Seventeenth Arab Chemistry Conference

Energy and Materials for a Sustainable Environment

Organized by the

Société Chimique de Tunisie
Tunisian Chemical Society

March 16 - 18, 2015

Laico Hotel, Yasmine Hammamet, Tunisia

Lectures and Communications Abstracts
List of Participants

Tunisian Chemical Society - Short Program of the ACC17

Monday 16 March 2015	
09.00 - 09.30	**Opening Ceremony**
09.30 - 10.10	**Lecture 1** **Pr. Jean-Marie Basset** *KAUST Catalysis Research Center, King Abdullah University, Saudi Arabia*
10.10 - 10.40	**Lecture 2** **Pr. Umit B. Demirci** *Institut Européen de Membranes, Montpellier, France*
10.40 - 11.10	**Coffee break**
11.10 - 11.40	**Lecture 3** **Pr. Pierre Dixneuf** *University, of Rennes, Bretagne, France*
11.45 - 12.45	**Oral Communications - First Session - OC 01 : OC 04**
13.00	***Lunch***
15.00 - 15.30	**Lecture 4** **Dr. Ali Madouri** *Laboratoire de Photonique et de Nanostructures MARCOUSSIS, France*
15.30 - 16.00	**Lecture 5** **Pr. Franck Dumeignil** *Université Lille 1, France*
16.00 - 17.00	**Oral Communications - Second Session - OC 05 : OC 08**
17.00 - 18.00	**Coffee break + Poster Session 1 (P 1 - P 44)**
18.00 - 19.00	**Oral Communications - Third Session - OC 09: OC 12**
19.30	***Dinner***
Tuesday 17 March 2015	
08.30 - 09.10	**Lecture 6** **Pr. Xiang-Hua Zhang** *Institut des Sciences Chimiques de Rennes - Université de Rennes I, France*
09.10 - 09.40	**Lecture 7** **Dr. Fabrice Odobel** *Faculté des Sciences et des Techniques de Nantes, France*
09.45 - 10.45	**Oral Communications - Fourth Session - OC 13 : OC 16**
10.45 - 11.45	**Coffee break + Poster Session 2 (P 45 - P 88)**
11.45 - 12.15	**Lecture 8** **Pr. Ahmed Galel** *Kuwait University, Kuwait*
12.15 - 12.45	**Lecture 9** **Dr. Hafedh Kochkar** *CNRSM, Technopôle Borj-Cédria, Tunisie*
13.00	***Lunch***
13.30	**Excursion to some touristic area in the capital**
20.30	***Gala Dinner***
Wednesday 18 March 2015	
08.30 - 09.00	**Lecture 10** **Pr. Abdulrahman Al-Warthan** *College of Science at King Saud University, Saudi Arabia*
09.00 - 09.30	**Lecture 11** **Pr. Zouhaier Ksibi** *Faculté des Sciences de Tunis, Tunisie*
09.30 - 10.45	**Oral Communications - Fifth Session - OC 17 : OC 21**
10.45 - 11.45	**Coffee break + Poster Session 3 (P 89 - P 130)**
11.45 - 12.15	**Lecture 12** **Pr. Adel MNIF** *CNRSM, Borj Cédria, Tunisie*
12.15 - 12.45	**Lecture 13** **Pr. Hanifa Easa Taher** *College of Engineering, UAE University of Al-Ain, United Arab Emirates*
12.45 - 13.00	**Awards distribution and Closing Ceremony**
13.00	***Lunch and leaving the hotel***

Being on time is the main factor for conference's success

FOREWORD

On behalf of the organizing committee, I am pleased to welcome you in Hammamet for this major event held under the auspices of the Tunisian Chemical Society (SCT) and the Union of Arab Chemists.

This is the Seventeenth edition of Arab Chemistry Conference, which is involved with more than 10 Arab and European countries in addition to the Tunisian universities and with the participation of more than 200 participants from various countries will discuss themes specializes in the science of chemistry dealing with energy and materials for a sustainable environment.

As will be held on the sidelines of the conference Thirty-fourth session of the Supreme Council of the Union of Arab Chemists

Many thanks for all participants and for all invited speakers who firmly accepted to contribute lectures at the event.

Happy 17th Arab Chemistry Conference at all.

Adel MEGRICHE

President of the
Tunisian Chemical Society

SCIENTIFIC COMMITTEE

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Abdelhamid SAYARI	Department of Chemistry, University of Ottawa, Ottawa, Canada
Abdulrahman AL-WARTHAN	King Saud University - Saudi Arabia

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Taïcir BEN AYED	Institut National des Sciences Appliquées et de Techologie - Tunisie
Imed LAAJIMI	Société Chimique de Tunisie

Sunday 15 March 2015	
15.00	**Welcoming participants, distribution of documents and check in**
19.00	***Dinner***

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Monday 16 March 2015 (Morning)						
09.00 - 09.30	**Opening Ceremony**					
09.30 - 10.10	**Lecture 1** **Pr. Jean-Marie Basset** *KAUST Catalysis Research Center, King Abdullah University, Saudi Arabia* **The metathesis of alkanes and related reactions: From silica supported early transition metal hydrides to metal (methylidene)(methyl)**					*Chairpersons:* **Farhat Rezgui** *&* **Xiang-Hua Zhang**
10.10 - 10.40	**Lecture 2** **Pr. Umit B. Demirci** *Institut Européen de Membranes, Montpellier, France* **Chemical hydrogen storage materials: Focus on boron and/or nitrogen-based compounds**					
10.40 - 11.10	**Coffee break**					
11.10 - 11.40	**Lecture 3** **Pr. Pierre Dixneuf** *University, of Rennes, Bretagne, France* **Catalytic transformations of biomass into polyamide precursors: The impact of ruthenium catalysts on green chemistry**					*Chairperson:* **Med Moncef Chaabouni**
Oral Communications - First Session						
	Room A *Chairperson :* *M.L Efrit*		**Room B** *Chairperson :* *N. Bellakhel*		**Room C** *Chairperson :* *T. Boubaker*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
11.45 - 12.00	**OC1A**	F. Odeh	**OC1B**	S. Charfeddine	**OC1C**	S. Teka
12.00 - 12.15	**OC2A**	I. Soltani	**OC2B**	H. Doghri	**OC2C**	F. Walha
12.15 - 12.30	**OC3A**	F. Laatar	**OC3B**	I. Elaissaoui	**OC3C**	S. Aouadi
12.30 - 12.45	**OC4A**	N. Bouazizi	**OC4B**	N. Ben Salah	**OC4C**	A. Al-Bawab
13.00	***Lunch***					

