016

Monday 19 December 2016	
09.00 - 09.30	**Opening Ceremony**
09.30 - 10.10	Plenary Lecture 1 **Cyrille KOUKLOVSKY** *Inst. de Chimie Moléculaire et des Matériaux d'Orsay, Université de Paris-Sud, France*
10.15 - 10.45	Invited Lecture 1 **Kalthoum AZZABI** *Centre National Universitaire de la Documentation Scientifique et Technique*
10.45 - 11.30	**Coffee break + Poster Session 1 (P1 - P43)**
11.30 - 13.00	**Oral Communications – Session 1 : OC1 - OC6**
13.00	***Lunch***
14.30 - 15.10	Plenary Lecture 2 **Azaiez BEN AKACHA** *Faculty of Science of Tunis, University Tunis El Manar, Tunisia*
15.15 - 16.30	**Oral Communications – Session 2 : OC7 - OC11**
16.30 - 17.30	**Coffee break + Poster Session 2 (P44 - P86)**
17.30 - 18.45	**Oral Communications – Session 3 : OC12 - OC16**
19.30	***Dinner***
Tuesday 20 December 2016	
09.00 - 09.40	Plenary Lecture 3 **Philippe C. GROS** *SRSMC, Université de Lorraine, CNRS, 54506 Vandoeuvre-Lès-Nancy, France*
09.40 - 10.40	**Coffee break + Poster Session 3 (P87 - P129)**
10.45 - 12.15	**Oral Communications – Session 4 : OC17 - OC22**
13.00	***Lunch***
14.30 - 15.10	Plenary Lecture 4 **Jean-Marc GRENECHE** *Université du Maine, FST, Le Mans - France*
15.15 - 16.30	**Oral Communications – Session 5 : OC23 - OC27**
16.30 - 17.30	**Coffee break + Poster Session 4 (P130 - P172)**
17.30 - 19.00	**Oral Communications – Session 6 : OC28 - OC33**
19.30	***Dinner***

Wednesday 21 December 2016	
08.30 - 09.10	Plenary Lecture 5 **Salim BARKALLAH** *Group Leader, Cambridge Isotope Laboratories*
09.15 - 10.30	**Oral Communications – Session 7 : OC34 - OC38**
10.30 - 11.30	**Coffee break + Poster Session 5 (P173 - P215)**
11.30 - 13.00	**Oral Communications – Session 8 : OC39 - OC44**
13.00	***Lunch***
15.00 - 15.40	Plenary Lecture 6 **Jean-Claude LEPRETRE** *Lab. de physicochimie des matériaux et des interfaces - LEPMI, Grenoble - France*
15.45 - 16.45	**Coffee break + Poster Session 6 (P216 - P256)**
17.00 - 19.30	**Ordinary General Assembly of the Tunisian Chemical Society**
19.30	***Dinner***
21.00	**Election of the new National Bureau of the Tunisian Chemical Society** **Counting of votes and announcement of results**
Thursday 22 December 2016	
09.00 - 10.45	**Oral Communications – Session 9 : OC45 - OC58**
10.45 - 11.15	**Coffee break**
11.15 - 11.45	Invited Lecture 2 **Nabil KHELIFI** *Responsible for Springer's Publishing Program in the Middle East and North Africa*
12.00	***Awards distribution and closing ceremony***
13.00	***Check Out, Lunch and leaving the hotel***

FOREWORD

The Tunisia Chemical Conference (TCC 2016) is organized by the Tunisian Chemical Society (SCT). Held every two years, this conference is taking place for the nineteenth time in the 38 years age of the Tunisian Chemical Society. As a chair man of the TCC 2016, and on behalf of the Organizing Committee, I am delighted to warmly welcome you all in Hammamet for this event held under the auspices of the Ministry of Higher Education and Scientific Research, the University of Tunis El Manar and the Faculty of Sciences of Tunis.

Attended by more than 400 participants from different countries, many of them visiting Tunisia for the first time, the TCC 2016 will cover many fields and topics of Chemistry through 6 plenary and 2 invited lectures, 141 oral and 252 poster communications.

I would like to thank the presenters and an international panel of world renowned keynote speakers for their active participation and for their willingness to share their latest research and idea.

I want to thank all my colleagues of the Organizing and Scientific Committees for their support, their help and their availability. Without their efforts, this conference would not be possible.

Let's hope that our conference will produce a synergy that affects the different areas of chemistry research.

Keep up the good work and enjoy your stay in this lovely resort of Hammamet.

Adel MEGRICHE

Chairman of TCC 2017
President of the Tunisian Chemical Society

SCIENTIFIC COMMITTEE

Adel MEGRICHE (Chairman)	Tunisia	University of Tunis El Manar, FST-Tunis
Moheddine ABDELLAOUI	Tunisia	Institut National de Recherche et d'Analyse Physico-chimique, Sidi Thabet
Rym ABIDI	Tunisia	Université de carthage, FSB-Bizerte
Salim BARKALLAH	U.S.A	Cambridge Isotope Laboratories, Andover, USA
Nizar BELLAKHAL	Tunisia	University of Carthage, INSAT-Tunis
Mohamed BELHOUCHET	Tunisia	University of Sfax, FSS-Sfax
Azaiez BEN AKACHA	Tunisia	University of Tunis El Manar, FST-Tunis
Taïcir BEN AYED	Tunisia	University of Carthage, INSAT-Tunis
Hatem BEN ROMDHANE	Tunisia	University of Tunis El Manar, FST-Tunis
Ridha BEN SALEM	Tunisia	University of Sfax, FSS-Sfax
Latifa BERGAOUI	Tunisia	University of Carthage, INSAT-Tunis
Taoufik BOUBAKER	Tunisia	University of Monastir, FSM-Monastir
Bechir CHAOUACHI	Tunisia	University of Gabès, ENIG-Gabès
Samia CHERIF	Tunisia	University of Tunis El Manar, ISSBAT-Tunis
Mohamed Lotfi EFRIT	Tunisia	University of Tunis El Manar, FST-Tunis
Jean-Marc GRENECHE	France	Université du Maine, Faculté des Sciences et Techniques, Le Mans, France

SCIENTIFIC COMMITTEE (continuation)

Philippe C. GROS	France	University of Lorraine, President SCF Lorraine - France
Halim HAMMI	Tunisia	University of Carthage, CNRSM-Borj Cedria
Ismail KHATTECH	Tunisia	University of Tunis El Manar, FST-Tunis
Mohamed KHITOUNI	Tunisia	University of Sfax, FSS-Sfax
Cyrille KOUKLOVSKY	France	Université de Paris-Sud, Institut de Chimie Moléculaire et des Matériaux d'Orsay Société Chimique de France - President of the Organic Chemistry Division
Jean-Claude LEPRETRE	France	Laboratoire de physicochimie des matériaux et des interfaces - LEPMI - Grenoble, France
Adel M'NIF	Tunisia	University of Carthage, CNRSM-Borj Cedria
Farhat REZGUI	Tunisia	University of Tunis El Manar, FST-Tunis
Mehrez ROMDHANE	Tunisia	University of Gabès, ENIG-Gabès
Ezzeddine SRASRA	Tunisia	University of Carthage, CNRSM-Borj Cedria
Hassib TOUNSI	Tunisia	University of Sfax, FSS-Sfax
Med Abderrahmane SANHOURY	Mauretania	Faculty of Sciences and Technics, USTM Nouakchott, Mauritania

ORGANIZING COMMITTEE

Halim HAMMI (Chairman)	University of Carthage, CNRSM-Borj Cedria
Mohamed Mouldi ABDELKAFI	Tunisian Chemical Society
Rym ABIDI	Université de carthage, FSB-Bizerte
Mohamed BELHOUCHET	University of Sfax, FSS-Sfax
Nizar BELLAKHAL	University of Carthage, INSAT-Tunis
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Bechir CHAOUACHI	University of Gabès, ENIG-Gabès
Mohamed Lotfi EFRIT	University of Tunis El Manar, FST-Tunis
Mohamed KHITOUNI	University of Sfax, FSS-Sfax
Imed LAAJIMI	Tunisian Chemical Society
Hatem MAJDOUB	University of Monastir, FSM-Monastir
Adel MEGRICHE	University of Tunis El Manar, FST-Tunis
Amine MNIF	University of Tunis El Manar, FST-Tunis
Farhat REZGUI	University of Tunis El Manar, FST-Tunis
Mehrez ROMDHANE	University of Gabès, ENIG-Gabès

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Monday 19 December 2016 (Morning)							
09.00 - 09.30	**O p e n i n g C e r e m o n y**						
09.30 - 10.10	**Plenary Lecture 1** **Cyrille KOUKLOVSKY** *Institut de Chimie Moléculaire et des Matériaux d'Orsay, Université de Paris-Sud, France* **Synthesis of New Aminoacids: Applications in Peptide Synthesis**						*Chairperson:* **Adel Megriche**
10.15 - 10.45	**Invited Lecture 1** **Kalthoum AZZABI** *Centre National Universitaire de la Documentation Scientifique et Technique* **Role of CNUDST as a Provider of Scientific and Technical Information**						
10.45 - 11.30	**C o f f e e b r e a k + Poster Session 1 (P 1 - P 43)**						
Oral Communications - Session 1							
	Room A - *Chairperson : N. Ben Hmida*		**Room B -** *Chairperson : H. Tounsi*		**Room C -** *Chairperson : H. Ben Romdhane*		
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*	
11.30 - 11.45	**OC-01 A**	S. ABID	**OC-02 B**	M. ABID DERBAL	**OC-01 C**	B. AOUR	
11.45 - 12.00	**OC-02 A**	L. BOUBAKRI	**OC-04 B**	L. AYDI	**OC-02 C**	S BEN CHEIKH	
12.00 - 12.15	**OC-03 A**	N. BOUZAINI	**OC-05 B**	Y. BAHROUNI	**OC-03 C**	S. BEN MAHMOUD	
12.15 - 12.30	**OC-04 A**	A. CHAARI	**OC-06 B**	J. BAIZIG	**OC-04 C**	M. DARDOURI	
12.30 - 12.45	**OC-05 A**	M.R. CHEBBI	**OC-07 B**	C. BARHOUMI	**OC-05 C**	A. EL MAHDI	
12.45 - 13.00	**OC-06 A**	S. CHEKIR	**OC-08 B**	W. BELAM	**OC-07 C**	A. JEBNOUNI	
13.00	***L u n c h***						

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Monday 19 December 2016 (Afternoon)						
14.30 - 15.10	**Plenary Lecture 2** **Azaiez BEN AKACHA** *Faculty of Science of Tunis, University Tunis El Manar, Tunisia* **Chlorophosphites: Preparations, Conformationally Study by (^{31}P, ^{13}C, ^{1}H NMR, DFT, X-Rays) and Precursors Synthesis of Heterocyclic Organophosphorus Compounds**				*Chairperson:* **Mohamed Lotfi Efrit**	
Oral Communications - Session 2						
	Room A - *Chairperson : I. Chehidi*		**Room B -** *Chairperson : M. Belhouchet*		**Room C -** *Chairperson : H. Ben Romdhane*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
15.15 - 15.30	**OC-07 A**	M. CHERIF	**OC-09 B**	I. BEN BELGACEM	**OC-08 C**	N. KAABI
15.30 - 15.45	**OC-08 A**	I. CHNITI	**OC-10 B**	M. BEN HADJ SALAH	**OC-09 C**	R. MEJRI
15.45 - 16.00	**OC-09 A**	K. CHOUAIB	**OC-11 B**	A.L. BEN HAFSIA	**OC-10 C**	H. TALMOUDI
16.00 - 16.15	**OC-10 A**	M. DEBBABI	**OC-12 B**	M. BEN HAMDEN	**OC-11 C**	M.Z. SLIMANI
16.15 - 16.30	**OC-11 A**	Y. DGACHI	**OC-13 B**	I. BEN MESSAOUD	**OC-12 C**	A. ZRELLI
16.30 - 17.30	**C o f f e e b r e a k + Poster Session 2 (P 44 - P 86)**					
Oral Communications - Session 3						
	Room A - *Chairperson : S. Barkallah*		**Room B -** *Chairperson : M. Khitouni*		**Room C -** *Chairperson : L. Atrous*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
17.30 - 17.45	**OC-12 A**	F. EBNOU	**OC-14 B**	C. BEN M'LEH	**OC-13 C**	F. AYARI
17.45 - 18.00	**OC-13 A**	M. ELALIM	**OC-15 B**	S.BOUGHANMI	**OC-14 C**	E. BEN KHALIFA
18.00- 18.15	**OC-14 A**	M. GARDELLY	**OC-16 B**	M. BOUCHAOUR	**OC-15 C**	R. BESGHAIER
18.15 - 18.30	**OC-15 A**	S. GHANAY			**OC-16 C**	N. AMEUR
18.30- 18.45	**OC-16 A**	K. HAMROUNI				
19.30	***D i n n e r***					

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Tuesday 20 December 2016 (Morning)						
09.00 - 09.40	**Plenary Lecture 3** **<u>Philippe C. GROS</u>** *SRSMC, Université de Lorraine, CNRS, 54506 Vandoeuvre-Lès-Nancy, France* **From ruthenium to iron sensitizers: A challenge towards more ecofriendly hybrid solar cells**					*Chairperson:* **Salim Barkallah**
09.40 - 10.40	**C o f f e e b r e a k + Poster Session 3 (P 87 - P 129)**					
	Oral Communications - Session 4					
	Room A - *Chairperson : F. Rezgui*		**Room B -** *Chairperson : F. Touati*		**Room C -** *Chairperson : M. Ben Sik Ali*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
10.45 - 11.00	**OC-17 A**	M. HASSINE	**OC-17 B**	J. CHEKIR MZALI	**OC-17 C**	F. BOUJELBENE
11.00 - 11.15	**OC-18 A**	A. HFAIEDH	**OC-18 B**	M.K. CHINE	**OC-18 C**	A. BOULARES
11.15 - 11.30	**OC-19 A**	N. JEBLI	**OC-19 B**	S. CHOUCHENE	**OC-19 C**	K. CHAIR
11.30 - 11.45	**OC-20 A**	D. KANZARI MNALLAH	**OC-20 B**	O. DACHRAOUI	**OC-20 C**	H. CHEMINGUI
11.45 - 12.00	**OC-21 A**	D. MARROUKI	**OC-21 B**	K. DAMMAK	**OC-21 C**	M. CHKIOUA
12.00 - 12.15	**OC-22 A**	E. KENICHE	**OC-22 B**	S. DGACHI	**OC-22 C**	H. AZAARI
13.00	***L u n c h***					

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Tuesday 20 December 2016 (Afternoon)						
14.30 - 15.10	**Plenary Lecture 4** **<u>Jean-Marc GRENECHE</u>** *Université du Maine, FST, Le Mans - France* **Mössbauer spectrometry as a local tool to investigate solid state physics and chemistry: Applications of Mössbauer spectrometry**					*Chairperson:* **Farhat Rezgui**
Oral Communications - Session 5						
	Room A - *Chairperson : M. Askri*		**Room B -** *Chairperson : B. Chaouachi*		**Room C -** *Chairperson : A. Mnif*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
15.15 - 15.30	**OC-23 A**	A. MEDDEB	**OC-23 B**	W. DRIDI	**OC-23 C**	A. DRIDI
15.30 - 15.45	**OC-24 A**	H. MTIRAOUI	**OC-24 B**	A. ELMHAMDI	**OC-24 C**	C. ELAZIZI
15.45 - 16.00	**OC-25 A**	N. OMRI	**OC-25 B**	S. FESSI	**OC-25 C**	W. ESSAFI
16.00 - 16.15	**OC-26 A**	O. OUERFELLI	**OC-26 B**	R. FEZAI	**OC-26 C**	A. FAJRAOUI
16.15 - 16.30	**OC-27 A**	S. RAOUAFI	**OC-27 B**	A. GHARBI BAKLOUTI	**OC-27 C**	Z. GOUID
16.30 - 17.30	**C o f f e e b r e a k + Poster Session 4 (P 130 - P 172)**					
Oral Communications - Session 6						
	Room A - *Chairperson : M.L. Efrit*		**Room B -** *Chairperson : M. Abdelhédi*		**Room C -** *Chairperson : R. Zarrougui*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
17.30 - 17.45	**OC-28 A**	H. RIGUENE	**OC-28 B**	A. GUESMI	**OC-28 C**	S. ISMAILI
17.45 - 18.00	**OC-29 A**	N. SBEI	**OC-29 B**	A. HARCHANI	**OC-29 C**	W. JAHOUACH
18.00 - 18.15	**OC-30 A**	I. TALBI	**OC-30 B**	F. HMIDA	**OC-30 C**	A. LABIDI
18.15 - 18.30	**OC-31 A**	A. TOUMI	**OC-31 B**	N. HOSNI	**OC-31 C**	
18.30 - 18.45			**OC-32 B**	H. IBN GHARSALLAH	**OC-32 C**	A. LEMSI
18.45 - 19.00			**OC-33 B**	S. JEBRI	**OC-33 C**	S. MANAI
19.30	***D i n n e r***					

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Wednesday 21 December 2016 (Morning)						
08.30 - 09.10	**Plenary Lecture 5** **Salim BARKALLAH** *Group Leader, Cambridge Isotope Laboratories* **Stable Isotope Labeled Compounds: Applications in Organic Synthesis, Environmental Studies, Medical Research, Diagnostics and Drug (Re)Discovery**					*Chairperson:* **Azaiez Ben Akacha**
Oral Communications - Session 7						
	Room A - *Chairperson : T. Ben Ayed*		**Room B -** *Chairperson : D. Hellali*		**Room C -** *Chairperson : N. Bellakhal*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
9.15 - 9.30	**OC-34 A**	L. ALOUI	**OC-34 B**	I. KADRI	**OC-34 C**	R. MEJDOUB
9.30 - 9.45	**OC-35 A**	M. BOUBAKER	**OC-35 B**	S. KEFI	**OC-35 C**	A. MEJRI
9.45 - 10.00	**OC-36 A**	A. JELASSI	**OC-36 B**	M. KHABBOUCHI	**OC-36 C**	S. MEZGHICH
10.00 - 10.15	**OC-37 A**	H. KRAYNI	**OC-37 B**	S. KHELIFI	**OC-37 C**	M. MEZNI
10.15 - 10-30	**OC-38 A**	F. MANNAI	**OC-38 B**	S. KOUASS	**OC-38 C**	R. MIADI
10.30 - 11.30	**C o f f e e b r e a k + Poster Session 5 (P 173 - P 215)**					
Oral Communications - Session 8						
	Room A - *Chairperson : H. Mejdoub*		**Room B -** *Chairperson : A. Haddad*		**Room C -** *Chairperson : M. Romdhane*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
11.30 - 11.45	**OC-39 A**	K. MKADMINI HAMMI	**OC-39 B**	I. LABIDI	**OC-39 C**	
11.45 - 12.00	**OC-40 A**	Z. MZOUGHI	**OC-40 B**	M. MAAMAR	**OC-40 C**	M. MRAD
12.00 - 12.15	**OC-41 A**	I. SAIDI	**OC-41 B**	J. MAAMRI	**OC-41 C**	N. NAGHMOUCHI
12.15 - 12.30	**OC-42 A**	F. TALEB	**OC-42 B**	N. MAHBOULI REHOUMA	**OC-42 C**	
12.30 - 12.45			**OC-43 B**	M. MAHFOUDH	**OC-43 C**	S. REKIK
12.45 - 13.00			**OC-44 B**	M. MATHLOUTHI	**OC-44 C**	M. SAIDI
13.00	***L u n c h***					

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Wednesday 21 December 2016 (Afternoon)		
15.00 - 15.40	**Plenary Lecture 6** **Jean-Claude LEPRETRE** *Laboratoire de physicochimie des matériaux et des interfaces - LEPMI, Grenoble - France* **The crucial role of organic materials towards the development of lithium battery**	*Chairperson:* **Nizar Bellakhal**
15.45 - 16.45	**C o f f e e b r e a k + Poster Session 6 (P 216 - P 256)**	
17:00 - 19:30	**Ordinary General Assembly** **of the Tunisian Chemical Society**	
19.30	***D i n n e r***	
21:00	**Election of the new National Bureau of the Tunisian Chemical Society** **Counting of votes and announcement of results**	

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Thursday 22 December 2016						
Oral Communications - Session 9						
	Room A - *Chairperson : M. Abdelhedi*		**Room B -** *Chairperson : W. Sta*		**Room C -** *Chairperson : N. Bellakhal*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
09.00 - 09.15	**OC-52 B**	A. RAYES	**OC-45 B**	M. MEKNI ABROUGUI	**OC-45 C**	W. SASSI
09.15 - 09.30	**OC-53 B**	D. RTIBI	**OC-46 B**	R. MESSAOUDI	**OC-46 C**	S. TRABELSI SOUISSI
09.30 - 09.45	**OC-54 B**	S. SAID	**OC-47 B**	S. MRAD	**OC-47 C**	M. ZAGHDOUDI
09.45 - 10.00	**OC-55 B**	H. SEHIMI	**OC-48 B**	S. NAJEH	**OC-48 C**	M. ZANNEN
10.00 - 10.15	**OC-56 B**	S. SOUDANI	**OC-49 B**	H. NEFZI TOUIL	**OC-49 C**	R. ZARROUGUI
10.15 - 10.30	**OC-57 B**	S. TRABELSI	**OC-50 B**	R. OUESLATI OMRANI		
10.30 - 10.45	**OC-58 B**	H. ZOUAOUI	**OC-51 B**	M. RAJAH		
10.45 - 11.15	**C o f f e e b r e a k**					
11.15 - 11.45	**Invited Lecture 2** **<u>Nabil KHELIFI</u>** *Responsible for Springer's Publishing Program in the Middle East and North Africa* **How to get your paper published in a scientific journal: Tips from a Springer Editor**				*Chairperson:* **Halim Hammi**	
12.00	**Awards distribution and Closing Ceremony**					
13.00	**Lunch and Check out**					

Speakers' Abstracts

Cyrille KOUKLOVSKY

Professor of Chemistry

Date and place of birth	29 march 1964, Paris, France
Citizenship	French
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Equipe Méthodologies, Synthèse et Molécules Thérapeutiques (MSMT)
Institut de Chimie Moléculaire et des Matériaux d'Orsay
Bâtiment 410
Université de Paris-Sud, F-91405 Orsay, France

Education

199&-1994 Postdoctoral associate *Supervisor :* Professor Steven V. Ley
University of Cambridge, Great Britain
« *Total Synthesis of Rapamycin* »

1986-1989 PhD graduate student in organic chemistry
Supervisor : Dr. Yves Langlois
Institut de Chimie des Substances Naturelles, CNRS, Gif-sur-Yvette, France
« *Asymmetric cationic Diels-Alder reactions* »

1982-1986 Undergraduate student in chemistry, Université Paris-Sud, Orsay, France

Diploma

19 May 2000 Habilitation, Université Paris-Sud, Orsay, France
14 November 1989 Thesis, Université Paris-Sud, Orsay, France

Academic Career

2003- Professor of Chemistry, Université Paris-Sud, Orsay, France

1994-2003 CNRS, Chargé de recherches
Appointment : Laboratoire de Synthèse des Substances Naturelles, Université de Paris-Sud, Orsay, France
Supervisor : Professor Yves Langlois
« Asymmetric [3+2] cycloadditions of camphor-derived oxazolines and synthetic applications »

Research interests

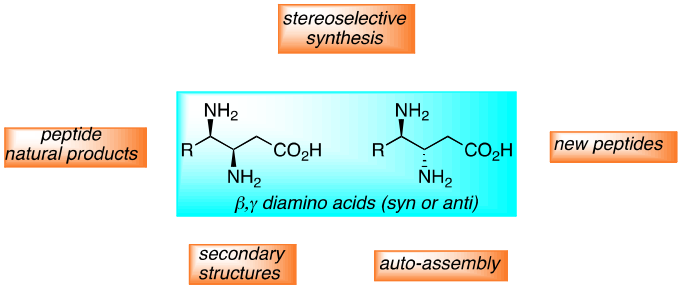
* Total synthesis of natural products (alkaloids)
* Aminoacid chemistry: synthesis of new aminoacids and peptides
* Synthetic methodologies: cycloadditions and alkene hydroamination reactions

Synthesis of New Aminoacids: Applications in Peptide Synthesis

Prof. Cyrille Kouklovsky

Equipe Méthodologie, Synthèse et Molécules Thérapeutiques
Institut de Chimie Moléculaire et des Matériaux d'Orsay
Université de Paris-Sud, F-91405 Orsay, France

The quest for new amino acids in an ongoing challenge in organic and bioorganic synthesis. Non-proteinogenic amino acids can induce numerous modifications while incorporated in peptides: proteolytic stability, secondary structure, biological activity, etc.
In the recent years, we have introduced b,g-diamino acids as original, modular building blocks for peptide synthesis. The present lecture will present our recent developments in the chemistry of b,g-diamino acids, including their stereoselective synthesis and their application in peptide synthesis, with some exemples in secondary structure determination and peptide auto-assembly.



Project under the responsability of Dr. Valérie Alezra

References:

1. "Efficient synthesis of both diastereomers of β,γ-diamino acids from phenylalanine and tryptophan" Auberger, N.; Stanovych, A.; Thétiot-Laurent, S.; Guillot, R.; Kouklovsky, C.; Cote Des Combes, S.; Pacaud, C.; Devillers, I.; Alezra, V. *Amino Acids* **2016**, *48*, 2237-2242.
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3. "Original β,γ-Diamino Acid as an Inducer of a γ-Turn Mimic in Short Peptides " Thétiot-Laurent, S.; Bouillère, F.; Baltaze, J.-P.; Brisset, F.; Feytens, D.; Kouklovsky, C.; Miclet, E.; Alezra, V. *Org. Biomol. Chem.* **2012**, *10*, 9660-9663.
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Rôle du CNUDST en tant que Fournisseur d'Information Scientifique et Technique

Kalthoum AZABI

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Le Centre National Universitaire de Documentation Scientifique et Technique (CNUDST) a été créé par la loi n°78-59 du 29/12/1978 portant loi de finances pour la gestion 1979 et notamment son article 33, sous forme d'établissement public à caractère administratif relevant de la tutelle du Ministère de l'Enseignement Supérieur et de la recherche scientifique.
En tant que centre d'appui à la recherche, le CNUDST est le principal fournisseur d'Information Scientifique et Technique (IST) qu'il met à la disposition de la communauté scientifique du pays.

Missions :
Centre intégré d'information scientifique et technique, le CNUDST a pour mission essentielle de :

- fournir l'information et la documentation scientifique et technique notamment aux chercheurs, quel que soit leur domaine d'activité.
- Collecter, traiter et diffuser la production et les résultats de la recherche scientifique et du développement technologique entrepris en Tunisie ou portant sur la Tunisie.
- Permettre un accès convivial à un fonds documentaire couvrant une partie importante de la recherche scientifique et technologique à l'échelle mondiale.
- Abriter la bibliothèque virtuelle de la recherche.
- Pratiquer la veille documentaire.

Pour ce faire, des applications sous forme de bases et de banques de données ont été développées dans le but de :

- Valoriser les résultats des recherches et contribuer à la promotion de la production scientifique tunisienne en assurant sa visibilité sur le web.
- Assurer une veille documentaire permanente sur les ressources documentaires accessibles sur le web à titre gratuit ou à tarif préférentiel (programmes en faveur des pays en développement).
- Mettre à la disposition des chercheurs la littérature scientifique mondiale nécessaire à leur activité, par la conclusion de licences d'accès à des revues en texte intégral et des bases et banques de données. Dans ce cadre, le CNUDST met à la disposition de la communauté scientifique tunisienne des ressources électroniques en majorité en texte intégral. Ces ressources consistent en des abonnements à plus de 10.000 revues scientifiques ainsi que des bases de données bibliographiques et bibliométriques.

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- Navigation sur internet : outre la navigation sur Internet, les utilisateurs de la bibliothèque peuvent accéder aux ressources électroniques à partir des postes mis à leur disposition dans les locaux du CNUDST.
- Reproduction de documents : photocopie, impression, numérisation
- Commande de documents à l'étranger : Le CNUDST met à la disposition du public un service payant pour répondre aux demandes d'articles, de thèses, de rapports de congrès publiés à l'étranger et non disponibles en Tunisie. Le délai de livraison dépend du type de documents demandés (articles, thèses etc.…) et de leur disponibilité
- Formations : Le CNUDST organise périodiquement au profit des professionnels de la documentation et des chercheurs des sessions de formation dans le domaine de la documentation, de la recherche bibliographique, de la publication des articles scientifiques, des études bibliométriques…soit grâce à ses moyens propres ou en collaboration avec des éditeurs internationaux.

Azaiez BEN AKACHA

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Adress : Département de chimie, Faculté des sciences de Tunis
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University corse:

June 1979 : Maitrise de chimie (Faculty of sciences of Tunis)
March 1981 : DEA of organic chemistry (Faculty of sciences of Tunis)
April 1987 : Thesis of organic chemistry (Faculty of sciences of Tunis)
February 2004 : Habilitation Universitaire in chemistry (Faculty of sciences of Tunis)

Professionnel carreer:

October 1980 : Assistant délégué - ENIG-Gabès
March 1981 : Assistant - ENIG-Gabès
October 1982 : Assistant- Faculté of sciences of Tunis
January 1988 : Maître-Assistant - Faculty of sciences of Tunis
September 2004 : Maître de Conférences - Faculty of sciences of Tunis
December 2010 : Professor - Faculty of sciences of Tunis

Research activity: Superviser (Master and Thesis) in « Laboratory of Selective Organic and Heterocyclic Synthesis-Biological Activity Evaluation » Chemistry department, Faculty of sciences of Tunis - University Tunis El Manar.

Research theme:

- Organophosphorus chemistry : Chlorophosphine, allenic, hydrazone, hydrazid, thiosemicarbazone, cyanomethylphosphonate, α-ketophosphonate, β-ketophosphonate, amide, thioamide, amidine, pyrazole, diazaphosphole, diazaphospholine, 1,3,2-dioxaphosphorinane, cyclophosphamide, oxazoline, thiazoline, indole…..
- Synthesis of heterocyclic molecules based on Sulfur, Nitrogen and Phosphorus.
- Complexes synthesis by organophosphorus reagents.
- Mechanisms By Phosphorus-31 NMR, structure by mulinuclear NMR, DFT, X-rays.
- Biological evaluation of organophosphorus compounds.
- Contribution to the international effort to fight cancer and AIDS through the analysis of some phosphorus nitrogen compounds (National Cancer Institute (NCI) - Bethesda-Maryland-USA).

Scientific production:

- Thesis : 6
- Masters: 12
- Communications: 32
- Publications : 35
- Review : Phosphorylated pyrazoles (166 references) by Azaiez Ben Akacha: Manuscrit at the Faculty of Sciences of Tunis, 1989.
- Précis de synthèse de quelques réactifs organophosphorés utilisés en synthèse organique by Salim Barkallah and Azaiez Ben Akacha, Manuscrit at the Faculty of Sciences of Tunis, 1992.

CHLOROPHOSPHITES: PREPARATIONS, CONFORMATIONALLY STUDY BY (^{31}P, ^{13}C, ^{1}H NMR, DFT, X-RAYS) AND PRECURSORS SYNTHESIS OF HETEROCYCLIC ORGANOPHOSPHORUS COMPOUNDS

Pr. Azaiez Ben Akacha

Laboratory of Selective Organic and Heterocyclic Synthesis-Biological Activity Evaluation
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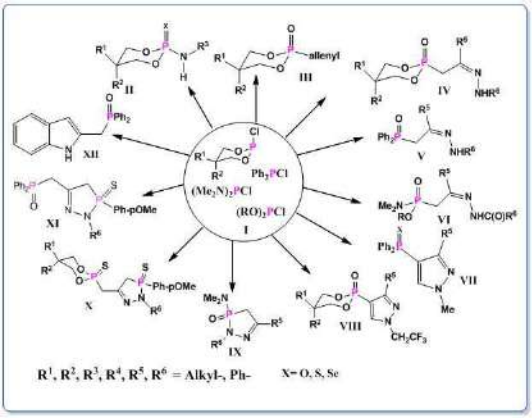
2092 -Tunis-Tunisia

Organophosphorus compounds have important properties as Anticancer (cyclophosphamide, isophosphamide) antibiotic (Phosphonomycin), Paget's disease (bisphosphonates: sodium aldronat, etidronat), Corrosion inhibitors (phosphonates, phosphites), fertilizers (phosphates), insecticides, flamme retardant, asymetric synthesis precursors, catalysis and Wittig olefination [1] etc.

Since its establishment in 1970, our laboratory has specialized in the synthesis of heterocyclic molecules containing Nitrogen, Oxygen, Sulfur and Phosphorus elements [2-6]. The theme of this conference is preparing chlorophosphines (**I**) and their applications in the synthesis of phosphorus products. Indeed, we have prepared a serie of chlorophosphine and studied stereoisomerism by multinuclear NMR (^{31}P,^{1}H,^{13}C) and DFT. Chlorophosphines are synthetic precursor to phosphorus compounds (**II-XII-Scheme-1**). The detection and evolution of intermediates of chemical reactions involved are followed by NMR ^{31}P spectroscopy.



Scheme 1: Organophosphorus compounds synthesized

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Philippe C. GROS

Philippe C. Gros studied chemistry at the University of Lyon and obtained his Ph.D in 1992 (Dr P. Le Perchec). He then worked for two years as a postdoctoral fellow on an Isochem-CNRS program on phosgene derivative chemistry. In 1994 he entered the CNRS as Researcher in the Laboratory of Pr. Paul Caubère at the Nancy University. He received his Habilitation (HDR) in 2000 and has been appointed Research Director in 2006. His is now first class Research Director and Head of the CNRS UMR SRSMC in Nancy. He is the co-author of 105 papers in international journals (h-index 25). His current research interests include the design of new metallating agents (organolithiums, ate-complexes) for functionalization of heterocycles, structure and reaction mechanism investigations, transition metal-catalyzed cross-couplings for ligand synthesis, and the design of photo- and electroactive organic and organometallic materials for various applications including solar energy.

FROM RUTHENIUM TO IRON SENSITIZERS: A CHALLENGE TOWARDS MORE ECOFRIENDLY HYBRID SOLAR CELLS

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The interest in organometallic complexes for optical applications is growing continuously. The development of such applications implies the careful design of metal complexes with appropriate photophysical properties to ensure efficient light harvesting based on strong and broadband molecular absorption or exciton generation in the condensed phase and excited state charge or energy transfer. Ruthenium polypyridine complexes have long been considered as lead compounds due to their ideal photophysical and geometrical properties. And used with success in Dye-sensitized Solar Cells (DSSCs) with efficiencies in the 9-12 % range. Our group has reported several new ruthenium complexes with improved absorption domains thanks to ligand tuning.[1] While ruthenium-based complexes have been widely investigated and used in many different lab scale applications it is a scarce metal. In contrast iron, belonging to the same group of the periodic table, is naturally abundant, of low cost and low toxicity and thus appears as an ideal substitute.

However, the replacement of ruthenium by iron is extremely challenging since in Fe-pyridine complexes an ultrafast non-radiative deactivation of the ^{1,3}MLCT states into the low-energy metal-centered quintuplet $^5T_2$ make Fe-pyridine unexploitable for applications requiring higher free energies.

Our group has reported new carbene-based ligands for the stabilization of the ^{3}MLCT state in iron complexes. We have shown that several of our iron complexes can sensitize the TiO_2 semiconductor in a laboratory DSSC, leading to measurable photocurrent and power conversion efficiency.[2] We have obtained the longest ^{3}MLCT state ever reported for iron(II) complexes.[3,4]

The conference will present our works on the preparation of ruthenium and iron complexes with focus on the chemical tuning of electronic and photophysical properties as well as their applications in DSSCs.

Key words: ligands, sensitizers, ruthenium, iron, excited states, solar cells

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Actual position: Directeur de Recherche CNRS at UMR CNRS 6283
Director of Laboratoire Physique de l'Etat Condensé UMR CNRS 6807 (01-01-2008 31-12-2011)
Director of UMR CNRS 6283 since 01/01/2012 (resulting from the aggregation of 4 ex-UMRs)
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PhD : 1987 Le Mans France
Alexandre von Humboldt Fellow in Karlsruhe (Germany) 1988-1989
Invited Professor at Santiago de Compostela (Spain) 1992

Key-words: amorphous metallic alloys, nanocrystalline alloys, magnetic nanoparticles, amorphous and crystalline fluorides, nanostructured milled and alloyed powders, functionalized nanoparticles, mesoporous structures magnetic nanostructures, magnetic frustration, non colinear magnetism, surface phenomena, interface phenomena, grain boundaries, core shell architectures, MOF, redox phenomena, physical biogeochemistry 57Fe Mössbauer spectrometry, numeric simulation

Publications: ~ 500 papers (~ 9000 citations and H = 43 WoS, H=51 Google Scholar, H=45 Scopus)

102 Invited conferences at International Conferences and Schools and National Events

French Representative at the International Board on Applications of Mössbauer Effect since 1998
Secretary of the International Board on Applications of Mössbauer Effect 2002-2007
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Referee: Phys. Rev. B, Phys. Rev. Letters, J. Appl. Physics, J. Appl. Lett., J. Phys.: Condensed Matter, J. Phys. D: Applied Physics, J. Magn. Magn. Mater., Hyper. Inter., Material Science and Engineering, Materiala Scripta, Eur. J. Phys. AP, Solid State Science, Corrosion Science, Physica B, European Journal of Mineralogy, J. Alloys Compds, Chem. Mater., ...

French Responsible of Bilateral Agreements: Picasso (2) Spain; Procope (1) Germany; Stefanik (1) Slovak Republik; CNRSPAS (6) Poland; CNRS-SAS (3) Slovak Republic; CNRS-Ireland (2); CNRS-RAS (2) Romania; ECOS NORTE (3) Colombia; CNRSKOCEF (1) South Korea; CMEP Algeria (1); CMEP Morocco (1)

French Responsible of NATO projects (2) and partner of FUNMIG (EU 6thPCRDT)
Partner of 5 ANR projects (Research French National Agency) and 2 Regional Projects
Member of scientific organizing committees of national and international conferences
Organizer of 2 Maghrebin Schools on Nanomagnetism and NanoMaterials
Organizer of 2 French Speaking Mössbauer Group Workshops
Organizer of 4 Schools devoted to Mössbauer spectrometry and its applications
Member of the Editorial Board of Hyperfine Interactions, Mössbauer Effect Reference Data Journal
Editor of Journal of Alloys and Compounds, Nanomagnetism, Hyperfine Interactions

Teaching duties: Magnetism, Nanomaterials and nanomagnetism in French/English Masters Le Mans, Nantes, Poitiers but also in Algeria, Marocco, Cameroon and Lebanon, and in Spanish: Santiago de Compostela and Bilbao (Spain), Tunja, Cali, Medellin, Bucaramanga, Barranquilla (Colombia), Lima (Peru), UNAM Mexico (Mexico).

Mössbauer spectrometry as a local tool to investigate solid state physics and chemistry: Applications of Mössbauer spectrometry.

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Since its discovery about 60 years ago, Mössbauer effect has been intensively developed: more specifically, ^{57}Fe Mössbauer spectrometry has received great attention to study Fe containing materials, because of their wide field of applications. Indeed, the high sensitivity to the electronic and magnetic environment of probe nuclei makes this technique very attractive and suitable in materials science, in addition to its non-destructive and non-invasive character.

After some preliminaries on the Mössbauer effect, the origin of the different hyperfine parameters and their physical meaning, we report some advances and trends in instrumental and experimental current facilities.

Some remarks have to be added on the fitting procedures of the hyperfine structures observed from zero-field and in-field Mössbauer spectra: this point is crucial and remains a difficult task in most of studies. Indeed, different codes are currently able to facilitate *a priori* the modelling of the hyperfine structures, but one should emphasize some misinterpretations due to texture effects, broadened and overlapped lines and mixture of static and dynamic effects.

Different examples concerned will then illustrate how ^{57}Fe Mössbauer spectrometry is a relevant local probe technique in solid state chemistry and physics, covering thus many topics. We first review its contribution in inorganic and organic chemistry to distinguish valency states, spin states and coordination allowing transition phase dependent phenomena and electronic relaxation phenomena to be studied. Then, we report some selected examples in non-crystalline structures, including thus chemically disordered systems, topologically disordered structures as amorphous and nanocrystalline structures. Finally, we report how in-field ^{57}Fe Mössbauer spectrometry is relevant to investigate magnetic structures and dynamical superparamagnetic phenomena in different magnetic nanoarchitectures such as magnetic nanoparticles, functionalized nanoparticles, nanostructured powders and hollow nanoparticles.

Salim BARKALLAH

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EDUCATION:

- **1990 Maitrise de chimie** : Faculte des Sciences de Tunis
- **1992 DEA de chimie Organique:** Faculte des Sciences de Tunis
  Supervisor: Prof. Belgacem Baccar
- **93/94: Teaching Assistant a l'ENIG** *: teaching general and Introductory organic chemistry*
- **94/95 Fulbright Scholarship** *:* University of North Caroliana at Chapel Hill-USA
  Worked on Spiroxyphosphrane in olifination chemistry. Studied the mechanism of first olefination reaction using spiroxyphosphoranes.
  Supervisor: Prof. Slayton Evans Jr
- **96-2000: Teaching Assistant** at Faculte des Sciences de Tunis et Bizerte : *teaching general and organic chemistry*- Research towards PhD
- **Janvier 2000: Doctorat en Chimie organique** : Faculte des sciences de Tunis,
  Supervisor: Prof. Hedi Zantour

POSTDOCTORAL EXPERIENCE

- ***September 2000- December 2001, Postdoctoral Fellow***
  Department of Chemistry, University of Pennsylvania-Philadelphia-USA
  Research advisor: Professor Marisa C. Kozlowski.
- ***January 2002- October 2003, Postdoctoral Fellow***
  Department of Biochemistry and Biophysics, Medicine school -University of Pennsylvania
  Research Adviser: Professor Dewey G. McCafferty

INDUSTRIAL EXPERIENCE 2003-Present

- Cambridge Isotope Laboratories- *Andover (Greater Boston) , MA –USA*
- **Position: Senior Chemist: 2003-2008**
- **Position: Senior Chemist II 2008- 2014**
- **Managerial position: Group Leader 2014- to present**
  Supervises a group of 3 chemists (Bachelor, Masters and PhD holders)

PUBLICATIONS

- ***Publications 14*** in the following Journals:
  J. Soc. Chim. Tunisie; J. Am, .Chem. .Soc.; Tetrahedron Letters; Bioorg. Med. Chem..; Biopolymers: Peptide Science; J. Soc. Alg. Chim; Phosphorus, Sulfur and Silicon, Acta Cryst.
- ***Patent 1: "LYSINE ISOTOPOLOGUES, COMPOSITIONS COMPRISING THE SAME AND METHODS OF SYNTHESIS" WO 2014/150909 Al; 25 September 2014 (25.09.2014)***
- ***Text Book 1: L'essentiel de l'isomérie et des mécanismes réactionnels***
  Published by CNUDST 2001- Editions CLE
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Stable Isotope Labeled Compounds: Applications in Organic Synthesis, Environmental Studies Medical Research, Diagnostics and Drug (Re)Discovery.

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In contrast to radioactive materials, stable isotope products are safe to use, manufacture and transport without risk or special regulations. A spectacular surge in the use and application of stable isotope labeled compounds in different areas of science has been noticed in the past two decades.

My presentation will describe examples of the use of stable isotope labeled compounds in a variety of applications pertaining to daily human life and well being.

In particular, I will highlight applications in organic chemistry where the isotope D, ^{13}C, ^{15}N and ^{18}O have been incorporated into organic molecules.

My talk will address the use of stable isotopes in five main applications: (1) determining reaction mechanisms and kinetics, (2) quantitation and analysis of contaminant and pollutants in the environment and food chain, (3) medical research areas of proteomics and metabolomics, (4) medical diagnostics and finally (5) new drug candidates.

Jean-Claude LEPRETRE

Full Prof UGA,

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Short CV. J.-C Leprêtre is 54, got his PhD (G. Cauquis Lab in Grenoble) in 2004, and worked for 12 years as maître de conferences in LEOPR Lab at the University J. Fourier of Grenoble. After he got his HDR in 2004 in A. Deronzier's lab, he was recruited as full professor in 2005 in the LEPMI (G-INP) in Grenoble. After two years as assistant director, he became director since 2015.
J.-C. Leprêtre has published more than 60 papers in peer reviewed journals (H Index 20,), and participated to more than to 30 conferences in international congresses. Due to its researches he has few patents at the national and international levels.

Scientific background in the context of the project
Key words*: electrochemistry, chemical electrical storage and conversion, organic and inorganic chemistry,*
J.-C. Lepretre's lab (LEMPI) has a recognized expertise in electrochemistry, particularly in view to study and elaborate electrochemical devices dedicated to the conversion and storage of electrical energy. The systems studied concern particularly lithium batteries and PEMFC focusing on the redox mechanisms involved in view to identify the overall redox mechanism and/or to synthesize a better material exhibiting higher performances (durability, capacity…). In this context, Pr J.-C. Lepretre developed new organic based materials. The first ones involve elaboration of new materials in order to reach higher performances. This concerns separators (D. Karabelli *et al* Electrochimica Acta 169 (2015) 32–36 ; C. Iojoiu *et al* Fuell Cell, 10(5), (2010) 778-789), electrode interfaces (F. Alloin *et al* Electrochimica Acta 112(2013), 74-81) and electrolyte (M. Bolloli *et al* Electrochimica Acta 169 (2015) 159-170 ; E. Perricone et al, Journal of Power Sources, 239 (2013), 217-224). The second aspect is dedicated to the multi spectroscopic investigation in view to precisely determine the nature of the species formed during the electrochemical process as illustrated by works dedicated to lithium/sulfur battery (S. Wallus *et al* Advanced Energy Material 5 (16), (2015) 1500165; Barchaz *et al*, Journal of Power Sources 199 (2012), 322-330). The third aspect concerns new organic materials which can be involved as organic cathode materials. In this context, new phenothiazine based polymeric material have been developed exhibiting competitive performances compared to classical cathodic inorganic material (Godet Bar et al Physical Chemistry Chemical Physics 17 (2015) 25283-25296).. Other materials are actually investigated involving coordination materials. First results show that this kind of material can be involved in cathode battery without any additive (binder and carbon)

The crucial role of organic materials towards the development of lithium battery

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Nowadays, lithium battery (polymer and ion) exhibit capacity and durability which have to be improved particularly for high energy requiring systems (electrical car). The improvement/increase of theses capacities can be reached by using high potential materials. However, this strategy requires organic materials (electrolytes) exhibiting both chemical and electrochemical stability while preserving their high ionic conductivity.

In this field, we elaborated original polymer electrolytes displaying high electrochemical stability whereas the controlled presence of an ionic function (sulfonate one) allows to maintain a relative high unipolar ionic conductivity (t^+=1). This can be obtained by using copolymer whose monomers have been selected to get material combining mechanical strength, conductivity and chemical stability.

Another alternative involving organic materials is based on the control of the interface between electrode material and the electrolyte (liquid) by the formation of a solid electrolyte interface (S.E.I). In this context, rather than using sacrificial compounds (vinyl carbonate), we investigated the use of diazonium salts. It is well known that such compounds through an electrochemical process (reduction) can lead to a covalently fixed films onto the surface of numerous materials (whose thickness can be easily controlled). In this context using diazonium salts functionalized by different lithium solvating groups, we achieved to efficiently protect the electrolyte towards the electrode reactivity allowing considering aqueous electrolyte.

The organic chemistry can also help to elaborate redox battery material. Contrary to inorganic ones, these materials exhibit numerous advantages i) a lower toxicity (compared to Mn…), a lower cost (compared to strategic metal as Co), these material are not specific to a cation (the more abundant Na^+ can be used rather than Li^+), their functioning potential can be tuned by to a adapted chemical modification.

In this context, we recently developed new organic polymeric materials base of phenothiazine. Their performances which compete with inorganic materials in terms of capacity and durability, only slightly depends on the nature of the cation of the electrolyte (Na^+ vs Li^+).

Nabil KHELIFI

Dr. Nabil Khélifi holds a BSc in Natural Sciences and a MSc in Earth & Environmental Sciences from the University of Sfax in Tunisia (2004). He received fellowships from the global change System for Analysis, Research and Training (START) in 2005 and the German Academic Exchange Service (DAAD) from 2006 to 2010 to continue with his PhD studies in Marine Geosciences at the University of Kiel in Germany.

After his PhD in 2010, Dr. Khélifi received a postdoctoral research grant from the German Science Foundation (DFG) to start his self-designed research projects at the GEOMAR Ocean Research Centre in Kiel, Germany on reconstructing past changes in oceanography and climate in the North Atlantic and the Mediterranean Sea using marine sediment samples retrieved by the International Ocean Drilling Program (IODP) and applying for a miniferal and geochemical proxy methods. He presented his research findings at many international conferences and published in highly ranked scientific journals. Dr. Khélifi also received funding from the European Science Foundation (ESF) and some European universities to co-organize two workshops on Pliocene climate in Bordeaux, France (2009) and Bristol, UK (2013). He also received the Swiss Government Excellence Scholarship (SGES) to continue with his research projects at ETH Zurich, Switzerland in early 2014.

Although his interest in scientific research remains strong, Dr. Khélifi decided in March 2014 to pursue his career as a Publishing Editor with Springer in Heidelberg, Germany. He is mainly responsible for developing the Springer's publishing program in the Middle East & North Africa (MENA). The program currently consists of developing 15 peer reviewed journals and publishing about 40 scientific books per year. Dr. Khélifi also helps researchers in MENA countries publish their work by delivering educational seminars for authors, reviewers, and journal editors to help improve publication output and quality. Dr. Khélifi is also a visiting lecturer at the University of Carthage, Tunisia and King Saud University, KSA giving MSc and PhD courses in geo-communication/-presentations and techniques for paper publishing, as well as career development training and professional development/soft skills workshops.

How to get your paper published in a scientific journal: Tips from a Springer Editor

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Preparing a research paper for publication in an academic journal is not an easy task and also very competitive. After producing data and generating ideas from your research, how do you write a clear and concise paper that attracts the attention of journal editors? How should you prepare a cover letter? How should you respond to reviewer report? We share here with early career scientists our advice and knowledge on how to effectively write and structure a paper, prepare a cover page, select the appropriate journal, and answer to reviewer comments.

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I. Saidi, P. Waffo-Téguo, H. Ben Jannet *FSM - Monastir* A new iridoid and derivatives isolated from *Citharexylum spinosum* L. growing in Tunisia	**OC41A**
M. Saidi, N. Bellakhal *INSAT - Tunis* Evaluation of degradation kinetics of NBD-Cl removal from water by electro-fenton treatment	**OC44C**
W. Sassi, L. Dhouibi, P. Berçot, M. Rezrazi *IPEIG - Gafsa* Electroplating of hybrid and nanocomposite Ni-W alloys as a coating againt copper corrosion used into automobile industries	**OC45C**
N. Sbei, B. Batanero, F. Barba, B. Haouas, L. Fuentes, M.L. Benkhoud *FST - Tunis* A convenient synthesis of new biological active 5-imino-4-thioxo-2-imidazolidinones involving acetonitrile electrogenerated base	**OC29A**
H. Sehimi, M.F. Zid *FSG - Gabès* A new 2-D Mn(II) coordination polymer : $(C_5H_8N_3)_2[Mn_2(C_2O_4)_3].3H_2O$	**OC55B**
M.Z. Slimani, C.N. Likos, A.J. Moreno *Chlef University, Algeria* Novel Scenario of Glass Formation in Polymeric Materials	**OC11C**
S. Soudani, C. Jelsch, C. Ben Nasr *FSB - Bizerte* Structural, Hirshfeld surface and spectroscopic studies of the noncentrosymmetric 1-ethylpiperazinediium pentachloroantimonate (III) monohydrate	**OC56B**
I. Talbi, C. Alayrac, J.F. Lohier, S. Touil, B. Witulski *FSB - Bizerte* From ynamides to organic electronics : A New Synthesis of 2-(Tosylamido)- and 2,5-Bis-(tosylamido)-thiophenes	**OC30A**
F. Taleb, M. Ben Mosbah, E. Elaloui, Y. Moussaoui *FSG - Gabès* Thermal and spectroscopic analysis of lignin from coffee spent	**OC42A**
H. Talmoudi, A. Haj Said, N. Khenoussi, L. Schacher, A. Dominique *FSM - Monastir* Preparation and characterization of nanofibers based MOF-5 and HKUST-1	**OC10C**

Name	Ref
A. Toumi, S. Boudriga, S. Haddad, M. Askri *FSM - Monastir* An efficient synthesis of novel dispiroindolizidine derivatives *via* one-pot three-component 1,3-dipolar cycloaddition reaction	**OC31A**
S. Trabelsi, H. Marouani *FSB - Bizerte* Crystal structure, Hirshfeld surface analysis and characterization of a new hybrid dichromate compound $[C_9H_{14}N]_2Cr_2O_7$	**OC57B**
S. Trabelsi Souissi, N. Souissi, N. Bellakhal *IPEIM - Tunis* Caracterisation of corrosion products of steel E235 used in shock absorber industry in Tunisia	**OC46C**
M. Zaghdoudi, S. Hammami, F. Fourcade, D. Floner, H. Maghraoui Meherzi, A. Amrane, F. Geneste *FST - Tunis* Direct electrochemical reduction of dimetridazole as pretreatment to enhance biodegradability	**OC47C**
M. Zannen, M. Dietze, H. Khemakhem, M. Es-Souni *FSS - Sfax* Toward Opto-electronic $Bi_{0.5}Na_{0.5}TiO_3$ Lead-free thin films	**OC48C**
R. Zarrougui *INRAP - Sidi Thabet* Highly efficient and eco-friendly extraction of neodymium using, undiluted and non-fluorinated ionic liquids. Direct electrochemical metal separation	**OC49C**
H. Zouaoui, M. Krichen, J. Bouaziz *FSS - Sfax* Structure, microstructure and mechanical changes of ceramic products of clay and non plastic mineral mixtures	**OC58B**
A. Zrelli, B. Chaouachi *ISSAT - Gabès* Microfiltration flat sheet membranes from waste polymer: Synthesis and characterization	**OC12C**

Oral Communications' Abstracts

Program of
Monday 19
December 2016

NEW FLUORENYL PHTHALOCYANINES FOR OPTICS

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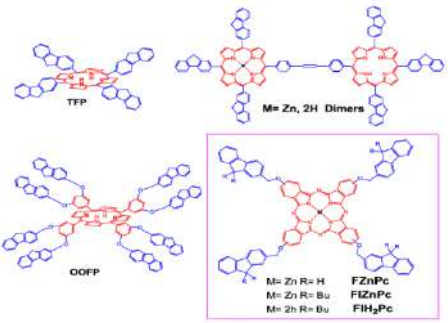
In 2004, we have reported the synthesis of porphyrin possessing four fluorenyl arms **TFP**.[1-5] Surprisingly, **TFP** exhibited a remarkably high quantum yield (24%), compared to the reference tetraphenylporphyrin demonstrating the capacity of the fluorenyl units to enhance quantum yields. Then, to exploite this efficiency, a series of porphyrin dendrimers bearing fluorenyl dendrons was prepared: new porphyrin **OOFP** possessing eighth fluorenyl pendant arms, [6] and the **SOFP** possessing sixteen fluorenyl arms.[6, 7]
As an application, we tested the corresponding platinum (II) complexes for the fabrication of red Organic Light Emitting Diodes (OLEDs).[8-10] Also, supramolecular assemblies possessing **12** or **24 fluorenyl arms**, using these efficient building blocks have been obtained by our group.[11]
Very recently, we synthesised smaller units possessing 6 fluorenyl arms like **dimers**,[10] as well as **trimers**. Encouraged by these results, we recently decided to change the acceptor core macrocycle and to study this effect of the in luminescence. So, a new series of symmetrical **phthalocyanines (Pc)** possessing four fluorenyl arms was prepared and studied. It was found that the excitation energy transfer occurs from the four fluorenyl units to the phthalocyanine core, following what the compounds emit intense red light exhibiting a high quantum yield (42% for **FlZnPc**).



Key words: phthalocyanine, porphyrin, fluorenyl, dendrimers, dimers, trimers, red emission, energy transfer

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AN APPROPRIATE ONE-POT SYNTHESIS OF PYRANO[3, 2 C] CHROMENE DERIVATIVES CATALYZED BY AMMONIUM ACETATE: CHARACTERIZATION, ANTIOXIDANT, ANTIBACTERIAL AND ANTI-INFLAMMATORY ACTIVITIES

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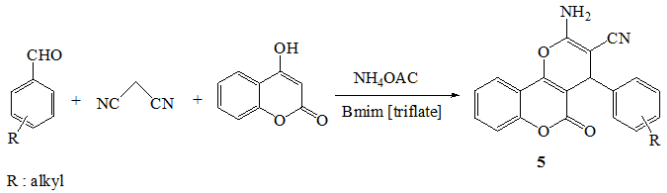
Multicomponent reactions (MCRs) are widely used to produce elaborate biologically active compounds. MCRs, particularly those performed in green and eco-friendly media offered significant advantages over conventional linear-type syntheses by virtue of their convergence, productivity, facile execution and high yield. herein, we aim to develop a simple, efficient and eco-friendly procedure for the synthesis of , pyrano[3,2-c]chromene derivatives by using a one-pot three component condensation of 4-Hydroxycoumarin, various arylaldehydes and malonitrile in 1-butyl-3-methylimidazolium triflate as green media is described[1]. The obtained compounds **5** were investigated for antioxidant activities by super oxide radical and DPPH (2,2-Diphenyl-1-picrylhydrazyl)[2]. Furthermore**,** compounds 5 were evaluated for anti-inflammatory activity by indirect haemolytic and lipoxygenase inhibition assays. Most of the new compounds 5 exhibited moderate antibacterial activity.



Scheme: Synthesis of pyrano[3,2-c]chromene derivatives **5**.

Keywords: Multicomponent reaction; 4-Hydroxycoumarin; pyrano[3,2 c]chromene; Ammonium acetate; antibacterial and anti-inflammatory activities.

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A CONVENIENT PROCEDURE FOR THE SYNTHESIS OF ENANTIOPURE SCHIFF BASES DERIVATIVES: THEIR PHYSICAL, CHEMICAL AND BIOLOGICAL PROPERTIES

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An efficacious and environmentally friendly procedure for the synthesis of Schiff bases has been described. These new chiral imines have been synthesized by the condensation of 2-hydroxynaphthaldehyde with chiral α-aminoacid phenylhydrazides derivatives using three different methods with a comparative study of their yields. These compounds have been confirmed by crystallographic, spectroscopic and spectrometric analysis. The crystallographic data reveals that these molecules adopt the canonical zwitterionic form. The synthesized compounds have been screened for their antibacterial activity on several species of pathogenic bacteria. The physical properties under room temperature indicated that all the ligands are considered as organic semiconductors with available gap energy.

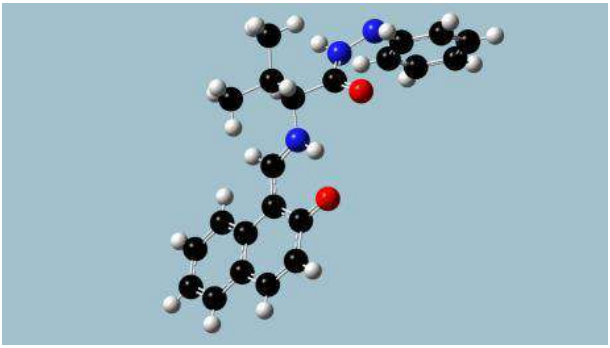

Fig. X-ray diffraction rays of the Schiff base

Key words: Schiff bases, ligands, condensation, α-aminoacid phenylhydrazides, organic semiconductors, gap energy.

ESTERIFICATION OF FATTY ACIDS WITH POLYOLS IN THE PRESENCE OF ACID CLAYS

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Esters of alcohols and long-chained carboxylic or "fatty" acids have a variety of applications in industry such as in food and cosmetic emulsifiers and lubricants, in flavors and fragrances, and as components of/or intermediates for food products (e.g. cooking oils). Polyol-fatty acid esters have strong implications in such industries as foods, cosmetics, and polymers.

Heterogeneous catalysts based on clays have received considerable attention in different chemical processes due to their environmental compatibility, low cost, selectivity, thermal stability and recyclability [1]. They are one of the most widely studied solid acid catalysts for many organic transformations, under milder reaction, especially in esterification reaction [2].

In this work, it is proposed to study the esterification of polyols by some typical fatty acids such as fatty acids in the presence of clays as catalysts. The catalytic role of each major physico-chemical properties of these clays, for instance specific surface area, porosity, surface acidity was studied. The best operating conditions were optimized through a development of experiments which take into account the amount of catalyst, the reaction temperature and the molar ratio.

Keywords: Esterification, heterogeneous catalyst, fatty acids, polyols, polyesters.

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FACILE ROUTE TO γ-LACTAMS FROM ALLYL AMINES CATALYZED BY NICKEL BORIDE

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γ-lactams are important heterocyclic motifs found in various biologically active compounds and marketed drugs. Among available methods for the synthesis of γ-Lactams, the intramolecular substitution of an amine at a carbonyl group is one of the widely utilized approaches. By reducing a nitro moiety to a primary amine which substitute immediately anethyl carbonated moiety in δ position we have been able to synthesize γ-Lactams with a good yield. Even though, Nickel Boride is a very important catalyst agent, few approaches of using it in the synthesis of γ-Lactams have been reported so far. In this work γ-Lactams were synthesized through two different reducing agents catalyzed with Nickel Boride in simple and facile experimental conditions.
The structures of all the prepared compounds were confirmed by ^{1}H, ^{13}C NMR spectral data.

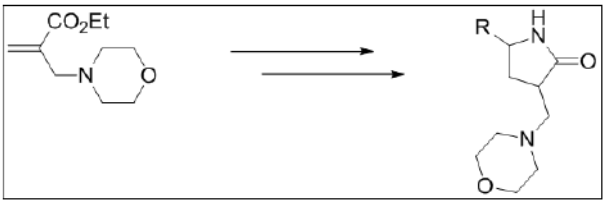

Fig: γ-Lactam synthesis from allyl amine

Key words: γ-Lactams, Nickel Boride, nitro compounds, allyl amine.

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DESIGN AND SYNTHESIS OF TRIAZOLE-LINKED COUMARINOPYRAZOLE CONJUGATES

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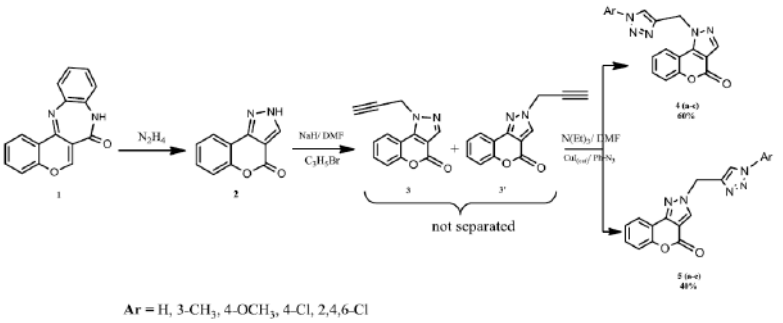
Owing to the fact that the presence in tandem of two linked privileged heterocycles within a same framework could contribute to provide a significant positive effect on the overall biological efficiency in the resulting molecule,[1] we have designed and synthesized a series of novel hybrids derivatives (**4** and **5**) containing a 1,2,3-triazole nucleus linked to a coumarinopyrazole moiety. Thus and starting from a useful key intermediate the benzopyranobenzodiazepine 1,[2] a two steps reaction led to a mixture of regioisomeric *N*-propargyled species 3 and 3'. The so obtained mixture of both dipolarophiles was treated by mean of an alkyne/azide cycloaddition type "click" chemistry protocol giving rise to the corresponding cycloadducts **4a-e** and **5a-e**. All the compounds structures were elucitated on the basis of ^{1}H, ^{13}C and 2D-NMR techniques and HRMS analysis.



Keywords: coumarinopyrazole, triazole, *1,3*-dipolar cycloaddition, click chemistry.

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MICROWAVE ASSISTED SYNTHESIS OF NEW BENZOCHROMENOPYRIMIDINONE-1,2,3-TRIAZOLES HYBRIDS

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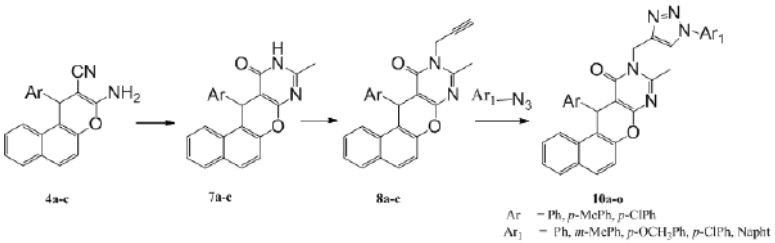
As our research is devoted to the synthesis of diverse heterocycles as anti-infective agents, we identified the chromene, pyrimidine and triazole (separately) as good antimicrobial,[1] anticoagulant[2] and antiviral agents.[3] Keeping this in view, we synthetized new prototypes using click chemistry by combining triazole, pyrimidine, pyran and synthesized hybrid molecules consisting of triazoles along with pyrimidines and pyran moieties, so we have employed some newly synthesized hybrid benzochromenopyrimidin-4(4*H*)-ones **10a-o** derived from 1,3-dipolar cycloaddition reaction of terminal alkynes **8a-c** with a sries of arylazides under classical conditions and microwave irradiation in the presence of copper(I) catalyst. Structures of all the prepared compounds were elucidated on the basis of extensive spectroscopic means including 1D and 2D-NMR.



Keywords: Pyrimidinone-triazole hybrids, Click Chemistry, Microwave Irradiation.

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ANTIMICROBIAL ACTIVITY OF HIGHLY FLUORINATED THIO(DITHIO)CARBAMATES

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Despite the large number of antibiotics available for the treatment of bacterial infections, antibacterial resistance continues to threaten global health systems [1,2]. This has triggered increasing research efforts to design and develop innovative antibacterial and antifungal agents that could overcome such antimicrobial resistance. Recently, we have described the synthesis of highly fluorinated thio(dithio)carbamates [3,4]. In this presentation, we will describe the evaluation of the *in vitro* antimicrobial activity of some of these fluoroalkyl thio(dithio)carbamates as well as their non fluorinated analogs against a panel of nine bacterial and three fungal strains. Thiocarbamate (**1i**) and dithiocarbamate (**2i**) showed both the lowest MIC values (3.9 µg/mL) and the widest spectrum of antibacterial activity (Fig. 1).

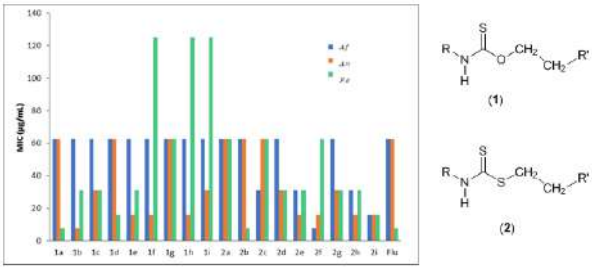

Fig. 1 Structures of thiocarbamates (**1**) and dithiocarbamates (**2**) and comparison of *in vitro* antifungal activity of thio(dithio)carbamates (**1** and **2)** with the control drug fluconazole (Flu) against three fungi after 72 h incubation.

Keywords: Thio(dithio)carbamates, fluoroalkyl, antimicrobial activity.

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SYNTHESIS, ANTICOAGULANT AND ANTI-α-GLUCOSIDASE ACTIVITIES OF NOVEL C-2 ARYLIDENE DERIVATIVES FROM THE NATURAL OLEANOLIC ACID

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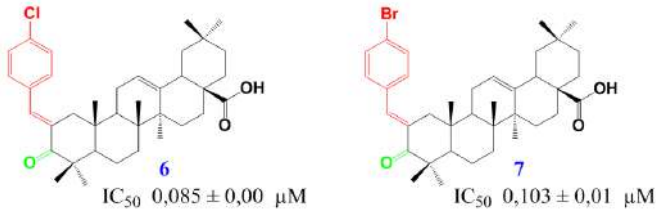
The optimization of synthesis conditions of novel C-2 arylidenes of the natural oleanolic acid isolated from *Olea europaea* L. [1] was described in an attempt to develop potent anticoagulant and anti-α-glucosidase agents. The Claisen-Schmidt reaction boric acid-catalyzed of oleanolic acid **1** with a series of arylaldehydes afforded regiospecifically the 2-arylidene-3-oxoolean-12-en-28-oic acids **3**–**12**. The reaction was assisted by microwave irradiation. The new products were obtained in almost quantitative yields. Their chemical structures were elucidated on the basis of extensive spectroscopic methods including ESI-HRMS, 1D and 2D-NMR. The *in vitro* anticoagulant activity of all the compounds was assessed by measuring the activated partial thromboplastin time (aPTT). Most the compounds displayed significant anticoagulant activities in a concentration-dependent manner. On the other hand, oleanolic acid **1** and their derivatives were screened for their ability to inhibit the α-glucosidase, an enzyme causing diabetes. Our findings showed that *2-(4-chlorobenzylidene)-3-oxoolean-12-en-28-oic acid* (**6**) and *2-(4-bromobenzylidene)-3-oxoolean-12-en-28-oic acid* (**7**) inhibited α-glucosidase in a non competitive manner with IC_{50} values of 0,085 ± 0,00 and 0,103 ± 0,01 µM, respectively.



Keywords: *Olea europaea* L., Oleanolic acid, C-2 arylidenes, Anticoagulant, Anti-α-glucosidase.

References

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Synthesis of new anticoagulant pyranopyrimidine derivatives from 2-amino-3-cyanopyrane

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Anis ROMDHANE and Hichem BEN JANNET

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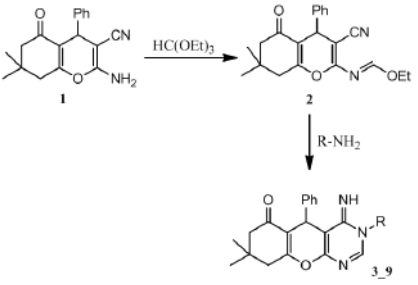
Heterocycles including the pyrimidine scaffold have often proved efficiency in different therapeutic areas as antibacterial[1], antiviral[2], antitumor[3], and anti-inflammatory[4]. On another hand, heterocycles incorporating the pyrane nucleus, are used extensively in medicinal chemistry. Referring to several reports claming that the fusion of a pyranopyrimidine ring to other heterocyclic scaffold could introduce modification of the biological properties of parent molecule. In this context, we were interested in the synthesis of novel pyranopyrimidine derivatives **3-9** *via* the condensation of 2-amino-3-cyanopyranes **1** with ethyl orthoformate affording the iminoether **2** which in turn reacts with a series of primary amines. in order to valorize biologically the target molecules **3-9**, we evaluated their anticoagulant activities. The structure-activity relationship has been also investigated.



R= Ph, 3-CH_3-Ph, 2-Cl-CH_2-Ph, 4-Et-Ph, 3-Cl-Ph, 4-OCH_3-Ph, Naphtyle

Keywords: pyrane, pyranopyrimidine, pyrimidine, 2-amino-3-cyanopyrane.

References

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Tetrahydropyranodiquinolin-8-amines as New Acetylcholinesterase Inhibitors for Alzheimer's Disease Therapy

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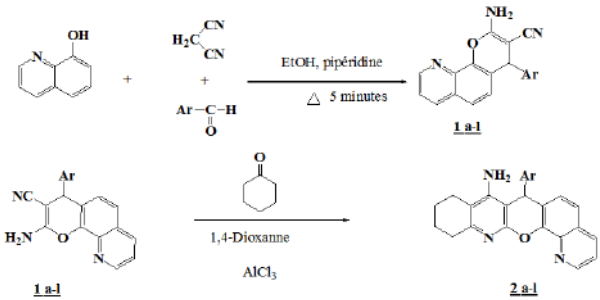
Alzheimer's disease (AD) is the most common neurodegenerative disorder, affecting round 35 million people worldwide. AD is characterized by progressive memory loss, decline in language skills and other cognitive impairments. Although the causes and mechanisms underlying the progress of AD are still not fully understood, there is a general consensus about the complexity of the disease. In the present work we report the straightforward two-step synthesis and biological assessment of novel Tetrahydropyranodiquinolin-8-amines as acetylcholinesterase inhibitors properties (Scheme1).



Scheme1

The biological evaluation of the compound **2**, identified compound -**2h**- (Ar= *m*-$OCH_3C_6H_4$) displayed a mixed-type inhibition of human acetylcholinesterase (IC_{50}= 0.75±0.01 μM).

Keywords: Alzheimer's disease; pipéridine; Tetrahydropyranodiquinolin-8-amines; cholinesterase inhibitors.

SYNTHESIS AND CHARACTERIZATION OF ORGANOCHALCOGENOPHOSPHORUS COMPOUNDS CONTAINING DIFFERENT CYCLIC AMINO GROUPS

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Organophosphorus compounds are well-known for their use as pesticides, insecticides and recently as prodrugs due to their activity as acetylcholinesterase inhibitors [1]. In particular, phosphine chalcogenides of the formula R_3PE (E = O, S, Se) are attracting considerable interest owing to their biological activity and selective affinity towards metal ions [2,3]. In this work, we will describe the synthesis of new organochalcogenophosphorus compounds bearing different cyclic amino groups (**L^3**, **L^4**, **L^7-L^{10}** and **L^{13}**, **L^{14}**). These phosphoramide derivatives were fully characterized by multinuclear (^{1}H, ^{13}C, ^{31}P and ^{77}Se) NMR and IR spectroscopic techniques. The use of these compounds as potential ligands towards different metal ions will also be discussed.

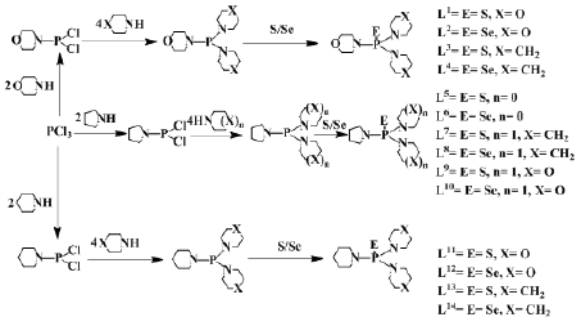

Fig. General pathway for the synthesis of different organophosphorus compounds.

Keywords: Organophosphorus, piperidinyl, morpholinyl, NMR.

References

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SYNTHESIS AND CHARACTERIZATION OF PHOSPHORAMIDES BEARING DIFFERENT CYCLIC AMINO GROUPS

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[b)] *Laboratory of structural organic chemistry: Synthesis and physicochemical studies, Coordination Chemistry group, Department of Chemistry, Faculty of Sciences of Tunis, El Manar University Campus I 2092, Tunis, Tunisia*

Phosphoramides are increasingly attracting considerable attention due their potential applications in industry as flame retardants, in medicine as prodrugs, in catalysis and organic synthesis as chiral ligands [1,2]. It was shown that these properties depend largely on the nature of substituents on the phosphorus atom [3]. In this work, we will report the synthesis of new phosphoramides containing different cyclic amino groups (**L^2**, **L^4**, **L^5**and **L^6**) (Fig. 1). These phosphoramides were fully characterized by multinuclear (^{1}H, ^{13}C and ^{31}P) NMR and IR spectroscopic techniques. The effect of the sequence of amine addition on the reaction yields as well as use of these compounds as potential ligands towards hard metal ions will also be discussed.

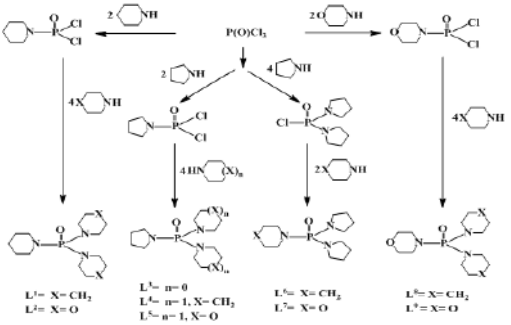

Fig. 1 General pathway for the synthesis of different phosphoramides.

Keywords: Phosphoramides, piperidinyl, morpholinyl, ^{31}P NMR.

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ARYLATION OF 3-ACETYL COUMARIN: COUPLING OF COUMARINIC TOSYLHYDRAZONE WITH ARYL HALIDES

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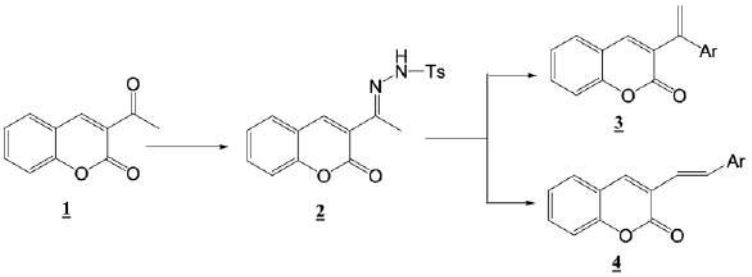
Palladium-catalyzed cross-coupling reactions are recognized as some of the most powerful and reliable methods for the formation of C-C bonds. In a broad sense, a cross-coupling reaction is the combination of an electrophile and a nucleophile in the presence of a transition-metal catalyst. In the last decade, a new class of palladium-catalyzed C-C bond-forming cross-coupling has come into play that features a different type of nucleophile and also a different mechanism. In these new reactions, the nucleophilic coupling partner is a diazo compound which is the tosylhydrazone. In our case, we have used a coumarinic tosylhydrazone **2** in the aim to obtain new arylated coumarin derivatives 3-(1-phenylvinyl)-2*H*-chromen-2-ones **3** and 3-styryl-2*H*-chromen-2-ones **4**. All the synthesized compounds were characterized by spectroscopic means such as 1D (^{1}H, ^{13}C) and MS.



Keywords: tosylhydrazone, cross-coupling, palladium, arylation, coumarin

UNPRECEDENTED 3-*O*-METHYL-3-*C*-TRIFLUOROMETHYL-D-RIBONO- (AND L-LYXONO)-γ-LACTONES SYNTHESIZED BY NUCLEOPHILIC TRIFLUOROMETHYLATION OF D-HEXOSE-DERIVED CYCLIC KETONES

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Université de Monastir, Laboratoire de Chimie Hétérocyclique, Produits Naturels et Réactivités, Faculté des Sciences de Monastir, Avenue de l'Environnement, 5000 Monastir, Tunisia

3-*O*-Methyl-3-*C*-trifluoromethyl-D-ribono-(and L-lyxono)-γ-lactones have been prepared from protected D-hexoses (gluco, galacto) by multi-step routes from D-glucose. The synthetic strategy includes the following steps: regioselective oxidation, nucleophilic trifluoromethylation [1] with the Ruppert-Prakash reagent of 3-keto hexofuranose derivatives attacked stereoselectively from the less hindered face, protective group manipulations, and regioselective oxidation of a hemiacetalic hydroxyl. Base-catalyzed hydrolysis of two related D-ribonolactones afforded 3-*O*-Me-3-*C*-CF_3-D-ribonic acid [2].

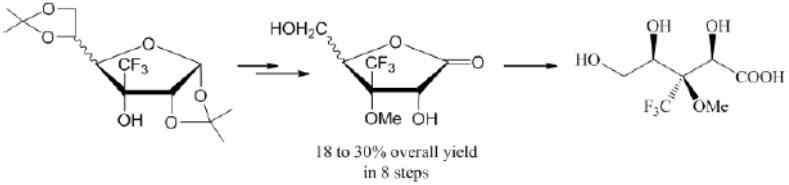

Fig. Synthesis of 3-*O*-Me-3-*C*-CF_3-D-ribonic acid

Key words: Nucleophilic trifluoromethylation/ Lactones/ Stereoselectivity.

References

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FACILE SYNTHESIS OF 2-METHYL-4-ARYL-1H-PYRROLE-3-CARBONITRILES BY CATHODIC REDUCTION OF ACETONITRILE

Kaouthar Hamrouni[b], Belen Batanero[a], Fructuoso Barba[a], Taieb Saied[b], Mohamed Lamine Benkhoud[b]

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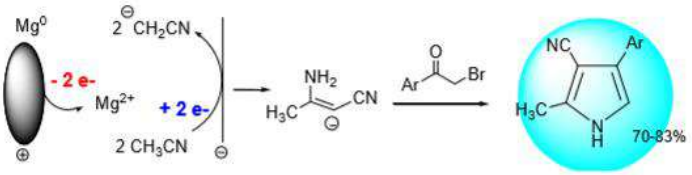
A new series of polyfunctionalized pyrroles has been obtained in very good yield when the product of reductive electrolysis of dry acetonitrile (3-aminocrotonitrile anion), is added to different phenacyl bromide solutions at low temperature under an argon atmosphere. The reactive anion is achieved by amperometric reduction of the solvent at stainless steel cathode and using a magnesium sacrificial anode.



Scheme: electrosynthesis of 2-methyl-4-aryl-1H-pyrrole-3-carbonitriles

Keywords: Electrogenerated anions, Magnesium sacrificial anode, Trisubstituted heterocycles, Phenacyl bromides

CRYSTAL STRUCTURE AND THERMAL BEHAVIOUR OF PIPERAZINEDIIUM TETRAAQUA BIS(SELENATO/SULFATO) CUPRATE(II) DIHYDRATE $(C_4H_{12}N_2)[Cu(S_{0.39}Se_{0.61}O_4)_2(H_2O)_4]\cdot 2H_2O$

Marwa ABID DERBEL, Walid REKIK, Houcine NAÏLI

Laboratoire physico-chimie de l'État Solide, Département de Chimie, Faculté des Sciences de Sfax, Université de Sfax, BP 1171, 3000 Sfax, Tunisie.

A new organic-inorganic hybrid material has been synthesized and cristallographically characterized by single-crystal X-ray diffraction. At room temperature, this new compound crystallizes in the monoclinic system (space group $P2_1/n$) with the following unit-cell parameters: a = 6.9783(2) Å, b = 11.7101(4) Å, c = 10.6238(3) Å, β = 104.431(2)° and Z = 2. Its crystal structure is built from anionic units $[Cu(S_{0.39}Se_{0.61}O_4)_2(H_2O)_4]^{2-}$, piperazinediium cations $(C_4H_{12}N_2)^{2+}$ and free water molecules. All these entities are linked together by two types of hydrogen bonds: Ow-H…O and N-H…O. The IR spectroscopy confirms not only the partial substitution of the sulfur by the selenium but also the piperazine protonation. The thermal decomposition of the precursor proceeds through four stages giving rise to the copper oxyselenate.

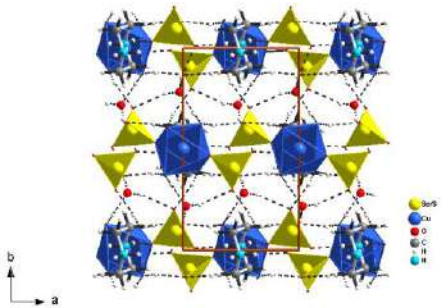


Fig. Projection of the structure of $(C_4H_{12}N_2)[Cu(S_{0.39}Se_{0.61}O_4)_2(H_2O)_4]\cdot 2H_2O$ along the crystallographic c-axis

Key words: Hybrid material, Crystal structure, IR spectroscopy, Thermal decomposition.