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Monday 16 March 2015 (Afternoon)						
15.00 - 15.30	**Lecture 4** **Dr. Ali Madouri** *Laboratoire de Photonique et de Nanostructures MARCOUSSIS, France* **Large area CVD graphene growth for energy and safety**					*Chairpersons:* **Hatem Ben Romdhane** *&* **Fabrice Odobel**
15.30 - 16.00	**Lecture 5** **Pr. Franck Dumeignil** *Université Lille 1, France* **Biomass valorization: Biorefineries & catalysis**					
Oral Communications - Second Session						
	Room A *Chairperson :* *M. Khitouni*		**Room B** *Chairperson :* *K. Jarraya*		**Room C** *Chairperson :* *S. Djemal*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
16.00 - 16.15	**OC5A**	H. Kchih	**OC5B**	K. Haddad	**OC5C**	I. Ayadi
16.15 - 16.30	**OC6A**	N. Kallel Kchaou	**OC6B**	I. Hedfi	**OC6C**	R. Banani
16.30 - 16.45	**OC7A**	A. Bouaziz	**OC7B**	A. Kedim	**OC7C**	M. Ben Abdelkader
16.45 - 17.00	**OC8A**	L. Ben Tahar	**OC8B**	M. Khemakhem	**OC8C**	M.A. Ben Ali
17.00 - 18.00	**Coffee break + Poster Session 1 (P 1 - P 44)**					
Oral Communications - Third Session						
	Room A *Chairperson :* *T. Ben Ayed*		**Room B** *Chairperson :* *J. Belgaied*		**Room C** *Chairperson :* *M.Dammak*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
18.00 - 18.15	**OC9A**	O. Bechambi	**OC9B**	A. Massoud	**OC9C**	S. Ben Mahmoud
18.15 - 18.30	**OC10A**	S.K. Shakshooki	**OC10B**	A. Midouni	**OC10C**	H. Benmoussa
18.30 - 18.45	**OC11A**	M. Zouari	**OC11B**	M.F. Nsib	**OC11C**	B. Ben Salem
18.45 - 19.00	**OC12A**	M. Elnewehy	**OC12B**	S. Mohammed	**OC12C**	N. Aliouane
19.30	***Dinner***					

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Tuesday 17 March 2015						
08.30 - 09.10	**Lecture 6** **Pr. Xiang-Hua Zhang** *Laboratory of glasses and Ceramics, Institut des Sciences Chimiques de Rennes, Université de Rennes I, France* **Non-oxide glasses and glass ceramics for thermal imaging and energy application**				*Chairpersons:* **Ridha Ben Salem** *&* **Pierre Dixneuf**	
09.10 - 09.40	**Lecture 7** **Dr. Fabrice Odobel** *Faculté des Sciences et des Techniques de Nantes, France* **Engineering of NiO-based photocathodes for photovoltaic and solar fuel production**					
Oral Communications - Fourth Session						
	Room A *Chairperson :* *H. Majdoub*		**Room B** *Chairperson :* *E. Srasra*		**Room C** *Chairperson :* *H. Tounsi*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
9.45 - 10.00	**OC13A**	M.A.G. Zayed	**OC13B**	A. Bouzieh	**OC13C**	M. Ghazouani
10.00 - 10.15	**OC14A**	N. Boussatha	**OC14B**	S. Chamali	**OC14C**	S. Ibrahimi
10.15 - 10.30	**OC15A**	R. Abu-Zurayk	**OC15B**	C. Elazizi Riahi	**OC15C**	S. Karoui
10.30 - 10.45	**OC16A**	M. Srasra	**OC16B**	S. Essid	**OC16C**	Kh. NASR
10.45 - 11.45	**Coffee break + Poster Session 2 (P 45 - P 88)**					
11.45 - 12.15	**Lecture 8** **Pr. Ahmed Galel** *Kuwait University, Kuwait* **Energy and materials for sustainable environment**				*Chairpersons:* **Mehrez Romdhane** *&* **Ali Madouri**	
12.15 - 12.45	**Lecture 9** **Dr. Hafedh Kochkar** *Centre National des Recherches en Sciences des Matériaux, CNRSM, Technopôle Borj-Cédria, Tunisie* **Advanced nanomaterials an ideal ''nanotools''. The major challenges for further developments**					
13.00	***Lunch***					
13.30	**Excursion to some touristic area in the capital**					
20.30	***Gala Dinner***					