Mechanical properties and microstructure of AA2017

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Alloy 2017 is a hard aluminum alloy strengthened by heat treatment. Because of its higher strength, finer weldability and ductility, hard aluminum alloy 2017 has been widely used in the field of aeronautics and astronautics. Aluminum alloy 2017 was given solid solution treatments at 500°C for both times 2h and 6h followed by quenching in water and in air. It was then artificially aged at 175°C for 8h. Subsequently, hardness measurements, tensile tests were carried out on the heat treated alloys to determine the mechanical properties on samples with different orientation rolling direction, RD and transverse direction, TD. The results of solid solution treated at 500°C for 6h, quenching in water then artificially aged at 175°C for 8h indicate the highest yield and tensile strength. In general, the increase in strength was accompanied by a decrease in ductility. Fracture, on a microscopic scale, was predominantly ductile comprising micro-void nucleation, growth and coalescence. Besides, the analysis of the fracture surface of AA2017 is made by scanning electronic microscopy.
Effects of quenching in water and artificial aging on microstructures of AA2017 were characterized by optical microscopy (OM) and scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) techniques.
The effect of aging, on the mechanical properties of aluminum alloy, was investigated by mean of hardness, and tensile tests. The obtained results show that, the quenching rate after solution heat treatment, aging time and temperature, play a very important role in the precipitation-hardening such as Al_2Cu (θ') and the Al_2CuMg (S-phase) associated with aging process of the aluminum alloy 2017.

Keywords: AA2017, quenching, artificial aging, microstructure, mechanical properties

CRYSTAL STRUCTURE AND PHYSICOCHEMICAL PROPERTIES OF A NEW ORGANIC NITRATE SINGLE CRYSTAL $(C_7H_9N_2O)HNO_3$

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Crystals of 2-aminobenzamidium nitrate has been prepared and grown at room temperature. This compound crystallizes in the orthorhombic system with non-centrosymmetric $P2_12_12_1$ space group. The unit cell dimensions are a = 9.553(2) Å, b= 4.901(2) Å, c= 19.004(2) Å with V= 889.75(4) $Å^3$ and Z = 4.The structure has been solved using direct method and refined to a reliability R factor of 0.0434.

The structure of this compound was determined by using X-ray data collection on a suitable single-crystal. Fig.1 and Fig. 2 shows the atomic arrangement of the studied compound. In this arrangement, 2-aminobenzamidium cations $(C_7H_9N_2O^+)$ and nitrate anions $(NO_3)^-$ are interconnected by a system of hydrogen bonds building a three dimensional network.

Also, the UV-visible, thermal behavior and the IR spectroscopic studies of this new compound are discussed.

Key words: Hybrid; X-ray diffraction; Crystal structure; infrared spectroscopy; Thermogravimetric analysis, optical properties.

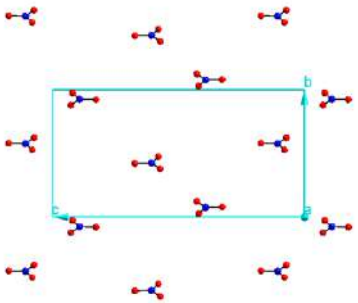

Fig.1. Projection of the anionic arrangement along the a-axis

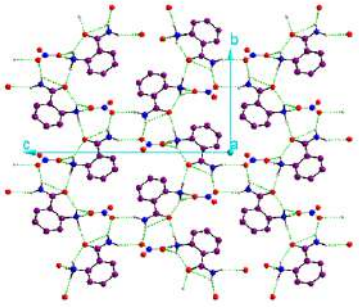

Fig.2. Projection along the a-axis of the atomic arrangement in $(C_7H_9N_2O)HNO_3$

SYNTHESIS AND PHYSICO-CHEMICAL CHARACTERIZATION OF A NEW OCTAMOLYBDATES $K_2(C_6H_{15}NO_3)_2[Mo_8O_{24}]$

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Polyoxometalates (POMs) as early-transition-metal oxide clusters have received much attention in various fields such as catalysis, ion exchange, electrochemistry, magnetism and medicine for their enormous variety of structures and unique properties **[1-4]**. Because of this importance, it was of interest to synthesize a new member of octamolybdates $K_2(C_6H_{15}NO_3)_2[Mo_8O_{24}]$ (1) and to investigate its structural characterization by single crystal X-ray diffraction, IR, UV and thermogravimetric analysis.
The title compound crystallizes in the triclinic system, space group P-1 with a = 9.826 (2) Å, b = 9.842(3) Å, c = 10.743(4) Å, α= 86.458(2)°, β= 71.167(3)°, γ = 89.294(5) °, Z = 11.

Keywords: polyoxometalate, octamolybdates polyanions, Crystal Structure.

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Structural and thermal characterization of nanocrystalline $Fe_{71}Nb_6B_{23}$ powder synthesized by mechanical alloying

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[b)] Laboratoire des matériaux céramiques et procédés associés, Université de Valenciennes (France)

Nano-based compounds have unique electrical, magnetic, optical, catalytic, mechanical or biological properties and received much attention in the recent years because of various applications [1-4]. In this paper, Structural and thermal characterization of nanocrystalline $Fe_{71}Nb_6B_{23}$ powder produced by mechanical alloying. Characterizations of the prepared powders were carried out by scanning electron microscopy (SEM), X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS) and laser granulometry. During the milling process, the powder particles are subjected to highly energetic compressive impact forces. The repeated fracturing and cold welding of the powder particles lead to reactions between solid components of the initial mixture. The results show the formation of several binary and ternary solid solutions as Nb(B), Fe_3B Fe(Nb, B) and bcc-Fe phase.

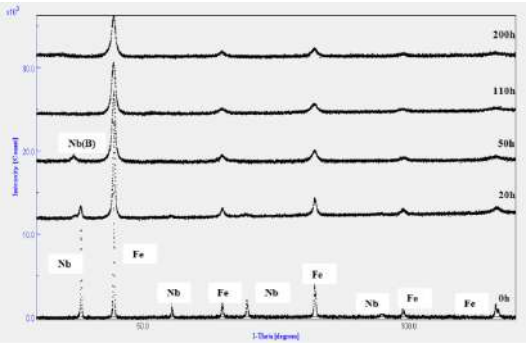

Fig. XRD patterns of the $Fe_{71}Nb_6B_{23}$ powders milled for various times.

Key words: Mechanical alloying, Nanocrystalline materials, Fe-Nb-B, DRX

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Synthesis and Physico-Chemical Characterizations of the Nanomaterial $Li_{2,8}Hf_2Si_{1,8}P_{1,2}O_{12}$

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The Nanomaterial $Li_{2,8}Hf_2Si_{1,8}P_{1,2}O_{12}$ was synthesized by sol-gel process. The formed white gel was dried at 100 °C in order to obtain the preliminary precursor which was then analyzed by coupled DTA-TGA (Figure1) to determine its crystallization temperature and as a result leading to the estimation of its calcined temperature. Furthermore, the calcined powder, obtained by heating the dehydrated precursor at temperature higher than its crystallization temperature, has been characterized by other physicochemical techniques: XRD, CIS and IR-FT. The XRD analysis (Figure 2) shows the obtaining of only the single phase without any detection of other phases and the unit cell parameters have been refined in the rhomboedric system with sapce group $R\bar{3}c$: a = b = 8.849(3) Å ; c = 22.057(3) Å. In addition, the crystallite size calculation, 14.01 nm, confirm the nanostructure of the studied material. whereas the complex impedance spectroscopy (Figure 3) results have highlighted the best total electric conductivity, 6.66 10^{-5} $S.cm^{-1}$ at 303 K. However concerning the IR-FT study (Figure 4) we can conclude that the results of vibration mode frequency assignment tentatives tend to be compatibles with bibliographic studies.

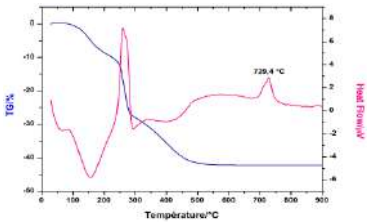

Figure 1: *The thermogram DTA-TGA coupled of the Nanomaterial* $Li_{2,8}Hf_2Si_{1.8}P_{1.2}O_{12}$

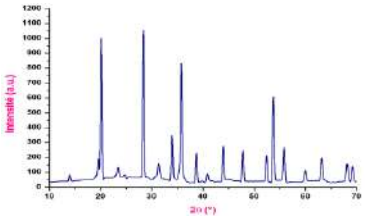

Figure 2: *The X-Ray Powder diffratogram of the nanomaterial* $Li_{2,8}Hf_2Si_{1.8}P_{1.2}O_{12}$

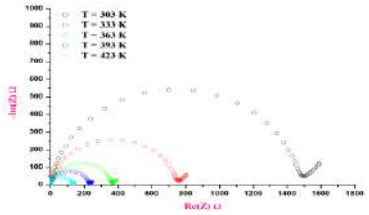

Figure 3 : *The impedance diagram of the nanomaterial* $Li_{2,8}Hf_2Si_{1.8}P_{1.2}O_{12}$

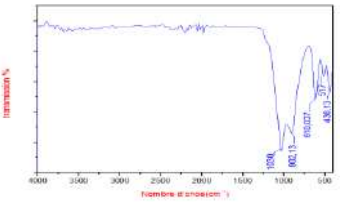

Figure 4 : *The infrared absoption spectrum of the nanomaterial* $Li_{2,8}Hf_2Si_{1.8}P_{1.2}O_{12}$

ELABORATION AND CHARACTERIZATION OF A NEW NANOCOMPOSITE $(ZnO)_{0.25}(SiO_2)_{0.5}(TiO_2)_{0.25}$

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The nanocomposite $(ZnO)_{0.25}(SiO_2)_{0.5}(TiO_2)_{0.25}$ has been successfully elaborated using sol-gel method. The obtained sample has been characterized by various techniques such as X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Raman Spectroscopy, UV-Vis transmittance spectroscopy, Photoluminescence (PL), BET surface area analysis, and Scanning Electron Microscopy (SEM).The X-ray diffraction (XRD) analysis of mixture sintered at 1200°C for 2h revealed the appearance only anatase phase of TiO_2 and willemite Zn_2SiO_4 phase. Optical measurements reveal a high transmittance in the visible range and direct band value about 3.32 eV.

Keywords*:* Sol-gel; ZnO; SiO_2; TiO_2; nanocomposite.

Synthesis and thermal behavior of a new rubidium hydrogen sulfate arsenate $Rb_4(SO_4)(HSO_4)_2(H_3AsO_4)$

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Single crystals of $Rb_4(SO_4)(HSO_4)_2(H_3AsO_4)$ are prepared from an aqueous solution of rubidium carbonate, sulfuric acid and arsenic acid, according to the following reaction:

$$RbHSO_4 + Rb_3H(SO_4)_2 + H_3AsO_4 \longrightarrow Rb_4(SO_4)(HSO_4)_2(H_3AsO_4) + H_2O$$

Slow evaporation of water at 298 K yielded colorless, transparent and stretched parallelepiped crystals after a few days later with a size of about (0,047 × 0,076 × 0,219) mm^3. It is noted that the compound is stable in air and its formula is determined by chemical analysis and confirmed by refinement of the crystal structure.

The thermogravimetry analysis (TGA) and the differential scanning calorimetric (DSC) show the presence of a structural phase transition of the title compound at 407 K which is confirmed by the variation of fp and σ_{ac} as a function of temperature. Thermal decomposition of this compound takes place at much higher temperatures, with an onset of approximately 522 K.

Ac impedance measurements revealed that, upon heating, the compound undergoes at 409 K a sharp increase in conductivity ($\sigma_{428K} = 1.048\ x10^{-4}\ \Omega^{-1}cm^{-1}$) from a low temperature protonic phase to a superprotonic conductivity phase. The activation energies calculated from the modulus (ΔE_f) and impedance (ΔE_σ) spectra respectively are approximately equal, suggesting that the transport properties in this material above and below the superprotonic phase transition (407 K) are probably due to an H^+ protons hopping mechanism.

Structural, Electron diffraction investigation and Mössbauer properties in $LaBaFeTiO_{6-\delta}$ compound

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The new double perovskite oxide of the formula $LaBaFeTiO_{6-\delta}$ has been prepared by sol-gel process and has been investigated by X-ray powder diffraction, Electronic diffraction, high resolution scanning transmission and Mössbauer spectroscopy. The Reitveld refinement of the X-ray diffraction data show that our average sample has been crystallized in the cubic system with Pm-3m space group. However the electronic diffraction shows extra week satellites can explain by trigonal local structure with R-3c space group. To determine the origin of this distortion and extra week a model structure has been suggested. Iron and Titanium are generally in octahedral environments, with stretched octahedral. The Mössbauer measurement effected at room temperature and 77 K showed a paramagnetic behavior with iron in Fe^{3+} high-spin state

Keywords: perovskite oxide, sol-gel reaction, SEM, X-ray diffraction, Electronic Diffraction, Mössbauer spectroscopy.

DESIGN AND CHARACTERIZATION OF CERMET ELABORETED FROM KAOLIN AND ALUMINUM POWDERS

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A cermet is a composite material composed of ceramic and metallic materials. Composite were elaborated from kaolin and aluminum powder mixtures by the press drying-sintering process. The evolution of structure and surface properties was followed by tensile strength, total porosities, hardness and scanning electron microscopy. The rupture strength changes arbitrarily with the variation of the percentage of aluminum. The best mechanical properties are obtained for 4, 7 and 10 wt % of metal doping in the kaolin matrix.

Key words: kaolin, Aluminum, Cermet, Sintering, Mechanical properties

Synthesis and characterization of inorganic foams made from alkali-activated Tunisian clay

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This study reports the results of our investigation on the possibility of producing foam geopolymer using Tunisian clay mineral. Raw and calcinated clays were mixed with an alkaline activator solution (a mixture of sodium silicate and NaOH) and silica fume (foaming agent). The in situ reaction between silica fume powder and alkali activator solution at 70°C in geopolymers formation caused porous structures. After geopolymerization process the synthesized foam was investigated by X-Ray diffraction, FTIR spectroscopy and scanning electron microscopy. Physical properties such as porosity, fire resistance and mechanical strength were checked from the completely dried samples. SEM analysis was conducted to evaluate the microstructure of successfully foamed geopolymers. Compressive strength of all foamed specimens was lower than 2 MPa, which is suitable for using as fireproof coating and fire resistant panels.

Key words: Tunisian clay, Inorganic foam, alkali-activation, fire resistance.

Synthesis, Crystal Structure, Infrared spectroscopy, Hirshfeld Surface Analysis and Antibacterial Activity of a New Organic–Inorganic Hybrid Compound $[C_6H_{16}N_2](ClO_4)_2.H_2O$

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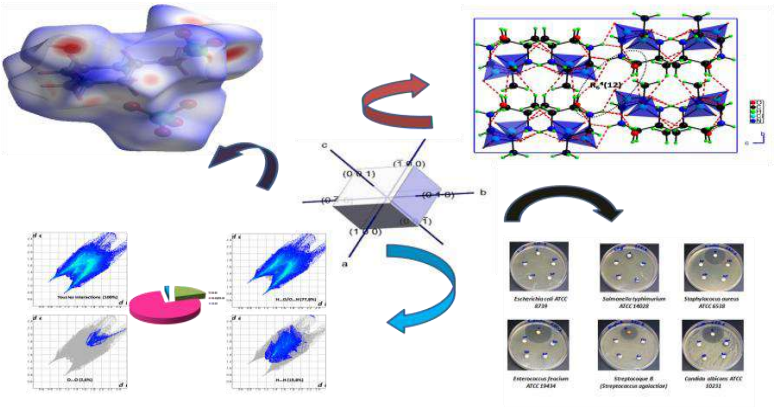
A novel organic–inorganic hybrid compound $[C_6H_{16}N_2](ClO_4)_2.H_2O$ has been grown by slow evaporation method and characterized by powder X-ray and single-crystal XRD methods, IR, UV–Vis and Hirshfeld surface. Single-crystal XRD analyses show that 2,6- dimethylpiperazine-1,4-diium perchlorate monohydrate crystallizes in the orthorhombic space group Pcab with unit cell parameters: a = 13.1195(5) Å, **b** = 12.4058(5), Å, **c** = 16.8438(8) Å and Z = 8. The organic cations and anionic entities are interconnected by the $R^4_6(12)$ motifs hydrogen bonds so as to build a three dimensional network. Three dimensional Hirshfeld surface analysis and two dimensional fingerprint maps disclose that the structure is subjugated by H…O/O…H and H…H contacts. The strongest hydrogen bonding interactions related to O–H…O and N–H…O represent the highest fraction of 77.8 % followed by these of the H…H type contributing 19.8 %. In addition, the compound shows promising antimicrobial activity against three pathogenic microbes. This compound is also characterized by IR and UV-Vis spectroscopies.

Graphical Abstract :



DOES THE PRESCENCE OF PHOSPHORUS HAVE AN EFFECT ON PORTLAND CEMENT QUALITY?

Sonia Boughanmi[a], Islem Laabidi[a], Adel Megriche[a], André Noant[b]

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Many research have been developed on the effect of minor elements on the Portland cement (process, mineralogy, reactivity..), especially in the last decade when wastes valorization become a common practice industry and the trace elements content in cement has increased [1,2]. A big part of these studies highlight the effect of phosphorous on the main clinker phase and its effect on Portland cement reactivity [3].
The aim of this presentation is to control the effect of phosphorus on a series of industrial clinkers manufactured in the same heat treatment on the same rotary kiln cement and underwent the same cooling conditions. The chemical composition of the samples was determinated by X-ray fluorescence, their mineralogical compositions by X-ray diffraction/Reitveld analysis, the cement reactivity was controlled by isothermal calorimetry, conductimetry and compressive strength.

Key words: P_2O_5, industrial, clinker, heat treatment, rotary kiln cement, cooling conditions, X-ray fluorescence, X-ray diffraction/Reitveld analysis, cement reactivity.

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Physical Properties of ZnO by Thermal Evaporation

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Zinc oxide (ZnO) thin films were deposited on glass Pyrex and on Si(100) substrates using thermal evaporation. The structural, morphological, optical, electrical and mechanical properties of these ZnO thin films were compared systematically after investigation using X-ray diffraction, Scanning Electronic Microscopy, Spectrophotometer technique, four point and nanoindentation. These ZnO films were found to be homogenous and polycrystalline in nature. Optical band gap was estimated to be around 3.3 eV.

Optical, electrical and mechanical studies of ZnO/glass films show a decreasing of transmittance, conductivity and hardness when the temperature of glass substrate increases. Whereas, the hardness of ZnO samples deposited on silicon substrate is improved with 50%, the resistivity is reduced as soon as the silicon substrate's temperature increases.

Key words: Thermal evaporation, Glass, silicon, XRD, SEM, spectrophotometer

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NUMERICAL INVESTIGATION OF THE PLASTIC DEFORMATION OF POLYMERS DURING TWO TURN EQUAL CHANNEL ANGULAR EXTRUSION

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Among various severe plastic deformation methods, equal channel angular extrusion (ECAE) introduces large plastic deformation in a sample by moving it through two intersecting channels without substantial change in its dimensions. In this work, an analysis of the high density polyethylene behavior during two turn Equal Channel Angular Extrusion (2-ECAE) has been investigated by finite element using 90° and 120° dies. ~~To this end~~,For this aim, the material parameters of an elasto-viscoplastic phenomenological model were derived from compressive tests at different temperatures and strain rates. A good agreement was found between the identified model and experimental results. Then, the effects of the channel angles, the length and thickness of the intermediate channel on the equivalent plastic strain and the pressing force have been analyzed. Recommendations on process conditions were ~~proclaimed~~ suggested at the end of this work.

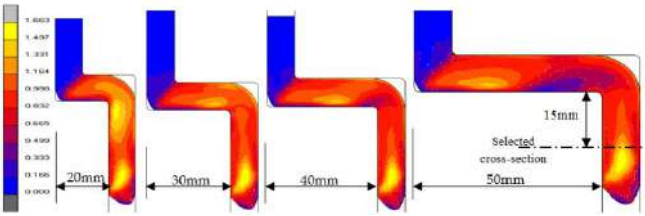

Fig. Equivalent plastic strain contours for HDPE of the samples during 2-ECAE process using 90° die for different lengths of the intermediate channel.

Key words: ECAE, HDPE, Finite element, Plastic deformation, Pressing force.

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Cellulose nanofibers extracted from Alfa plant as bio-reinforcement for Polyurethane matrix

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This work concerns the study of the morphological, mechanical and thermal properties of nanocomposite films made by polyurethane matrix reinforced with cellulose nanofibers which extracted from Alfa plant by chemical treatments and ultra-sonication.

The morphology of the films and the fibers was studied by scanning electron microscopy (SEM) while the thermal behavior and the mechanical properties of nanocomposites were determined using thermogravimetric analysis (TGA) and tensile tests respectively.

Key words: cellulose nanofibers, Alfa plant, bio-reinforcement, nanocomposite, Polyurethane

STUDY OF THE INTERACTION OF METHYLENE BLUE WITH POLYSTYRENE -CO- STYRENE SULFONATE

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The interaction of the cationic phenothiazine dye, the Methylene Blue (MB) with poly-(sodium styrene sulfonate)*f*–co-(styrene)*1-f*, (PSSNa *f*), has been investigated by spectrophotometric method. The polyelectrolyte induced metachromasy resulting in a blue shift of the absorption maxima of the dye, in agreement with the formation of dye H-aggregates. The stability of the PSSNa-MB complexes was studied as a function of polyelectrolyte chain length, polyelectrolyte electrostatic charge density *f*, polyelectrolyte concentration, NaCl salt addition, tetrahydrofurane (THF) addition and THF treatment (consisting first to add to water, THF, which is then evaporated and replaced by the same amount of water) [1]. The stoichiometry of PSSNa-MB complex evaluated by the molar ratio method was found 4:1 for the fully charged PSSNa *f*=1, which is a high value in comparison to other systems (ex polyacrylic acid) where the stoichiometry was 2:1 [2]. This indicates that there is less overcrowding of the bound dyes on the PSSNa chain because of the aromatic rings steric effects. Reversal of metachromasy was observed upon salt and THF addition, while THF treatment does not affect the complex and allows recovering the initial complex. Finally, thermodynamic parameters of the interaction between the polyelectrolyte and the dye at different temperatures, namely free energy ΔG, the enthalpy ΔH and the entropy ΔS have been evaluated to determine the binding constant and as a consequence the stability of the complex.

Key words: Methylene Blue, poly(styrene-*co*-sodium styrene sulfonate), metachromatic complex, Thermodynamic parameters**.**

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Removal of heavy metals by new polymers bearing aminoalkylated and organophosphorus groups

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The study of the new structures mainly useful in the synthesis of more efficient and selective new extractants led to the development of new series of macromolecules. Hence, in our present work, the polystyrene was chemically modified to synthesize novel substituted polymers bearing functional groups such as amine and organophosphorus. The synthesized resins were then tried to evaluate their efficiency for extracting metallic ions such as Cr^{3+}, Fe^{3+}, Ni^{2+}, Cd^{2+}, Cd^{2+} and Pb^{2+}…. from aqueous solutions. The obtained resins were characterized via attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), elemental analysis (EA), thermogravimetric analysis (TGA), thermodynamic analysis (DTA) and differential thermogravimetric analysis (DTG).

Keywords: Polystyrene, chelating groups, ions metallic extraction.

DEVELOPMENT OF LEATHER WASTES BASED ANTIMICROBIAL COMPOSITE MATERIALS

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Nowadays, the disposal of thousands of tons of leather wastes is one of the main problems of leather industries. The recovery of these considerable amounts of solid wastes has become a major challenge for sustainable development. In fact, it raises many concerns regarding the environmental effects and escalating landfill costs. In the other hand, consumers request new active and smart materials which are creating new challenges for footwear industry. Self-sterilizing fabrics could have potential benefits to reduce disease transfers in hospital environment and provide personal hygiene and comfort. As a result, the conversion of leather wastes into valuable materials with antimicrobial activity could be a great interest for both industry and consumers. In this field, many attempts have been made to prepare commercially acceptable leather substitutes by combining leather fibers or wastes with natural or synthetic fibers and binders [1,2].

The main purpose of this work is to develop new antimicrobial composite materials that can meet several requirements such as: environment protection, economy of raw materials and the production of high quality materials in terms of physical and chemical as well as mechanical proprieties. The structure of the obtained composite has been thoroughly analyzed by several complementary analytical methods. Several mechanical tests were used in this work to determine the properties of leather composite, such as tear strength, thickness and tensile strength.

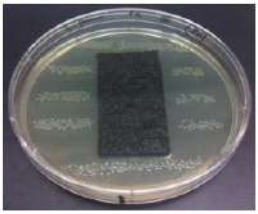

Fig. Screening evaluation of antimicrobial activity using the Agar Diffusion Method

Key words: Leather wastes, Valorization, Antimicrobial composite material

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NEW 2,7-DISUBSTITUTED CARBAZOLE BASED CONJUGATED MODELS: SYNTHESIS, CHARACTERIZATION AND APPLICATION ON ORGANIC FIELD-EFFECT TRANSISTOR

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Two new semi-conducting models based on 2,7-disubstituted carbazole and coupled with anthracene were synthesized via diamine condensation and Wittig-Horner reactions. The effect of substitution of the linker vinylene group (-CH=CH-) in molecule M1 by azomethine (-CH=N-) ~~in~~ to obtain the molecule M2 on physicochemical and electrical properties was reported in this study.

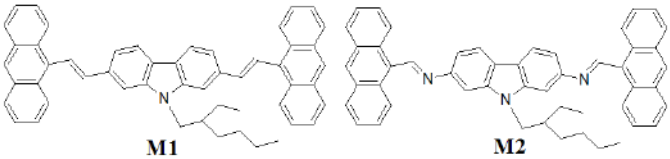

Fig. Structures of the models M1 and M2.

The analysis of both molecules TGA and DSC, show a good thermal stability up to 300 and 310 °C for M1 and M2 respectively and high melting temperature (T_m) (233 and 182°C for M1 and M2 respectively).
The UV–Vis absorption spectra of the models (in chloroform solution and in thin films) show absorption maxima in the range of 345 – 450 nm. The two materials had blue and blue-green emission. The replacement of vinylene group (-CH=CH-) by the azomethine linkage seems to contribute for increasing inter-chain π–π stacking interaction in solid state resulting a decrease of the π–π * transition energy. The carrier mobilities were extracted from the electrical characteristics measured in organic field-effect transistors (OFET) configuration.

Key words: Organic Semi-Conductors, Carbazole, Anthracene, Conjugated Models, UV–Vis absorption, Organic Field-Fffect Transistors (OFET), carrier mobility, Organic Electronics.

Synthesis of nanosized manganese dioxide prepared by the polyol method. Catalytic properties of a new nanocomposite-based on MnO_2 nanoparticles/organic polymer.

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δ- MnO_2 (Birnessite type) nanoparticles have been successfully synthesized via a polyol reflux process through oxidizing $MnCl_2$ with H_2O_2 in basic conditions using the polyvinylpyrrolidone (PVP) as a surfactant polymer. Morphological studies shown the synthesized particles to be a nanospherical structure depending on experimental conditions (reflux temperature, molar ratio (n (OH^-)/n (Mn (II))...). The FTIR spectrum confirms the presence of Mn-O bands. As a result we have used these nanoparticles to make a new and ecofriendly nanocomposite called MnO_2/PES-NH_2. In fact, the PES-NH_2 is the Polyethersulfone amine which is used in this case to prepare a new work-electrode for electrochemical measurements. Electrochemical analysis of MnO_2/PES-NH_2 nanocomposite was found to exhibit the highest specific capacitance .Electrochemical measurements (cyclic voltammetry, impedance spectroscopy and galvanostatic coulometry) proved that the as synthesized nanocomposite was a good electrode material for a supercapacitor.

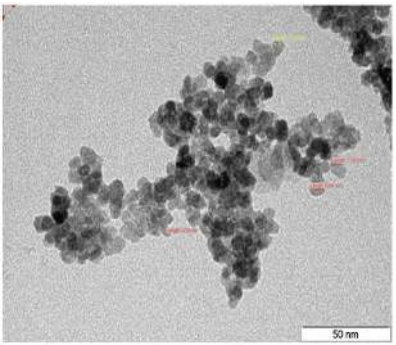

Fig. TEM image of the product obtained at reflexing temperature of 120°C for 1h with molar ratio OH/Mn=5 and PVP/Mn =0.6%

Key words: Electrochemical capacitors, Pseudocapacitance Manganese dioxide , Nanostructured materials .

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Effect of anion type in the performance of ionic liquid/poly(vinylidene fluoride) electromechanical actuators

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Low voltage actuators based on poly(vinylidene fluoride) (PVDF) with 10, 25 and 40% 1-hexyl-3-methylimidazolium chloride ([C6mim][Cl]) and 1-hexyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([C6mim][NTf2]) are prepared by solvent casting in order to evaluate the effect of anion size in the bending properties. Independently of the ionic liquid type and content, its presence leads to the crystallization of PVDF in the β-phase. The addition of ionic liquid into the polymer matrix decreases significantly its degree of crystallinity and the elastic modulus. It is also confirmed the good miscibility between PVDF and IL, determined by the interaction of the CF2 groups from the PVDF chains with the imidazolium ring in the ionic liquid (IL). The AC conductivity of the composites depends both on the amount of ionic liquid content and anion size. The bending movement of the IL/PVDF composites is correlated to their degree of crystallinity, mechanical properties and ionic conductivity value and the best value of bending response (0.53%) being found for IL/PVDF composite with 40 wt% of [C6mim][Cl] at an applied voltage of 10 V square signal.

Keywords: PVDF, Ionic liquid, Actuators, Bending

PREPARATION AND CHARACTERIZATION OF NANOFIBERS BASED MOF-5 AND HKUST-1

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In the last decade, coordination polymers with infinite structures have been extensively studied. These compounds are organometallic structures known as "Metal Organic Framework (MOF)". Their highly diverse structures, wide surfaces, large porous size ranges and their functionality; offer a great potential for various applications especially in the field of catalysis, storage and gas separation [1-3].
Among the most representative metal organic frameworks, we can cite the HKUST-1 and MOF-5. They are prototypes of this new class of porous materials. These materials are constructed by assembling inorganic building units (consisting of metal cations) and organic entities also known as linkers.
Despite the huge potential especially in the separation, there are very few reports about the growth of MOFs in continuous films or synthetic membranes [4, 5]. Thus, this new type of porous substrate with easily adjustable structural parameters (such as porosity, thickness, size and specific surface area) can be investigated. More recently, new methods have been placed on the preparation of supported MOFs thin films and membranes, including electrospinning [6], [7].
Besides, the electrospinning is a simple and versatile technique which can produce continuous fibers with diameters ranging from a few micrometers to tens of nanometers [8]. This technique is based on stretching of a jet of polymer solution through electrical loads.The nanofibers so obtained could be used in several areas. Among these, catalysis, tissue engineering, protective textiles, filtration, biomedical, pharmaceuticals, optoelectronics, environmental engineering, may be mentioned.
By combining the high production rate of fibers with the simplicity of electrospinning process, the nanofibers can be produced from various types of materials (organic, inorganic or hybrid) [9].
We describe in this work the electrospinning of composite consisting of a polymer/metallic cation (Zn^{2+}, Cu^{2+}, ...) and the optimal operating conditions for the immersion of the obtained nanofiber in a solution containing different linkers (H_2BDC, H_3BTC, ...). The obtained mats were characterized using Fourier Transform InfraRed spectroscopy, powder X-ray diffraction and scanning electron microscopy (SEM).

Keywords: MOF-5,HKUST-1, electrospinning, immersion

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Novel Scenario of Glass Formation in Polymeric Materials*

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We present computer simulations of concentrated solutions of unknotted nonconcatenated semiflexible ring polymers. We investigate the slow dynamics of the centers-of-mass of the rings in the amorphous cluster phase. Scattering functions reveal a striking decoupling of self- and collective motions.
Correlations between centers-of-mass exhibit slow relaxation, as expected for an incipient glass transition, indicating the dynamic arrest of the cluster positions. However, self-correlations decay at much shorter time scales. Our results reveal a novel scenario of glass formation in a simple monodisperse system, characterized by self-collective decoupling, soft caging, and mild dynamic heterogeneity.

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MICROFILTRATION FLAT SHEET MEMBRANES FROM WASTE POLYMER: SYNTHESIS AND CHARACTERIZATION

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In order to see the influence of some operating parameters on the polymeric membrane properties, a parametric study was done. These parameters are polymer concentration, solvent, non solvent and additive. The results showed that it's better to prepare membranes with high polymer concentration and xylene as solvent. In this case, we obtained membranes with a large pores size (2.53 µm). The effect of non-solvent was treated. The use of unrefined solvent minimizes shrinkage of the membrane. In our case, this shrinkage was about 90% in the case of the thickness. About the modification of the membrane surface, we were used lignin and PEG. In the case of PEG, hydrophilic membrane was obtained (contact angle of 63.25°). For against, lignin improves the hydrophobicity since the contact angle was increased and reached 94.17°

Key words: polymeric membrane, characterization, LEP, hydrophobicity.

Synthesis and characterization of low cost hybrid organomaterial for organic pollutants removal from aqueous solution as alternative to wastewater reuse

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Surfactant modified bentonite was prepared using hexadecyltrimethyl-ammonium (HDTMA) bromide at three CEC levels (1, 2 and 3CEC), obtained organobentonite labeled H_1-bent, H_2-bent and H_3-bent were characterized by several methods such as X-ray diffraction (XRD), Infrared spectroscopy (IR), thermal analysis (TGA-DTG), Brunauer-Emmett-Teller (BET) and transmission electron microscopy (TEM). These methods were used to visualize the interlayer structure of organoclays. XRD patterns showed the changes in the d(001) spacings, which gave details of the arrangement of surfactant in the organoclays, IR spectroscopy showed the existence of HDTMA functional groups on bentonite surface. The BET surface area significantly decreased after the modification due to the coverage of the pores of Na-bent. Acid-base potentiometric titration and mass titration behavior of all samples were investigated. The point of zero charge (PZC) estimated by these both methods was about 6.4 for Na-bent and 6.8, 7.7 and 8.2 for H_1-bent, H_2-bent and H_3-bent respectively. Bentonite surface which has hydrophilic property in nature converts to hydrophobic after organophilisation. The hybrid material develops more positive surface charges and great basal spacing layers; therefore these materials can be very useful to remove anionic pollutants from wastewater.
Accordingly HDTMA-bentonites and Na-bent were then tested as adsorbents for the removal of an organic compound, the anionic reactive dye (reactive blue II (RBII)), which is very used in textiles industries yet it is carcinogen and very toxic, from aqueous solutions. The amount of adsorbed HDTMA-bentonites was found to be around 4 times higher than that of Na-bent, 98% of dye was removed by H_3-bent without any further modification when only 24% was removed by Na-bent. The results indicate that HDTMA-bentonite is an effective and a low-cost adsorbent for the removal of anionic dye. It was proved that HDTMA-modified minerals have high anion sorption capacity.

Keywords: Bentonite, organoclay, surfactant , anionic dye, adsorption isotherms, thermol analysis

Selective extraction of 2,5-hexanedione by molecular imprinting: optimization of synthesis parameters

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Biological monitoring of exposure to chemical pollutants is often achieved through the analysis of their urinary metabolites. Due to the urine matrix complexity, the analysis of the biological samples requires a pretreatment step, in order to concentrate sample analytes, reduce interferences and improve the analytical method sensitivity. Sample preparation techniques such as liquid and solid phase extraction are commonly used. However, compared to these classic methods, the Molecularly Imprinted Polymers (MIPs) have many advantages: low cost of synthesis, easy manufacturing and high selectivity. For these reasons, they offer an interesting alternative as a new and efficient sorbents for selective extraction of several pollutants and their metabolites from biological samples. The aim of this study was to prepare a selective MIP for the extraction of 2,5-hexanedione a biological indicator of exposure to hexane. The "porogenic" solvent (which promotes the formation of pores in the polymer), and the functional monomer to use for the synthesis were optimized to improve the recognition performance of the MIPs. Different molar ratios of the cross-linker to the functional monomer were evaluated. The synthesized imprinted and non imprinted polymers were characterized with infrared spectroscopy to check their structures and confirm the success of the polymerization process.
The obtained results showed that the acetonitrile is the better "porogenic" solvent. The acrylic acid was found to be a suitable monomer with a good affinity to the template and a higher retention capacity of the MIP in comparison to the NIP. The molar ratio (0.8 : 3 mmol) between the cross-linker and the functional monomer showed the optimum retention and the best selectivity.

Key words: Molecular imprinting, Synthesis, 2,5-hexanedione, Biological monitoring, hexane

TEMPERATURE AND FLOW EFFECT ON THE FORMATION OF CORROSION PRODUCT FILM ON THE CUPRONICKEL 90/10 SURFACE IMMERGED IN SYNTHETIC SEAWATER

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Cu-Ni 90/10 alloys used in seawater desalination shell-heat exchangers and condensers exhibit a good resistance toward corrosion. This resistance results from the formation of a protective oxide film that forms naturally and quickly when exposed to seawater. Unfortunately, some Cupronickel tubes suffer from severe corrosion due to the combination of heating and evaporating process of seawater.

The aim of this study was to evaluate the temperature effect on corrosion behaviour of 90/10 Cu-Ni from 20°C to 80°C by electrochemical techniques in artificial seawater under stirring conditions. The corroded surfaces were then analysed by optical microscopy, SEM and μ-Raman Spectroscopy. OCP, Rp and EIS measurements show that the alloy exhibits a resistance toward corrosion after immersion in synthetic seawater which increases with increasing temperature up to T< 60°C. The composition of the film formed on the tube immersed in seawater after 48h of immersion has revealed an enrichment of the oxide film in Ni. μ-Raman spectroscopy shows that whatever the temperature, the oxide film is composed of Cu oxide and Ni oxide covered by a calcareous layer. At 80°C, OCP decreases in the cathodic direction and Rp is reduced. This result was attributed to a lower dissolved O2 and then to a less resistive oxide film that gives rise to a higher corrosion rate.

Key words: desalination, cupronickel, corrosion, seawater, OCP, Rp, Raman spectroscopy

PRODUCTION OF ADIPIC ACID BY GREEN ROUTE

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Adipic acid is an important bulk chemical product for different industrial chemistry processes. It is largely used in the manufacture of polyurethanes, plasticizers and particularly nylon 6,6 polyamide. The aim of this work is to study the activity of supported gold and iron nanoparticles on cyclohexene oxidation by O_2. For this, we prepared Au/TiO_2 and Au/ZrO_2 by deposition precipitation with urea (DPU) with 1 wt% of gold, and the bimetallic catalyst ($Au\text{-}Fe/TiO_2$, $Au\text{-}Fe/Al_2O_3$) are prepared by deposition under tension (UPD). The catalysts are characterized by different techniques: TEM (EDX), XRD, DR/UV-Vis and ICP. The test reactions of these catalysts show the production of adipic acid.

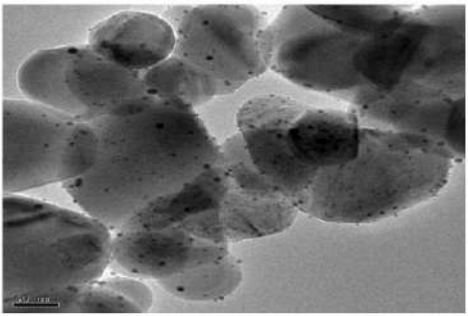

Fig. TEM image of bimetallic catalyst $Au\text{-}Fe/TiO_2$

Key words: Adipic acid, oxidation, gold, iron, nanoparticles, titania.

Program of Tuesday 20 December 2016

SYNTHESIS OF NEW ANTI-PROLIFERATIVE (*N*-SUBSTITUED)-CONVOLVINE DERIVATIVES *VIA* PALLADIUM-CATALYZED BUCHWALD-HARTWIG COUPLING REACTION

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A series of (*N*-substituted)-convolvine derivatives have been synthesized by the palladium catalyzed C–N Buchwald-Hartwig coupling reaction. In this context, the reaction of convolvine with 3-bromoquinolin-2-(1H)-one was applied as a model[1]. Then, various nucleophiles including aryls and heteroaryls have been used successfully. In all the cases, the reaction took place in 1,4-dioxane and proceed in good to excellent yield using palladium acetate as a catalyst, Xphos as a ligand and ^{t}BuONa as a base. Investigations on further expanding the scope of the method to other related heterocycles are in progress in our laboratory. Upon completion of the synthesis of analogues of convolvine, the *in vitro* anti-proliferative activity of all products was evaluated against the human colon carcinoma HCT-116 cell line at the concentration of 10^{-5} M. Our findings showed interesting preliminary anti-proliferative potential of most compounds tested.

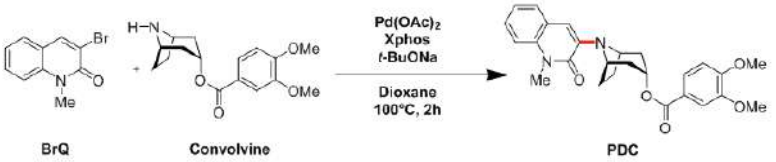

Fig. Coupling reaction of convolvine with bromoquinolein-2-one

Key words: Convolvine, Synthesis, palladium-catalyzed, HCT-116.

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Synthesis of (benzo)furan derivatives *via* Palladium-Catalyzed Desulfitative

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The discovery of green chemistry processes is a great challenge and hot topic from both academic and industrial points of views. Indeed, the simplification of chemical processes along with the decrease of waste and cost is an important ideal for human development. In this line, the catalytic direct functionalization of C-H bonds has emerged recently as one of the most promising and powerful methods for the construction of C-C bonds because of the generation of lower amounts of waste compared to that of traditional useful crosscouplings. [1]
Herein, we report a novel approach for the synthesis of (benzo)furan derivatives. This reaction consists of a palladium-catalyzed direct desulfitative C-H bond arylation of (benzo)furan with benzenesulfonyl chlorides. Recently, metalcatalyzed C-H bond activation was introduced as one of the most attractive methods for the modification and the synthesis of heterocycles. [2]

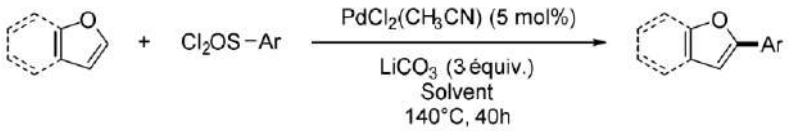

Fig. Pd-catalyzed desulfitative C-H bond arylations in which benzenesulfonyl chlorides are used as coupling partners

Key words: (benzo)furan, palladium-catalyzed, desulfitative

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DIRECT REGIOSELECTIVE DIPHOSPHONYLATION OF CYCLIC ENAMINES WITH P-CHLORODIPHENYLPHOSPHINE: ONE-POT SYNTHESIS OF β, β'-BIS(DIPHENYLPHOSPHORYL)- AND β, β'-BIS(DIPHENYLPHOSPHOROTHIOYL) CYCLOALKANONES

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In recent years, organic phosphorus compounds has evoked the attention of chemists[1] due to their numerous application domain[2].
In view of this particular interest, an efficient, facile and straightforward regio- and diastereoselective process has been developed for the synthesis of symmetrical *trans*-α,α'-bis(diphenylphosphoryl)- and α,α'-bis(diphenyl-phosphorothioyl)-cycloalkanones, through the reaction of cyclic enamines with excess P-chlorodiphenylphosphine in the presence of triethylamine, followed by oxidation or sulfurization and hydrolytic work up.

The synthetic routes of the proposed compounds are outlined in the figure above (fig 1).

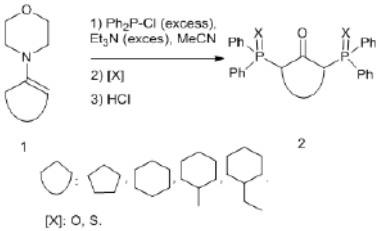

Fig.1 Reaction conditions and scope studies.

Key words: Enamines, P -chlorodiphenylphosphine, α,α' bis(diphenylphos-phoryl)-cycloalkanones, bis(diphenylphosphorothioyl)-cycloalkanones.

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SYNTHESIS AND REACTIVITY OF 5,5-DIALKYL-2-CHLORO-1,3,2-DIOXAPHOSPHORINANE

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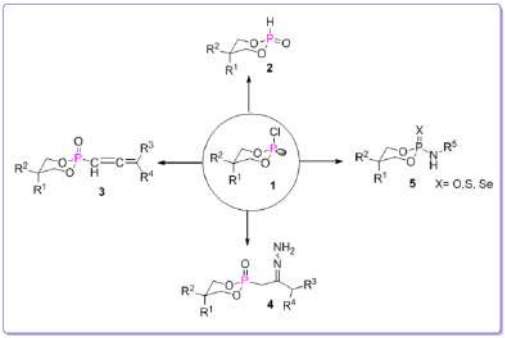
Organophosphorus compounds exhibit interesting applications in pharmaceuticals, agrochemicals, plasticizers, Wittig olefination, measuring enantiomeric excess and catalysis [1]…

5,5-dialkyl-2-chloro-1,3,2-dioxaphosphorinanes **1** were synthesized and studied by NMR (^{1}H,^{31}P,^{13}C). Diastereoisomeric products were obtained and analyzed by computational method DFT in order to determine the predominant conformation. Obtained chlorophosphines were used as excellent precursors for the synthesis of some organophosphorus compounds [2-3]: dialkylphosphites **2**, allenes **3**, hydrazones **4** and cyclophosphamides **5 (Scheme-1)**. The steps of reactions were followed by ^{31}P NMR.



Scheme-1: The synthesized organophosphorus compounds

Key words: 1,3,2-dioxaphosphorinane, dialkylphosphite, hydrazones, cyclophosphamide, heterocyclic synthesis.

References :

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SYNTHESIS OF γ-DIKETONES FROM CYCLIC ENONES AND THEIR REACTIVITY

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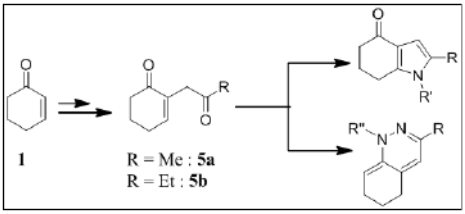
1,4-diketones are widely used as a synthetic intermediate in the preparation of many heterocyclic compounds [1]. In this work, we proposed a new strategy for the synthesis of γ-diketones **5a,b** from cyclohex-2-enone **1** via Baylis-Hillman reaction. In addition, we examined their reactivity using primary amine and some derivatives of hydrazine. Several types of heterocycles were synthesized and should show potential biological activities such as anti-inflammatory, anticancer and antimicrobial [2].
The structures of all of the prepared compounds structures were confirmed by ^{1}H, ^{13}C NMR spectral data.



Key words: γ-diketones, indole, pyridazine.

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DEVELOPMENT OF NEW ANTIBACTERIAL AZIRIDINES AND FORMULATION WITH CYCLODEXTRINES

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Background: Antibiotics, as miraculous drugs, have been used extensively to confront fatal infection, even without prescriptions. However, the inappropriate and disproportionate use of antibiotics have led to the emergence of new drug-resistant bacteria1, which causes a high risk of serious diseases and dramatically aggravates the clinical complications in hospitals.
Methods: By using the peptide coupling protocol, a simple straightforward synthesis of functionalized aziridines has been developed. By means of this synthetic strategy from readily available N-phtaloyl acide and 2-methylbenzosulfonate aziridine using DCC as coupling agent, new tosylates aziridines could be obtained. The coupling reactions occurred without ring opening of the three membered ring.
Results: This work describes new results of our ongoing research targeting new derivatives of biological interest. All the compounds were screened for their antibacterial activity, they all showed comparable moderate to good growth inhibitory activity with reference to Tetracyclin and Gentamicin.
Conclusion: In conclusion, we reported the synthesis and a preliminary antibacterial evaluation of novel functionalized tosylaziridines. The synthetic strategy relies on the coupling reactions between tosylaziridines and amino acids. Moreover and besides showing interesting antibacterial activities, the series of novel compounds can be further improved to serve as potential drug against nosocomial diseases.

Keywords: Aziridines, phtaloyl acide, strained heterocycles, Antibiotics.

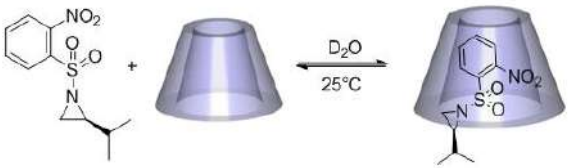

Figure 1: Formulation of aziridine.

TOWARDS FLAVANONES AND FLAVONES FROM FUNCTIONALIZED TETRAHYDRONAPHTALENES

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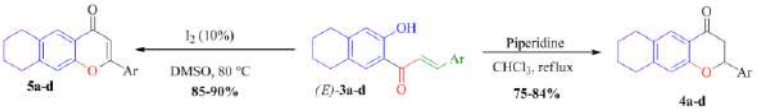
Flavonoids such as flavones and flavanones are a class of natural products isolated from a wide range of vascular plants. Their extensive spectrum of biological activities, including anti-cancer and anti-bacterial ones, have attracted considerable interests in medicinal chemistry.[1]

As part of the synthesis of novel heterocyclic compounds exibiting pharmacological interest, we have developed an efficient two-step sequence for the synthesis of a new series of flavanones **4a-d** and flavones **5a-d**, using the tetrahydronaphtalene **2,** previously prepared through a Robinson annulation of the δ-diketone **1**.[2, 3]

The first synthetic step provided access to chalcones **3a-d** *via* a Claisen-Schmidt condensation between the tetrahydronaphtalene **2** and various aromatic aldehydes using potassium hydroxide in ethanol at room temperature.[3]

In the second step, favanones **4a-d** have been prepared via an intramolecular oxa-Michael addition on **3a-d** in the presenceof potassium hydroxide or both piperidineandpotassium hydroxide in chloroforme**.** Alternatively, the reaction of **3a-d** with diiode in refluxing DMSO, afforded flavones **5a-d** in high yields.[4,5]



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1, 5-BENZODIAZEPIN-2-ONES: INVESTIGATION OF A FAMILY OF PHOTOLUMINESCENT MATERIALS

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Abstract: Photoluminescent materials, that are now ubiquitous in our everyday life, have particularly attracted the attention of the scientific community these past few years due to potential important applications such as in bioimaging, sensing, or optoelectronics. In this context, relatively few different families of molecules have been reported to exhibit fluorescence in the aggregated or solid-state through the excited-state intramolecular proton transfer (ESIPT) photochemical process.[1] The preparation and subsequent determination of photochemical properties of an underexplored family of 1,5-benzodiazepin-2-one derivatives are reported. From these data and X-ray diffraction analysis study, it emerged that photoluminescence (in the range 520-655 nm) was mostly attributed to ESIPT. The photoluminescent potential of 1, 5-benzodiazepin-2-onestheir facile access, and functionalization were demonstrated through the preparation of two fluorogenic probes for the selective detection of biothiols.[2]

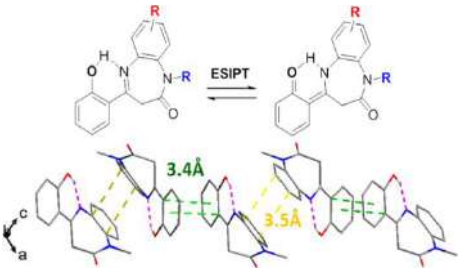

Fig.1: ESIPT Principle

Key words: 1, 5-benzodiazepines, Photoluminescent, ESIPT, Biothiols

References

[1] Hasan Mtiraoui,[a] Rafik Gharbi;[a] Moncef Msaddek,[a] Yann Bretonnière,[b] Chantal Andraud,[b] Cyrille Sabot,[c] Pierre-Yves Renard,[c] ; *J. Org. Chem.* **2016**, 81, 4720-4727.

MECHANISM AND NUMBER OF ADDUCTS OF PHOTO-ADDITION OF TRYPTOPHANE METHYL-ESTER TO [60]FULLERENE

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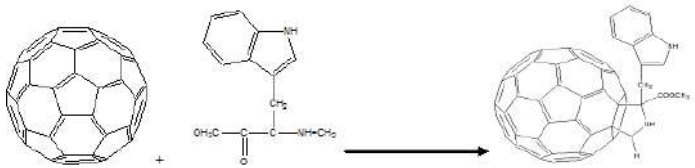
Many derivatives have been synthesized by photo-addition reaction of many polar groups on the [60]Fullerene (C_{60}) [1-2]. Among these derivatives the Fulleropyrrolidines (FP) [3], which are obtainable from Tryptophan methyl ester (TrpME) by [2+3]cyclo-addition reaction involving the oxygen in its singlet state. The understanding of the reactional mechanism help to control their synthesis.
In order to determine the mechanism and the number of adducts involved in the photo-addition of TrpME to C_{60} we investigated the kinetics of FP formation during irradiation of C_{60} in the presence of a large excess of TrpME (1/100 equiv/equiv).
Recently, it has been shown that visible light irradiation of a mixture of C_{60} and TrpME in the presence of oxygen is a direct route to sequentially synthesize higher Fulleropyrrolidine poly-adducts. However, the involved mechanism and the maximum number of pyrrolidine adducts per C_{60} molecule remained to be ascertained.
Using high resolution mass spectrometry and LC-MS, we show here that the most probable mechanism involves a first step of azomethine ylide (AMY) formation followed by its [2+3] cyclo-addition to C_{60}, and that the tetra-adduct is the highest Fulleropyrrolidine poly-adduct (FPPA) obtained under these conditions. Some FPPA and AMY dimers are also identified in the final mixture.



The theoretical study carried on the formation reactions of AMY and Fulleropyrrolidine mono-adduct, clearly shows that the Tryptophan methyl-ester would react more easily under his zwitterionic form, than in its molecular form, with important energetic gain (-30.120 kCal/mol).

Key words: [60]Fullerène, Photo-addition, Tryptophane-méthyle-ester, Fulleropyrrolidine, HPLC-MS

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SYNTHESIS OF FLUORINATED AND NONFLUORINATED AZIRIDINE-2- CARBOXYLATES: A COMPARATIVE STUDY OF THEIR REACTIVITIES

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α-Trifluoromethyl α-amino acids (α-Tfm AAs) and β-Aminoalcohols are versatile precursors for the synthesis of variety of nitrogen-containing compounds which are useful in synthetic and biological fields[1]. The aim of this work was to prepare a new fluorinated aziridine in enantiomerically pur form and nonfluorinated aziridines in racemic form, and to compare their different reactivities.

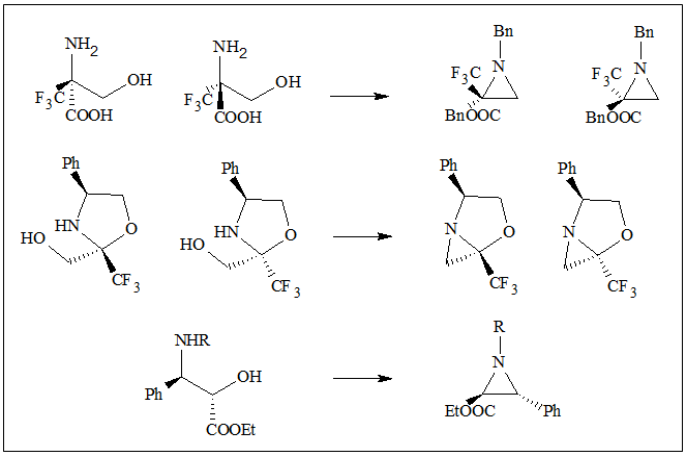

Fig. Conversion of amino acids and aminoalcohols into aziridines

Keywords: Amino Acids, β-Aminoalcohols.

Reference

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SYNTHESIS AND OPTICAL PROPERTIES OF NEW PENTACYCLIC HELICENES

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Helicene are polycyclic hydrocarbons (HAP) composing of benzene or other aromatic rings that fused at the ortho-position resulting in helical shaped molecules. For many years, they were regarded as little more than an academic curiosity. More recently, their extraordinary optical and electronic properties have rekindled an interest in these non-planar condensed aromatic in fields as diverse as liquid crystals[1], asymmetric synthesis and polymer synthesis[2,3].
In this work, new helically chiral pentacyclic systems containing a cyano group at a selected position (Fig.1) have been prepared in good yields via a three-step sequence involving coupling reactions and oxidative photocyclization[4]. The optical properties of the pentacyclic helicenes have been investigated and show interesting behaviour.

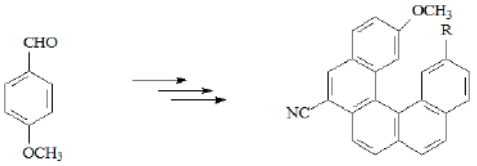

Fig 1. Synthesis of new [5]Helicenes

Key words: Helicenes, couplings, Photocyclization.

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DEVELOPMENT OF A SELECTIVE EXTRACTION METHOD OF HYDROXYTYROSOL

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Hydroxytyrosol was the well known antioxidant in a variety of natural sources. The main source in the human diet was olive oil. This molecule was used in the pharmaceutical field due to its antioxidant properties.
In this work, the extraction of this molecule was done using a new extraction technique: The microwave. To attempt this goal, the optimization of physical parameters was carried out by analyzing the obtained extracts using GC-MS apparatus. Adjust the microwave in medium power during 5 min, the treatment of Chemlali two-phase olive pomace could produce an extract rich in hydroxytyrosol (~ 1.9 g of hydroxytyrosol / Kg of two phase Chemlali olive pomace).
Furthermore, and in order to enhance our research work, we have studied the stability of the mayonnaise after enrichment by 65-195 micrograms of hydroxytyrosol. This study was conducted for 4 weeks and the mayonnaise samples were stored protected from light and at room temperature. The results obtained show that 195µg of hydroxytyrosol may be of great benefit to improving the stability of the mayonnaise against oxidation.

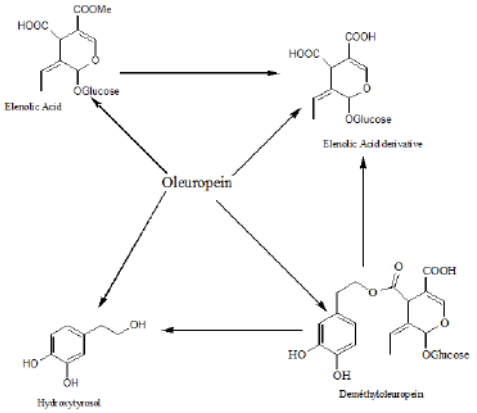

Fig: Various molecules derived from oleuropein

Key words: Hydroxytyrosol, microwave, GC-MS, two phase olive pomace

A CONVENIENT SYNTHESIS OF NEW BIOLOGICAL ACTIVE 5-IMINO-4-THIOXO-2-IMIDAZOLIDINONES INVOLVING ACETONITRILE ELECTROGENERATED BASE

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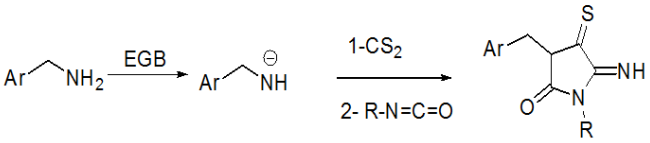
3-Benzyl-5-imino-4-thioxo-2-imidazolidinones are obtained in good yields by using commercially available starting compounds when, under low temperature conditions, a solution of dry acetonitrile is electrolyzed at a graphite sheet as cathode to produce cyanomethyl anion as EGB, that is further reacted with benzylamines, CS_2 and isocyanates. The use of a magnesium rod, as sacrificial anode, is definitive to avoid 3-aminocrotonitrile anion formation. Mechanism proposal is given where cyanide is formed 'in situ' by decomposition of the first electrogenerated organomagnesium derivative.



Keywords: Imidazolidinones; Magnesium anode; Electrogenerated base (EGB); Cyanide; Carbon disulfide; Isocyanates.

FROM YNAMIDES TO ORGANIC ELECTRONICS : A NEW SYNTHESIS OF 2-(TOSYLAMIDO)- AND 2,5-BIS-(TOSYLAMIDO)-THIOPHENES

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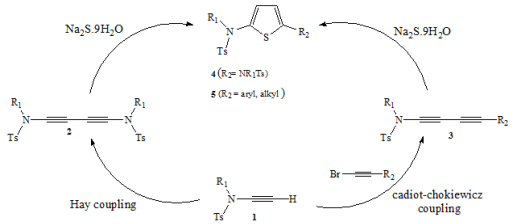
Substituted thiophenes are an important class of molecules for the synthesis of agrochemicals, dyes, and pharmacologically active compounds. In particular, 2-aminothiophenes present an interesting biological and medicinal activities and they are frequently used as the scaffold motif in optoelectronic materials field.
Owing to their promising properties, 2-amino and 2-amidothiophenes with electron-withdrawing/donating substituent have been largely studied. Unfortunately, despite their synthetic importance, their synthesis methods are still quite limited [1,2].
Within this work, we developed a new strategies for the synthesis of 2-(tosylamido)thiophenes **4** and **5** starring from ynamides as versatile building blocks.
The first part was dedicated to the synthesis, crystal structure, optical, electrochemical and thermal properties of the ynamide-derived buta-1,3-diynes.
Both precursors **2** and **3** are readily available from terminal ynamide **1** via Hay or Cadiot−Chodkiewicz cross-coupling reactions.
The conversion of diyne to their corresponding 2,5-bis(tosylamido)thiophene using 3 equiv of $Na_2S{\cdot}9H_2O$ in the optimized conditions (solvents, temperatures, and reaction times) was described in the second part.



Key words: Thiophene, diyne, ynamide, optoelectronics.

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AN EFFICIENT SYNTHESIS OF NOVEL DISPIROINDOLIZIDINE DERIVATIVES *VIA* ONE-POT THREE-COMPONENT 1,3-DIPOLAR CYCLOADDITION REACTION

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Indolizidines with different degrees of unsaturation constitute the core structural element found in a large family of alkaloids that occupy an important and privileged position in modern organic chemistry due to their remarkable and attractive pharmacological properties [1]. On the other hand, thiazolidinone derivatives are fascinating scaffolds which are found in a wide range of naturally occuring alkaloids and synthetic products exhibiting a variety of biological and pharmacological activities [2].

In the continuity of our studies on the development of cycloaddition reactions [3], we present herein the synthesis of an extended series of novel dispiroindolozidine derivatives through three-component 1,3-dipolar cycloaddition of *(Z)*-5-arylidene-1,3-thiazolidine-2,4-diones **1** with azomethine ylides generated *in situ* from isatin **2** and 1,2,3,4-tetrahydroisoquinoline **3** by 1,5-prototropic shift route. The structure of the spiroadducts **4** and **5** was confirmed by spectroscopic data. This methodology can be an ideal tool for the preparation of biologically active functionalized dispiroindolizidines.

Scheme

Key words: Three-component 1,3-dipolar cycloaddition, Dispiroindolizidines, Azomethine ylides.

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Syntheses, Vibrational Spectroscopy and photoluminescence properties of $NH_4HErP_3O_{10}$

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The triphosphate of $NH_4HErP_3O_{10}$ [1] was synthesized by the flux method. The obtained powder was identified by X-ray diffraction (XRD) powders, IR spectroscopy, RAMAN spectroscopy and photoluminescence. Indeed, the $NH_4HErP_3O_{10}$ compound crystallizes in the triclinic system (space group P-1), the cell parameters are: a = 6.956 (9) Å; b = 7.678 (8) Å; c = 8701 (2) Å; α = 106.38 (9) °; β = 105.86 (7) °; γ = 82.63 (7) °; and Z = 2 (PDF ICDD- No. 046-0280). The shape of the bands observed on both FTIR absorption and Raman spectra is characteristic of triphosphate of $NH_4HErP_3O_{10}$. The absorption spectrum of the Er^{3+} ion in $NH_4HErP_3O_{10}$ which is recorded in the [230 nm -1800 nm] range allowed us to determine the levels of excited energy and assign transitions of the fundamental $^4I_{15/2} \rightarrow$ excited levels. The value of the energy gap was determined. The fluorescence spectrum of the Er^{3+} ion in $NH_4HErP_3O_{10}$ is recorded in the visible and IR region (λ_{ex} = 488nm). The appearance of bands which are centered at 548 nm, 659 nm and 1530 nm is assigned to the electronic transitions $^4S_{3/2} \rightarrow {}^4I_{15/2}$, $^4F_{9/2} \rightarrow {}^4I_{15/2}$ and $^4I_{13/2} \rightarrow {}^4I_{15/2}$, respectively. The excitation spectrum of the Er^{3+} ion in $NH_4HErP_3O_{10}$ is recorded in the UV-visible domain (λ_{em} = 659nm). All the bands are characteristic to the ion Er^{3+}. The decay time of the $^4F_{9/2}$ (659nm) level under λ_{ex} = 488nm was determined.

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VO_2(B) NANOSPHERES FOR VISIBLE LIGHT PHOTOCATALYSIS

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The exploration of new photocatalyst materials with high photocatalytic efficiency under visible light have been considered to be one of the important investigative fields for the exploitation of visible solar radiation in the solar energy conversion [1], and for the elimination of various organic pollutants in aqueous phase as well as in gaseous media [2-3].
Herein, we reported a general, efficient, and environmentally friendly synthetic strategy for obtaining VO_2(B) nanospheres via a simple one-step solvothermal method. The crystalline phases, the size and the morphology of the as-synthesized samples were identified by x-ray diffraction analysis and scanning electron microscope, respectively. The structural and optical properties were studied by Fourier transform infrared and diffuse reflectance spectroscopy. The texture and surface analysis of the nanospheres were examined by Brunauer-Emmett-Teller gas adsorption method. The photocatalytic activity of mesostructured nanospheres has been investigated by evaluating the discoloration/degradation of methylene blue (MB) under visible light irradiation. The nanospheres exhibited enhanced photocatalytic activity in degradation of MB compared with the bulk vanadium pentoxide. Indeed after 135 min of reaction, the nanospheres were able to degrade about 92% of the MB in solution while bulk V_2O_5 presents a degradation percentage of about 44 %. The enhanced photocatalyst activity was assigned to the high performance of absorption in the visible range and their spherical morphology. In fact, nanospheres can promote the separation efficiency of electron-hole pairs, thus improving their response to visible light.

Key words: nanospheres, vanadium dioxide, photocatalysis, solvothermal.

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Superprotonic behavior of the new solid acid: $K_{0.47}(NH_4)_{0.53}H_2(PO_4)_{0.52}(AsO_4)_{0.48}$

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The new compound $K_{0.47}(NH_4)_{0.53}H_2(PO_4)_{0.52}(AsO_4)_{0.48}$ crystallizes in the tetragonal system $I\bar{4}2d$ with lattice parameters a = 7.606(5) Å and c = 7.401(5) Å. A calorimetric study of the title compound shows two distinct endothermal peaks which are detected at 248 and 490 K. Samples were examined by impedance and modulus spectroscopy techniques. The first transition (248 K) is attributed to a antiferroelectric -paraelectric type. A high temperature phase transition (490 K) leading to a superionic-protonic phase was found, characterized by an unusual high conductivity. The conductivity relaxation parameters associated with the high disorder protonic conduction have been determined from analysis of the M'/M''_{max} spectrum measured in a wide temperature range. Transport properties of this material appear to be due to the proton hopping mechanism.

APPLICATION OF DOEHLERT MATRIX TO DETERMINE THE OPTIMAL CONDITIONS OF CHEMICALLY DEPOSITED BISMUTH SULFIDE THIN FILMS

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Bismuth sulfide (Bi_2S_3) is considered to be one of the attractive materials for photovoltaics, optoelectronics applications and thermoelectric cooling devices [1]. This is due to its small forbidden energy gap between 1.25 and 1.70 eV, near to the range of theoretically maximum attainable energy conversion efficiency [2].
The semiconductors thin films of bismuth sulfide were deposited by chemical bath deposition commonly called CBD on glass substrates at low temperature using triethanolamine as a complexing agent, bismuth nitrate and thiourea solutions in an aqueous basic medium. Optimization of the deposition conditions was carried out by the use of the experimental design methodology with Doehlert approach. Three principal experimental factors were investigated: bath temperature T (°C), time deposition t (min) and the ratio of the precursors concentrations ($R = \frac{[TU]}{[Bi]}$). The experimental response of interest is the ratio of bismuth moles to the bismuth sulfide moles present in the film ($n = \frac{nBi}{nBi2S3}$).
The chemical analysis of bismuth was performed by inductively coupled plasma atomic emission spectroscopy (ICP-AES). The accuracy and the calibration curve of the method were validated according to French Standard NF T 90-210. The prepared films were characterized using X–ray diffraction (XRD) and UV-Vis spectroscopy.
The optimal conditions found for having a near stoichiometric amorphous film of bismuth sulfide are T=84°C, t =86 min and R= 13. The bandgap energy was estimated to be 1.26 eV.

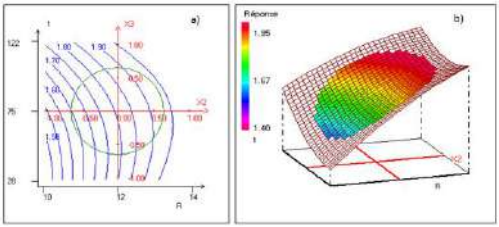

Figure: (a) Contour plots of time deposition t (min) versus ratio of the precursors concentration R at a fixed temperature T = 84°C;
(b) Corresponding three-dimensional plot.

Key words: Bismuth sulfide; Thin films; Chemical bath deposition; Doehlert Matrix.

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CORRELATION OF STRUCTURE AND ION CONDUCTION IN $La_2Mo_{2-y}S_yO_9$ ($0.1 \leq y \leq 0.3$) OXYGEN ION CONDUCTORS

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Research in the field of fast oxide-ion conductors is very active, due to their important types of application, among which solid oxide fuel cells (SOFCs), oxygen sensors, or oxygen pumping devices. For the aim to increase anionic conductivity at lower temperature, Goutenoire et al. Discovered in 2000 a new family of fast O^{2-} conductors **[1, 2]**, which exhibit anionic conductivity comparable to that of stabilized zirconium (YSZ8%). The parent compound of this new family, hereafter called LAMOX, is lanthanum molybdenum $La_2Mo_2O_9$. Powder-solid state reaction route using La_2SO_6 as sulfur source was used to prepare compositions of the solid solution $La_2Mo_{2-y}S_yO_9$. This isovalent substitution suppress the phase transition which occurs in $La_2Mo_2O_9$ around 580°C from a low temperature α monoclinic ordered form (space group $P2_1$) to a high temperature (more conducting) cubic form **[3]** and stabilize the β-type disordered form at room temperature from and above y = 0.1 (5 mol % S). Single phases were only obtained in the substitution range extending up to y = 0.3 (15 mol % S). The solid solution $La_2Mo_{2-y}S_yO_9$ has been studied using temperature - controlled X-ray powder diffraction, differential thermal analysis, thermogravimetric analysis, complex impedance spectroscopy such as Infrared spectra, Raman diffusion and electric analysis. In fact, the influence of the amount of sulfur on the transport mechanisms is studied in both low-temperature (Arrhenius) and high-temperature (Vogel –Tammann−Fulcher) regimes. All the S series (y= 0.1, 0. 2, 0.3) follows a cubic symmetry (space group $P2_13$) and a regular Vegard-type evolution of crystallographic parameters.

Key words: LAMOX, X-ray powder diffraction, phase transition, Ionic conductivity, Infrared, Arrhenius and Vogel –Tammann−Fulcher

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ORGANIC TEMPLATED METAL HALIDES: STRUCTURE, THERMAL BEHAVIOR AND MAGNETIC PROPERTIES

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Two organic templated metal halides with the general formula $(C_6H_9N_2)_2[M^{II}Br_4]$ with M^{II} = Cu (compound 1) and Zn (compound 2) were synthesized by the slow evaporation method. The grown crystals were subjected to the following characterization techniques: single crystal X-ray diffraction, thermal analysis (ATG-TD) and temperature dependent magnetic susceptibility. The crystals of $(C_6H_9N_2)_2[CuBr_4]$ belong to the triclinic$P\bar{1}$space group, whereas $(C_6H_9N_2)_2[ZnBr_4]$ crystallize in the orthorhombic system with Pbcn space group. Molecular structures of the coordination compounds consist of $[MBr_4]^{2-}$ anions and 2-amino-6-methylpyridine cations linked together via non-covalent interactions essentially hydrogen bonding, π···π stacking and Br···Br interactions which lead to three-dimensional supramolecular architecture. The thermal decomposition curves reveal two steps weight loss giving finally metal oxide. Compound 1 exhibits a paramagnetic behavior with weak antiferromagnetic interactions at low temperature.

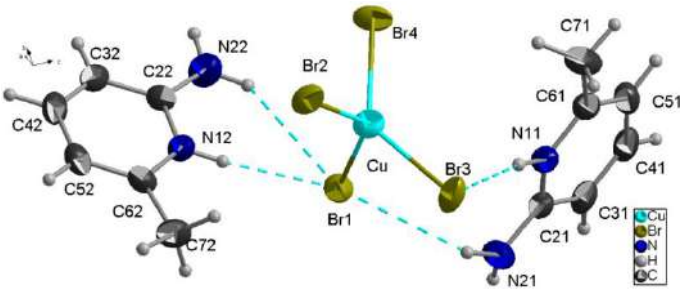

Fig: The asymmetric unit of 1 showing the atom-numbering scheme involving N-H···Br hydrogen bonding.

Key words: Metal halides, crystal structure, intermolecular interactions, thermal behavior, Magnetic properties.

Synthesis, structural study and simulation of conduction path of $Na_{0.45}K_{1.55}Cu_3(MoO_4)_4$ compound

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Single crystals of a new triple molybdate, sodium potassium copper(II) tetra--[molybdate(VI)], $Na_{0.45}K_{1.55}Cu_3(MoO_4)_4$ have been synthesized by a solid-state reaction and its crystal structure has been determined. The title compound crystallizes in the monoclinic space group $P2_1/c$ with a = 5.097 (2) Å, b = 14.594 (3) Å, c = 19.905 (3) Å, β = 94.00 (2)°, V = 1476.9 (7) ($Å^3$), Z = 4, $R(F^2)$ = 0.025 and $wR(F^2)$ = 0.063. K2 and Na1 cations are located at the same general site with occupancies of 0.55 and 0.45 respectively. The structure was formed by infinite $Cu_3Mo_3O_{23}$ type ribbons. These ribbons are connected by tridentate tetrahedron which shared vertices to built layers disposed parallel to (001) plan. All of these layers connected by vertices to form a three-dimensional framework. The structure model is supported by bond-valence-sum and charge-distribution CHARDI methods [1-2]. The most likely conduction paths of monovalent alkali cations was studied using the bond-valence-sum (BVS) model. A structural relationship has been established between the framework of one-dimensional $Rb_2Cu_2(MoO_4)_3$ structure [3], the two-dimensional structure $Ag_2Cu_2(MoO_4)_3$ [4] and our three-dimensional structure.

Key words: BVS, CHARDI, Conduction paths.

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EFFECT OF IMPREGNATION pH ON THE CATALYTIC ACTIVITY OF CuO / CeO_2 / $MgAl_2O_4$ IN CO-PROX

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In general, the catalytic activity of a catalyst depends on several factors such as the nature of the support, the addition of the promoter, the particle size and the nature of the pretreatment. Various catalysts have shown good performance for CO-PROX process. Copper-ceria based catalysts have been widely used in this process. It has been shown that their catalytic properties depend strongly to the characteristics of contact established between the constituent oxides [1].
The objective of this work is to highlight the influence of pH preparation of CuO / CeO_2 / $MgAl_2O_4$ catalyst on its catalytic activity in CO-PROX. Therefore, the CuO / CeO_2 / $MgAl_2O_4$ catalyst was prepared by the impregnation method, at three different pH values: 4, 8 and 10.
Comparing the CO conversion curves and selectivity of CO_2 of the three prepared catalysts during the CO-PROX oxidation reaction it is possible to conclude that catalyst prepared at pH = 8 gives the better catalytic activity as shown in Figure 1. The differences in behavior of the three prepared catalysts were explained based, firstly, on the type of contacts between the three oxides and secondly on the analysis results of their catalytic activities complemented by operando-DRIFTS [2].

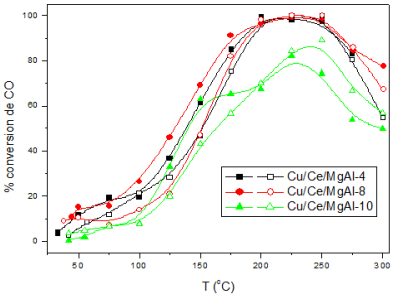

Figure1: CO conversion of CuO/CeO_2/$MgAl_2O_4$ prepared at pH=4, 8 et 10

Keywords: CuO/CeO_2/$MgAl_2O_4$, CO-PROX; catalytic activity, DRIFTS.

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ADVANCED $Pd/Ce_xZr_{1-x}O_2/SBA-15$ CATALYSTS FOR METHANE COMBUSTION FROM BIOHYBRID NANOMATERIALS

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For applications in gas turbines and boilers, there is an urgent need for the development of effective and stable catalysts for natural gas combustion. Palladium has been reported to be the most active catalyst for methane combustion when operating in excess of oxygen [1]. Palladium dispersion and palladium-support interaction play an important role on catalytic performance [2]. In addition, cerium and zirconium are effective promoters for noble-based catalysts because they can enhance dispersion, stability and oxygen storage capacity [3]. In this work, SBA-15 is used as a support due to its relative high surface area and organized mesoporous structure. The addition step of bio-polymer is changed in order to modify the Pd-support interaction and consequently the structure of the active phase.

The $Pd/Ce_xZr_{1-x}O_2/SBA-15$ catalysts are prepared by sol-gel method with palladium acetate, cerium nitrate, zirconium oxynitrate, Pluronic acid P123, tetraethylorthosilicate (TEOS) and a bio-polymer as precursors. The bio-polymer introduction step during the catalyst synthesis is changed from the sol-gel solution (Pd1) to the dried gel (Pd2) or to the calcined catalyst (Pd3). N_2-physisorption, UV-visible, H_2-chemisorption, MET-EDX, DRX and TPR are the main techniques used to characterize the prepared solids. Furthermore, total methane oxidation is used to test their catalytic activity.

According to N_2-physisorption and H_2-chimisorption results, better BET surface area and higher metal dispersion are obtained on Pd3 compared to Pd1 and Pd2. The XRD, UV-Visible, MET-EDX, and TPR results show that the formation of $Ce_xZr_{1-x}O_2$ solid solution, the $Ce_xZr_{1-x}O_2$ particle size, the the $Ce_xZr_{1-x}O_2$ crystallisation, the reduction temperatures of the PdO phase depends greatly on the bio-polymer introduction step probably due to the different Pd-support interactions.

The catalytic activity results show that the bio-polymer introduction step modifies significantly the activity of the catalytic sites (see Fig.1). Globally, the improvement of palladium dispersion and PdO reducibility increases the activity in methane combustion.

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SYNTHESIS, CRYSTAL STRUCTURE AND PHYSICOCHEMICAL STUDY OF $[p\text{-}(NH_3)C_6H_4NH_3]_3P_6O_{18}.6H_2O$

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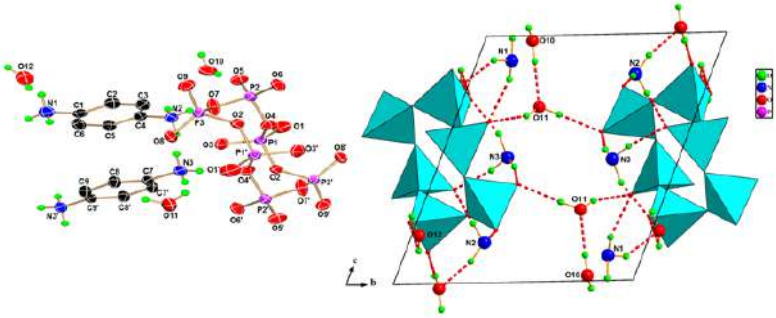
This communication reports synthesis, crystal structure and physiocochemical study of the new hybrid $[p\text{-}(NH_3)C_6H_4NH_3]_3P_6O_{18}.6H_2O$ prepared in aqueous solution. This compound crystallizes in a triclinic $P\bar{1}$ unit cell with a = 9.269(2) Å, b = 9.922(19) Å, c = 11.061(2) Å, α = 65.652(18)°, β = 74.073(17)°, γ = 76.325(17)° and V = 882.3(3) $Å^3$, Z = 1. Its centrosymmetrical structure shows two non-identical organic cations and a symmetrical $P_6O_{18}^{6-}$ anion which has a chair configuration. Cohesion of these species is ensured by hydrogen bonding and π…π stacking interactions. Thermal stability and spectroscopic properties of this hybrid organic-inorganic material are given.



Disposition and organization of all components of the structure of $[p\text{-}(NH_3)C_6H_4NH_3]_3P_6O_{18}.6H_2O$

Key words: Crystal structure, Hybrid material, Cylohexaphosphate, Spectroscopic study.

Bioactive Fluor-glasses for use as Bone substitute: Effect of Spark Plasma Sintering process on the Microstructure and Mechanical Characteristics

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To improve the mechanical properties of bioactive fluor-glasses compacts were produced by a non-conventional technique, spark plasma sintering (SPS). This original method was used to consolidate such glass powders at non-conventional, very low temperatures (T < 450°C) so as to preserve the vitreous structure. The fluor-glasses (Fx) have been successfully conducted by spark plasma sintering process, resulting in a dense bioactive glass compacts. Besides, the mechanical behaviour of glass powders was ameliorated according to the fluorine amount added in the vitreous network.
The microstructure, Vickers microhardness and density are described. Finally, the resulting mechanical properties determined by SPS are significantly higher than those usually reported in the literature.

Key words: Bioactive Glasses, Fluor, Density, mechanical behaviour, SPS, Bone substitute.

Synthesis, structure, Hirshfeld surface analysis and infrared spectroscopy of a novel organic-inorganic hybrid material $[C_8H_{14}N_2]SO_4$

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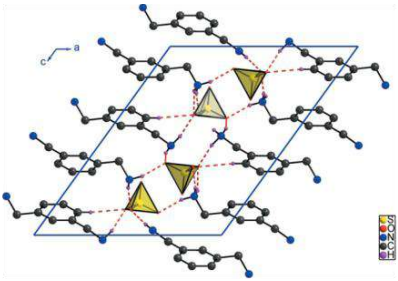
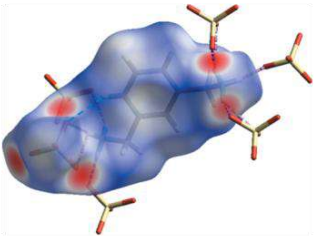
A new organic-inorganic hybrid material denoted [1,3-phenylenebis(methylene)]bis(azanium) sulfate, $(C_8H_{14}N_2)(SO_4)$ has been synthesized and the structure was determined by single crystal X-Ray diffraction analysis. This compound has been crystallized in the monoclinic system, space group $P2_1/c$, with a = 12.841(1) Å, b = 6.0989(5) Å, c = 15.9642(9), β = 125.791(4) °, V = 1014.15(13) A^3 and Z = 4. Its atomic arrangement can be described as infinite (100) sheets of alternating organic and inorganic entities, the m-xylylenediaminium cations are connected to the sulfate anions by N—H…O and asymmetric bifurcated N—H…(O,O) hydrogen bonds, generating a three-dimensional network. A weak C—H…O interaction also occurs. The Hirshfeld surface analysis and the two-dimensional fingerprint maps indicate that the packing is dominated by H…O/O…H and H…H contacts. The infrared spectrum of this crystal reported from 4000 to 400 cm^{-1} confirmed the presence of the principal bands assigned to the internal modes of the organic cation and sulfate anion.



Projection of $(C_8H_{14}N_2)(SO_4)$ along the b axis. H atoms not involved in hydrogen bonding have been omitted.



Hirshfeld surface mapped over d_{norm} of $(C_8H_{14}N_2)(SO_4)$.

Keywords: crystal structure, m-xylylenediaminium, sulfate, hydrogen bonding, Hirshfeld surface analysis, fingerprint maps.

SYNTHESIS, CRYSTAL STRUCTURE AND PHYSICOCHEMICAL PROPERTIES OF NEW ORGANOAMMONIUM DIPHOSPHOPENTAMOLYBDATE (VI): $(C_6H_{14}N)_4(NH_4)_2[P_2Mo_5O_{23}]\cdot H_2O$

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The reaction of Cyclohexylamine with ammonium molybdate tetrahydrate and phosphoric acid yields the title compound: $(C_6H_{14}N)_4(NH_4)_2[P_2Mo_5O_{23}]\cdot H_2O$ (1). This diphosphopentamolybdate compound was structurally characterized by single crystal X-ray diffraction, energy dispersive analysis, IR, thermal stability analysis, cyclic voltammetry analyzes and UV–Vis. The crystal structure of (1) also contains the polyanion units $[P_2Mo_5O_{23}]^{6-}$ around the $(C_6H_{14}N)^+$, $(NH_4)^+$ cations and lattice water. The $[P_2Mo_5O_{23}]^{6-}$ subunits and cations are connected together through hydrogen bond. The crystal shape morphology was simulated using the Bravais–Friedel–Donnay–Harker model.

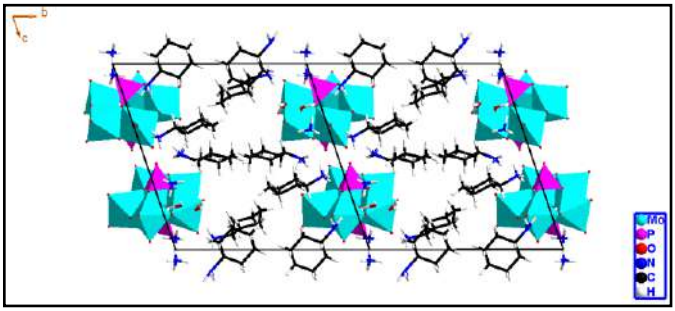

Fig. View along the a axis of compound (1)

Key words: Diphosphopentamolybdate, Synthesis, Crystal structure, Characterizations, Crystal Morphology.

HYDROTHERMAL SYNTHESIS AND CHARACTERIZATION OF A NEW ORGANIC-INORGANIC HYBRID DAWSON POLYOXOTUNGSTATE

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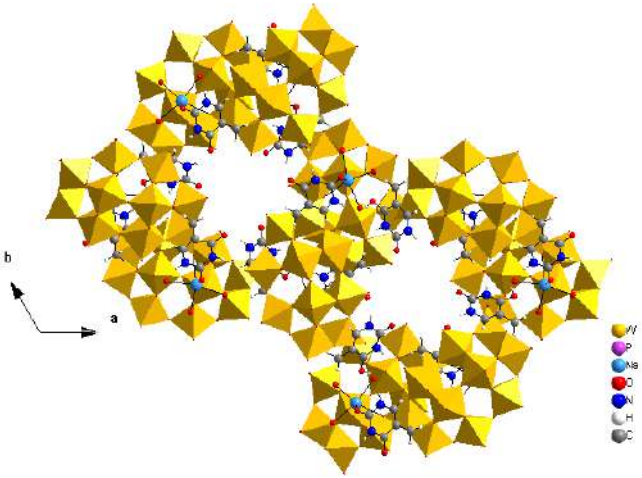
A novel organic–inorganic hybrid compound based on Wells-Dawson polyoxotungstate, $Na(C_5H_6N_2O_2)_4[HP_2W_{18}O_{62}]$ (1) have been isolated by the conventional solution method and characterized by single-crystal X-ray diffraction, infrared, ultraviolet spectroscopy and thermogravimetric analyses. This compound crystallized in the hexagonal system, space group P63/m, with a = b =27.992(1) Å, c = 21.248(3) Å, γ = 120(7)°, V = 14418.4(1)Å^3 and Z = 12. The crystal structures of the compounds exhibit three-dimensional supramolecular assembly based on the extensive hydrogen bonding interactions between organic cations, water molecules and Wells-Dawson polyoxoanions.

Keywords: Polyoxotungstate hybride Well-Dawson type, hydrothermal condition.



A packing view of $Na(C_5H_6N_2O_2)_4[HP_2W_{18}O_{62}]$ *along the c axis*

SEMI-HARD MAGNETIC PROPERTIES OF NANOPARTICLES OF COBALT FERRITE SYNTHESIZED BY THE CO-PRECIPITATION PROCESS

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The nanoparticles ferromagnetic materials $CoFe_2O_4$ have been synthesized by the co-precipitation method and annealed at various temperatures from 773 to 1223 K. The influence of the heat treatment on the structural, morphological and magnetic properties of $CoFe_2O_4$ nanoparticles was investigated. The structure, morphology and magnetic properties of the annealing samples were reached by mean of X-ray diffraction (XRD), scanning electron microscopy (SEM) techniques and measurement of hysteresis loop. The average crystallite size, lattice parameter and X-ray density were calculated from X-ray diffraction data. All samples have the cubic spinel structure with average crystallite sizes increasing from 30 to 178 nm with the temperature. The measurements of the hysteresis loop, performed at both 10 K and 300 K, indicate a ferromagnetic behavior of nanoparticles. Also the measurement at 300 K shows that the sample annealed at 1023 K is magnetically harder than the other samples. The coercive fields are 1.58 kOe against the values inferior at 0.61 kOe for the other samples. Indeed, the remanent magnetization is 31.85 emu.g^{-1} for the hard magnetic sample against 13.02 and 17.29 emu.g^{-1} for the soft magnetic samples annealed at 773 and 1223 K, respectively. The origin of the hard-soft magnetic properties of nanoparticles $CoFe_2O_4$ is discussed in terms of the co-existence of secondary phases β-Fe_2O_3, CoO_2 and the distribution of Fe^{2+} and Co^{2+} ions within the lattice.

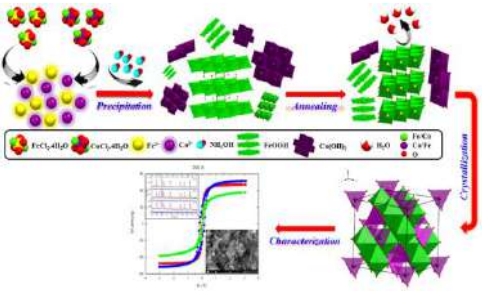

Fig. Representative diagram of the formation of the ferromagnetic nanoparticles of $CoFe_2O_4$

Key words: Nanoparticles, $CoFe_2O_4$, DRX, hard-soft magnetic, Ferromagnetic.

Microstructure and magnetic properties of nanostructured $(Fe_{0.75}Al_{0.25})_{100-x}B_x$ alloys prepared by mechanical alloying

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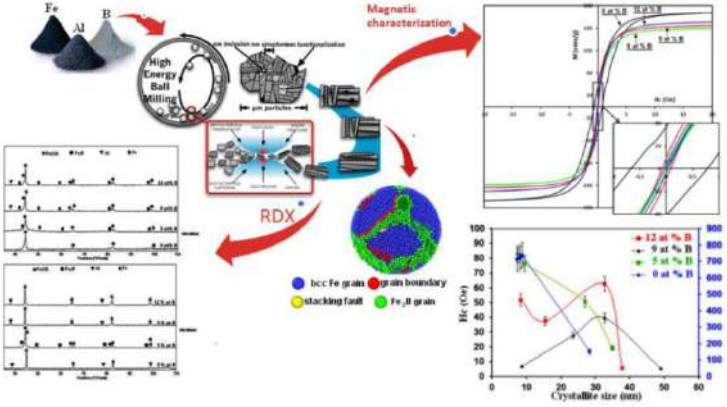
New magnetic nanocrystalline powders $(Fe_{0.75}Al_{0.25})_{100-x}B_x$ with (x=0, 5, 9 and 12 at %) have been elaborated by mechanical alloying. Fe–Al binary compounds have been widely studied due to their interesting magnetic properties from both fundamental and applied points of view [1]. The boron content and milling time affect the magnetic and structural properties of these alloys [2]. Morphological, microstructural, structural and magnetic characterizations of the powders milled several times were investigated by scanning electron microscopy, X-ray diffraction, vibrating sample magnetometer and SQUID techniques. Increasing the boron content, the formation of two crystalline phases were found, a majority one corresponding to a FeAl bcc phase and another one in low proportion, corresponding to a Fe_2B phase which needed more milling time [3, 4]. Increasing the milling time, the crystallite size decreases to the nanoscale, the microstrain increases and the lattice parameters of the disordered solid solution, increase. A decrease in coercivity (Hc) was observed with increasing the boron content. These variations are explained on the basis of crystallite size and strain variation in the samples during milling.



Keywords: FeAl alloy; High energy ball milling; X-ray diffraction; nanostructure; Magnetic proprieties.

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Synthesis, characterization and thermochemical study of "A" type carbonate phosphocalcium hydroxyapatites

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Apatites are a large class of mineral compounds with the general formula $M_{10}(XO_4)_6Y_2$, where M is generally a divalent cation (Ca^{2+}, Sr^{2+}, Ba^{2+}, Pb^{2+}, etc), XO_4 is a trivalent or tetravalent anion (PO_4^{3-}, VO_4^{3-}, AsO_4^{3-}, SiO_4^{4-}, etc) and Y can be a halide, hydroxyl or carbonate group.

Incorporation of carbonate ion in hydroxyapatite can occur in two different sites replacing either OH^- or PO_4^{3-}, leading to "A" or "B" type carbonate apatite respectively. The physico-chemical properties of these materials are highly affected by the carbonate localization and the chemical composition.

"A" type carbonate phosphocalcium hydroxyapatites having the general formula $Ca_{10}(PO_4)_6(OH)_{(2-2x)}(CO_3)_x$ with $0 \leq x \leq 1$, were prepared by solid gas reaction in the temperature range of 700-1000°C. The obtained materials were characterized by X-ray diffraction and infrared spectroscopy. The carbonate content has been determined by CHN analysis.

The heat of solution of these products was measured at T = 298 K in 9 wt% nitric acid solution using an isoperibol calorimeter. A thermochemical cycle was proposed and complementary experiences were performed in order to access to the standard enthalpies of formation of these phosphates. The results show a decrease of this quantity as the ratio of substitution increases.

Estimation of the entropy of formation allowed the determination of standard Gibbs free energy of formation of these compounds. The study showed that the substitution of hydroxyl by carbonate ions contributes to the stabilisation of the apatite structure.

Key words: Carbonate hydroxyapatites; Isoperibol calorimeter; Heat of solution; Enthalpy of formation; Entropy of formation; Gibbs free energy of formation.

^{210}Po concentration of cigarettes consumed in Tunisia and their estimated dose contribution due to the smoking

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Almost 90% of pulmonary cancer death cases can be assigned to smoking [1]. Tobacco smoke contains several carcinogenic components; different radionuclides of natural origin are present in a significant quantity besides the considerable quantity of toxic chemical compounds [2].
Among the several poisonous chemicals found in cigarettes, polonium-210 is the major radioactive element of interest because it can be inhaled with cigarette smoke due to its low volatilization temperature.
In this study, the polonium 210 activity concentrations of cigarettes were investigated for 13 brands of cigarettes consumed in Tunisia. This natural radionuclide alpha emitter was determined by alpha spectrometry using PIPS, a silicon semi-conductor, after chemical treatment and spontaneous deposition in stainless disc.
The activity of 210 Po was calculated and the results showed concentrations ranging from 11,77 to 25,31 mBq per gram of dry tobacco. These values indicate that tunisian tobacco contains lower concentration of polonium in comparison with tobacco from other countries.
The average annual committed effective dose for smokers was calculated using the dose per unit intake conversion factors and is estimated to be 106,53µSv for 20 cigarettes per day for Tunisia.

Key words: Polonium 210; Radionuclide; Radiochemical analysis; Alpha Spectrometry; Cigarette smoking; Radiation Dose.

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CORROSION BEHAVIOR OF TITANIUM OXYHYDROXIDE GEL ELECTRODEPOSITED BY CHRONOAMPEROMETRY ON A PURE COPPER SUBSTRATE

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Objectives:
- Electrochemical elaboration of titanium oxyhydroxide $TiO(OH)_2$ films on pure copper by chronoamperometery.
- Determination of physical and chemical properties of the deposits and evaluation of its corrosion resistance in a 3% NaCl medium.

Techniques:
- Electrodeposition of $TiO(OH)_2$ titanium oxyhydroxide gel on pure copper (1 cm^2 surface area) was carried out in an aqueous bath of titanium oxysulfate $TiOSO_4$ by chronoamperometry by imposing a potential of -1852 mV vs. Hg / Hg_2SO_4, saturated K_2SO_4 for one hour.
- Scanning electron microscopy (SEM) associated to an energy dispersive spectroscopy (EDS) were used to study the gel morphology and related composition distribution. Additionally, the X-ray fluorescence spectrometer (XRF) was used to determine the approximate composition of the gel.
- The corrosion test was performed in a 3% NaCl medium by using potentiodynamic polarisation and electrochemical impedance curves.

Results:
- The SEM observations of the coated copper samples show that a thin films of a $TiO(OH)_2$ gel with fewer cracks cover partially the surface of copper.
- The comparative study of the electrochemical behavior of copper with and without $TiO(OH)_2$ coatings in chloride media shows that despite of the fact that the deposit is unstable and is partially dissolved during immersion in 3% NaCl, there is an amelioration of corrosion potential and attenuation of peak intensities (relating to the formation of cuprite Cu_2O and cuprous oxide CuO) of the coated sample in camparison with bare copper.

Keywords: electrodeposition, titanium oxyhydroxide gel, copper, chronoamperometry, corrosion resistance.

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PHOTO-FENTON TREATMENT OF NICOTINIC ACID WASTEWATERS

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Aromatic compounds in the form of pyridine base are used in pharmaceuticals. On example can be the nicotinic acid which is a pyridine compound substitued with carboxylic group. Nicotinic acid is used as a drug, hence the major water contamination by this acid comes from its use as a drug.
In this work, the degradation of nicotinic acid in aqueous solutions by photo-Fenton process has been investigated. The main variables affecting the photo-Fenton processes as well as initial pH, Fe^{2+} dosage and H_2O_2/Fe(II) mole ratio were investigated, respectively to evaluate their influence on the efficiency of the process. The effects of these experimental parameters on the kinetics and on the efficiency of the degradation have been evaluated. Under optimal conditions, photo-Fenton process achieved more than 97% of TOC removal.

Key words. Photo-Fenton process, Nicotinic Acid wastewaters, TOC removal, hydroxyl radicals.

Green synthesis of zinc oxide (ZnO) nanoparticles using *Lauris nobilis* plant extract and their optical properties

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The present study involves green synthesis of ZnO nanoparticles (Nps) using aqueous *Lauris nobilis* plant extract as fuel (green chemistry process). The obtained ZnO Nps were characterized by UV–Vis spectroscopy, Photoluminescence (PL), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscope (SEM) analysis. Powder XRD studies indicated the formation of pure Wurtzite structure with maximum absorption of 375 nm corresponding to band gap energy of 3.30 eV. Photoluminescence study revealed the blue emission at 402, 447, 469 and 483 nm and the green emission at 529 nm respectively, upon excitation at 360 nm because of oxygen deficiencies and zinc interstitials. We suggested that plants represent an important bioresource of ZnO (Nps).

Keywords: Green synthesis,optical properties, ZnO nanoparticles, *Laurus nobilis* extract

OPTIMIZATION OF MORPHOLOGY AND PERFORMANCE OF FINNED HOLLOW FIBER FOR DIRECT CONTACT MEMBRANE DISTILLATION USING COMSOL MULTIPHYSICS

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The performance of membrane distillation depends on both membrane and module characteristics. This study explores the design of hollow fiber membranes used in direct contact membrane distillation (DCMD) to enhance Temperature Polarization Coefficient (TPC) and permeation flux.
Three different hydrophobic PVDF hollow fiber membrane respectively with 2, 4 and 6 fins were carried out with a series of three-dimensional numerical simulation using COMSOL Multiphysics. The simulation results showed that the membrane with four fins had an improvement of 32% in TPC compared to the hollow fiber membrane with two and six fins.
The enhancement of permeation flux was up to 2-fold for the design of membrane with four fins.

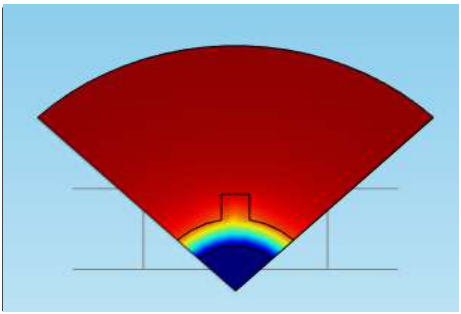

Fig. Temperature profile in the (xy) direction for hollow fiber membrane with four fins.

Key words: Membrane distillation, Finned hollow fiber, optimization, DCMD, numerical simulation.

OTOCATALYTIC DEGRADATION OF ORGANIC POLLUANTS IN AQUEOUS SOLUTION: KINETICS AND ANALYTICAL STUDIES

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Nowadays, the chemical compounds such as pesticides, pharmaceuticals, veterinary, drugs, cosmetics, dyes… have been extensively used and their amount has increased drastically. Owing to their potential persistence, toxicity, carcinogeniticity, their presence in the various environmental compartments such as soils, atmosphere and surface waters have raised increasing concerns and interest. Therefore, their environmental fate, namely their degradation processes, transfer and the elucidation of the transformation metabolites are of great interest. A large number of studies are available regarding their degradation, fate and behavior in different compartments such as water, soil and food with the aim to reduce their potential negative effect. Moreover, it has been found that some of the xenobiotics are highly persistent and that their complete elimination is difficult to achieve with the existing water treatment facilities.

Currently, heterogeneous photocatalysis is a process that is growing rapidly in environmental engineering and for removing organic pollutants such as pesticides from water. In this present work, we are interested in the degradation of chloridazon, an herbicide of the family of pyridazinones that present high water solubility by heterogeneous photocatalysis using titanium dioxide TiO_2 under excitation at 365 nm.

The main objective is to optimize the conditions for total degradation or mineralization of the solution in order to decontaminate the water. The aim of this work was the assessment of the impact of different parameters on photodegradation of chloridazon. TiO_2 P25 degussa was used as a catalyst and photodegradation occurred in the aqueous solution under excitation at 365nm. The effect of different TiO2 concentrations, herbicide concentrations and irradiation times were investigated. The analyzeswere performed by (HPLC). Similarly, it has been shown that the ions Cl^-, SO_4^{2-}, NO_3^-, CO_3^{2-} have an effect on the photocatalytic degradation on the basis of the results. We concluded that this pollutant disappeared completely after 20 minutes of irradiation. The structure elucidation was performed by using HPLC/ESI/MS and HPLC/ESI/MS^2 techniques in positive mode and through the complete study of the various fragmentation pathways.

Keywords : Chloridazon – degradation – Heterogeneous photocatalysis – TiO

STUDY OF COPPER CORROSION AND INHIBITION IN COOLING SEA WATER SYSTEAM

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Copper has a long history of service in marine and salt water systems, heat exchangers, because of its good machinability, high thermal and electrical conductivity and exceptional corrosion resistance, however under severe conditions, it can corrode at a very high speed [1]. Among several means of protection against the corrosion of copper, the addition of inhibitors in the medium is the most common method to slow down this phenomenon. In this study Na_3PO_4 has been studied. The aim of the present work is first to study the electrochemical behavior of copper in different synthesized environments at different temperature, and evaluate in second place pure copper inhibition mechanism in a synthesized marine environment and in Gabes sea water used in the cooling circuits of Gabes chemical group. The study was conducted from stationary and transient electrochemical techniques: Cyclic Voltammetry (CV), Potentiometry (OCP) and Electrochemical Impedance Spectroscopy (EIS), complemented by simple surface analysis methods; An Energy Dispersive X-Ray (EDAX) And Scanning Electron Microscope (SEM).

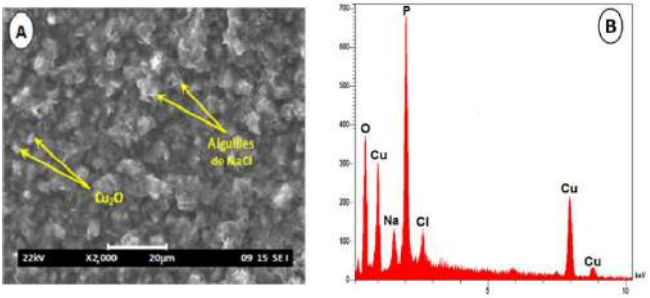

Fig. (a)Morphology of the sample surface in a scanning electron microscope, (b) Qualitative and quantitative EDX analyses of copper electrode surface in 3%NaCl and inhibitor

Key words: Copper, corrosion, sea water, inhibitors, electrochemical techniques.

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EFFECT OF PARTIAL REPLACEMENT OF PORTLAND CEMENT POWDER FROM '*PHOENIX DACTYLIFERA L.*' STONES ON THE MECHANICAL AND CHEMICAL BEHAVIOR OF THE MORTAR

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Université de Monastir

The development of a binder industry mortar biomass originates from the urgency requested by the professionals of the environment and climate to develop an eco material that brings add value compared to ordinary binder Portland cement.

So we assessed the influence of the addition of the date stones (DS) from *'Phoenix Dactylifera L.'* on the mechanical properties of these specimens and chemical towards aggressive environment such as acids.

The aim of this experimental study is to compare the behavior of cementitious mortars developed from a mixture of CEM II / A-L 32.5 N and different proportions of DS and therefore highlight the influence of DS on the strength and durability of the mortars and try to bring a better understanding of the chemical reactions responsible for the increase of sustainability.

Techniques such as X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) were used to determine the structure of the new formulation.

After curing specimens at 28 days in water, they were immersed in the same acid solutions concentration of 5% (HCl, CH3COOH). Mass measurement tests were performed at different ages.

The results are used to demonstrate the beneficial effect on the compressive strength and the durability of such an addition of (DS), while having a retarder or a natural setting accelerator according to the addition rate.

Keywords: Sustainability, Mortar Biomass (Date Stones), compressive strength, chemical attack, setting time.

INFLUENCE OF FORMULATION ON THE OXIDATIVE STABILITY OF WATER-IN-OIL EMULSIONS

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Oxidation is ubiquitous in lipids and causes degradation of organoleptic and nutritional qualities of foods. Lipid oxidation depends on various parameters (temperature, light, transition metals, lipid dispersion state …) that have to be controlled during food processing and storage. In this context, lipid oxidation was followed by measuring the content of primary oxidation products, for lipids in bulk phase and in water-in-oil emulsions. Different edible oils were chosen for their contents of α-linolenic acid (18:3 n-3). Emulsions were formulated at varying polyglycerol polyricinoleate (PGPR)/distilled monoglycerides concentration ratios (surfactant ratio), with or without the presence of pro-oxydant metals or chelators in the internal aqueous phase. In all experiments, the aqueous volume fraction (40%) and the droplet mean diameter (1 µm) remained constant. Besides this study, an innovative and rapid method based on differential microcalorimetry was developed for monitoring the kinetics of lipid oxidation [1]. The oxidability of the studied oils was related to their content in α-linolenic acid according the following order: linseed oil> camelina oil> rapeseed oil> olive oil. The rate of lipid oxidation increased with the iron sulfate concentration in the internal aqueous phase. The iron valence or its substitution by copper had no significant impact on the oxidation kinetics. However, both chemical nature of the counter ion (molecular weight, chelating force) and proportion of PGPR surfactant used to stabilize the emulsions were influential parameters [2]. In general terms, our results suggest that surfactants at the water-oil interface do not prevent pro-oxidant species to interact with lipids in the continuous phase but that their organization at the oil/water interface is a key parameter for controlling lipid oxidation.

Key words: Lipid oxidation, Water-in-oil emulsions, Emulsion formulation, Conjugated diene hydroperoxides, thermal analysis, Microcalorimeter.

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EXPLOITATION OF A NATUREL DYE IN A THERMOPLASTIC POLYMER

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National School of Engineers of Sfax

The use of synthetic colors causes a lot of problems on the environment and on human health. Most of the dyes are toxic and responsible for pollution [1]. Their direct discharge into the environment is a major source of pollution. In order to minimize the dangers, natural dyes are used. Therefore, intense research in the recent past has focused on the search for dyes from plants, animals, insects and minerals [2]. In this work we are interested in the plastics industry that aims to seek an effective solution to minimize problems caused by plastics. So we try to apply a natural origin dye in these polymers to minimize their dangers.

Fig. Dye extracted from flowers of the *Acacia cyanophylla*

Key words: Acacia cyanophylla, natural dye, polymer

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[2] Zibbu and Batra, 2010

EXPERIMENTAL AND THEORETICAL STUDY OF ZINC(II) COMPLEXES WITH TRIOCTYLPHOSPHINE CHALCOGENIDES

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In the recent years, the preparation and investigation of well-defined nanocrystals of ME semiconductors (M= Zn, Cd or Hg, E= O, S or Se) have been the focus of considerable attention because of the ability to fine-tune their electronic and optical properties for possible applications in lasers and photovoltaic's [1]. These ME nanoparticles can be prepared from metal complexes with organophosphorus ligands [2]. In this work, zinc(II) complexes of the type $[Zn(TOPE)_4](ClO_4)_2$ and $[Zn(TOPE)_2Cl_2]$ (TOPE = Oct_3PE with E = O, S or Se)) have been synthesized and characterized by multinuclear (^1H, ^{13}C and ^{31}P) NMR, conductivity and IR techniques. The complex formation was confirmed by a coordination chemical shift of the ^{31}P NMR signals towards lower field compared to those of the free ligands. The spectroscopic data show that the ligand is coordinated to the zinc center through the chalcogenide atom of the P=E group. The use of the complex $[Zn(TOPSe)_2Cl_2]$ as a suitable single source for the preparation of TOPO-caped ZnSe will be presented and discussed. In addition, a geometry optimization through DFT/B3LYP theoretical study was carried out in order to support and complement the experimental data and to further investigate the nature of the chalcogenide-Zn interaction. The results show a good agreement between the experimental and theoretical data.

Fig. DFT/B3LYP optimized structures of $[ZnCl_2(TOPE)_2]$

Keywords: Phosphine chalcogenide, zinc complexes, NMR, DFT/B3LYP, ZnSe

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Kinetic study of the effect of organic acids on the anaerobic digestion of margin

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There are multistep reactions involved in anaerobic digestion for degrading complex biodegradable materials (1). In this study, the effect of for selected C1 to C4 organic acids on biogas production and microbial growth in anaerobic digestion was evaluated using batch assays. These C1 to C4 compounds included formic acid, acetic acid, propionic acid, lactic acid, pyruvic acid and butyric acid. Each organic acid was tested as a single substrate in individual batch experiment at different concentrations range from 1000 mg COD/L to 8000 mg COD in mesophilic conditions at a temperature 35 C.
The specific microbial growth rate was estimated using Monod and Haldane equations based on the coupled stoichiometric reactions between biogas production and microbial growth
Anaerobic digestion of acetic acid and formic acid presented self-inhibition with inhibition constants 677 and 2765, 15 mg COD/L ,respectively, while the other acids tested did not inhibit anaerobic digestion of the margin.
Among the organic acids tested, it was found that the acetic acid has specific maximum growth rate the highest (μ_{max} = 0.28 d-1). While butyric acid, propionic acid, lactic acid, pyruvic acid and formic acid have μ_{max} values: 0.077; 0.072; 0.13; 0.085 and 0.083 d-1 respectively.
The half saturation constant (K_S) found for the acetic acid and formic acid are low compared to previously proposed values indicating that the low affinity of microorganisms on these acids. According to the values of the constants inhibitions found for formic acid and acetic acid, it is found that the inhibitory effect of acetate is more intense than that of formic acid.

Key words: . Anaerobic digestion,. Methanogenesis,Monod and Haldane equations

RADIOLYSIS OF PHARMACEUTICALS IN AQUEOUS SOLUTIONS AND WASTEWATER

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Several pharmaceutical compounds have been detected in natural aqueous systems in many countries, especially anti-inflammatory medicines. In this purpose, the degradation efficiency of these compounds under gamma irradiation was evaluated using advanced oxidation process (AOP) as an alternative to conventional water treatment technologies. In fact, irradiation of aqueous solutions by ionizing radiation produces several reactive radicals, essentially hydroxyl radical (˙OH), to destroy recalcitrant organic pollutants in water.
Pharmaceuticals considered in this study are aqueous solutions of paracetamol, ibuprofen and aspirin at different concentrations 0.1-1 mmol/L, which were treated by application of irradiation doses from 3 to 10 kGy with 6.1 kGy/h rate.
The main parameters influencing irradiation performance are absorbed doses and initial concentrations. Significant modifications attributed to these parameters appeared in the variation of chemical oxygen demand (COD) removal, concentration of radio-induced radicals and the pH value changes of treated compounds. Variation curves of concentrations as a function of irradiation dose describing the experimental data and the required dose for elimination of these pollutants by ionization were released. Preliminary degradation mechanisms are suggested based on analytical study using spectroscopic and chromatographic techniques, namely EPR and HPLC. Spectroscopic results (EPR) indicated the transformation of irradiated compounds into quinones molecules. The kinetic study showed that the degradation is pseudo-first rate to the contaminant.
The obtained results revealed the destruction of pharmaceutical compounds until total degradation in optimal conditions (mineralization), which improve the efficiency of this process in water remediation. Finally, pilot plant and industrial scale irradiation facilities demonstrated the applicability of radiation technology on large scale.

Key words: Pharmaceuticals, AOP, Gamma irradiation, hydroxyl radical, EPR.

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Copper (II) uptake by natural adsorbents: Chitin, Chitosan and Chitosan-EDTA

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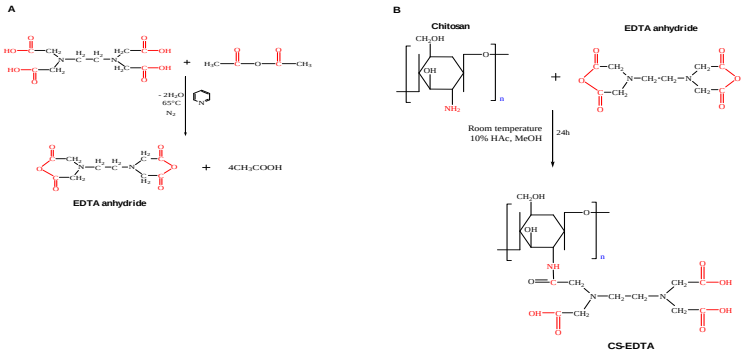
Wastewater contains dissolved copper ions released at different concentrations. This heavy metal in mineral effluent when exceeding trace concentrations is harmful and carcinogenic for living organisms **[1].** The copper ions should be removed from water or wastewater physically or chemically due to the fact that they are not biodegradable. Several techniques have been proposed for the removal of copper ions from wastewater and water including chemical precipitation, membrane filtration, ion exchange, electrolysis and adsorption **[2].** Chitosan is a linear cationic aminopolysaccharide obtained from chitin after deacytelation. It has been used in different fields such as textile **[3]**, drug delivery **[4]**, and environmental protection **[5]**. The present work focuses on the study of the copper uptake by natural adsorbents (chitin, chitosan and chitosan-EDTA ((scheme 1)) powders. Chitosan –EDTA has a significantly higher capacity of adsorption than chitosan and chitin due to the presence of chelating groups. The sorption of the metal by natural polymers was evaluated by intra particle diffusion model at different concentrations of Cu^{2+}; pH of the solution in the sorption process was studied. The sorption of Cu^{2+} by chitin, chitosan and chitosan-EDTA is strongly pH dependent, indicating an ion exchange mechanism.



Scheme 1: Synthesis of EDTA anhydride (A) and CS-EDTA (B).

Keywords: chitin, chitosan, sorption, copper, intraparticle, wastewater treatment.

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Electrochemical study of a conductive polymer

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In this work, thin films of polypyrrole were electrodeposited by cyclic voltammetry on FTO electrode. The developed material was characterized by infrared and Raman spectroscopy. An electrochemical study was achieved by modifying various parameters such as the monomer concentration, the concentration of the supporting electrolyte, the cycle number and the scanning speed. The surface analysis by scanning electron microscopy (SEM) shows a rough surface formed by agglomerates of differents sizes. [1] The transmission and reflection spectrum allowed us to determine the band gap energy of the elaborate thin films (Eg = 2,23eV) [2]. The resistance of the charge transfer was determined by electrochemical impedance spectroscopy where it is equal to 17,7 Ω.

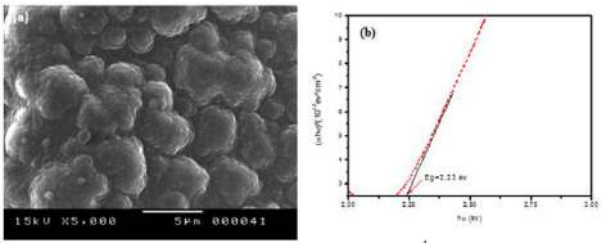

Fig. (a) MEB et (b) band gap energy

Key words: Thin films , Polypyrrole, Elecropolymérisation , cyclic voltammetry

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COMPLEXING PROPERTIES OF CALIX[4]ARENE AMIDE DERIVATIVES

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The complexing properties of three ligands was carried out in acetonitrile and monitored by UV spectrophotometry. Their considered targets were several heavy metal cations (Hg^{2+}, Cd^{2+} and Pb^{2+}) and trivalent lanthanides (La^{3+}, Nd^{3+}, Eu^{3+}, Gd^{3+} and Yb^{3+}). The three derivatives (**L1**, **L2** and **L3)** form mononuclear complexes with cations of heavy metals and lanthanides, except in the case of calixarene **L2**, which formed simultaneously mononuclear and binuclear species with Pb^{2+}. The results showed that complexation is the main factor affecting extraction with the two first ligands, in contrast with the third ligand.[1]

The assessment of the complexation equilibria were based on the use of UV spectrophotometry, although conductimetric measurements were also used to obtain preliminary estimations of the stoichiometry of the formed complexes.

Fig. Chemical stuctural of derivatives L1-L3.

Keywords: Calixarene, Complexation, Stability constant, Extraction.

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Program of Wednesday 21 December 2016

ISOLATION AND STRUCTURE ELUCIDATION OF TWO NEW PHENYLPROPANOIDS AND ANTIOXIDANT ACTIVITY FROM TUNISIAN *PITURANTHOS TORTUOSUS (APIACEAE)*

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Natural products from medicinal plants, as pure compounds, have attracted the attention in recent years because of their diverse pharmacological properties that are mainly due to their structural variability. *Pituranthos tortuosus* (family Apiaceae) is a small shrub without leaves. This aromatic plant, which grows naturally in North Africa, was selected and investigated for its bioactive compounds. Powdered of aerial parts were extracted successively in petroleum-ether, ethyl acetate and methanol using maceration method at room temperature. The antioxidant activity of methanol extract was evaluated by three complementary assays, including DPPH radical-scavenging activity, reducing power and metal chelating activity. This extract was then subjected to column chromatography and eluted with solvent mixtures of increasing polarity (petroleum ether, ethyl acetate and methanol) to isolate phytoconstituents. The structure of the isolated compounds was established using spectroscopic methods (UV, ^{1}H-NMR, ^{13}C-NMR, DEPT, HMBC, HSQC and COSY) as well as by comparison of their NMR data with those reported in the literature. Three compounds were isolated from the methanol extract of the aerial parts identified as mannitol, elemicin and 6-methoxyelemicin. Among these compounds, elemicin and 6-methoxyelemicin were isolated, for the first time, from the genus *Pituranthos* from the methanol extract.

Key words: *Pituranthos tortuosus*, NMR, Apiaceae, elemicin, phytoconstituent

POLYPHENOL CONTENT IN GLOBE ARTICHOKE [CYNARA CARDUNCULUS L. VAR. *SCOLYMUS* (L.) FIORI] AS AFFECTED BY GENOTYPE AND PLANT PART

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Globe artichoke is an ancient herbaceous plant native to the Mediterranean Basin in which it provides an important contribution to the agriculture economy. Artichoke heads are particularly rich in polyphenols whose therapeutic properties are well documented. The aim of the present study was to evaluate the effect of genotype and plant part on polyphenol content and antioxidant activity. The content of total polyphenols (TPC) was significantly affected by the two factors under study (genotype and plant part). The results indicated that the stem from "Blanc Oranais" variety contains the highest levels of total phenols, whereas flavonoids seem to be more abundant in the leaves. All samples analyzed showed a good antioxidant capacity using two different methods. The edible part of the three cultivars was associated with a lower EC_{50} than either the floral stems and bract or the leaves. These results suggest that globe artichoke by-products might represent a potential source of natural compounds, which could be used as nutraceuticals or ingredients in the design of functional foods.

Key words: Total polyphenol, flavonoid, antioxidant, globe artichoke, plant part, genotype

Chemical and biological investigation of essential and fixed oils isolated from *Acacia cyclops*, *A. mollissima* and *A. cyanophylla* cultivated in Tunisia

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Little is known about the chemistry of most *Acacia* species, although the genus is quite large and widespread in the warm sub-arid and arid portions of the world. The *Acacia* genus comprises approximately 1350 species **[1]**.

Fatty acid and phenolic compositions of the three species of *Acacia cyclops*, *A. mollissima* and *A. cyanophylla* oils were studied. These oils were compared in terms of physicochemical properties and fatty acid composition to Soybean oil. The oil content of *Acacia* seeds is 8.85%, 11.13% and 7.16%, respectively. Fatty acid composition identified by GC and NMR, was essentially dominated by linoleic acid, oleic acid, and palmitic acid.

On the other side, the volatile oils from fresh flowers of *Acacia mollissima*, *A. cyclops* and *A. cyanophylla* obtained by hydrodistillation were analyzed by GC-MS and 18 (94.7% of the total oil composition), 23 (97.4%) and 24 (97.6%) compounds were identified in these oils, respectively. The essential oil of *A. cyanophylla* was largely composed of hydrocarbons (n-octadecane (12.2%), tetradecanol (13.2%)), that of *A. mollissima* was dominated by hydrocarbon sesquiterpenes (56.7%) and represented by two stereoisomers, the (*E,Z*)-α-farnesene (5.2%) and (*E,E*)-α-farnesene (51.5%). Differently to the other two species, the volatile oil of *A. cyclops* was composed primarily by oxygenated sesquiterpenes (27.8%), apocarotenoids (14.2%) and a major fraction (44.6%) dominated by the n-nonadecane (29.6%).

The studied essential oils expressed high antioxidant activity with both DPPH and ABTS assays (IC_{50} = 4-39 µg.mL^{-1}), as well as interesting inhibitor properties against the α-glucosidase (IC_{50} = 62-89 µg.mL^{-1}) and varying antibacterial activity according to *Acacia* species or to the nature of microorganisms.

Key words: *Acacia*, essential oil, fixed oil, fatty acid, DPPH, ABTS, α-glucosidase antibacterial activity

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NEW *O*-METHYLATED COUMARIN FROM ROOTS OF TUNISIAN *RUTA CHALEPENSIS (RUTACEAE)*

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The phytochemical screening of the roots of *Ruta chalepensis* was conducted for the determination of alkaloids kokusaginine, skimmianine, arborinine, γ-fagarine, dictamnine and maculosidine, as well as coumarins chalepensin, chalepin, rutamarin, bergapten, isopimpinellin, xanthotoxin, xanthylétin, imperatorin. The new *O*-methylated coumarin osthol was isolated for the first time from the species *Ruta chalepensis* from the ethanolique extract. The structures were established by spectral methods (^{1}H-NMR, ^{13}C-NMR, DEPT, HMBC, HSQC, COSY), and HRMS as well as by comparison of their NMR data with those reported in the literature.

Key words: *Ruta chalepensis*, NMR, Rutaceae, Osthol, alkaloid, coumarin.

THERMAL AND MORPHOLOGICAL PROPERTIES OF FIBERS EXTRACTED FROM *CACTUS* STEMS

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A new process was used to extract fibers-network from Cactus stem (Fig.1). The obtained fibers were characterized by stereomicroscopy and scanning electron microscopy (SEM) in order to evaluate the morphological characteristics. The thermal properties were studied using thermogravimetric analysis (TGA) under nitrogen and oxygen. The *Cactus*-fibers were characterized by high thermal stability. According to the chemical analysis, the rate of cellulose in the trunk [1] was more important than those of cladodes and other wood, non-wood and some annual plants [2-3].

Fig 1. *Cactus* plants "*Opuntia ficus-indica*"

Key words: *Cactus*-fibers, cellulose, chemical analysis, morphology, thermal property.

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OPTIMIZATION EXTRACTION OF POLYSACCHARIDE FROM TUNISIAN *ZIZYPHUS LOTUS* FRUIT BY RESPONSE SURFACE METHODOLOGY

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Response surface methodology using a Box-Behnken design was employed to optimize extraction temperature, extraction time and ratio of water to material to obtain a maximum polysaccharide yield with high uronic acid content and antioxidant property from edible *Zizyphus lotus* fruit. The optimal conditions were an extraction time of 3h 15min, an extraction temperature of 91.2°C and an amount of water to solid ratio of 39 mL/g. Under these conditions, the experimental extraction yield of the uronic acid content and 2,2-diphenyl-1-picrylhydrazyl scavenging ability (IC_{50}) were 18.88%, 41.89 and 0.518 mg/mL, respectively. Chemical analysis revealed that the extract was composed of 97.92% carbohydrate of which 41.89% was uronic acid. The extracted polysaccharides, with an average molecular weight of 2720 kDa, are composed of arabinose, rhamnose, glucose, fructose, galactose and xylose. Moreover, the polysaccharides exhibited a significant reducing power and anti-lipid peroxidation activities.

Key words: *Zizyphus lotus* fruit; polysaccharide; response surface methodology; Chemical analysis; average molecular weight; antioxidant property.

OPTIMIZATION OF POLYSACCHARIDES ULTRASOUND EXTRACTION FROM TUNISIAN *SUAEDA FRUTICOSA*: *IN VITRO* ANTIOXIDANT AND *IN VIVO* ANTI-INFLAMMATORY ACTIVITIES

ZEINEB MZOUGHI[a], AMEL ABDELHAMID[b], SAWSSEN AMAMOU[C], KHAOULA MKADMINI HAMMI[a,d] , ABDERRAHMAN BOURAOUI[b], HATEM MAJDOUB [a]

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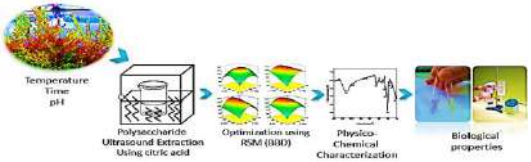
In this study, we investigate the ultrasound-assisted extraction of polysaccharides using citric acid solution from *Suaeda fruticosa* leaves. Response surface methodology using a Box-Behnken design was employed to optimize extraction conditions (temperature, time and pH) in order to obtain a maximum polysaccharide yield with high uronic acid content, low degree of methoxylation and significant total antioxidant property. Fourier transform infrared spectroscopy was used to determine degree of methoxylation and galacturonic acid of polysaccharides. The extraction yield and the galacturonic acid content were improved by higher temperature and lower pH values. The optimum operating conditions for extraction were as follows: Temperature, 90°C; Time, 40 min and pH 2.5. Under these conditions, the experimental extraction yield, uronic acid content, yield of galacturonic acid, degree of methoxylation and total antioxidant activity were 33.22%, 26.05%, 7.40%, 20.32% and 25.61 mg ascorbic acid equivalents/g dry matter, respectively. The phenol-sulfuric acid assay showed that total sugar and uronic acid contents in the extracted polysaccharide were about 66.76% and 26.05%, respectively. The macromolecular characteristics of the extracted biopolymer were identified by size exclusion chromatography (SEC/MALS/VD/DRI) equipped with a triple detection: multiangle light scattering, viscometer and differential refractive index detectors. Furthermore, the polysaccharides exhibited a significant hydroxyl radical scavenging capacity, total antioxidant capacity and Ferric reducing power activity values. Lastly, the extracted showed a good potential in anti-inflamatory effect particularly on the first 3 h after carrageenan injection, with an oedema inhibition of 59.08% and 73.68%, respectively, at the dose of 100 mg/Kg.



Keywords: *Suaeda fruticosa* leaves, Polysaccharide, Response surface methodology, Chemical analysis, Antioxidant property, Anti-inflamatory effect.

A NEW IRIDOID AND DERIVATIVES ISOLATED FROM *CITHAREXYLUM SPINOSUM* L. GROWING IN TUNISIA

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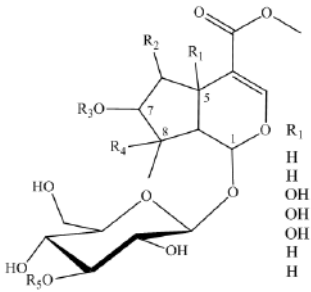
Citharexylum spinosum L. is a tree belonging to the *Verbenaceae* family [1]. It is used as a source of anti-allergic, antifungal and anthelmintic substances. In addition, *C. spinosum* demonstrated significant antiulcer, antihypertensive and hepatoprotective effects [2]. These data from the literature led us to choose to contribute to its phytochemical study and locate if possible bioactive principles. The phytochemical investigation of this plant led to the isolation of a new iridoid named 5-deoxypulchelldioside **(1)** together with six analogs, 5-deoxypulchelloside I **(2)**, Pulchelloside I **(3)**, lamiide **(4)**, lamiide-7-*β*-*O*-acetate **(5)**, lamalbide **(6)** and 8-epiloganin **(7)** (Fig. 1). These products were isolated from the aqueous extract of the bark of this tree trunk by preparative high performance liquid chromatography (HPLC). Their structures were established by spectroscopic means such as NMR (1D and 2D) and high-resolution mass spectrometry (ESI-HRMS).



R_1	R_2	R_3	R_4	R_5	
H	OH	H	H	Glu	: 5-deoxypulchelldioside **(1)**
H	OH	H	H	H	: 5-deoxypulchelloside I **(2)**
OH	OH	H	H	H	: Pulchelloside I **(3)**
OH	H	H	OH	H	: lamiide **(4)**
OH	H	Ac	OH	H	: lamiide-7-*β*-*O*-acetate **(5)**
H	OH	H	OH	H	: lamalbide **(6)**
H	H	H	H	H	:8-epiloganine **(7)**

Figure 1. Structure of Iridoids isolated from the tree *Citharexylum spinosum* L.

Key words: Iridoids, 5-deoxypulchelldioside, *Citharexylum spinosum* L.

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THERMAL AND SPECTROSCOPIC ANALYSIS OF LIGNIN FROM COFFEE SPENT

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Lignin is considered as an important constituent of vascular plants and is second in abundance after the cellulose, among the naturally occurring polymers (18-40 wt. %). the physical-chemical characteristics of lignin differ considerably and are depending on the extraction method used and original source. This paper is devoted to the study of chemical structure, thermal and spectroscopic analysis of Klason lignin isolated from coffee spent.
The structure of lignin was studied by three non-destructive (FT-IR, 13C NMR and UV/visible) and one destructive (thermogravimetric analysis) methods. Elemental analysis determination was performed in order to determine the empirical formulae for the isolated lignin.

Fig. Isolation and characterization of Klason lignin from coffee waste.

Key words: Klason Lignin, coffee spent, TGA/DTG, 13C NMR.

SYNTHESIS, CRYSTAL STRUCTURE, SPECTROSCOPIC STUDY AND THERMAL DECOMPOSITION OF A TWO NEW CO-BASED HYBRID MATERIALS, $(C_4H_{12}N_2)[Co(H_2O)_6](S_{0.685}Se_{0.315})_2$ (1) et $(C_4H_{12}N_2)[Co(H_2O)_6](S_{0.80}Se_{0.20}O_4)_2$

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Two new Co-based hybrid materials, $(C_4H_{12}N_2)[Co(H_2O)_6](S_{0,685}Se_{0,315}O_4)_2$ (**1**)and $(C_4H_{12}N_2)[Co(H_2O)_6](S_{0,80}Se_{0,20}O_4)_2$ (**2**), have been synthesized by slow evaporation method at room temperature and crystallographically characterized. They crystallizes isotypically in the monoclinic system, space group *C*2/*c*, with the following unit-cell parameters: a = 14.2559(19), b = 9.5066(13), c = 13.6759(19) Å, β = 114.930(4) °and Z = 4 for (**1**) and a = 14.2235(19), b = 9.4901(14), c = 13.666(2) Å, β = 114.863(4)° and Z=4 for (**2**). Their crystal structure consist of metallic cationoctahedrally coordinated by six water molecules $[Co(H_2O)_6]^{2+}$,piperazinediiumcations$(C_4H_{12}N_2)^{2+}$and sulfate/selenate anions $(S_xSe_{1-x}O_4)^{2-}$ linked together via two types of hydrogen bonds, Ow–H...O and N–H...O. The spectroscopic study by IR and Raman confirms not only the substitution of the sulfur by the selenium but also the piperazine protonation. The thermogravimetric measurement indicates that the dehydration occurs in only one step by the departure of six water molecules giving rise to an anhydrous phase.

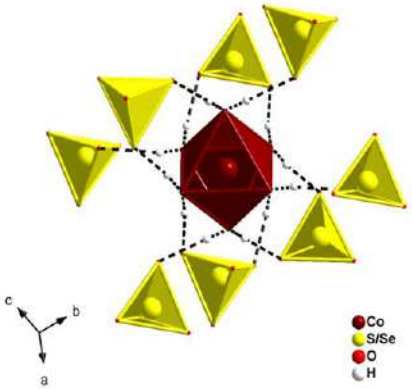

Fig.1: *Liaisons hydrogène entre l'octaèdre de Co2+ et les tétraèdres*

Key words: X-ray diffraction, thermal studies, IR and Raman spectroscopy.

Thermodynamic assessment of the ($LiNO_3$ + $NaNO_3$+$TlNO_3$) ternary system

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The experimental determination of phase diagrams of ternary systems requires a very high number of experimental measurements and the use of several techniques. To target the necessary experiments to study these systems, we choose a method which based on thermodynamic calculation. Indeed, the compilation of all the experimental data relative to the three pure components and to their binary mixtures allows studying the coherence between the thermodynamic data and the phase diagram.
A thermodynamic assessment of the ($LiNO_3$+$NaNO_3$+$TlNO_3$) ternary system is realized, in this work, for the first time. The ($LiNO_3$+$NaNO_3$), ($NaNO_3$+$TlNO_3$) and ($LiNO_3$ +$TlNO_3$) binary systems, have been optimized earlier in previous works [1-3]. The parameters describing the Gibbs energies of all the binary phases are then used to estimate the phase diagram of ($LiNO_3$+ $NaNO_3$+$TlNO_3$) ternary system. In order to verify the results of our assessment, a vertical section, X_{NaNO3} = 0.20, is studied experimentally by simultaneous direct and differential thermal analysis technique (STA/DTA). The experimental results are then compared to the calculation. A good agreement was obtained.

Key words: Alkali nitrate, Phase diagrams, Thermodynamic assessment, STA/DTA.

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INTERACTION RAW TUNISIAN KAOLIN - PHOSPHORIC ACID

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The physical and chemical properties of kaolin determine its use as an industrial mineral. such as in ceramics, toothpaste, cosmetics, paper, paint extender, for facial masks or soap, plastic filler, cracking catalysts or cements, as adsorbents in water and wastewater treatment [1]. their usage are governed by several factors including the particle size, particle shape, surface area, porosity, acidity and surface chemistry, together with other physical and chemical properties. All of these mineral properties are affected by various methods including mechano-chemical activation [2][3], intercalation [4], thermo-chemical treatment [5], and chemical activation [6].

In this study, a crude Tunisian kaolinite clay (from the Tabarka region - Northern of Tunisia) was subjected to refluxing with phosphoric acid under different operating conditions. Acid activation was carried out with H_3PO_4 (28% and 54%), at temperatures of 60°C and 90°C and with times of treatment ranging from 2 to 12h. The structure of acid leached kaolinite clay was characterized by, XRD, FTIR, SEM, ^{29}Si and ^{31}P NMR technique, and Brunauer, Emmett, and Teller (BET) measurements. The chemical composition was determined by ICP analysis technique. The results of X-ray diffraction show that the kaolin acid treatment at 90 ° C provoked a destruction of clay structure after 6h with phosphoric acid 28% and 4h with phosphoric acid 54%. hereafter, the forming an amorphous silica phase is detected, their confiramtion is effected by ^{29}Si NMR analysis. FTIR studies indicate that acid treatment under reflux conditions leads to the removal of the octahedral Al^{3+} cations for the two types of phosphoric acid 28 and 54%. In fact, the chemical analysis show that leaching of Al^{3+} ions increased progressively with severity and times of the reaction. The acid treatment with H_3PO_4, 54% at 90°C increased ŧ the surface area from 24 m^2/g to 150 m^2/g. Solids elaborated by acid treatments can be used as promising adsorbents and catalyst supports.

Key words: Kaolin; acid activation; phosphoric acid; XRD; surface area

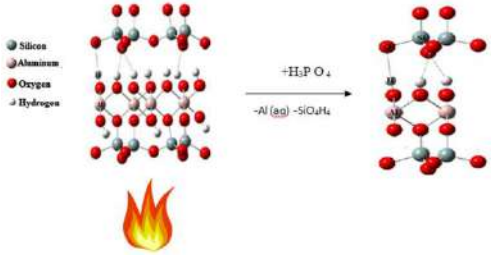

Fig. interaction kaolin with phosphoric acid

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Catalytic Wet Peroxide oxidation of azo dye using Al-Fe-PILC as catalyst

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Advanced oxidation processes (AOP) have been extensively used in the treatment of wastewater from the textile industry, as it has been seen that dye degradation and removal is very hard to be carried out. Among these AOPs, heterogeneous Fenton-like catalyst and several attempts have been made using supporting materials.

Therefore, Al-Fe-Pillared clay used in this investigation, prepared from locally Tunisian clay, seems to be an efficient catalyst for Fenton-like processes. Synthesis and characterization of the heterogeneous catalyst Fenton Fe-Al-PILC was describe. It is used in heterogeneous Fenton reaction to degrade Congo red dye. In order to optimize reaction conditions, effects of pH, initial catalyst concentration and amount of H_2O_2 were investigated. Kinetic study was carried out to calculate the order of Congo red degradation reaction and the rate constants by initial rate and integration methods.

Results showed that the degradation yield could reach 95,75%. It was observed that H_2O_2 had almost negligible effect on iron leaching. The kinetic studies revealed that the degradation profile is well fitted by the first-order kinetic model.

Key words: Fenton, Advanced oxidation processes, Congo Red, Al-Fe-PILC, Kinetic study, heterogeneous catalyst

Co-substitution effect on the structural, electrical and electrochemical properties of nanostructured $Co_xNi_{1-x}MnP_2O_7$ (x=0, 0.25, 0.5, and 0.75)

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Cobalt-Nickel-manganese pyrophosphate nanostructures with formula $Co_xNi_{1-x}MnP_2O_7$ were prepared via the hydrothermal method at 150 °C, followed by the calcination at 500 °C. The structural analysis of $Co_xNi_{1-x}MnP_2O_7$ samples was carried out by X-ray diffraction (XRD) and using the Rietveld method. Morphological characterizations were performed using a scanning electron microscope (SEM). The effect of Co-substitution on the structural, electrical and electrochemical properties of $Co_xNi_{1-x}MnP_2O_7$ is reported. The electrochemical results show that the specific capacity increases from 59 to 205 $mAh.g^{-1}$ with increasing Co-content.

This study shows the Co-substitution effect on the mixed electrical conductivity. The dependence between the temperature and the electrical conductivity with direct current, for both pure and spiked samples Co^{2+}, obeys the Arrhenius law. The increase of Co-amount led to decrease the activation energies (Ea (x = 0.00) = 1.96 eV to Ea (x = 0.75) = 0.88 eV) and an increase in the total conductivity. The frequency dependence of ac-conductivity for the materials, exhibits a Jonscher's universal power law. The improved electrical conductivity and electrochemical proprieties of $Co_xNi_{1-x}MnP_2O_7$ nanomaterials imply their promising potential applications in the energy storage.

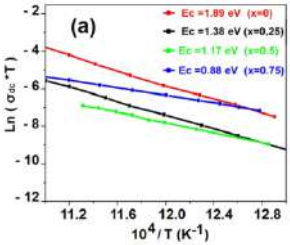

Fig.a: Arrhenius plot of the electrical conductivity of $Ni_{1-x}Co_xMnP_2O_7$ (x= 0, 0.25, 0.5 and0.75

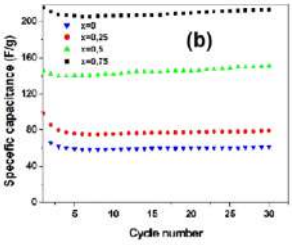

Fig.b: $Ni_{1-x}Co_xMnP_2O_7$ Cycling performance.

Keywords: Inorganic compounds, Electrical properties, Electrochemical properties.

THE EFFECT OF LIMESTONE FILLER ON HRS PORTLAND CEMENT PROPRTIES DURING HYDRATION PROCESS

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The sulfate attack is a common form of concrete deterioration, it occurs when concrete comes in contact with water containing a high concentration of sulfate ions. There are many ways to protect against this type of chemical attack such as the sulfate resisting Portland cement (HRS cement) which is used especially in Tunisia [1].
Some work has been carried out on the effect of limestone additive on HRS Portland cement paste, but there is no general agreement on the relative effects of different amounts of calcium carbonate on cement paste properties [2]. The objective of the present work is to assess the effect of various amounts of calcium carbonate on the hydration of HRS cement in order to explain the physico-chemical changes occurring during hydration. This study is assured by using a number of HRS Portland cement as commercial.

Key words: HRS Portland cement, Hydration, limestone filler, additive, Sulfate attack.

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TREATMENT OF A TUNISIAN TEXTILE EFFLUENT CONTAINING BROMOTHYMOL BLUE DYE BY ANODIC OXIDATION AT BORON DOPED DIAMOND ELECTRODE

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In the present study, Anodic Oxidation (AO), an Advanced Oxidation Process (AOP), was applied to degrade and mineralize a synthetic solution of a textile dye, Bromothymol Blue (BTB), at a Boron Doped Diamond (BDD) anode. An industrial discharge, sampled from a Tunisian industry containing BTB, was then treated at the same operating conditions by AO. The effect of key parameters such as current density, reaction medium pH, treatment time, bubbling and cathode nature were investigated for the synthetic BTB solution. Degradation was optimal and it reached 99% after 60 min of BTB treatment at pH 3, using a carbon felt as a cathode, operating at 60 mA cm^{-2} as current density and maintaining bubbling during the electrolysis. BTB mineralization was investigated under these optimal conditions by monitoring the decolorization, the Chemical Oxygen Demand (COD) and the Total Organic Carbon (TOC) during 6 h of treatment. There upon the optimization of the BTB synthetic dye solution, AO method was applied to oxidize the Tunisian textile effluent. The energy consumption of the AO was also calculated for the treatment of both BTB synthetic solution and the Tunisian textile effluent. The obtained results insure the effectiveness of the AO treatment using BDD anode, considering it as a promising environmentally friendly technology for the remediation of wastewaters containing BTB textile dye.

Key words: Bromothymol Blue, Advanced Oxidation Process, Anodic Oxidation, Boren Doped Diamond anode, Textile effluent

APATITE AND CARBONATE SEPARATION IN FROTH FLOTATION USING ANIONIC AND CATIONIC COLLECTORS

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As the requirement of phosphate ore increases, high grade phosphate resources in Tunisia are exhausted. Thus, at present researches focused on non exploited phosphate, the big and fine rejects' recovery. In order to valorize the low grade phosphate, Tunisia has begun a research program of suitable process. This work investigated the flotation response of phosphate and carbonate using different collectors (phosphoric ester or alkyl hydroxamic acid and amine).
The experiments were performed using pure and artificially mixed minerals as well as a real phosphate ore. Their separation mechanisms were studied by means of zeta potential measurements, infrared analysis and adsorption measurements. The results indicated that anionic collectors exhibited an excellent performance in the flotation separation of phosphate from carbonate in acid to neutral medium of the pulp. The P_2O_5 grade of the concentrate increased to 29.84% with an acceptable carbonation rate to reach the transformation exigences. The zeta potential and infrared studies showed that the adsorption mechanism is different using phosphoric ester from hydroxamic acid at the phosphate/aqueous interface. And it was greater with carbonate than that at phosphate surface, which was the essential reason of the separation.

Keywords: phosphate, big rejections, carbonate, valorization, flotation, separation.

Synthesis and crystal structure of 4-(2-ammonioethyl) morpholin-4-ium dichloridodiiodidocadmate/ chloridotriiodidocadmate (0.90/0.10)

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[c)] Dipartimento di Chimica, Universita di Parma, Parco Area delle Scienze 17A, I-43124 Parma, Italy.

The crystal structure of the title compound, $(C_6H_{16}N_2O)[CdCl_{1.90}I_{2.10}]$, a new organic-inorganic hybrid salt synthesized in the form of single crystals, consists of discrete statistically distributed dichloridodiiodidocadmate /chloridotriiodidocadmate anions (occupancy ratio 0.90:0.10) and 4-(2-ammonioethyl)morpholin-4-ium cations, $[NH_3(CH_2)2NH(CH_2)_4O]^{2+}$. The cations are linked by intermolecular N—H…O hydrogen bonds, forming corrugated chains extending parallel to the c axis. The $[CdCl1.90I2.10]^{2-}$ tetrahalidocadmate anions lie between the chains to maximize the electrostatic interactions and are connected with the organic cations via N—H…Cl and C—H…Cl(I) hydrogen bonds developing in the ab plane and leading to the formation of a three-dimensional network structure. The tetracoordinate Cd(II) atom has a distorted tetrahedral conformation, with a τ_4 index of 0.87.

ELABORATION OF MODIFIED ELECTRODE BY ELECTROPHORETIC DEPOSITION OF HYBRID MATERIALS

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Metal-Organic Frameworks (MOFs) are hybrid porous materials resulting from a supramolecular assembly of inorganic cations and organic linkers [1, 2]. Even there are more than 20 000 MOF structures that have been reported and studied to date, their elaboration as thin films still be a challenging problem. In this frame, MOFs were deposited on smooth substrates, such as gold [3] and ITO [4]. Many different ways are used for preparing MOFs film. Electrophoretic deposition is one of the most powerful techniques to fabricate hybrid thin films [5].
In this study, a novel functionalized metal–organic frameworks (MOF) was synthesized. The obtained product was characterized by infrared spectra; thermal analysis and zeta potential measurements. These MOFs were then deposited on indium tin oxide electrode by electrophoretic deposition to form a modified electrode. The prepared films were monitored by Fourier X-ray diffraction (XRD) and optical microscopy.

Key words: Coordination polymers, MOFs, electrophoretic deposition

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STRUCTURAL CHARACTERIZATION, SPECTROSCOPIC, THERMAL, AC CONDUCTIVITY AND DIELECTRIC PROPERTIES AND ANTIMICROBIAL STUDIES OF $(C_8H_{12}N)_2[SnCl_6]$

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A new inorganic-organic hybrid material produced from 2,6-dimethylanilinium cations and tin halide $(SnCl_6)^{2-}$ has been synthesized and structurally determined by X-ray diffraction method. The title compound crystallizes in the monoclinic system, space group C2/m with a = 19.8772(4), b = 6.9879(1), c = 8.3001(2) Å, β = 98.487(2) ° and V = 1140.26(4) $Å^3$. The crystal structure is built up of sheets of $(SnCl_6)^{2-}$ octahedral anions and 2,6-xylidinium cations. The optical band gap was calculated and found to be 4.11 eV. At high temperature this compound exhibits a structural phase transition at 338 K. This has been characterized by differential scanning calorimetric and dielectric studies. Measurements of AC conductivity as a function of frequency at different temperatures indicated the hopping conduction mechanism. The bioassay results showed that the structure exhibits significant antibacterial activity.

Key words: Crystal structure, spectroscopies, fluorescent properties, phase transition, antibacterial activity.

EFFECT OF AMORPHIZATION DEGREE ON MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF PORTLAND CEMENT PASTE

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In this report, the authors have shown that besides the particle size reduction, prolonged high-energy ball milling of Portland cement leads to mechanically induced phase transformation from the crystalline to the amorphous state. This research has also investigated the improvement of the mechanical and microstructural properties of cement pastes incorporating mechanically activated cement milled for various durations, as partial replacement of the unprocessed cement. Results have shown that compressive strength increases with prolonging mill duration as well as the amorphization degree of the processed cement. The setting times and porosity are significantly reduced, and the degree of hydration is improved by increasing the mill duration and the amorphization degree of the modified cement. The most significant results were observed when 10 wt.% of amorphous cement was added to the unprocessed cement. In this experimental condition, the compressive strength was increased by 107% (at 28 curing days), the initial and final setting times were reduced about 3 times, the porosity was reduced by 52% and the early degree of hydration was improved by 162%.

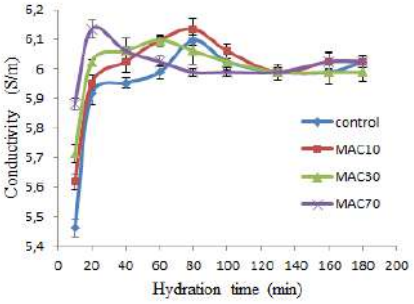

Fig 1. Electrical conductivity development of liquid solution

Key words: mechanical activation, amorphization degree, Portland cement.

Bis(tetraethylthiophosphoramidoyl)methylamine, electrochemical ligand for simultaneous iron and copper bivalent cations detection

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Exploration of chemosensor for bivalent cations, particularly iron and copper, is an important research subject attracting a widespread attention due to the pivotal roles of such species in several biological processes [1-2]. For all these reasons, scientific community devoted a huge interest to develop new electrochemical sensors, which are considered economical, easy to handle and highly sensitive systems [3]. In this work, we report the synthesis of the ligand, (tetraethylthiophosphoramidoyl)methylamine and its exploitation as a bivalent metallic cation sensor. The electrochemical studies reveal a sensitive detection process toward copper and iron cations. The chelation process was accompanied by dramatic changes in the redox properties of free ligand. Interestingly, the ligand shows a simultaneous sensing behavior towards iron and copper cations (figure below). The oxidation peak potentials of the two complexes can be well separated allowing the sensitive detection.

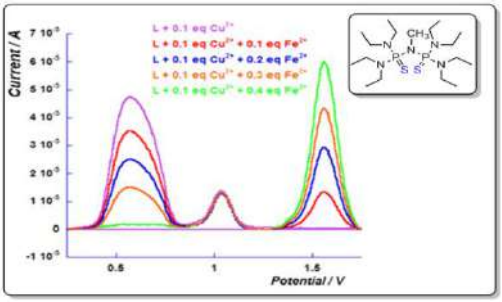

Fig. Differential pulse voltammograms of the successive additions of the iron perchlorate cation to the solution of ligand (1 mM) in the presence of 0.1 equivalent of copper perchlorate cation

Keywords: Thiophosphoramide ligand; cation chelation; simultaneous detection; voltammetry technique

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Study of the photochemical reactivity of veterinary antibiotics found in surface water

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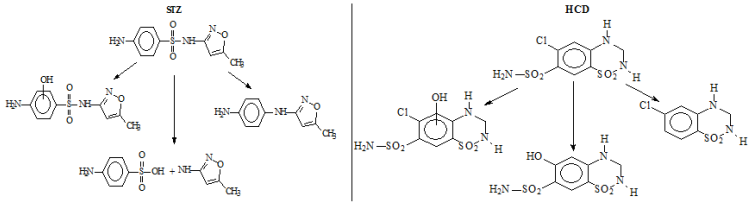
Nowadays, the emergence of pharmaceuticals in the aquatic and terrestrial environment have been a major concern. They have been detected in sewage-treatment plants, sediments, and soils as well as at surface and drinking water. So far, there is limited information in the literature on the fate of these compounds when they are exposed to solar light in the various environmental compartments. The objective of the present study is to investigate the degradation process of two different antibiotics Sulfamethoxazole (**STZ**), Hydrochlorothiazide (**HCD**) in aqueous solutions when exposed to simulated solar light. We mainly concentrate our effort on the kinetic studies by evaluating the degradation quantum yield as well as the effect of various parameters such as oxygen concentration, pH and the presence of inorganic ions. The main effect was observed by molecular oxygen parameter. We also make an important effort in the elucidation of the main intermediate and stable by-products. A lot of information is available on the stability and fate of parent compounds and not so many on their transformation products. These may present a toxicity level higher than the precursor substrate and should be identified and analysed. The structure elucidation was obtained by using the HPLC/ESI/MS and HPLC/ESI/MS^2 techniques in negative as well as positive modes and through the complete study of the various fragmentation pathways. The main involved photochemical processes were i) the scission of the bridge through a photohydrolysis process, ii) selective hydroxylation of the aromatic moiety iii) Desulfonation process and iiii) in the case of HCD to dechloration reaction. A mechanism was then proposed in the light of the kinetic and analytical studies.



Keywords : antibiotics, sunlight, photolysis, Sulfamethoxazole, Hydrochlorothiazide

Synthesis of organo-materials. Study of the effect of: pH, time, initial concentration and surfactant properties.

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The synthesis of organo-materials is based on the modification of raw materials: a Tunisian illite and a zeolite X (synthesized from illite), by three cationic surfactants: DTAB, HDTMA and HDPy. The modification of these raw materials has focused on the study of the various parameters influencing the synthesis by the "batch" method: the effect of pH, time and the initial concentration of surfactant. In addition, we interested to compare the three surfactants in order to understand the effect of the length of the chain surfactant (DTAB/HDTMA) and the effect of the nature of the polar head (HDTMA/HDPy).

The study concluded that the surfactant-adsorbent interaction occurs at the external surfaces. This interaction is influenced by the pH, contact time, the initial concentration, the properties of the surfactant (length of the hydrocarbon chain, CMC, nature of the polar head) and the adsorbent properties (S_{BET}, CEC). Indeed, for three surfactants, the adsorption capacity is better on the zeolite as illite. Furthermore, the increasing of the length of the surfactant chain increases the adsorption capacity. For the same length of chain, aromatic polar heads promote and enhance the adsorption capacity.

The DTAB adsorbs on monolayers with maximum adsorption capacities obtained respectively from the initial concentrations of 100% of the CEC of the illite and 150% of the CECE of the zeolite.

The HDTMA and the HDPy, at low initial concentrations, are adsorbed as a monolayer which evolves in bilayer at high concentrations. The maximum adsorption is obtained at initial concentrations of 200% CEC of the illite and of 250% CECE of the zeolite.

The adsorption of the three surfactants is a rapid kinetics, at optimum pH of 8.5 and which the isotherms of the study of the effect of the initial concentration, shows a good correlation with the Langmuir model.

Key words: Illite, Zéolithe X, Organo-materials, Surfactants, DTAB, HDTMA, HDPy, CEC, CECE, monolayer, bilayer.

Modeling and simulation of solar phosphate drying

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U.R Environment, Catalysis and Process Analysis, ENIG

In this work, we will design and model a solar dryer for the phosphate drying of the Company of phosphate Gafsa (CPG).
This dryer is composed of a cylindro-parabolic solar concentrator and a tunnel dryer. By carrying out mass and energy balances, we have established a theoretical model describing the operation of the system and by developing a program, on MATLAB, we were able to simulate the operation of this dryer.
The results obtained show the considerable solar contribution to the energy required for drying.
These results are encouraging to continue this work on the experimental aspect.
The installation of the process is shown in the following figure:

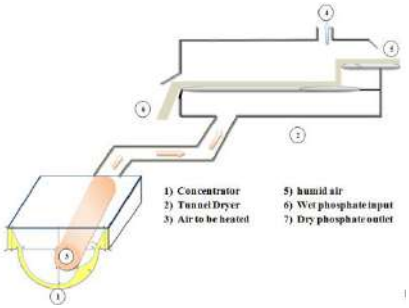

Fig. Schema descriptive of the drying installation

Key words: solar drying of phosphates, cylindro-parabolic concentrator, tunnel dryer, humidity.

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Evaluation of physicochemical properties and corrosion inhibition performance of γ-glycidoxypropyltrimethoxysilane coating modified with Nitrate salts ($NaNO_3$ and $Ce(NO_3)_3$)

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This work investigates the effect of nitrate salts ($NaNO_3$ and $Ce(NO_3)_3$) on the properties, especially, the anti-corrosion performance of γ-glycidoxypropyltrimethoxysilane (γ-GPTMS) film deposited on AA2024-T3 aluminum alloy.

The deposition of γ-glycidoxypropyltrimethoxysilane (γ-GPTMS) films on AA2024-T3 was performed via electrodeposition. The electrodeposited coatings were performed under potentiostatic control at -0.80 V/SCE.

γ-GPTMS films over AA2024-T3 surface were characterized by physical, microscopic and spectroscopic techniques : Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray Spectroscopy (EDS), Infrared Spectroscopy (FTIR) and Thermogravimetric analysis (TGA). The electrochemical behaviors of coatings were assessed in chloride neutral solution by means of potentiodynamic polarization studies (PDS) and electrochemical impedance spectroscopy (EIS).

The results proved the influences of $NaNO_3$ and $Ce(NO_3)_3$ on the of γ-GPTMS film properties. $NaNO_3$ improved the corrosion resistance of γ-GPTMS coating due to the contribution of the nitrate ions reduction activity in the promotion of γ-GPTMS condensation reaction.

$Ce(NO_3)_3$ ameliorated the anticorrosion performance of γ-GPTMS film mainly due to the healing ability of cerium ions.

Furthermore, EIS data revealed that the $Ce(NO_3)_3$ enriched γ-GPTMS coating could provide superior corrosion protection compared to the sol–gel film which was incorporated with $NaNO_3$.

Key Words: γ-GPTMS, AA2024-T3, corrosion, $NaNO_3$, $Ce(NO_3)_3$

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Equilibrium, kinetics and thermodynamics studies of textile dyes adsorption on modified Tunisian clay

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The adsorption capacity of two anionic textile dyes (RR120 and BB150) on DMSO intercalated Tunisian raw clay was investigated with respect to contact time, initial dye concentration, pH and Temperature. The equilibrium data were fitted into Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherms. The kinetic parameters were calculated using pseudo-first order, pseudo second-order, intra-particle diffusion and Elovich kinetic models. The thermodynamic parameters ($\Delta H°$, $\Delta S°$ and $\Delta G°$) of the adsorption process were also evaluated.

Keywords: Tunisian clay, DMSO, adsorption isotherm, kinetics model, anionic dyes.

DEVELOPMENT OF AN ASYMETRIC ULTRAFILTRATION MEMBRANE FROM NATURALLY-OCCURING KAOLIN CLAY

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This work concerns the development and characterization of support and ultrafiltration membranes from naturally occurring- kaolin clays as principal components. The preparation and characterization of porous tubular supports, using kaolin powder with corn starch as poreforming agent, is reported. It has been found that the average pore size was about 1 µm while the pore volume was 44% for supports sintered at 1150°C with a flexural strength of about 15 MPa. The deposition of the active layer was performed by slip casting method. The rheological study of various coatings with different concentration of kaolin powder, polyvinyl alcohol (PVA) and water under different conditions regarding temperature and stirring time was done. After drying at room temperature for 24 h, the membrane was sintered at 650°C. The average pore diameter of the active layer was 11 nm and the thickness was around 9 µm. The determination of the water permeability shows a value of 78 l/h.m2.bar. This membrane can be used for crossflow ultrafiltration. The application of the cuttlefish effluent treatment shows an important decrease of turbidity, inferior to 1.5 NTU and chemical organic demand (COD), retention rate of about 87%. So, it seems that this membrane is suitable to be used for wastewater treatment.

Key words: Kaolin clays; Sintering; Ceramic supports; Ultrafiltration membrane; Cuttlefish effluent.

EVALUATION OF DEGRADATION KINETICS OF NBD-Cl REMOVAL FROM WATER BY ELECTRO-FENTON TREATMENT

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This thesis project deals with the electrochemical oxidation of carbon-halogen bond. It's known that the presence of halogen decreases the biodegradability of the molecule. The great oxidation ability of this process is due to the large production of hydroxyl radical (OH°) by electrochemically induced Fenton's reagent. The electrochemical advanced oxidation method Electro-Fenton has been applied to remove NBD-Cl (4-Chloro-7-nitrobenzofurazan) in water. This organochlorine compound is released from industrial waste of pesticides and residual pharmaceuticals into natural waters and usually causes suspected undesirable effects on aquatic organisms. The degradation of this organic compound by Electro-Fenton process was showed using a carbon felt cathode and platinum anode. The evolution of the concentration during treatment was followed up by high performance liquid chromatography (HPLC). The influence of operating conditions on the degradation of NBD-Cl by Electro-Fenton step, such as current density and catalyst concentration, has been investigated and discussed. NBD-Cl mineralization was investigated under these optimal conditions by monitoring the Chemical Oxygen Demand (COD) during 6 h of treatment. Degradation reaction obeyed apparent first-order reaction kinetics, with absolute rate constant determined as $3{,}32 \times 10^5$ $M^{-1}s^{-1}$ by competitive kinetics method taking Benzoic Acid as reference compound. Moreover, a simultaneous generation of inorganic ions (chlorides and nitrates) was observed and 3 short chain carboxylic acids (oxalic, benzoic and propionic acids) were identified and monitored during 6h of electrolysis. The results confirm the efficiency of the Electro-Fenton process to degrade organochlorine pollutants in water.

Keywords: Organochlorine, biodegradability, degradation, AOPs, electrochemical oxydation.

Program of Thursday 22 December 2016

"Chemical and viscoelastic properties of clay suspensions treated by hydrocycloning process"

Mariem Mekni Abrougui[a b*], Ana Belén Bonhome-Espinosa[c], Juan de Dios García Durán[c], Srasra Ezzeddine[a]

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[b)] *Faculté des Sciences de Tunis, Université de Tunis El Manar, Tunisia*
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Several studies and research focus on clay minerals, which are considered as key components for applications in many industrial sectors such as ceramics industries, petroleum, water treatment, and pharmacy. In their natural state, most of the bentonite deposits are heterogeneous. They consist of mixed or interlayered smectite with illite and/or kaolinite and other impurities.**[1]** Generally, impurities affect the performance of bentonite in industrial applications. As a consequence, the need of developping purification techniques is a priority in this area. Several techniques can be used to separate the fine clay fraction of associated impurities. Among these techniques, the hydrocyclone separation in bentonite clays provides a high efficiency in a short period of time with the minimum number of experiments and low operating cost **[2].**

The main objective of this study was to use raw Tunisien clay from Zaghouan (North East Tunisia) and purified with "hydrocyclone" in presence of 2% and 5% of sodium carbonate. The product obtained after hydrocycloning was characterized by XRD, FTIR, and SEM. The chemical composition was determined by X-ray fluorescence. XRD diffractograms show that the Ca-montmorillonite fraction was converted to Na-montmorillonite after addition of (2%, 5%) Na_2CO_3. In addition, a significant reduction of the quantity of quartz and calcite is observed, which is consistent with the obtained IR and XRD results. Finally, the rheological properties of the clay suspensions were determined with a controlled-stress rheometer. The rheological results demonstrate a significant increase of the yield stress of the suspensions varying from 0,72 Pa in raw clay to 1,47 (7,67) Pa for the clay purified with 2% (5%) Na_2CO_3. Thus, demonstrating that this process allows conferring a stronger plastic behavior and higher viscosity to the suspensions. The clay obtained after hydrocycloning can be used as drilling mud and, in consequence, we expect that the present contribution can contribute to the economic development of Tunisia.

Key words: Clay , Caracterisation, Hydrocyclone, Rheology.

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SYNTHESIS AND CHARACTERISATION OF THE NEW HYBRID $(C_6H_{20}N_3)_2(Cr_2O_7)_3$

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In recent years, the attention in organic-inorganic hybrid compounds has increased**.** The new crystal $(C_6H_{20}N_3)_2(Cr_2O_7)_3$ has been synthesized by a slow evaporation at room temperature and is characterized by a single-crystal X-ray diffraction, this compound crystallizes in the triclinic system, space group P -1, with the following unit cell parameters a=8.7148(4) (Å), b= 12.4875(6) (Å), c= 16.0986(7) (Å), Z=4. Crystal structure was solved with a final reability factor R=0,0413 for 5266 independent reflections. The sample was characterized by thermal analysis, X-ray powder diffraction, IR and UV-visible.

The asymmetric unit of the title compound contains two cations and three dichromate dianinon, in the crystal anion chain and the cation are forever linked into a tridimensional network by N-H...O hydrogen bond interaction.

Thermophysical properties of the binary mixtures of N,N-Dimethylacetamide with 1-propanol and 1-butanol

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[b)] *Departamento de Química Física, Facultad de Ciencias, Universidad de Zaragoza, 50009 Zaragoza, Spain.*

The thermophysical behavior of the binary mixtures N,N-dimethylacetamide + 1-propanol and N,N-dimethylacetamide + 1-butanol has been studied through the measurement of density, ρ, speed of sound, u, refractive index, n_D, and kinematic viscosity, ν, over the entire mole fraction range at the temperatures (283.15, 298.15 and 313.15) K and at atmospheric pressure. Excess volumes, V^E, excess isentropic compressibilities, κ_S^E refractive index deviations, Δn_D, and viscosity deviations, $\Delta\eta$, were also calculated and correlated with the Redlich-Kister equation. The negative values of κ_S^E and $\Delta\eta$ at all temperatures, for both mixtures indicate the existence of significant interactions between the components. The values of V^E are found to be negative for the N,N-Dimethylacetamide with 1-propanol, whereas an inversion of the sign at 283.15 and at 298.15 K is obtained for the N,N-Dimethylacetamide with 1-butanol, values turn positive at 313.15 K. The Δn_D behavior is found to be opposite to the behavior of V^E for both mixtures. Further, it was noticed, that interaction between unlike molecules become weaker and dispersive forces become more important for increased chain alkanol.

Key words: density, viscosity, speed of sound, refractive index

Standard enthalpy of «formation of $AgTl(NO_3)_2$, solid at 298 K

Sondes NAJEH, Dalila HELLALI and Hmida ZAMALI

University Tunis El Manar, Faculty of Sciences of Tunis, Department of Chemistry, Lab Materials, Crystal Chemistry and Applied Thermodynamics (LMCTA) LR15ES01.

Alkali and pseudo-alkali nitrates such as molten salts are potential phase change materials (PCM) for the storage and the transfer of thermal energy.
The knowledge of their thermodynamic properties and their phase diagram is necessary to predict their thermal behavior.
A previous investigation of the binary phase diagram ($AgNO_3$+$TlNO_3$) mentioned the existence of a congruent intermediate compound $AgTl(NO_3)_2$.
The aim of this work, is to determine, by solution calorimetry, the enthalpy of "formation" referred to the solid pure nitrates ($AgNO_3$ and $TlNO_3$) of the solid $AgTl(NO_3)_2$.
Thermodynamic data of the latter compound and specially the standard enthalpy of "formation" have never been determined elsewhere. This enthalpy of "formation" is essential for the optimization of the phase diagram.

VANADIUM OXIDE NANOTUBES: HYDROTHERMAL SYNTHESIS AND ELECTROCHEMICAL PROPERTIES

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In the past decades, nanostructured materials have been at the forefront of applied research because of their unique properties, which are different from their bulk materials [1]. Vanadium oxide nanotubes (VOx-NTs), one of the most interesting one-dimensional nanoscale electrode materials, have received considerable attention because of their higher discharge capacity and better cycling performance than the corresponding traditional materials [2].
The present communication deals with the electrochemical performances of vanadium oxide nanotubes VOx-NTs. Electrochemical measurements were carried out on thin films deposited on ITO modified glass electrodes and revealed reversible redox behavior corresponding to a reduction of vanadium oxide, with charge-discharge cycling corresponding to the reversible lithium ions charge/discharge into the crystal lattice of the nanostructures. In order to identify the electrochemical reaction kinetics of electrode materials, EIS measurements were carried out for 50 cycles in the frequency range between 0.01 Hz and 1 MHz. Nyquist plots consisted of a small intercept at high frequency, two partially overlapped semicircles in the high and medium frequency regions and a long low frequency line which corresponds to the Warburg impedance of Li^+ diffusion. Thus, the EIS curves were fitted to the equivalent circuit in order to find Warburg parameter and to evaluate the diffusion coefficient value ($D_{Lithium}$). The $D_{Lithium}$ is found to be varying between 5.64×10^{-9} and 1.47×10^{-10} cm^2 s^{-1}.