Being on time is the main factor for conference's success

Wednesday 18 March 2015 (Morning)						
08.30 - 09.00	**Lecture 10** **Pr. Abdulrahman Al-Warthan** *College of Science at King Saud University, Saudi Arabia* **Biogenic synthesis of metallic nanoparticles and prospects toward green chemistry**				*Chairpersons:* **Ahmed Galal** *&* **Taïcir Ben Ayed**	
09.00 - 09.30	**Lecture 11** **Pr. Zouhaier Ksibi** *Faculté des Sciences de Tunis, Tunisie* **Effect of chemicals on the environment and on health**					
Oral Communications - Fifth Session						
	Room A *Chairperson :* *R. Abidi*		**Room B** *Chairperson :* *R. Abdelhédi*		**Room C** *Chairperson :* *H. Feki*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
9.30 - 9.45	**OC17A**	M. Stouri	**OC17B**	N. Omri	**OC17C**	S. Guiza
9.45 - 10.00	**OC18A**	A. Siaï	**OC18B**	W. Raissi	**OC18C**	H. Benaissa
10.00 - 10.15	**OC19A**	I. Tlili	**OC19B**	K. Abderrazek	**OC19C**	A. Nafady
10.15 - 10.30	**OC20A**	A.A. Azzaz	**OC20B**	I. Abidli	**OC20C**	A. Youssef
10.30 - 10.45	**OC21A**	R. Bouchrit	**OC21B**	N. Ben Hafeith		
10.45 - 11.45	**Coffee break + Poster Session 3 (P 89 - P 130)**					
11.45 - 12.15	**Lecture 12** **Pr. Adel MNIF** *Laboratoire de Valorisation des Matériaux Utiles, CNRSM, Borj Cédria, Tunisie* **Silica sand techno-scientific and economic issues**				*Chairpersons:* **Abdulrahman Al-Warthan** *&* **Adel Megriche**	
12.15 - 12.45	**Lecture 13** **Pr. Hanifa Easa Taher** *College of Engineering, UAE University of Al-Ain, United Arab Emirates* **Utilization of green technologies in the biofuel production from lipid-rich feedstocks**					
12.45 - 13.00	**Awards distribution and Closing Ceremony**					
13.00	***Lunch and leaving the hotel***					

Speakers' CVs

& Abstracts

Jean Marie BASSET

Professor
Director, Catalysis Research Center

Research Interests

Professor Basset works to evidence the possible relationships between homogeneous and heterogeneous catalysis. For that purpose, he developed "surface organometallic chemistry," a new field of chemistry that tries to answer questions, such as are the concepts of the reactivity of molecular chemistry applicable when an organometallic molecule reacts with a surface? Or, can the structures of molecular surface species be rationalized on the same basis as those of molecular species in solution? As a result of these questions, he is now able to design any catalyst for any catalytic reaction by a single site approach.

His work resulted in the discovery of a number of new catalytic reactions, such as the metathesis of alkanes, which transforms any paraffin into its lower and higher homologues, the cleavage of paraffin by methane, the coupling of methane into ethane and hydrogen, the Ziegler-Natta de-polymerization, which transforms polyethylene into diesel range gasoline, the transformation of ethylene to propylene, and the removal of traces of arsenic from water.

His research interests are also strongly linked to the needs of the Kingdom of Saudi Arabia in terms of energy and environment: dry reforming of methane (CO_2 + CH_4->H_2 and CO), transformation of methane to carbon and hydrogen (CH_4 ->C + H_2), the one-step transformation of crude oil to ethylene and propylene, and the direct oxidation of ethane to ethylene.

Biography

Prof. Jean Marie Basset is a professor in the Physical Sciences and Engineering Division and the director of the KAUST Catalysis Research Center (KCC). Presently, this lab includes 10 professors and 160 people. He joined KAUST in September 2009. Catalysis is a strategic domain for the world and particularly Saudi Arabia. It is a critical enabling science for the kingdom's energy future. Improvement in the catalytic processes across the chemical and petroleum industries will increase resources and energy utilization efficiencies and reduce waste. Needs for breakthroughs in catalysis are imperative requirements to meet these energy challenges "beyond petroleum": the use of biomass, CO_2, and CO utilization, and efficient photocatalysts for water splitting are crucial domains for catalysis. To meet these challenges, the field must advance from catalyst discovery by trial and error to catalysis by design.

Prof. Basset served as scientific director of the School of Chemistry, Physics and Electronics at the University of Lyon in France. He was appointed at CNRS in 1987, became directeur de Recherches Classe Exceptionnelle in 1994, and funded his laboratory of surface organometallic chemistry from 1994 to 2009. Prof. Basset's Lyon lab was home to 50 scientists, including Nobel laureate Yves Chauvin. Jean Marie came to CNRS in 1971 and has occupied several positions, including vice director of the Institute of Catalysis (Lyon). He also founded the consortium "Actane" on alkane activation with 11 university labs and five companies. Since 1992, he also has served as scientific director of L'École Supérieure de Chimie Physique Electronique de Lyon, which trains 450 chemists in a 3-year scholarship program.

Prof. Basset founded and served as president of the network Integrated Design of Catalytic Nanomaterials for a Sustainable Production (IDECAT), the only European network of excellence in catalysis, which includes 40 labs and 20 companies. A distinguished author of more than 600 journal articles (with an H-index of 58), he has been invited to more than 400 conferences at international symposia and congresses. Prof. Basset holds various professional memberships, is the recipient of several international and national awards, and is doctor honoris causa of several universities worldwide.

THE METATHESIS OF ALKANES AND RELATED REACTIONS: FROM SILICA SUPPORTED EARLY TRANSITION METAL HYDRIDES TO METAL (METHYLIDENE)(METHYL).

J.-M. Basset , Manoja K. Samantaray , Yin Chen, Emmanuel Callens , Nassima Riache, Edy Abou-Hamad, Aaron J. Rossini, Cory M. Widdifield, Raju Dey, L. Cavallo, Lyndon Emsley, Farhan A. Pasha

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Alkane metathesis is a catalytic reaction which transforms directly a given paraffin into its lower and higher homologues:

$$2\ C_nH_{2n+2} \rightleftharpoons C_{n-i}H_{2(n-i)+2} + C_{n+i}H_{2(n+i)+2}$$

$i = 1, 2...n-1$

It was discovered by the group of J.M. Basset using a tantalum hydride supported on silica, $(^{o}SiO)_2TaH_x$, that is a single site system.[1] This silica supported tantalum hydride catalyst has been the result of a systematic and broad investigation of the reactions between organometallic complexes and oxides surfaces which is usually called *surface organometallic chemistry*.[2] In the continuity, an alumina-supported tungsten hydride $W(H)_3/Al_2O_3$, was elaborated giving better activity. The mechanism proposed with these metal hydrides were the occurrence of a multifunctional catalyst in which the C-H bond activation was occurring by σ-bond metathesis with the M-H followed by two processes of : α-H elimination producing a metallocarbene followed by a β-H elimination producing an olefin. The reaction of the olefin with the metallocarbene would produce the classical cleavage of the double bond by a metalla-cycle. Unfortunately we were not able with the hydrides precursors to isolate any metallo-carbene either with Ta or with W.[3]

We have developed recently a new approach of this reaction with monohapto silica supported tantalum tetra methyl and tungsten pentamethyl. In both cases we were able to identify the surface methylidenes of Ta and W.[4-9] With this new generation of catalysts the TON of alkane metathesis was increased to a factor of 10 and more importantly the reaction could be applied to cycloalkanes such as cyclo-octane. With these new catalysts C-H bond activation occurs by σ-bond metathesis with the metal-methyl and a new multi-functional mechanism is proposed.