Key words: Hydrothermal synthesis, Nanocomposite; Electrochemical properties.

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STRUCTURAL, THERMAL AND OPTICAL PROPERTIES OF PHOSPHATE GLASSES DOPED WITH SiO_2 OXIDE

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During the recent years, phosphate glasses have attracted a considerable technological interest due to their particular properties which makes them suitable for large applications such as: biomaterials, glass to metal seals, optical waveguides, amorphous semi-conductor….

The present work deals with the study of structural, optical and thermal properties of phosphate glasses doped with SiO_2 oxide having a general formula: $(0,9\text{-}x)NaPO_3\text{-}xSiO_2\text{-}0,1ZnO$ ($0,02 \leq x \leq 0,1$ mol%).

Glasses have been prepared using a conventional melt-quenching technique. Samples were investigated by means of density and refractive index measurements, differential scanning calorimetry (DSC), Fourier-transformed infrared (FTIR), Raman and UV-Visible spectroscopy.

The variations of density and refractive index of the glass series were attributed to the structural changes of the glass network resulting from the incorporation of SiO_2 oxide.

The increase of glass transition temperature (T_g) values reflects an increase of the rigidity of the glass network when SiO_2 oxide is gradually introduced. These results were correlated with FTIR and Raman investigations which revealed the enhancement of the vitreous network when P-O-P bonds were replaced by P-O-Si.

The variation of the optical band gap (E_g) of the amorphous materials indicated an increase in the energy gap with the glass composition. This behavior can be explained by the formation of non-bridging oxygen, which is probably due to the distortion of phosphate chains when SiO_2 oxide is introduced.

Key words:
Phosphate glasses, distortion of phosphate chains, spectroscopic analysis, optical band gap.

SYNTHESIS, CHARACTERIZATION, AND STYRENE OXIDATION ACTIVITY OF TUNGSTEN SUPPORTED ON SILICA

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A series of supported tungsten catalysts were synthesized on performed SiO_2 support via sol gel method. Sodium Tungstate, tetraethylorthosilicate (TEOS) were used as Tungsten and Silica sources, respectively. The resulting materials were characterized by techniques such as Fourier transform infrared spectroscopy and X-ray diffraction. The electronic structures and the coordination state of the supported tungsten oxide phases were determined with UV–vis spectroscopy. For comparison, UV–vis DRS spectra of well-defined compounds references (Sodium tungstate (Na_2WO_4, $2H_2O$), ammonium metatungstate ($(NH_4)_6H_2W_{12}O_{40} \cdot xH_2O$) are used. The UV-vis spectrums were depended on tungsten loading and catalyst pH. The catalysts were used for oxidation of styrene with H_2O_2 as oxidant in CH_3CN as solvent. It was found that the catalytic activity and the selectivity of the product depended on, the metal content, the pH and styrene/H_2O_2 molar ratio. The heterogeneous nature of the reaction was tested and the leaching problem was observed with some catalysts.

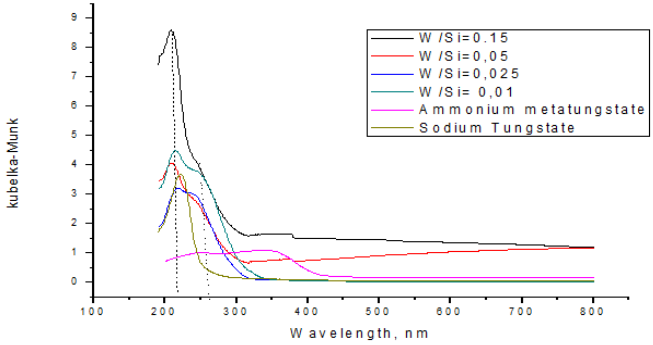

Fig. Diffuse –reflectance UV-vis spectra of W/SiO_2 samples

Key words: Tungsten, oxidation, styrene, leaching.

Synthesis and crystal structure of a novel inorganic-organic hybrid compound $[(3\text{-}NH_3CH_2C_5H_4N]CdI_{2.71}Cl_{0.29}$.

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A new organic – inorganic hybrid complex with the monodentate ligand 3-aminomethylpyridine, $[(3\text{-}NH_3CH_2C_5H_4N]CdI_{2.71}Cl_{0.29}$, has been prepared and characterized by single X-ray-diffraction, thermal analysis and spectroscopic studies. The compound crystallizes in the monoclinic system, with space group group $P2_1/c$, a = 9.6691 (12), b = 11.4172 (14), c = 12.8633 (15) Å, β = 104.4874 (17)°, V = 1374.9 (3) $Å^3$, Z = 4. Each Cd^{II} atom is tetracoordinated in a distorted tetrahedral environment defined by one $[(3\text{-}NH_3CH_2C_5H_4N]$ through the unprotinated nitrogen atom and three I/Cl atoms (occupancy ratio approxymately 0.90:10). The partial presence of chloride in this sites reflects a small increase of Cd – I/Cl bond length when compared by Cd – I in other reported structures. In the crystal, the $(3\text{-}NH_3CH_2C_5H_4N]CdI_{2.71}Cl_{0.29}$ molecules are linked via N – H…Cl hydrogen bonds, forming a supramolecular layers parallel to (a, c) plane. Layers such formed, cross the unit cell at x = ¼ and x = ¾ and are organized in an anti parallel manner. This justifies the formation of a centrosymmetric structure. The π-π interactions between pyridine rings may be neglected and the cohesion and the stability of the structure is ensure by the hydrogen bonds and Van Der Waals interactions.

THERMODYNAMIC ASSESSMENT OF THE (KNO_3+$NANO_3$+$TLNO_3$) TERNARY SYSTEM

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Phase diagrams of binary and/or higher order systems are an important source of information in industrial application. However, the determination of phase equilibria in ternary and high order systems is experimentally difficult and requires enormous resources in time and equipments.
The use of a method which is based on thermodynamic calculation reduces the number of experimental measurements.
We are interested in the present work, in the assessment, for the first time, of the (KNO_3+$NaNO_3$+$TlNO_3$) ternary system using the parameters describing the Gibbs energies of all binary phases. The (KNO_3+$NaNO_3$), ($NaNO_3$+ $TlNO_3$) and (KNO_3+$TlNO_3$) binary systems have been optimized earlier in previous works [1-3].
A vertical section X($TlNO_3$) = 0.7 is also experimentally studied, using simultaneous simple and differential thermal analysis (STA/DTA) technique.
A satisfactory agreement between the experimental and calculated data is obtained.

Keywords: Assessment, Calculation, Phase diagram, STA/DTA, Potassium nitrate, Sodium nitrate, Thallium nitrate.

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[3] D. Rtibi, D. Hellali, D. Boa, H. Zamali : C.P. (19th TCC 2016).

Effect of nickel substitution on electrical and microstructural properties of $CaCu_3Ti_4O_{12}$ ceramic.

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In this paper we present the effect of substitution and sintering parameter (temperature and duration) of $CaCu_3Ti_4O_{12}$ composition on the grain growth, morphological evolution, dielectric constant and resistivity which depend on temperature and frequency. The effects of Ni additive on crystallite structure, microstructure and dielectric properties of the CCNTO ceramics were particularly studied. Compositions with formula $CaCu_{3-x}Ni_x$ Ti_4O_{12} with x= 0, 0.025, 0.05, 0.075, 0.1, 0.2 and 0.3 were prepared by conventional solid state reaction method. The samples were studied by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Dielectric measurements were taken at room temperature in the 10^2Hz -10^7Hz frequency range. XRD analysis confirmed the formation of a single phase material in the samples calcined at 1000°C for 12h with $x \leq 0.2$ and indicated the presence of secondary phases such as CuO, $CaTiO_3$ and TiO_3 in other with x>0.2. In all compositions, structure remained cubic. The dielectric properties permittivity; the loss factor and the resistivity of these ceramics were improved and interpreted.

Key words: $CaCu_3Ti_4O_{12}$, dielectric properties, colossal dielectric, CCNTO, Microstructures.

A new 2-D Mn(II) coordination polymer: $(C_5H_8N_3)_2[Mn_2(C_2O_4)_3].3H_2O$

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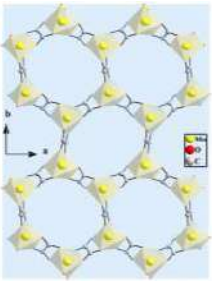
Coordination polymers continue to attract a great deal of attention due to their fundamental importance in many processes.
The single-crystal X-ray structure of a new Mn(II)-oxalate coordination polymer $(C_5H_8N_3)_2[Mn_2(C_2O_4)_3].3H_2O$ (1) is reported. This compound crystallizes in the monoclinic space group P21/n, a= 9.756(3) Å, b= 16.743(5) Å, c= 15.420 (1), β= 97.53(5)°, V= 2497.1(11) Å3 and Z= 4. The asymmetric unit consists of a complex anion $[Mn2(C2O_4)_3]^{2-}$, two protonated 2,6-diamipyrine cations $[C_5H_8N_3]^+$ and three water molecules. The complex anion is formed by two Mn(II) atoms bridged by an oxalate ligand, each Mn(II) atom being further coordinated by one more oxalate ligand in a distorted octahedral geometry.
The 3-D structure of (1) is composed of infinite manganese-oxalate 2-D honeycomb layers (Fig. 1), perpendicular to the *c* axis, formed by infinite manganese-oxalate rings and held together by hydrogen bonds via the water molecules of crystallization and 2,6-diaminopyridinuim cations, entrapped between the layers (Fig. 2).



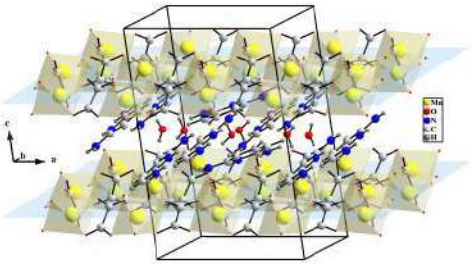


Fig. 1 A honeycomb layer **Fig. 2** A perspective view of the structure of (1)

Key words: manganese, oxalate, 2-D coordination polymer, honeycomb.

Structural, Hirshfeld surface and spectroscopic studies of the noncentrosymmetric 1-ethylpiperazinediium pentachloroantimonate (III) monohydrate

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Halogenoantimonates (III) feature a rich diversity of solid state structures. Sb^{3+}–halide units are highly dynamic building blocks. The properties and structural characteristics of some of these organic–inorganic salts, defined by the general formula $R_aM_bX_c$ (R-organic cation; M-antimony(III); X-chlorine; a; b; c- stoichiometric coefficients (c=a+3b)), have recently been reviewed [1–3].

1-Ethylpiperazinediium chloride pentachloroantimonate (III) monohydrate, $C_6H_{16}N_2SbCl_5.H_2O$, crystallizes in the orthorhombic system, in the non-centrosymmetric space group *Pca2*$_1$ and consists of isolated $[C_6H_{16}N_2]^{2+}$ cations, square pyramidal $[SbCl_5]^{2-}$ anions and lattice water molecules. O-H···Cl hydrogen bonds link the $[SbCl_5]^{2-}$anions and water molecules to form double chains stretching along the [101] direction. The chains in turn are linked to the organic cations via N-H···Cl, C-H···Cl, C-H···O and N-H···O hydrogen bonds to form a three-dimensional network (Fig. 1). This structure presents an example of a general square pyramidal complex ion containing a stereo-chemically active lone pair of electrons. The interactions variability of the two independent cations and ten chloride anions is analyzed via Hirshfeld surface analysis (Fig. 2). Solid state ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray structure, and vibrational absorption bands were identified by infrared spectroscopy. DFT calculations allowed the attribution of the NMR peaks and IR absorption bands.

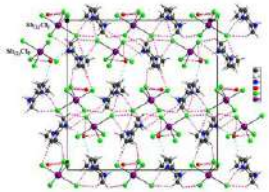

Fig. 1. Projection along the *b*-axis of the crystal packing of the title compound. The dotted lines indicate hydrogen bonds.

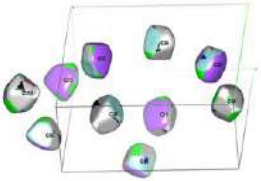

Fig. 2. Hirshfeld surfaces around the ten chloride anions colored according to the atom in contact.

Keywords: Chemical synthesis; X-ray diffraction; NMR; Crystal structure; IR

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Crystal structure, Hirshfeld surface analysis and characterization of a new hybrid dichromate compound $[C_9H_{14}N]_2Cr_2O_7$

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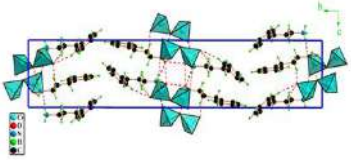
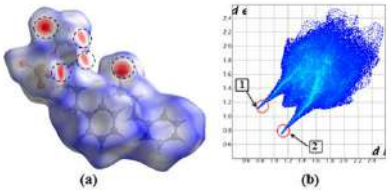
The design and synthesis of new solid materials with controlled structure are a current challenge in solid state chemistry. Recently a large number of dichromates of organic bases have been prepared and used as reagents in mild selective oxidation processes of organic substrates [1], so as to exhibit some interesting crystal structure and some special properties in several areas, such as nonlinear optical (NLO), catalysis and biology [2]. We report in this work the development and study of a new organic dichromate $[C_9H_{14}N]_2Cr_2O_7$. This compound crystallizes in the monoclinic space group $P2_1/c$ with the following parameters a = 7.9379(2) Å, b = 36.2439(16) Å, c = 7.5753(3) Å, β = 96.069(2)°, V = 2167.20(14) $Å^3$ and Z = 4. The structure consists of discrete dichromate anions ($Cr_2O_7^{2-}$) with eclipsed conformation stacked in layers parallel to (a, c) plane at y = 0 and ½. These anions are linked via the 3-phenylpropylammonium cations by N—H…O and C—H…O hydrogen bonds, forming a two-dimensional supramolecular network. Hirshfeld surface analysis for visually analyzing intermolecular interactions in crystal structure employing molecular surface contours and 2D fingerprint plot have been used to examine molecular shapes.

In order to give more information on the crystal structure, we have studied the vibrational proprieties of our compound using infrared absorption, UV-visible, solid state NMR and thermal analysis.



Projection of the crystal structure of $[C_9H_{14}N]_2Cr_2O_7$ along the *a*-axis



Hirshfeld surface d_{norm} (a) and full fingerprint plot (b) of $[C_9H_{14}N]_2Cr_2O_7$

References

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Structure, microstructure and mechanical changes of ceramic products of sintered clay and non-plastic clay mixtures

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Five clay and non-plastic mineral mixtures (M1, M2, M3, M4 and M5) from Tunisia were investigated for their possible ceramic applications. X-ray diffraction (XRD), thermal analysis (DTA/TG), dilatometry, scanning electron microscopy (SEM) and physical-mechanical analyses were used to assess the phase evolution and microstructure of the mixtures sintered between 900 and 1150°C. The decrease of water absorption and the increase of the bending strength of the samples were due to the formation of both the anorthite and mullite phases and the glassy phase helping to fill the pores. M1 and M2 had the highest values of bending strength at 1150°C. According to the European Norm EN 14411, these mixtures are suggested for porcelainized and fully vitrified stoneware thanks to their low water absorption (*0.09* and *0.2%*, respectively). M3, M4 and M5 mixtures (with respective water absorption of *2.74*, *1.68* and *1.74%)* are suitable for the production of vitrified stoneware.

Electroplating of hybrid and nanocomposite Ni-W alloys as a coating against copper corrosion used into automobile industries

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Alloy's electroplating had widely used as sacrificed anodes to protect substrates from corrosion phenomena. In this work, we have investigated the effect of corrosion inhibitors and nanoparticles used as additives into the electroplating bath of Ni-W. Results proved that additives had enhanced the system Cu-Ni-W properties (thickness, hardness, conductivities and electrochemical behavior).

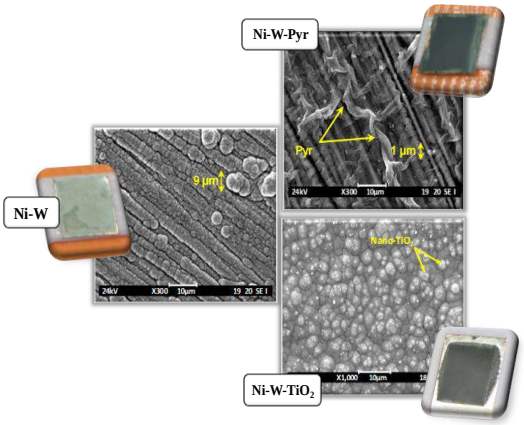

Fig. SEM micrographics of the pure, hybrid and nanocomposite Ni-W coating onto copper substrate [1, 2].

Key words: Electroplating, nanocomposites, inhibitors, corrosion

References

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CARACTERISATION OF CORROSION PRODUCTS OF STEEL E235 USED IN SHOCK ABSORBER INDUSTRY IN TUNISIA

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Most metals are not stable in contact with air. The various constituents of this one (oxygen, relative humidity, pollutants) has lead to the establishment of corrosion process. In particular, when a liquid film is formed and disappears cyclically at the surface of a metal object, there is an atmospheric corrosion process. This type of corrosion depends primarily on the exhibition medium, ie climatic conditions (humidity, temperature and pollutant content).
In this study we focused on the characterization of corrosion products identified by different analytical techniques on the surface of four iron plates E235 from the various stages of production of shock absorbers in Tunisia (raw sample, washed sample, sample treated with phosphate and painted sample).
The results obtained by X-ray diffraction, spectroscopy UV and Raman and fluorescence X have revealed the presence of ferrite (Density 7.87 $g.cm^{-3}$) in the first three samples. The magnetite Fe_3O_4 (density 5.20 $g.cm^{-3}$) and hematite (α Fe_2O_3 density 5.30 $g.cm^{-3}$) are products of iron corrosion and have been detected in the two first samples that are not treated against corrosion. In the third plate treated by phosphating (to ensure a much shorter processing time and to obtain an even layer of paint) we have identified mainly hopeite ($Zn_3(PO_4)_2$,4 H_2O) which forms a protective layer on the sample surface.

Key words: steel E235, shock absorber, corrosion, ferrite, magnetite, hematite.

Direct electrochemical reduction of dimetridazole as pretreatment to enhance biodegradability

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The relevance of an electrochemical pretreatment for the removal of dimetridazole, a biorecalcitrant antibiotic, was investigated [1]. The electrochemical behaviour of dimetridazole was examined by cyclic voltammetry showing an electroactivity in reduction. Direct electroreduction was performed in a home-made flow cell at -0.45 V/SCE on a graphite felt electrode. After a single pass through the cell, the analysis of electrolyzed solutions showed a total degradation of dimetridazole (99.5% elimination yield). The results were promising as the biodegradability of the effluent estimated from the BOD_5/COD ratio increased from 0.13 to 0.24. In the second part, main degradation by-products were identified.

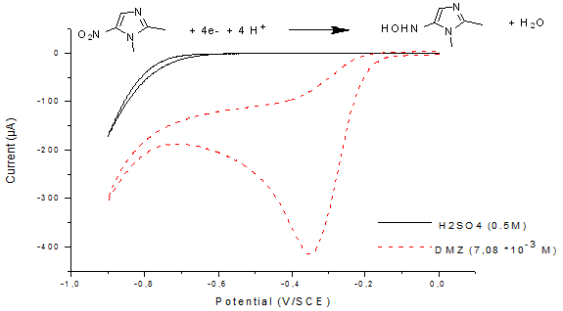

Fig. Cyclic voltammetry of DMZ solution (100 mg L^{-1}). Voltammograms were recorded at 100 mV s^{-1} in H_2SO_4 (0.5 M) on a glassy carbon electrode (7 mm^2), under argon atmosphere

Key words: Dimetridazole- Biodegradability- Direct electroreduction- Carbon felt electrode

References

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Toward Opto-electronic $Bi_{0.5}Na_{0.5}TiO_3$ Lead-free thin films

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[(2)] *Institute for Materials and Surface Technology, University of Applied Sciences, Kiel, Germany*

Sodium bismuth titanate doped rare earth elements, $Na_{1/2}[Bi_{1-x}\ RE_x]_{1/2}TiO_3$ (RE: La, Er, Eu and Ho) thin films were grown on Pt/Ti/SiO2/Si substrates via a sol-gel process. Our results indicate that thermal annealing in air after each layer of coating is effective in promoting crystallization of the film at a relatively low temperature of 650 °C. The resulting film is dense and well crystallized in the rhombohedral perovskite phase, and has good electrical and ferroelectric properties. Also, doped with optical active elements, the materials exhibit a very interesting optical properties. The BNT and NBT-doped rare earth elements films are expected to be a promising candidate for lead-free piezoelectric applications.

Keywords: Sodium bismuth titanate, thin films, opto-electronic applications.

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Highly efficient and eco-friendly extraction of neodymium using undiluted and non-fluorinated ionic liquids. Direct electrochemical metal separation.

Ramzi Zarrougui[a]

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A new approach for highly efficient extraction and recovery of neodymium from acidic medium by new undiluted ionic liquids (ILs) as extractants is developed. The distribution ratio D_{Nd} and the extraction efficiency %*E* was measured as a function of nitric acid concentration, extraction time, nature of the IL, concentration of the aqueous feed and molar quantity of complexing agent. The %E is close to 100% when the HNO_3 concentration is greater than 6M. The extraction process follows solvation mechanism, suggesting the involvement of three molecules in the extraction of one (Nd^{3+})-nitrate complex. The nature of bonding in the extracted complexes was investigated by various spectroscopic techniques. The recovery process of metal was performed by direct electrodeposition from the IL phase. The electrodeposition of metal was realized by potentiostatic electrolysis at −2V. The current efficiency evaluated from the mass change of the anode was more than 83%. The particules were obtained as small crystallite rods with various size approximately 3–70μm and diameters range of 0.5–30μm. The liquid-liquid extraction and the direct electrodeposition of neodymium by these H-phosphonate based ILs are a new efficient and eco-friendly process.

Graphical abstract.

Key words: Ionic liquids, Rares earths, Green separation, liquid-liquid extraction, Neodymium, Electrodeposition.

Poster Communications' List

(alphabetically authors' names)

Name	Ref
S. Abid, X. Zhang, O. Mongin, F. Paul, B. Jamoussi, C. Paul-Roth *FSB - Bizerte* Fluorenyl macrocycles for light-emitting molecular materials	**PC 01**
M. Abidi, S. López-Bernabeu, F. Huerta, F. Montilla, S.B. Hentati, E. Morallón *FSB - Bizerte* Chemical oxidative polymerization of 2-amino-4-tert-butylphenol and spectroelectrochemical characterizations	**PC 02**
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Posters Communications' Abstracts

FLUORENYL MACROCYCLES FOR LIGHT-EMITTING MOLECULAR MATERIALS

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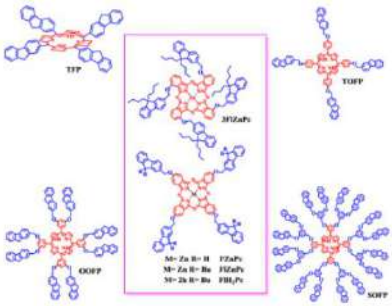
In 2004, we have reported the synthesis of a free porphyrin possessing four pendant fluorenyl arms directly connected at the meso positions **(TFP)** wich exhibited a high quantum yield **(24%)** demonstrating the good capacity of the fluorenyl units to enhance fluorescence by increasing the radiative process.[1-5] By the fact, that **(TFP)** exhibited good red chromaticity and enhaced emission efficiency. So, we have recently tested the corresponding platinum (II) complexes for the fabrication of red Organic Light Emitting Diodes **(OLEDs)**.[6-8] Then, three generations of porphyrin dendrimers bearing four **(TOFP)**, eight **(OOFP)** and sixteen **(SOFP**) fluorenyl units have been reported.[8, 9] The target was to obtain highly luminescent soluble organic compounds.
More recently, we designed a new series of fluorenyl phthalocyanines to improve the capacity of fluorenyl units to enhance quantum yield on various macrocyles. Their photophysical properties have been studied and improved the efficiency of the energy transfer from the donor to the acceptor and all the compounds were characterized by a red emission with a capacity to enhance a high quantum yield.



Key words: fluorenyl, Phthalocyanine, porphyrin, quantum yield, energy transfer…

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Chemical oxidative polymerization of 2-amino-4-tert-butylphenol and spectroelectrochemical characterizations

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Several studies have been concerned with the mechanistic aspects of the chemical oxidative polymerization of o-aminophenol. The synthesized poly(o-aminophenol), poly(OAP) shows a ladder structure with phenoxazine-type units [1, 2].

2-amino-4-tert-butylphenol monomer was polymerized from acidic medium using chemical oxidation procedures. In order to gain information on the chemical structure and redox behavior of the electroactive material *in situ* spectroscopic characterization techniques were employed. *In situ* FTIR and *in situ* UV-vis spectroscopies as well as ex situ XPS, strongly suggest that the chemical structure of poly(2-amino-4-tert-butylphenol) contains both phenoxazine rings and azo moieties. The former, which are more prevalent, arise from the usual *para*-coupling of aniline-derivative monomers [3], while the latter are probably stimulated by the presence of a voluminous alkyl group adjacent to the more reactive *para*- position. A possible chemical structure of such material has been depicted in Fig. 1.

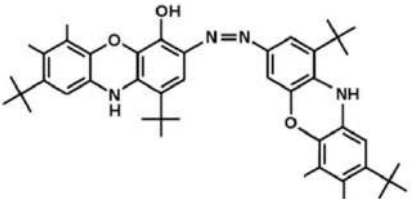

Fig. 1. Representation of the reduced form of a poly(2-amino-4-tert-butylphenol) fragment, as derived from *in situ* spectroscopic results.

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Corrosion inhibition of steel by prickly pear: Extraction, Characterization and Electrochemical Studies

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Aqueous and Methanol extracts of prickly pear was tested as corrosion inhibitor for steel using electrochemical impedance spectroscopy (EIS) in three different medias.
Impedance measurements showed that there are two phenomena in the inhibition process. The obtained results show that this plant extract could serve as an effective inhibitor for corrosion of steel in NaOH (0.1M), NaOH+NaCl (0.1M) and NaOH+NaCl (0.5M) medias. The aqueous extract obtained give inhibition around 63.3% in NaOH (0.1M) and the methanol extract obtained give inhibition around 82.2% in NaOH+NaCl (0.5M). The experimental data obtained from EIS method shows a frequency distribution and therefore a modelling element with frequency dispersion behavior, a constant phase ($CPE_{\alpha,Q}$) has been used. Graphical methods are illustrated by synthetic data to determine the parameter of CPE (α, Q). Chromatographic analysis, on chemical families present in the crude extract, were also carried out to find the main constituents responsible for corrosion inhibition properties of the plant extract.
In another hand, a theoretical study was performed in order to determine active sites in the studied molecules.

Keywords: steel, barrier properties, mechanical properties, EIS, DFT calculations, surface analysis.

Olefin Cross Metathesis for the synthesise of new glycostéroides

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The work presented here will focus on the synthesis of a new series of glycosteroids bearing a longer spacer between the carbohydrate and the steroid moieties. The Strategy of synthesis as showed on fig.1, the simple addition of allylamine to the two easy accessible acetylated carboxymethylglycoside lactones (CMGLs) containing a glucose or a cellobiose skeletons furnished in high yields the corresponding olefins **1a** and 1**b**. Interestingly, these two compounds with terminal double bond make a cross metathesis reaction with the readily accessible allyl cholesteryl ether **2**[1]. This reaction provided the corresponding adducts **3a** and **3b** in good wield.

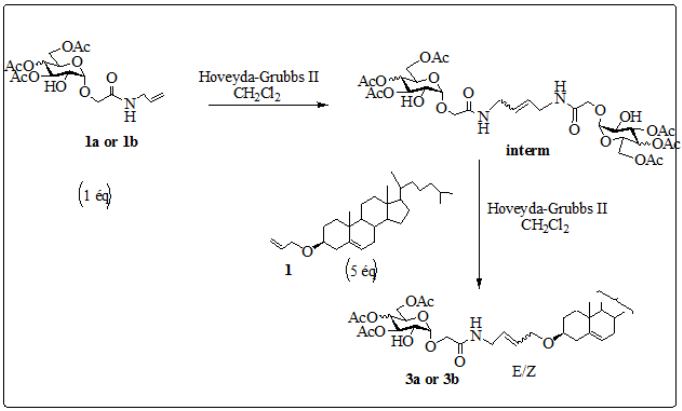

Fig.1 Cross Metathesis for the synthesise of new glycostéroides

Key words: cross metathesis, allyl cholesteryl ether, 'Grubbs-Hoveyda' catalyst

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Phytochemical Study and biological activity of the plant *Sixalix atropurpurea* subsp. maritima

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The study of the species Sixalix atropurpurea subsp. maritima covered a phytochemical screening to identify different families of chemical compounds in the leaves of this plant. Five Crude extracts were prepared, namely petroleum ether extract, dichloromethane extract, ethyl acetate extract, butanol extract and finally the aqueous extract.

Dosages of total polyphenols performed on these crude extracts were determined from calibration curves of gallic acid. The results show that the butanol extract and the acetate extract are richer in polyphenols than other extracts. The petroleum ether extract is free of polyphenol.

The evaluation of the antibacterial activity was determined by the diffusion method on Mueller-Hinton agar. Four bacteria were tested Staphylococcus aureus, Escherichia coli, and Pseudomonas aeruginosa. The results show that the three samples: butanol, acetate and dichloromethane showed moderate activity against E. coli, on the other hand the other two bacteria have resisted.

Keywords: *Sixalix atropurpurea* subsp. maritima, phytochemical screening, bacteriological activity.

CHEMICAL COMPOSITION, ANTIOXIDANT AND ANTIMICROBIAL ACTIVITIES OF ESSENTIAL OIL OF WILD TUNISIAN *PITURANTHOS CHLORANTHUS* (APIACEAE)

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The chemical compositions of essential oils of an endemic species of North Africa *"Pituranthos chloranthus"* collected, at the fruiting stage, from two areas of the South-East of Tunisia (Boughrara and Naffatia), were identified. The essential oils extracted from the seeds and stems by hydrodistillation, were analysed by gas chromatography/mass spectrometry (GC/MS). Seventy compounds were identified accounting for 91.8 to 99.6% of the whole essential oil showing the occurrence of monoterpene hydrocarbons. The most abundant compounds in the seeds oils collected from Boughrara and Naffatia respectively were α-pinène (32.17%; 37.73%) and β –pinene (27.83% ; 30.67%), while α -pinène (10.95%; 8.93%), β–pinene (8.1% ; 6.5%), β-phellandrene (17.19% ; 10.97%) and α-phellandrene (14.13% ; 7.24%) were found to be the major compounds in the stems oil. The antioxidant activities of essential oils was evaluated by three complementary assays, including 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical-scavenging activity, reducing power and Metal (Fe^{2+}) chelating activity. The obtained results revealed that the stems oils collected from Naffatia showed the highest reducing power with an EC_{50} value of 0.19 mg/ml. However, the greater metal chelating activity was recorded for the stems essential oils collected from Boughrara (IC_{50}=0.16 mg/ml). Nevertheless, all the studied essential oils of *P. chloranthus* did not display a noteworthy DPPH-radical scavenging activity. Finally, the antimicrobial activity of *P. chloranthus* essential oil was evaluated against six bacterial strains by the agar diffusion method and by determining the inhibition zone. Our results showed that the stems oils of two provenances exhibited a strong growth inhibitory activity against *klebsiella pneumonia.*

Key words: *Pituranthos chloranthus,* essential oil, chemical composition, antioxidant activity, antimicrobial activity.

Synthesis, structural and vibrational investigation for Tetrakis(2,6-diethylanilinium) decabromodibismuthate(III) hexahydrate $[C_{10}H_{16}N]_4Bi_2Br_{10}.6H_2O$

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The new organic-inorganic compound, Tetrakis(2,6-diethylanilinium) decabromodibismuthate(III) hexahydrate, has been synthesized and characterized by single-crystal X-ray diffraction at room temperature. It crystallizes in the monoclinic system ($P2_1/c$ space group). The structure consists of discrete dinuclear $[Bi_2Br_{10}]^{4-}$ anions, $[C_{10}H_{16}N]^+$ cations and water molecules. The organic part consists of two organic cations which orient their two amine groups to the neighboring anions. The inorganic entity is made up by $[Bi_2Br_{10}]^{4-}$ dimers composed of two equivalent irregular octahedra sharing one edge. The water molecules are sandwiched between the $[Bi_2Br_{10}]^{4-}$ anions and the neighboring $[C_{10}H_{16}N]^+$ cations. The crystal packing is governed by N(OW)-H…Br and N-H…OW hydrogen bonds and π–π interactions to built a three dimensional network (Figure). The nature of the inorganic polyhedra distortion, which can be attributed to the stereo-activity of the Bi(III) lone electron pair, has been studied. The infrared and NMR spectra were calculated by Density Functional Theory (DFT) using the B3LYP method with the 6-311++G** basis set. A good consistency was found between the calculated and experimental data.

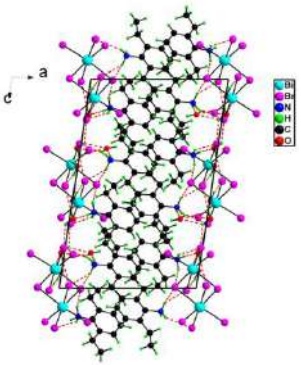

Figure: Projection of the $[C_{10}H_{16}N]_4Bi_2Br_{10}.6H_2O$ structure along the *b*-axis

Keywords: X-ray diffraction; Crystal structure; IR spectroscopy; CP-MAS NMR; DFT calculations

Synthesis, crystal structure and spectroscopic studies of a new 2-methylpiperazine-1,4-diium pentachlorobismuthate(III) monohydrate: $[C_5H_{14}N_2]BiCl_5.H_2O$

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We report in this work the synthesis, structural investigation and spectroscopic characterization of the organic-inorganic one-dimensional hybrid compound; 2-methylpiperazine-1,4-diium pentachlorobismuthate(III) monohydrate: $[C_5H_{14}N_2]BiCl_5.H_2O$. This compound crystallizes in the triclinic system with the space group P-1, with cell parameters: a = 8.041(3), b = 8.425(2), c = 10.912(2) Å, α = 80.65(2), β = 88.09(2), γ = 80.85(3)°, Z = 2 and V = 720.1(3) $Å^3$. The structural unit consists of one 2-methylpiperazine-1,4-diium dication, $[BiCl_5]^{2-}$ anions and water molecule. The anionic part built up by corrugated chains parallel to the *a*-axis of corner –joined $[BiCl_6]$ octahedra. These chains are themselves interconnected by N–H…Cl and N-H…OW hydrogen bonds originating from the $[C_5H_{14}N_2]^{2+}$ entities and waternmolecule to forming three-dimensional network (Figure). The ^{13}C CP-MAS-NMR spectrum shows five isotropic resonances, confirming the existence of five non-equivalent carbon atoms. This result is in good agreement with X-ray structure. The vibrational bands are identified by infrared spectroscopy.

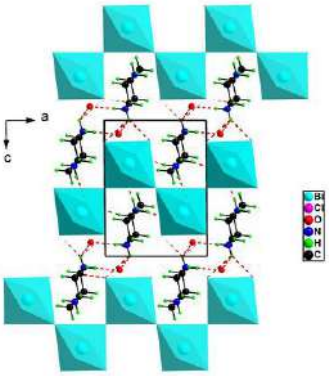

Figure: Projection of the $[C_5H_{14}N_2]BiCl_5.H_2O$ structure along the *b*-axis

Keywords: Halogenobismuthate(III); Crystal structure; Infrared spectroscopy; ^{13}C CP-MAS-NMR

DFT STUDY OF PHYSICOCHEMICAL PROPERTIES OF P_{n+1} AND AlP_n ($1 \leq n \leq 14$) CLUSTERS

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In this work we have investigated the properties of pure phosphorus and aluminum doped phosphorus nanomaterials (clusters) in the range size of 2 to 15 atoms by using the first principles Density Functional Theory (DFT) within the generalized density functional approximation (GGA) implemented in SIESTA simulation package. The calculated binding energy per atom as a function of size increases as the system size increase which indicating that the systems continue to gain energy during the growth process. In general the binding energies increase as the cluster size increases because the overall stability is expected to increase as the cluster grows larger and eventually reach to the bulk value of binding energy in bulk crystal. The stabilities and the related physicochemical properties of different AlP_n materials have been discussed in terms of their structures bond distances and the effect of the position of doping atom. The evolution of the electronic structure can be probed by calculating the characteristics of the energy gap between high occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO). The theoretical HOMO–LUMO gaps are generally decreases as the cluster size increases. This indicates that the AlP_n clusters with large size seem to be approaching the gap closure characteristic of a metallic state which is enhanced by the doping Al atom. On the other hand, we note that the systems with high value of HOMO-LUMO are less reactive and have the stronger chemical stability. The vertical ionization potential (VIP), vertical electron affinity (VEA) and chemical hardness (η) of P_{n+1} and AlP_n are simulated theoretically, analyzed and discussed for all the ground-state clusters. We obtained that the VIP values decreases as the cluster size increases indicating the high metallic character of the large size clusters. The VEA values are increased tendency with clusters sizes. This indicates that the P_{n+1} and AlP_n clusters with large sizes will liberate more energy when they capture one electron. The magnetic properties of P_{n+1} and AlP_n clusters are discussed according to the value of the total magnetic moment calculated for the lowest energies structure.

Key words: DFT, Al-P, electronic properties, magnetic properties, chemical properties.

SYNTHESIS AND SPECTROSCOPIC CHARACTERIZATION OF NOVEL CHIRAL CYCLOSULFAMIDES *N-N'*-SUBSTITUTED

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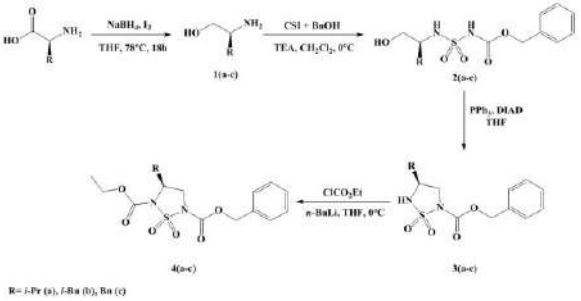
Cyclic sulfamides are an important class of compounds that are structurally similar to ureas and sulfonamides, which have been the subject of immense interest over the past decade [1]. They have been shown remarkable chemical, medicinal and pharmaceutical properties [2]. Cyclosulfamides have been recognized as analogs possessing a broad spectrum which exhibit as non hydrolyzable peptidomimetics [3], inhibitors of noroviruses [4], HIV-1 protease [5], human skin chymase [6], and human leukocyte elastase enzyme and cathepsin G [7], also as both agonists and antagonists of serotonin [8], histamine receptors [9], and constrained di- and tripeptides [10, 11].
Herein we report a convenient synthesis of novel *N-N'*-substituted 1,2,5-thiadiazolidine 1,1-dioxides starting from chiral amino acids in five step (reduction, carbamoylation - sulfamoylation, intermolecular cyclization via the Mitsunobu reaction and acylation).



Scheme 1. Synthesis of chiral cyclosulfamides

The structures of all the synthesized compounds were established on the basis of spectroscopic methods.

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Nucleation and Growth of Zn and Zn–Fe alloy electrodeposition from chloride bath

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The electrodeposition Zn-Fe alloy coating possess many excellent proprieties, such as excellent corrosion resistance, good welding and has been considered to be an alternative for pure zinc coating of steel products.
Phase transformations in alloys are generally controlled by nucleation and growth process, the slowest of which is the rate-limiting step.
Use adequate electrochemical techniques: cyclic voltammetry, chronoamperometry; the potentiometric electrochemical impedance microscopy and optical coatings obtained to determine:
*The synthesis of potential coatings imposed shows germination, growth and recovery of these germs. The deposition of Zn–Fe behaved anomalously and followed the mechanism of three-dimensional (3D) nucleation. With the increase of deposition potential, the grain growth changed from progressive to instantaneous.
*The SEM observations of the deposits obtained have shown the diversity of morphology as a result of their mode of germination and growth. They confirm that the deposits are more regular and homogeneous in thickness in the case of current zinc imposed unlike imposed potential. Incorporation of oxides or hydroxides in the coating for the impressed current alloy.
*The smallest corrosion rate is recorded for the potential alloy imposed against by imposed current speeds are similar for both types of deposits.

Key words: electrodeposition, Zn–Fe alloy, corrosion, surface modification, zinc, nucleation and growth, electrocrystallization.

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Synthesis and characterization of a novel inorganic/organic hybrid material based on strandberg type polyoxoanions and copper cation

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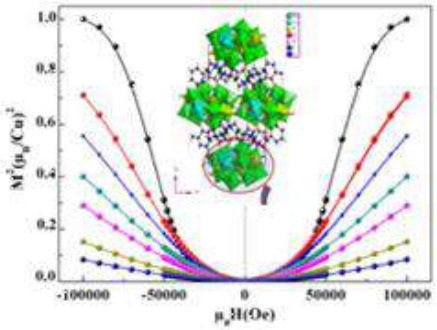
Polyoxometalates (POMs), as one kind of significant metal oxide cluster have been attracting extensive interest in fields such as catalysis, electrochemistry and magnetism **[1, 2]**. Among those, Keggin and Anderson-type POM linked transition-metal complex frameworks have been studied extensively, but similar studies on Strandberg-type POMs are limited till date. As a contribution to the elaboration of this compound family, we report in the present work, the synthesis and the characterization of novel Strandberg type molybdophosphonate complex $(C_5H_7N_2)_3[Cu(H_2O)_3HP_2Mo_5O_{23}].2H_2O$. The single crystal X-ray diffraction analysis reveals that this novel compound is composed of $[Cu(H_2O)_3HP_2Mo_5O_{23}]^{3-}$ anions, three distinct 2-aminopyridinium cations as counter-ions and two distinct crystallization water molecules. The crystal packing is stabilized by H-bonds and π-π interactions, resulting in a 3D framework. In addition, this new complex display good luminescent property at room temperature and the plot of the Kubelka-Munk function against energy shows that this hybrid compound is a potential semiconductor material. Moreover, the magnetization measurements reveal an antiferromagnetic interaction between the copper cationwith the Néel temperature at 7 K.

Keywords: Copper(II); Polyoxometalate; Crystal structure; Spectroscopy; Magnetic properties.



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Synthesis, structure and characterization of a novel Co(II) –P_2Mo_5 hybrid complex

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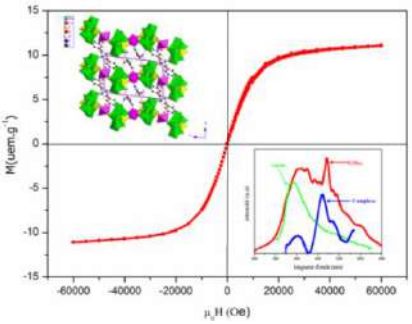
Polyoxometalates (POMs) have become one of the most heterogenous growing areas in inorganic chemistry, not only because of their structural versatility, but also owing to their encouraging applications in the fields of magnetism, photochemistry and electrochemistry **[1-2].** Among the versatile POMs building blocks, {P2Mo5} cluster form a noticeable structure that is characterized by its relatively small size and their low electronic density **[3]**. Therefore, the design of transition metal complex modified $\{P_2Mo_5\}$ hybrids is a meaningful and challenging work. Taking this into account, we report here the design, synthesis, and characterization of novel Strandberg type polymer complex $(C_6H_{10}N_2)_2[Co(H_2O)_4P_2Mo_5O_{23}].6H_2O$. The single crystal X-ray diffraction analysis reveals that the structure of the complex exhibits $1D_\infty$ $[P_2Mo_5O_{23}]^{6-}$ anion chains separated by Co^{2+} cations. The protonated 3-(ammoniomethyl)pyridinium dication and the uncoordinated water molecules, located in the void spaces, connect adjacent chains into a 3D supramolecular framework through hydrogen bonds. The analysis of the optical reflectance data of this material reveals optical direct transition with band gap of 2.64 eV. In addition, this new complex display good luminescent property at room temperature and the magnetization versus temperature curve shows a PM – FM transition at 235 K. The hysteresis curve yields a saturation magnetic moment value μ_S^{exp} of 5.40 μ_B.

Keywords: Cobalt(II); Polyoxometalate; Crystal structure; Spectroscopy; Magnetic properties



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Poly(imide-triazolium): a new candidate for polymer electrolytes

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Poly(ionic liquid)s or polymeric ionic liquids (PILs) are a new class of polymer electrolytes which combines the properties of ionic liquids (ILs) and polymer architecture, and kept considerable attention in recent years.[1]
Fast advances in the chemistry, physics, and macromolecular architecture of PILs have prompted the development of new polyelectrolytes that have found applications in many fields, such as batteries, supercapacitors, solar cells, environment (water treatment), gas separation, catalysis, microelectronics (transistors, OLEDs), bio-related applications and so on...[2]
1,2,3-Triazolium-based poly(ionic liquid)s (TPILs) were the most recent addition to the pool of PIL materials.[3] Their synthesis benefits from the versatile, robust and orthogonal nature of CuAAC in combination with efficient quaternization and anion metathesis reactions. In this work we report the synthesis of a new poly(imide-triazolium) via the post-polymerization modification of its precursor the poly(imide-triazole). (Scheme 1) This material can be considered as a new candidate of solid polyelectrolytes for gas separation membranes.

Scheme 1: Synthetic method of Poly(imide-triazolium)

Key words: Poly(ionic liquid), CuAAC, poly(imide-triazole), poly(imide-triazolium)

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Coupling an advanced oxidation process with a biological treatment for the mineralization of Enoxacin

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Advanced oxidation processes (AOPs) present a great alternative for the treatment of water polluted by toxic and persistent compounds. However, the mineralization of such molecules by AOPs remains expensive and time consuming. To overcome these drawbacks, the present study examined the coupling of an AOP with a conventional biological treatment, consisting on an activated sludge culture, to treat Enoxacin, an antibiotic belonging to the family of Fluoroquinolones. The Enoxacin exists in some industrial livestock manure at a content of milligrams per liter and it is poorly degraded by treatment plants and water purification. The presence of this antibiotic in the water, even at trace levels, causes disturbances in the aquatic ecosystem. Therefore, the aim of this study is to assess the relevance and the feasibility of coupling the photo-Fenton process with a conventional biological treatment for Enoxacin mineralization.

Initially, the work was devoted to the monitoring of the mineralization, the oxidation and the biodegradability improvement of Enoxacin during its irradiation, in optimal operating conditions. A pre-treatment duration was fixed according to the found results and only 60 min of irradiation by the photo-Fenton process were needed to improve the biodegradability of the target molecule. Subsequently, an activated sludge culture for the irradiated solutions was carried out during 10 days and showed that 2 days of culture were sufficient to significantly reduce the TOC values, reaching 69 % of final mineralization yields.

Key words: Enoxacin, photo-Fenton, biodégradability, coupling, activated sludge, culture.

1,3-DIPOLAR CYCLOADDITION OF A CHIRAL NITRONE TO (*E*)-1,4-DICHLORO-2-BUTENE: A NEW SYNTHETIC ROUTE TOWARD (2*S*,3*S*,4*R*)-4-HYDROXYISOLEUCINE.

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Our group has reported an efficient synthesis of (2*S*,3*R*,4*R*)-4-OH-Ile with total control of the stereochemistry [1], and approaches towards analogues of (2*S*,3*R*,4*S*)-4-OH-Ile, prepared either from D-glucose [2], or by cycloaddition, including a direct access to 4(*S*)-4-hydroxy-L-ornithine [3]. We report herein a stereocontrolled synthesis of (2*S*,3*S*,4*R*)-4-OH-Ile by a 1,3-dipolar cyloaddition reaction between a (–)-menthone-derived nitrone and (*E*)-1,4-dichlorobut-2-ene [4].

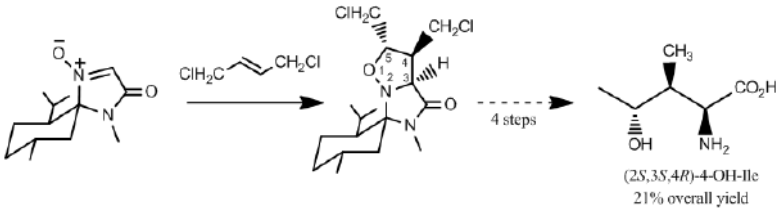

Fig. Synthesis of (2*S*,3*S*,4*R*)-4-OH-Ile

Key words: menthone, chiral nitrone, 1,3-dipolar cycloaddition, isoxazolidines, 4-hydroxyisoleucine.

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Stereoselective synthesis of 1,2,3-triazolyl-functionalized isoxazolidines, via two subsequent 1,3-dipolar cycloadditions, as precursors of un-natural amino-acids

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Our previous reports showed the potential of stereocontrolled cycloadditions to access original amino acids [1] as enantiopure (2*S*,3*R*,4*R*)-, or (2*S*,3*S*,4*R*)-4-hydroxyisoleucine [2]. As the simultaneous creation of several stereogenic centers impacts favorably atom economy, these cycloaddition routes are advantageous, as compared for example with a multi-step route from D-glucose to analogues of (2*S*,3*R*,4*S*)-4-hydroxyisoleucine. With these considerations in mind, we reasoned that the cycloadducts obtained could be elaborated further by application of a simple and high-yielding protocol from the panel of methods referred to as "click-chemistry" [3]. We report herein a stereoselective synthesis of isoxazolidines, by 1,3-dipolar cycloaddition of nitrone derived from (–)-menthone with allyl bromide and a subsequent functionalization with 1,2,3-triazole moieties by Cu(I)-catalyzed azide-alkyne cycloaddition (CuAAC) applied to an azidomethyl intermediate.

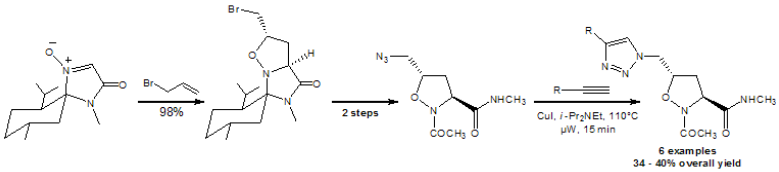

Fig. Synthesis of 1,2,3-triazolyl-functionalized isoxazolidines

Key words: menthone; chiral nitrone; 1,3-dipolar cycloaddition; isoxazolidine; 1,2,3-triazole; click chemistry.

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SYNTHESIS, PHYSICOCHEMICAL CHARACTERIZATION, CRYSTAL STRUCTURE AND HIRSHFELD SURFACE ANALYSIS OF A NOVEL ORGANIC DICHROMATE: $(C_6H_{18}N_2)[Cr_2O_7]$

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A novel organic dichromate named N,N,N′,N′-tetramethylethylenediammonium dichromate of formula $(C_6H_{18}N_{22})[Cr_2O_7]$ (I) was synthesized and characterized by FT-IR, UV, EDX and single-crystal XRD. Crystallographically independent dications in this crystalline phase have also been investigated in terms of their corresponding Hirshfeld surface. In the crystal, the cations and anions form a layered arrangement parallel to (110) plane. Normal and bifurcated N-H…O hydrogen bonds between the cations and anions and additional weak C-H…O interactions lead to the formation of a three-dimensional network structure. The three-dimensional Hirshfeld surfaces (3D-HS) **[1]** analysis shows closely similar Hirshfeld surface shapes for the two dications in the compound, reflecting similar intermolecular contacts and similar conformations **(figure)**. The two-dimensional fingerprint plots (2D-FP) **[2]** are quite asymmetric, due to the presence of more than one component (cation and anion). 3D-HS and 2D-FP reveal that the structure is dominated by H…O (63%) and H…H (37%) contacts.

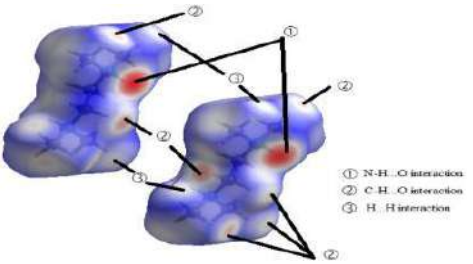

Figure: View of the three-dimensional Hirshfeld surfaces (3D-HS) for the two dications of (I). (3D-HS mapped with d_{norm}).

Key words: Crystal structure; N,N,N′,N′-Tetramethylethylenediammonium; Dichromate; Hirshfeld surface analysis; Fingerprint plots.

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Synthesis and Characterization of a New Organic Cation Sulfate: $[C_{12}H_{20}N_2][HSO_4]_2 .H_2O$

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A new organic cation sulfate, $[C_{12}H_{20}N_2][HSO_4]_2.H_2O$, was synthesized in aqueous solution. The structure was determined by X-ray diffraction method. The title compound crystallizes in the triclinic system P-1 with lattice parameters a = 7.73691 (19), b = 8.2371 (3), c = 14.2753 (5) (Å), α = 82.472 (3), β = 75.022 (2), γ = 76.614 (4) ° and V = 852.52 (5) $Å^3$. The structure is found to form infinite pairs of $[HSO_4]^-$ anions interconnected by O-H…O hydrogen bonds. These pairs are associated via O(W)-H…O and O-H…O(W) to form corrugated ribbons spreading along the b-axis direction **(Fig. 1)**. Solid-state ^{13}C and ^{15}N CP-MAS NMR spectra are in full agreement with the X-ray structure. The vibrational absorption bands were identified by infrared spectroscopy. DFT calculations allowed the attribution of the NMR peaks. The electronic properties such as HOMO and LUMO energies were carried out.

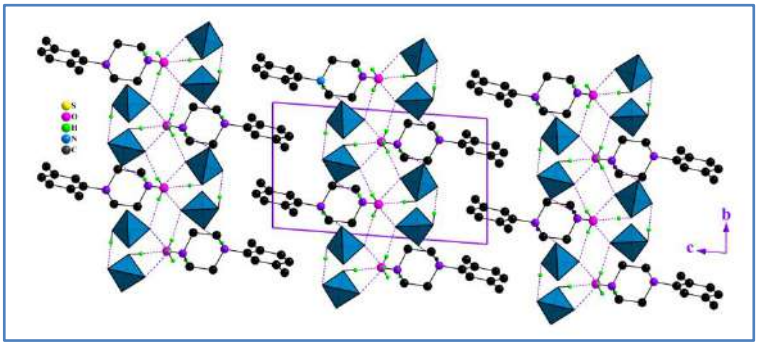

Fig. 1: Projection of the structure of $[C_{12}H_{20}N_2][HSO_4]_2.H_2O$ along the a axis

ULTRASONIC ACTIVATION OF THE ARYLATION REACTION OF STYRENE CATALYZED BY IRON

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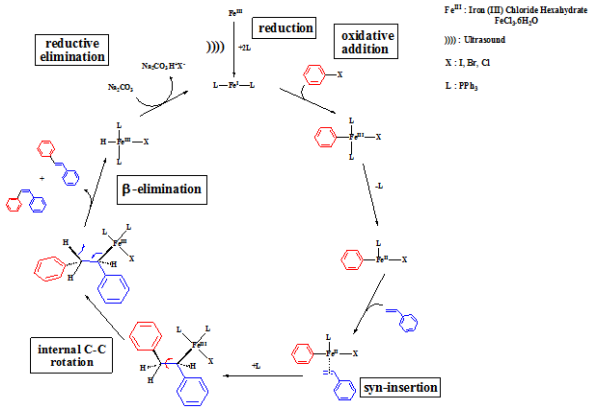
In recent work in our laboratory, we tested the arylation reaction of styrene in the presence of $Pd(OAc)_2$ and $PdCl_2$ **[1,2]** to expand the fields of application. In this work, we took the same reaction but in the presence of a new type of catalyst iron $FeCl_3.6H_2O$ given that it is advantageous from an economic view point, non-polluting and non-toxic. Our objective in this study was to investigate the influence of reaction parameters on the arylation of sp^2 hybridized carbon styrene. We propose also to study the effect of sonochemical activation by comparing the results to those obtained in classic mode (thermal). The yields obtained illustrate the effectiveness of our catalytic system and especially under ultrasonic irradiation. In parallel, we have noticed an improvement in selectivity arylation of β-carbon position of styrene. In this respect, we propose reaction mechanisms that could explain the results obtained.



Scheme. Proposed mechanism for the carbon sp^2 hybridized arylation reaction of styrene catalyzed by iron under Ultrasound.

Key words: Stilbene, iron, Arylation, Ultrasound.

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Organobentonite Synthesize and Physicochemical properties

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[2]*Laboratoire Physico-chimie des Matériaux Minéraux et leurs Applications Centre National de Recherches en Sciences des Matériaux, Technopôle BorjCédria, B.P. 73, Soliman, 8027, Tunisie.*
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Surfactant modified bentonite was prepared using hexadecyltrimethylammonium (HDTMA) bromide at three CEC levels (1, 2 and 3CEC), obtained organobentonite labeled H_1-bent, H_2-bent and H_3-bent were characterized by several methods such as X-ray diffraction (XRD), Infrared spectroscopy (IR), thermal analysis (TGA-DTG), (BET), Scanning electron microscope (SEM), transmission electron microscopy (TEM). X-ray photoelectron spectroscopy (XPS).. These methods were used to provide new visions into the interlayer structure of organoclays and to determine their physicochemical properties. XRD patterns showed the changes in the d(001) spacings, which gave details of the arrangement of surfactant in the interlayer's space of organoclays, IR spectroscopy showed the existence of HDTMA functional groups on bentonite surface. The BET surface area significantly decreased after modification due to the coverage of the pores sites into Na-bent.

Acid-base potentiometric titration and mass titration behavior of all samples were investigated. The point of zero charge (PZC) estimated by these both methods was about 6.4 for Na-bent and 7.8, 8.2 and 8.9 for H_1-bent, H_2-bent and H_3-bent respectively. Bentonite which has hydrophilic property in nature converts to hydrophobic after organophilisation. Hybrid material develops more positives surface charges with hydrophobic character and great basal spacing layer; therefore these materials can be very useful in industry applications and as adsorbent materials to remove anionic pollutants from wastewater.

Keywords: Bentonite, organoclays, surfactant, TEM, XPS, Potentiometric titration

NONLINEAR HAMMETT AND BRÖNSTED PLOTS IN REACTIONS OF MICHAEL ACCEPTORS WITH PIPERIDINE IN WATER

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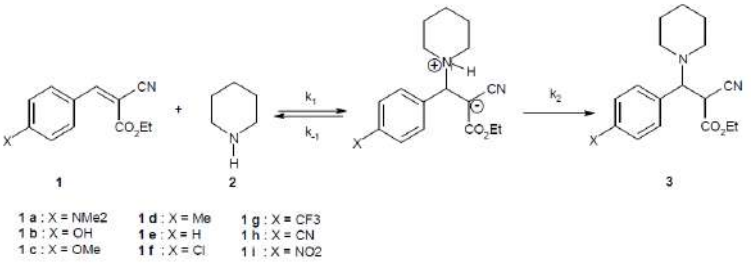
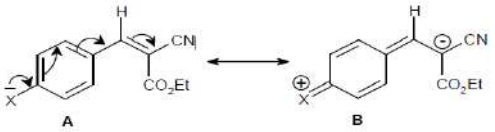
The seconde-order rate constants for the reactions of Micheal acceptors **1a-i** with piperidine have been determined photometrically in water at 20°C (scheme1).



Scheme 1

Hammett (log k *vs* σ) and Brönsted (log k *vs* pKa) plots for these systems show clear evidence of nonlinear behavior. However, the corresponding Yukawa- Tsuno plot is linear with r value of 0.64.

As can be seen, the origine of the negative deviation for the **1a-d** (X= NMe_2, OH, OMe, Me) that we propose is stabilization of the ground state through resonance interaction between the electron-donating substituant on the benzoyl moiety and the double band C=C as illustrated by resonance structures A and B.



Key words: kinetics, Hammett's equation, Correlation of Brönsted, Substituent effects, Michael acceptors.

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AN EFFICIENT SYNTHESIS OF NEW FUNCTIONALIZED 5-VINYL-1,2,4-TRIAZOLES FROM IMIDATES

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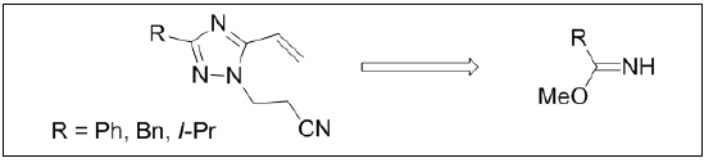
Triazole compounds have paying strong interest due to their pharmacological [1,2] and agricultural activities such as anticancer, anticonvulsant, antimicrobial, anti-inflammatory, antioxidant, antitubercular and pesticidal properties [3].
We propose in this work to present an efficient synthesis of vinyl-1,2,4-triazoles according to the following reaction scheme.



Many types of substituted hydrazines can lead to two regioisomeric triazoles. To determine the exact position of the pattern 2- cyanoethyl in the triazole we used two-dimensional NMR experiments HMBC.

Key words: Triazole dervatives, 2- cyanoethylhydrazine.

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Assessment of cadmium accumulation in pepper and agricultural soils

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Contamination of soils with heavy metals is a common problem throughout the world. The presence of heavy metals can lead to risks contamination of water, soil, transfers in the chain food, or reduce the biological activity of the soil (Sterckeman and al., 2008).

It is known that serious systemic health problems can develop as a result of excessive accumulation of dietary heavy metals such as Cd, Cr, and Pb in the human body (Oliver, 1997). Heavy metals are extremely persistent in the environment; they are nonbiodegradable and nonthermodegradable and thus readily accumulate to toxic levels (Goulding and al., 1998).

Heavy metals can accumulate in the soil at toxic levels due to the long-term application of wastewater (Bohn et al., 1985). One important dietary uptake pathway could be through crops irrigated with contaminated wastewater.

Soils irrigated by wastewater accumulate heavy metals such as Cd, Zn, Cr, Ni, Pb, and Mn in surface soil (Baize et al., 2002). Zinc and cadmium are generally considered as rather mobile metals, migrating to depth in soil solution as free ions (Citeau and al., 2003) but easily intercepted in subsurface horizons by clay-iron fabrics (van Oort and al., 2006). Lead is considered as a low- or non-mobile element (Semlali and al., 2004), strongly accumulating at the soil's surface due to complexation with organic matter.

For these reasons, it is necessary to understand the impact of cadmium in soils and develop techniques to determine the potential risks related to contaminated soils.

In this study an assessment is done concerning the impact of soils on heavy metals contamination of pepper. In this work, we have studied the distribution and migration of cadmium in different soil horizons it was applied on a site widely cultivated with pepper in Algeria. The soil was subjected to chemical characterisation, total metal quantification (Cd) sequential extraction as well metal speciation in 2011.

Cadmium concentrations exceeded the acceptable limits in soils, with strong surface accumulations and a rapid decrease of the contamination with depth. However, in the edible portion of pepper, the cadmium concentrations were higher than the permitted limits of the european standard.As for the plants grown, the study indicates a potential ecological risk of pepper contamination by cadmium.

At the end, the study of the localization of cadmium has also to follow the evolution of this metal as a function of depth. This could inform us about the potential risks associated with the contamination of the soil and the food chain in Hammam Boughrara.

Keywords: pepper, cadmium, soil contamination, sequential extraction.

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CRYSTAL STRUCTURE AND PHYSICOCHEMICAL PROPERTIES OF A NEW ORGANIC MONOHYDROGEN MONOPHOSPHATE: $(C_3H_9N)\ HPO_4$

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A new organic monohydrogenphosphate $(C_3H_9N)\ HPO_4$ is prepared by reacting H_3PO_4 acid with organic molecule 3-ethoxypropylamine. This compound crystallizes in the monoclinic crystal system, with space group $P2_1/c$. Unit cell parameters are a = 7.4896(20) A°, b = 11.7921(20) Å, c = 8.0342(20) Å, β= 110.130(20) Å, with, Z = 4. The structure is solved, using the direct methods and refined to a reliability R factor of 0.0566. The structure is built up from infinite inorganic $[HPO_4]^{2-}$ zigzag chains (Fig 1), parallel to the $\vec{c}$ direction localized at y = (2n + 1)/4 and between which are located the organic cations $(C_3H_9N)^{2+}$ (Fig 2). The cohesion of the framework is ensured by two types of hydrogen bonds N—H---O and O—H---O. The thermal properties of the compound are investigated as well as the IR properties supported by group theoretical analyses.

Key words: Hybrid; X-ray diffraction; Crystal structure; infrared spectroscopy; Thermogravimetric analysis.

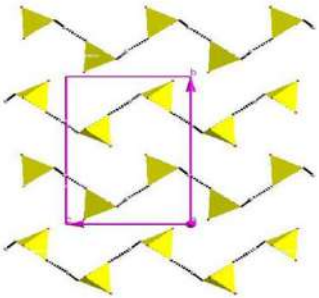

Fig.1. Projection of the anionic arrangement along the a-axis

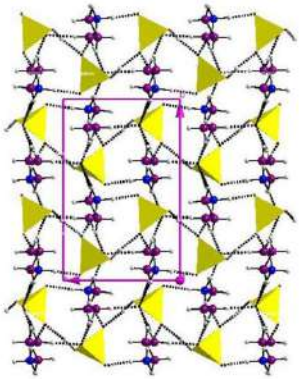

Fig.2. Projection along the a-axis of the atomic arrangement in :(C_3H_9N) HPO_4

A NEW MEMBER OF ARSENOMOLYBDATES: SYNTHESIS, AND PHYSICO-CHEMICAL PROPERTIES

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Polyoxometalates (POMs) have been a center of research interest for a long time, as they exhibit a great variety of structures and rich diversity of remarkable properties (1–2). In the past decades, an important advance in polyoxometalate chemistry is the polyoxometalate-based organic–inorganic hybrid materials. (3-4).
In this context, New organic–inorganic hybrid compound, with formula $Na_2(C_7H_{10}N_2)_2$ $[As_2Mo_6O_{26}].nH_2O$, was prepared and characterized by IR and UV–visible spectroscopies, TG analysis and X-ray diffraction techniques. The title coumpound crystallizes in the triclinic system, space group P-1 with a = 8.100(2) Å, b = 12.877 (4) Å, c = 20.275 (3) Å, α= 98.055°, β= 96.006°, γ= 93.455°, V= 2076 $Å^3$ and Z = 2.

Keywords: Polyoxomolybdates Hybrids, X-ray diffraction.

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Structural and thermal properties of $Fe_{55.8}Co_{14.2}Nb_6B_{24}$ nanostructured powder prepared by mechanical alloying

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The solid state reaction induced by mechanical alloying (MA) has been an intensive research subject because of its great potential for producing nanostructured and amorphous materials for technological applications [1]. A considerable attention has been devoted to Fe-based nanostructured alloys due to their exceptional mechanical and magnetic properties that originate from the fineness of the crystal size at the nanometer scale and the increased density of grain boundaries [2-3]. The mechanical milling of high purity elemental powders in planetary ball mill (Fritsch pulverisette 7) was used to prepare nanocrystalline alloy $Fe_{55.8}Co_{14.2}Nb_6B_{24}$. Structural properties, morphology changes and thermal stability have been investigated by X-ray diffraction, scanning electron microscopy (SEM) and differential scanning calorimetry (DSC). Slight contamination from milling tools and atmosphere were detected.

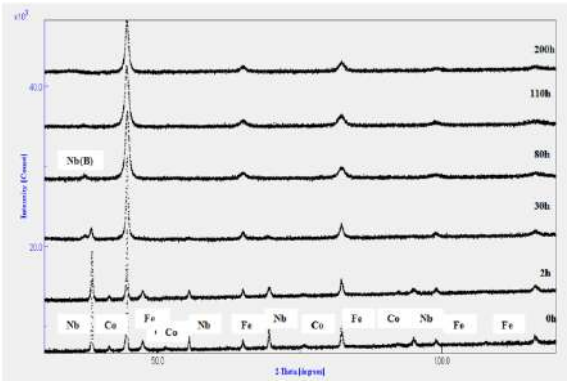

Fig. XRD patterns of the $Fe_{55.8}Co_{14.2}Nb_6B_{24}$ powders milled for various times.

Key words : Mechanical alloying, Fe-based alloy, DRX , DSC

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Synthesis and structural study of a new hybrid compound $(C_6H_{13}N_4)ZnCl_3$

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In the course of recent years, inorganic-organic hybrid compounds provide a class of materials displaying interesting technological importance [1].
In this work we report the synthesis of a new hybrid zinc complex $(C_6H_{13}N_4)ZnCl_3$. This compound crystallizes in the monoclinic system space group $P2_1/n$ with a= 7.789(5) ***Å, b=*** 13.793(5) ***Å,*** c= 10.431(5) ***Å,*** β = 90.000(5)**º,** ***Z=4,*** R (F) = 2.54% and wR (F^2) = 7.25%.
The title compound was prepared from aqueous solution and its structure was determined. The coordination geometry around the Zn atom is a tetrahedron, with the central Zn atom bounds to three Cl atoms and to one N atom of the hexamethylenetetramine ligand (fig).
In this material, the crystal packing is ensured by hydrogen bonds and π-π stacking interactions.

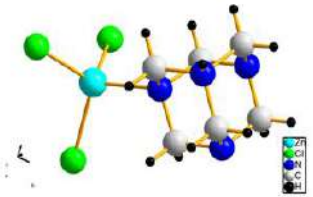

Fig. Asymmetric unit of the title compound

Key words: X-Ray Diffraction, Coordination Compound, Zinc Complex

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Synthesis, Structural Study and Physico-Chemical Characterization of a New Nitrate: $[C_6H_7ClN]NO_3$

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The synthesis, characterization, and understanding of the properties of energetic materials are the goal of many research groups. Because of the presence of both the oxidizer and the fuel parts in the same molecule, nitrate salts find applications in explosive, pyrotechnics and propellant formulations **[1]**. Nitrates are mainly produced for use as fertilizers in agriculture because of their high solubility and biodegradability and are also found to be powerful oxidizing agent that decompose and ignite at elevated temperature to give oxygen as one of the major products **[2]**. In this work, we report the synthesis and the characterization of a new organic nitrate, $[C_6H_7ClN]NO_3$.

The physicochemical properties of the title compound are discussed on the basis of a single-crystal X-ray diffraction investigation. The crystal structure determination reveals that the structure consists of one 3-chloroanilinium cation and one nitrate anion **(Fig. 1)**. The atomic arrangement can be described by corrugated layers of inorganic entities, made up of NO_3^- anions and NH_3^+ ammonium groups, developed parallel to the (a, b) plane at z = 1/4 and z = 3/4. The bodies of the organic entities are anchored between these layers and connect them via hydrogen bonds to form an infinite three-dimensional network **(Fig. 2)**. This compound is also characterized by infrared spectroscopy, solid state NMR and electronic properties such as HOMO and LUMO energies were carried out.

Keywords: Chemical synthesis; X-ray diffraction; NMR; Crystal structure; HOMO and LUMO energies

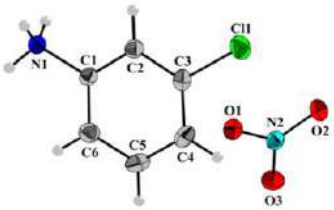

Fig. 1: Asymmetric unit of $[C_6H_7ClN]NO_3$.

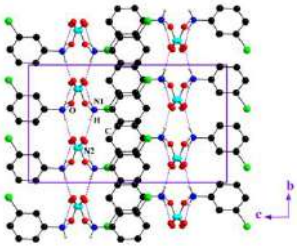

Fig. 2: The crystal packing of $[C_6H_7ClN]NO_3$ viewed along the *a* axis.

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Synthesis, characterization and crystal structure of a new compound $Na_3Bi(AsO_4)_2$

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During the exploration of the Na_2O-Bi_2O_3 -As_2O_5 system, single crystals sodium (I) bismuth (III) trisorthoarsenate (V), $Na_3Bi(AsO_4)_2$, have been prepared by solid state reaction and characterized by X-ray diffraction techniques and infrared spectroscopy. The title compound crystallizes in the monoclinic space group $P2_1/n$ with a=5.512 (1) Å; b=19.246 (1) Å; c=7.148 (1) Å, β=92.09 (1)° and Z=4.
Crystals of the title compound were grown by melting a mixture of Na_2CO_3, Bi_2O_3 and $(NH_4)H_2AsO_4$ in a molar ratio of Na : Bi : As = 6 : 1 : 3 in a porcelain crucible. After grinding, the obtained powder was heated at 350°C during 12 h in free air to remove volatile species and then kept for 7 days at 830 °C. A slow cooling to room temperature led to colorless plate crystals of $BiAsO_4$ together with colorless parallelepiped crystals of $Na_3Bi(AsO_4)_2$.
The main structural feature can be described as infinite anionic layers, made up of AsO_4 tetrahedra and BiO_5 polyhedra sharing corners, with Na^+ cations being located between the layers.

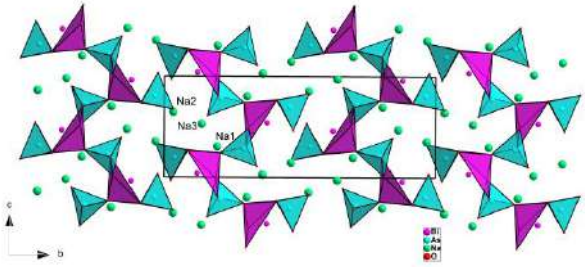

Fig. View of the structure $Na_3Bi(AsO_4)_2$ along[100] diretion

Key words: solid-state reaction synthesis, crystal structure refinement, IR spectroscopy, X-ray diffraction.

SYNTHESIS, CRYSTAL STRUCTURE, VIBRATIONAL SPECTROSCOPIC AND THEORETICAL STUDIES OF A NEW COPPER COMPLEX: $[C_6H_{16}N_2O]CuCl_4$

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During the last decades, the hybrid materials have devoted much attention owing to their special structural features and physical properties[1]. Among these compounds, the halide-transition metal complexes such as halocuprates have drawn growing interest due to their interesting structural and magnetic properties[2]. Herein, we report the synthesis, structure and characterization of a novel copper (II) halide complex: N-(2-hydroxyethyl)piperazine-1,4-diium tetrachlorocuprate. This compound crystallizes in the monoclinic space group *P*c, with the following parameters: a = 7.713 (1) Å, b = 6.336 (1) Å, c = 13.381 (3) Å, β = 108.888 (2), Z = 2 and V = 618.71 (2) $Å^3$. Its crystal structure consists of $[CuCl_4]^{2-}$ anions surrounded by $[C_6H_{16}N_2O]^{2+}$ cations. Three-dimensional frameworks of the compound are produced by N-H...Cl, N-H...O and O-H...Cl hydrogen bonding. Theoretical calculations were performed to study the molecular structure and the vibrational spectrum. All calculations were carried out at the DFT/B3LYP/LanL2DZ level of theory using Gaussian 09 program [3]. Good consistency is found between the calculated and the experimental vibrational wavenumbers.

Keywords: Tetrachlorocuprate; Crystal structure; DFT; Vibrational spectrum.

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Application of Polyaniline (PANI) as Hole Transport Material (HTM) in Quantum Dots Light Emitting Diodes (LEDs)

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Polyaniline (PANI) has been investigated as alternative hole transporting material (HTM) and hole injecting contact for Quantum Dot (QD) Light Emitting Diodes (LEDs) [1]. PANI thin films have been prepared by electrochemical polymerization on ITO substrates at room temperature. The polymerization of PANI was investigated in acidic medium using cyclic voltammetry methodology (CV) in potential range from -0.2 V to 1.2 V at scan rate of 20 mV/s [2]. The surface morphology and the thickness of the films have been observed using scanning electron microscopy (SEM). Different PANI layers were used as HTM in LEDs based on core/shell CdSe/CdS/ZnS QDs with ITO/PANI/QDs/BCP/Al structure. Prepared QDLEDs emits green light with a turn on voltage of 4 V and a maximum external quantum efficiency (EQE) of 10^{-2} %. The normalized electroluminescence (EL) intensity of the working device was studied at different voltages.

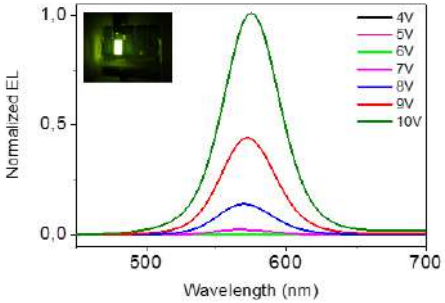

Fig. Electroluminescence spectra of the device at different voltages.

Key words: Polyaniline, electropolymerization, quantum dots, light emitting diode

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A GREEN METHODOLOGY FOR OBTAINING THE ENANTIOMERICALLY PURE CHIRAL WHITESELL AUXILIARIES

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Lipases are effective biocatalysts in several biotransformations tolerate access to chiral molecules under mild and eco-compatible conditions [1]. The enzymatic kinetic resolution is commonly used, they catalyze both transesterification in organic solvents (acetylation or alcoholysis), or by enzymatic hydrolysis [3].
In this work, we are interested to the enzymatic resolution of (*cis/trans*)-2-phenylcyclohexanol **1**, and that, based on the potential interest of the *trans*-2-phenylcyclohexanol (withesell's auxiliary) which play an important role in asymmetric synthesis [4]. We present two enantiocomplementary paths, promoted by *CAL-B* catalyzed kinetic resolution, affording success fully both enantiomers of this auxiliary. The first one, is the enzymatic transacetylation of a racemic mixture (+/-)-(*cis/trans*)-**1**, obtained by simple reduction of the corresponding ketone, which show that only the stereoisomer *trans*-**1** react with high enantioselectivity, 99%ee at 42% of conversion. And the last one, is the *CAL-B* hydrolysis assisted by carbonate salts, affording the other enantiomer, and only the *trans*-**1** stereoisomer with high enantioselectivity, 99%ee at 36% of conversion. These both approaches show high enantio- and diastereoselectivity of the *CAL-B.*

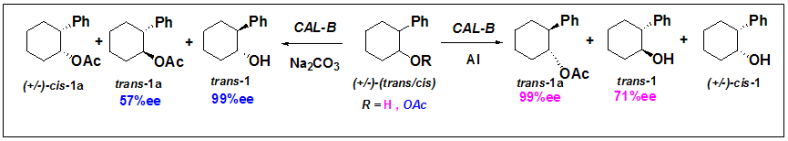

Fig1. Acylation/deacylation of trans/cis-phénylcyclohexanol

Key words: Whitesell's auxiliary, Enzymatic kinetic resolution, *CAL-B*, Acetylation, Alkalin-enzymatic hydrolysis.

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STUDY OF THE PHYSICAL AND RHEOLOGICAL BEHAVIOUR OF CHEMICALLY SYNTHESISED HYDROGEL

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In this study, we aimed to identify the effect of composition on the synthesized superabsorbent properties based on polyvinylpyrrolidone (PVP) and agar. Therefore, different compositions were prepared by varying the amount of Polyvinylpyrrolidone (PVP), agar and the thermal initiator Azobisisobutyronitrile (AIBN). Network parameters such as molecular weight between crosslinks $\overline{M_c}$, cross-linking density $v_{e,}$ mesh size ξ and elastic modulus G' were determined using swelling experiments and rheological analyses. It was found that increasing PVP content leads to an increase in the crosslinking density and a decrease in pore size. However, increasing agar and AIBN molar ratio leads to a decrease in the elastic modulus and cross-linking density.[1]

The polymer was characterized by Fourier transform infrared spectra analysis, X-ray Diffraction and thermogravimetric analyser.

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ELABORATION AND CHARACTERIZATION OF (TiO_2-SiO_2) NANOCOMPOSITE FOR NANOFILTRATION LAYER

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Although several studiesexplored the use of nanocomposites as additives in membrane structures, mixed matrix membranes still suffer from difficulties in synthesis and applications. In this work a new method of use nanocomposites as filter layer is proposed by using TiO_2/SiO2 nanocomposite with best performance. The nanocomposite TiO_2/SiO2 has been successfully elaborated using sol-gel technique and sintering at different temperatures. The obtained samples have been characterized by various techniques such as X-ray diffraction (XRD), Fouriertransform infrared spectroscopy (FTIR), Raman spectroscopyand photoluminescence (PL). The found results have been encouraging to use (TiO_2/SiO2) nanomaterial in nanofiltration membrane applications thanks to its porous texture and good properties.

Keywords*:* Sol-gel; TiO_2; SiO_2; nanocomposite; nanofiltration.

Ricinus communis extract effect on archeological bronze corrosion in aqueous chloride medium

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The protection of metals against corrosion was the concern of researches for a long time. Use of inhibitors is one of the most practical methods for protection against corrosion. A number of organic compounds have been reported as effective corrosion inhibitors . But, most of them are highly toxic to both human being and environment. It then becomes necessary to look for molecules that can provide the required properties to be used (active at low concentrations, etc.).Recent years several research teams focused on the study of a new class of corrosion inhibitors or green inhibitors.This work aims to develop a plant to our heritage vegetable Ricinus Communis (RC) used as a corrosion inhibitor of archeological bronze in corrosive environments. The effect of the addition of the inhibitor was performed with the stationary polarization curves in potentiostatic mode.

The extrapolation of Tafel lines in the corrosion potential has access to electrochemical parameters (Ecorr, βa, βc and Icorr).

It was found that the corrosion potential varied slightly after the extract addition.

The comparison of the electrochemical behavior of (Cu10Sn) in chloride medium with and without addition of the RC extract shows that it seems considered as a mixed inhibitor since it affects simultaneously both anodic and cathodic partial reactions.

The corrosion current was equal to 398,1 $mA.cm^{-2}$ for the control system, it decreased for the system –NaCl+ RC. The inhibition efficiency was lower than 55%.

Structural study of a new proton conducting solid: $Rb_4(SO_4)(HSO_4)_2(H_3AsO_4)$

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Single crystals of $Rb_4(SO_4)(HSO_4)_2(H_3AsO_4)$ are prepared by slow evaporation method at room temperature from an aqueous solution of rubidium carbonate, sulfur acid and arsenic acid. The structural properties of the crystal were characterized by X-ray single analysis (performed at room temperature), which revealed that $Rb_4(SO_4)(HSO_4)_2(H_3AsO_4)$ (abbreviated to RbHSAs) crystallizes in space group $P2_1$ with lattice parameters: a = 5.8682 (1) Å; b = 13.5799(3) Å ; c = 11.8098(3) Å and β = 94.7370(10)°. The compound has a unit cell volume 937.90(4) $Å^3$ and two formula units per cell, giving a calculated density of 2.741. The structure was solved from 5693 independent reflections and refined with 218 parameters yielded weighted residuals of 0.0853 and 0.0352 based on F^2 and F values, respectively. This structure is characterized by the presence of different groups SO_4^{2-}, HSO_4^- and H_3AsO_4 and the cation Rb^+. The cohesion of the crystal structure in this compound is provided by two types of bonds: ionic bonds linking the four Rb^+ cations to the anion $[(SO_4)(HSO_4)(H_3AsO_4)]^{4-}$ and hydrogen bonds connecting the sulphate groups and arsenates. The atomic arrangement of this structure can be described as resulting of the association of SO_4 tetrahedra and AsO_4 via asymmetric hydrogen bonds. Rb^+ cations are interposed between the chains while ensuring the stability of the structure. The protons involved in hydrogen bonds are located in asymmetrical positions between the oxygen atoms, giving an ordered character to the structure. It is noted that all the oxygen atoms in the tetrahedron $S(1)O_4$ are involved in hydrogen bonds. The tetrahedra $S(1)O_4$ alternate with the AsO_4 and interconnected with hydrogen bonds to form a chains in zigzag according to a direction.