These new catalysts are able to achieve cleavage of alkanes by methane, coupling of methane to ethane and hydrogen, hydro-metathesis of olefins, direct transformation of ethylene to propylene.

References

[1] Vidal, V.; Theolier, A.; Thivolle-Cazat, J.; Basset, J. M. Science, 1997, 276(5309)
[2] Modern Surface Organometallic Chemistry Jean -Marie Basset, Rinaldo Psaro, dominique Roberto et Renato Ugo (Editor), (Wiley) (2011)
[3] Basset, J.-M.; Callens, E.; Riache, N. Alkane Metathesis. In Handbook of Metathesis, 2nd Ed. Grubbs, R. H., Wenzel, A. G., O'Leary, D. J., Khosravi, E., Eds. Wiley: Weinheim, 2015; Vol. 1, pp 33-70.
[4] Yin Chen, Bin Zheng, Edy Abou-Hamad, Ali Hamieh, Bilel Hamzaoui, Kuo-Wei Huang, Jean-Marie Basset , Chemical Communications, 2014, 50, 11721-11723
[5] Yin Chen, Samy Ould-Chikh, Edy Abou-Hamad, Emmanuel Callens, Janet C. Mohandas, Syed Khalid, Jean-Marie Basset, Organometallics 02/2014; 33(5):1205–1211
[6] Yin Chen, Emmanuel Callens, Edy Abou-hamad, Jean-Marie Basset, Journal of Organometallic Chemistry 07/2013
[7] Manoja K. Samantaray, Emmanuel Callens, Edy Abou-Hamad, Aaron J. Rossini, Cory M. Widdifield, Raju Dey, Lyndon Emsley, and Jean-Marie Basset, J. Amer. Chem. Soc., 136 , 3 (1054-1061), 2014,
[8] Manoja K. Samantaray, Raju Dey, Edy Abou-Hamad, Ali Hamieh, and Jean-Marie Basset, ACS catalysis, submitted 2014
[9] Yin Chen, Edy Abou-Hamad, and J.M. Basset, submitted 2014.

Umit B. DEMIRCI

Dr. Umit B. DEMIRCI received his PhD in Physical Chemistry (specialty in heterogeneous catalysis) in 2002 at the University of Strasbourg 1. From 2002 to 2007, he worked at different places and gained expertise in metal-based catalysis (*i.e.* research engineer in a small company but detached to the car manufacturer PSA Peugeot Citroën; researcher at TÜBITAK in Turkey; and post-doctoral fellow at University of Strasbourg 1).

In 2007, Dr. Umit B. DEMIRCI became Associate Professor at the University of Lyon 1 where he worked on "chemistry and materials for energy". In 2011, he had the opportunity to join the University of Montpellier 2 with part of the group in which he was involved. Since then, he works at the European Institute of Membranes (UMR ENSCM/UM2/CNRS 5635) where he continues his activity on "(inorganic) chemistry and materials for energy". In fact, since 2007, he has specialized in heterogeneous catalysis, and boron- and nitrogen-based materials for liquid- and solid-phase chemical hydrogen storage, and has developed his activity with national/international collaborations.

Dr. Umit B. DEMIRCI is member of the French Chemical Society (SCF), topic-leader in the national research group HySPaC supported by CNRS, member of the national council for universities CNU33, and associate editors of 2 international journals.

Chemical Hydrogen Storage Materials: Focus on Boron and/or Nitrogen-Based Compounds

Umit B. Demirci

Institut Européen des Membranes, Université de Montpellier, ENSCM, CNRS UMR5635, Place Eugène Bataillon, CC047, 34090 Montpellier, France
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In chemical hydrogen storage, boron and/or nitrogen-based materials (also reported as B–N–H compounds) have shown to be promising, especially owing to their high gravimetric hydrogen density. Sodium borohydride $NaBH_4$ and ammonia borane NH_3BH_3 are typical examples with 10.8 and 19.5 wt% H. In our group, we worked on the synthesis of this kind of materials and on their hydro-/thermo-lytic dehydrogenation from 2007 to 2012.

In 2011, we have started an activity on a derivative of ammonia borane, *i.e.* hydrazine borane $N_2H_4BH_3$ (15.4 wt% H). On the one hand, it has attracted our attention as, unlike ammonia borane, the N-containing moiety, *i.e.* N_2H_4, can be dehydrogenated in aqueous solution in the presence of an active and selective bi-metallic catalyst. Our main achievements will be presented. On the other hand, like ammonia borane, hydrazine borane can be decomposed under heating (from 60°C). Nonetheless, the unwanted N_2H_4 forms as by-product and a shock-sensitive residue is "dangerously" recovered upon heating at >300°C. This made us thinking about its chemical modification with the synthesis by ball-milling of alkali and alkali-earth derivatives, *i.e.* the hydrazinidoboranes.

More recently, in 2013, we have started to scrutinize the potential of hydrazine *bis*borane $N_2H_4(BH_3)_2$ for solid-/liquid-state hydrogen storage. Our very recent results demonstrate that the potential of this material for chemical hydrogen storage is low, but would be high for other energy applications.

To sum up, it is a great opportunity for me to attend to the ACC17 event in Hammamet, Tunisia, where I will present:

- A large introduction on chemical hydrogen storage;
- Our main achievements with respect to B–N–H compounds, with a special focus on very recent results.

Pierre DIXNEUF

Emeritus professor of Chemistry, UMR 6226 CNRS
University of Rennes
Institut Sciences Chimiques de Rennes, France
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http://scienceschimiques.Univ-rennes1.fr/catalyse/personnels/interface/PHD.htm

- Doctorat es sciences 1971 with Prof René Dabard University of Rennes on ferrocene chemistry
- Post doc with Prof M. F. Lappert University of Sussex UK, 71-72,on NHCarbene-metal complexes
- Professeur at the university of Rennes in 1978, Prof Classe exceptionnelle 1994

Research topics at the boundary of metal complexes and catalysis

- Carbon-rich organometallics and new molecular architectures

Chemistry of model systems for catalysis: carbenes, vinylidenes, allenylidenes, cumulenes, Carbynes

Ruthenium-arene and ruthenium-Cp organometallic complexes. Design of catalysts

- Homogeneous catalysis especially with ruthenium catalysts since 1985

Functional carbonates, oxazolidinones in organic synthesis, heterocycles via catalysis: vinylidenes in catalysis.

Alkene metathesis, catalytic biomass transformation, Selective formation of C-C bonds with Ru catalysts

-Catalutic C-H bond activation/functionalization and catalysis in water

Scientific activity

- > 440 papers in international journals, 50 since 2011
- 8 patents
- 4 books (ruthenium fine chemistry, 2004 Springer, vinylidene & allenylidenes, 2008 Wiley, Metal-Catalyed reactions in water, 2013 Wiley, Ruthenium in Catalysis, 2014 Springer)

Awards and Distinctions

Since 2000 Member of the « **Institut universitaire de France** »

1990 A. von Humboldt prize for Research
1994 Académie des Sciences award
1996 Doctor Honoris Causa, Waterloo, Canada
2000 Prix Le Bel (French Chemical Society)
2000 Member of the «Institut universitaire de France»
2001 Grignard-Wittig Prize (Germany)
2006 Grand prix IFP académie des sciences
2006 Sacconi medal (Italy)
2014 Prix Catalan of the Spanish Chem Soiciety
2014 Prize of the Chinese Chemical Society

University, National, European responsabilities

Director of a CNRS research unit "organometallics and catalysis 86-99
first Director of l'Institut de Chimie de Rennes 2000-03
vice president for research of the university of Rennes jan 2001-Dec 2004
Deputy director for CNRS-Department of Chemical Sciences in Paris (nov 96-Dec 99)
Europe : COST, ERASMUS, IDECAT Network programmes

Journal Advisory, Editorial boards

Previously Organometallics, Chemical communications, Advances in Synthesis and catalysis 00-10
1997 - Editor board of " Topics in Organometallic Chemistry "series, Springer
2010- Green Chemistry

Catalytic Transformations of Biomass into Polyamide Precursors: The Impact of Ruthenium Catalysts on Green Chemistry.

Pierre H. Dixneuf, Xiaowei Miao, Cédric Fischmeister, Christian Bruneau

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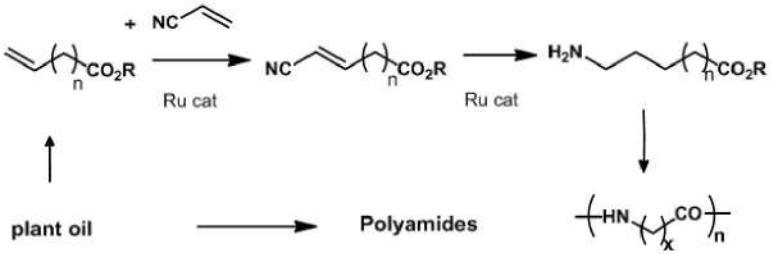
Selected metal catalysts have the potential to transform molecules arising from biomass, via sequential catalyses, into well-known useful intermediates for industry via clean, atom and energy economical processes. Thus they can significantly contribute to the development of green chemistry giving higher value to renewable resources. **Molecular ruthenium catalysts** constitute one of the most powerful catalyst family for efficient alkene metathesis with tolerance of a variety of functional groups.

The objective of this presentation is to demonstrate that molecular ruthenium-alkylidene catalysts can transform unsaturated esters, arising from plant oils, into precursors of polyamides that are currently used in industry, but arising from fossil derivatives. It will be shown that the catalytic cross-methathesis of unsaturated esters and acrylonitrile can be performed and leads to linear nitrile esters. Further catalytic hydrogenation affords the expected amino esters, the polyamide precursors. This whole process can be organized via tandem ruthenium-catalyses. Several directions will be pointed out for the potential of metal catalysis



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University education

1982 Mastership of Physics. University of Sciences- Tunis
1983 Master of Science: Material Science at Paris VII University
1987 Doctorate: Material science at Paris VII University

Experience

1988 Post-doctorate at Laboratory of microstructures and Micro-electronics CNRS.
1989 Research Engineer: Laboratory de Photonics and Nanostructures CNRS.
1995-2008
Manager of the Technology platform LPN CNRS
Expert of clean room conception for :
French Conseil Régional Franche-Compté.
Beijing Academy of Science
Tunisian ministry of high education and research
Algerian ministry of high education and research
1988-2013
Team manager of technological research for materials:
standard thin films, 2D materials (Graphene, and beyond) and micro & nanofabrication of devices (5 ANR research contracts, 2 RTRA, 1 NanoSaclay).

Expertise:

Management of technology facilities (scientific)
Management of Clean room facilities (technical)
Process
Thin film deposition (PVD, CVD, ALD, Thermal evaporation, e-beam evaporation)
Synthesis of 2D materials : CVD Graphene for photovoltaic applications
Characterization: (Raman, IR Spectrometry, SEM), electrical measurements (Hall effect, Electrical conductivity, VDP,..)
Micro et nanofabrication of MEMS-like devices (Cantilevers, AFM tips, membranes 10nm thick …)

Collaborations

SPCSI- IRAMIS (CEA): Grafting of neural on Graphene electrodes.
LGEP (SUPELEC High School) : Graphene as top electrode for photovoltaic cells.
LMSC (Univ. Paris Diderot): solid nanopores (in graphene) fabrication and integration for biology usage.
LPA-ENS : Magnetotransport and THz devices on Graphene.
University of Science at Tunis: Photo Luminescence and Raman spectroscopy on graphene and its applications on semiconductors (QD).