The optimization of Acid Red 18 mineralization by Electro-Fenton Process

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The oxidation of Acid Red18, in aqueous solutions by electro-Fenton method was optimized. The reactor is an undivided glass electrochemical cell with a graphite-felt cathode and a platinum anode. The electrolyses were performed using different supporting electrolytes such as Na_2SO_4, NaCl, $NaNO_3$, and using different catalysts such as $CuSO_4$, $AgSO_4$, and FeSO4 in order to define the optimum degradation parameters. The value of 250 mA as applied current and Fe^{2+} as a catalyst with a concentration of 0.5 mM are found to be the optimum operating parameters for the efficient mineralization of $6.6X10^{-2}$mM of AR18. The evolution of its concentrations with processing time shows a pseudo first order kinetics. The repeatability of the experiments was investigated. Progress of decolorization through treatment time was monitored; it achieves 100% decolorization for the AR18 synthetic solution and 98% for an industrial effluent containing AR18. Mineralization of the dye solutions were examined by the estimation of the chemical oxygen demand removal, which achieves 73% for the synthetic AR18 solution and 50% for the industrial effluent after 4h of treatment. The total organic carbon indicates 23.5% of mineralization yields, after 4h of treatment and decay in the mineralization current efficiency was observed to be 23.29%. The ions chromatography analysis has shown that all nitrogen is converted to NO_3^- , however sulfate ions, were not detectable in the medium.

VALORIZATION OF ALUMINUM SCRAPS IN THE VIEW OF OBTAINGING A CERMET

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This work presents the implementation of cermet based on kaolin and aluminum powder mixtures. The operating conditions for producing composite materials have been optimized in order to reach good mechanical strength. The search for optimal development conditions has been carried out. The powder was uniaxially hot pressed under 150 MPa pressure to obtain cylindrical pellets. Mechanical properties, hardness and total porosities could be varied by different amounts of metal precursor. The total porosity and the hardness of the cermet sintered at 1350°C for 2h increase from 16,3 to 30,25% and 115 to 120 (at pH=4),respectively, with the addition of aluminum from 0 to 20 wt.%, whereas the mechanical strength, the best compression resistance is obtained for 4, 7 and 10 wt.% of commercial aluminum at pH=4.

Key words: kaolin, Aluminum, Cermet, Sintering, Mechanical properties

Application of Doehlert experimental design for boron removal by adsorption on freshly magnesium hydroxide

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Water contamination by boron becomes an important problem due to its widespread applications in industrial processes such as detergents, cosmetics, glass, fertilizers in agriculture. Boron is an essential element for plants, animals and humans but the excess and the deficiency of this element are toxic. The World Health Organization defined an upper limit of 2,4 $mg.L^{-1}$ of boron in drinking water. In the present study, boron removal by adsorption on freshly magnesium hydroxide was investigated and a Doehlert experimental design was applied to determine optimum adsorption conditions. Sorption uptake was highly dependent of initial boron concentration, magnesium amount and pH. In addition adsorption experiments were carried out to examine the effect of contact time and temperature. The maximum removal efficiency was reached within 10 min. According to the Doehlert design, the maximum boron removal was achieved under a pH 11 and a concentration of magnesium equal to 3,5 $g.L^{-1}$. For an initial boron concentration of 5 $mg.L^{-1}$, the boron removal efficiency reached 90%.

Key words: Boron removal, freshly magnesium hydroxide, adsorption, doehlert experimental design

ISOBARIC VAPOR–LIQUID EQUILIBRIUM FOR BINARY SYSTEMS OF C_1–C_4 *N*-ALKANOL + DIMETHYL DISULFIDE AT 40 AND 101.325 kPA

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Boiling temperature of pure liquids and isobaric vapor–liquid equilibrium (VLE) data for binary systems of dimethyl disulfide + methanol, dimethyl disulfide + ethanol, dimethyl disulfide + 1-propanol and dimethyl disulfide + 1-butanol were measured over complete composition range at p = 40 and p = 101.325 kPa using Fisher's ebulliometer. All the considered binary systems exhibit azeotropic points.

The experimental data were correlated by the Wilson activity coefficient model and were compared with original UNIversal Functional Activity Coefficient (UNIFAC) predictive model with satisfactory results. The experimental and calculated coordinates of invariant points are shown in table 1.

Table 1. Azeotropic Coordinates, Composition, $x_{1,az}$, and Temperature, T_{az} : Experimental and UNIFAC Predictions

System	p / kPa	T_{az} / K		$x_{1,az}$	
		Exptl.	UNIFAC	Exptl.	UNIFAC
Dimethyl disulfide (1) + methanol (2)	40.00	315.1	314.8	0.115	0.121
	101.325	336.9	337.0	0.110	0.098
Dimethyl disulfide (1) + ethanol (2)	40.00	327.8	328.3	0.242	0.175
	101.325	349.9	350.0	0.185	0.164
Dimethyl disulfide (1) + 1-propanol (2)	40.00	341.3	341.9	0.478	0.425
	101.325	366.1	364.5	0.369	0.366
Dimethyl disulfide (1) + 1-butanol (2)	40.00	351.1	350.9	0.759	0.743
	101.325	378.5	375.4	0.677	0.616

The effect of pressure upon the azeotropic composition was also studied. For the same pressure, increasing the carbon number of the n-alcohols causes the azeotropic point to move to dimethyl disulfide rich region. Molecular interactions between the unlike molecules in the binary liquid mixtures are discussed.

Key words: Dimethyl disulfide, Alkanol, Vapor–liquid equilibrium, azeotrope, Wilson, UNIFAC

STUDY OF THE INTERACTION OF METHYLENE BLUE WITH POLYSTYRENE -CO- STYRENE SULFONATE

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The interaction of the cationic phenothiazine dye, the Methylene Blue (MB) with poly-(sodium styrene sulfonate)*f*–co-(styrene)*1-f*, (PSSNa *f*), has been investigated by spectrophotometric method. The polyelectrolyte induced metachromasy resulting in a blue shift of the absorption maxima of the dye, in agreement with the formation of dye H-aggregates. The stability of the PSSNa-MB complexes was studied as a function of polyelectrolyte chain length, polyelectrolyte electrostatic charge density *f*, polyelectrolyte concentration, NaCl salt addition, tetrahydrofurane (THF) addition and THF treatment (consisting first to add to water, THF, which is then evaporated and replaced by the same amount of water) [1]. The stoichiometry of PSSNa-MB complex evaluated by the molar ratio method was found 4:1 for the fully charged PSSNa *f*=1, which is a high value in comparison to other systems (ex polyacrylic acid) where the stoichiometry was 2:1 [2]. This indicates that there is less overcrowding of the bound dyes on the PSSNa chain because of the aromatic rings steric effects. Reversal of metachromasy was observed upon salt and THF addition, while THF treatment does not affect the complex and allows recovering the initial complex. Finally, thermodynamic parameters of the interaction between the polyelectrolyte and the dye at different temperatures, namely free energy ΔG, the enthalpy ΔH and the entropy ΔS have been evaluated to determine the binding constant and as a consequence the stability of the complex.

Key words: Methylene Blue, poly(styrene-*co*-sodium styrene sulfonate), metachromatic complex, Thermodynamic parameters**.**

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trans-2,5-Dimethylpiperazine-1,4-diium bis(perchlorate) dihydrate: Crystal structure, Characterization and Hirshfeld surface analysis

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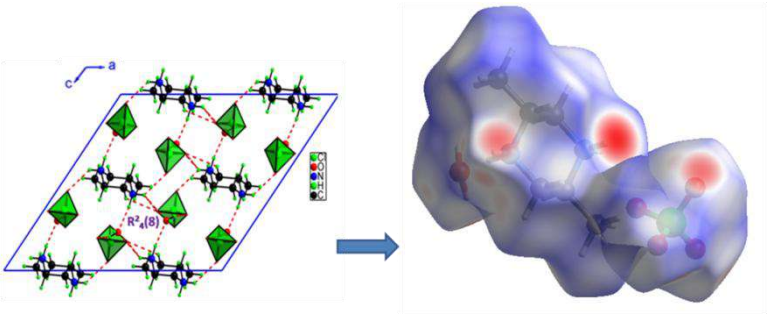
The crystal structure of a new organic–inorganic hybrid compound **$[C_6H_{16}N_2](ClO_4)_2.2H_2O$** was determined. The new *trans*-2,5-Dimethylpiperazine-1,4-diium bis(perchlorate) dihydrate has been synthesized and characterized by powder X-ray and single-crystal XRD methods, IR and UV–Vis. Single crystal X-ray diffraction study on grown crystal reveals that they belong to monoclinic crystal system with space group C2/c. The unit cell dimensions are: a = 16.8603(8) Å, b = 7.2655(3) Å, c = 14.4534(6) Å, β = 128,751 (1)° with V = 1380,78(10) $Å^3$ and Z = 4. The structure has been solved using a direct method and refined to a reliability R factor of 0.028. The asymmetric unit of the title hydrated molecular salt, $C_6H_{16}N_2^{2+}.2ClO_4.2H_2O$, contains a half dication (completed by inversion symmetry), a perchlorate anion and a water molecule. The extended structure consists of infinite chains of formula $[(ClO_4)H_2O]_n^{n-}$ ions extending along the b axis linked by Ow—H…O (w = water) hydrogen bonds. These chains are cross-linked by the dications via N—H…Ow and weak C—H…O hydrogen bonds, thus forming a three-dimensional supramolecular network.

FT-IR spectrum confirms the existence of the functional groups in the synthesized material. Three-dimensional Hirshfeld surface analysis and two-dimensional fingerprint maps reveal that the structure is dominated by H…O/O…H and H…H connections.

Graphical Abstract :



IMPACT OF AN INDUSTRIAL EFFLUENT ON THE DISTRIBUTION OF CARBONATED SPECIES IN AQUATIC ENVIRONMENT

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Industrial activities located in the Gulf of Gabes region are holding a strategic position in the Tunisian national economy. Unfortunately, these industries reject several gas, liquid and solid effluents. Some of these byproducts are discharged to the marine environment. This had a negative impact on the fragile marine ecosystem which has been so far characterized by its biological richness. Among the activities that contribute to this environmental crisis is the aluminum fluoride industry that discharges an acid fluoride rich liquid effluent. The pollution curried by this effluent could have a direct and indirect effect on many marine species.

This work focuses on the effect of a fluorine rich industrial effluent on carbonated species in seawater. The distribution of carbonated species in seawater will be predicted for several pollution scenarios. This allows assessing some of the detrimental environmental impact of the effluent discharge on some aquatic species sensitive to carbonated entities in seawater.

Key words: Industrial effluent, marine environment, carbonated species, distribution, environment impact.

Effect of chloride on the composition of the passive films formed at stainless steel (UNS N-08028) in industrial phosphoric acid solutions under natural conditions

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Chlorine in phosphate rock is found in the acid as chloride ions, which are most corrosive towards stainless steel [1]. In this sense, austenitic stainless steels are a good choice for phosphoric acid solution contaminated with 0.34wt% of $SO4^{2-}$, 0.026wt. % Cl^- and 0.32 wt. % F^- . Behaviour of a highly alloyed austenitic stainless steel (UNSN-08028) was evaluated in 50 wt. % industrial phosphoric acid medium at 80°C. The corrosion resistance was also investigated by increasing the chloride concentration up to 0.46% in order to improve the resistance of the passive film.
The main conclusions drawn from the research are summarized as follows:
When the content of chloride in the acid exceeds a critical threshold which was estimated at 0.24%, the phosphate layer is locally altered. This causes the deterioration of the passive film in the active sites and the formation of shallow pits, at the bottom of which precipitated calcium sulfate. This ensures repassivation. With the increase of immersion time and / or the content of chloride, pits' repassivation becomes difficult. The attack is initiated and the material lifetime becomes limited. Thus, the passivation / depassivation process of Sanicro 28 steel in industrial phosphoric acid medium depends essentially on the polyphosphate layer's quality and the precipitation of $CaSO_4$ at the bottom of pits.

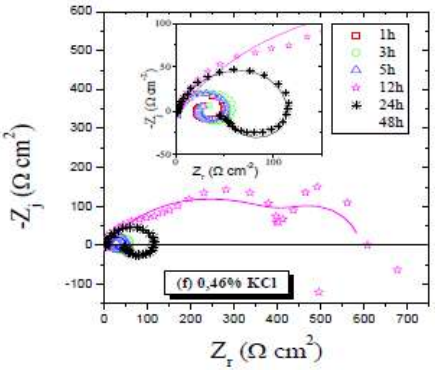

Fig1. Nyquist diagrams of Sanicro28 after 1, 3, 5, 12 and 24 h of immersion the 54 wt.% H_3PO_4 contaminated solution with 0.34 wt.% H_2SO_4 , 0.46 wt.% KCl and 0.32 wt.% HF.

Key words: Stainless steel, Chloride, EIS, Raman spectroscopy, XPS, Passive film

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DESULFATION OF POLYSACCHARIDES FROM SEA URCHIN *PARACENTROTUS LIVITUS*: ANTI-PROLIFERATIVE ACTIVITY AGAINST HUMAN CANCER CELL LINE

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The extraction of sulfated polysaccharides from *Paracentrotus lividus* was performed by papain digestion [1]. The extract was characterized by using size exclusion chromatography (SEC/MALS/VD/DRI), GC-MS, and FTIR Spectroscopy. The native carbohydrate exhibits a high molecular weight (≥2200 KDa) and it is composed mainly of arabinose, accompanied by other neutral monosaccharides (mostly galactose, glucose and fucose), significant amounts of uronic acids (18.4%) and higher proportions of sulfate (22.4%).
On the other hand, the polysaccharide SP inhibits strongly the proliferation of hepatic cancer cell human HepG2 up to 94.2 % at the concentration of 20 µg/mL. This activity could be attributed to the presence of sulfated groups in the structure of SP. In order to confirm this hypothesis, a desulfation was carried out versus time to eliminate sulfate groups. For this, methanol was used as a sulfate acceptor for the removal of sulfates from polysaccharides, when, the desulfation was carried out at 100 °C for 4 h [2]. The sulfate was removed up to 94.2 %. FTIR spectra displayed consistent decreases in intensities of bands at 1247 cm^{-1} and 854 cm^{-1}. The present strategy for the chemical modification of sulfated polysaccharides could be very important for the development of further anti-retroviral activities.

Key words: Paracentrotus lividus; Polysaccharide; desulfation; anti-proliferative activity

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Staudinger reductive amination: A powerful tool for heterobifunctional surfaces elaboration

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The design of surfaces with controlled morphology, both in dimension and shape, and also with regards to overall surface energy, is very important towards the design of sensors, catalysts, biomimetics, as well as for controlling surface wettability. Towards this last aim, the design of highly hydrophobic surfaces are very desirable for textiles, coatings, membranes, anti-icing, anti-fogging, and anti-biofouling applications [1].
One engineering route towards this type of surface design is the electrodeposition of conducting polymers [2]. This strategy has been reported as a very efficient way to get highly structured surfaces with controlled wettability [3]. The morphology and wettability of the final surface is deeply linked to the monomer chemical structure. As a consequence, to develop new surfaces, it is hardly needed to develop new monomers.
In this work, we report for the first time, the use of the Staudinger reductive amination to functionalize the monomer, this reaction followed by an amidification step which allows developing original heterobifunctional monomers. Here, we describe the synthesis of new heterobifunctional monomers and there electrodeposition of conductive surfaces. The properties of the newly formed surfaces were investigated for wettability, morphology and roughness analyses. All data were compared and the impact of the monomer chemical structure was discussed.

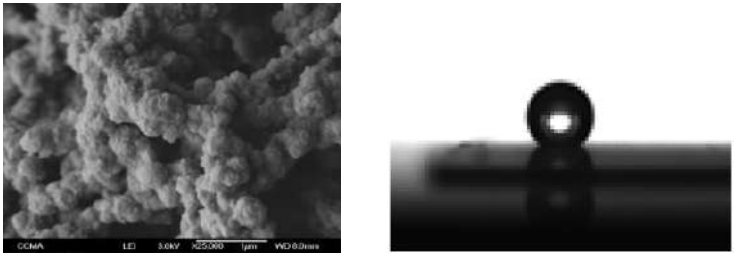

Fig.1: Example of SEM image for EDOT-C12, scale bar=1µm

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NEW BIS(OXAZOLINE)–VANADYL COMPLEXES SUPPORTED IN LAPONITE CLAY AS HETEROGENEOUS CATALYSTS FOR ASYMMETRIC OXIDATION OF METHYL PHENYL SULFIDE

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Chiral bis(oxazoline) ligands are used for the first time to promote the enantioselective vanadium-catalyzed oxidation of sulfides with alkyl hydroperoxides. Several bis(oxazoline)-VO complexes have been prepared and supported by cation exchange in Laponite clay. The substituent in the oxazoline ring, and the type of hydroperoxide are relevant parameters that control the activity and selectivity of the resulting catalysts. *Tert*-butyl hydroperoxide is more reactive but less enantioselective than cumenehydroperoxide, both with the neat and supported VO-bis(oxazoline) complexes. Activities and enantioselectivities obtained with the heterogeneous catalysts are always lower than in solution, and in general better and more consistent results are obtained with box(^{i}Pr) ligand which seems to be also the best chiral auxiliary in homogeneous phase, leading to a modest but significant enantioselectivity of 20% ee in heterogeneous phase and of 28% in homogeneous phase. The recovered of the best catalyst shows a decrease in the catalytic activity and in the enantioselectivity, in agreement with some decomplexation of the chiral ligand, whereas the supported vanadium species remain stable and recoverable.

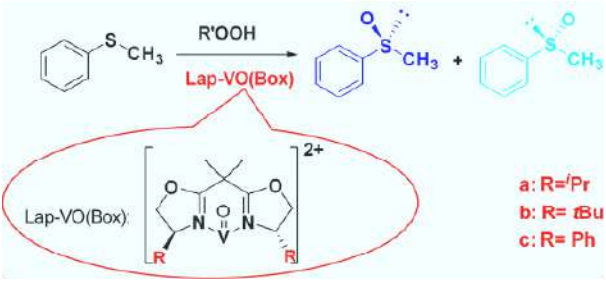

Fig. Asymmetric oxidation of methyl phenyl sulfide catalyzed by Lap-VO(Box)

Keywords: Laponite, bis(oxazoline), vanadium, catalyst, oxidation, asymmetry.

SYNTHESIS OF NEW ORGANOTIN COMPOUNDS

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Research Unit of Environmental Chemistry and Structural Molecular (a)

Organotin compounds showed countless applications in modern technology, especially in catalysis in the polymer chemistry(1). Similarly anticancer activity was reported by Hadjikakou and Hadjiliadis (2).
Our work aims the synthesis and structural study of new organotin compounds from organic ligands and their complexation with tin salts. The synthesis will be carried out by the three conventional methods, the synthesis wet, dry and hydrothermally.

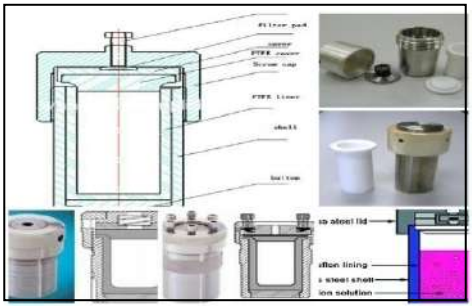

Fig. Hydrothermal Synthesis

Key words: organotin compounds, hydrothermal synthesis, crystal structure, X-ray diffraction, …..

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Study of diffusion of isotropic solvents into polymer network from swelling measurements

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The aim of this work is the synthesis and the characterization by swelling in isotropic solvents at equilibrium of crosslinked poly(PEA) network. The synthesis of photo-chemically polymer networks is carried out by exposing the mixture of compound: photoinitiator (Darocur 1173), the crosslinking agent (HDDA) and the ~~precursor~~ monomer (2-phenoxyethyl acrylate), under UV radiation. Polymer networks obtained are characterized by different physico-chemical techniques (DSC, TGA and FTIR). The swelling behavior of these networks was further investigated in different isotropic solvents ~~and for a~~ at different temperatures. The coefficient diffusion is calculated. ~~We deduce~~ The relation between the temperature and the diffusion process was determined. The Arrhenius parameters were also estimated. The Van't Hoff relationship was used to compute the entropy and enthalpy of sorption, and generally, the sorption process was found endothermic, and spontaneous.

Key words: isotropic solvents, crosslink network, swelling, coefficient diffusion.

InSitu elaboration, Characterization, Topology study and thermal stability of new Alkaline Earth Tetrazol compounds

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The [2+3] Cycloaddition between an azide and a nitrile, is a reaction of a great interest because of its efficiency, except that it presents a disadvantage of producing highly toxic and explosives intermediates, fortunately another synthesis protocol appears, which is called *InSitu* hydrothermal synthesis. It was applied in the synthesis of most of the products based on tetrazole. The compounds reported in this work are mono or dinuclear complexes, with hybrid and mono, di or polymeric forms, comprising alkaline earth metal and tetrazole ligands.
In this work we report five alkaline earth tetrazole compounds including a barium, calcium, magnesium and strontium: (I): $[Ca(C_4H_{18}N_{18})_2(H_2O)_6].4H_2O$, (II): $[Ba(C_4H_{18}N_{18})_2(H_2O)_8]$ and (III): $[Sr(C_5H_3N_6)_2(H_2O)_4].2H_2O$. (IV): $\{[Mg(C_2HN_9)(H_2O)_3][Mg_2(C_2HN_9)_2(H_2O)_6]0.5\{H_2O\}n$, and (V): $(C_5H_3N_6)[Mg(H_2O)_6]$. We report also herein TGA-DSC thermal behaviour studies as well as infrared and Raman spectroscopic analysis. This family of compounds has been the subject of some investigations for their intriguing esthetic structures and topologies. The both structures are synthesized under hydrothermal conditions, and crystals were obtained after slow evaporation of an aqueous solution at room temperature. At first sight the two structures of I and II are isomorphic with a dimeric unit that is strongly connected via hydrogen bonds, while the study of network of these two structures reveal two different topologies bcu-x/ 14-c net for compound I and fcu net topology for II. At second sight the crystal structure of III is composed of infinite 2D metal−organic layers that extend via hydrogen bonds into an 3D supramolecular framework, and the topological analysis of III reveal a 3,12-c binodal topological type. The structure of IV is amixed dimer–polymer based on magnesium ion centres and can be regarded as the first example of a magnesium–tetrazolate polymer in the crystalline form. The structure shows a complex three-dimensional hydrogen bonded network that involves magnesium–tetrazolate dimers, solvent water molecules and one-dimensional magnesium–tetrazolate polymeric chains. The crystal structure of (V) reveals a mixed anion-cation ribbons running along one direction and generating a 4,8-cflu network.

Key words: Alkaline earth, Tetrazole complex, Tetrazole polymer, In-Situ elaboration, Toplogy, Thermal stability

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Comparative Study of Noble Metal Doped-Zirconia Catalysts (Pt /Pd/ Ru – ZrO_2) for Catalytic Wet Air Oxidation of BPA in Aqueous Solution

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Bisphenol A (BPA) is used in the industry as a monomer in the production of polycarbonate and epoxy resins and as a varnish on the packaging of food products and pharmaceuticals [1]. Despite the widely use of this molecule, many reports show its repercussions on human health and it is classified as an endocrine-disrupting chemical (EDC) with estrogenic properties [2]. The effective removal of BPA was studied in the present work by catalytic wet air oxidation (CWAO) which is regarded as one of the most promising AOP techniques. Aqueous solution of (BPA) (10 mg/L) was degraded at 100°C and 20 bar of air in the presence of zirconia based metal catalysts. Zirconia was synthesized by sol-gel method then different metals (Pt/ Pd / Ru) with low amount (3wt%) were loaded by impregnation methods. The prepared catalysts were then characterized by X-ray diffraction (DRX), N_2 adsorption-desorption and hydrogen temperature-programmed reduction (H_2-TPR). Results of catalytic tests showed a significant enhancement of the catalytic activity of zirconia doped with Ru comparing to those doped with Pt and Pd.

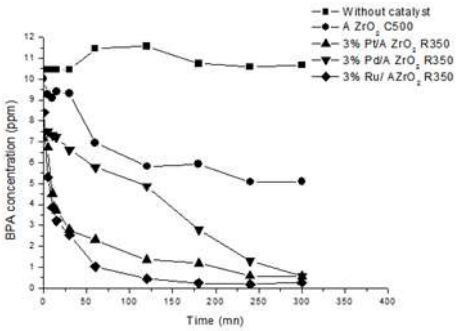

Fig. 1 BPA degradation as a function of oxidation time

Key words: CWAO, BPA, metallic catalysts, zirconia, sol-gel

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Encapsulation Of Anticancer Drug Doxorubicine Inside Dendrimers: A Therotical Study

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The drug molecule vectorization is a very promising approach in terms of both medical and economical factors for the delivery of active substances with low bioavailability. Symmetrical and branched macromolecules such as star polymers and dendrimers, seem to be more attractive solutions. Indeed, these structures can effectively combine a high stability in biological media and the ability to encapsulate active ingredients. Thanks to the well-defined architecture, they can achieve a high level of reproducibility, while avoiding the problem of polydispersity. In recent years, many dendritic structures have been proposed; however, the design of new effective dendritic nanocarriers is still relevant. This is due to many reasons such as related to biocompatibility, encapsulation efficiency of therapeutic agents, as well as economic reasons. Considering this, we present theoretical results based on density functional theory to study the encapsulation drugs by dendrimers .

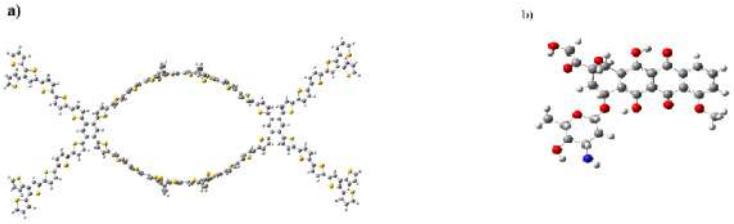

Figure: Geometry of (a) dendrimer and (b) Doxorubicine (C, O, H, N and S atoms are represented as: gray, red, white, blue, yellow).

Keywords : Dendrimers, DFT, anticancer drugs..

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TOTAL, SOLUBLE AND INSOLUBLE DIETARY FIBRE IN GLOBE ARTICHOKE (CYNARA SCOLYMUS L) AS AFFECTED BY GENOTYPE AND PLANT PART

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Dietary fibre is an important component of human's nutrition. It is the common name for all carbohydrate components occurring in foods that are non-digestible in the human small intestine. Dietary fibre plays an important role in decreasing the risks of many disorders such as constipation, diabetes, cardiovascular diseases (CVD), diverticulosis and obesity. This work provides new data on total (TDF), insoluble (IDF) and soluble (SDF) dietary fibre contents of different plant part (intermediate bract, external bract, leave and stem) of two globe artichoke varieties "Violet d'Hyère" and Blanc Oranais). Results of study showed that the variation in DF content was significantly influenced by plant part and artichoke genotype. Total dietary fibre content in different fibre sources ranged between 44,62% and 77,53% DW, where the lowest TDF content was determined for the leave of "Violet d'Hyère" variety and the highest for the external and intermediate bract of "Blanc Oranais" variety. The content of TDF in "Blanc Oranais" was significantly higher comparing with "Violet d'Hyère" variety.

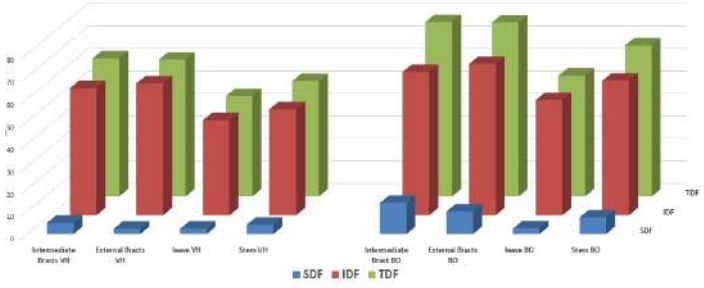

Fig. Variation in total, soluble and insoluble dietary fibre (DF) content among different plant part (intermediate bract, external bract, leave and stem) of two globe artichoke varieties "Violet d'Hyère" and "Blanc Oranais"

Key words: Total dietary fibre, soluble and insoluble fibre globe artichoke, plant part, genotype

N-HETEROCYCLIC CARBENES PALLADIUM COMPLEXES CATALYZED SUZUKI MIYAURA AND SONOGASHIRA CROSS COUPLING REACTIONS

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N-heterocyclic carbenes palladium complexes have become a very important class in N-heterocyclic carbene chemistry and they show good catalytic activity for some C-C coupling reaction, such as Suzuki Miyaura and Sonogashira reactions [1]. This coupling reaction has been utilized in the synthesis of a natural product, pharmaceuticals and biologically active molecules [2]. In this work, we synthesize diaryl derivatives using cross-coupling reaction of aryl chlorides and bromides with phenyl boronic acid catalyzed by palladium complexes in toluene and DMF/H_2O. On the other hand palladium complexes based on phosphine ligands exhibited very high activity for sonogashira reaction of aryl bromides with phenylacetylene in the presence of DMF as a solvent and KOtBu as a base. The reaction can be carried out under argon atmosphere at 100°C. To demonstrate the general applicability the sonogashira products have been synthesized in good to excellent yields 62 – 100% under mild conditions.

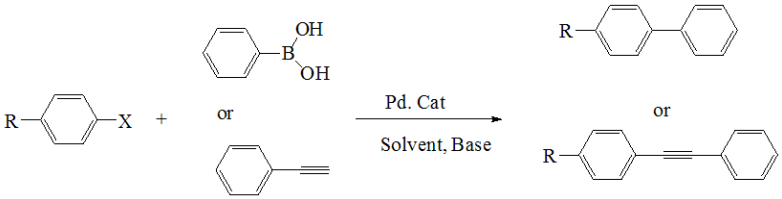

Fig. Sonogashira and Suzuki reactions catalyzed by Palladium complexes.

Key words: Cross coupling, palladium complexes, Sonogashira reaction, Suzuki Miyaura reaction.

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Synthesis and reactivity of ferrocenylallylamine derivatives

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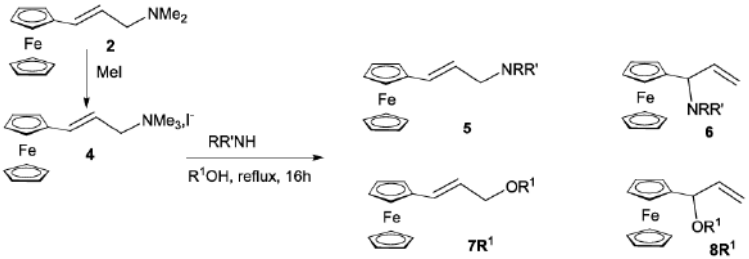
Many efforts have been devoted to the synthesis of allylamines [1] because of their interesting biological activities [2] and because they are valuable synthetic intermediates [3]. However, although ferrocene derivatives are of great interest in many fields like asymmetric catalysis, [4] biology, [5] materials chemistry,..., only very few ferrocene-containingallylamines have been described to the best of our knowledge [6]. Thus, our research focuses on an efficient and straightforward general synthesis of new ferrocenyl allylic amines.

((2-ferrocenylvinyl)methyl)dimethylamine **2**was obtained in good yields with perfect regio and stereoselectivity by reaction of dimethylmethylideneammonium salts on vinylferrocene. After methylation of amine 2, ((2-ferrocenylvinyl)methyl)trimethylammonium **4** was obtained in high yields and used to transfer ferrocene-containing allyl group on various amines. By controlling reaction conditions, various linear and branched ferrocenylallylamines could be obtained with good yields and selectivities.



Scheme: synthesis of allylammonium 4 and its reaction with various amines in alcoholic solvents.

Key words: Vinylferrocene, Allylamines,regioselectivity,amine.

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PLANNING OF THE REAL AGRI-FOOD SUPPLY CHAIN WITH THE COST OF WASTE DEGRADATION

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The supply chain of agricultural products has received a great deal of attention lately due to issues related to public health. Something that has become apparent is that in the near future the design and operation of agricultural supply chains will be subject to more stringent regulations and closer monitoring, in particular those for products destined for human consumption (agri-foods). The supply chain of agri-foods, as any other supply chain, is a network of organizations working together in different processes and activities in order to bring products and services to the market, with the purpose of satisfying customers' demands. This work is concerned with the planning of a real agri-food supply chain for milk products for the city of Tlemcen in Algeria. More precisely the problem is to redesign the existing supply chain and to optimize the distribution planning. Furthermore, environmental costs of road transportation in terms of CO2 emissions are taken into account in the computations. The management of perishable products is a relevant issue in the ASC management domain, since the sellers cannot wait for the best favorable market conditions unless the quality and safety of their products deteriorate. These products must therefore be rapidly shipped from the sellers to the customers. In addition to these factors, the variability in the demand and price of these products with limited shelf life adds complexity to the supply planning of these already critical supply networks. Food safety has become the subject of significant public and regulatory attention in various countries. Food safety is often an experience or credence attribute that consumers cannot detect through search activities prior to purchase.
The entire problem is decomposed into three problems, and each problem is solved in sequential manner, with LINGO optimization solver (Version12.0).

Key words: Agri_food Supply chain; distribution network; optimization; CO2 emissions .

SYNTHESIS AND CHARACTERIZATION OF WILLEMITE POWDER

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Divalent metal orthosilicates are compounds of general formula M_2SiO_4 widely applied in various technologies. This work is a contribution to the study of these materials. It is dedicated to the preparation of Zn_2SiO_4 (willemite) by the sol-gel method. The synthesis was held at different temperatures (500 - 1100°C) for 6 hours of calcination. The calcined samples were characterized by X-ray diffraction (XRD), infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The results showed that the synthesized Zn_2SiO_4 powders are mainly composed of α-Zn_2SiO_4 in the presence of β-Zn_2SiO_4. The formation of willemite was also highlighted by the presence of infrared vibration bands characteristic of SiO_4 and ZnO_4 groups. We noticed an improvement of the Zn_2SiO_4 crystallinity with increasing calcination temperature. In addition, an increase of the average crystallite size was noted.

Keywords : Synthesis, Sol-gel, Willemite, XRD, Crystallite

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SYNTHESIS OF NEW DISPIROPYRROLIDINE DERIVATIVES *VIA* ONE-POT AZOMETHINE YLIDE CYCLOADDITION. EXPERIMENTAL AND COMPUTATIONAL APPROACH TOWARD REGIO- AND DIASTEREOSELECTION

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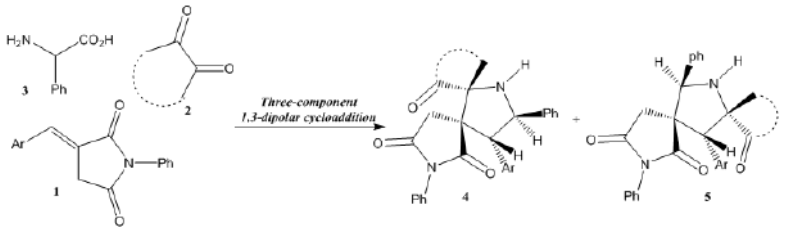
Multifunctional polycyclic spiropyrrolidine represent a privileged heterocyclic skeleton **that** exist in a large family of naturally occurring alkaloids and synthetic products exhibiting a plethora of biological activities [1]. On the other hand, the pyrrolidine-2,5-dione scaffold is a central component of numerous alkaloids as well as a great variety of synthetic compounds endowed with diverse bioactivities [2].

Prompted by the synthetic interest and applications of spiropyrrolidine and pyrrolidine-2,5-dione skeletons as part of our research on *N*-spiroheterocycles, we describe herein the synthesis of a novel series of dispiropyrrolidines *via* one-pot multicomponent 1,3-dipolar cycloaddition reaction of *(E)*-3-arylidene-1-phenyl-pyrrolidine-2,5-diones **1** with azomethine ylides. The latter have been generated *in situ* by reaction of various cyclic ketones **2** and 2-phenylglycine **3**. The stereochemistry of these *N*-heterocycles has been confirmed by several X-ray diffraction studies. To enlighten the observed regio- and stereoselectivity of the [3+2] cycloaddition, DFT calculations at the B3LYP/6-31G(d,p) level were employed.



Scheme 1. Synthesis of dispiropyrrolidine derivatives **4** and **5**

Key words: Dispiropyrrolidines, DFT study, 1,3-dipolar cycloaddition.

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HIGHLY STEREOSELECTIVE CONSTRUCTION OF NOVEL DISPIROINDOLIZIDINE DERIVATIVES *VIA* MULTICOMPONENT 1,3-DIPOLAR CYCLOADDITION

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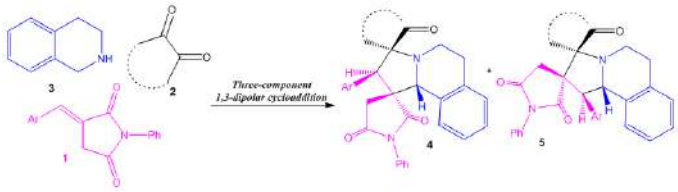
Functionalized indolizidine as structural motifs have drawn tremendous interest in the area of synthetic organic chemistry and medicinal chemistry since they are the ubiquitous substructures in a broad range of bioactive natural isolates and pharmaceutically important compounds [1]. On the other hand, substituted pyrrolidine-2,5-dione-based scaffolds represent a large family of compounds that have been the subject of synthetic interest owing to their medicinal promise and overall versatility as natural product building blocks [2].

Herein, in continuation of our efforts on the design and efficient methods for the synthesis of new *N*-spiroheterocycles, we report herein the synthesis of an extended series of novel dispiroindolozidine derivatives by a one pot three component 1,3-dipolar cycloaddition reaction of *(E)*-3-arylidene-1-phenyl-pyrrolidine-2,5-diones **1** with azomethine ylides generated *in situ* from various cyclic ketones **2** and 1,2,3,4-tetrahydroisoquinoline **3** by 1,5-prototropic shift route. The stereochemistry of the spiroadducts **4** and **5** has been confirmed by several X-ray diffraction studies.



Scheme 1: Synthesis of dispiroindolizidines **4** and **5**

Key words: Three-component 1,3-dipolar cycloaddition, Dispiroindolizidines, Azomethine ylides.

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Synthesis and Characterization of New Nonionic Surfactants

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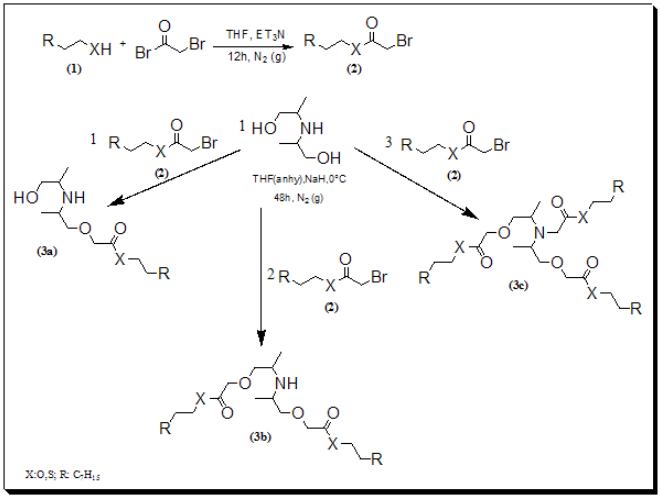
In this paper, a series of new mono-, bi- and tri-catenary nonionic surfactants (3) were synthesized from reaction of hydrocarbon thiols and alcohols with bromo acetyl bromide in anhydrous THF at 0°C for 48h under N_2 atmosphere. This reaction leads to the formation of intermediates (2) [1]. We add to these intermediates bis (2-hydroxyl propyl) amine to obtain surfactants (3), according to literature procedure (scheme 1) [2].



Scheme 1

Surface activity of these new surfactants should be evaluated on the base of surface tension (γs) measurements. We will also discuss the influence of oxygen and sulfur on yield and surface tension measurements.

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Synthesis, X-Ray Structure, Spectroscopic, Electrochemical Properties and DFT Calculation of Tetrakis(Cyclohexylammonium) Chloride Hexachlorobismuthate Dihydrate

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A novel bismuthates(III) complex, $(C_6H_{14}N)_4[BiCl_6]Cl.2H_2O$, is synthesized and characterized by X-ray diffraction, FT-IR spectroscopy, DFT and cyclic voltammetry. Its structure consists of protonated cyclohexylamine cations, discrete $[BiCl_6]^{3-}$ anions, chlorine anions and water molecules. These components are connected into a three-dimensional network via hydrogen bonds. In addition, molecular geometry in the electronic ground state, vibrational wanenumbers and Natural bond orbital (NBO) analysis were performed by means of density functional theory (DFT/B3LYP) with LanL2DZ basis set. Theoretical results are in good agreement with experimental data. The NBO analysis reveals that the N–H....O, N–H....Cl and O-H…Cl intermolecular interactions significantly influence crystal packing in this molecule. Moreover, the electrochemical behavior of this complex was investigated in DMSO by cyclic voltammetry (CV).

Keywords: bismuthates(III) complex, Crystal structure, FT-IR, DFT, NBO, cyclic voltammetry.

Synthesis and characterization of $CaCu_{3-x-y}Ni_xMg_yTi_4O_{12}$ By solid-state reaction

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A new material named $CaCu_3Ti_4O_{12}$ has been recently studied for their important properties and applications in different field. Perovskite type compounds with $CaCu_{3-x-y}Ni_xMg_yTi_4O_{12}$ formula (x=0.1/0.15/0.2 and y=0.1/0.2/0.3) were prepared by a conventional solid state reaction method. The samples were calcined in air at 1000°C and 1100°C for 12h and 24h and then sintered at 1100°C for 12h and 24h. Solid solutions were studied by X-ray diffraction (XRD), dielectric measurement and Raman spectroscopy. The X-ray diffraction revealed that these compounds crystallize in the cubic system with space group Im3 with lattice parameter a=7.4 A° and Z=2. Single-phase powders were characterized by Rietveld refinement. Dielectric properties of the CCNMTO ceramics were particularly studied in wide rang frequency 1KHz - 1MHz at room temperature. The dielectric properties, permittivity and the loss factor of these ceramics were interpreted.

Key words: $CaCu_3Ti_4O_{12}$, Dielectric properties, colossal dielectric, CCNMTO.

SYNTHESIS OF AURONES AND RELATED COMPOUNDS UNDER SOLVENT FREE CONDITIONS

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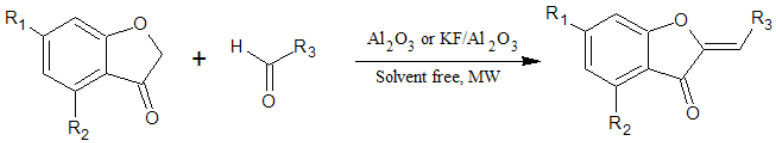
Aurones (2-benzylidenebenzofuran-3(2*H*)-ones) are natural compounds occurring as yellow dyes in plants and constitute a little part of flavonoid[1]. Aurones exhibit a wide variety of biological activities[2] : they must be described as inhibitor of enzymes (acetylcholinesterase, tyrosinase) and also as anticancer, antibacterial, antifungal, and insect antifeedant agents.
The most common synthesis of aurones is the oxidative rearrangement of chalcones in the presence of toxic metal salts[3] like thallium or mercury oxidants. The use of heavy metal salts can be problematic for the preparation of drugs because traces of heavy metals can influence the results of the biological tests.
In order to achieve synthesis of new aurones under green conditions, we have extended the scope of our work on the 3-coumaranone condensation[4] using solvent-free condition on alumina or on alumina-KF, under MW irradiation, according to the Scheme 1. We were interested in the synthesis of novel aurones carrying 1,3-benzodioxol and ferrocenyl groups.



Schema1. Condensation of 3-coumaranones **1-5** with aldehydes **a-b**.

Condensation under solvent-free conditions of 3-coumaranones with aldehydes allowed the synthesis of new potentially biological aurones. All the reactions were stereospecific and the stereochemistry obtained for all compounds was Z, corresponding to the configuration of natural aurones.

Key words: aurones, ferrocene, solventless reaction.

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SYNTHESIS OF NEW CHIRAL BIS-IMIDAZOLIDIN-4-ONES: COMPARISON BETWEEN CLASSIC METHOD AND GREEN CHEMISTRY CONDITIONS

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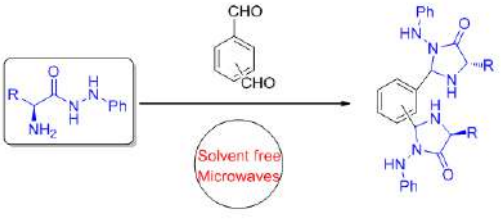
Novel bis-imidazolidin-4-ones were synthesized in moderate to good yields through the cyclocondensation of ortho-, meta- and para-phthalaldehydes with various substituted phenylhydrazides. These nitrogenated cyclic compounds were isolated as a mixture of two diastereoisomers and were prepared in green chemistry conditions using microwave irradiation.



R = Me, *i*-Pr, *i*-Bu, Bn, $H_3CS(CH_2)_2$

Fig. Synthesis of the bis-imidazolidin-4-ones

Key words: bis-imidazolidin-4-one, cyclocondensation, phenylhydrazide.

Highly luminescent lanthanide-based coordination polymers with 5-methoxy-isophthalate as ligand

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Reactions in water between lanthanide chlorides and the di-sodium salt of 5-metoxy-isophtalic acid, Na_2(mip), lead to the first two series of lanthanide-based coordination polymers based on this ligand. The first series has general chemical formula $[Ln(mip)(Hmip)(H_2O)_5{\cdot}H_2O]_\infty$ with Ln = La - Ce. The second family is constituted by compounds with general chemical formula $[Ln_2(mip)_3(H_2O)_8{\cdot}4H_2O]_\infty$ with Ln = Sm - Er plus Y. Luminescent properties of the compounds that belongs to this family have been studied. The Tb-based compound presents one of the brightest luminescence reported to date for a lanthanide-based coordination polymer. The weak intermetallic energy transfer evidenced on the second family of compounds allows easy and predictable color tuning of the hetero-bi-metallic powders.

Figure 1. Extended coordination polyhedrons of Y1 (left) and Y2 (right) in $[Y_2(mip)_3(H_2O)_8{\cdot}4H_2O]_\infty$.

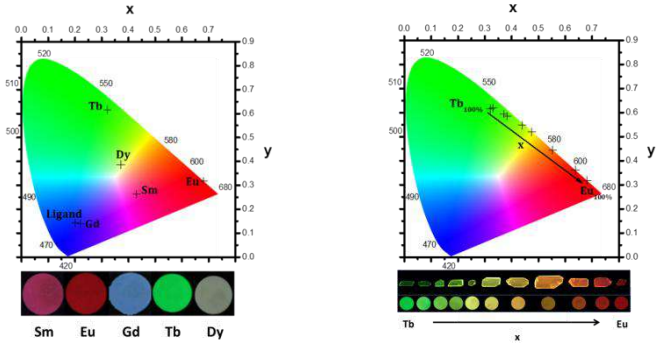

Figure 2. Left: Colorimetric coordinates and pictures under UV irradiation of $[Ln_2(mip)_3(H_2O)_8{\cdot}4H_2O]_\infty$ with Ln = Sm-Dy (λ_{exc} = 312 nm). Right: Colorimetric coordinates and pictures of pellets of microcrystalline powders and of single crystals (middle) under UV irradiation of $[Tb_{2-2x}Eu_{2x}(mip)_3(H_2O)_8{\cdot}4H_2O]_\infty$ with $0 \leq x \leq 1$.

TREATMENT OF AQUEOUS WASTES CONTAMINATED WITH HERBICIDE 2,4-D BY ANODIC OXIDATION ON BORON DOPED DIAMOND

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In this work, the treatment of effluents polluted with pesticide 2,4-D is studied using electrolytic and photoelectric technologies. Results show that photo-electrolysis with Boron Doped Diamond anodes is more efficient than single electrolysis and influence of potential radically oxidant produced on the anode surface from sulphate and chloride and activated by UV light can explain the better results. Both, electrolysis and photo-electrolysis can lead to the complete mineralization of the 2,4-D.

Keywords: 2,4-D, electrolysis, photo-electrolysis.

WASTEWATER TREATMENT BY NOBLE METAL/MESOPOROUS TiO_2 PHOTOCATALYSTS

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Photocatalysis plays an important role in dealing with today's challenging demand for wastewater treatment technologies. Among the photocatalysts, TiO_2 is believed to be the most promising presently known material due to its superior photocatalytic activity, non-toxicity, long-term stability, and low price. However, the lifetime of reactive oxygen species is relatively short under UV irradiation, which often led to a partial mineralization of organic pollutants. Mesoporous TiO_2 has been considered to be a promising photocatalyst owing to its advantages such as high specific surface area and multidimensional framework. The modification of TiO_2 with precious metals can alter the charge-transfer properties between TiO_2 and the surrounding environment, thus improving the performance of TiO_2 nanomaterials-based devices.

In this study, mesoporous TiO_2 prepared by a templating sol-gel method was modified with precious metals such as Ag, Au and Pd. Photocatalytic activity of doped and undoped mesoporous TiO_2 were evaluated under simulated solar light irradiation on real wastewater effluents sampled in the output of wastewater treatment plant located in the North of France. This study clearly demonstrated that the doped TiO_2 photocatalysts present better activity than undoped one.

Key words: Photocatalysis, mesoporous TiO_2, wastewater, precious metals.

CONFORMATIONL STUDY AND HIRSHFELD SURFACE ANALYSIS OF 1-CYCLOHEXYLPIPERAZINE-1,4-DIIUM DICATION IN THE LAYRED ORGANIC DICHROMATE, $(C_{10}H_{22}N_2)[Cr_2O_7]$

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[c)] *University of Carthage, Preparatory Institute for Engineering Studies of Nabeul, 8000 Mrazka, Nabeul, Tunisia.*

Structure of 1-cyclohexylpiperazine-1,4-diium dichromate(VI), $(C_{10}H_{22}N_2)[Cr_2O_7]$ (1), was determined from single-crystal X-ray diffraction data. Compound (1) is triclinic ($P\bar{1}$) with a = 10.351 (2) Å, b = 12.766 (3) Å, c = 6.111 (1) Å, α = 91.50 (2)°, β = 104.26 (3)°, γ = 94.91 (2)°, V = 778.8 (3) Å3, and Z = 2. In the crystal structure 1-cyclohexylpiperazine-1,4-diium (1-CHPP) dications and dichromate dianions are linked together by N–H···O hydrogen bonds to form $\{(C_{10}H_{22}N_2)[Cr_2O_7]\}n$ infinite chains lying parallel to the (100) plane and running along the c axis **[1]**. The intermolecular N–H···O hydrogen bonds link these chains into a two-dimensional network structure consolidated through C–H···O weak interactions. In the organic dication, both piperazinium and cyclohexyl rings possess spatial chair conformations and the orientation of N-cyclohexyl bond is equatorial (equatorial conformer) **(Fig.(a)**). The possible stable conformers of 1-CHPP dication were also studied theoretically by B3LYP hybrid density functional theory method together with 6-31G(d) basis set. Comparison between the crystallographic and theoretical results indicates that the equatorial conformer is the most stable form of 1-CHPP dication. The Hirshfeld surface (HS) analysis and the two-dimensional fingerprint (FP) maps reveal that the packing of (1) is dominated by H…O/O…H (51.6%) and H…H (48.4%) contacts **(Fig.(b)**).

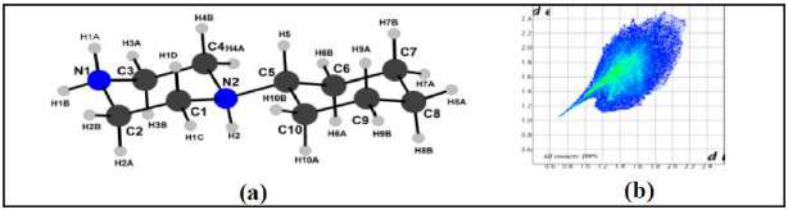

Fig. (a) Equatorial conformer of 1-CHPP dication, and (b) FP map of (1).

Key words: Dichromate, X-ray diffraction, Crystal structure, Hirshfeld surface, B3LYP

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SYNTHESIS, SPECTROSCOPIC CHARACTERIZATION, CRYSTAL STRUCTURE AND HIRSHFELD SURFACE ANALYSIS OF METHYL 1-(4-FLUOROBENZYL)-4-METHOXY-5-OXOPYRROLIDINE-3-CARBOXYLATE

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Synthesis of methyl 1-(4-fluorobenzyl)-4-methoxy-5-oxopyrrolidine-3-carboxylate (I) was achieved through a conjugate addition-lactamization sequence. Its single crystal was obtained by slow evaporation at room temperature. Preliminary analysis was done spectroscopically by means of NMR (^{1}H, ^{13}C), EI, CI, HRMS and FT-IR. Further the structure was confirmed by X-ray crystal structure analysis. Compound (I) crystallizes in monoclinic *P21/n* space group with the unit cell parameters *a*=10.188 (8) Å, *b*=7.741 (7) Å, *c*=17.239 (6) Å, *β*=94.50 (3)°, *V*=1355.6 (4) Å3 and *Z*=4. In the molecular structure of (I) the pyrrolidine ring adopts a conformation intermediate between envelope and half-chair and its mean plane makes an angle of 75.37 (2)° with the phenyl ring. In the crystal, molecules are arranged into centrosymmetric dimers via pairs of C10-H10…O1(*1-x, -y, 1-z*) hydrogen bonds to form infinite chains lying parallel to the [010] direction and running along the *a* axis. These chains are connected into a three-dimensional architecture by a network of C-H…O, C-H…π and π-π interactions. An intramolecular C7-H7A…O1 hydrogen bond is observed, generating an *S(5)* ring motif **[1](Fig.(a))**.The prevalence of C-H···O, C-H··· π and π-π interactions is confirmed by an analysis of the Hirshfeld surface (HS) **(Fig.(b))** and fingerprint plots (FP).

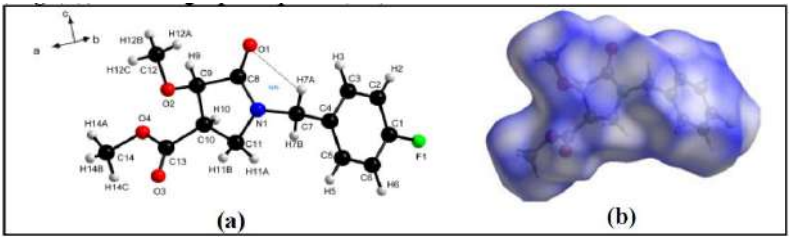

Fig. (a) Molecular structure of (I) showing the atom labeling scheme, and (b) Hirshfeld surface of (I) mapped with *dnorm*.

Key words: Oxopyrrolidine, X-ray analysis, Crystal structure, Hirshfeld surface.

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Design and synthesis of novel antimicrobial pyrimidinone hydrazones

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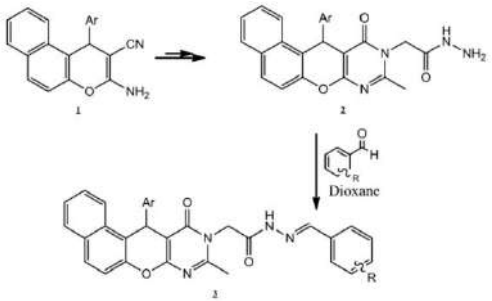
Hydrazone derivatives are found to be an important class of compounds in organic and medicinal chemistry. They draw considerable attention, due to their extensive applications in pharmaceutical and biological activities such as anticancer, anti-inflammatory, antibacterial, antifungal, anti HIV, antimalarial, antitubercular and potential inhibitor for various enzymes.[1] On other hand, pyrimidinones were found to exhibit a wide spectrum of biological and pharmacological activities, they are considered as good antimicrobial agents.[2] These observations prompted us to join these two skeletons (pyrimidinone and hydrazone) in the same molecule in order to access to new hybrid molecules with potent biological activities.[3] In this context, the key intermediates 2-amino-3-cyanonaphtopyrane **1** were used as starting material to access to the pyrimidinone hydrazides **2** which in turn were condensed with a series of arylaldehydes to afford the corresponding hybrid molecules 2-pyrimidinones hydrazones **3**. On the other hand, the antimicrobial activity towards some Gram+ and Gram- bacteria of all synthesized derivatives was evaluated and discussed.



Ar : H, CH_3, OCH_3
R: H, 4-CH_3, 4-OCH_3, 4-Cl, 3,4-$(OCH_3)_2$, 3-C_2HF_4O

Keywords: naphtopyrane, hydrazone, pyrimidinone, 2-amino-3-cyanonaphtopyrane

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Optical and vibrational study of $Na_3La_{(1-x)}Er_{(x)}(PO_4)_2$ (x= 10%, 20% and 50%) monophosphate

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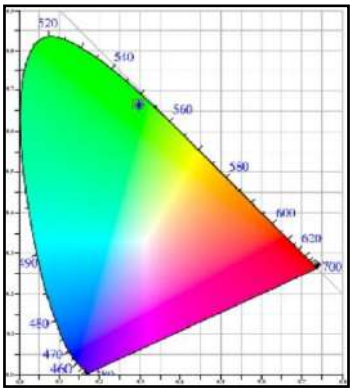
Powders of monophosphate $Na_3La_{(1-x)}Er_{(x)}(PO_4)_2$ were synthesized by a conventional solid state reaction. These powders were identified by X-ray diffraction (XRD) powders, IR spectroscopy, RAMAN spectroscopy and photoluminescence. In fact, a comparison with the literature, the compound revealed that is isotypic with a $Na_3Ce(PO_4)_2$ (PDF no. 088-2378) [1]. Indeed, it crystallizes in the orthorhombic system with space group $Pca2_1$. Absorption spectra were performed in the UV-Visible and IR range. The values of the energy gap for this monophosphate were determined. The spectroscopic properties of the Er^{3+} ion in $Na_3La_{(1-x)}Er_{(x)}(PO_4)_2$ were performed in the UV-visible and IR range at room temperature. All obtained bands are characteristic to the erbium ion. The decay time profiles of green emission ($^4S_{3/2}$ level) under λ_{ex} = 488 nm were investigated. The evolution of the chromaticity coordinates of the green emission of the Er^{3+} ion in these compounds is shown in the chromaticity diagram. We found that the value of the quantum yield is high (> 90%). It is suggested that this compound may have optical applications.



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APPLICATION OF DIELS-ALDER REACTION TO LIQUID- LIQUID INTERFACE

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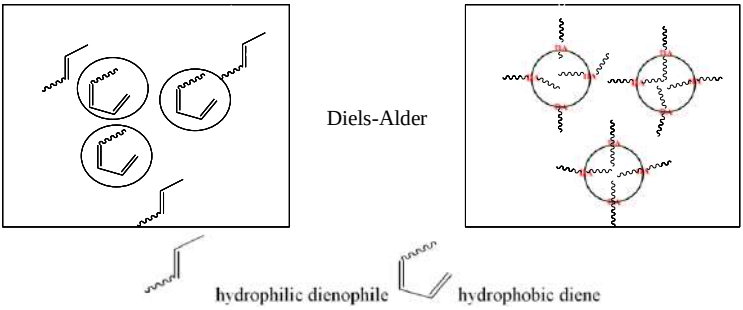
The aim of this work is the realization of the Diels-Alder reaction at the interface of an oil / water system and its application to micro encapsulation in developing heat-sensitive membrane microcapsules.



Polycondensation Diels Alder oil / water interface

The Diels-Alder synthesis occurs in three steps :

- The synthesis of hydrophilic dienophile using polyethylene glycol with different molecular weights according to alcohol - maleic anhydride reaction.
- The synthesis of hydrophobic diene from either hydrophobic oligomers grafted to a pyrrole molecule, or by modifying octanoic acid with furan derivatives.
- The Diels-Alder reaction occurs between a diene and a dienophile hydrophobic.

Several syntheses of adducts were developed without solvent according to Diels-Alder reaction between diene and dienophiles in type emulsions: water in oil or oil in water.

The products obtained were characterized with different techniques such as chromatography TLC thin layer, FTIR infrared spectroscopy, thermal analysis by differential scanning calorimetry DSC and optical microscopy with image acquisition system.

Keywords: Diels-Alder reaction; diene; dienophile; pyrrole; furan; polyethylene glycol

Microwave assisted synthesis and biological activity of new pyrrole and pyrazole derivatives

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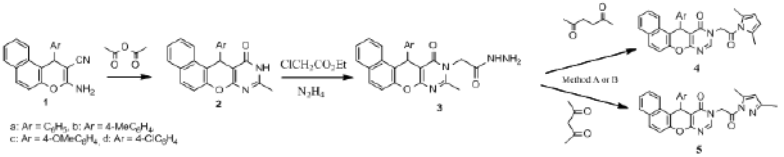
This study deals with the development of new free catalyst-solvent synthesis of new pyrrole and pyrazole derivatives along with benzochromene and pyrimidinone rings under microwave irradiation and classical conditions, keeping in view the high spectrum of biological activities of pyrrole and pyrazole compounds such as anticandidal,[1] antifungal[2] and antiviral activities.[3]

The treatment of **1** with acetic anhydride containing catalytic drops of orthophosphoric acid furnished the isolable products **2**. The obtained chromenoyrimidinones **2** were *N*-alkylated by ethylchloroactetate to give the corresponding esters, which were subsequently treated with hydrazine hydrate to give hydrazides **3**. Then, we have performed the cyclocondensation of the pyrimidine precursors **3** with some diones under both MW irradiation (method A) and classical heating (method B) conditions to give our new pyrrole **4** and pyrazole **5** derivatives. Structures of all the prepared compounds were elucidated on the basis of extensive spectroscopic methods including (^{1}H, ^{13}C) NMR and ES-HRMS. They were also screened for their antimicrobial and α-glycosidase activities.



Scheme 1. Synthesis of benzochromenopyrimidinone, hydrazides, pyrrole and pyrazole derivatives

Keywords: Pyrroles, Pyrazoles, Microwave irradiation, Free catalyst-solvent, Biological activities.

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SYNTHESIS, CRYSTAL STRUCTURE AND ELECTRICAL PROPERTIES OF $K_{0.12}Na_{0.54}Ag_{0.34}Nb_4AsO_{13}$

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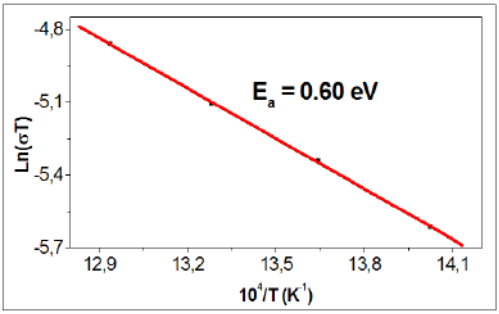
In recent years, inorganic metal arsenates possessing open-framework structures have been part of intensive research activities due to their structural diversity and their potential applications in the fields of ionic conductivity. The architecture and the size of the tunnels have a great influence on the cations motion in the crystal structure. Therefore, structural studies of new materials are the key to understand and predict their ionic transport properties. In this context, we explored the A_2O–Nb_2O_5–As_2O_5 (A = alkali metal or/and Ag) system. A new compound $K_{0.12}Na_{0.54}Ag_{0.34}Nb_4AsO_{13}$ was synthesized via the solid-state reaction. The structure can be described as infinite chains arranged along [100] and connected via $Nb(2)O_6$ octahedra to form a three-dimensional framework. This architecture delimits large cavities (hexagonal section) where the potassium, silver and sodium ions are included. The electrical characterization of the title compound was performed using AC impedance spectroscopy. Ionic conductivity study versus temperature is undertaken and the activation energy deduced from the slope is 0.60 eV.



Arrhenius plot of conductivity of $K_{0.12}Na_{0.54}Ag_{0.34}Nb_4AsO_{13}$ sample.

Structural analysis of pectic polysaccharides of myrtle fruits (*Myrtus communis* L.)

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The fruits of myrtle (*Myrtus communis* L.), an edible species used for food, industrial and medical were harvested in the Annaba region (Northeast, Algeria). After treatment 85% ethanol (v / v). The solid was removed by centrifugation at 9000 rpm for 30 min, using a solid / liquid ratio 1:40 (w / v) at 80 ° C for 24 h. Pectic polysaccharides were solubilized from the skin of fruit myrtus by sequential extraction with water at 60 °C (WSP) and the EDTA solution at 60 °C (CSP).
Analysis of monosaccharide composition by exchange chromatography high performance anion pulsed amperometric detection (HPAEC-PAD) showed the presence of galacturonic acid as the major compound.
The results of 1 H NMR spectroscopy showed the presence of esterified galacturonic acids.

Key words: Myrtle; *Myrtus communis*; Analysis: HPAEC-PAD; 1 H NMR spectroscopy.

Biotransformation and Cytotoxic Activity of Natural Oleanolic acid form *Olea europaea* L.

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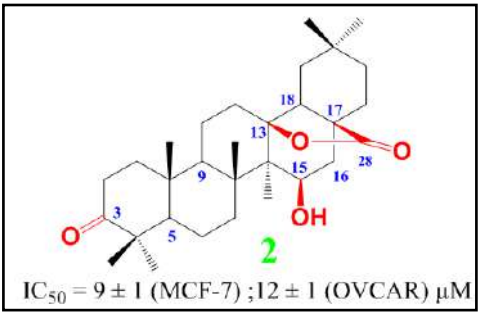
Microbial transformation of triterpenoids has provided new derivatives that are potentially useful for pharmacological studies [1]. In these biotransformation processes, several reactions that are difficult to achieve by chemical means have been accomplished, such as introduction of hydroxyl groups into remote positions of the molecules, selective ring cleavage through a Baeyer-Villiger-type oxidation and carbon skeleton rearrangements involving a methyl group migration [2]. In this work, the biotransformation of oleanolic acid **1** by *Saccharomyces cerevisiae* produced five metabolites **2**–**6** which were separated by column chromatography. Their structures were elucidated by extensive analyses of their spectroscopic data, and also by chemical correlations. The cytotoxic activity of all compounds against the human breast adenocarcinoma cell line MCF-7 and ovary cell line OVCAR was assessed using MTT assay. Significant results were noticed and discussed. Among all triterpenic derivatives, the metabolite **2** was found to have the highest cytotoxic effect against the human breast and ovary cells lines with IC_{50} values of 9 ± 1 and 12 ± 1 µM, respectively.



Keywords: *Olea europaea* L., Oleanolic acid, Biotransformation, Cytotoxic activity

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SYNTHESIS AND CHARACTERIZATION OF Al-DOPED ZINC OXIDE NANORODS WITH HIGH ADSORPTION PROPERTIES

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Doped and undoped zinc oxide (ZnO) nanoparticles can exhibit a wide range of morphologies and possess unique optical properties. Hence, ZnO has been investigated for a wide variety of applications ranging from optolectronic to photocatalysis [1, 2]. In this work, Al-doped zinc oxide nanorods were successfully synthesized by a solvothermal method using ethanol as solvent and the doping percentage in Al varied from 1 to 20%. Zinc acetate, sodium hydroxide and aluminum acetylacetonate were used as precursors.
The synthesized ZnO:Al samples were characterized using X-ray diffraction (XRD), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS) and BET measurements. XRD confirms that the obtained crystals have the wurtzite ZnO phase. TEM images showed nanorods with an average length that decreases when increasing the Al content (127 nm for undoped ZnO nanorods to 12 nm for ZnO:Al (20%) ones). Meanwhile, the BET specific surface area increased from 21 to 82 m^2/g when increasing the Al doping from 0 to 20%. The ZnO:Al (20%) nanorods were found to exhibit the highest adsorption capabilities towards some negatively charged dyes like Orange II, Rocelline or Alizarin (Fig. 1).

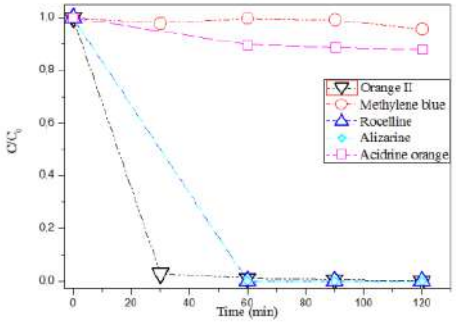

Fig. 1. C/C_0 evolution over time when varying the dye in contact with ZnO:Al (20%) rods.

Keywords: Solvothermal synthesis, Al-doped ZnO, Nanomaterials, Adsorption.

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Dielectric properties of a new proton conducting solid with a superprotonic phase transition

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Thermal behavior and electrical proprieties are given for a new compound with a superprotonic phase transition $Cs_{0.2}Rb_{0.8}H_2AsO_4$ (CRDA). The title compound is isostructural with the tetragonal phases of CsH_2AsO_4 (CDA) and RbH_2AsO_4 (RDA). A calorimetric study of the title compound shows two peaks, which are detected at 185 and 433 K. The first transition (185 K) is attributed to a ferroelectric–paraelectric type. A high temperature phase transition (433 K) leading to a superionic–protonic phase was found, characterised by an unusual high conductivity. Moreover, protonic conduction of the mixed compound determined by impedance and modulus spectroscopy has been studied in the temperature range 180–473K.

TREATMENT OF BIRNESSITE SURFACE BY HUMID AIR PLASMA: APPLICATION TO THE DEGRADATION OF COCHINEAL RED

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[b)] *Institut National des Sciences Appliquées et de Technologie (INSAT)*

The thin layers of birnessite ($Mn_7O_{13}.5H_2O$) are exposed to reactive species gliding arc plasma in humid air, which induces the treatment of the thin layers surface. Plasma treatment thin layer of birnessite was used for the degradation of Cochineal Red. The experimental results showed that 95% of the CR solution was completely decolorized by thin layer of birnessite treated by plasma compared to 80% of the same solution after interaction of thin layer of birnessite untreated. The decay kinetics always follows a pseudo-first order reaction. The application of the humid air plasma for the surface treatment of thin layers of birnessite improves the efficiency of treatment for Cochineal Red degradation.

Key words: Humid Air Plasma, Surface Treatment, Thin Layers of Birnessite, Cochineal Red, Degradation.

SYNTHESYS OF DIBENZYLIDENECYCLOHEXANONE BY MIXT ALDOLIC CONDENSATION

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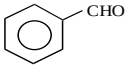
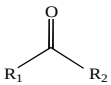
This work was realised for the object of organic synthesis, particulary the synthesis of α, β-unsaturated ketones, which are biologically active and shows a great pharmaceutical interest and find numerous applications in the treatment of several diseases such as cancer[1-3], paludism [4], rheumatism [5,6], and diabete [7].

Carbonyl compound α, β-unsaturated was obtained by an aldolic condensation, via Claisen-Schmidt reaction, in which the benzaldehyde (1) was used to react with enolisable Ketone "cyclohexanone" (2) under basic condition.



(1)



(2)

R_1 et R_2 = -(-CH_2-)$_5$-

Reagents of mixt aldolic condensation

The dibenzylidencyclohexanone was prepared by the method described above.

The reactivity of carbonyl and double band allow us to carryon addition reactions of bromine and 2, 4-dinitrophénylhydrazine, to obtain the corresponding addition products.

The identification of synthesised compound was accomplished by usual spectroscopic methods, such as, infrared (IR), UV-Visible, Nuclear Magnetic Resonance (^{1}H NMR), melting point and chemical tests.

Key words: Benzaldehyde, cyclohexanone, mixt aldolic condensation, Claisen-Schmidt, spectral methods.

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Synthesis of novel triazoles1,2,3- derivatives through [3+2] cycloaddition

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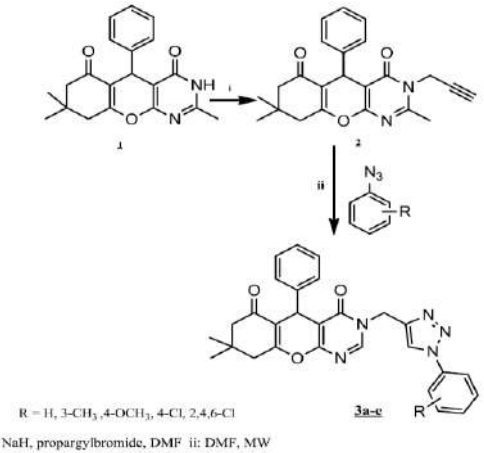
1,2,3-Triazoles have occupied an important role not only in organic chemistry but also in medicinal chemistry and are one of the key structural units found in a large variety of bioactive molecules[1] as antifungal, anti-allergic, anti-tubercular and anti-inflammatory agents. Thus, several methodologies exist toward the synthesis of triazoles and most of them endeavor azides as a key step. So far, the most versatile method to generate substituted 1,2,3-triazoles has been the Huisgen [3+2] cycloaddition between a terminal alkyne and an azide[2]. As part of our continued interest in the development of efficient methods for the synthesis of heterocyclic compounds of biological importance, we report herein a facile and efficient synthesis of novel series of 1,2,3-triazoles of 2,8,8-trimethyl-5-phenyl-3-(prop-2-yn-1-yl)-8,9-dihydro-3*H*-chromeno[2,3-d]pyrimidine-4,6(5*H*, 7*H*)-dione **3a-e** in good yields by a 1,3-dipolar cycloaddition reaction.



Keywords: 1,2,3-triazoles, pyrimidinone, 1,3-dipolar cycloaddition

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CRYSTAL STRUCTURE, THERMAL BEHAVIOR AND MAGNETIC PROPERTIES OF THE ORGANIC-INORGANIC MONONUCLEAR CO(II) COMPLEX WITH 2-AMINO-6-METHYLPYRIDINE LIGAND, $(C_6H_9N_2)_2[CoBr_4]$.

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Single crystals of the new organic-inorganic compound $(C_6H_9N_2)_2[CoBr_4]$ were grown by the slow evaporation method and characterized by single crystal X-ray diffraction, thermal analysis (ATG-TD) and temperature dependent magnetic susceptibility. The title compound belongs to the orthorhombic space group Pbcn. The inorganic layers consist of $[MBr_4]^{2-}$ anions linked together via Br···Br interactions. The organic 2-amino-6-methylpyridine cations are connected to the inorganic sheets by means of N-H···Br and C-H···Br hydrogen bonds. The thermal decomposition curves reveal a two steps weight loss giving finally metal oxide. The title compound exhibits a paramagnetic behavior with weak antiferromagnetic interactions at low temperature.

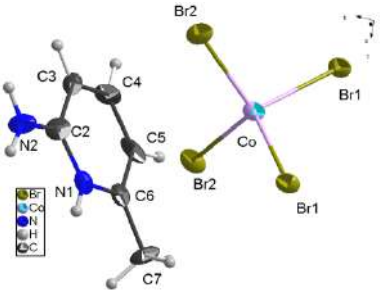

Fig: A view of the asymmetric unit in the crystal structure of $(C_6H_9N_2)_2[CoBr_4]$ salt, showing the atom-numbering scheme.

Key words: Metal halides, crystal structure, intermolecular interactions, thermal behavior, Magnetic properties.

Synthesis and Biological Evaluation of Benzochromenopyrimidinones as Cholinesterase for Alzheimer's Disease Therapy

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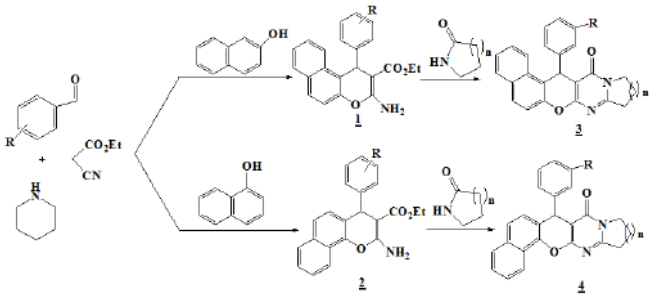
Alzheimer's disease (AD) has emerged as the main cause of memory and cognitive deficiency in aged persons. The Alzheimer's disease International (ADI) Association report from 2015 estimates that over 46 million people are currently affected by dementia. Given the epidemic expansion of AD, this number is expected to drastically increase in the future, reaching 131.5 million cases by 2050. Many efforts have been devoted to comprehend the complex etiology of AD, yet certain aspects of the pathogenesis are still not well understood. Nevertheless, several histopathologic been clearly identified in AD patients such as intracellular neurofibrillary tangles, composed of hyperphosphorylated tau protein and toxic amyloid plaques of aggregated β-amyloid (Aβ) peptide. In the present work we report the straightforward two-step synthesis and biological assessment of novel racemic benzochromenopyrimidinones as acetylcholinesterase inhibitors properties (Scheme1).



Scheme 1.

The biological evaluation of the compounds **3** and **4** identified compound -**3Bb**- (R = 3-OCH3, n=2) displayed a mixed-type inhibition of human acetylcholinesterase (IC_{50} = 1.28±0.03 μM).

Keywords: Alzheimer's disease; amyloid plaques; benzochromenopyrimidinones; cholinesterase inhibitors.

ELECTROPHILICITIES OF β-(4-X-PHENYL)-α-(CYANO) ETHYL ACRYLATE

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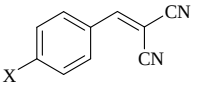
The kinetics of numerous chemical reactions can be described by the Mayr's equation (1), were k, E and N/s correspond to the second-order rate constant, the strength of the electrophile and the nucleophile-specific, respectively.

$$\log k\ (20\ °C) = s\ (N + E) \qquad (1)$$

In a recent work, we have shown that the logarithms of the second-order rate constants ($\log k_{calcd}$) calculated from eq. (1) for reactions of the p-X-substituted benzylidenemalononitrile with hydroxide ions in 50% H_2O-50% CH_3CN at 20 °C are linearly related to the logarithms of the experimental rate constants ($\log k_{exp}$). This behavior is described by the linear relationship (2).

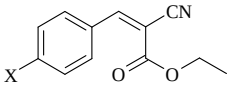
$$\log k_{exp} = 1{,}22 + \log k_{calcd} \qquad (2)$$

In this communication, we discuss how equation (2) can be employed to estimate the parameters of electrophilicities E of β-(4-X-Phenyl)-α-(Cyano) Ethyl Acrylate **2a-h** in aqueous solutions at 20 °C.



1a-h

1a: X = NMe_2 1e: X = Cl
1b: X = OMe 1f: X = CF_3
1c: X = Me 1g: X = CN
1d: X = H 1h: X = NO_2



2a-h

2a: X = NMe_2 2e: X = Cl
2b: X = OMe 2f: X = CF_3
2c: X = Me 2g: X = CN
2d: X = H 2h: X = NO_2

Key words: Substituent effects, Mayr's equation,Benzylidenemalononitrile, Alkaline Hydrolysis, Electrophilicity, Kinetics.

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STRUCTURAL CHARACTERIZATION, VIBRATIONAL PROPERTIES, HIRSHFELD SURFACE ANALYSIS AND MAGNETIC MEASUREMENTS OF A NEW HYBRID MATERIAL, TRICHLORIDO(1-ETHYLPIPERAZIN-1-IUM)COBALT(II)

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A new organic-inorganic hybrid material of formula $(C_6H_{15}N_2)CoCl_3$ was elaborated and characterized by X-Ray diffraction analysis. It crystallizes in the monoclinic space group P21/n with the following unit cell parameters a= 7.421 (3) Å, b= 18.160 (7) Å, c= 8.691 (4) Å, Z=4 and V= 1171.1 (8) $Å^3$. The title inorganic-organic hybrid compound is presented as a neutral zwitterionic species. The negative charge of the -CoCl3 group is balanced by the positive charge at the ethylated N2 nitrogen atom of the 1-ethylpiperazin-1-ium ring (Fig 1). The examination of the structure shows that the bi-dimensional frameworks are produced by N-H···Cl hydrogen bonding and Van der Waals interactions. Investigation of intermolecular interactions and crystal packing via Hirshfeld surface analysis reveals that H···Cl and H···H intermolecular interactions are the most abundant contacts in the crystal packing. IR, Raman and UV-Visible spectroscopies were also used to characterize this compound. Solution ^{13}C NMR spectroscopy results are in agreement with the X-ray structure. The thermal properties of the Trichlorido(1-Ethylpiperazin-1-ium)Cobalt(II) has been studied by differential scanning calorimetry (DSC). Moreover, magnetic susceptibility measurements indicate that the compound exhibits weak antiferromagnetic coupling interaction between the cobalt (II) centers. The ferromagnetic ordering is further confirmed by the magnetic field dependence of the magnetization between 2 and 30K (Fig 2).

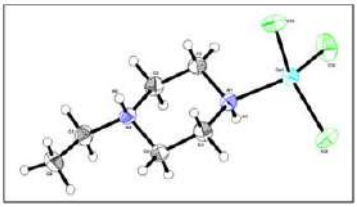

Fig 1. ORTEP view of $(C_6H_{15}N_2)CoCl_3$

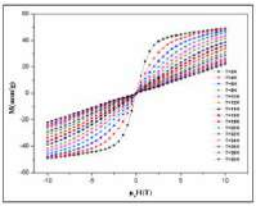

Fig 2. Magnetic isotherms of the $(C_6H_{15}N_2)CoCl_3$

Key words: Crystal structure, Hirshfeld analysis, IR-Raman Spectroscopy, NMR spectroscopy, Magnetic properties

Chemiclal and electrochemical copolymerization of aniline with piperazine on platinum electrodes: Characterization, and Application to the Detection of Dopamine and Ascorbic Acid

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Polyaniline (PANI) ranks among important conducting polymers with highly promising properties for different applications, However, it has been observed that some of its properties still need further improvement, it is electroactive only at low pH and it is insoluble in common solvents in its conductor state [1]. Several authors [2] have studied the synthesis of copolymers of aniline with other molecules such for example aminobenzenesulfonic acids [2]. In this work, the electrochemical polymerization of aniline in presence of piperazine was successfully synthesized from monomers in acidic medium. Several spectroscopic characterization techniques were employed to gain information on the chemical structure and redox behavior of the obtained material. Cyclic voltammetry, XPS and in situ FTIR spectroscopy were combined to study the composition of the copolymer and redox transitions mechanism. The poly(aniline-co-piperazine) was developed and applied for the determination of ascorbic acid and dopamine in perchloric acid solution.

Acknowledgements
Financial support from the Spanish Ministerio de Economía y Competitividad and FEDER funds (MAT2013-42007-P) and from the Generalitat Valenciana (PROMETEO2013/038) is gratefully acknowledged. S. Dkhili thanks the Ministry of Higher Education and Scientific Research of Tunisia for funding her stay at the University of Alicante.

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SYNTHESIS,CRYSTAL STRUCTURE AND CHARACTERIZATION OF A NEW CHROMIUM COMPLEX $(C_8H_9N_2)_3[Cr(C_2O_4)_3].3H_2O$

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The search of Tri(oxalato)chromium(III) compounds obtained through an impressive synthetic strategy based on supramolecular chemistry has attracted intense interest not only because of their appealing structural novelty, but also due to their fascinating properties and applications, such as catalysts, luminescence, NLO and magnetic [1]. Following these lines of investigation, we have focused our work on the synthesis and the characterization of a new compound of formula $(C_8H_9N_2)_3[Cr(C_2O_4)_3].3(H_2O)$.
This compound crystallizes in the monoclinic $P2_1/c$ space group with a = 13.401(2)Å, b = 14.978(4)Å, c = 16.934(2)Å, β = 91.23(2)° and Z= 4.
The structure is made up of $[Cr(C_2O_4)_3]^{3-}$ anion, three $(C_8H_9N_2)^+$ cations and three crystallization water molecules, which are held together by hydrogen bonds and π-π stacking interactions. The structure of the title compound consists of layers formed by $[Cr(C_2O_4)_3]^{3-}$ and free water molecules intercalated by organic cations $(C_8H_9N_2)^+$ (Fig. 1). The crystal packing is stabilized by N-H...O and O-H...O hydrogen bonds generating motifs such as $R^3_4(10)$.