Relevant publications :

✓ High frequency 3C-SiC AFM cantilever using thermal actuation and metallic piezoresistive detection. R. Boubekri et al. *Materials Science Forum Vol. 711 (2012) pp 80-83. doi:10.4028*
✓ A new approach for AFM cantilever elaboration with 3C-SiC S. Jiao a,b et al. *Mater Lett (2012), doi:10.1016/j.matlet.2012.02.128*
✓ Single-Shot Diffractive Imaging with a Table-Top Femtosecond Soft X-Ray Laser-Harmonics Source. Ravasio et al. PRL 103, 028104 (2009).
✓ Focused gold ions beam for localized epitaxy of semiconductor nanowires J. Gierak et al. Microelectronic Engineering 87 (2010) 1386–1390. doi:10.1016.
✓ Tailoring nanopores for efficient sensing of different biomolecules. O. AbdelGhani et al. MRS Proceedings 2010 1253 : 1253-K10-33 MRS 2010 DOI:doi.10.1557
✓ Hot electron cooling by acoustic phonons in graphene. A.C. Betz et al. DOI : 10.1103/PhysRevLett.109.056805. PRL 109,056805 (2012).
✓ Graphene-capped InAs/GaAs quantum dots
Hosni Ajlani[1,a)], Riadh Othmen[1], Meherzi Oueslati[1], Antonnella Cavanna[2] and Ali Madouri[2]
J. Vac. Sci. Technol. B 31, 020602 (2013); http://dx.doi.org/10.1116/1.4790365 .
✓ The improvement of InAs/GaAs quantum dot properties capped by Graphene
Kamel Rezgui[1,*], Riadh Othmen[1], Antonella Cavanna[2], Hosni Ajlani[1], Ali Madouri[2], Meherzi Oueslati[1]
Article first published online: 17 SEP 2013 DOI: 10.1002/jrs.4382

LARGE AREA CVD GRAPHENE GROWTH FOR ENERGY AND SAFETY

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SUMMARY
Large areas of graphene are grown by chemical vapor deposition technique using copper foil as a catalyst. We performed Raman spectroscopy investigation coupled with TEM and electrical measurements to point out its properties for devices. The technological lock is the transfer of the graphene onto a specific substrate. We improved a process for PMMA removal keeping the top surface of the graphene clean.

KEY WORDS: Graphene, CVD, Transfer, Raman spectroscopy, HRTEM, XPS.

1. INTRODUCTION

The remarkable properties of graphene have positioned this material on the roadmaps for electronic and optoelectronic applications. Nevertheless, to envision applications, large scale graphene must be synthesized with appropriate properties for specific applications such as sensors and photonic devices. For example, there is a strong research effort to replace rare earth ITO (*Indium Tin Oxide*)-based devices with graphene transparent electrode in optoelectronic devices such as solar cells.

2. EXPERIMENTAL RESULTS

The growth of graphene is optimized using different experimental conditions (temperature, CH4/H2 flow, pressure) to minimize defects shown on 1D band on Raman spectra. To characterize our CVD graphene, large area of it were transferred onto SiO2/Si substrates and Raman spectroscopy was performed to investigate the uniformity and defect density. Raman mapping (Figure 1), EFM and SEM image showed that our large area graphene were mostly monolayer (98%) with a very low defect density (ID/IG<<0.1) and with a few layers of small islands located on defects.[2]

Graphene is also an interesting material for many other applications as gas sensors. For that we fabricate a device for that and made experiments with oxygen and nitrogen exposure. The first measurements show clearly the variation of the resistivity during the exposure time.

The first experiments on graphene based devices show that the CVD graphene have a huge potential for many applications. The node of this technique is the transfer of the material onto a specific substrate. The actual state of the art doesn't guarantee free defect surface after transfer as we see on Raman maps. To improve the quality remains important but the control of the final quality after transfer is the big challenge.

3. CONCLUSION

We demonstrate that we grow large graphene areas (10x10 cm²) on copper foils with a good quality for many applications and gas sensors are good demonstrators for that. The transfer of the graphene onto substrates is successfully done with more than 95% of surface clean.

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[3] E. Pallecchi, F. Lafont, V. Cavaliere, F. Schopfer, D. Mailly, W. Poirier and A. Ouerghi «High Electron Mobility in Epitaxial Graphene on 4H-SiC(0001) via post-growth annealing under hydrogen» Scientific Reports 4, Article number: 4558doi: 10.1038/srep04558

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1. Carrier Summary

After obtaining his PhD in 1998 from Lille 1 University, Franck Dumeignil spent more than six years in Japan: a post-doctoral stay of 2 years at the NIMC in Tsukuba before obtaining a 2-year postdoctoral contract at the Tokyo University of Agriculture and Technology (TUAT), following which he became Associate Professor at a Centre of Excellence (Scientific Cluster).
He returned to France as a Full Professor at Lille 1 University, Sciences and Technologies where he piloted several research projects. In January 2010, he was appointed Deputy Director of UCCS, UMR CNRS 8181, which employs more than 200 people. He coordinated the PCRD7 European EuroBioRef Programme '*EUROpean Multilevel Integrated BIOREFinery Design for Sustainable Biomass Processing*' (www.eurobioref.org), gathering 29 partners from 15 countries for a global budget of €38 million (€23 million of European subvention) during 4 years (01/03/2010 - 28/02/2014). As part of that project, his research team was, in particular, involved in the development of a wide variety of catalytic reactions. Recently, he was awarded the ANR EQUIPEX REALCAT project (*'Advanced high throughput technologies platform for biorefineries catalysts design'*; €8.3 million) and created the Franco-Japanese International Associate CNRS Laboratory CAT&P4BIO (*'Innovative Catalysts for Oxidation Reactions and Processes, Biomass Conversion'*) of which he is Director. Finally, he is piloting the SP3 *'Catalysis and Biocatalysis'* of the *'Genesys of Lipids Biorefinery'* GENESYS program in the framework of the ANR ITE P.I.V.E.R.T. *'Picardy Plant Innovations, Teaching and Technological Research'* (2011-2020; €247 million).
Franck Dumeignil was also named Junior Member of the Institut Universitaire de France (University Institute of France, IUF) on October 1st 2010. The mission of IUF is to promote the development of a high-level academic research and encourage interdisciplinary projects. He has co-authored over 90 scientific articles, 25 patents, and 300 presentations (oral and poster) in national and international conferences.

2. Diplomas of the Université des Sciences et Technologies de Lille, France

- **Master 1 in Physical Chemistry** (Pass with **H**igh **H**onors, **V**aledictorian, 1994).
- **Master 2 in Spectrochemistry, Molecules, Solids and Reactivity** (Pass with **H**igh **H**onors, **V**aledictorian, 1995).
- **PhD in Spectrochemistry, Molecules, Solids and Reactivity** (1996-1998) obtained on the 25th of November 1998 at the Université des Sciences et Technologies de Lille with the **H**ighest **L**evel of **D**istinction.
- **Accreditation to Supervise Research** obtained on the 12th of December 2005.

3. Prices and Awards

- **2005 year's price of the best scientific paper awarded by the Japan Petroleum Institute:** Atsushi Ishihara, **Franck Dumeignil**, Danhong Wang, Xiangguo Li, Hisashi Arakawa, Eika W. Qian, Shinichi Inoue, Akinori Muto, Toshiaki Kabe, *"Investigation of the sulfur behavior on Mo-based HDS catalysts supported on a high surface area TiO_2 by ^{35}S radioisotope tracer method"*, Journal of the Japan Petroleum Institute, Vol. 48(1), p37-44 (2005).
- **Awarded as Junior Member of the Institut Universitaire de France (2010-2015)**
- **1st Poster Award at the CAT4BIO conference (2012):** Wenhao Wang, Cyril Pirez, Mickaël Capron, Sébastien Paul, Thirumalaiswamy Raja, Paresh L. Dhepe, **Franck Dumeignil**,* Louise Jalowiecki-Duhamel.
  "H_2 Production from Ethanol Steam Reforming over Ce-Ni Based Catalysts: Studies on Preparation Methods"
  Advances in catalysis for biomass valorization to fuels and chemicals" (CAT4BIO), July 8-11, Thessaloniki (Greece) (2012).
- **Awarded as Junior Honorary Member of the Société Chimique de France (2014-2018).**
- **Awarded as Fellow of the Royal Society of Chemistry, UK (from November 2014).**
- **Awarded as 'Etoile de l'Europe' by the French Ministry of Higher Education and Research (2014).**

BIOMASS VALORIZATION: BIOREFINERIES & CATALYSIS

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[a)] *Unité de Catalyse et de Chimie du Solide, UCCS (UMR CNRS 8181), Université Lille 1, Sciences et Technologies, Cité Scientifique, F-59650 Villeneuve d'Ascq, France*
[b)] *Institut Universitaire de France, 103, Boulevard Saint-Michel, 75005, Paris, (France)*

The development of biorefinery processes is of the upmost importance for establishing an economy based on bio-resources. Nevertheless, contrary to petro-resources of which the nature and composition variations are 'relatively' limited, under the term 'bio-resources' are gathered compounds of very different natures (cellulose, hemicellulose, oils, lignin…). A complete set of specific technologies must be developed in order to convert as smartly as possible each fraction. This implies the elaboration of a lot of processes based on chemistry, and especially on catalysis (enzymatic and chemical) as well as on thermochemistry. Within this frame, a good example is the EuroBioRef project *'EUROpean multilevel integrated BIOREFinery design for sustainable biomass processing*' [1], a new highly integrated, diversified and sustainable concept, which adopts a flexible and modular process design adapted to large-scale but also small-scale production units. To this respect, the EuroBioRef project will notably be presented, in which the central role of catalysis will be pointed out after describing the various challenges of this field in the context of biorefineries.

Acknowledgements. The evocated biorefinery concept has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) under grant agreement n°241718 EuroBioRef.

Key words: Biorefineries, catalysis, thermochemistry, biotechnology

References

[1] www.eurobioref.org

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MAIN PROFESSIONAL EXPERIENCE

Since 10/2002	**Research Professor at CNRS** (French National Center for Scientific Research). Director of the laboratory of glasses and ceramics.
07/2001 - 07/2002	**Chief technology officer** of UMICORE IR Glass
04/1996 – 07/2001	**President, CEO** and **founder**, acting also as technical director, of VERTEX S.A. specialized in the development and the production of infrared glass optics.
1988 – 1996	**Permanent researcher** at French CNRS (National Center for Scientific research).

EDUCATION

Sept 1985 - Oct 1988	Ph.D. Student at University of Rennes I, France.
Sep 1984 - Jul 1985	Master student at University of Rennes I, France.
Oct 1983 - Jul 1984	French language training at Shanghai Foreign language institute.
Sept 1979 - Jul 1983	University student at Zhejiang University, China.

SCIENTIFIC PRODUCTIONS

- 15 patents
- 49 invited conferences in international conferences
- 210 publications in international peer-reviewed journals

NON-OXIDE GLASSES AND GLASS CERAMICS FOR THERMAL IMAGING AND ENERGY APPLICATION

Xiang-Hua ZHANG, Laurent Calvez, David Lecoq,
Hongli MA, Michel Cathelinaud

Laboratory of glasses and ceramics, UMR CNRS-Université de Rennes I n°6226, 35042 Rennes cedex, France.

Non-oxide glasses include mainly fluoride glasses and chalcogenide glasses. Chalcogenide glasses are glasses containing at least one of the following elements: sulfur, selenium and tellurium. Their main properties are associated with the very large transmission window ranging from visible to far infrared region. This optical property can be significantly deteriorated by the presence of oxygen. This is the reason why chalcogenide glasses must be prepared in the absence of oxygen, in vacuum or in inert gases.

The most studied applications for these glasses are thermal imaging and chemical sensing[1]. More recently, chalcogenide glass ceramics with outstanding photoelectric properties have also been discovered. Fist results show already high efficiency for visible light photocatalytic applications[2].