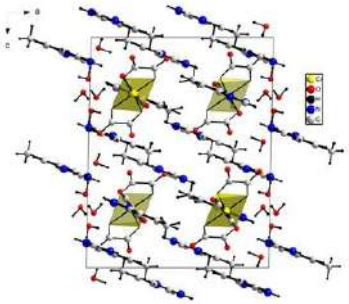

Fig. 1 Projection of the $(C_8H_9N_2)_3[Cr(C_2O_4)_3].3H_2O$ structure along the *b* axis

Keywords: Chromium(III) complexes, Crystal structure, X-Ray Powder diffraction, Infrared spectroscopy.

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Crystal Structure, Bond-Valence-Sum and Charge-Distribution validations of β-$Na_4Cu(MoO_4)_3$-Alluaudite type

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Cristallochimie et Thermodynamique appliquée.

Single crystals of the new variety of tetrasodium copper (II) tris[molybdate (IV)], have been synthesized by solid-state reactions and characterized by single-crystal X-ray. This alluaudite structure is characterized by the presence of infinite layer $(Cu/Na1)_2Mo_3O_{12}$ type parallel to the (100) which are linked by MoO_4 tetrahedra, forming a three-dimensional framework containing two types of hexagonal tunnels in which Na^+ cations reside. Cu1 and Na1 atoms are located at the same general site with occupancies of 0.5. Na2 and Na4 atoms (4e) sits on a twofold axis, Na1 is located in the center of inversion (4a) and all other atoms are at general positions. The title compound is isotypic with $Na_5Sc(MoO_4)_4$ and $Na_3In_2As_3O_{12}$. The most likely conduction paths of monovalent alkali cations was studied using the bond-valence-sum (BVS) model. The structure model is supported by bond-valence-sum (BVS) and charge-distribution CHARDI methods [1-2]. β-$Na_4Cu(MoO_4)_3$ is compared and discussed with $K_4Cu(MoO_4)_3$ [3] and α-$Na_4Cu(MoO_4)_3$ [4] structures.

Key words: BVS, CHARDI, Alluaudite-type, sodium, copper (II), molybdate(VI).

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HYDROTHERMAL SYNTHESIS AND STRUCTURAL CHARACTERISATION OF TWO NEW DMAP OCTAMOLYBDATES: $(C_7H_{11}N_2)_2[MO_4O_{13}]$ AND $(C_7H_{11}N_2)_2[MO_4O_{13}]. H_2O$

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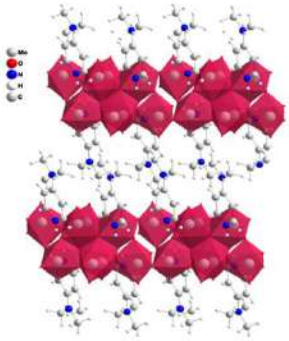
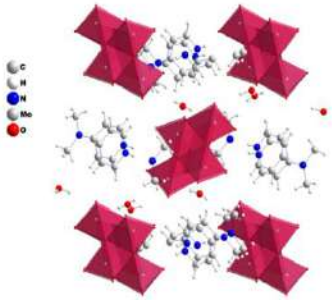
Two new compounds $(HDMAP)_2[Mo_4O_{13}]$ (**1**) and $(HDMAP)_2[Mo_4O_{13}]. H_2O$ (**2**) have been hydrothermally synthesized. Compound (**1**) crystallized in the triclinic system, space group
P-1 with a = 8.204(6) Å; b = 12.179(5) Å; c = 12.966(9) Å; α = 115.15(7)°; β = 96.29(5)°;
γ = 91.28(8)° and Z = 2.
Compound (**2**) Crystallized in the monoclinic system, space group $P2_1/C$ with a = 12.593(9) Å; b = 13.955(5) Å; c = 15.458(6) Å and β = 111.64(5)° and Z = 4. The structures of both compounds were determined by single crystal X-ray methods.
The crystal structure of (1) consists of one dimensional chains constructed from $[Mo_8O_{26}O_{4/2}]_n^{4n-}$ monomers and mono-protoned organic cations $(C_7H_{11}N_2)$ dimethyl-aminopyridinium (HDMAP). The crystal structure of (2) consists of the well known β-octamolybdate anions who are separated by HDMAP cations and occluded water molecules from witch an extensive hydrogen-bonding network is created. The relationship between the two structures is discussed.



Projection according to the b direction of $(HDMAP)_2[Mo_4O_{13}]$ structure (**1**)



Projection of the structure: $(HDMAP)_2[Mo_4O_{13}]. H_2O$ (**2**)

A Hirshfeld Surface Analysis, Crystal Structure, Physicochemical studies and magnetic properties of a new organic cuprate(II) complex: $(C_6H_{18}N_3)_4[CuCl_5]_2[CuCl_4]_31.42(H_2O)$

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$(C_6H_{18}N_3)_4[CuCl_5]_2[CuCl_4]_31.42(H_2O)$ has been prepared and characterized by various physicochemical techniques. Single crystal X-ray diffraction structural analysis has revealed that the title compound belongs to the orthorhombic system with space group *Cmca*. Its unit cell dimensions are: a = 24.286 (2) Å, b = 14.3082 (14) Å, c = 16.6160 (16) Å, with Z = 4, V = 5773.8 (10) Å^3 and its crystal structure was determined and refined down to R = 0.024 and wR (F^2) = 0.059. The structure of the title compound contains three crystallographically independent Cu(II) ions coordinated to chlorine anions in various fashions. Cu(1) is five-coordinated, in a distorted square pyramidal fashion, while Cu(2) and Cu(3) are tetracoordinated in square planar and distorted tetrahedral fashions, respectively. The entities are interconnected by means of hydrogen bonding [O(W)-H···Cl, N-H···Cl, C-H···Cl and C-H···O(W)] forming a three-dimensional network. Intermolecular interactions were investigated by Hirshfeld surfaces. The vibrational absorption bands were identified by infrared spectroscopy. The magnetic susceptibility of Cu(II) complex was measured in the range of 2.00 - 299 K. The complex shows the paramagnetic nature. The effective magnetic moment values change from 2.64 BM (at 2 K) to 3.88 BM (at 299 K). The decrease of magnetic moment values is characteristic for weak antiferromagnetic interactions between the nuclear units within the crystal lattice.

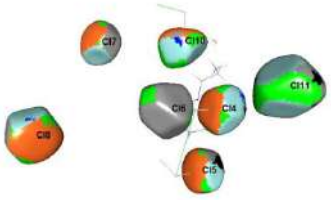

Fig. 1 View of the Hirshfeld surface around the seven independent chloride atoms.

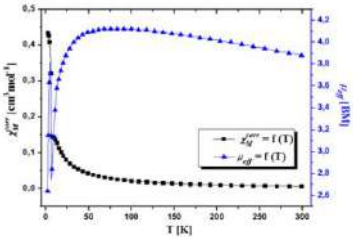

Fig. 2 Dependences of χ_M^{corr} and μ_{eff} vs. T.

Key words: Cu(II) complex; X-ray diffraction; Coordination compound, Hirshfeld surface, Magnetic properties.

New ante and post functionalization strategies for hydrophobic surfaces elaboration

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Highly hydrophobic surfaces present a wide range of interest for both industrial and scientific communities. These applications can be found in various fields, including water proof textiles, self-cleaning windows, anti-fogging or anti-icing glass, biomedical applications (anti-bioadhesion surfaces) or in the environmental domain to develop oil/water separation membranes[1,2]. Due to this wide field of applications, obtaining these properties became a challenge for researchers.

Examples from the nature (Lotus, Tarot, Echeveria) reveal two particularly important parameters: the surface roughness and the surface energy. Various methods can be found to control these parameters[3]. In this work we focus on the use of electrodeposition to get highly structured surfaces. The surface energy can be tuned by functionalization.

Which can be performed before or after the polymerization. The present work report for the first time, the use of a Staudinger reaction variation to reach hydrophobic feature. This reaction can be used for both Ante and Post functionalization strategies. The surfaces obtained by both strategies were prepared and characterized for their wettability, roughness and morphology. The results allow to compare the ante and post functionalization by their positive and negative points to develop hydrophobic surfaces.

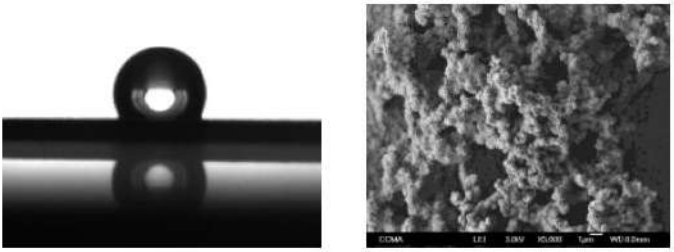

Fig. 1: The morphology of the hydrophobic surface obtained

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Synthesis, crystal structure and physico-chemical characterization of m-Xylylenediaminium diaquabis [dihydrogendiphosphato (2)] cobaltate(II) dihydrate

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Among a variety of organic inorganic hybrid materials, diphosphate compounds containing transition metals showed
promising properties in diverse areas such as catalysis [1], magnetism [2], conductivity, ion-exchange or second-order non-linear optics [3-4]. Here, we report a new diphosphate of mixed organic-metal cations: $(C_8H_{14}N_2)[Co(H_2P_2O_7)_2(H_2O)_2]\cdot 2(H_2O)$ (I). the CoII ion lies on an inversion center and is coordinated by two bidentate diphosphate ligands and two water molecules in a slightly distorted octahedral coordination geometry. The mxylylenediaminium cation is located on a twofold rotation axis. In the crystal, a three-dimensional supramolecular assembly is constructed by O-H…O and N-H…O hydrogen bonds between the organic cations, complex anions and uncoordinated water molecules.

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AN EFFICIENT SYNTHESIS AND CHARACTERIZATION OF α-PHOSPHONATES COMPOUNDS

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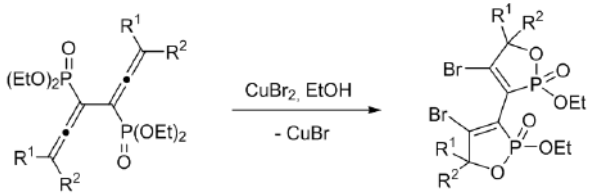
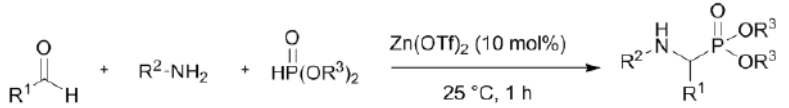
The chemistry of organophosphorus compounds has attracted a great deal of attention because they constitute an important group of organic reagents and useful synthetic intermediates with a wide range of biological activities [1]. In light of these successes, we report in the present work, the synthesis of novel phosphorus bis-allene derivatives from the reaction of diyne diols with diethyl chlorophosphite. The cyclisation of the obtained bis-allenylphosphonates, performed in the presence of copper (II) bromide, gave bis-oxaphospholenes in satisfactory yields.



In continuation of our studies on the preparation and potential synthetic applications of multifunctional phosphonates [2], we report in second part, a simple and green methodology for the synthesis of α-aminophosphonates, in good yields, through the zinc triflate-catalyzed one-pot multi-component reaction of aldehydes, amines and dialkylphosphites, at room temperature, under solvent-free conditions. It is important to mention here that although several metal triflates.



Key words: Bis-oxaphospholenes, bis-allenylphosphonates, α-aminophosphonates.

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Efficient and Green one-pot multi-component synthesis of α-aminophosphonates catalyzed by zinc triflate

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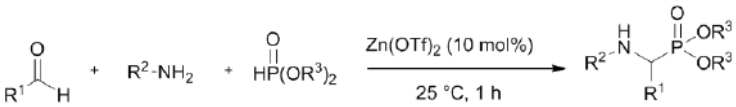
α-aminophosphonates, which are structural analogs of α-aminoacids and transition state mimics of peptide hydrolysis [1, 2], are an important class of compounds in medicinal chemistry with a wide range of biological effects as enzyme inhibitors [3], HIV protease [4], antibiotics [5], anticancer, and antithrombotic agents. With this in mind, and in the continuation of our interest to develop efficient protocols for the synthesis of biologically active phosphonates, we report herein a simple and green methodology for the synthesis of α-aminophosphonates, in good yields, through the zinc triflate-catalyzed one-pot multi-component reaction of aldehydes, amines and dialkyl phosphites, at room temperature, under solvent-free conditions. To further elaborate the scope of this methodology, and in order to investigate the diastereoselectivity of the protocol, we examined the reaction of (*S*)-α-methylbenzylamine. In all these cases, the reaction furnished a mixture of two diastereoisomers (*R*,*S*)- and (*S*,*S*) in an approximate 7:3 ratio, with predominance of the (*R*,*S*)-isomer. The diastereoisomeric ratio was determined by ^{31}P NMR spectroscopy and the configuration was assigned by analogy with results reported in the literature.



Key words: α-Aminophosphonates; Kabachnik-Fields reaction

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ENCAPSULATION OF VANADIUM-LIGAND BOX INTO LAPONITE R-D: APPLICATION AS HETEROGENOUS CATALYSTS IN THE ASYMMETRIC EPOXIDATION OF STYRENE USING TBHP

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[b] *Instituto de Síntesis Química y Catálisis Homogénea. Facultad de Ciencias, C.S.I.C.-Universidad de Zaragoza-50009 Zaragoza, Spain.*

The box-vanadium is covalently immobilized on laponite through a direct and simple procedure. The Box-Vanadium catalysts were characterized by different techniques such as N_2 absorption-desorption, X-ray diffraction, FT-IR, RAMAN, they were screened as heterogenous chiral catalysts in the asymmetric epoxidation of styrene using Tert-butyl hydroperoxide TBHP as oxidant and Acetonitrile as solvent.

The encapsulated complexe Box-Vanadium show, in general, high conversion and good enentiometric excess.

The Py-Box-Vanadium supported catalysts suggested high catalytic activity: the selectivity of 57% to styrene oxide at a conversion in excess of 100% could be obtained.

The heterogenised box catalystis demonstrated to be a re-usable and non-leaching catalytic system.

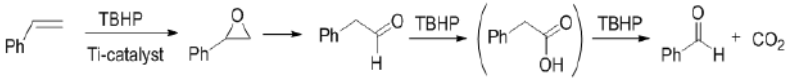

Fig. 1: Epoxidation of styrene

Key words: laponite, vanadium, catalysts, epoxidation

Synthesis, structure and optic nonlinear of a new phosphate $K_6Bi_{13}(PO_4)_{15}$

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Isabelle Ledoux-Rak[c] and Habib Boughzala[a]

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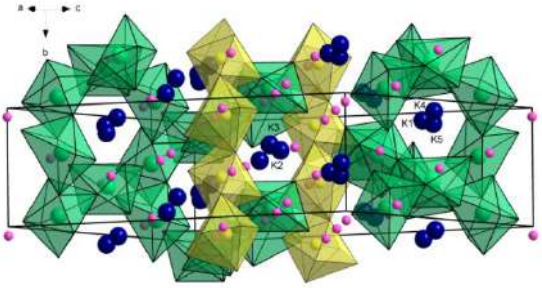
Before the environmental problems caused by fossil energy sources, more work is devoted to research other less polluting sources such as inorganic materials with open structures. Indeed, the structures of these materials containing large channels that accommodate mobile cations are highly sought after in the world of innovative materials. Some compounds may thereby be used in various applications such as in medicine, catalysis, fuel cells, the energy storage and in optical and optoelectronic domains (scintillators and lasers).

This new phosphate $K_6Bi_{13}(PO_4)_{15}$ has been synthesized by solid state reaction and characterized by single-crystal X-ray diffraction. This compound crystallizes in the noncentrosymmetric monoclinic system space group C2 with a=17.637(8) Å, b = 6.9261(8) Å, c = 22.385(4)Å, β = 104.35(2)° and Z = 2. The crystal structure was made up of BiO_n (n=5,6,7,8) polyhedra and PO_4 tetrahedra sharing corners and edges to form an open three-dimensional framework. This architecture delimits tunnels along the [001] direction where K atoms are located. Potassium migration pathways in the anionic frameworks were simulated using the bond valence sum map (BVSM) model. The noncentrosymmetry of the space group was confirmed by second harmonic generation experiments carried out at 1.06 µm on a powder sample.

Keywords: Bismuth phosphate, crystal structure, X-ray diffraction, transport pathways simulation, nonlinear optics.



Projection of $K_6Bi_{13}(PO_4)_{15}$ structure along the [101] direction shown cavities wherein potassium ions are located (the PO_4 tetrahedra have not been drawn for the sake of clarity).

THERMODYNAMIC PROPERTIES OF CH_4 THERMAL PLASMAS MIXED WITH H_2

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This paper is devoted to the calculation of equilibrium compositions, thermodynamic properties : mass density, enthalpy and specific heat at constant pressure of methane CH_4 thermal plasmas mixed with hydrogen H_2. These data are computed in the temperature range 300–30 000 K at atmospheric pressure. The overall results show that the influence of the presence of hydrogen on the thermodynamic properties is important in this temperature range.

Figure 1 shows the specific heat Cp obtained for CH_4-H_2 mixtures as a function of the temperature at atmospheric pressure.

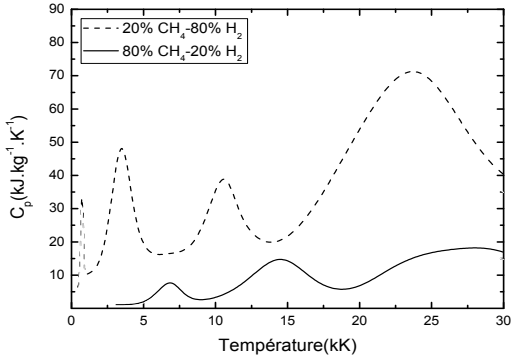

Fig 1. Specific heat at constant pressure a mixture CH_4-H_2.

Key words: thermodynamics of plasmas, methane, hydrogen, thermal plasma,

References

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AC CONDUCTIVITY AND ANTIOXIDANT STUDY OF $[p\text{-}(NH_3)C_6H_4NH_3]_3P_6O_{18}.6H_2O$

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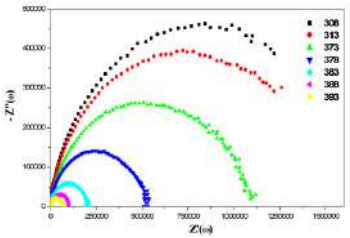
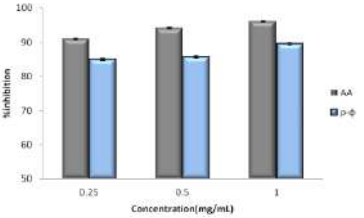
During the last decades, many research activities have focused on the synthesis of organic-inorganic hybrid materials. Thus, to benefit from these materials, it is very useful to have a good understanding of their structural characteristics and some properties which can lead to certain applications. We report in this communication the DC and AC electrical conductivities of $[p\text{-}(NH_3)C_6H_4NH_3]_3P_6O_{18}.6H_2O$. Modulus analysis and dielectric constants have been investigated. The AC conductivity obeys the universal power law. The DC electrical conductivity indicates a semiconductor behavior. The kind of the observed conduction is protonic by translocation. This compound has also been screened for its antioxidant activity in vitro, using 1,1-diphenyl-2-picrylhydrazyl, reducing power, hydroxyl scavenging ability and ferrous ion chelating (FIC) methods and with ascorbic acid as reference.



Complex impedance diagrams



DPPH Radical scavenging activity

Key words: Hybrid material, Complex Impedance, AC Conductivity, Dielectric Modulus, Antioxidant activity.

DFT study of the structure and reaction of ketamine with different derivatization groups

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The structures of ketamine and the reaction of different derivatization groups (MSTFA, MBTFA)[1] have been investigated at the DFT/B3LYP level using 6-311+G (d, p) basis set[2]. Analysis resultants of frontier molecular orbital interaction and the hardness of all molecules, provide a good prediction of experimental results.

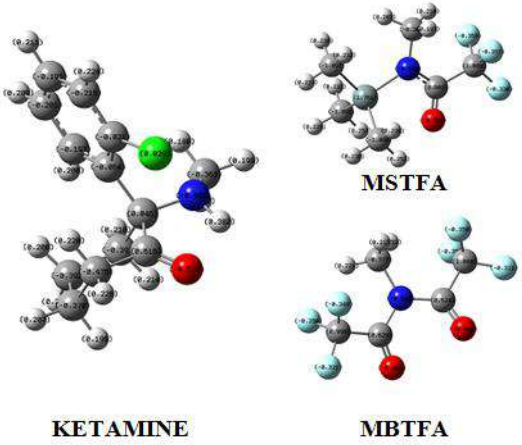

Fig. Structures of ketamine, MSTFA and MBTFA

Key words: DFT calculation, Ketamine, derivatization.

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AMBIDENT BEHAVIOR OF 5-NITRO-3-X-THIOPHENES IN σ-COMPLEXATION ON PARA-SUBSTITUTED PHENOXIDE ANIONS

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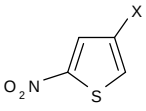
Kinetics of the reactions of 3-5-dinitrothiophene **1** and 3-cyano-5-nitrothiophene **2** with a series of para-X-substituted phenoxide anions **3a-d** (Y= OMe, Me, H and Cl) have been investigated photometrically in aqueous solution at 20 °C.

Two unsubstituted electrophilic centers of thiophenes **1** and **2** were identified. Analysis of the experimental data in terms of Brønsted relationships reveals that the reactions mechanism likely involves a single electron transfer (SET) process. The excellent correlations (R^2 > 0.9910) of the rate constants with oxidation potentials, E^o, values is an additional evidence that reactions between thiophene **1** (or **2**) and phenoxide anions **3a-d** are proceeding by initial electron transfer. It is of particular interest to note that the systems studied in this communication provide a rare example of a SET mechanism in σ-complexation reactions. Evaluation of the kinetic data by the correlation equation (1),yields the electrophilicity parameters (E),which allow a comparaison of the electrophicities of thiophenes with those of other classes of ambident compounds.

$$\log k\ (20\ °C) = s\ (E + N) \qquad \textbf{(1)}$$



1: X = NO_2
2 : X = CN

Key words: Ambident reactivity, SET process, Mayr's relationschip, Kinetic, σ-complexation

References

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Synthesis of new substituted quinolines and methylbenzenesulfonohydrazone coumarin derivatives

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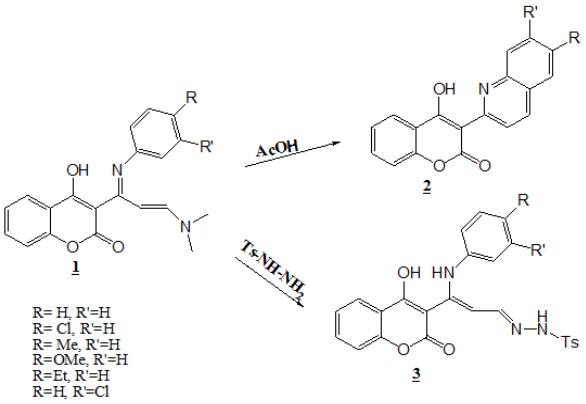
DMFDMA is a versatile reagent widely used in the heterocyclic chemistry because it transforms functional groups to N-methylated groups[1]. In many previous works, these later are great intermediates for the synthesis of new heterocycles[2]. It was also indicated that these compounds are excellent substrats to nucleophilic substitution reactions[3]. These observations have been guiding us for the development of new quinolines and hydrazone derivatives starting from coumarinic enamines **1**. In this frame work, new coumarinic quinolones derivatives **2** and new methylbenzenesulfonylhydrazone derivatives **3** were synthesized in a very reduced time and an economical way with very good yields. All the synthesized compounds were characterized by spectroscopic means such as 1D (^{1}H, ^{13}C) and MS.



Keywords: DMFDMA, hydrazones, coumarin, methylbenzenesulfonylhydrazones, quinolines

References

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Crystal structure and physicochemical properties of a new organic-inorganic hybrid material $C_6H_{16}N_2(NO_3)_2$

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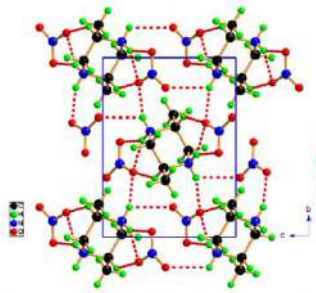
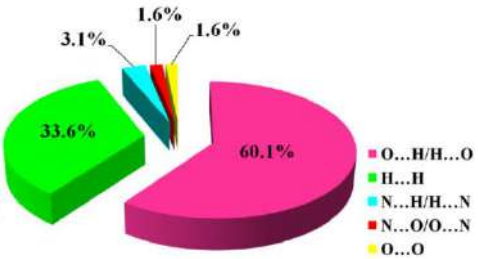
Piperazine and its derivatives are widely used due to their interesting biological and pharmacology proprieties **[1]**. In this work, we report the preparation, the structural investigation, Hirshfeld surface analysis, infrared spectroscopy and thermal analysis of a new organic nitrate $C_6H_{16}N_2(NO_3)_2$. The compound crystallizes in the monoclinic space group P21/c (Z = 2) with lattice parameters: a = 7.035(8) Å, *b* = 10.027(10) Å, *c* = 8.311(8) Å and β = 116.149(8) °. The structure was solved using a direct method by means of 1059 reflections with $I > 2\sigma(I)$ and refined to a reliability R = 0.034 (Rw = 0.09). Intermolecular N-H…O and C-H…O hydrogen bonds link the organic and inorganic entities into a bidimensional network. The diprotonated piperazine ring adopts a chair conformation, with the methyl groups occupying an equatorial position, with puckering parameters: Q = 0.6083 Å, θ = 90 ° and φ = 166 ° **[2]**.



Projection of $C_6H_{16}N_2(NO_3)_2$ along the a axis. The H-atoms not involved in H-bonding are omitted.



Percentage of various intermolecular contacts contributed to the Hirshfeld surface.

Keywords: Organic-inorganic hybrid; piperazine; bidimensional network; Hirshfeld surface; Infrared spectroscopy; Thermal analysis.

References

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Synthesis and characterization of a novel organic-inorganic hybrid material ($C_8H_{12}N)NO_3$

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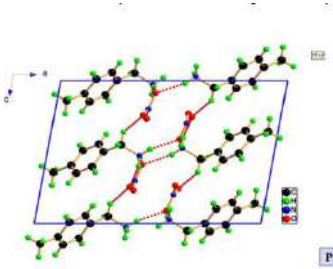
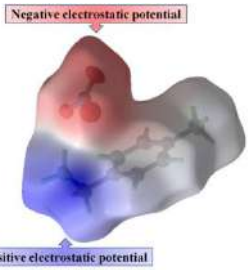
In this work, we present a collective experimental and theoretical study on crystal structure of a new organic-inorganic hybrid material. The title compound crystallizes in the monoclinic space group ($P2_1/c$, Z = 4) with unit cell parameters: a = 15.097(2) Å, b = 5.812(10) Å, c = 10.486(2) Å and β = 99.75(2)°. The atomic arrangement of the title compound can be described by two-dimensional framework. The organic cations are related to the nitrate anions throughout multiple bifurcated N—H···(O,O) and weak C—H···O hydrogen bonds, with donor-acceptor distances varying between 2.900 (2) and 3.234 (2) Å. Each organic entity is surrounded to three different nitrate anions through five N—H···O hydrogen bonds forming $\mathbf{R}_1^2$ (**4**) and $\mathbf{R}_4^2$ (**8**) motifs. Hirshfeld surfaces and two-dimensional fingerprint plots estimate the intermolecular interactions accountable for the generation of crystal packing. The infrared spectrum of this compound reported from 4000 to 400 cm^{-1} confirmed the presence of the principal bands assigned to the internal modes of the organic cation and nitrate anions. Impedance spectroscopy analysis was used to analyze the electrical behavior of sample as a function of frequency at different temperatures. This compound was also characterized by differential scanning calorimetry.



Projection of $(C_8H_{12}N)NO_3$ along the b axis. The H-atoms not involved in H-bonding are omitted.



Hirshfeld surfaces mapped over the electrostatic potential of the title compound.

Keywords: Organic-inorganic hybrid, Two-dimensional network, Hirshfeld surface, Infrared spectrum, Electrical behavior and differential scanning calorimetry.

Study of the Solid Phase Extraction SPE and Chromatographic analysis of Polycyclic Aromatic Hydrocarbons in an aqueous matrix

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Soufiane Touil, Mohamed Ridha Driss*

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Many organic pollutants are dangerous to human, flora and fauna. Among these pollutants we are interested to polycyclic aromatic hydrocarbons. This group is included in a list of priority pollutants of US-EPA [1].
Solid-phase extraction (SPE) procedure followed by HPLC-Fluorescence detection was evaluated for the determination of trace amounts of these pollutants in waters samples. Different parameters affecting extraction efficiency such as: volume of elution solvent, volume of water sample … were studied and optimized.
SPE was carried out on different cartridges and high recoveries were test educing different quantity of sorbent phase such as C18 [2] for the treatment of 500 mL water sample. The described method was then tested on spiked tap, mineral, sea and traited water (78.57 % – 93.33 %) for the examined PAHs in high volume of water samples. Among the studied compounds, different PAHs were detected in water at different concentration level.

Key Words: PAHs, SPE, HPLC.

References

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SYNTHESIS OF (2S,4R)-2-AMINO-4-HYDROXY-*N*-METHYLPENTANAMIDE DERIVATIVE OF UNNATURAL AMINO ACID

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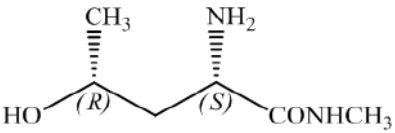
Amino acids are building blocks essential for life, as their association through amide bonds leads to peptides and proteins. They are the focus of many studies, in particular in the field of bioorganic chemistry. The design of peptide analogues has therefore attracted significant interest and synthetic unnatural α-amino acids have found many applications, for example, as starting materials for the total synthesis of natural products, as chiral auxiliaries, as ligands for metal-catalyzed reactions, or for the design of drugs or bioactive molecules. Isoxazolidines offer a general route to natural and unnatural amino acids, through opening of the isoxazolidine ring, usually by reductive cleavage of the weak N–O bond. We herein describe the synthesis of (2*S*,4*R*)-2-amino-4-hydroxy-*N*-methylpentanamide by 1,3-dipolar cycloaddition of a chiral nitrone with allyl bromide.



Key words: Amino acid/ Isoxazolidine/ Nitrone/ Allyl bromide.

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Bioactive Boron-Glasses as orthopedic implants Microstructure and Mechanical Properties after Spark Plasma Sintering

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This research describes a new approach for the synthesis, of bioactive glasses associated with boron (Bx), by melting method have been successfully conducted by spark plasma sintering process, resulting in a dense bioactive glass compacts. Besides, the sintering behaviour of glass powders with different amount of boron ranging from 0% to 20% in mass at 50MPa was studied. The microstructure, Vickers microhardness, fracture toughness and density are described. The suitable sintering condition under the pressure of 50 MPa is 420°C for 2 min.
A maximum value of the density was reached at 420°C-50MPa-2min for B_{20}. Although, the mechanical properties of bioactive boron-glasses (Bx) decreased with increase in the boron amount added into the vitreous structure.

Key words: Bioactive Glasses, Boron, Density, mechanical proprieties, SPS, Bone substitute.

INFLUENCE OF THERMAL TREATMENT ON THE SYNTHESIS OF MAGNESIUM ORTHOSILICATE AND ITS GRANULOMETRY

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The magnesium orthosilicate (Mg_2SiO_4, forsterite) is an important compound of the olivine family that has interesting physical and chemical properties. This work is a contribution to the study of its preparation by the sol-gel method [1-2]. The effect of calcination temperature for a short time (30 minutes) on Mg_2SiO_4 formation and its size was examined. The various synthesized powders were characterized by X-ray diffraction, Infrared spectroscopy and scanning electron microscopy. We also determined the distribution of particle sizes by a laser-diffraction particle sizer. The results showed that the sample calcined at 500°C is Mg_2SiO_4 with a unimodal particle size distribution centered at 122.8 microns. The dispersion index I (indicator of particle size uniformity) is 1.84. With increasing temperature, well crystallized compounds were obtained. Their particle size distributions remain unimodal and slightly shift toward smaller particles. A decrease of the dispersion index was also observed, indicating the formation of Mg_2SiO_4 particles with more uniform size. This study showed that 900°C can be chosen as appropriate energy saving temperature for the preparation of a pure and well crystallized forsterite in only 30 minutes of annealing time.

Keywords: Synthesis, Sol-gel, Forsterite, Particle, Size Distribution

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Comparison of Fenton and Electro Fenton processes for degradation of a cytostatic agent

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As anticancer drugs allow people to live longer and to achieve higher standards of living, the consumption of these compounds is expected to increase considerably. As a consequence, they are more likely to be present in the different environmental matrices in higher concentrations. They have been regarded as potential carcinogenic, genotoxic, mutagenic and teratogenic compounds because of their none-selective mode of action. Indeed, they affect both cancerous and healthy cells. It is, however, not known how and to what extent these substances affect organisms and the environment in the long run. That is why they should have special consideration. Hence, pharmaceutical industry effluent generated from these substances should be treated and degraded efficiently.

The elimination of these cytotoxic drugs and other pharmaceuticals by conventional wastewater treatments is often incomplete and inneficient, that is why other alternatives need to be tested. Advanced oxidation processes may yield good results in terms of decontamination. They are environmentally friendly methods based on the large production of hydroxyl radicals ($OH^{\bullet}$) which react with any organic pollutants and thus lead to their mineralization. For this purpose, we have tested and compared the influence of two advanced oxidation processes, Fenton and Electro Fenton processes, in order to eliminate a cytostatic agent generated from the wastewater of oncology industry. We have also used a factorial experimental design to determine the influent parameters in the degradation of the anticancer by the electrochemical treatment.

Key words: anticancer drugs, advanced oxidation processes, degradation, mineralization, wastewater treatment.

PHYTOCHEMICAL STUDY OF LOTUS PUSILLUS MEDIK (FABACEAE)

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The genus *Lotus* (tribe Loteae, family leguminosae) comprises about 120–130 species in the Algerian flora and is represented by fifteen species most of which occur in the Sahara [1]. In folk medicine, plants of genus Lotus are used as contraceptives, prophylaxis and treatment of sexually transmitted disorders and peptic ulcers [2]. *Lotus pusillus* Medik. named also *Lotus halophilus* Boiss has a good antimicrobial activity against Gram-positive, Gram-negative bacteria and fungi. Phytochemical investigation of the aerial parts of *Lotus pusillus* Medik. Resulted in the isolation and identification of three known compounds. Liquid-Liquid fractionation of the crude extract followed by chromatographic purification resulted in the isolation of Soyacerebroside I (**1**), 1-*O*-(9Z,12Z-octadécadiénoyl)-2-*O*-(9Z,12Z,15Z-octadécatriénoyl)-3-*O*-(6′ sulfoquinovopyranosyl)-glycérol (**2**) and a saponoside was identified as Azukisaponine V.

The molecular structures of isolated compounds were established by spectroscopic analysis particularly NMR 1D (^{1}H and ^{13}C J-modulated) and 2D (COSY, HSQC J-modulated, HMBC, TOCSY and ROESY), mass spectrometry EI-MS and by comparison with literature data.

Key words: *Lotus pusillus*, Leguminosae, maltol, saponin

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STRUCTURES OF NEW COMPOUNDS CONTAINING PYRAZOLE AND 1,3,4-OXADIAZOLE MOIETIES

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The multi-step reactions starting from the malonic acid dihydrazide **1** afford the hydrazones **2** and **4**. The Vilsmeier-Haack reagent [1] then leads to symmetrical di-pyrazoles **3** and **5**.

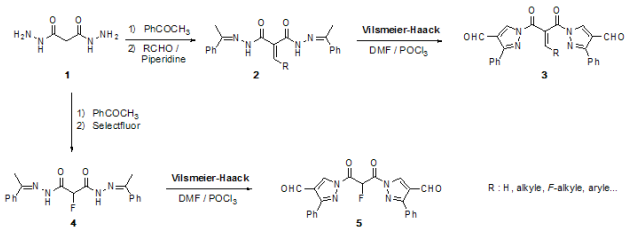

Fig. 1 Synthetic pathways to symmetrical pyrazoles

Different means of cyclization are used in order to transform the dihydrazides **6** into 1,3,4-oxadiazoles **7**, **8** and **9** [2].

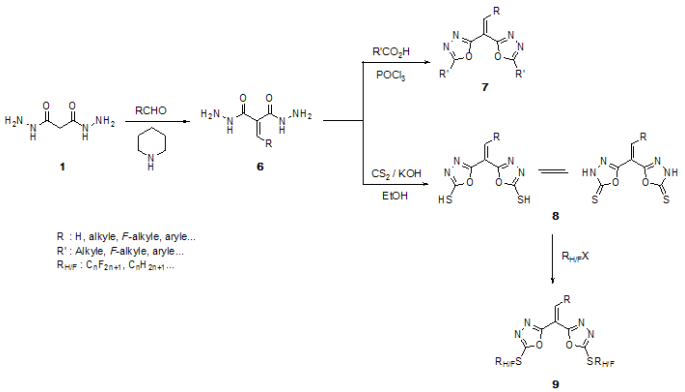

Fig. 2 Structures of different 1,3,4-oxadiazole derivatives

Key words: Pyrazole, 1,3,4-oxadiazole, fluorine, cyclization.

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Synthesis, characterization and Hirshfeld surface analysis of m-xylylenediaminium bis(perchlorate) dihydrate

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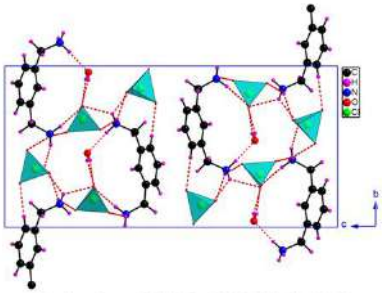
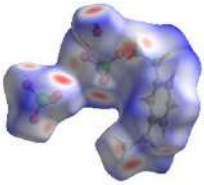
The crystal structure of a new compound $(C_8H_{14}N_2)(ClO_4)_2.H_2O$ has been obtained by slow evaporation at room temperature and characterized by X-ray single crystal analysis, Hirshfeld surface analysis, differential scanning calorimetry (DSC), IR and Raman spectroscopies. The title compound crystallized in the monoclinic system, space group $P2_1/c$, with a = 5.5781(10) Å, b = 11.1137(3) Å, c = 23.0731(7) Å, β = 95.414(1) °, V = 1424.0(3) Å^3 and Z = 4. The asymmetric unit of the title compound contains one m-xylylenediaminium cation, two perchlorate anions and one water molecule. The atomic arrangement of the title compound can be described by two-dimensional network. Therefore, chlorine atoms, Cl2, are discrete and Cl1 form with water molecules corrugated channels extending along the ***a***-axis at z = ¼ and ¾. These inorganic entities are allied to organic cations $[C_8H_{14}N_2]^{2+}$ through intricate hydrogen bonding. The intermolecular interactions were further evaluated by using the three-dimensional Hirshfeld surfaces and two-dimensional fingerprint plots. The hydrogen bonding interactions associated to O–H…O, N–H…O and C–H…O represent the top fraction of 69.1 % followed by these of the H…H type contributing 17.5 %.



Projection of $(C_8H_{14}N_2)(ClO_4)_2.H_2O$ along the a axis.



Hirshfeld surface mapped over d_{norm} of $(C_8H_{14}N_2)(ClO_4)_2.H_2O$.

Keywords: crystal structure, m-xylylenediaminium, perchlorate, hydrogen bonding, Hirshfeld surface analysis.

AB INITIO INVESTIGATION OF STRUCTURAL, ELECTRONIC AND MAGNETIC PROPERTIES OF $TbNi_5$ COMPOUND

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The spin-polarized, electronic and magnetic properties of $TbNi_5$ inter- metallic compound have been calculated by employing the full-potential linear augmented plane waves (FP-LAPW) within the density functional theory (DFT) [1] and implemented in the WIEN2k package [2]. In this approach, the exchange-correlation potential (XC) is described by the spin-polarized generalized gradient approximation of Perdew-Burke-Ernzerhof with Hubbard U-correction (PBE-GGA+U) [3–5]. The Hubbard term of Coulomb repulsion in the 4f –shell of Tb was taken as U = 3.5 eV and J = 0.7 eV. The electronic structure such as band structure and density of states have been investigated. Total and local magnetic moment of Tb and Ni are also estimated it is shown that the total magnetic moment of this alloy is vigorously contributed by the local moment of terbium.

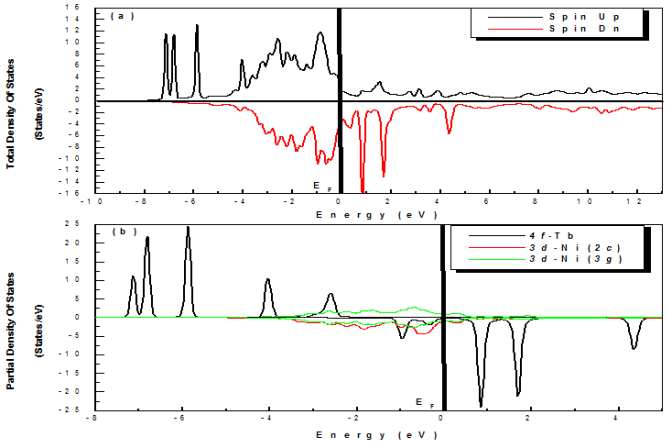

Fig.: Spin-dependent total (a) and partial (b) densities of states of the equilibrium $TbNi_5$ compound alloy, using GGA+U scheme.

Keywords : Intermetallic; $TbNi_5$; Electronic structure; Density of states;· Magnetic moment.

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Effect of various physico-chemical parameters on the removal of contaminated copper (Cu (II)) ions from aqueous solutions

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The potential of coffee ground waste (CGW) as a low-cost natural agricultural waste bio-mass for the removal of copper (Cu (II)) ions from aqueous solutions wastested in batch experiments as a contaminated water purification process. The effect of various physico-chemical parameters such as initial pH, initial Cu (II) ion concentrations, contact time, and particle size was sought. The optimum pH for adsorption was measuredat 5. For all initial Cu(II) ion concentrations (50 $mg.l^{-1}$ to 500 $mg.l^{-1}$), 30 min was selected as optimal contact time to reach the maximum adsorption rate. Kinetics data were best described by the pseudo-2^{nd}-order model. The experimental data were well fitted with Freundlich, Temkin, Langmuir,Harkins-Jura andHalsey isotherm models. The Langmuir model was the best with a high correlation coefficient value (R^2 = 0.998).The maximum quantity of adsorbed Cu (II) ions (q_m), and Langmuir adsorption equilibrium constant (K_l) were respectively 7.92 $mg.g^{-1}$ and 0.081 at 298 K. Different active functional groups which were responsible for the Cu (II) ion adsorption process were identified through FTIR.

Reduction of 1,2-dicarbonyl compounds and their N-phenylimine derivatives by sodium cyanide under aprotic conditions

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Some aromatic 1,2-dicarbonyl compounds, i.e. 9,10-phenanthrenequinone, acenaphthe- nequinone and benzil, and their corresponding N-phenyl monoimines, have been reduced, using dry acetonitrile as a solvent, in the presence of sodium cyanide as a reducing agent. Comparative potentiostatic preparative-scale electrolysis is described.

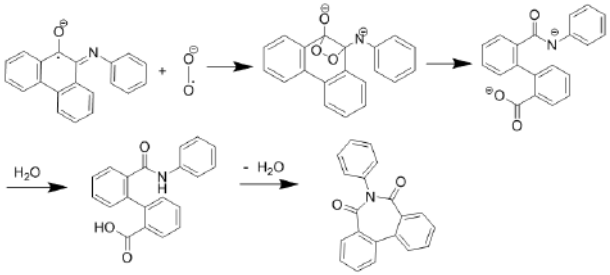

Figure 1: Electron transfer between the monoimine of 9,10 phenanthrenequinone and the oxygen

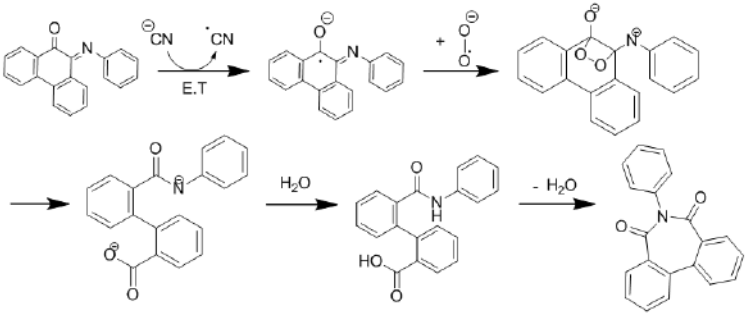

Figure 2: Reduction of the monoimine of 9,10 phenanthrenequinone by sodium cyanide

Keywords: Homogeneous electron transfer Radical anion Cathode Superoxide anion Oxygen

EFFECT OF DIVALENT TRANSITION METAL ON STRUCTURAL, MORPHOLOGICAL, MAGNETIC AND ELECTROCHEMICAL PROPERTIES OF $Na_2M_2M'(PO_4)_3$, (M and M'= transition metals)

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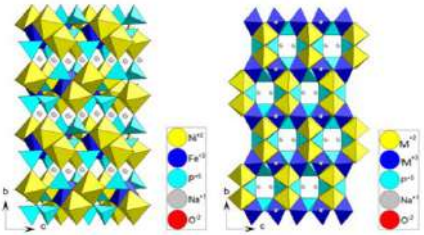
Na-ion batteries have regained considerable interest recently as an alternative to Li-ion batteries for electric storage applications because of the low cost and natural abundance of Na resources [1].
The best candidates for cathodic materials in Na-ion batteries are phosphate based materials, because of their thermal stability and higher voltage due to the inductive effect, we can mention olivine $NaFePO_4$
Accordingly, novel poly anion compounds considered as alternative materials for $NaFePO_4$ of chemical formula $\mathbf{Na_2M_2M'(PO_4)_3}$ (M and M' = transition metals) have been prepared by using auto-combustion method [2]. Hence, Rietveld refinement analyses of powder X-ray diffraction data confirmed allotropic crystal structural transition from monoclinic to orthorhombic symmetry with changing divalent transition metal ion [3], (Fig. a,b).
In addition, the microstructure and particle size of the as-prepared materials were investigated, using field emission gun scanning electron microscope. Moreover, the magnetic studies have also been carried out. Finally, the electrochemical performance of the assembled Na ion batteries was systematically explored and compared in terms of the specific capacity, columbic efficiency and cycle life [4].



Key words: Na-ion batteries, poly anion compounds, in situ XRD

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NEW DIPHOSPHOPENTAMOLYBDATE $K_5[HP_2Mo_5O_{23}]\cdot 10H_2O$: SYNTHESIS, STRUCTURE, AND CHARACTERIZATION

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Université de Monastir, Faculté des Sciences Monastir, 5000 Monastir, Tunisia

A new diphosphopentamolybdate (VI) $K_5[HP_2Mo_5O_{23}]\cdot 10H_2O$ has been prepared employing solution method and characterized by single crystal X-ray diffraction, energy dispersive analysis, infrared spectroscopy, thermal stability analysis, cyclic voltammetry analysis, and ultraviolet visible spectroscopy. The compound crystallizes in the orthorhombic, space group *Pcab*: a = 15.9661(10) Å, b = 19.0832(10) Å, c = 20.0970(9) Å, V = 6123.27 (10) Å^3, Z = 8. The crystal structure contains the polyanion units $[HP_2Mo_5O_{23}]^{5-}$ around the potassium cations and lattice water. The $[HP_2Mo_5O_{23}]^{5-}$ subunits are connected together through K−O−Mo bridges and hydrogen bonds. The crystal shape morphology was simulated using the Bravais–Friedel–Donnay–Harker model.

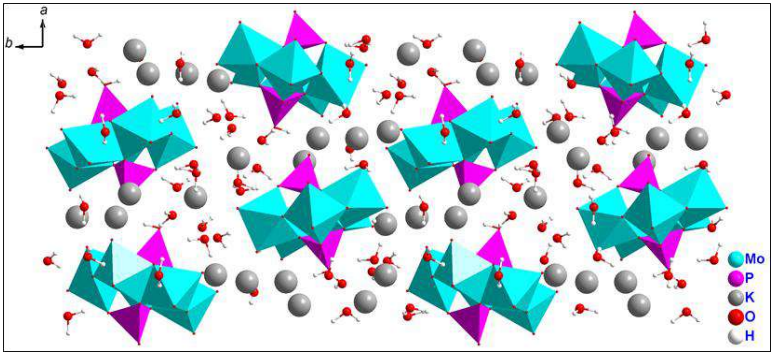

Fig. View of $K_5[HP_2Mo_5O_{23}]\cdot 10H_2O$ along the *c* axis.

Key words: Diphosphopentamolybdate, Synthesis, Crystal structure, Characterizations, Crystal Morphology.

SYNTHESIS, CRYSTAL STRUCTURE, SPECTROSCOPIC CHARACTERIZATION AND HIRSHFELD SURFACE ANALYSIS OF MANGANESE COMPLEX BASED ON 3-AMINO-1,2,4-TRIAZOLE LIGAND

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The 3-amino-1,2,4-triazole (Hatz)-based manganese complex, $[Mn(H_2O)_2(Hatz)Cl_3].H_2O$ (1), was prepared by slow evaporation and characterized through IR spectroscopy and single-crystal X-Ray diffraction. The title complex feature a mononuclear complex with the asymmetric unit composed of unique Mn(II) atom, one $Hatz^+$ cationic ligand, two coordinated water molecules and three Cl^- anionic ligands. An additional unicoordinated water molecule of crystallization is, however, present **(Figure**). The metal center of the studied complex has distorted octahedron geometry. In the crystal structure individual complexes are interconnected through strong hydrogen bonds of types N—H…Cl and N—H…O into 1D chains running parallel to the *a*-direction of the unit cell. The three-dimensional Hirshfeld surface (3D-HS) analysis **[1]** and the two-dimensional fingerprint plots (2D-FP) [2] reveal that the structure is dominated by H…O, H…Cl and H…H contacts.

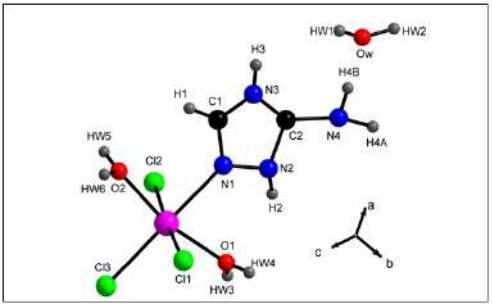

Fig. The asymmetric unit of (1) showing the atom-numbering scheme.

Key words: Manganese complex, crystal structure, Hirshfeld surface analysis,

References

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ISOLATION, STRUCTURAL ELUCIDATION AND TOTAL SYNTHESIS OF CONVOLVINE: A TROPANE ALKALOID FROM THE TUNISIAN *CONVOLVULUS DORYCNIUM* L.

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The present study describes the isolation, the structural elucidation and the total synthesis of convolvine **1** from the roots of the plant *Convolvulus dorycnium* growing in Tunisia. This compound was isolated from the *n*- butanolic extract. Its structure was elucidated on the basis of spectroscopic evidences including, 1D and 2D-NMR (COSY, HSQC, HMBC and NOESY), ES-HRMS, and comparison with literature[1]. To strengthen the structure assignment of the natural convolvine **1** from the Tunisian *C. dorycnium*, we envisioned to perform its synthesis which would match with the proposed structure of natural convolvine. In the retrosynthetic analysis toward the convolvine, we assumed that a demethylation reaction of the *N*-methylconvolvine **4** might be appropriate to obtain the desired structure[2]. The intermediate **4** in turn can be derived from the simple acylation of the commercially available tropine. The total synthesis of the convolvine **1** was performed in the aim to confirm the structure of the isolated one. The strategy involved a four steps sequence starting from tropine.

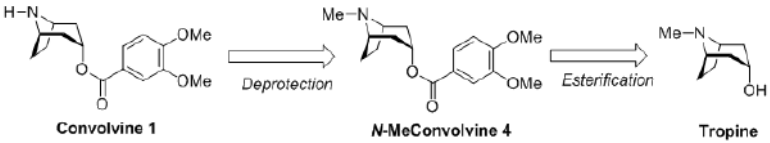

Fig. Retrosynthetic analysis of Convolvine **1**.

Key words: *Convolvulus dorycnium* L., Convolvine, NMR, Synthesis.

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MODELING OF SUNFLOWER OIL BLEACHING AND OPTIMISATION OF THE QUANTITIES OF BLEACHING EARTH USED IN THE REFINING PROCESS

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The refining process of crude edible oil includes four operations; degumming, neutralization, bleaching and deodorization. After its degumming and neutralization operations during the refining process, the crude edible oil still contains undesirable substances. Those substances limit the conservation of oil and degrade its quality by altering the taste and color properties of this product, which affects its market value by giving it an aspect that is not appreciated by the consumer. This color is due to the presence of pigments in the crude edible oil, such as chlorophyll-a and β-carotene. Among undesirable substances, the crude edible oil contains also soap residues, free fatty acids, phosphatides and metals at trace concentrations.

The Bleaching operation is carried out by means of porous materials with a strong adsorption power, which are clays called bleaching earths, activated by heating with strong acids or microwave irradiation. These materials eliminate the undesirable elements such as the dyes (chlorophyll-a and β-carotene) and other residues (soap residues, traces of heavy metals contained in the crude edible oil.

The region of Bejaïa (Algeria) has two edible oil refineries (COGB-Labelle and Cevital). They produce huge quantities of waste called spent bleaching earths, which are discharged directly in the rubbish dump without any treatment that would prevent contamination of the environment. This presents a serious problem regarding the solid wastes management, storage and the harmful effects they would cause. To solve the problem of waste products, the edible oil refining factories have opted for the reduction of bleaching earths used. Moreover, the reduction of amount of bleaching earth added to the bad controls of temperature and contact time causes an incomplete decolorization. Indeed, during the deodorization step that occurs by high temperature processing (240-270 °C), all the thermally degradable pigments, undesirable elements or volatile compounds, such as, aldehydes, ketones and alcohols are removed by vacuum distillation. Therefore, these substantial color reductions increase air pollution.

The aim of this study is to propose mathematical models governing the bleaching of the sunflower oil over a commercial bleaching earth used in edible oil refining factory (Cevital, Bejaia, Algeria) in order to optimize the bleaching parameters, which are temperature, clay dosage and contact time. Besides, this study contributes to a better control of the most influencing physicochemical parameters (temperature, clay dosage and contact time) on the decolorization step - while keeping a better quality of oil - in order to reduce risks of air pollution and spent bleaching earth that presents a potential threat to our region.

The modesl were developed using the Matlab programming language. The input variables are the oil temperature (X_1 in °C), the clay dosage (X_2 in % w/w) and the contact time (X_3 in minutes). The output parameter is the bleaching capacity (Y in % uptake). The reliability of the selected models was analyzed using four statistical criteria: the residual variance, the coefficient of determination (r^2), the Student test and the Fisher–Snedecor test

The developed models allowed predicting the bleaching capacity representing the removal of the β-carotene and chlorophyll-a pigments as a function of the bleaching parameters. These parameters were optimized: temperature (80°C), clay dosage (1%) and contact time (25 min).

Keywords: Modeling, bleaching, sunflower oil, pigments, solid wastes.

EFFICIENT AND GREEN ONE-POT SYNTHESIS OF ARYLPHOSPHONATES

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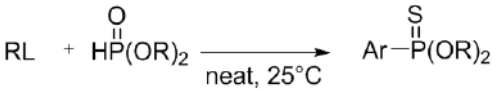
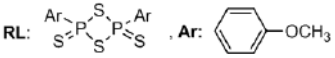
In recent years the synthesis of organophosphorus compounds has attracted much attention due to their wide applications in organic synthesis, medicinal chemistry, and material science[1]. In light of the potential importance of arylphosphonates derivatives and in the continuation of our interest to develop efficient protocols for the synthesis of biologically active phosphonates, we report herein a highly efficient, cost effective, general, P-arylation protocol for the synthesis of arylphosphonates and their derivatives. Arylphosphonates are obtained by condensation of various H-alkylphosphonates with lawesson's reagent at room temperature, under solvent-free conditions in excellent yields. This synthetic strategy offers significant advantages such as good yields, versatile substituents, mild reaction conditions, easy work-up and environmental safety, what make this protocol more amenable for high throughput library synthesis.





Key words: green synthesis, P-arylation, room temperature

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Degradation of Acetic Acid by Advanced Oxidation Using the Fenton System Fe (II) / H_2O_2

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The last twenty years many studies published in the literature devoted to the emergence of new treatment processes; including the advanced oxidation processes (AOP) occupy an important place, in fact, they are very interesting for the degradation of recalcitrant organic molecules. These techniques are complementary to the usual methods of flocculation, precipitation, activated carbon adsorption or membrane processes. Advanced oxidation aims for a complete mineralization of organic pollutants into CO_2, H_2O and other inorganic compounds such as Cl^-, SO_4^{2-}, NH_4^+, etc ...

This study aims the application of Fenton system as oxidation process of an organic pollutant (acetic acid) in aqueous solution, the choice of conditions implementation of the reactive is important, in order to generate enough hydroxyl radicals to oxidize contaminants. Degradation of acetic acid is possible. However, a significant difference in reactivity was observed using different ratios of [H_2O_2] 0 / [Fe (II) namely 2, 10, 20 and 39.

All experiments carried out are characterized by a very rapid oxidation to the first few minutes associated with significant production of hydroxyl radicals resulting from the reaction between Fe (II) and H_2O_2. A second phase characterized beyond that time, by a slight decline in the rate of oxidation of acetic acid. Degradation rates obtained were 22%, 50%, 72% and 80% respectively for reports 2, 10, 20 and 39.

Key words: advanced oxidation, acetic acid, degradation, Fenton system

Eco-Friendly Solvents for Palladium-Catalyzed Desulfitative C-H Bond Arylation of Heteroarenes

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The Pd-catalyzed regioselective direct arylation of heteroarenes in which benzenesulfonyl chlorides were used as coupling partners through a desulfitative cross-coupling performed in diethyl carbonate (DEC) or cyclopentyl methyl ether (CPME) as green and renewable solvents or even in neat conditions instead of dioxane or dimethylacetamide (DMA)[1]. Under these solvent conditions, the reaction proceeds with a wide range of heteroarenes. C_2- or C_5- arylated products were obtained with furan and pyrrole derivatives. Benzofuran was also arylated regioselectively at the C2- position, whereas the reaction proceeds selectively at the C3- or C4-positions if thiophenes and benzothiophenes were used. [2]

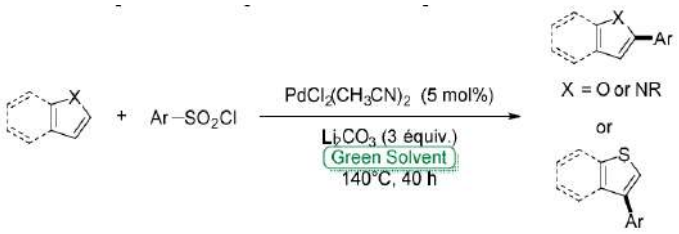

Fig. Pd-catalyzed desulfitative C-H bond arylations in which benzenesulfonyl chlorides are used as coupling partners

Key words: Eco-Friendly Solvents, DEC, CPME, arylation, desulfitative, palladium-catalyzed

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Supramolecular and lamellar architecture of new organically templated metal sulfate with 2-amino-4-methylpyrimidine: Crystal structure and thermal behavior

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Supramolecular and lamellar hybrid materials based on covalent and non-covalent interactions has been one of the most active areas of research in recent years due to their intriguing structural diversities and potential applications [1]. In the field of crystal engineering, many attempts have been made to control the molecular arrangements in crystals by simultaneous use of coordination bonds of a transition metal ion and intermolecular hydrogen bonds [2]. Building blocks possessing such non-covalent interaction sites can produce one-, two- and three-dimensional molecular arrangements with long-range order [3].

In this field, we succeed to synthesis a new hybrid double sulfate salts, incorporating aromatic 2-amino-4-methylpyrimidine as template and containing transition metals, with the general formula $(C_5\ H_8\ N_3)_2[M^{II}(H_2O)_6](SO_4)_2{\cdot}2H_2O$ (M^{II} = Co and Cu) provided a new structure topology that gathers between the supramolecular aspect and the lamellar aspect, which is not very much encountered in the literature. The network structural are stabilized by hydrogen bonds and weak interaction. Indeed, inorganic molecules are interconnected through O–H·····O hydrogen bonds while aromatic amines are bounded to the mineral part through N–H·····O hydrogen bonds and they interact together through π–π interactions. On our side, the thermal study assured by TG-DSC shows good stability of precursors.

Key words: Lamellar structure, supramolecular, thermal study

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CRYSTAL STRUCTURE AND PHYSOCOCHEMICAL PROPERTIES OF A NEW ORGANIC-INORGANIC HYBRID DAWSON $(C_4H_7N_2)_{12}$ $[P_2W_{18}O_{62}]_2$

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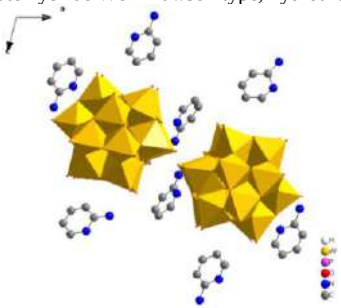
Tungsten (W) is a complicated metal to study, not only because of its ability to form complexes with various coordination number and geometries, but also since it has a tendency to form clusters and poly-nuclear complexes with a verity of metal ions.
Heteropolytungstates (HPT), with general formula $[XmWnOp]^{q-}$ (X = Si, P, B, Co, Fe, Cu..) are the poly-condensed products of tungstate (WO_4^{2-}) anion, formed at intermediate pH in presence of a hetero-atom and is one of the intensely studied area of polyoxometalates (POMs) chemistry [1,2,3].
A novel organic–inorganic hybrid compound based on Wells-Dawson polyoxotungstate, $(C_4H_7N_2)_{12}[P_2W_{18}O_{62}]_2$ (1) has been hydrothermally synthesized and characterized by single-crystal X-ray diffraction, infrared, ultraviolet spectroscopy and thermogravimetric analyses. This compound crystallized in the monoclinic system, space group P21/n, with a = 33.271(4)Å, b =14.643(15)Å, c = 33.584(3) Å, β= 105 (9)°, V = 15801.7(3)Å^3 and Z = 4. The crystal structures of the compounds exhibit three-dimensional supramolecular assembly based on the extensive hydrogen bonding interactions between organic cations, water molecules and Wells-Dawson polyoxoanions.

Keywords: Polyoxotungstate hybride Well-Dawson type, hydrothermal condition.



A packing view of $(C_4H_7N_2)_{12}[P_2W_{18}O_{62}]_2$ along the b axis

Reference:

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APPLICATION OF THE RECHTSCHAFFNER AND DOEHLERT DESIGN FOR ELABORATION OF $NiFe_2O_4$ THIN FILMS BY CBD

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Actually, thin films of spinel ferrite with form MFe_2O_4 (M = Ni^{2+}, Co^{2+}, etc.) have attracted a huge attention due to their wide applications in various fields such as magnetic refrigeration, magnetic resonance imaging and high density data storage [1]. In this work, thin films of nanocrystalline ferrimagnetic $NiFe_2O_4$ have been elaborated by chemical bath deposition (CBD) on stainless steel (SS) substrate. The effect of principal experimental parameters on the film growth has been studied using the experimental design methodology. Furthermore, the Rechtschaffner design has shown that the bath and annealing temperature were the main influential parameters. The optimal operating parameters of depositions have been investigated using the Doehlert matrix. Complimentary techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM) and VSM magnetometer were carried out in order to investigate phase formation. Moreover, XRD studies revealed the formation of a single phase of $NiFe_2O_4$ with preferential orientation in the (3 1 1) plane.

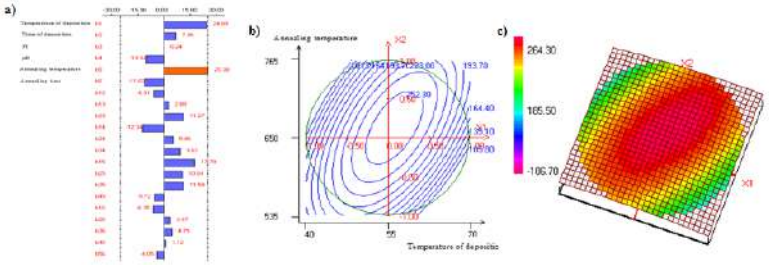

Fig: a) Graphical analysis of the different factors effect; **b)** Isoresponses curves of $NiFe_2O_4$ films saturation magnetization; **c)** corresponding three-dimensional plot.

Key words: $NiFe_2O_4$, CBD, Rechtschaffner design, Doehlert Matrix, Magnetic.

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SYNTHESIS AND STRUCTURAL STYUDY OF THE TETRAGONAL $NaCo_4(AsO_4)_3$

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A novel arsenate $NaCo_4(AsO_4)_3$ has been synthesized by solid state reaction and its structure was determined by single-crystal X-ray diffraction. It is isostructural to $NaMg_4(VO_4)_3$ [1] with tetragonal symmetry, space group $I\bar{4}2d$, a=6.854(2) Å, c=18.949(2) Å and Z=4. The structure was solved and refined to final agreement factors R(F2)=2.20%, Rw(F2)=5.60%, S(F2)=1.13. The obtained structural model was supported by both bond-valence-sum (BVS) and charge-distribution (CD) validation tools [2, 3].

The asymmetric unit consists of a Co_2O_{10} dimer formed by two edge-sharing CoO_6 octahedra, two independent AsO_4 tetrahedra and one Na atom. Each O atom connects two CoO_6 octahedra and one AsO_4 tetrahedron (Fig. 1). The three-dimensional network encloses cavities in which Na^+ cations are located (Fig. 2). The possible motion of monovalent cations in the anionic framework is simulated by means of a generalization of the BVS model.

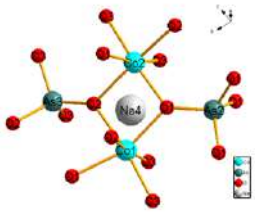

Fig. 1: The asymmetric unit of $NaCo_4(AsO_4)_3$.

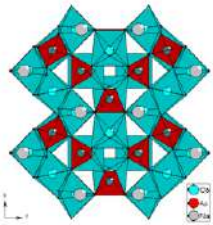

Fig. 2: Projection of structure along the [100] direction.

Key words: $NaCo_4(AsO_4)_3$, BVS, CHARDI. CHARDI.

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SEQUENTIAL EXTRACTION OF PECTIN FROM *PRUNUS AMYGDALUS* PEELS: PHYSICOCHEMICAL CHARACTERIZATION AND ANTIOXIDANT ACTIVITY

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In Tunisia, the *Prunus Amygdalus* is used in the folk medicine for the treatment of diseases. Many researchers are interested in the extraction of bio products from almond peels (hull and shell) and evaluation of their biological and pharmacological activities[1,2]. In this work, we are interested in the study of the antioxydant activity of polysaccharide extracted from *Prunus Amygdalus* shell. The almond used in this researches are collected from Sfax in Tunisia, their shells are dried and grinded. In order to obtain pure pectin, first we prepared the alcohol insoluble solids (AIS) using the ethanol (96%). Water, ammonium oxalate and hydrochloric acid were used to extract different pectic fractions sequentially. Water was found to be the most effective extracting agent with the highest extracting yield (9%). The Water Soluble Pectin (WSP) exhibited high galacturonic acid content (98.5%), high neutral sugar (86.1%) and degree of esterification (44.1%). All of this characteristics have contributed to the excellent thickening properties of WSP. Moreover, the different pectic fractions are characterized by Fourier Transform Infrared Spectroscopy (FTIR). The antioxidant activity was determined with three different analytical assays (DPPH, ABTS and FRAP radical scavenging assay). The WSP showed better DPPH (70.60, 77.38, 83.48 and 86.02% at the concentration of 0.6, 1.25, 2.5 and 5 mg/mL, respectively), FRAP (2468.8, 3888.8, 4568.8, 4668.8 and 4768.8 µmol/L at the concentration of 0.6, 1.25, 2.5, 5 and 10 mg/mL) and ABTS (62, 69.42, 75, 80.14 and 82.28% at the concentration of 0.6, 1.25, 2.5, 5 and 10 mg/mL) radical scavenging capacity. These results showed that *Prunus Amygdalus* peels had a very interesting antioxidant activity and may comprise the raw materials for new drugs.

Keywords: *Prunus Amygdalus*; sequential extraction pectin; physico-chemical characterization; antioxidant activity.

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Synthesis, Characterization and Enhancement of the Bioactivity of a New Co(II)-Hexaborate Complex Based Organic Cation

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A new Co(II)-hexaborate containing $[Co\{B_6O_7(OH)_6\}_2]^{2-}$ building blocks decorated with 1-cyanopiperazinium organic cation; $(C_5H_{11}N_3)[Co\{B_6O_7(OH)_6\}_2].4H_2O$ (1-CPCoB6) was synthesized from aqueous solution and the crystals were grown by the slow evaporation technique and characterized by means of X-ray diffraction, IR, UV–Vis, fluorescence spectroscopy measurements, elemental analysis and TDA-TG thermal analysis. The powder and single crystal XRD analysis revealed that the isolated pure crystals belong to the triclinic system. The structure exhibits interesting porous 3D hydrogen-bonded network built by $[Co\{B_6O_7(OH)_6\}_2]^{2-}$ monomers. The cyanopiperazinium dications are filled in the free void channels of the inorganic framework and interact by means extensive hydrogen bonds. This hydrated mixed hexaborate thermally decomposed in multiple steps involving dehydration and release of organic cation leading to a novel unknown phase which undergoes crystallization. Moreover, the fluorescent and antimicrobial properties of 1-CPCoB6 have also been carried out, showing a red photoluminescence and a promising enhancement of biological activity of reported material in comparison with its parent reactants.

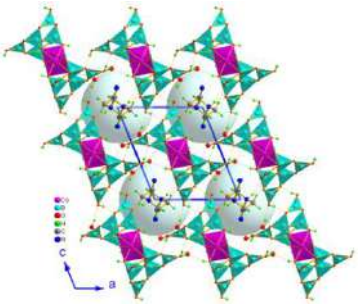

Fig. View of the three-dimensional crystal structure of 1-CPCoB6 viewed along b-axis

Key words: Hydrated Co(II)-hexaborate. X-ray diffraction. Thermal analysis. Photoluminescence. Antibacterial activity

Transition-Metal-Free Nucleophilic Allylic Substitutions of α-(acetoxyalkyl) 2-cyclohexenone by malonic acid derivatives

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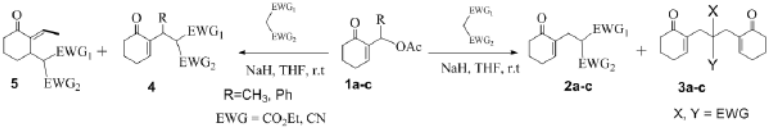
University of Tunis El Manar, Faculty of Sciences Tunis, Laboratory of Structural Organic chemistry

Allylic compounds are useful intermediates in organic synthesis. Correlatively, the Morita-Baylis-Hillman (MBH) acetates have been more employed in the nucleophilic allylic substitution than the corresponding alcohols because the hydroxyl group is considered as an inefficient leaving group that requires a prior activation.[1] In this context, we have previously reported the study of their behavior towards carbon- or heteroatom-nucleophiles in the presence of various catalysts or under transition-metal-free.[2,3] As a continuation of our ongoing research program on the chemistry of MBH, we report herein a transition-metal-free mild nucleophilic substitution of cyclic MBH acetates with malonic acid dervatives.

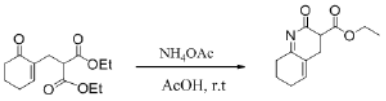
Upon treatment of the acetate **1a** (R = H) with stabilized carbanions derived from malonic acid derivatives (EWG = COOEt, CN), in THF at room temperature, a mixture of the monoallylation products **2a-c** and the bis allyltion products **4a-c** have been isolated in good yields (Scheme 1).



Scheme 1. Reaction of the MBH acetates **1a-c** with acid malonic derivatives

Under the previous conditions (THF, rt), the nucleophilic substitution of **1b** (R =Me) or **1c** (R = Ph) with the same stabilized carbanions, provided a mixture of the kinetic compounds **5** and the thermodynamic ones **4** (Scheme 1).

Finally, as a preliminary synthetic application of the allylation products **2,** we have shown that the conversion of **2a** (EWG = COOEt) into hexahydroquinoline derivative **4a** can be efficiently performed in the presence of an excess of ammonium acetate in acetic acid (Scheme 2).



Scheme 2. Conversion of the allylation product **2a** into hexahydroquinoline derivative **6**

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SEMI CONDUCTING CONJUGATED POLY-2,7- CARBAZOLES: SYNTHESIS, CHARACTERIZATION AND APPLICATION ON ORGANIC FIELD-EFFECT TRANSISTOR.

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[b]*Institute of Chemistry and Processes for Energy, Environment and Health (ICPEES) CNRS-University of Strasbourg, ECPM, 25, rue Becquerel, 67087 Strasbourg 02*
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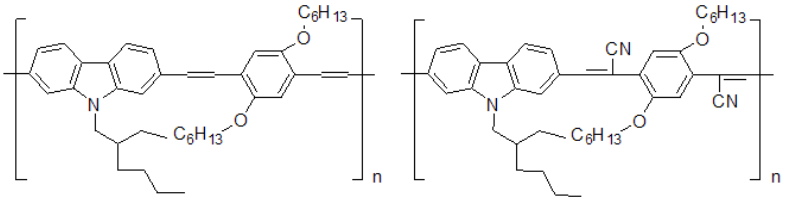
Two new light-emitting conjugated oligomers involving N-(2-ethylhexyl)-2,7-(bisformyl)carbazole and functionalized Hydroquinone moieties in the main chain, were synthesized via Wittig (P_1) and Knoevenagel (P_2) polycondensation.



Structures of the polymers P_1 and P_2

The synthesized polymers are completely soluble in common organic solvents such as tetrahydrofuran (THF) and chloroform.
The effect of cyano (CN) side groups on the physicochemical and electrical properties of the oligomeric PPV derivative was studied.
The two materials exhibit good thermostability up to 220 °C for P1 and 200 °C for P2. The UV/vis absorption and photoluminescent spectra in solution and in the film state show maximum peaks at around 328-465 and 400-570 nm, respectively.
The LUMO and HOMO energy levels of these materials were measured by cyclicvoltammetry. The hydroquinone segments are regarded as electrontransport material swhereas the carbazole units act as positive carriertransporters.
The carrier mobilities were extracted from the electrical characteristics measured in organic field-effect transistors (OFET) configuration.

Key words: Organic Semi-Conductors, Semi-conducting polymers, Carbazole, UV–Vis absorption, photoluminescence, Organic Field-Fffect Transistors (OFET), carrier mobility, Organic Electronics.

Cadmium immobilization in aqueous medium using calcium hydroxyapatite

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Calcium phosphates, especially apatites, are much studied in the removal of heavy metal ions, fluoride, nitrobenzene, protein. Their availability, ion exchange property, adsorption affinity, and their ability to establish bonds with organic molecules of different sizes, have conferred to these materials to be efficient matrixes for water purification.

The aim of this work was to investigate the efficiency of the hydroxyapatite in removal of cadmium from aqueous media by the precipitation process.

Phosphocalcium hydroxyapatite having the general formula $Ca_{10}(PO_4)_6(OH)_2$ was prepared by double decomposition in aqueous medium. The solution of diammonium hydrogen phosphate was added, drop by drop, during 90 minutes, to a boiling solution of calcium nitrate with a molar ratio of reactants Ca/P = 1.67. The precipitation pH has been adjusted to 11 by adding ammonia solution. The obtained precipitate was ignited at 800°C for 24 hours under wet Argon atmosphere. X-ray diffraction and IR spectroscopy techniques were used for the characterization and purity control of the obtained samples.

Depollution experiments consist in adding various amounts of the dissolved apatite into the contaminated water at room temperature. Incorporation of the cadmium in the apatite structure starts by the pH adjustment. The mixture was stirred for one hour. The solid was separated from the mother solution by filtration and the residual cadmium was quantified using the absorption atomic spectroscopy.

The results show that whatever the initial concentration of the pollutant, the removal of Cd^{2+} increased with increasing the apatite quantity. The optimum dose of the phosphate at the equilibrium stage decreases when the initial Cd^{2+} increases. The kinetic study shows that the equilibrium was quickly reached. The maximum of cadmium retention was observed in the first half hour of the reaction. The latter reached 2.1 moles per mole of apatite.

Key words: Water purification; Phosphocalcium hydroxyapatite; Cadmium removal.

Biological investigation of organic extracts from three Tunisian *Acacia* species

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Acacia has a wide range of ecological amplitudes and is distributed in many regions all over the world. The genus includes more than 1350 species **[1]**. In spite of the huge number of *Acacia* species, there are very few researches regarding the phytochemistry of these plants **[2]**.
Acetylcholinesterase, xanthine oxidase, 5-lipoxygenase, α-amylase inhibitory activities as well as, antioxidant and cytotoxic potentialities of various organic extracts from seeds and pods of three Tunisian species of *Acacia*, *A. cyclops*, *A. mollissima* and *A. cyanophylla*, were investigated.
At a concentration of 50 µg.mL^{-1}, EtOAc extract of seeds from *A. mollissima* and n-BuOH extract of seeds from *A.cyanophylla* were able to strongly inhibit XOD activity with 98.40% and 98.57%, respectively. Moreover, EtOAc and n-BuOH extracts from pods of *A. mollissima* showed the highest activity against the 5-lipoxygenase enzyme.
Seeds and pods extracts of *A. cyanophylla* exhibited strong activities ranging from 82.11 to 90.93% against α-amylase. However, most of the tested extracts have not been good acetylcholinesterase inhibitors.
Butanolic extracts of pods showed lowest IC_{50} in the DPPH activity indicating its highest antioxidant activity and this was confirmed also with the ABTS assays.
The twelve extracts of *Acacia* were tested for their cytotoxic activities in vitro towards the MCF-7, HCT-116, IGROV 1 and OVCAR 3 cell lines, the most active extracts were those from *A. cyclops* against IGROV 1 ranging from 12.9 to 16.1 µg.mL^{-1}.

Key words: *Acacia*, extracts, anti-acetylcholinesterase, anti-xanthine oxidase, anti-5-lipoxygenase, anti-α-amylase, antioxidnat, cytotoxic.

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AZA-MICHAEL ADDITION OF AMINES TO β-FUNCTIONALIZED DIALKYL (ALLYLOXYMETHYL)PHOSPHONATES

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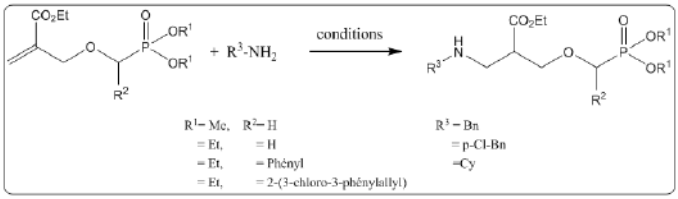
Aza-Michael addition reaction has been extensively studied and various researchers have reported the utility of this reaction towards the synthesis of various pharmacological active compounds [1] as well as natural products [2].
The conjugate addition of a nitrogen nucleophile to a α,β-unsaturated carbonyl compound leads to the formation of a β-amino carbonyl compound, which have been considered as versatile nitrogen-containing intermediates such as β-amino acids, β-amino alcohol and β-lactam. In continuation of our earlier studies [3] on acrylic derivatives we wanted to extend the aza-Michael reaction on the conjugate addition of amines to β-functionalized dialkyl (allyloxymethyl)phosphonates (scheme-1).



Scheme- 1 : Aza-Michael addition of amines to dialkyl (allyloxymethyl)phosphonates

Several tests were conducted. Aza-Michael adducts are obtained in good yields using triethylamine us catalyst in acetonitrile at room temperature. We note that, even with the use of excess of amines, bis-Michael addition product no observed.

Key words: Aza-Michael addition**,** Allyloxymethylphosphonate

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OPTICAL AND ELECTRICAL PROPERTIES OF THE MOLYBDATE $NaAl(MoO_4)_2$

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Polycrystalline powder of $NaAl(MoO_4)_2$ **[1]** was prepared by a solid state reaction. Although the material previously studied, its physical properties have not been studied. In our work, it was characterized by X-ray diffraction (XRD), Rietveld refinement, differential thermal analysis (DTA), and Fourier transform infrared (FTIR), and Raman spectroscopy. XRD patterns and Rietveld refinement showed that the powder is monophasic with a yavapaiite type monoclinic structure **[2]**. The optical properties were investigated using ultraviolet–visible (UV–Vis) absorption spectroscopy. Kubelka-Munk model and inversion method were used to determine the band gap energies of the sample. Direct band gap (3,77eV) and indirect band gap (3,55eV) derived from the diffuse reflectance measurements shows that this compound can be classified among the semiconductors materials. Electrical properties were studied using complex impedance spectroscopy in the frequency range 40 Hz – 5 MHz and temperature range (693–883 K). The impedance data were fitted to an equivalent circuit containing two elements (R // C) and (R // CPE) connected in series corresponding respectively to the contributions of grain (R and C) and grain boundaries (R_{jg} and CPE_{jg}).

Fig: The equivalent circuit allows the establishment of correlations between electrochemical system parameters and characteristic impedance elements.

Key words: Band gap, Rietveld refinement, X-ray diffraction, Equivalent circuit, Electrical properties.

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SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE MODIFIED SULFATED ZIRCONIA POWDER BY CHROMIUM AND NICKEL USING HYPERCRITICAL SOLVENT

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Solid acids are nowadays among the most important heterogeneous catalysts. Among this class, sulfated zirconia attracts the attention of many researchers because it exhibits an exceptional acidity and high catalytic properties in many reactions such as esterification [1] n-paraffin isomerization [2].
In this work, we are interested in preparing two series of nanocrystalline catalyst Cr/ ZrO_2-SO_4^{2-} and Ni/ ZrO_2-SO_4^{2-} by sol-gel route in one step, using hypercritical solvent evacuation conditions. Nickel and chromium are chosen as the dopants of sulfated zirconia for their hydrogenate-deshydrogenate properties and good reactivity in many reactions.
Interesting results have been obtained using this novel drying method in the case of this class of materials. All samples present superfine crystallites and stabilize the tetragonal ZrO_2 phase before and after heating and even at high calcination temperature. The adopted preparation method and the use of the dopant metals allow a well retention of sulfate species at high temperatures. Infrared spectroscopy shows the presence of S $\overset{\cdots}{=}$ O bond characteristic of Brønsted acid sites. Textural analysis reveals a large surface area and a homogenous mesoporosity of all aerogels (Fig 1). However, chromium confers to sulfated zirconia a more developed surface area. XPS spectroscopy shows the presence of an important amount of sulfur at the surface notably with the use of nickel. These nanocrystalline modified sulfated zirconia by chromium and nickel may be promoting heterogeneous catalysts for many reactions.

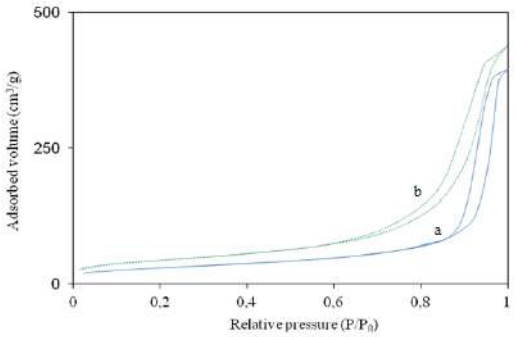

Fig 1 : Adsorption-desorption isotherm of (a) Ni/ ZrO_2-SO_4^{2-} and (b) Cr/ ZrO_2-SO_4^{2-}

Keywords: Nanocrystalline, Aerogel, Chromium, Sulfated zirconia, Nickel

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Thermodynamic assessment of the ($LiNO_3$ +$TlNO_3$) binary system

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Alkali and pseudo-alkali nitrates are used for several applications, and especially for the storage and the transfer of thermal energy. Knowledge of their phase diagrams and thermodynamic properties is important for a better use.
The phase diagram of the ($LiNO_3$+$TlNO_3$) binary system has been the subject of some previous studies leading to different experimental results [1-4]. A method which based on thermodynamic calculation allows resolving these differences. Indeed, the compilation of all the experimental data relative to pure nitrates and their binary mixtures allows studying the coherence between the thermodynamic data and the phase diagram.
We are interested in this work to optimize, for the first time, the thermodynamic parameters in the ($LiNO_3$+$TlNO_3$) binary system. The calculated phase diagram and the thermodynamic functions agree well with the experimental data.

Key words: Alkali nitrate, Phase diagrams, Calculation, Thermodynamic parameters.

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DEGUMMING OF SOYBEAN OIL BY PHYSICAL-ADSORBED PHOSPHOLIPASE A1 ONTO UNMODIFIED BENTONITE

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Traditional degumming processes cannot guarantee the achievement of low phosphorus contents required for physical refining of edible oil, the yield loss, the apparatus requirement and the energy expenditure of these processes are also great. Enzymatic oil degumming is a suitable process for the physical refining, in which a phospholipase is used to convert the nonhydratable phosphatides into hydratable form [1, 2]. However, this process utilizes an enzyme solution for degumming which is lost after a single cycle and it would be advantageous to recycle the enzyme by immobilizing it on a solid support [3]. In the present study, we investigated the ability of an immobilized phospholipase A_1 (PLA_1) in degumming soybean oil mainly by reducing phosphorus content. The enzyme was immobilized by simple adsorption onto the bentonite without any further modification. The optimal immobilization conditions and the properties of free and immobilized PLA_1 were assessed. Thereafter, the PLA_1-bentonite system was characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD) and Scanning Electron Microscopy. A notable result was that the free and immobilized PLA_1 had a high activity in neutral buffer solution (pH 7), while the temperature was found to shift to 55 °C for immobilized PLA_1. The enzyme activity was enhanced by a factor of 2 to 10 than its maximum normal activity. In terms of specific activity, PLA_1 adsorbed onto the bentonite had a specific activity equaled to 80% of its free form. The activation energies of the free and immobilized PLA_1 were estimated to 37.27 $kj.mol^{-1}$ and 4.3 $kj.mol^{-1}$, respectively. Moreover, we demonstrated that the PLA_1 adsorbed onto the bentonite was still active after being used for 4 cycles and the phosphorus content was reduced to less than 38 ppm. Thus, adsorption of PLA_1 onto the bentonite may be effective in removing phosphorus components from vegetable oil.

Key words: Immobilization, Phospholipase A1, bentonite, degumming process

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NICKEL-COBALT COATING ELECTRODEPOSITED FROM A SULFATE BATH BY PULSED CURRENTS TECHNIQUE

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The effect of pulse plating on the properties of Ni-Co alloys was presented in this study especially on the surface morphology, microstructure, composition and crystal size. An improvement in corrosion resistance and a high quality of Ni-Co coatings were detected with low content of nickel in the bath using pulse-plating mode compared to deposits produced by continuous currents [1].

The analytical characterization of Ni-Co coating was investigated by scanning electron spectroscopy (SEM) and X-ray diffraction (XRD) [2]. The corrosion performance of Ni-Co alloy coatings were evaluated in 3% NaCl solution by means of potentiodynamic polarization. The results showed that the deposits morphology was quite different. It changed from irregular deposit with imperfections obtained by continuous currents (cc), to smooth and regular surface with an average of grain size about 6 nm for the deposit obtained by pulsed currents (pc). The electrochemical measurements revealed that the protection of carbon steel substrate against corrosion was better with Ni-Co alloys obtained by the pulse electrodeposition mode than by the continuous currents coated from the same bath composition.

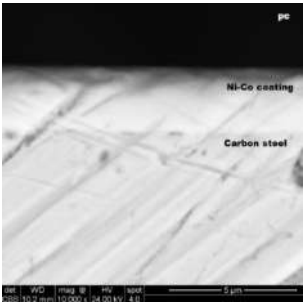

Fig. Cross-section micrographs of Ni-Co deposit in pulsed currents on carbon steel substrate.

Keywords: Ni-Co alloys, electrodeposition, continuous currents (cc), corrosion, pulsed currents (pc).

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CHEMICAL ANALYSIS AND ANTIMICROBIAL EFFECTS OF ESSENTIAL OIL FROM *ECHINOPS SPINOSUS* GROWING IN TUNISIA.

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Echinops spinosus roots (ES) are traditional and popular medicinal plant in Tunisia. In this study, the essential oil of *Echinops spinosus* roots was obtained by hydro-distillation. The essential oil yield was 1.22% (w/w) and volatil compounds were identified by GC–MS analysis. The essential oil components were alpha caryophyllene (1.522 %), caryophyllene oxide (5.217 %), thujone (0.328 %), agarospirol (1.625 %), eucalyptol (0.321 %), γ-cadinene (27.224), 5-(3-buten-1-ynyl)-2,2'-bithienyl (21.334 %) and 2,2',5',2''-terthiophene (18.024 %). A study of antibacterial activity of *Echinops spinosus* roots essential oil showed that this plant can be a source of bioactive compounds. The essential oil from *Echinops spinosus* roots has a high antimicrobial activity in front of *E.faecalis* by inhibition diameter value 15.5 mm by minimal inhibition concentration value 20µg/mL.

The characterization and the antimicrobial analysis of essential oil led to use *Echinops spinosus* as resource for pharmaceutical and food industries.

Fig. *Echinops spinosus*

Key words: *Echinops spinosus*, antimicrobial activity, essential oil.

Synthesis and Characterization of a New Cyclohexaphosphate, $(C_9H_{14}N)_4(H_3O)_2(P_6O_{18})$

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The physicochemical properties of the title new cyclohexaphosphate, $(C_9H_{14}N)_4(H_3O)_2(P_6O_{18})$ prepared by an acid/base reaction between cyclohexaphosphoric acid $H_6P_6O_{18}$ and 2,4,6-trimethylaniline, are discussed on the basis of an X-ray crystal structure investigation. The crystal structure determination reveals that the complete cyclohexaphosphate anion, with chair conformation, is generated by crystallographic inversion symmetry. The asymmetric unit consists of two organic cations, one half-anion and one hydronium cation. The atomic arrangement can be described by thick layers built by P_6O_{18} anions and H_3O^+cations parallel to the (a, b) plane. Each cyclohexaphosphate group is connected to its adjacent neighbors by six hydronium ions through strong O-H…O hydrogen bonds. The protonated amines are located between these successive inorganic layers, in order to balance the negative charge of the inorganic framework by establishing N-H…O hydrogen bonds with the O atoms of the P_6O_{18} anions. The three-dimensional cohesion is assured by different interactions (electrostatic, H-bonds, van der Waals) established between the various molecular components. This compound is also characterized by infrared spectroscopy and solid state MAS-NMR.

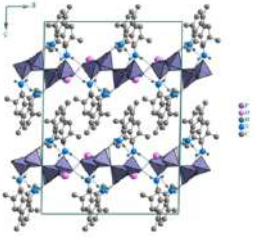

Fig. 1: The three-dimensional network of $(C_9H_{14}N)_4(H_3O)_2(P_6O_{18})$, projected along the b-axis

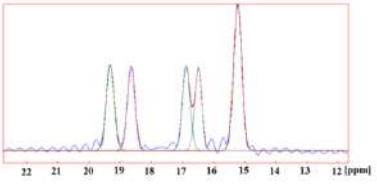

Fig. 2: Simulated and experimental signals of the aliphatic carbon resonances domain of the ^{13}C CP-MAS NMR spectrum of $(C_9H_{14}N)_4(H_3O)_2(P_6O_{18})$.

Synthesis, Structure and Physicochemical Studies of $(C_6H_7ClN)_6P_6O_{18}.0.5(H_2O)$

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Since the preparation and identification of $Li_6P_6O_{18}.6H_2O$ [1], this salt has been used as starting material to prepare other cyclohexaphosphates. Organic phosphate materials have attracted considerable interest in recent years because of their structural diversity, stability and potential uses in various fields.

A new cyclohexaphosphate, $(C_6H_7ClN)_6P_6O_{18}.0.5(H_2O)$, has been prepared at room temperature and characterized by physicochemical methods. In the atomic arrangement, the cyclohexaphosphate anions are linked to the water molecules forming inorganic layers parallel to the (a, b) plane. The organic cations are anchored between these layers and connect them via hydrogen bonds to form an infinite three-dimensional network. The structure cohesion is ensured by N-H…O, C-H…O and O-H…O hydrogen bonds. The number of ^{13}C ^{15}N and ^{31}P CP–MAS NMR lines is in full agreement with the crystallographic data. DFT calculations allow the attribution of the experimental NMR lines. The vibrational absorption bands were identified by infrared spectroscopy.

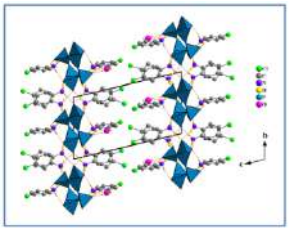

Fig. 1: Projection of the structure of $(C_6H_7ClN)_6P_6O_{18}.0.5(H_2O)$ along the a-axis

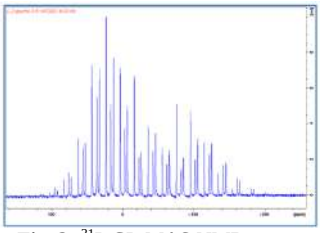

Fig. 2: ^{31}P CP-MAS NMR spectrum of $(C_6H_7ClN)_6P_6O_{18}.0.5(H_2O)$.

Reference

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Effect of Severe Plastic Deformation on the Strain Hardening of AA6060-T6

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The aim of this work was to study the effect of severe plastic deformation imparted by equal channel angular pressing (ECAP) on the strain hardening capability of AA 6060-T6. Cylindrical samples of AA6060-T6 (10 mm in diameter, 60 mm in length) were subjected to ECAP deformation using a Φ=90º and ψ= 37° die for up to 12 passes following route Bc. The mechanical properties of the deformed material were measured after each pass by hardness and tensile tests. The Hollomon analysis was used to investigate the strain hardening behavior of AA6060-T6 samples. All deformed samples showed lower strain hardening exponent in comparison with the as received. Moreover, the strain hardening rate increased with increasing number of passes. The original grain size of the as-received AA6060-T6 of about 70 μm was drastically reduced after ECAP, reaching grain sizes close to 500 nm after 12 passes.

Keywords: Equal channel angular pressing (ECAP), AA6060-T6, Microstructure, Mechanical properties, Strain hardening behavior.

Preparation and characterized of inexpensive and easily available Tunisian heterogeneous catalysts Al-Fe-Pillared clays

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The placement of metal oxide pillars between clay mineral layers modifies their physical chemical properties, including surface area, acidity, and catalytic activity. Aluminum is the most commonly used pillar cation, but the use of Fe offers a distinct opportunity to expand the range of catalytic behavior.

The purpose of this study was to prepare Al-Fe-pillared smectite and to characterize the resulting materials.

Al-Fe-pillared clay was synthesized from Tunisian clay precursors according to a common procedure: grinding, sieving, Na exchange, pillaring, drying and calcinations. Smectite suspension was mixed with different pillaring solutions containing Al and Fe oligomers with Fe/(Al+Fe) percent ratios: 1; 5; 10 and 50%. The structural and textural properties of synthesis catalysis have been determined by X-ray diffraction, nitrogen adsorption-desorption isotherms, Transmission Electron Microscopy (MET), X-ray fluorescence, CEC and infrared spectroscopy.

XR diffraction showed an increase in the basal spacing up to 19A°. They have a high thermal stability. Textural characterization show a decrease in BET surface area indicating that a portion of the iron inserted into the clay structure is deposited on the surface, interacting with the tetrahedral sheets of the clay, without destroying the original framework. That change in the amount of iron on the surface of the material leads to a substantial increase in the adsorption and catalysis activity due to changes in the structure of surface sites available for the reaction to take place.

Key words: clay, Al-Fe pillared clays, oligomers, catalysts.

Inexpensive and easily available Tunisian natural's heterogeneous catalysts were prepared and characterized: Al-Fe-Pillared clays

Selma Khelifi, Fadhila Ayari, Dalila Ben Hassan Chehimi

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Key words: clay, Al-Fe pillared clays, oligomers, catalysts.

SYNTHESIS, CHARACTERIZATION AND ANTIBACTERIAL ACTIVITIES OF TRANSITION METAL COMPLEXES WITH ISONICOTINIC HYDRAZIDE AND 2- (PHENYLCARBAMOYL) BENZOHYDRAZIDE.

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This work concerned the study of the reactivity of the metal complexes of Co(II), Ni(II), Cu(II) and Zn(II) with respect to two ligands therapeutic in particular the isonicotinohydrazide and the 2-(Phenylcarbamoyl)benzohydrazide.This complex were isolated with the state from powder by adopting three methods of synthesis, in fact, the traditional synthesis (with backward flow), the synthesis by microwave and the last synthesis hydrothermal. They were characterized by methods of analysis such as gravimetric, the IR spectroscopy and spectroscopy UV-VISIBLE. The two ligands were revealed to act like agents bidentate chelating [1]. The antibacterial activity of the ligands and their complexes was evaluated *in-vitro* against two bacteria pathogenic (Escherichia Coli and staphylococcus aureaus) by the method of diffusion on agar. The able antibacterial of the compounds is relatively weak for the bacterial stock with gram negative (E.coli) [2]. On the other hand an inhibition is notable for the bacterial stock with gram positive (S.aureus) with the various concentrations (50mg/ml, 100 mg/ml).

Key words: Isonicotinohydrazide, 2-(Phenylcarbamoyl)benzohydrazide, Metal omplexes, Antibacterial Activity.

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Synthesis characterization and antimicrobial activity studies of some transition metal complexes from Aspirin and Paracetamol.

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Novel complexes of Fe (I11), Cu (11), Co(II) and Zn (11) with two therapeutic ligands (paracetamol and aspirin) have synthesized by hydrothermal process.

The obtained compounds were thoroughly characterized by elemental analyses, Infrared spectroscopy and nuclear magnetic resonances (^{1}H and ^{13}C). The two ligands have been found to act as bidentate chelating agents. Aspirin complexes coordinate through the carbonyl oxygen of the carboxyl and the ester groups, while paracetamol complexes coordinate through the oxygen of the hydroxyl and the amide groups.

Antibacterial screening of the complexes against *Bacillus substilis, Serratia species and Escherichia coli*, was also investigated. The metal complexes were found to have varied degree of inhibitory effect against the bacteria.

Key words Aspirin, Paracetamol, Metal Complexes, Antibacterial Activity.

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Structural, Elastic and thermodynamic Properties of Perovskites $AHfO_3$ (A=Ba, Ca, Pb And Sr) From First Principles Calculations.

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The structural and elastic constants of series of hafnium perovskites oxides are studied by first principles approach, where cation A is systematically varied in order to see its effect on elastic, mechanical properties and Debye temperature. The calculated elastic constants of $AHfO_3$ (A=Ba, Ca, Pb, and Sr) using the pseudopotentiel method with local density approximation (LDA) are in good agreement with the available data in the literature. The discussions of the changes of the elastic constants with respect to the cation sizes are given based on the nature of the crystal structure. Details and explanations for such dependencies are discussed.

Keywords: Perovskite, ab initio calculation, elastic properties, thermodynamic properties.

FRUITS OF *RUTA CHALEPENSIS* AS A SOURCE OF KETONES AND ESTERS

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Kossentini Mohamed[a], and Zouari Sami[a].

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The aim of the present work is to provide more information on the variation of essential oil composition of *Ruta chalepensis* collected from the South East of Tunisia (Sdira and Thoujene) at the fruiting stage. Essential oils were extracted from the leaves, stems, and fruits by hydrodistillation and analyzed by gas chromatography–flame ionization detector (GC–FID) and GC–mass spectrometry (GC–MS) techniques. Nineteen compounds were identified from the different plant organs, where ketones and esters constituted the main chemical classes. The most abundant compounds in the leaves and stems essential oils were 2-undecanone (23.0–37.7%), 2-nonanone (16.7–23.0%) and octyl acetate (11.0–26.8%), while 2-undecanone (57.5–58.4%) and 2-nonanone (19.0–23.3%) were found to be major compounds in the fruits essential oil.

Key words: *Ruta chalepensis*, ketone, ester, 2-undecanone, 2-nonanone.

Equal channel angular pressing technique for the formation of ultra-fine grained structures in Fe-48%Ni

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The evolution of the microstructure and the mechanical response of a Fe-48%Ni alloy submitted to severe plastic deformation using equal channel angular pressing (ECAP) was investigated. A stacking of 9 samples was introduced and pressed in the die with an inner angles of Φ=90º from 1 to 2 passes at room temperature following route A. The present study will be concentrated on analyzing the initial state (N=0), the peripheral plates and the central plate of the stack after the two passes of ECAP. The microstructure, misorientation boundaries and texture were evaluated by means of X-ray diffraction (XRD) and electron backscattered diffraction (EBSD). During the first ECAP pass, the microstructure developed a large fraction of low angle boundaries associated with subgrain formation. A heterogeneous ultra-fine grain structure was obtained in different samples in each pass. Microhardness test was used to characterize mechanical properties. Significant increase in hardness was observed from the first pass.

Keywords: Equal channel angular pressing (ECAP), Fe-Ni alloy, Electron backscattered diffraction (EBSD); Microstructure.

SYNTHESIS, CRYSTAL STRUCTURE, SPECTROSCOPIC, THERMAL AND DIELECTRIC PROPERTIES OF A NOVEL SEMI-ORGANIC PENTACHLOROANTIMONATE (III)

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A new organic-inorganic hybrid material of formula $(C_{10}H_{15}N_2F)_5(SbCl_5)_5.2H_2O$ was synthesized and characterized by X-Ray diffraction analysis. It crystallizes in the monoclinic space group $P2_1/c$ with the following unit cell parameters a=15.819(4) Å, b=17.685(3) Å, c=30.529(4) Å, Z=4 and V=8540(3) $Å^3$. The examination of the structure shows that the three-dimensional frameworks are produced by N–H…Cl, N–H…O, C-H…Cl and N-H…F, C-H…F hydrogen bonding and Cl…Cl interactions. IR, Raman and UV-Visible spectroscopies were also used to characterize this compound. In addition, the fluorescent properties of this compound have been investigated in the liquid state at room temperature. Differential scanning calorimetry (DSC) has revealed a structural phase transition of the order-disorder type around 370 K. Dielectric investigations revealed a step-wise change of the electric permittivity at Ttr characteristic of the crystal in the high-temperature phase. The evolution of dielectric constant as a function of temperature of the sample has been investigated in order to determine some related parameters. Measurements of AC conductivity as a function of frequency at different temperatures indicated a hopping conduction mechanism and/or reorientational motion.

Keywords: Complex compound, Crystal structure, Spectroscopies, Dielectric properties, Phase transition

SYNTHESIS OF CHALCONES VIA CLAISEN–SCHMIDT CONDENSATION REACTION CATALYZED BY SILICA-H_2SO_4 UNDER ULTRASOUND IRRADIATION

Achraf Lahyani, Manef Chtourou, Mohamed Hédi Frikha, Mahmoud Trabelsi

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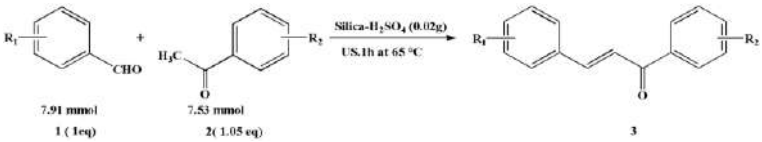
The synthesis of 1,3-diaryl-2-propenones was carried out by using ultrasonic irradiation in the presence of silica-H_2SO_4, which was used as a catalyst in solvent-free conditions. The advantages of this process are the high yields (82–98%) it produces, the cost effectiveness of the catalyst, and the simple work-up and purification of products achieved via a non-chromatographic method.

Key words: Trans-chalcones; Silica-H_2SO_4; Solvent-free; Ultrasound irradiation; Claisen-Schmidt condensation.



PERFORMANCE EVALUATION OF ELECTRODEIONIZATION PROCESS

Aicha Lakehal[a], Salima Lakehal[b,c]

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Electrodeionization (EDI) is being applied more and more to produce ultrapure water [1-6], especially in the semiconductor industry. The continuous electrical regeneration of the ion-exchange (IEX) mixed bed is the main advantage of this recent technology. EDI couples two well known effects: electrodialysis and IEX. In spite of its rapid development, there are no established theories, design equations nor clear mechanisms of regeneration and transport [1,2,5]. The present research work deals with Electrodeionization (EDI) process efficiency. We have investigated the influence of the applied voltage V, the flow rate Q, the concentration C and the salt nature on cell efficiency by measuring current intensity I, and inlet and outlet concentrations of the treated solution.
The main finding was that an original, empirical, and simple equation between the mass transfer flux J and the flow rate Q is established: $J = K\,Q^{n} \approx K\,Q^{0.5}$
This remarkable equation is obtained with various examined salts. This is an important result because it presents strong analogies with the habitual equations of electrochemical hydrodynamics. The other interesting results are: the Nernst-Planck equation is globally respected and there is an optimal working voltage. At higher voltage, the concentration polarization is easily obtained. We observe water dissociation and neat decrease in efficiency. This problem in EDI may be easily solved by the addition of sacrificial amphoteric salts, which improve seriously the efficiency.

Key words: Electrodeionization, water, ion-exchange

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THEORETICAL INVESTIGATION ON ORGANOLANTHANIDE COMPLEXES

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The chemistry of lanthanide has grown rapidly in recent years and along with this an improved understanding of lanthanide-nitrogen bonding. The lanthanide amidinates and guanidinates comprise a relatively new class of rare earth metal complexes containing *N*-chelating ligands [1]. Guanidinate anions $[(RN)_2CNR_2]^-$, which present an interesting ligand and receive considerable attention as supporting ligands in organometallic chemistry. So understanding the bonding between the lanthanide and these ligands is important to better understand the structures, properties and chemical reactivity of lanthanide compounds [2].
Compared to the number of experimental studies, the number of theoretical studies for organolanthanide guanidinate complexes is limited. Considering the importance of these compounds, we carried out a DFT (density functional theory) calculations on this type of compounds that were synthesized by Zhang and coworkers. The results were compared with the X-ray structures that were available from the literature; the calculations reproduced quite well the experimental structural features in these complexes exhibiting distorted tetrahedron geometry. The calculated evolution of the Ln-guan bond as a function of the cation showed that lanthanide-ligand distances increased with the increase of the ionic radius; but for lanthanide-guan Hirschfield charge Cp_2Dy-guan made the exception where we found that the bond in this complex was the most ionic.

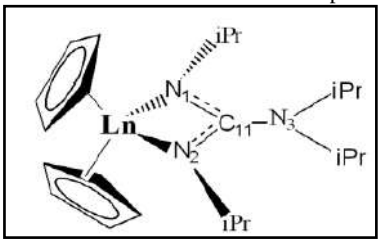

Fig. The studied systemsLn= Lu, Yb, Y, Dy et Gd

Key words: DFT, guanidinate, lanthanide

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PHOTOVOLTAIC CELLS BASED ON PHTHALOCYANINE WITH DIFFERENTS STRUCTURES

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This paper presents the theoretical investigation of electronic and optical properties for organo-metallic systems donor-acceptor formed phthalocyanine with different metals (Fe, Ni, Co, Cu, Na, Mn). The geometric and electronic properties of different systems in their fundamental state are studied using functional theory of density (DFT), while the optical characteristics obtained by the functional theory of dependent density time (TDDFT). The software Amsterdam Density Functional (ADF Version 2014) is used for this study. The present results indicate that high conversion efficiency would be expected when phthalocyanine dimer were used as solar cell materials.

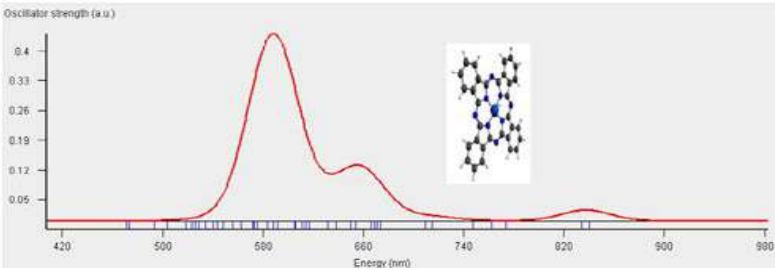

Fig. absorption spectrum for Co Pc

Key words: DFT, absorption, phthalocyanine.

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EFFICIENT AND GREEN SYNTHESES OF NOVEL FUNCTIONALIZED γ -PHOSPHONYLOXIMES

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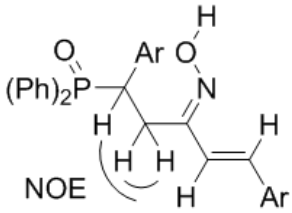
Oxime have been frequently used in organic synthesis as a key intermediate not only in the preparation of natural products such as erythromycin derivatives[1a] and perhydrohistrionicotoxin[1b] but also in agrochemicals,[2a] medicinal chemistry,[2b] toxicology,[2c,d] and mainly in the preparation of cephalosporin derivatives known with their potent antibacterial activity.[2e] With this in mind, and in the continuation of our interest to develop efficient protocols for the synthesis of biologically active phosphonates [3], we report herein a simple and green methodology for the synthesis of novel γ-phosphonyloximes functionalized. The stereochemistry (E,Z) assigment of derivatives was based on ^{31}P NMR spectrum and NOE experiments, irradiating the methylene protons.



Key words: Phosphonates, Phosphines oxides, Oxime, Isomer, NMR

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Catalytic wet air oxidation of para-hydroxybenzoic acid by γ-Al_2O_3 and Ce/γ-Al_2O_3

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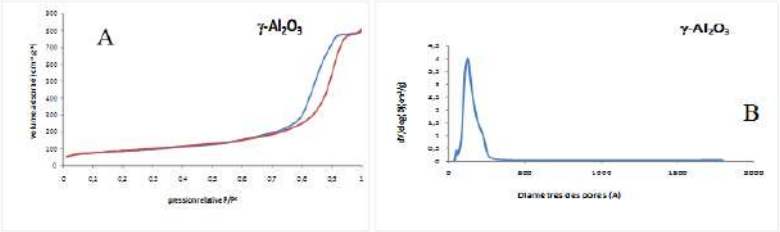
This work focuses on the catalytic wet air oxidation (CWAO) of para-hydroxybenzoic acid p-HBZ, in aqueous media, using catalysts based on aluminum oxide synthesized by a sol gel. The modification of the catalysts by adding cerium seems like a good alternative to improve the catalytic performance of the synthesized catalysts. The textural properties of matrices synthesized based on alumina shows the isothermal adsorption-desorption of nitrogen at 77 K and the porous material distributions (Figures A and B). Isotherms γ-Al2O3 is of type (IV) having a hysteresis loop of the form H1 often obtained with rigid spherical particles of uniform sizes stacks [1]. Treating p-HBZ acid by oxidation in the catalytic wet air (CWAO) was studied under pressure of 50 bars of air and at 140°C. The catalytic performances of catalysts developed γ-Al2O3 and Ce/ γ-Al2O3 were examined and compared to the CWAO tests of para-hydroxybenzoic acid. The catalysts prepared seem to be active with a conversion of 35% for γ-Al2O3 and a better conversion for Ce/ γ-Al2O3.



Figures (A) adsorption-desorption of nitrogen at 77K and (B) porous Distributions g-Al_2O_3

Keywords: Aluminum oxide Al_2O_3; CWAO ; parahydroxybenzoic acid.

Référence

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IDENTIFICATION OF SMALL MOLECULE AS α-GLUCOSIDASE INHIBITORS

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Glucosidases are largely involved in the human metabolic system. Thus, glucosidase inhibitions have many potential therapeutic applications and have been implicated in several diseases such as diabetes[1], cancers [2] and Gaucher's disease[3]. One special category of potent glucosidase inhibitor is naturally occurring and synthesized azasugars such as deoxynojirimycin, acarbose, voglibose and miglitol[4].
The objective of the present work was to identify molecules as potent α-glucosidase inhibitors.
Further, in silico screening of the database was carried out by applying the Lipinski rule of five and the hits obtained were then subjected to virtual screening by docking, to obtain the final hits potentially with potent α-glucosidase inhibition activity. The best hits obtained from the docking studies were subjected to the adsorption, distribution, metabolism, and excretion (ADME) study to compare the drug-likeness properties with standard drugs.
The screening resulted in 3 hits with XP > 9.753, these hits may be further developed as novel potential and selective inhibitors.

Key words: α-glucosidase, Inhibitors, Virtual screening, Docking, Diabetes.

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ASSESSMENT OF POSSIBLE EFFICACY OF AQUEOUS LEAVES EXTRACT OF *PSORALEA BITUMINOSA* L. FOR ANTIHYPERGLYCAEMIC ACTIVITY

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Psoralea bituminosa L. (C.H. Stirton, Fabaceae, Psoraleeae; Stirton 1981) (*P. bituminosa*) (syn. *Bituminaria bituminosa*L.), commonly known as the Arabian pea or pitch trefoil, is a perennial herb species widely distributed in the Mediterranean region. It has been used as animal feed especially for dairy goats[1]. Moreover, the plant is a rich source of secondary metabolites with considerable pharmacologic properties. *P.bituminosa* is known for producing furanocoumarins such as psoralen and angelicin used in the treatment of skin diseases[2,3], pterocarpans such as bitucarpin A and B with antitumor activity against colon cancer[4,5].

In the north-west region of Algeria (Tlemcen), some population is found to use leaf decoction of *P. bituminosa* to treat diabetes, but up to now, there are no scientific data available for its antidiabetic effect.

The aim of this study was to assess for a possible antihyperglycaemic activity of aqueous leaves extract of *P. bituminosa* both in normal and streptozotocin (STZ)-induced diabetic rats.

The aqueous extract was screened for its phytochemicals and revealed the presence of tannins, alkaloids, flavonoids, anthocyans, terpenes and sterols.

The results of acute toxicity showed that the rats had good tolerance to high doses of extract (up to 1.5 g/kg) and no mortality was observed. The extract has shown a good blood glucose lowering effect in the oral glucose tolerance test. After 21 days of daily oral administration of the extract to streptozotocin induced diabetic rats, the aqueous extract can reduce hyperglycemia reaching over more than 31%.

Keywords
Diabetes mellitus, *Psoralea bituminosa* L., Aqueous extract, Phytochemical, Hyperglycaemia..

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MOBILITY OF N^+IONS IN A HELIUM GAS BY A QUANTAL TREATMENT OF THE CROSS SECTIONS

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[b)] *Badji Mokhtar University, Annaba, Algeria (b)*
[c)] *Mohamed-Cherif Messaadia University, Souk Ahras, Algeria (c)*

This work follows recent experimental measurements of low-temperature mobility of nitrogen ions N^+ in helium performed at Tokyo Metropolitan University by the group of Pr Tanuma. More specifically, this work tries to determine theoretically the mobility in He ($1s^2$) of ground and excited N^+ ions at low temperatures: T=4.3 and 77 K. Initially, the mobilities are determined with quantum-mechanical transport cross sections without the spin-orbit effects. Then, these effects are introduced in the computation of the potential-energy curves that dissociate asymptotically into the interactions $N^+(^3P)$+He and $N^+(^1D)$+He. This PhD project will mainly focus on the behavior of the reduced mobility with the ratio E/N of the electric field and the gas density and try to explain its observed decrease near the value E/N~6Td.

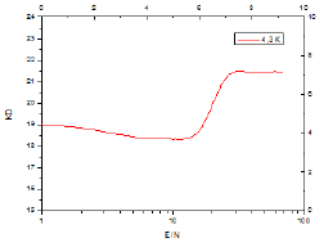

Fig. Mobility of the N^+ (3S) and N^+ (3P) ions in to He as a function of E/N at low temperatures T= 4.3K

Key words: Chapman-Enskog model; Potential-energy curves; Transport cross sections; Three-temperature theory; Mobility and diffusion coefficients.

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STUDY OF BISPHENOL-A MIGRATION FROM POLYCARBONATE BABY BOTTLES BY SOLID PHASE EXTRACTION AND GAS CHROMATOGRAPHY-MASS SPECTROMETRY

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The films and plastic containers are widely used in the food industry due to their flexibility, varying sizes and shapes, relatively light weight, their stability and low production cost [1]. Bisphenol-A (BPA) based polymers are among the most used plastic resins as packaging materials [2]. In this study, we have developed an extraction method of BPA leaching from baby bottles to food contents, using water as aliment simulant. Determination of migrant BPA was conducted by Solid Phase Extraction (SPE) followed by N-Methyl-N-(trimethylsilyl) trifluoroacetamide (MSTFA) derivatization and gas chromatography coupled to mass spectrometry (GC-MS) analysis. The linearity was verified between 0.05 and 5 mg L^{-1} (R^2> 0.998). The limit of detection and limit of quantification were estimated at 0.006 mg L^{-1} and 0.02 mg L^{-1}, respectively. The repeatability of the derivatization method has been proven. The effects of temperature and usury on the migration of BPA were studied

Key words: Bisphenol-A, Solid phase extraction (SPE), GC-MS, baby bottles.

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Study of adsorption of Brilliant Green by modified clays

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Organophilic clays synthesized using two surfactants; hexadecyltrimethylammonium bromide (HDTMA) and HDPyridine (HDPy) were introduced into Tunisian pure clay at various concentrations (from 1 to 3 times the cationic exchange capacity). The results of obtained samples show clearly the intercalation of surfactants in clay and this can be observed particularly by a significant increase in its basal distance.
The adsorption phenomenon of dye textile Brilliant Green in aqueous solution by modified clays has been studied as a function of various parameters, either the stirring time, the concentrations of adsorbents balance in water, the pH and temperature.

Keywords: organophilic clay, surfactant, HDTMA, HDPy, intercalation, dye textile, adsorption.

Sythesis of 1,5 bis-propargyloxy-sulfide

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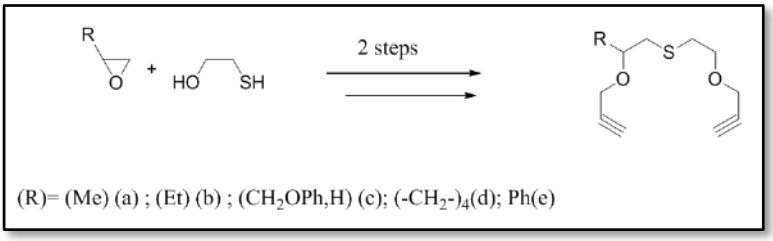
Propargyl ethers are important starting materials for a wide range of organic reactions[1]. The reactivity of alkyne allows them to be used in many areas such as coordination chemistry[2] polimers[3] The most commonly used method is Williamson synthesis for the preparation of propargyl ethers, this type of ether synthesis which involves the use of strongly basic metal alkoxide anions and highly active propargyl halides. Herein, we report the synthesis of novel 1,5 bis-propargyloxy-sulfide substituted obtained starting from the products of opening of epoxides by the mercaptoéthanol[4] (Scheme 1).



Scheme 1: Sythesis of 1,5 bis-propargyloxy-sulfide

Key words: epoxide, mercaptoethanol, propargyl bromide.

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Synthesis and Characterization of some novel bis(1,3,4)-Oxadiazole

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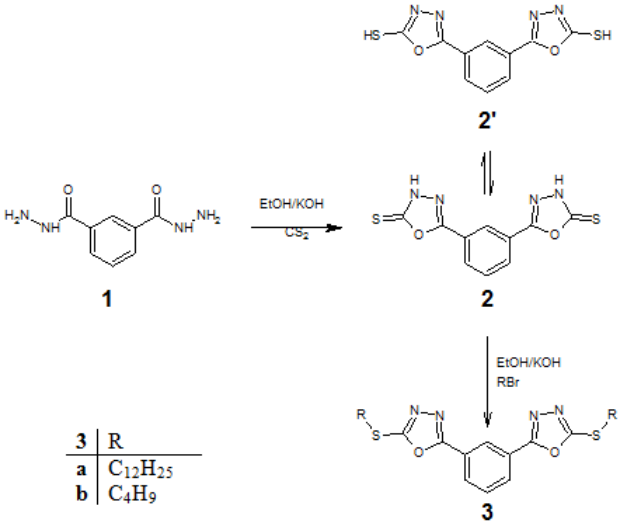
During the last decades, a number of oxadiazole derivatives have been synthesized and tested for their biological activities [1-4]. Starting from isophthalic dihydrazide **1,** we describe in this work the synthesis and characterization of a series of 1,3-bis(1,3,4-oxadiazole-2-dialkylthiol)benzene derivatives **3**. These newly synthesized compounds were characterized by conventional spectroscopic techniques. IR spectra indicated that, among the two expected tautomers **2** and **2'** of the intermediate**,** the "thione" form **2** is predominant.



3	R
a	$C_{12}H_{25}$
b	C_4H_9

1 **2** **2'** **3**

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Synthesis and crystal structure of 4-(2-ammonioethyl) morpholin-4-ium dichloridodiiodidocadmate/ chloridotriiodidocadmate (0.90/0.10)

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The crystal structure of the title compound, $(C_6H_{16}N_2O)[CdCl_{1.90}I_{2.10}]$, a new organic–inorganic hybrid salt synthesized in the form of single crystals, consists of discrete statistically distributed dichloridodiiodidocadmate/chloridotriiodidocadmate anions (occupancy ratio 0.90:0.10) and 4-(2-ammonioethyl)morpholin- 4-ium cations, [NH3(CH2)2NH(CH2)4O]2+. The cations are linked by intermolecular N—H…O hydrogen bonds, forming corrugated chains extending parallel to the c axis. The $[CdCl1.90I2.10]^{2-}$ tetrahalidocadmate anions lie between the chains to maximize the electrostatic interactions and are connected with the organic cations via N—H…Cl and C—H…Cl(I) hydrogen bonds developing in the ab plane and leading to the formation of a three-dimensional network structure. The tetracoordinate Cd(II) atom has a distorted tetrahedral conformation, with a τ_4 index of 0.87.

SYNTHESIS OF FULLERENE NANOTUBES

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Fullerene nanowhiskers are the thin crystalline fibers that are composed of fullerene molecules with diameters less than 1 μm, and fullerene nanotubes (FNTs) are the tubular fullerene nanowhiskers. The FNTs will find various applications in the field of transistors, solar cells, catalysts, chemical synthesis templates, MEMS devices, and so forth in future.
C_{60}NTs were prepared by the LLIP method [1,2]. In preparing C60NTs, pyridine was used as a good solvent of C60, and isopropyl alcohol (IPA) was used as a poor solvent of C_{60}. Fullerene powder (99.5% pure, MTR Ltd.) are dissolved in pyridine and this solution was ultrasonicated to obtain a C_{60}-saturated pyridine solution. The undissolved C_{60} powder was removed by syringe filter (PUREDISC 25TF/0.45μm PTFE) and this C60-saturated pyridine solution was irradiated with UV light and stored in an incubator. Pyridine solution saturated with C_{60} was put into a transparent glass bottle and isopropyl alcohol was gently added to the C_{60}-saturated pyridine solution. After, shaking the bottle, it was stored in an incubator at 10°C for 2 hours. The synthesized C60NTs were vacuum-dried at 120°C.

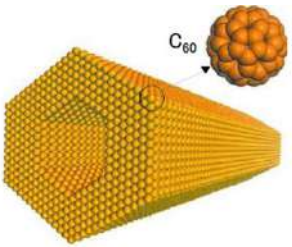

Fig. model of a fullerene nanotube composed of C_{60} molecules.

Key words: fullerene nanotubes, water, solvent ration, temperature, light.

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σ-omplexation reaction of 2,6-dimethoxy-3,5-dinitropyridine with para-X-phenoxide anions in aqueous solution: Structure-Reactivity and Mechanism

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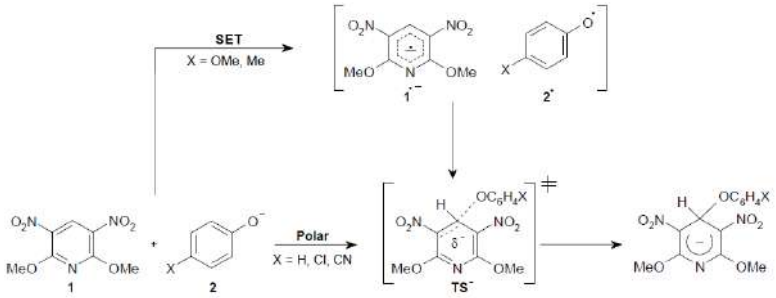
In this communication, we have presented our study to the σ-complexation reaction of 2,6-dimethoxy-3,5-dinitropyridine **1** with para-X-substituted phenoxide anions (X = OMe, Me, H, Cl, CN) **2** in aqueous solutions at 20 °C. During this study the applicabilities of various structure-reactivity correlations were discussed and found some importing results. The effect of phenoxide substituted on the reaction rate constant was examined, leading to nonlinear Hammett correlation with large negative ρ values for the phenoxide anions possessing an electrodonating group. A single electron transfer mechanism is suggested.



Possible mechanistic pathway for the reactivity of 2,6-dimethoxy-3,5-dinitropyridine **1** with para-X-substitutent phenoxide anions **2** in aqueous solution.

Key words: 2,6-dimethoxy-3,5-dinitropyridine, σ-complexation, single electron transfer, kinetics.

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LAYER-BY-LAYER METHOD FOR THE GROWTH OF SURFACE MOUNTED METAL-ORGANIC FRAMEWORKS

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Metal organic frameworks (MOFs) are a class of crystalline materials consisting of metal ions or clusters coordinated to multidentate organic linkers to form one-, two-, or three-dimensional structures. This new family of materials has attracted considerable interest because of its vast applications in gas storage [1], catalysis [2] and drug delivery [3]. The deposition of these coordination polymers as thin films onto conducting substrate will be a new direction for the elaboration of modified electrodes for electrocatalysis and for sensor development [4]. These films can be prepared with many different ways. Layer by layer deposition is one of the powerful techniques to prepare thin films [5]. In this work, we report the synthesis of functionalized coordination polymer (MOF-5). The obtained product was characterized by infrared spectra; thermal gravimetric analysis and zeta potential measurements. Due to its charged surface, thin films of MOF-5 are successfully deposited on indium tin oxide electrode using layer by layer deposition. The films were monitored by Fourier X-ray diffraction (XRD) and optical microscopy. The described method is a feasible way for surface modification by MOF's thin films.

Key words: Coordination polymers, MOFs, layer by layer deposition

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Analysis of Residues Polychlorinated Biphenyles (PCB) and Organochlorine pesticides (POC) in treated waste water by gaz chromatography (GC-ECD)

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This study is interested to the developpement of a new a protocol in order to analyze the residue of 9 congeners of polychlorinated biphenyles (PCB) and 6 organochlorine pesticides (POC) in treated waste water by gaz chromatography (CPG-ECD) [1]. The samples were collected from 3 sites in Tunisia. This study has allowed us to became familiar with several technics and methodologies of analysis of organic compound.

The protocol used for the treatment of water is solid phase extraction (SPE). It's easy to applied and gived a good recoveries between 70 and 94%.

Levels of PCB and POC measured in samples shown a general contamination with a variable concentration. The highest concentration for highly chlorinated congeners was in relation to their high bioaccumulation [2].

Key words: PCB, POC, SPE, GC-ECD.

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ANION COMPLEXATION BY AMIDO DERIVATIVES OF P-TERT-BUTYL CALIX[4]ARENE

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[b)] *Chemistry Department, Faculty of Sciences and Arts at El-Rass, Qassim University, Kingdom of SAUDI ARABIA*

The complexation of anions by artificial receptors and the search for anionic recognition by specific receptors is developed in the field of supramolecular chemistry.

We recently reported ^{1}H NMR studies of the complexation of selected anions (spherical halides, planar trigonal nitrate and tetrahedral hydrogen sulphate) by a series of amido derivatives of p-tert-butyl calix[4]arenes (organic-based type) and more particularly. The stability constants, log β_{ij}, varying from 0.94 to 2.05 were determined for the mononuclear complexes. Interpretation of all spectroscopic data obtained by the computer program "ChemEqui for Calculations" led to the determination of the apparent stability constants logβ_{ij} of the formed complexes in $CDCl_3$ and the type of dissolved species.[1]

We present a subsequent study on the complexation of F^-, Cl^-, Br^-, I^-, NO_3^- et HSO_4^- by the di(2-pyridylmethyl) amide calix[4]arene **L1**, tetra(2-pyridylmethyl) amide calix[4]arene **L2**, tetra(2-pyridylethyl) amide calix[4]arene **L3** in order to evidence the influence of the residues attached to the calix unit by amido functions.

Fig. Chemical structural of derivatives L1-L3.

Keywords: Calixarenes, Anion complexation, ^{1}H NMR.

ISOLATION AND CHARACTERIZATION OF BIOMENERALS FROM BARBARY FIG PADS

Faten Mannai[a], Hanadi Elhleli[a], Mohamed Ammar[a],
Elimame Elaloui[a,b], Younes Moussaoui[b,c]

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This work aimed to study the presence of biomenerals "calcium oxalates" in Barbary fig or *Opuntia ficus-indica.* This xerophyte plant was characterized by the presence of whewellite [1]. Crystals are isolated from Barbary fig pads in alkaline solution. The structure and the morphological properties of isolated crystals were studied with scanning electron microscopy (SEM) coupled by spectroscopy dispersion of X-ray energy (SEM-EDS). Infrared spectroscopy was used to analyze the chemical composition of crystalline materials. X-Ray diffraction analysis was investigated to identify the crystalline nature of crystals. The thermal stability of isolated biomenerals was performed by thermogravimetric analysis (TGA). The results show that calcium oxalate was characterized by high thermal stability.

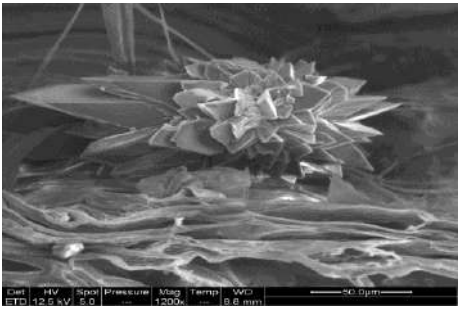

Fig.1. Calcium oxalate crystal in the Cactus-fibres [1].

Key words: *Opuntia ficus-indica*, biomenerals, calcium oxalates, morphological properties, thermal analysis.

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CRYSTAL STRUCTURE OF 2,5-DIMETHYLANILINIUM HYDROGEN MALEATE

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The crystal structure of the title salt, $C_8H_{12}N^+.C_4H_3O_4^-$, consists of a 2,5-dimethylanilinium cation and an hydrogen maleate anion. In the anion, a strong intramolecular O—H…O hydrogen bond is observed, leading to an S(7) graphset motif. In the crystal, the cations and anions pack in alternating layers parallel to (001). The ammonium group undergoes intermolecular N—H…O hydrogen-bonding interactions with the O atoms, of three different hydrogen maleate anions. This results in the formation of ribbons extending parallel to [010] with hydrogen-bonding motifs of the types $R_4^4(12)$ and $R_4^4(18)$.

Key words: crystal structure, 2,5-dimethylanilinium cation, maleate anion, hydrogen bonding.

SYNTHESIS OF NEW BENZOFURAN DERIVATIVES FROM FUNCTIONALIZED TETRAHYDRONAPHTHALENES

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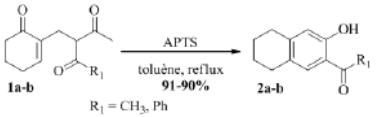
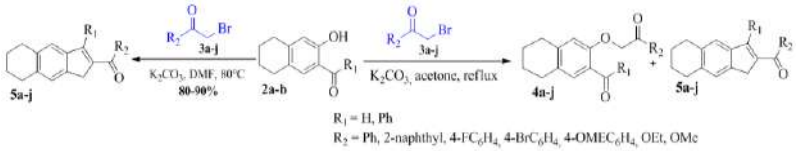
Benzofuran derivatives are nowadays an important class of organic compounds that occur in a great number of natural products,[1] and used in synthetic pharmaceuticals. Many of the natural benzo[b]furans have physiological, pharmacological, and toxic properties, and therefore there is continuing interest in their chemical synthesis.[2]

As part of the synthesis of novel heterocyclic compounds exhibiting pharmacological interest, we have developed an efficient a *one-pot* synthesis of a new series of benzofuran derivatives **4a-j** using functionalized tetrahydronaphthalenes (THN) **2a-b** that were previously prepared through a Robinson annulation of the δ-diketones **1a-b**.[3, 4]



The reaction of THN **2a-b** and bromoacetones **3a-j**, under the influence of K_2CO_3, in dry acetone at reflux, gave the *O*-alkylation products **4a-j** along with the benzofuran derivative **5a-j**.[5] Interestingly, under the previous conditions but in DMF at 80°C, the title reaction provided exclusively the benzofuran derivatives **5a-j** within 2 h in high yields (80-90%).[6]



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Study of the treatment of wastewater containing fluoride by neutralization with hydrated lime

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Aluminum fluoride industries produce aluminum fluoride (AlF_3). This product reduced greatly of CO_2 emission in the aluminum processing industry. However, aluminum fluoride industry rejects water effluent in the marine environment. This liquid discharge is relatively rich in fluoride. It is necessary to treat this effluent in order to respect the environmental standards before discharge in the sea. Several techniques have been developed for the treatment of fluoride effluents. A physicochemical process associated to a membrane treatment can achieve this goal when the concentration of fluorine is high. Each step requires an optimization to get the best result.
This work studies the drying effect of hydrated lime on the removal of fluoride in the aluminum fluoride industry effluent. The variations in conductivity, pH and the residual content of fluoride were followed when various lime excesses for neutralization at a laboratory scale. The best conditions of removal of fluoride were identified.

Key words: industrial wastewater, fluoride, treatment, hydrated lime, neutralization.

AMINO-INDOLONE-*N*-OXIDES : SYNTHESIS AND ANTIPLASMODIAL ACTIVITIES

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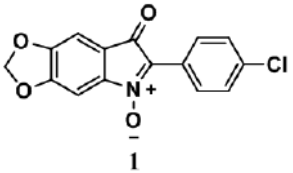
The emergence of the resistance of *Plasmodium falciparum* (*P. falciparum*) has been a major problem for the treatment of malaria in the last four decades making predictions of the future patterns of this difficult disease [1,2]. Recent reports showed a decreased susceptibility to artemisinin of *P. falciparum* in the Great Mekong region in Asia [3]. These indications prompt the need for new antimalarial drugs which ideally should act with novel mechanisms of action, on novel targets and/or should represent new chemotypes even though parasite resistance may evolve again. Indolone-*N*-oxides (INODs) display antimalarial properties *in vitro* and *in vivo*, but identified leads such as 6-(4-chloro-phenyl)-5-oxy-[1,3]dioxolo[4,5-f]indol-7-one **1**, suffer from very poor aqueous solubility [4]. In this study, structural modifications have been made by introducing various amino groups to produce sufficiently water soluble and active compounds for further pharmacological and pharmacokinetic studies. A series of 12 new ether, ester and amide derivatives of the indolone-*N*-oxide was synthesized. Compounds were evaluated for *in vitro* activity against chloroquine-resistant (FcB1) strain of *Plasmodium falciparum* (*P.f.*), as well as for cytotoxic concentration (CC_{50}) on MCF7 cell line. The amino-ether-indolone-*N*-oxides had the most potent antiplasmodial activity *in vitro* (IC_{50}/nM < 25).



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PREPARATION OF 2-CHLORO-5, 5-DIMETHYL-1,3,2-DIOXAPHOSPHORINANE AND STUDY OF THEIR REACTIVITY

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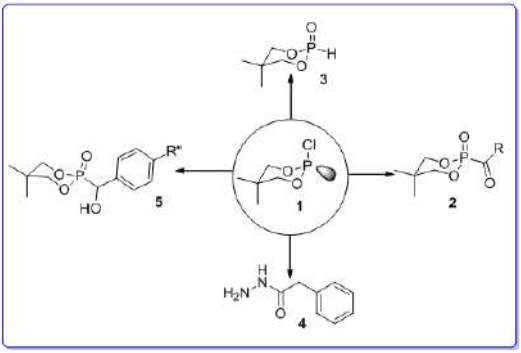
α-Ketophosphonates are one of the most interesting classes of organophosphorus compounds.They exhebit biological activities and they are used as precursors for the synthesis of biphosphonates, which are used against paget deseases. In this work we have prepared a family of α-ketophosphonates[1-3] from 5,5-dimethyl-2-chloro-1,3,2-dioxphosphorinane **1** using one-pot process. Their reactivity was then examined using primary amines and phosphite (Scheme 1).



Scheme-1: The synthesized organophosphorus compounds

Key words: 2-chloro-1,3,2-dioxaphosphorinane, α-ketophosphonate, hydrazide, dialcoxyphosphite, Hydroxyphosphonate.

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Bis(tetraethylselenophosphoramidoyl)methylamine as a chelating ligand for heavy metallic cations

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The coordination chemistry of the imidodiphosphorus acids of type $\{(XPR_2)(YPR'_2)\}NH$ (X,Y: O,S,Se; R,R' : alkyl, aryl) has become an active area of research due to their selective complexation properties towards 'hard/soft' metal cations [1-2]. In contrast, a little attention has been paid to the coordination chemistry of the neutral ligands of the type $RN\{P(E)R'_2\}_2$ and few examples of their complexes have been described in literature [3-4].

Herein, we describe the synthesis strategy of a new neutral bidentate ligand, bis(tetraethylselenophosphoramidoyl)methylamine, $MeN[P(Se)(NEt_2)_2]_2$. Complexation event was characterized by various electrochemical techniques such as CV, DPV and chronoamperometry. The results reveal that the reported ligand is able to chelate various heavy metallic cations (M^{2+}: Zn^{2+}, Cd^{2+}, Pb^{2+}, Hg^{2+} and Fe^{2+}) forming high stable complexes. From DPV results, we notice that addition of each cations underwent important oxidation potential shifts up to 1070 mV. 1:2 metal-ligand stoichiometry was also confirmed.

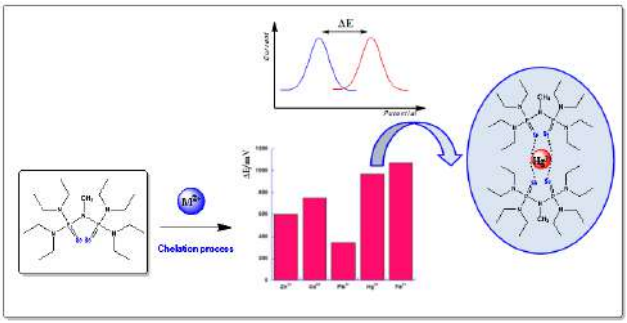

Fig. Potentiometric response of the chelation process

Keywords: Seleno-phosphoramide ligand; chelation process; heavy metal cations; voltammetry technique.

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Imidazolium-based ionic liquid type dependence of the bending response of polymer actuators

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Actuators based on polymer blends of poly(vinylidene fluoride) (PVDF) with 40 % of different ionic liquids (IL) are prepared by solvent casting. [C_2mim][Cl], [C_6mim][Cl], [C_{10}mim][Cl], [C_2mim][NTf_2], [C_6mim][NTf_2] and [C_{10}mim][NTf_2] were selected in order to evaluate the effect of anion and cation sizes in the bending properties. The microstructure, mechanical and electrical properties of the blend depend on the IL type, which in turn leads to a different bending response. In particular, the mechanical properties are independent on the IL type but the AC conductivity of the composites depend more on the anion type than on the size of the alkyl chain connected to the imidazolium based cation. Thus, the bending response of the IL/PVDF composites is correlated with the anion and cation sizes and a maximum bending response of 0.3 % is achieved for a 10 volts square signal in the IL/PVDF composite with 40 wt% content of [C_2mim][NTf_2].

Key words: PVDF, ionic liquid, actuator, electrical conductivity, bending

Halogenated Flame retardants compounds in urchin and sediment from Bizerte Lagoon by GC-MS-MS

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In the last decades, halogenated flame retardants (HFRs) such as brominated flame retardants, (RFBs) metoxylated brominated diphenyl ethers (MeO-PBDEs) and Dechloranes (DP) threat the environment by their persistence, bioaccumulation and potential toxicity [1]. We report the first study of the HFRs analysis in sediment and urchin samples collected from Bizerte Lagoon.
Accelerated solvent extraction (ASE) was used to extract samples in the presence of DCM Hexane (1:1, v/v) as a solvent. Regarding urchin, two steps of purification were used: the first one using acid sulfuric concentrated (H_2SO_4) and the second one by solid phase extraction (SPE). The extraction and the purification of the sediment proceed simultaneously using a selective pressurized liquid extraction (SPLE). Finally, the extracts were concentrated and re-dissolved in toluene [2]. Compounds were detected by CG-MS-MS machine.
Pollutant concentration follows this order MeO-PBDEs > DP > PBDEs. They were detected with variable levels due to the collection of samples from different zone such as industrial, agricultural…

Key words: PBDEs, MeO-PBDEs, Dechloranes, GC-MS-MS.

References

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MODELING OF THE DRYING KINETICS OF A FOOD PRODUCT BY SOLAR ENERGY

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Drying is one of the main techniques of preservation of agricultural and food products. This theoretical study focuses on optimizing an indirect type of solar dryer running in forced convection. It consisted of developing a mathematical model to describe the thermal behavior and the energetic power of the dryer.
This mathematical model has been obtained by application of heat transfer laws and mass in the various slices constituting the solar dryer. This model is validated by several studies on tomato which after being dried and ground, is of great interest to the food industry. It therefore consists on a system of equations to solve it using a simulation tool called MATLAB.
We have achieved results that describe the variation of temperature and humidity according to time for different screens and different temperatures and air speed drying. The results show that the disposition of product in the drying cabinet as well as that of the drying temperature and the air speed is very important parameters that influence the drying process in very important ways.

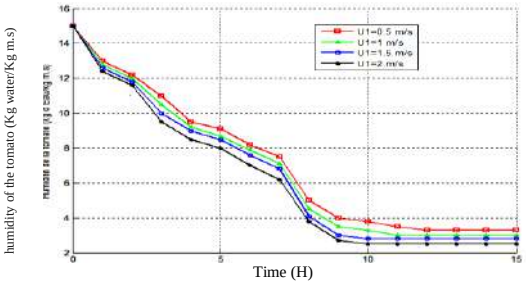

Fig. 1 The influence of air speed drying the product moisture

Key words: drying, solar dryer, agricultural, food product, optimizing.

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SYNTHESIS, CRYSTAL STRUCTURE, VIBRATION SPECTRAL, AND DFT STUDIES OF A NEW ORGANIC-INORGANIC HYBRID MATERIAL: (2-HAMP)$_2$[CoBr$_4$]

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Single crystals of a new organic–inorganic hybrid compound (2-HAMP)$_2$[CoBr$_4$], (2-HAMP = 2-protonated aminopyridinium cation) was synthesized and characterized by X-Ray diffraction at room temperature, DTA–TG measurement, FT-IR and FT-Raman spectroscopies and optical absorption techniques. Its crystal structure is a packing of alternated organic and inorganic layers parallel to (a, b) plane. The different components are connected by a network of N/C-H⋯Br hydrogen bonds and halogen⋯halogen interactions. These hydrogen bonds give notable vibrational effects. Theoretical calculations were performed using density functional theory (DFT) for studying the molecular structure, vibrational spectra and optical properties of the investigated molecule in the ground state. The optimized geometrical parameters obtained by DFT calculations are in good agreement with single crystal XRD data. The energy and oscillator strength calculated by Time-Dependent Density Functional Theory (TD-DFT) results complements with the experimental findings. The simulated spectra satisfactorily coincide with the experimental UV-Visible spectrum. The results show good consistent with the experiment and confirm the contribution of metal orbital to the HOMO–LUMO boundary. Thermal analysis studies indicate the presence of three phase transitions at 68, 125 and 172°C, which are confirmed by X-ray powder diffraction as a function of temperature.

Key words: Organic template; Metal halides; DFT calculations; Optical absorption, Thermal studies and phase transitions.

NMR SPECTRAL ASSIGNMENT OF NEW γ -PHOSPHONYLATED OXIMES

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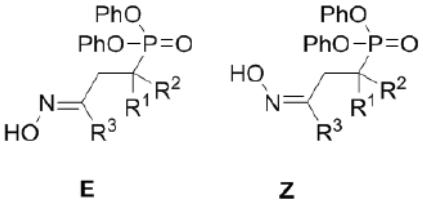
Oximes are important building blocks for the synthesis of numerous pharmaceuticals, biologically active compounds and natural products such as erythromycin derivatives[1] and perhydrohistrionicotoxin also for their industrial applications in the areas of agrochemicals,[2a] medicinal chemistry,[2b] toxicology, and in the preparation of cephalosporin derivatives with potent antibacterial activity. In view of the above, and in the continuation of our studies on the synthetic applications of phosphonyl ketones, we report here for the first time the synthesis of γ -phosphonyloximes from the base catalyzed reaction of hydroxylamine hydrochloride with γ –ketophosphonates. The stereochemistry (E and Z) of a series of new γ-phosphonylated oximes was assigned on the basis of the chemical shifts of carbons in α positions with respect to the C=N moiety. This carbon rings in a field strongest when it is in the syn-position relative to the OH group carried by the nitrogen when it is in anti to the same reason. The synthesized compounds could have promising applications as precursors of new phosphoruscontaining polymers, which are known for their improved flame retardancy as well as improved thermo- and thermo-oxidative resistance. These studies are ongoing in our laboratory and will be reported in due course.



Schemes 1

Key words: Oxime, ketophosphonate, stereochemistry.

References

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BIACTIVATION OF THE DARZENS REACTION BY THE PHASE TRANSFER CATALYSIS AND UNDER ULTRASONIC IRRADIATION

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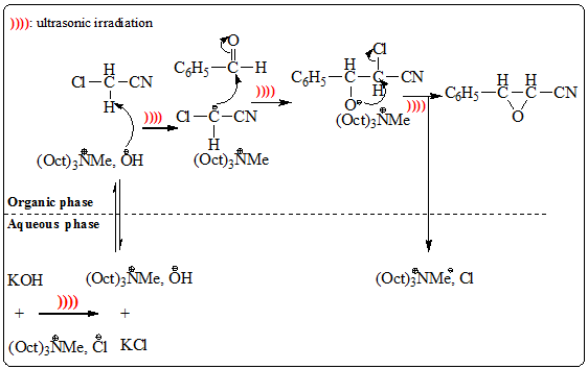
Currently, sonochemical processes are more widely developed in organic synthesis. However, the possibility of improvements in many types of reactions suggests that there is still a long way to go to achieve the ideal procedure [1]. Many improvements are still needed to fully develop the general coupling procedures to good results. In this regard, we studied the carbonyl compounds with α-haloester to form and α, β-epoxy ester in the presence of Aliquat-336 and under ultrasonic irradiation under optimized conditions at room temperature. Yields are increased by sonochemical biactivation and catalysis phase transfer presence for a very short duration. The isolated products have a high purity. In this regard, we suggest the reaction mechanisms that could explain the results.



Scheme: Proposed mechanism for the Darzens reaction of α–chloroacetonitrile with benzaldehyde under ultrasonic irradiation in the presence of PTC.

Key words: Darzens, ultrasonic irradiation, Phase Transfer Catalysis, biactivation.

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SONOCHEMICAL ACTIVATION OF THE NOZAKI-HIYAMA-KISHI REACTION OF HALIDES AND ALDEHYDES CATALYZED BY Cr/Ni

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New concepts are outlined for formations C-C bond catalyzed by bimetallic systems have emerged. The development of the Nozaki-Hiyama-Kishi reactions catalyzed by Cr/Ni combination is being pursued with strong interest in the presence of ultrasound at room conditions. It has been shown that the ultrasonic irradiation promoted an efficient enantioselective Nozaki-Hiyama-Kishi reaction of various halides and aldehydes [1]. With this novel method, the addition of various organochromium intermediates to aromatic and aliphatic preceded under catalytic conditions increases the rate and improves the yields of the reaction. In this context, a reactionnel mechanism is proposed.

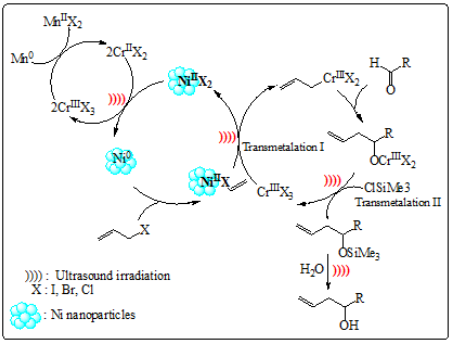

Fig. Proposed mechanism of the Nozaki-Hiyama-Kishi cross-coupling reaction catalyzed by Cr/Ni under ultrasound.

Key words: Nozaki–Hiyama–Kishi reaction; organochromium; ultrasonic irradiation; Cr/Ni combination.

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EFFICIENT MILD OXIDATION OF 5-HYDROXYMETHYLFURFURAL TO 5-HYDROXYMETHYL-2(*5H*)-FURANONE

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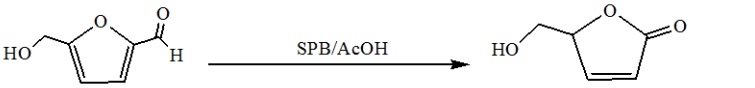
The 5-hydroxymethyl-2(*5H*)-furanone is a versatile chemical intermediate used to produce a variety of products such as unsaturated (+)-Muscarine and certain analogues of prostacyclin and chrysanthemic acid. In this study, we have successfully tested a sodium perborate ($NaBO_3.4H_2O$) known by its acronym SPB in the oxidation of 5-hydroxymethylfurfural (5-HMF) using acetic acid (AcOH) as solvent. The SPB has been proven to be an efficient reagent for the oxidation of 5-HMF into 5-hydroxymethyl-2(5H)-furanone in one step. A reaction mechanism has been proposed for this purpose. In this work a variety of important reaction parameters, such as SPB amount, reaction temperature, and time were explored. The maximum yield to 5-hydroxymethyl-2(*5H*)-furanone was achieved in 94% at room temperature in 24h with 5-HMF conversion of 100% was obtained in SPB/ 5-HMF mol ratio = 1.5 under optimal reaction conditions. Then it could be obtained in a high yield of 87% at 50°C after a short reaction time of 4h. At high temperature (100°C) 5-hydroxymethyl-2(*5H*)-furanone yield decrease dramatically.

Key words: 5-HMF, 5-hydroxymethyl-2(*5H*)-furanone, SPB, AcOH solvent.



5-Hydroxymethylfurfural 5-Hydroxymethyl-2(*5H*)-furanone

Scheme. First chemoselective oxidation of 5-hydroxymethylfurfural into 5-hydroxymethyl-2(*5H*)-furanone

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PREPARATION AND CHARACTERIZATION OF POLY (N-2-VINYLPYRROLIDONE-CO-VINYL ACETATE) USING RADICALER POLYMERERIZATION

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The aim of this study is to prepare and characterize the poly (N-2-vinyl pyrrolidone-co-vinyl acetate) Poly (VP/VAC) copolymers. It has been prepared and characterized two vinyl copolymers (CP_a) and (CP_b) by radical copolymerization using the couple of comonomers N-2-vinyl pyrrolidone (VP) /vinyl acetate (VAC). The mass copolymerization (AIBN 1%) leads to white solid copolymer (CP_a). The (CP_b), a slightly yellowish copolymer was prepared in solution in dioxane (0.3% AIBN). The obtained copolymers were recrystallized from petroleum ether. These copolymers were characterized by IR, RMN ^1H, DSC and the $\bar{M}_v$ molecular masses were determined.

Key words: Radical Copolymerization, Poly (VP/VAC), Copolymer, Vinyl acetate.

FUNCTIONALIZATION OF SILICA NANOPARTICLES BY QUERCETIN AND RUTIN

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Currently, nanotechnology, especially devoted to medicine, pharmacy and biology has grown significantly. Indeed, a large number of functional nanomaterials have been developed for use in the medical field. In this regard, we have prepared homogeneous silica nanoparticles with a nanometer size, a surface area ranging from 517 to 964 m^2/g and a hydrodynamic diameter varying from 327 to 370 nm. These characteristics show that the synthesized nanoparticles are compatible for biological application. The grafting of quercetin (Fig.1) and rutin on the silica nanoparticles was performed using 3-(triethoxysilyl) propyl isocyanate as a coupling agent.

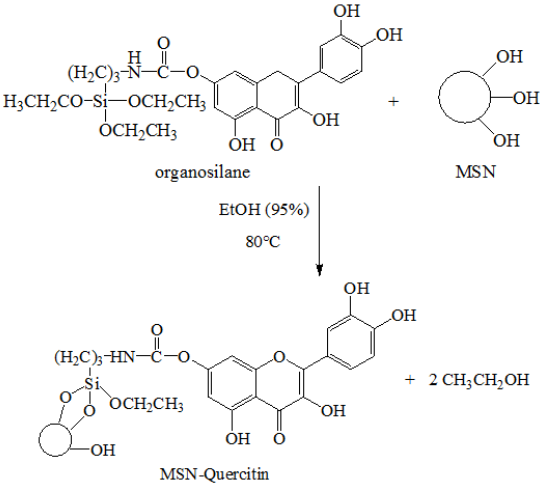

Figure 1. Grafting reaction of Quercetin

Synthesis and characterization of a new noncentrosymmetric dihydrogenmonophosphate $[C_3H_6N_3S_2]H_2PO_4$

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Organic-inorganic hybrid materials with noncentrosymmetric structures are interesting for their application in various fields such as quadratic non-linear optical materials research [1]. Their abilities to combine the high cohesion of inorganic host matrices with the enhanced polarizability of organic guest chromophores within one molecular scale leads to a better performance of optical signal processing devices [2]. Here, we report the synthesis and the crystal structure of a new hydrogen phosphate with an organic cation $[C_3H_6N_3S_2]H_2PO_4$. This compound crystallizes in the noncentrosymmetric orthorhombic space group Pna21 with the lattice parameters a = 7.662 (1), b = 26.553 (3), c = 4.4258 (6) Å, V = 900.4 (2) $Å^3$, and Z = 4. The framework is built upon layers parallel to (0 1 0) made up from $[H_2PO_4]^-$ entities associated by O-H...O strong hydrogen bonds. The organic cations are located between the layers and connect them via N-H… O and C-H…O hydrogen bonding network (Fig). Results from solid state ^1H, ^{31}P, ^{13}C and ^{15}N spectroscopy are in good agreement with the X-ray structure. Density functional theory calculations allowed for the assignment of the carbon and nitrogen peaks to the independent crystallographic sites.

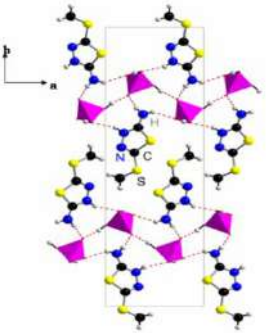

Fig. The atomic arrangement of $[C_3H_6N_3S_2]H_2PO_4$ along the c-direction.

Key words: X-ray diffraction, Infrared Spectroscopy, NMR Spectroscopy, DFT calculations.

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Synthesis, crystal structure and characterization of a new Octane-1,8-diammonium bis(dihydrogenmonophosphate)

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Recent years have witnessed an explosion of great interest in hybrid organic-inorganic framework solids not only for their intriguing architectures and topologies, but also for their potential applications in optical, electrical, magnetic, and micro-porous materials [1, 2]. In this work, the synthesis and the structure of a new $[H_3N\text{-}(CH_2)_8\text{ -}NH_3][H_2PO_4]_2$ is described. This compound crystallizes in the orthorhombic system, with the centric space group Pbcn and the following unit cell parameters: a = 7.2887 (4), b = 9.1728 (6), c = 23.2169 (19) Å, Z = 4 and V = 1552.23 (18) $Å^3$. The crystal structure has been solved and refined to R = 0.074 and R(w) = 0.173. The $[H_2PO_4]^-$ entities are associated by O---H...O strong hydrogen bonds to form layers which are interconnected with the organic entities via (N)-H...O hydrogen bonds. (**Fig)** In this atomic arrangement, H bonds between the different species play an important role in the three-dimensional network. This compound has also been characterized by infrared spectroscopy, solid state MAS-NMR and thermal analysis.

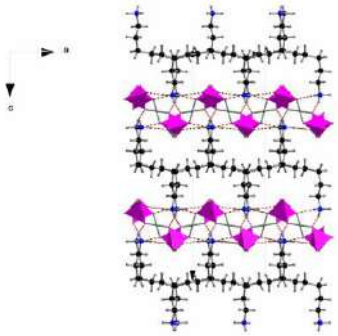

Fig. The atomic arrangement of $[H_3N\text{-}(CH_2)_8\text{ -}NH_3][H_2PO_4]_2$ along the b-direction.

Key words: X-ray diffraction, Infrared Spectroscopy, NMR Spectroscopy, ATD, ATG and DSC.

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Thermophysical properties of the binary mixtures of N,N-Dimethylacetamide with 2-propanol and 2-butanol

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Various thermophysical properties such as density, ρ, kinematic viscosity, ν, speed of sound, u, and refractive index, n_D, were measured for the pure N,N-dimethylacetamide, 2-propanol, 2-butanol and their binary mixtures over the whole composition range at the temperatures 283.15, 298.15 and 313.15 K and at atmospheric pressure. Experimental values were used to calculate the excess values of molar volume, V^E, isentropic compressibility, κ_S^E and deviations in viscosity, $\Delta\eta$, and refractive index, Δn_D. These properties were correlated with the Redlich-Kister equation. The least-square method was used to determine the adjustable parameters. Excess molar volumes are positive for both mixtures. Refractive index deviations are positive for N,N-dimethylacetamide + 2-propanol and negative for N,N-dimethylacetamide + 2-butanol. Viscosity deviations and excess isentropic compressiblities are negative for all systems. These results were interpreted based on strength of specific interactions, size and shape of molecules.

Key words: density, viscosity, speed of sound, refractive index

Solution and solid-state fluorescence of 2-(2'-hydroxyphenyl) 1,5benzodiazepin-2-one(HBD) borate complexes

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A new family of fluorescent 1,5-benzodiazepin-2-one (HBD) borate complexes was prepared in good yields[1], and fully characterized by means of MS, NMR and IR spectroscopy, as well as X-ray crystal structure analysis for compound 13. Unlike their uncomplexed congeners, most of these cyclic boranils were emissive both in solution and solid state, with maxima in the range of 426 596 nm. A systematic study of substituent effects revealed that the presence of a halogen atom specifically at position 8 of the fused-aromatic ring system led to an increase in fluorescence intensity in solution while electron rich substituents tended to extinguish the photoluminescence[2]. Finally, a proof-of-concept study highlighted that the amide moiety of the benzodiazepinone framework could be functionalized with a chemical handle useful for subsequent specific modifications.

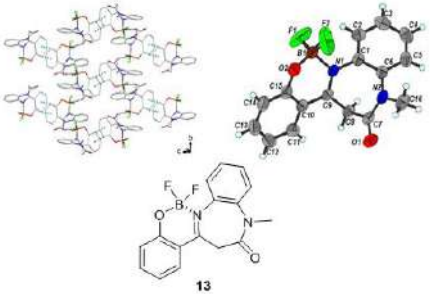

Fig.1 Crystal structure of compound **13**

Key words: 1,5-benzodiazepin-2-one, solid-state fluorescence, Boranils

References

[1] Hasan Mtiraoui,[a] Rafik Gharbi;[a] Moncef Msaddek,[a] Yann Bretonnière,[b] Chantal Andraud,[b] Cyrille Sabot,[c] Pierre-Yves Renard,[c]; J. Org. Chem. **2016**, 81, 4720-4727.

[2] Hasan Mtiraoui,[a] Rafik Gharbi;[a] Moncef Msaddek,[a] Yann Bretonnière,[b] Chantal Andraud,[b] Cyrille Sabot,[c] Pierre-Yves Renard,[c,] RSC Adv., **2016**, 6, 86352-86360

EFFECT OF OZONE TREATMENT ON DEGRADATION OF POLYSACCHARIDES FROM TUNISIAN *ARTHROCNEMUM INDICUM*: PHYSICO-CHEMICAL PROPRIETIES AND ANTIOXIDANT ACTIVITIY

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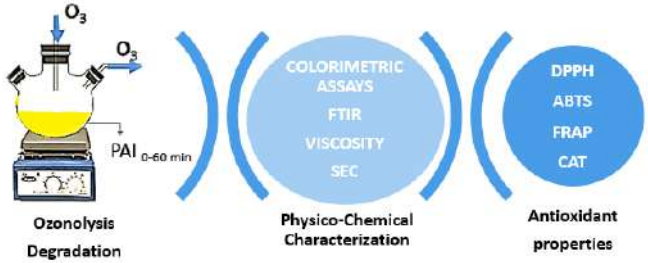
The depolymerisation of crude carbohydrates from Tunisian *Arthrocnemum indicum* leaves was investigated. In this study, the ozonolysis treatment for various reaction times (0,15,30,45 and 60 min) was used as degradation method in order to obtain the lower molecular weight product with a stronger antioxidant activity and a higher purity. Total sugar and uronic acid contents were determined by colorimetric assays. The intrinsic viscosity as well as the macromolecular characteristics of the different degraded polysaccharides were carried out by an Ubbelohde viscometer and size exclusion chromatography, respectively, before and after ozonolysis treatment. These experiments showed that the intrinsic viscosity of the degraded samples decreased exponentially with increase in reaction time, and molecular weight (Mw) was established and confirmed well without changing the structure. The surface color analysis indicated that the color of the native polysaccharide was different from that of degraded polysaccharide fractions. Furthermore, the antioxidant properties *in vitro* (scavenging abilities for $ABTS^{+\bullet}$ and $DPPH^{\bullet}$, Ferric reducing power activity and total antioxidant property) of polysaccharides proved to be significantly enhanced after ozonolysis depolymerisation (ranged from 25.90 to 64.51%, 68.71 to 83.57%, 1069.8 to 2388.8 µmol/L and 16.87 to 56.25 mg ascorbic acid equivalents/g dry matter, respectively).



Keywords: *Arthrocnemum indicum*, Carbohydrates, Ozonolysis treatment, Depolymerisation, Antioxidant properties.

Chromium Speciation in environmental and aqueous samples using Electrothermal atomic absorption

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Chiraz Hannachi,[b] Bechir Hamrouni,[b] and Habib Nouaïgui[a]

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Hexavalent chromium has been identified as an inhalation carcinogen. Recently, threshold limit values in workplace airborne have been lowered for this element. Consequently, the current available analytical methods are not enough sensitive or of high cost. In this paper a simple, cost effective, sensitive and reproducible solid phase extraction electro-thermal atomic absorption spectrometry method for hexavalent chromium quantification in airborne and aqueous samples is proposed. The method was validated with respect to linearity, accuracy and precision. The detection and quantification limits were respectively as low as 0.1 and 0.4 $\mu g \cdot L^{-1}$. The linearity ranged from 0.5 to 50.0 $\mu g \cdot L^{-1}$, with a regression coefficient exceeding 0.998. The extraction recovery exceeded 98 %. The developed procedure was tested on real airborne and aqueous samples collected from workplaces. The method performances suggest that the proposed method could be an interesting alternative to high cost analytical techniques for the monitoring of the occupational exposure to hexavalent chromium compounds.

Keywords: Airborne and aqueous samples, occupational monitoring, environmental assessment, solid phase extraction, hexavalent chromium speciation, electro-thermal atomic absorption spectrometry.

REACTIVITY OF SUBSTITUED OLEFINS WITH SECONDARY AMINES IN WATER: KINETIC STUDY AND REACTION MECHANISM

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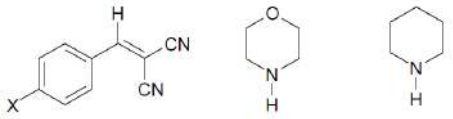
The kinetic of the reactions of the olefins **1a** and **1b** with secondary amines (morpholine **2** and piperidine **3**) have been determined in water at 20°C. The resulting second-order rate constants were employed to determine the nucleophile-specfic reactivity parameters N and s of the two secondary amines **2** and **3** according to the correlation equation (1).

$$\log k\ (20\ °C) = sN + sE \qquad (1)$$

As can be seen, the effect of substituents on the reactions rate was examined quantitatively on the basis of kinetic measurements, leading to a good linears correlations of log k *vs.* Hammett's substituted contants (σ) and of log k *vs.* pK_a of secondary amines.

Finally, we have reexamined the values of N/s of morpholine and piperidine by applying Mayr's relationship. A mechanism is proposed to explain the formation of coupling products.



1 **2** **3**

1a X = NMe_2
1b X = OMe

Key words: olefins, secondary amines, σ-complexation's process, Mayr's relationship, Structure-reactivity correlations.

References

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A HIRSHFELD SURFACE ANALYSIS, SUPRAMOLECULAR STRUCTURE AND MAGNETIC PROPERTIES OF A NEW CU(II) COMPLEX WITH THE 4-AMINO-6-METHOXYPYRIMIDINE LIGAND

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A new Cu(II) complex with the bridge bidentate ligand 4-amino-6-methoxypyrimidine, $[Cu(C_5H_7N_3O)(H_2O)(NO_3)_2]$, has been prepared at room temperature and characterized by single crystal X-ray diffraction and IR spectroscopy. The compound crystallizes in the monoclinic space group C2/c with lattice parameters a = 17.783 (4), b = 11.131 (3), c = 12.594 (3)Å, β = 117.616(3)°, V = 2209.0(9) $Å^3$ and Z =8. The Cu(II) cation is hexa-coordinated, in distorted octahedral fashion, by two nitrogen atoms of two 4-amino-6-methoxypyrimidine ligands, one water oxygen atom and three oxygen atoms of two nitrate anions. In the atomic arrangement, the organic ligands and the 6-connected Cu centers are linked with each other to give a 1-D corrugated chain running along the b-axis direction. The chains are interconnected via O-H···O, C-H···O and N-H···O hydrogen bonds to form a three dimensional network. The analysis of contacts on the Hirshfeld surface shows that the crystal packing is driven mainly by the electrostatic interactions: the coordination of Cu(II) by O and N as well as strong hydrogen bonds. The vibrational absorption bands were identified by infrared spectroscopy. Magnetic properties were also studied to characterize the complex.

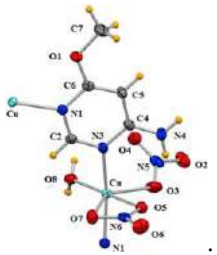

Figure 1: Molecular structure of the title compound showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

Key words: Hirshfeld surface, Enrichment ratio, Quantum mechanics, X-ray diffraction, Coordination compound, Magnetism.

STRUCTURAL AND SPECTROSCOPIC STUDIES OF BIS(2-AMINO-6-METHYLPYRIMIDINIUM-4-(1H)-ONE) AQUAPENTACHLORIDOINDATE(III) MONOHYDRATE

Wijdene Nbili, Kamel Kaabi, Chérif Ben Nasr

Université de Carthage, Laboratoire de Chimie des Matériaux, Faculté des Sciences de Bizerte, 7021 Zarzouna, Tunisia.

Physico-chemical properties of a new organic indate(III) $(C_5H_8N_3O)_2[InCl_5(H_2O)].H_2O$ are discussed on the basis of its X-ray crystal structure investigation. The asymmetric unit includes two independent 2-amino-6-methylpyrimidinium-4-(1**H**)-one cations, one aquapentachloridoindate dianion, and one crystallization water molecule. The In^{III} ion is in slightly distorted octahedral coordination geometry. In the crystal structure, the dimeric species formed by two metal complexes and two water molecules are connected to the 2-amino-6-methylpyrimidin-4-1**H**)-one cations through N–H···Cl hydrogen bonds to build 2D sheets parallel to the (b, a + c) plane. The ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray structure. The vibrational absorption bands were identified by infrared spectroscopy. DFT calculations allowed attributions of NMR signals and of the IR bands.

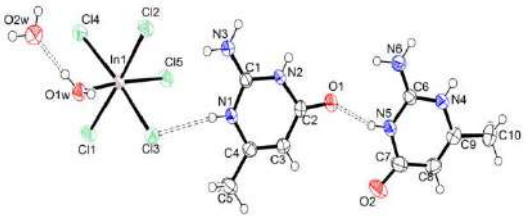

Figure. 1 Asymmetric unit of $(C_5H_8N_3O)_2[InCl_5(H_2O)].H_2O$ with the atom numbering scheme and thermal ellipsoids at 40 % probability

Key words: X-ray diffraction; Crystal structure; ^{13}C CP-MAS NMR; IR spectroscopy; DFT calculations.

NUCLEOPHILICITIES OF ALIPHATIC NITROALKALY ANIONS IN METHANOL

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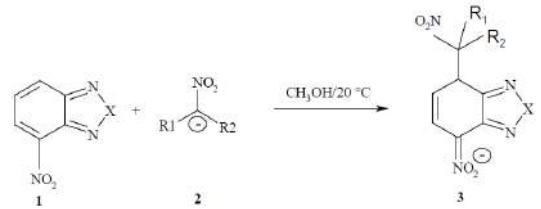
The objective of this work is the application of Mayr equation (1) to quantified the nucleophile specific N and s of aliphatic nitroalkaly anions **2A-C**. In this equation, the strength of the electrophiles is measured by the parameter E and the nucleophiles is represented by the two parameters N and s.

$$\log k\ (20\ °C) = s\ (E + N) \qquad (1)$$

k: constant of the second order ($M^{-1}.s^{-1}$).
E: parameter of électrophilie.
N/s: specific parameter of nucleophilicity.

The kinetics of the reactions between three nitroalkaly anions **2A-C** and 4-nitrobenzochalcogenadiazole **1a-c** were investigated photometrically in methanol at 20°C.
As can be seen, the second-order rate constants (log k) correlate linearly with the electrophilicty parameters E of the 4-nitrobenzochalcogenadiazole **1a-c**, allowing us to evaluate the parameters N and s of our nitroalkaly anions **2A-C.** On the other hand, the effect of basicity of these anions is discussed.



1
(a) X = S
(b) X = Se
(c) X = O

2
(A) R_1 = H, R_2 = H
(B) R_1 = CH_3, R_2 = H
(C) R_1 = CH_3, R_2 = CH_3

3

Key words: kinetics, Nucleophilicity, Equation of Mayr, Nitroalkaly anion.

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SYNTHESIS, CHARACTERIZATION AND ELECTRICAL PROPERTIES STUDY OF SULFATE FLUORAPATITES $Ca_4Na_6(SO_4)_6F_2$

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This work falls within the framework of systematic studies of the physicochemical properties of apatite, Here, we consider the sulfated apatite in order to show their interest in the field of solid oxide fuel cell type SOFC [1.3].

The synthesis of sulfated apatites of general formula $Ca_4Na_6(SO_4)_6F_2$ was carried out by reaction in the solid state at high temperatures. This apatite was characterized by X-ray diffraction (XRD), infrared absorption (IR) and Raman scattering spectroscopy.

These different techniques were used to verify the purity of the obtained apatite. It is an appatite crystallizes in the hexagonal system of P63 / m space group.

The study of the electrical properties of this apatite based on the complex impedance spectroscopy technique has shown that the conductivity increases with increasing temperature.In the temperature range studied the ionic conductivity checks the Arrhenius equation and Jonscher (figures 1 et 2).

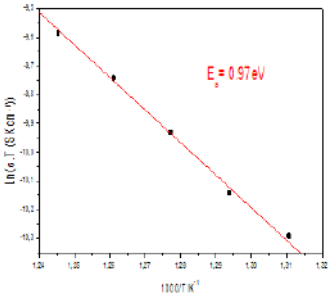

Figure 1. Arrhenius plot Ln(σT) = f(1000/T) of ionic conductivity of $Ca_4Na_6(SO_4)_6F_2$

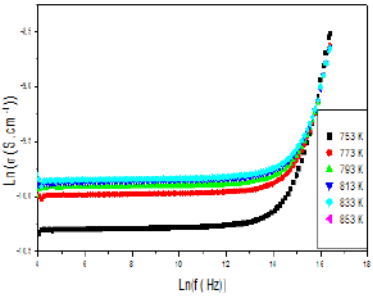

Figure 2. Frequency dependence ofconductivity at different temperatures of $Ca_4Na_6(SO_4)_6F_2$

Key words: Synthesis, Apatite, Sulfate, Impedance, Electrical properties.

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ADDITION OF TRYPTOPHAN-METHYL ESTER ON [60]FULLERENE: A SEMI EMPIRICAL METHOD AND DENSITY FUNCTIONNAL THEORY INVESTIGATIONS

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In order to synthesize carbon nanoparticles based for the vectorization of active ingredients, we suggested a reactional mechanism of the addition of Tryptophan methyl ester (TrpME) on [60]Fullerene (C_{60}) according a photo-addition reaction involving the oxygen in the singlet state.
In this investigation we studied the reactional mechanism by thermodynamic calculation of different formation reactions of azomethine ylide (AMY) and Fulleropyrrolidine mono-adduct (FP), and determined the relative stabilities of reactive intermediates formed by AM1 and DFT methods with 6-31G(d,p) basis set. A study has been also conducted on the AMY molecule formed during the reaction of methanal with TrpME, by a conformational analysis using DFT/B3LYP/6-31G(d,p). The five most stable conformations obtained for this molecule have been observed after a scan procedure dihedral angles of all values of D1-4 fixed, the most stable form has been registered for the D1 = 2.11°, D2 = -70.32°, D3 = 178.27° and D4=-3.71°.
The optimized geometries of FP molecule as well as the values of its thermodynamic constants obtained by AM1, HF and DFT/B3LYP, BPW91, SVWN5 methods with 6-31G(d,p) basis set has been also determined.
A detailed interpretation has been focused on the vibration frequencies and IR intensities of this product. FT-IR spectra observed in the region 100-4000 cm^{-1} and calculated by HF and DFT/6-31G(d,p) methods confirm our hypothesis suggesting the formation of the AMY followed by addition on the [60]Fullerene by the appearance of absorption bands $\nu(C_{aliphatique}\text{-}C\text{-}N)$, $\nu(C_{aromatique}\text{-}C\text{-}N)$ and $^{\delta}(N\text{-}H)$ coupled with $\nu(C\text{-}N)$.

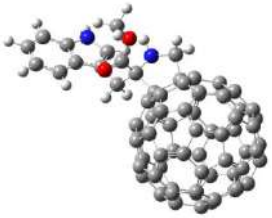

Fig. The fulleropyrrolidine mono-adduct.

Key words: [60]Fullerene, Tryptophan methyl ester, DFT, geometric optimization, thermodynamic properties, FT-IR spectra.

Concise Synthesis of Enantiopure (S)- and (R)-α-Trifluoromethyl Serine and Other Aza-Compounds from Chiral Trifluoromethyl Oxazolidine (Fox) via a Strecker-type reaction

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α-Trifluoromethyl α-amino acids and their derivatives have attracted considerable interest as natural α-aminoacid analogues, owing to unique propreties imparted by trifluoromethyl (Tfm) group, such as high electronegativity, electron density, steric hindrince, and hydrophobic character[1]. Aminoacids are rarely reported procedure in the litterature for stereoselective synthesis in enantiopure form. In this work, we show that the Strecker-Type reaction of chiral fluorinated oxazolidines (Fox) derived from (R)-phenylglycinol provides an expedient and convenient access to enantiopure side chain functionalized α-trifluoromethyl α-aminoacids such as α-Tfm Serine and α-Tfm- aziridine-2-carboxylates.

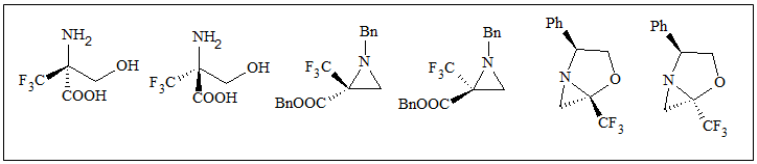

Fig. Various Aza-compounds prepared from Fluorinated oxazolidine Fox.

Keywords: Amino Acids, Oxazolidines, strecker reaction, aziridine-2-carboxylates.

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Calcium carbonate biominéralisation

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Biogenic crystals attract large attentions because of their superior properties on many aspects. While commonly involved in skeletal support or protection of cells and soft tissues, some biomineralized structures also have evolved to fulfill highly specific functions in demanding environments. The process of biomineralisation is realised with a minimum consumption of energy and a precise control on the polymorphism and crystal morphology [1] on a nanometer-scale by the use of organic macromolecules secreted by the organism. A deeper understanding of the mineralisation process and the mimicking of complex structures produced by nature in laboratory may have a significant impact on many fields such as the development of new composite materials, drug delivery...etc.

In this work, we used the Rietveld method and the anisotropic model of Popa to study the mechanism of action of polyacrylic acid on the growth of different polymorphs of calcium carbonate. The mechanism of action of the polyacrylic acid was clearly demonstrated when refining with anisotropic sizes model which shows a flattening effect on the crystallites in the case of vaterite and aragonite but not in the case of calcite (fig. 1). Then there is a strong interaction with PAA and $CaCO_3$ that could help understanding the growth of the natural bio-mineral.

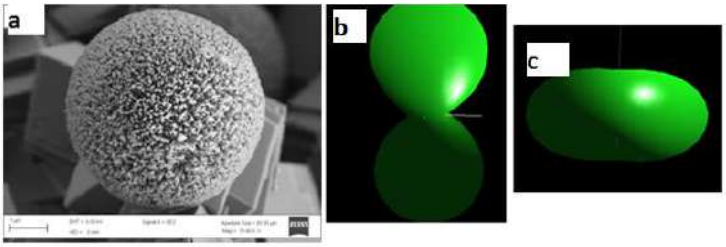

Fig 1: SEM image of vaterite particule (a) vaterite cristallite without PAA (b) Vaterite cristallite with PAA.

Key words: biomineralisation, calcium carbonate, Rietvel method.

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Modified Anionic Exchange membrane and electrodialysis

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Caused especially by agricultural and industrial activities, many countries confront an excess of nitrate in groundwater so they are concerned with minimizing their effects on human health and environment [1, 2].
In this work, we study the possibility to eliminate this excess of nitrate by electrodialysis using a new polymeric Anion Exchange Membrane: $PAEM_m$ (modified with nitrate complexant). We study the influence of electrodialysis time and current intensity on elimination of nitrate. Then, we examine the selectivity of $PAEM_m$ towards nitrate and different anions (alone and mixed with nitrate) while comparing it reaction or behavior with PAEM (without complexant) in order to understand the role of complexant inside the membrane. Finally, we have applied an application of this treatment on real water from a well of Cap bon, Tunisia and we found that about 93% of nitrate was eliminated after 1 hour and 25 minutes.

Key words: electrodialysis, modified anionic exchange membrane, elimination of nitrate.

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Synthesis, characterization and antibacterial activity of N-benzimidazole-O-acetyl-chitosan

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A novel chitosan derivative, N-benzimidazole-O-acetyl-chitosan (BZMACH) was prepared in acid acetic solution from imidate N-benzimidazole and shrimp-shell chitosan. The chemical structure and physical properties of BZMACH were characterized by FTIR, 1H NMR, DSC, TGA and XRD techniques. Besides, an enhancement of the antibacterial activity of chitosan against *Salmonella typhimurium* and *Pseudomoenas aeruginosa* bacteria has been also observed with this modification.

Key words: Chitosan, N-Benzimidazole-O-acetyl-chitosan, Characterization, antibacterial activity.

THE ELECTROPHILIE OF THE BENZOFURAZANS IN THE UNIVERSAL SCALE OF MAYR

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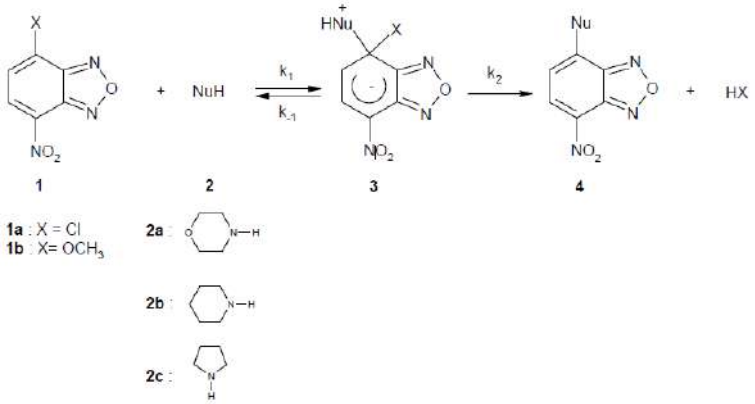
The kinetics of the reactions of 4-X-7-nitrobenzofurazan **1a** and **1b** (X = Cl and OCH_3) with various secondary amines **2a-c** have been studied by UV-visible spectroscopy in water and acetonitrile solution at 20 °C (Scheme 1).
The second-order rate constants (log k1) correlated linearly with the nucleophilicity parameters N and s of **2a-c** according to the correlation (1), allowing us to determine the electrophilicity parameter E for these benzofurazans **1a** and **1b**.

$$\log k1\ (20\ °C) = s\ (E + N) \qquad (1)$$



Scheme 1

Keywords: kinetics, electrophilicity, nucleophilicity, Mayr's equation, S_NAr mechanism

Références

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ASSESSMENT OF METAL POLLUTION OF ROADSIDE SOIL: CASE RN° 35, TLEMCEN, ALGERIA

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Laboratory of Condensed Matter and Sustainability (LMCDD) Liabes Djillali University of Sidi Bel Abbes, Faculty of Exact Sciences, Department of Chemistry.

Metal pollution of soils caused by road traffic is a serious environmental problem because of their biodegradation and bioaccumulation. The objective of this study is to assess the degree of contamination of 22 simples soils taken at 2 m from the floor by lead, cadmium and zinc, located on National Road 35 linking Remchi in Maghnia. the Analyses of heavy metals was carried out by dosing spectrometry flame atomic absorption, Hydrogen potential, electrical conductivity, organic matter and grain size are physicochemical parameters to consider in interpreting the content revealed pollutants.

Mean concentrations (mg / kg) of Cd, Pb and Zn in the soils of the study area are respectively 2.10, 75.58 and 567.31, while in soils of the control area were 1.92, 25.75 and 93.87, respectively. These results clearly show high levels of pollution related to road traffic.

These heavy metals contamination levels require remediation of these soils strategy to avoid their introduction into the environmental components near this busy road.

Key words: Soil, Road traffic, Metal pollution, Physicochemical parameters

Synthesis and characterization of new phenanthrene derivatives

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Phenanthrenes are versatile intermediates toward polycyclic aromatic hydrocarbons which are relevant for material sciences,[1] as well as toward helicenes,[2] an intriguing class of molecules which show remarkable chiroptical properties due to their helical pitch.
In this work, we describe the synthesis and characterization of new phenanthrene derivatives with cyano groups at selected positions on the aromatic rings. We have followed an efficient photochemical procedure for the construction of the phenanthrene derivatives[3] (Fig.1).

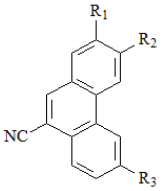

Fig 1. Synthesis of phenanthrenes derivatives

Key words: Phenanthrenes, Knovenagel condensation, Photocyclization.

References

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Synthesis and crystal structure of a new 4-fluorobenzylammonium dihydrogenphosphate: $[FC_6H_4CH_2NH_3]H_2PO_4$

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The synthesis and the structure of a new $FC_6H_4CH_2NH_3]H_2PO_4$, is described. The compound crystallizes in the orthorhombic system, space group Pbcn. Its unit cell parameters are a = 7.1630 (8), b = 9.1309 (10), c = 29.694 (3) Å, V = 1942.1 (4) $Å^3$, Z = 8. The crystal structure has been solved and refined to R = 0.078 and R_w = 0.017, using 1804 independent reflections. The asymmetric unit of the title compound, $[p\text{-}FC_6H_4CH_2NH_3]H_2PO_4$, contains one 4-fluorobenzylammonium cation and one dihydrogenphosphate anion. In the crystal, the $H_2PO_4^-$ anions are linked by O – H···O hydrogen bonds, as to build layers extending parallel to (001) planes. The $FC_6H_4CH_2NH_3^+$ lie between these anionic layers to maximize the electrostatic interactions and linked with the $H_2PO_4^-$ anions through N – H···O hydrogen bonds, forming a three dimensional supramolecular network. Two hydrogen atoms belonging to the phosphate anion are disordred, along the O···O direction involving two $H_2PO_4^-$ anions, over two positions with equal occupancy factors. This compound has also been chraracterized by infrared spectroscopy, solid state MAS-NMR and thermal analysis.

CRYSTAL STRUCTURE, PHASE TRANSITION AND THERMAL BEHAVIOR OF THE DABCODIIUM HEXAAQUAMANGANESE(II) BIS(SELENATE), $(C_6H_{14}N_2)[Mn(H_2O)_6](SeO_4)_2$

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The crystal structure of 1,4-diazabicyclo[2.2.2]octane (dabco)-templated manganese selenate, $(C_6H_{14}N_2)[Mn(H_2O)_6](SeO_4)_2$, was determined at room temperature from single-crystal X-ray diffraction. At 20 °C, it crystallises in the monoclinic symmetry, centrosymmetric space group $P2_1/n$, with the following unit cell parameters: a = 7.2373(4), b = 12.5600(7), c =10.1945(7) Å, β = 91.155(4)° and Z = 2. The structure consists of $[Mn(H_2O)_6]^{2+}$ and disordered $(C_6H_{14}N_2)^{2+}$ cations and $(SeO_4)^{2-}$ anions connected together by an extensive three-dimensional H-bond network. The title compound undergoes a reversible phase transition at 227 and 229 K in the heating and cooling cycle, respectively, which was detected by DSC and probably indicates the ordering of the organic part at lower temperature. The thermogravimetric analysis indicates that thermal decomposition of the supramolecular precursor proceeds through four stages giving rise to the manganese oxide.

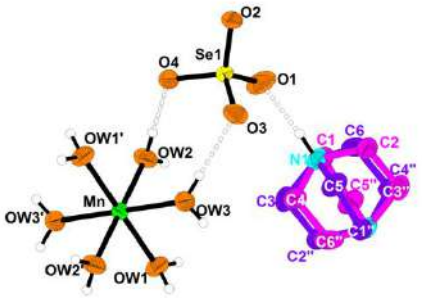

Fig. Asymmetric unit of the title complex expanded by symmetry to give complete cations

Key words: Hybrid materials, Supramolecular structure, phase transition, thermal decomposition.

Chemical composition, antioxidant and antimicrobial activities of the essential oil of *Trachyspermum ammi* L. growing in Tunisia

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In order to increase our knowledge about Trachyspermum ammi L., this work assesses the chemical composition as well as the in vitro biological activity of its essential oil. Thus, the essential oil obtained by hydrodistillation of the aerial parts of this species was analyzed by GC-FID and GC-MS. The main components of *T. ammi* L. oil were Thymol (60.90%), carvacrol methyl ether (18.93%), γ-terpinene (7.50 %), p-cymene (4.59 %), carvacrol (1.99 %) and α-pipene (1.31 %). The essential oil of *T. ammi* stood out for its antioxidant activity expressed on µg. ml^{-1}, 6.53 and 5.24, for DPPH and ABTS assays, respectively. Antimicrobial activity against several micro-organisms, including some ones infesting historical art craft, was also determined. These results suggested that essential oil of *T. ammi* processes antimicrobial and antioxidant proprieties, and are therefore a potential source of active ingredients for food and pharmaceutical industry.

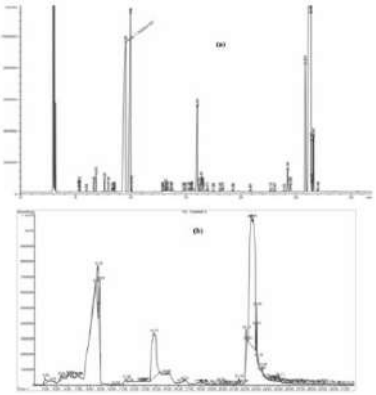

Fig.1. GC-FID and TIC (b) chromatograms of essential oil of *T.ammi*.

Key words: *Trachyspermum ammi* L., major constituents, thymol, antimicrobial, antioxidant.

THERMODYNAMIC MODELING OF THE (KNO_3+$TLNO_3$) BINARY SYSTEM

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Nowadays, the use of renewable energy has been the subject of much research. Alkali nitrates have a great interest in this area, especially in the storage and transfer of solar energy. These salts are characterized by low melting points, relatively high transition enthalpies and good thermal conductivities. Thallium nitrate, often encountered in industrial processes, has great similarities with alkali nitrates.The combination of these nitrates can provide materials with interesting properties.
The study of phase diagrams of binary and/or higher order systems contributes to give industrial information for a better use. However, in most cases, the reported data concerning the thermodynamic properties of these nitrates and especially their phase diagrams are incomplete, inconsistent or they present some discrepancies between the various published data.
Thermodynamic calculation is the approach that solves the differences between the thermodynamic data of binary systems and gives a more complete description of higher order systems.
The purpose of this work is to optimize, for the first time, the (KNO_3+$TlNO_3$) binary system using all the available experimental data.The coherence between thermodynamic properties and phase diagram of this system is studied. The results are then compared to those found experimentally.

Keywords: Potassium nitrate, Thallium nitrate, Optimization, Phase diagram

SYNTHESIS, CRYSTAL STRUCTURE AND SPECTROSCOPIC STUDIES OF A NEW NON-CENTROSYMMETRIC ORGANIC CATION NITRATE

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The synthesis, crystal structure and spectroscopic studies are reported for the salt *m*-anisidinium nitrate. A single-crystal X-ray investigation has shown that this compound crystallizes in the non-centrosymmetric space group Cc with the lattice parameters: a = 5.744(3) Å, b = 14.968(6) Å, c = 10.178(4) Å; β = 96.910(7)^{o} ; V = 868.6(6)Å3 ; Z = 4. The structure was solved from 1973 independent reflections with R1 = 0.033 and wR2 = 0.088. The hydrogen atoms of the protonated amine undergo hydrogen bonding interactions with oxygen atoms of three different nitrate anions forming 2-dimensional sheets. Solution NMR results are consistent with the X-ray structure. A measured optical band gap of 3.35 eV indicates that *m*-anisidinium nitrate is a wide-band-gap dielectric material.

Keywords: Organic-inorganic hybrid; Crystal structure; IR Spectroscopy; NMR spectroscopy; Band-gap

Advanced $Pd/Ce_xZr_{1-x}O_2/SiO_2$ Catalysts for methane combustion: Effect palladium amount

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For applications in gas turbines and boilers, there is an urgent need for the development of effective and stable catalysts for natural gas combustion. Palladium has been reported to be the most active catalyst for methane combustion when operating in excess of oxygen [1]. Palladium dispersion and palladium-support interaction play an important role on catalytic performance [2]. In addition Ce and Zr are effective promoters for noble-based catalysts because they can enhance dispersion, stability and oxygen storage capacity [3]. In this work, silica is prepared by sol-gel method in order to have high surface area. Consequently, attention is given only to the effect of palladium amount on the structure and activity of the $Pd/Ce_xZr_{(1-x)}O_2/SiO_2$ catalyst in methane combustion.

$Pd/Ce_xZr_{(1-x)}O_2/SiO_2$ catalysts (0.25, 0.5, 0.75 and 1 wt.% Pd) were prepared by combining sol-gel and impregnation methods. Palladium acetate, zirconium oxynitrate, cerium nitrate, tetraethylorthosilicate (TEOS) were used as precursors. N_2-physisorption, H_2-chemisorption, MET-EDX, DRX and TPR are the main techniques used to characterize these catalysts. Furthermore, total methane oxidation was used to test the catalytic activity of the prepared solids.

The results of nitrogen physisorption characterization, show that all adsorption-desorption isotherms are of type IV with a H4 hysteresis, which characterize mesoporous solids with cylindrical porous and a different average pore distributions. The XRD, UV-Visible, MET-EDX, and TPR results show that the particle size of transition metal oxides and the reduction temperatures of the PdO phase depend greatly on the palladium amount. The catalytic activity results show that the amount of palladium modifies remarkably the activity of the catalytic sites and that an optimum of palladium loading (0.5%) is necessary to maximize activity. Globally, the improvement of palladium dispersion and PdO reducibility increases the activity in methane combustion.

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Preparation of Highly Substituted Oxazol-2-ones: Reaction of 1,3-Oxazolidin-2-ones with NBS

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Nitrogen-containing heterocycles occur in various bioactive natural products and synthetic drugs. In particular, oxazolidin-2-ones[1] and oxazol-2-ones[2] have received extensive attention owing to their potential as versatile starting materials for various organic reactions and their presence in a large number of natural products and pharmacologically active agents. In this work, we have developed a facile and efficient two-step approach for tri-substituted functionalized oxazol-2-ones **3**. The first step involves the conversion of the corresponding amino alcohols **1** into *cis*-1,3-oxazolidin-2-ones **2** under mild conditions. These cyclic carbamates were then reacted with N-bromosuccinimide (NBS) in the presence of light to afford, through a one-pot bromination-dehydrobromination, the desired oxazol-2-ones **3** (Fig.1).

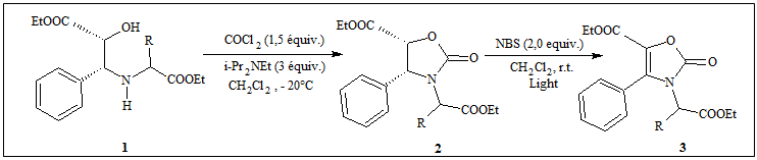

Fig.1 Synthesis of Oxazol-2-ones from Amino alcohol diesters via *cis*-1,3-Oxazolidin-2-ones

Keywords: β-Amino alcohol diesters, 1,3-Oxazolidin-2-ones, Oxazol-2-ones, N-bromosuccinimide.

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ULTRASOUND EFFECT ON SUZUKI REACTIONS USING PALLADIUM-IRON CATALYST IN DMF/WATER

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A simple and efficient procedure for Suzuki coupling of aryl bromides with aryl-boronic acids catalyzed by in situ-generated palladium-iron nanoparticles in water/DMF without any ligand in open air to produce a variety of functionalized biaryls and aryls has been developed. The boronic acids act here as the reducing agent for the formation of Pd-Fe nanoparticles. The reactions are remarkably fast under ultrasound irradiation [1] has been developed. The reaction was carried out at room temperature under ballon pressure of argon and gave the products in good yields. The regioselectivity of the studied reaction, in each case, was based on the ^{1}H NMR data. In this context, we propose the reaction mechanisms for these reactions in the presence of a catalyst.

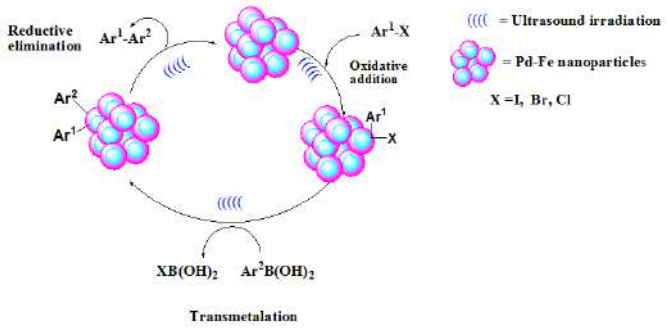

Fig. Proposed catalytic cycle for Suzuki cross-coupling reactions.

Key words: Suzuki coupling, Pd-Fe Nanoparticles, Ultrasonic activation, biaryls.

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CRYSTALLISATION, THERMAL ANALYSIS AND ACETAL PROTECTION ACTIVITY OF NEW LAYERED Zn(II) HYBRID POLYMORPHS

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Two new polymorphic mononuclear complexes, with analogous structural formula $(C_6H_9N_2)_2[Zn(H_2O)_6](SO_4)_2\cdot 2H_2O$, of zinc(II) templated by 2-amino-6-methylpyridinium ligand have been discovered. These polymorphic hybrid crystals, which differ only in terms of their crystal structures, were prepared in one step synthesis under ambient conditions and investigated for their thermal and catalytic properties. Single crystal X-ray diffraction of the obtained materials revealed that polymorphs 1 and 2 crystallise in the triclinic system, space group *P*-1. In an effort to further explore the form diversity of these compounds, the structural arrangements and intermolecular interactions such as hydrogen-bonding and π···π interactions are discussed from which supramolecular assembly was formed. Meanwhile, these new polymorphic forms can be described as the stacking of 3D layers where the interlayer distances are 13.23 and 12.59 Å for 1 and 2, respectively. The thermal behaviour of the precursors checked by TG-DTA analysis for both zinc sulfate polymorphs and variable temperature powder X-ray diffraction (VT-PXRD) show successive intermediate crystalline anhydrous phases upon dehydration. The catalytic activity of both polymorphic structures has been tested in the acetalisation reaction of aldehydes as a benchmark process. Interestingly, both complexes showed excellent activity with almost total conversion in many examples and using MeOH as solvent and as the unique source of acetalisation.

Key words: Polymorph hybrid materials, Crystal packing, Intermolecular interactions, lamellar structure, Thermal stability and Catalytic properties.

Relaxor behaviorin the Perovskite-type $Bi_{0.5}(Na_{1-x}Li_x)_{0.5}TiO_3$ ($0 \leq x \leq 0.2$) solid solution

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In the search of lead – free ferroelectric ceramics with improved properties, bismuth sodium titanate $Na_{0.5}Bi_{0.5}TiO_3$ (NBT) was thoroughly investigated in our group. Solid solutions of the perovskite-type compounds, with $Bi_{0.5}(Na_{1-x}Li_x)_{0.5}TiO_3$, formula, were prepared by solid state reaction. The results of X-ray diffraction (XRD) data evidenced for a single perovskite phase, with a solubility limit of (Na,Li) estimated to $x \approx 0.2$. The dielectric properties of sintered ceramics were investigated in wide temperature (25-800°C) and frequency (1kHz-1MHz) ranges. For low substitution rate, ($0 \leq x \leq 0.2$) the compounds exhibit diffuse phase transition and are characterised by high Curie temperature and a relaxor – like behavior. In the high temperature range, the thermal variation of permittivity is well described by a law $\frac{1}{\varepsilon} - \frac{1}{\varepsilon_m} = C(T - T_m)^{\gamma}$, where γ is close to 1.5. Such a relaxor behavior is interpreted in terms of cation disorder, due to the statistical repartition ofNa and Li, orcorrelated to the relaxation of polar clusters induced by the Na substitution by Li in the A site of the perovskite unit cell.

Key words: dielectric properties, Perovskite phase, Piezoelectric, Ferroelectrics

VERBASCOSIDE AND DERIVATIVES ISOLATED FROM *CITHAREXYLUM SPINOSUM* L.

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Citharexylum spinosum L. also called *Citharexylum quadrangular* Jacq. is a tree belonging to the *Verbenaceae* family. It was traditionally used as a diuretic, an antipyretic and to treat liver, gastro intestinal tract and antiarthritic disorders. It has also known, among others, by its immunomodulatory and antimicrobial activities [1]. The literature survey prompted us to study the phytochemical properties of this plant and to isolate if possible some bioactive secondary metabolites. The phytochemical investigation of this plant led to the isolation of five phenylethanoid glycosides, named Verbascoside **(1)**, Leucosceptoside A **(2)**, Martynoside **(3)**, Isoverbascoside **(4)** and plantainoside C **(5)**. Their isolation was carried out using a silica gel column and high performance liquid chromatography (HPLC). Their structures were established by extensive spectroscopic means including 1D and 2D NMR and spectrometric ESI-HRMS analysis.

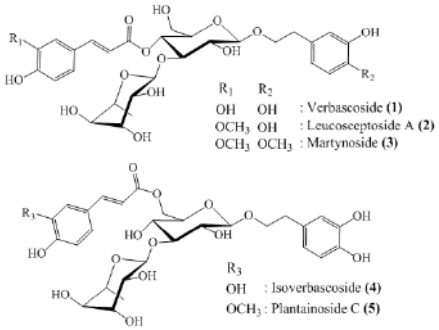

Fig. phenylethanoid glycosides isolated

Key words: *Citharexylum spinosum* L. Phenylethanoid glycosides, NMR.

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SYNTHESIS OF NOVEL SPIROOXINDOLOPYRROLIZIDINE LINKED 1,2,3-TRIAZOLES *VIA* CYCLOADDITION REACTION

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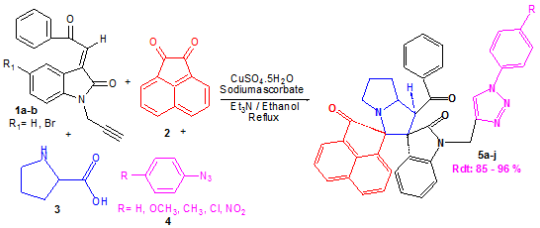
The 1,3-dipolar cycloaddition reaction of azomethine ylides with exocyclic alkenes and the Cu(I) catalyzed [3 + 2] cycloaddition of azides with triple bonds constitute facile approaches for the construction of five-membered nitrogen containing heterocycles [1]. Azomethine ylides can be generated *in situ* for the construction of highly functionalized spirooxindolopyrrolizidines which are featured in a large number of naturally occurring alkaloids, drugs and biologically active compounds [2]. On the other hand, the 1,2,3-triazole scaffold is a central component of numerous alkaloids as well as a great variety of synthetic compounds endowed with diverse bioactivities [3]. In the continuity of our studies on the development of cycloaddition reactions, we herein report the synthesis of highly diversified novel functionalized spirooxindolopyrrolizidine linked 1,2,3-triazole conjugates **via** a one-pot, four-component condensation of *(E)*-2-(1-propargyl-2-oxoindoline-3-ylidene)acetophenones **1**, acenaphthenequinone **2**, *L*-proline **3** and azides **4** using Cu(I) as a catalyst (**Scheme 1**). The stereochemistry of these *N*-heterocycles has been confirmed using an X-ray diffraction study.



Scheme 1. Synthesis of dispiropyrrolizidine linked with 1,2,3-triazoles

Key words: Spirooxindolopyrrolizidine, 1,2,3-Triazole, Dipolar cycloaddition.

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STRUCTURE-ACTIVITY TRENDS ANALYSIS OF SAPONINS IN THE PLANT GENUS ASTRAGALUS (FABACEAE)

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[c)] *Aix-Marseille Université, Marseille, France*

The genus *Astragalus* (Fabaceae) represents the most abundant plant taxon in the terrestrial world, and it is represented by 2500-3000 species distributed through the different continents. Chemical polymorphism and diversity of this wide genus have been subjected to several analyses of secondary metabolites which continuously revealed new molecules with different pharmacological activities. Among the invested secondary metabolites, saponins were found to be interesting by structural diversity, and various pharmacological activities. Structural diversity of saponins in *Astragalus* species are characterized by different hydrocarbon skeletons called aglycones. The most abundant saponin aglycone in *Astragalus* is cycloartane which can be ethoxylated. The different cycloartane skeletons can be further diversified by various substitution and different desmosylation levels, leading to the biosynthesis of saponins. Saponins of *Astragalus* were characterized by several pharmacological activities including immunomodulatory and cytotoxic properties. Structure-activity trends linking such pharmacological activities and several structural traits (including aglycone type, substitution types and levels) were statistically highlighted by means of ordination analysis and significance tests.

Key words: *Astragalus*; saponin; cytotoxic activity; immunomodulatory activity; structure-activity trends.

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Electrochemical Assisted Synthesis of Alkyl-2-(2-imino-4-oxothiazolidin-5-ylidene)Acetate Derivatives

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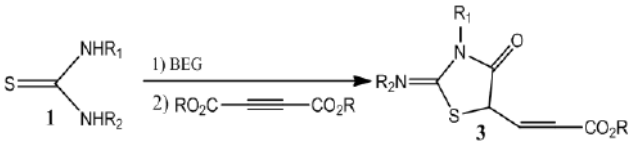
We report in a simple method for the synthesis of alkyl -2-(2-imino-4-oxothiazolidin-5-ylidene) acetate derivatives in good yields under mild conditions. The electrogenerated cyanomethyl base, obtained from electroreduction of neat acetonitrile assists the reaction between thiourea derivatives and dialkyl acetylene dicarboxylate. From the expected products **3/4**, the structure obtained from X-ray diffraction study confirmed that the main products are the five membred heterocycles **3**. Furthermore, a mechanism, to explain the reaction pathways, is proposed based on the thermodynamic data obtained from quantum calculations.



Keywords: alkyl-2-(2-imino-4-oxothiazolidin-5-ylidene) acetates; dialkyl acetylene dicarboxylate, thioureas; EGBs, X-ray, DFT calculation.

Enzymatic Synthesis of octyl acetate (Orange flavor) Improving the efficiency of the reaction by ultrasound

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University of Badji Mokhtar, Annaba - Algeria

Flavors and fragrances play an important role in food, pharmaceutical and cosmetic industries. This has led to the development of nature identical artificial flavoring compounds which can be used in different products to compensate the loss or boost the flavor. This work describes the study on the identification of nature identical artificial orange flavoring compound. This allows the development of artificial flavoring substances identical to natural ones and can be used in various industrial products.
This work describes the study on the identification of an identical artificial flavor than natural orange. The main objective of this study is the reaction yield of the synthesis of the ester of the orange aroma by enzymatic acylation of n-octanol with vinyl acetate in the presence of lipases (CRL and LPP).
The reaction was conducted without solvent to remain in the field of green chemistry. The obtained results allow us to optimize the reaction conditions that lead to better reaction yields. To enhance our study we also compared the ultrasound technical with and without ultrasound. The results obtained show that ultrasound technical may be a better and economical way to improve the reaction yield in the synthesis of octyl acetate (ester of the orange aroma) in less reaction time.

Key words: Transesterification, reaction yield, Ultrasonic synthesis, Lipase, Aromas.

Sythesis of β,β'-aminodiols and chemoselective protection of the amine group

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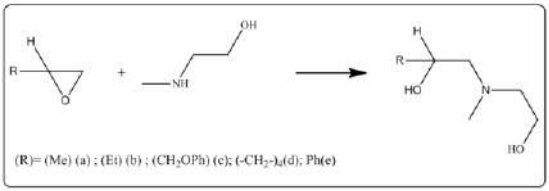
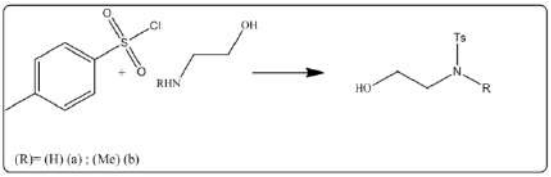
Epoxides are among the most versatile intermediates in organic synthesis because they can be subjected to diverse stereoselective transformations for the synthesis of higly valuable products.[1] The β-amino alcohols are bifunctional molecules that can be synthesized from the epoxide. They are also known under the name of β-blockers. They have received much attention in the pharmaceutical industry, given their importance in the treatment of human diseases [2]. In addition, they are involved in many biological processes and related to the numerous pathways to heterocycles [3]. This is the type of molecules that we were interested, to access to new β-amino alcohols (Scheme 1). We also succed to protect selectively with p-Toluenesulfonyl chloride too classes of amins in β-hydoxyamines (Scheme 2).



Scheme 1: Sythesis of β,β'-aminodiols



Scheme 2: Chemoselective protection of the amine group

Key words: Epoxide, 2-(Methylamino)ethanol, β,β'-aminodiols, p-Toluenesulfonyl chloride.

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(POLY)HALOBENZENESULFONYL CHLORIDES AS COUPLING PARTNERS: ONE STEP ACCESS TO (POLY)HALO-SUBSTITUTED BI(HETERO)ARYLS AND HECK PRODUCTS

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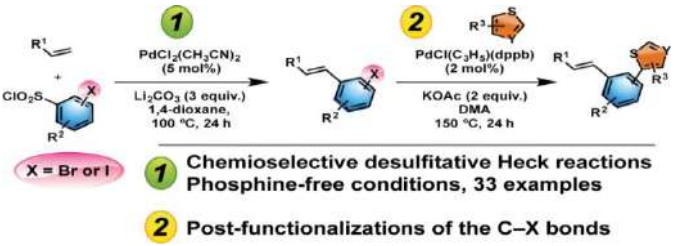
The reactivity of (poly)halo-substituted benzenesulfonyl chlorides for Pd-catalysed desulfitative arylation was investigated. 2-, 3- and 4-bromobenzenesulfonyl chlorides react nicely to afford arylated heteroarenes in moderate to high yields without cleavage of the C–Br bonds, allowing further transformations.Even 4-iodobenzenesulfonyl chloride, di- and tribromobenzenesulfonyl chlorides were successfully coupled with a range of heteroarenes and Styrene without cleavage of the C–Br or C–I bonds and very regioselective arylations were observed in all cases.



Key words: Palladium, catalysis, desulfitative Heck reaction, halobenzenesulfonyl chlorides, alkenes

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SYNTHESIS, CRYSTAL STRUCTURE, CHARACTERIZATION AND BIOLOGICAL ACTIVITIES OF A NEW DIMERIC TRINUCLEAR COPPER (II) COMPLEX: $Cu_3(C_{12}H_8N_2)_3P_6O_{18}$•2,5$H_2O$

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Single crystals of a new dimeric trinuclear copper (II) complex: $Cu_3(C_{12}H_8N_2)_3P_6O_{18}$•2,5$H_2O$, have been prepared by conventional aqueous solution synthesis, and characterized by X-ray diffraction, spectroscopy (Infrared, Raman and diffuse reflectance), thermal analysis, Hirshfeld surface analysis and antioxidant activities. The crystal structure determination reveals that the synthesized compound crystallizes in the triclinic space group $P\bar{1}$. Its crystal structure consists of dimmers with formula $[Cu_3(C_{12}H_8N_2)_3P_6O_{18}.H_2O]_2$ (Fig. 1a). Adjacent dimers are linked via hydrogen bonds (O-H…O and C-H…O) and π···π interactions as to form a three-dimensional network. The antioxidant properties were determined via the DPPH radical scavenging, ABTS radical scavenging, hydroxyl radical scavenging and ferric reducing power (FRP) using ascorbic acid as control. The results show that the synthesized compound possesses high antioxidant activities (Fig. 1b).

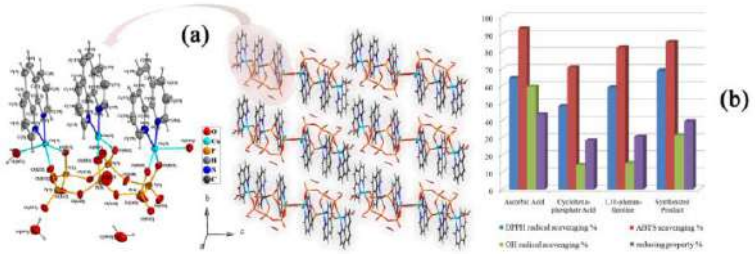

Fig. 1: (a) Atomic arrangement of $Cu_3(C_{12}H_8N_2)_3P_6O_{18}$•2,5$H_2O$ (b) Antioxidant activities

Keywords: Copper (II), cyclohexaphosphate; 1,10-phenanthroline, Crystal structure; Thermal analysis, Spectroscopy, Antioxidant activities.

Elaboration, structural study and validation (CHARDI and BVS) of a new variety β-$Na_9Cr(MoO_4)_6$ of alluaudite type.

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The title compound, β-$Na_9CrMo_6O_{24}$, was prepared by solid-state reaction, characterized by X-ray diffraction and MEB. The compound crystallizes in the monoclinic space group C2/c with a= 12.655 (2), b= 13.578 (2), c= 7.1405 (8), β (°) = 112.58 (2), V ($Å^3$) = 1132.9 (3), Z= 2, R (F2) = 0.028, Rw (F2) = 0.080. The structural unit of the compound β-$Na_9CrMo_6O_{24}$ consists of two tetrahedron MoO_4 and one octahedron MO_6 (M= Cr1/Na1) sharing by vertices. The Na^+ and Cr^{3+} are located in the same site with respectively 0.25 and 0.75 occupancies. The charge compensation is provided by Na^+ cations. This alluaudite-type structure [1-2] is constituted by an infinite layers formed by links between M_2O_{10} (M= Cr1/Na1) dimers and MoO_4 tetrahedra. The layers are related by sharing corners with MoO_4 tetrahedra leading to an open three framework with hexagonal-form cavities occupied by Na^+ cations.

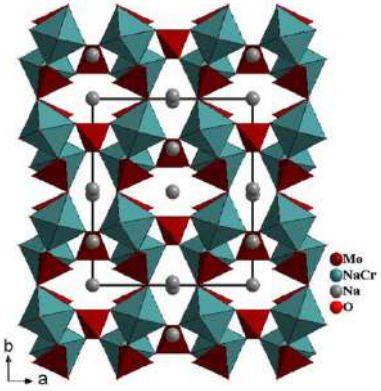

Fig. Projection of β-$Na_9CrMo_6O_{24}$ along the c axis.

Key words: Alluaudite structure, framework, crystal structure, BVS, CHARDI.

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Structural, Crystal Structure, Hirshfeld surface analysis and physicochemical studies of a new Chlorocadmate template by 1-(2-hydroxyethyl)piperazine

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In recent years, much attention has been paid to the design and construction of hybrid materials which represent one of the most fascinating developments in materials chemistry [1], fluorescent properties [2] and other potential applications [3] in molecular-based materials.

The synthesis, crystal structure and spectroscopic characterization of a new chlorocadmate template by the 1-(2-hydroxyethyl)piperazine ligand are reported. In these crystals, piperazinediium cations are in a chair conformation and are inserted between the inorganic layers through N-H…Cl, C-H…Cl, O-H…Cl and N-H…O hydrogen bonds to form infinite three-dimensional network (Fig. 1). Investigation of intermolecular interactions and crystal packing via Hirshfeld surface analysis reveals that H…Cl and C-H…H-C intermolecular interactions are the most abundant contacts of the organic cation in the crystal packing (Fig. 2). The crystal contacts enrichments reveals that the Cd^{++}…Cl^{-} salt bridges, the Cd…O complexation and O-H…Cl^{-} and N-H…Cl^{-} strong H-bonds are the driving forces in the packing formation. The ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray structure.

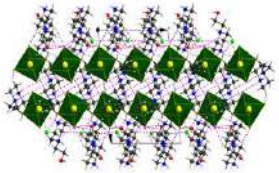

Fig. 1. Projection along the *b*-axis of the crystal packing of the title compound. The dotted lines indicate hydrogen bonds.

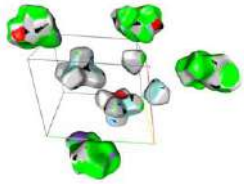

Fig. 2. View of the Hirshfeld surface around the individual entities (four cations, two $CdCl_5$ moieties, two Cl^{-} atoms).

Keywords: Crystal Structure; Hirshfeld surface, cadmium; NMR, IR

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Demonstration of an acid phosphate glass

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A new acid glass of potassium and copper phosphate is obtained by heat treatment at 560 K of a mixture of KH_2PO_4, P_2O_5 and CuCl. The glass obtained is characterized by X-rays and by Raman and IR spectroscopy. The X-ray diffractogram shows a halo of the diffusion towards (20-40) of 2Θ, characteristic of an amorphous structure. The Raman spectrum showed bands at 325, 497, 718, 904, 988, 1142, 1270 and 1329 cm^{-1}. The bands not coincide with any characteristic bands of KPO_3 (383, 671, and 1139 cm^{-1}), $K_2H_2P_2O_7$ (705 and 1041 cm^{-1}), and KH_2PO_4 (358 cm^{-1}, 916 cm^{-1}). By comparison, of the Raman and IR spectra with those of metaphosphate, polyphosphate and ultraphosphate, it appears that the product prepared is a polyphosphate. This hypothesis is confirmed by the presence of Raman lines at (1142, 718 cm^{-1}) and IR bands at (1216, 1102, 1125,893 and 770 cm^{-1}).

QUANTIFICATION OF THE NUCLEOPHILE-SPECIFIC PARAMETERS OF PYRROLIDINE IN MIXTURES ACETONITRILE-METHANOL

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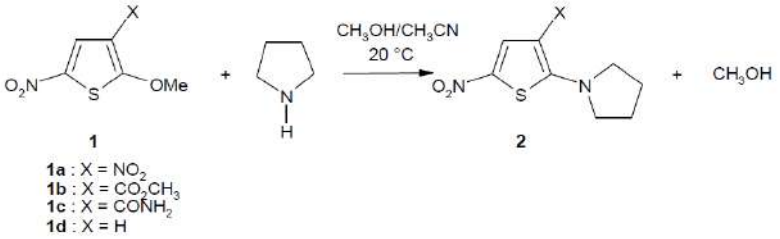
The kinetics of reaction of 2-methoxy-3-x-5-nitrothiophenes **1a-d** with pyrrolidine have been determined in various CH_3CN-CH_3OH mixtures at 20 °C. The resulting second-order rate constants have been employed to determine the nucleophile-specific parameters N and s of the pyrrolidine according to the linear free energy relationship (Eq. (1)).

$$\log k\ (20\ °C) = s_N\,(E + N) \qquad (1)$$

The N parameters are found to cover a range from 16.67 to 19.34 going from the most protic (CH_3OH) to the last protic solvent (CH_3CN).
As can be seen, linear corrolation betwen the nucleophilicity parameters N evaluated in the present work and the mole fraction of methanol values has been observed and discussed.



Key words: kinetics, S_NAr mechanism, Mary's equation, electrophilicity, nucléophilicitie

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STABILITY STUDY OF NIGELLA SATIVA LAYER FORMED ON THE STEEL SURFACE IN NEUTRAL SOLUTION

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El Mustafa Rezrazi[b], Ezzeddine Triki[a]

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The use of synthetic chemical compounds as corrosion inhibitor has proved to be highly efficient. Nevertheless, it presents a health and environmental hazard with high toxicity rates [1]. As a result, studies were conducted to extract safer molecules with protective ability against metal corrosion from natural products [2]. The inhibition of corrosion of mild steel in an aerated 0.5 M NaCl solution was studied using electrochemical impedance spectroscopy in the presence of different amounts of aqueous extract of *Nigella Sativa* seeds. The presence of this mixture in the solution decreases the corrosion current density. Long term immersion showed that lower amounts are more efficient than higher ones. And the inhibitor layer is more stable for smaller amount after 18 hours of immersion. The surface of examination and analyses confirm that a layer is formed on the metal surface.

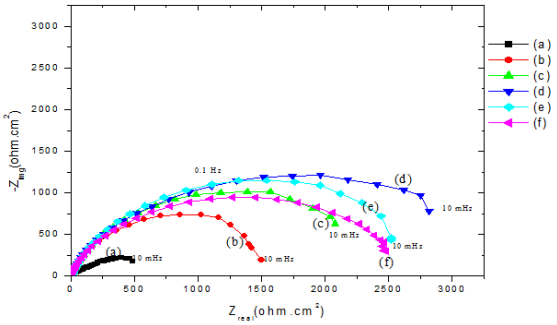

Fig. Impedance diagrams of the mild steel immersed in 0.5 M NaCl solution containing different NE concentrations;
a: 0 g.L^{-1}, b: 0.07 g.L^{-1}, c: 0.35 g.L^{-1}, d: 0.7 g.L^{-1}, e: 1.4 g.L^{-1}, f: 2.1 g.L^{-1}

Key words: Corrosion, EIS, Nigella Sativa, mild steel, inhibition.

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Selective Synthesis of Imidazolidine-2-thiones via ring expansion of Aziridine-2-carboxylates with Isothiocyanates

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Five-membered ring heterocyclic systems prepared from the reaction of aziridines with heterocumulenes have been widely studied due to their diverse biological activities[1]. A variety of N-substituted isothiocyanates were employed for the synthesis of imidazolidine-2-thiones **2**, involving the ring expansion of N-alkylaziridine-2-carboxylates **1**. The latter undergo ring opening and cyclization via completely regio- and stereoselective processes to afford the target *trans*-imidazolidine-2-thiones **2** and the *trans*-imidazolidine-4-thiones **3**, depending on the steric and electronic effects of the N-substituents on the aziridines and isothiocyanates (Fig.1).

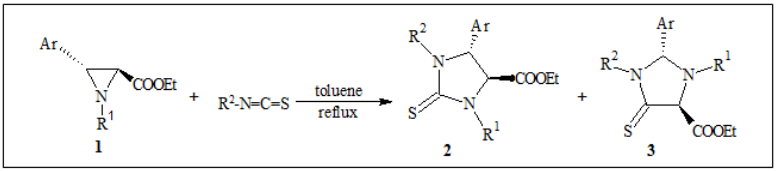

Fig.1 Synthesis of imidazolidine-2-thiones

Keywords: aziridines-2-carboxylates, imidazolidine-2-thiones, imidazolidine-4-thiones.

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GEOPOLYMERIC MORTARS BASED ON TUNISIAN CLAY FOR THE REPAIR OF CONCRETE STRUCTURES DAMAGED

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Fernando Pacheco-Torgal, José Barroso de Aguiar, Samir Baklouti

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Université de Sfax - Tunisie

The production of ordinary cement generates a huge amount of CO_2 (producing 1 tonne of ordinary Portland cement emits one ton of CO_2). To reduce the amount of CO2 emissions, a new geopolymer cement is developed as an alternative for the substitution of ordinary cement in construction. The geopolymeric cements are obtained by alkaline attack of an aluminosilicate material as metakaolin. In this study, mortars geopolymeric based on Tunisian clay from Medenine region, fly ash and metakaolin are prepared through using of an activating solution consisting of sodium silicate and calcium hydroxide and sodium hydroxide. These mortars are intended for the rehabilitation of concrete structures deteriorated. The mechanical characterization of these mortars showed that Tunisian clay mortars calcined possess sufficient mechanical performance to ensure rehabilitation.

On the reactivity of 3-(bromomethyl)butenolide: Selective preparation of *bis*-butenolide adducts

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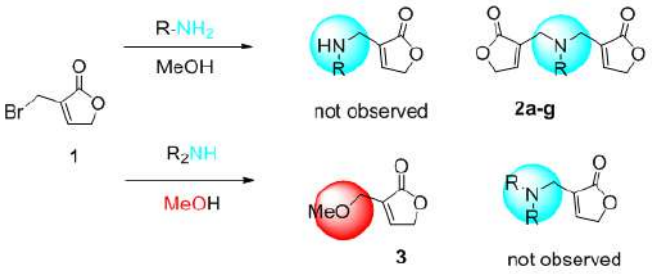
Substituted butenolides represent an important class of heterocyclic compounds, which can be considered as the basic core in a large number of natural products. The presence of the butenolide scaffold is also known to be associated with several biological activities.[1] The development of efficient methodologies to install various fragments at the butenolide core remains of high interest. In this communication, we describe the first lines of our study devoted to the coupling of various amines and 3-(bromomethyl)butenolide **1** as a common precursor. The reaction variety of primary amines in the methanol cleanly and selectively afforded 3,3'-arylamino *bis*-butenolide adducts **2**. In deep contrast, the use of secondary amines did not afford the expected amino derivatives but instead led to the formation of a C-O bond. The reactivity of the C-Br bond in butenolide **1**, is currently under investigation to extend the scope of our methodology.



Key words: 3-(Bromomethyl)butenolide, primary amines, 3,3'-arylamino *bis*-butenolide, 3-(methoxymethy)-5*H*-furan-2-one.

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PREPARATION AND CHARACTERIZATION OF NANO FIBER OBTAINED BY ELECTROSPINNING OF MIXED POLYMER / MOFs

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A novel class of extended metal–ligand networks with metal nodes and bridging organic ligands are called coordination polymers[1], also named as metal organic frameworks (MOFs) and were popularized by Yaghi et al. around 1995 [2, 3].
These compounds have attracted enormous attention due to their highly designable structure [1], their large pore surface area ranging from 1000 to 4500 $m^2 g^{-1}$ [4], pore size [5], and highly diversified structures and facilely tailorable functionality [6].
Typically, MOFs are synthesized by the assembly of inorganic units (isolated metal–atoms such as metal–oxygen clusters, chains or layers) with functionalized organic linker-molecules. The design network leads to the formation of one, two and three-dimensional extended frameworks [7].
The families of MOFs can be used for the synthesis of continuous membranes [8-9]. For this reason, some organic polymers have been developed for the deposition or blending of MOFs [6].
Thereby, Electrospinning is a new technique to produce continuous fibers with average diameters in the range of nanometers to a few micrometers [10].
In this work we combine the simplicity of electrospinning method and the advantages of MOFs materials to produce new hierarchical nanostructures mats with a total interior surface accessible. Thus, we describe in this paper the use of electrospinning technique to prepare fiber mats of PVA containing high loadings of MOFs particles and investigate their properties by performing their characterization using Fourier transform infrared spectroscopy, powder X-ray diffraction and scanning electron microscopy (SEM).

Keywords: MOFs, PVA, electrospinning

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EFFICIENT ROAD TO PREPARE C3-ARYLATED IMIDAZO[1,2-b]PYRIDAZINES DERIVATIVES

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In recent years, Pd-catalyzed direct arylation of several heteroaromatics using aryl halides, *via* a C−H bond activation, has become one of the most environmentally friendly methods for the C-C bond formation.[1] Phosphine-free $Pd(OAc)_2$ was found to catalyze very efficiently the direct arylation of imidazo[1,2-*b*]pyridazine at C3 position under a very low catalyst concentration. The reaction can be performed employing as little as 0.1–0.05 mol% catalyst with aryl bromides and chlorides. Moreover, examples of those couplings were reported using green, safe and renewable solvents without loss of efficiency.[2]

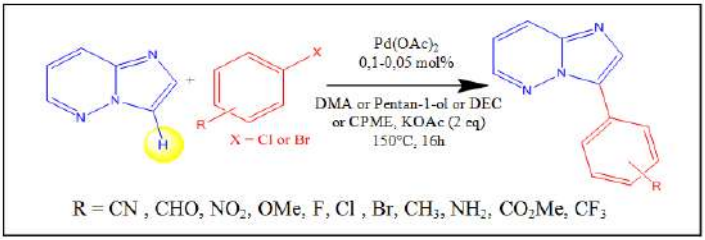

Fig. Scope of Aryl Bromides or Chloride in Pd-Catalyzed Direct Arylation of Imidazo[1,2-*b*]pyridazine.

Key words: Palladium, imidazo[1,2-b]pyridazine, green solvent

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DESIGN AND SYNTHESIS OF SET OF A MITOCHONDRIA-TARGETED AND APPLICATION IN PHOTODYNAMIC THERAPY

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The treatment of cancer with photodynamic therapy (PDT) based on the combination of photosensitizing molecules (Ps) which are able to concentrate in tumor cells, a focused light suitable wavelength (dependent on the Ps) and exciting molecular oxygen into its singlet form. This will be the combination of these three factors which will specifically target tumor tissue and to destroy them. Porphyrines are among the most promising photosensitizers to be developed.
However most of the chemists prepare many kind of photosensitizers type phthalocyanines and porphyrines but they find same problem of solubility and targets tumor cells [(1),(2)]
In these works, a set of hydrophobic Porphyrines are synthesised. And a developing of a mitochondrial addressing strategy by the controlled grafting of specific cationic groups will be the aim of an application in photodynamic therapy.

Keywords: Porphyrines, hydrophobic, photodynamic therapy of cancer

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CRYSTAL STRUCTURE, INVESTIGATION, DFT STUDIES AND THERMAL CHARACTERIZATION OF COBALT(II) COMPLEX WITH PIPERAZINEDIIUM CATION AS LIGAND

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The crystals of the new organic–inorganic material $(C_4H_{12}N_2)[CoBr_4]$ were grown by slow evaporation technique in aqueous solution and characterized by X-ray diffraction, infrared absorption, raman spectroscopy scattering, and thermal analysis. The structure is built from isolated $[CoBr_4]^{2-}$ anions and pepirazinediium $[C_4H_{12}N_2]^{2+}$ cations which are connected by a network of N-H …Br hydrogen bonds. Theoretical calculations were performed using density functional theory with the B3LYP/LanL2DZ [1] level for studying the molecular structure and vibrational spectra of the title compound. Good adhesion is observed between the calculated and experimental results of the structure, the IR and Raman spectra.

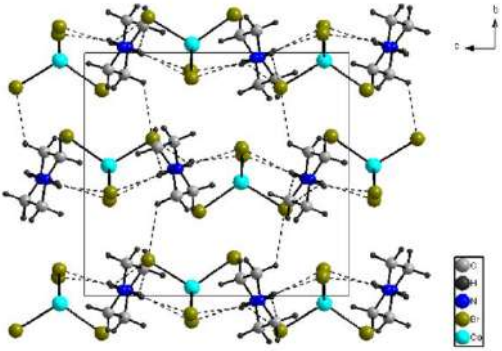

Fig.1 Projection of the crystal structure of $(C_4H_{12}N_2)[CoBr_4]$ along the a-axis.

Keywords: Crystal structure, DFT calculations, IR and Raman spectroscopy. U. V visible.

Reference

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COPPER N-HETEROCYCLIC CARBENE COMPLEX CATALYZED CLICK REACTION FOR SYNTHESIS OF TRİAZOLES

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Since the first Cu-NHC complex was discovered in 1993 by Arduengo and co-workers,[1] Cu-NHCs have been attracting continuous attention and a number of copper-NHC complexes have been synthesized so far.[2] The structural diversity, ease of modification as well as the low cost of Cu-NHCs offer excellent opportunities for their versatile applications[3]. The use of Cu-NHCs such as [(NHC)CuX] type complexes or their derivatives has proven successful in homogeneous catalysis. In this context we reported in this work, firstly, the synthesis of a new series of copper N-heterocyclic carbene complexes from different benzimidazolium based N-heterocyclic carbene salts using copper(I)oxide in water and under air. Then, to show usefulness of these comlexes we investigated the catalytic application of these complexes in click reaction for synthesis of triazoles. **Figure 1**

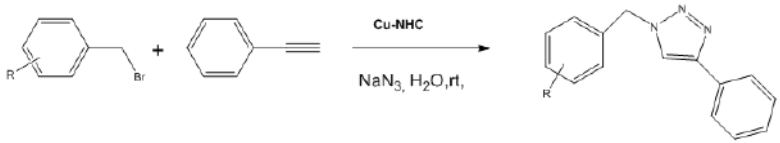

Figure 1: Cu-NHC catalyzed Huisgen Dipolar Cycloaddition of azides and alkynes

Key words: Cu-NHC carbene complexes, homogeneous catalysis, cycloaddition, click reaction.

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Simultaneous quantitative determination of ketamine, codeine and morphine and identification of 6-monoacetylmorphine and normorphine in plasma by gas chromatography/mass spectrometry

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A gas chromatography/mass spectrometry (GC/MS) method was developed and validated for the determination of ketamine, morphine and codeine and for identification of 6-monoacetylmorphine and normorphine in plasma.
This technique has been applied for the determination of ketamine and morphine in patients nephrectomized and receiving ketamine as an anesthetic and morphine as analgesic. Our method is applicable to pharmacological and toxicological studies on humans.
Protein precipitation was obtained by the addition of acetonitrile. The samples were extracted by methyl-t-butyl-ether (MTBE). After freezing, the supernatant was evaporated using a stream of nitrogen. The residu is then derivatized using N-methyl-N-trimethylsilyl-trifluoracetamide (MSTFA).

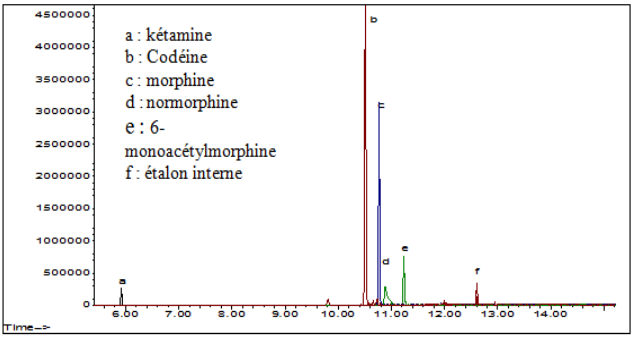

Fig. Chromatogram obtained after derivatization with the MSTFA

Key words: GC-MS, plasma, ketamine, morphine, codeine, 6-monoacetylmorphine, normorphine

SYNTHESIS OF MIXED ANHYDRIDES OF FATTY ACIDS: STABILITY AND REACTIVITY

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Mixed anhydrides obtained from the reaction between two different carboxylic acids are of an increasing interest in the chemical industry due to their high reactivity [1]. Unsymmetrical anhydrides, characterized by the general formula $R_1COOCOR_2$, can be easily prepared by the reaction of the fatty acid (R_1) with the acid chloride (R_2) in organic solvent and in the presence of triethylamine as a Bronsted base. These anhydrides should be stable and react as acylating agents.

Key words: fatty acid, mixed anhydride, symmetrical anhydride, stability, esterification.

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Synthesis and physico-chemical studies of a novel organic-inorganic hybrid material: $(C_3H_{12}N_2)CrO_4$

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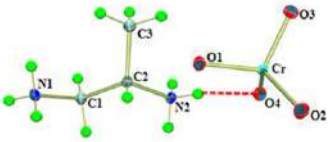
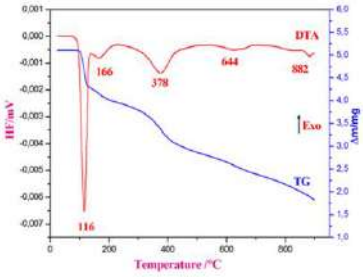
A novel organic inorganic hybrid material $(C_3H_{12}N_2)CrO_4$ **(I)** was synthesis by slow evaporation method and studied by means of a single-crystal X ray diffraction, Hirshfeld surface analysis, IR spectroscopy, UV-visible spectroscopy and thermal analysis.

The crystal structure of propane-1,2-diammonium chromate(VI) crystallizes in the monoclinic space group ($P2_1/c$, **Z** = 4) with unit cell constants a = 5.6462(2) Å, b = 15.8373(5) Å, c = 8.4442(3) Å, β = 106.779(1) ° and V =722.94(4) $Å^3$. The asymmetric unit of (I) consists of one chromate anion and one propane-1,2-ammonium dication. The structure of the compound consists of discrete chromate ions stacked in layers parallel to the (100) plane, separated by organic cations. The structural cohesion is established by a three-dimensional network of N—H…O and C—H…O hydrogen bonds. Furthermore, Hirshfeld surfaces and two-dimensional fingerprint plots estimate the intermolecular interactions accountable for the generation of crystal packing. The room temperature FT-IR of the title compound was analyzed. The UV-visible absorption spectrum is characterized by two strong absorptions assigned to the n→σ*, n→ π^*, the third band corresponds to d-d transitions of anions. Moreover, differential and thermogravimetric analysis (DTA/TG) has revealed a melting transformation, at the same time; the material begins to undergo a continuous decomposition in a wide temperature range.



An ORTEP view of $(C_3H_{12}N_2)CrO_4$



DTA/TG curves of **(I)**.

SYNTHESIS OF NEW PHOSPHONOAMIDE AND PHOSPHONOCAPROLACTAM DERIVATIVES VIA THE DIETHYL CHLOROPHOSPHATE-PROMOTED BECKMANN REARRANGEMENT OF γ-PHOSPHONYLOXIMES

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Phosphonoamides are well known by their promising applications as antibacterial, insecticide [1,2]. Some of them are also useful as antiwear and friction-reducing additives for lubricants and liquid hydrocarbon fuels [3]. On the other hand, phosphonocaprolactams are found to be used as modifying additives in the synthesis of phosphorus-containing polyamide-6 which have an improved ability for flame retardancy, as well as thermo- and thermooxidative resistance [4].

Herein, we report an efficient and straightforward synthesis of new phosphonoamide and phosphonocaprolactam derivatives, via the Beckmann rearrangement of γ-phosphonyloximes. The reaction proceeds smoothly in presence of diethyl chlorophosphate as a promoter, to afford the title compounds in satisfactory yields. A mechanistic rationalization of this reaction is provided by the prediction of its regiochemistry.

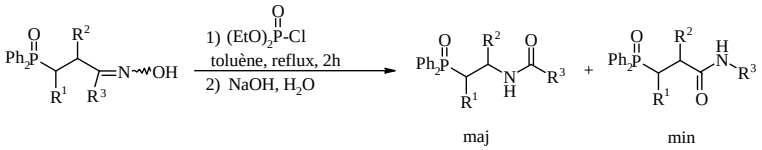

Fig. Synthesis of phosphonoamides and phosphonocaprolactams

Key words: Beckmann rearrangement, phosphonoamides, phosphonocaprolactams, γ-phosphonyloximes, diethyl chlorophosphate.

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EXPERIMENTAL AND DFT CHARACTERIZATION OF THE ORGANIC-INORGANIC ZN(II) COMPLEX WITH PIPERAZINE LIGAND,$(C_4H_{12}N_2)[ZNBR_4]$

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During the past few decades, self-assembly of organic and inorganic molecules has attracted much attention in chemistry, physics and material science owing to their abilities to combine the desired properties of organic and inorganic compounds, so as to exhibit some interesting crystal structures and special properties, such as second order nonlinear optical (NLO) response, magnetism and their technologic applications [1–3]. In the present investigation, we report the synthesis, crystal structure and FT-IR and FT-Raman spectroscopies of the title compound $(C_4H_{12}N_2)[ZnBr_4]$. The structure consists of inorganic entities $[ZnBr_4]^{2-}$ and organic moieties of $(C_4H_{12}N_2)^{2+}$, forming mixed organic–inorganic layers in (b, c) planes. Theoretical calculations were performed using density functional theory (DFT) for studying the molecular structure, vibrational spectra and optical properties of the investigated molecule in the ground state.

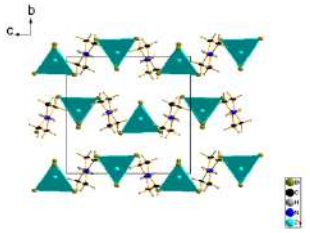

Fig. 1 : Projection of the crystal structure of $(C_4H_{12}N_2)[ZnBr_4]$ along the a-axis.

Keywords : Crystal structure; DFT calculations; IR and Raman spectroscopy.

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Structural, microstructural and thermal properties of nanostructured $Fe_{60}Al_{35}Sn_5$ alloy synthesized by mechanical alloying

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The process of nanocrystalline structure formation during mechanical milling was studied in Fe, Al and Sn powders and prepared in a high-energy ball mill under argon atmosphere. A detailed microstructural and morphologic study of powder samples was performed by X-ray diffraction experiments and scanning electron microscopy over time milling (in the 0-60 h range). Thus, analysis breaths line was performed to get an idea about the evolution and impact of the grain size, lattice strain and dislocation on the microstructure of powders. Experimental results demonstrated that a Bcc-Fe (Al, Sn) solid solution with a mean crystallite size about 16 nm was achieved. In addition, the thermal behavior of the milled powders was examined by differential scanning calorimetry (DSC).

STUDY OF THE LEAD RETENTION ON CACTUS RACKETS

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For the last decades, the tremendous industrial, agricultural and domestic wastes charged with contaminants have caused a dramatic pollution of the water all over the world. Over the years, the management of these pollutants has become a major environmental concern. To overcome this problem, various depollution techniques have been developed. Among these techniques, the retention of pollutant on a solid natural support is an effective and inexpensive alternative. In this work, the retention process of Pb(II) cations on prickly pear rackets (Ficus Indica Opunita) was studied. In particular, the influence of the process parameters (pH, adsorbent mass, contact time) on the adsorption rate was examined. A maximum retention rate (95%) of Pb(II) ions was obtained after 20 minutes of treatment, at a pH of 6.9, and with 0.1g of cactus fibers. The thermodynamic parameters ($\Delta H°$, $\Delta S°$, $\Delta G°$) were also determined, and the results showed that the adsorption is spontaneous and endothermic.

Keywords: Cactus racket, Retention, Lead ion, Adsorption rate.

References

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Recovery of High Yield Flavonoids Rich Extract from Two-phase Chemlali Olive Pomace

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Increasing interest in phenolic compounds in olive cake is due to their antioxidant and health-enhancing properties. In this study, the flavonoids in two phase olive pomace of the Tunisian olive cultivar Chemlali, were extracted by ethanol-water to obtain a flavonoid rich extract. The identification and quantification of flavonoids was based on separation by a high-performance liquid chromatography equipped with a diode array detector followed by liquid chromatography-mass spectrometry analysis. Five flavonoids were isolated andpurified using HPLC apparatus. Hesperidin and quercetin-3-*O*-arabinoglucoside were in high amounts (15.60 and 21.71 mg/Kg of fresh weight, respectively) followed by quercetin and luteolin (8.12 and 11.01 mg/Kg of fresh weight, respectively). The antioxidant activity of purified flavonoids was evaluated by measuring the radical scavenging effect on DPPH and by using ferric reducing antioxidant power assays. Bactericidal and fungicidal effects were determined using the NCCLS broth dilution. The minimal cidal concentrations (MCCs) values of ethyl acetate extract of two-phase Chemlali olive oil are ranged from 1.825 to 14.6 mg/mL and tested against pathogenic and phytopathogenic microbes. This activity was related to the richness of the extract of flavonoid compound especially quercetin-3-*O*-arabinoglucoside and hesperidin.

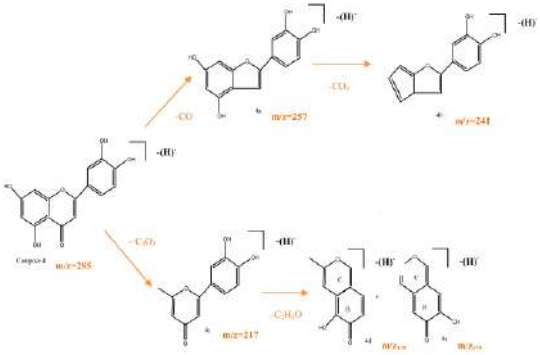

Fig. Proposal fragmentation of luteolin

Keywords: flavonoids, Chemlali, hesperidin, olive pomace, antioxidant activity.

Horner-Wadsworth-Emmons olefination mediates synthesis of novel Butadienylphosphonates derivatives

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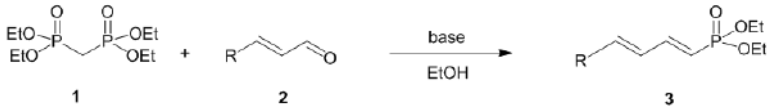
It's well established that 1,3-dienes are highly valuable and versatile reagents for the synthesis of a variety of cyclic and heterocyclic compounds. [1] We herein report a rapid access to novel series of substituted conjugated dienes using a straightforward approach based on the utilization of Horner-Wadsworth-Emmons (HWE) olefination [2] of unsaturated aldehydes as precursors. The reaction of methylenebisphosphonate anion of **1** with a variety of α,β-unsaturated aldehydes **2** proved successful and allowed to the synthesis of novel family of conjugated 1,3-dienes 3 featuring a terminal vinyl phosphonate moiety, isolated in good yields.



1 2 3

Key words: methylenbisphosphonates, HWE olifination, α,β-unsaturated aldehydes, butadienylphosphonates

References:

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Synthesis, Characterization and Combined Kinetic analysis of thermal decomposition of Hydrotalcite ($Mg_6Al_2(OH)_{16}CO_3.4H_2O$) under air atmosphere.

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[2] *Instituto de Ciencia de materiales de Sevilla, Espagne.*

TG study of synthesized hydrotalcite, $Mg_6Al_2CO_3(OH)_{16}\cdot 4H_2O$, was carried out at four rates of temperature linear increase and under air atmosphere. The kinetic parameters and the mechanism of its solid-state thermal decomposition reaction were evaluated from the TG data using combined kinetic analysis method. It was shown that thermal decomposition up to 650°C occurs in two separated stages. The first one is simple and corresponds to the release of four water molecules. The second stage is complex and corresponds, respectively, to the release of the hydroxyl groups and carbonates. The originality here is the kinetic study of each process of thermal décomposition of hydrotalcite. The apparent activation energies of dehydration, dehydroxylation and decarbonation are, respectively, equal to 149 $kJ.mol^{-1}$, 300 $kJ.mol^{-1}$ and 259 $kJ.mol^{-1}$. Dehydration and decarbonation occur according to three-dimensional diffusion (D3) mechanism while dehydroxylation process occurs according to two-dimensional diffusion (D2) mechanism.

Keywords: Hydrotalcite; TG; activation energy; combined kinetic analysis.

THERMOCHEMISTRY AND KINETIC STUDY OF THE SULFO-PHOSPHORIC ACID ATTACK OF PHOSPHATE ROCK

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Ismail KHATTECH and Mohamed JEMAL

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In previous work, we studied the effect of the solid / liquid ratio on thermochemical and the nature of the products formed after the sulfo-phosphoric attack a merchant phosphate used by SIAPE for the synthesis of phosphoric acid and phosphates' fertilizers in particular TSP. The results show that whatever ratio, the attack is accompanied by an exothermic phenomenon. Moreover, its gradual increase of this ratio leads to the formation of hemihydrate or dihydrate or a mixture of these two solid.

The present work is a contribution to the study by calorimetric (DRC) of the effect of temperature on the sulfo-phosphoric attack reaction of a natural phosphate (NP). For this, this parameter was varied from 25 ° C to 60 ° C in conditions very close to those used by the industry. Indeed, the etching solution contains 20% by volume of sulfuric acid (96% acid by weight) and 80% phosphoric acid solution at 20% by weight of P2O5.

The thermochemical study of the variation of the amount of heat ΔH_{mes} calculated by integration of the raw signal, depending on the temperature in the range 25-60 ° C shows that the overall enthalpy of the reaction increases in absolute value up 'at a temperature of 45 ° C and then it undergoes a fracture. This behavior was attributed to change of reaction mechanism of change of either side of this temperature by several authors [1-3]. Similarly, the kinetic study in this temperature range shows that the etching rate decreases with increasing temperature. In addition, the representation of Ln(k) = f (1/T) leads two different activation energy values of either side of this temperature (T = 45°C).

Keywords*:* Phosphate ore, Differential Reaction Calorimeter, Kinetic, Activation energy ·

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Energy storage property in lead free Gd doped $Na_{1/2}Bi_{1/2}TiO_3$ ceramics

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The Effect of Gadolinium ion incorporation on structure, dielectric and ferroelectric properties of lead-free $Na_{1/2}Bi_{1/2}TiO_3$ (NBT) ceramic was investigated. X-ray diffraction allowed the identification of a pure phase isostructural to NBT. Dielectric measurements showed more pronounced anomalies in the range of depolarization temperature when Gd was added. Antiferroelectric-like behavior with a double pinched hysteresis loop was observed versus temperature in the doped phase. The energy-storage density (W) was calculated using the P-E loops data and was found to vary from 0.45 $J.cm^{-3}$ at room temperature to 0.85 $J.cm^{-3}$ at 413 K, which is promising for energy storage application.

Keywords: Lead–free Sodium Bismuth Titanate, Ceramics, Energy storage.

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Gamma radiation induced degradation of Indigo Carmine dye on aqueous solutions

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In this study, the possibility of using gamma rays to degrade or decolorize reactive dye in water was investigated. A reactive dye, Indigo Carmine, in aqueous solutions was irradiated at doses of 0.1–5 kGy. The change of absorption spectra, the chemical oxygen demand (COD) was examined. The absorption bands at 611 and 297 nm decreased rapidly with increasing irradiation dose. The COD reduction for the dye solutions was approximately 90-95% at 1 and 5 kGy. Finally, a kinetic study based on spectrophotometric measurements showed that the degradation process is pseudo first order.

Keywords: gamma irradiation, Indigo carmine, COD

THEORETICAL LIMITS OF OPTICAL FIBRE CONICAL SOLAR FURNACE

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University of Tlemcen, Algeria (a)

This paper reports on an theoretical realization of a recently proposed solar optical fiber mini dish light concentrator connected to a conical solar furnace. The prototype dish is 21.8 cm in diameter. We present the daily power obtained at the output of the optical fiber, the power supply is estimated to be 25 W at the end. Then we show that the energy transported is diffused until the enclosure then disperse inside it, this energy is absorbed by the receiver. Temperatures higher than 800°K may be reached while maintaining very good efficiency.

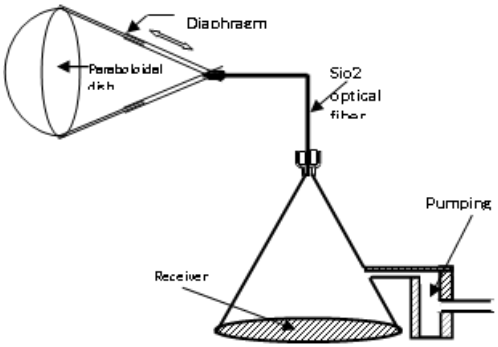

Fig. The coupling between the concentrator, the optical fiber and the solar furnace proposed

Key words: solar energy ; optical fiber; paraboloidal dish; conical solar furnace

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FACILE SYNTHESIS OF NEW PYRAZOLO[1,5-a][1,3,5]TRIAZINES

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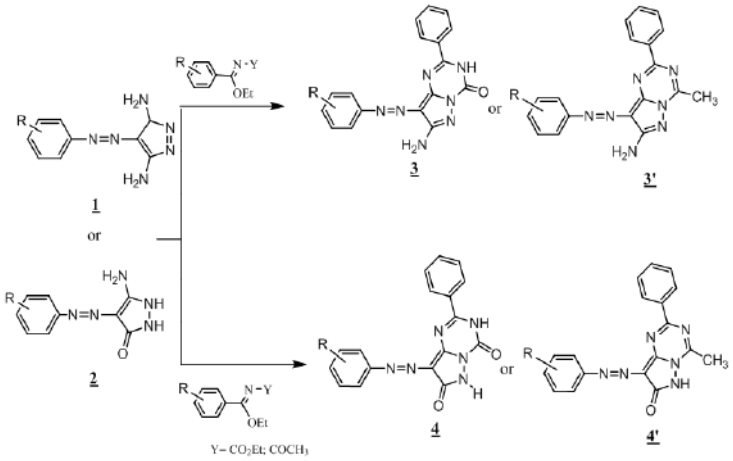
Recently, some new Pyrazolotriazine derivatives attracted organic chemists very much due to their biological and chemotherapeutic importance. For this reason, we propose in this work a new route to access at novel pyrazotriazine derivatives **3**,**3'**, **4** and **4'**.

These compounds are obtained respectively by condensation of compound **1** and **2** via N-acylated or N-ethoxycarbonylated imidates.



All compounds obtained are isolated after precipitation in reaction mixture with good yields and characterized by I.R, NMR ^{1}H and NMR ^{13}C.

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