The preparation, properties and applications of these materials will be presented.

Key words: Chalcogenide glasses, infrared, photocatalysis, photovoltaic.

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[1] Bruno Bureau et al, J. Non-Cryst. Solids, Vol. 345&346, 2004, pp276–283.
[2] Xianghua Zhang et al, J. Mater. Chem. A, 2014, 2, 17099.

Fabrice ODOBEL

Date of birth: June 6th, 1966 at Les Bouchoux (Jura, France)
Citizenship : French
Chimie et Interdisciplinarité, Synthèse, Analyse, Modélisation-CEISAM- UMR CNRS 6230
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Education:

Dr. Fabrice ODOBEL is a Chemical Engineer from "Ecole Nationale Supérieure de Chimie de Lille" (Diploma in 1990). He obtained a Ph.D. in Chemistry under the direction of Prof. Jean-Pierre SAUVAGE in 1993 at the University Louis Pasteur in Strasbourg (FRANCE). The research topic concerned the synthesis of a modular approach of porphyrin arrays for photoinduced electron transfer.

In 1993-1994, he spent one year at Columbia University as a postdoctoral fellow with Prof. Ronald BRESLOW in New York city (USA). He worked in the field of artificial enzymes and the research project aimed at designing cyclodextrine derivatives for the selective cleavage of a peptide sequence.

Appointment:

He is Research Director at CNRS (equivalent of full professor) from 2007. The core activity of his team focuses of molecular systems (organic and coordination chemistry) towards light induced properties. Summarily, the research activity addresses two different projects:

1. Photovoltaic devices (dye sensitized solar cells) for the transformation of solar energy into electricity.
   We develop sensitizers and prepare solar cells with both conventional n-type semiconductor (TiO_2, ZnO, etc...) and for the emerging technology based on p-type semiconductors (NiO).
2. -Molecular architectures for artificial photosynthesis.
   The goal in this area is to develop new molecular architectures reproducing the functions of charge photoaccumulation and of the Z scheme of natural photosynthesis. We also aim at solar fuel production with these artificial molecular systems.

Student supervisions:

From 2001, Dr. Fabrice ODOBEL has been supervising about 16 Ph.D. students and about 14 post-doctorates

Award and grants:

In 2003, he was awarded the Bronze Medal of CNRS (Organic Chemistry Division)
In december 2010, the AERES assessment comity ranked A+ the team led by F. Odobel at CEISAM.
From 2002 until now, Dr. Fabrice Odobel was involved in several multidisplinary international research projects as participant (5 projects) or as coordinator (6 projects) in the area of molecular photonics.

Dr. Fabrice ODOBEL regularly presents his research activity in lectures at several Universities in France and abroad (about 30 times) or in international conferences (about 30 times) including recently as invites speaker in Gordon Research Conference. Dr. Fabrice ODOBEL has about 130 peer-reviewed publications in International Journals with peers review journals + 3 patents and an "h index" factor of 35 (source: ISIS WEB of Knowledge).

Engineering of NiO-based photocathodes for photovoltaic and solar fuel production

Fabrice Odobel,[a] Florent Légalité,[a] Yann Pellegrin,[a] Errol Blart,[a]
Jérôme Fortage,[b] Alain Deronzier,[b] Marie-Noëlle Collomb,[b]
Mohammed Boujtita,[a] Denis Jacquemin,[a] Leif Hammarström[c]

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Dye-sensitized solar cells (DSCs), especially conventional n-type DSCs based on nanostructured TiO_2 films, have been extensively investigated for several decades and they now reach photoconversion efficiencies (PCE) around 13%.[1] Sensitization of p-type nanocrystalline semiconductors (p-SCs), such as NiO, have been much less investigated, but they have been receiving increasing attention, because they are the complementary photoelectrodes of conventional TiO_2-based photoanodes to build tandem photoelectrochemical cells.[2] Tandem cells present the potential to achieve high PCE, certainly above 15%. Furthermore, these photocathodes can be also used for solar fuel production.[2] Contrary to conventional dye-sensitized solar cells (n-DSCs), whose operation principle is based on the photoinjection of electron into the conduction band of a n-type SC such as TiO_2, in p-DSC the sensitizer excited-state decays by hole injection into the valence band of the p-SC. This contribution will be devoted to photocathodes (sensitized mesoporous NiO electrodes) and will focus on new materials for this emerging technology. More particularly, we will present new p-SC metal oxides, new classes of sensitizers based on organic dyes and transition metal complexes which exhibit significant PCEs. The second part will be devoted to the development of photoelectrosynthetic cells combining a sensitized NiO film with a suitable catalyst to drive chemical transformations (H_2 evolution) with the reducing equivalents produced at the photocathode.

Keywords: photocathode, p-type semiconductor, sensitizer, water splitting, dye-sensitized solar cells, photocatalysis.

References

[1] (a) S. Mathew, A. Yella, P. Gao, R. Humphry-Baker, F. E. Curchod, N. Ashari-Astani, I. Tav-ernelli, U. Rothlisberger, K. Nazeeruddin, M. Grätzel, Nat. Chem. 2014, 6, 242; (b) A. Hagfeldt, G. Boschloo, L. Sun, L. Kloo, H. Pettersson, Chem. Rev. 2010, 110, 6595.

[2] (a) F. Odobel, L. Le Pleux, Y. Pellegrin and E. Blart, Acc. Chem. Res., 2010, 43, 1063; (b) F. Odobel, Y. Pellegrin, J. Phys. Chem. Lett. 2013, 4, 2551 and references therein.

Ahmed GALAL

Prof. Galal earned his bachelor and master degrees in Chemistry from Cairo University and master and PhD from the University of Cincinnati, OH, USA. He joined the chemistry department of Cairo University as assistant professor on 1992 where he is currently a full professor. Professor Galal worked at several universities where he assumed Professor Visit ship including: Ulm, Erlangen, Ilmenau, and Free University (Germany); Cincinnati and Texas A&M (USA); Liverpool (UK); Polytechnic of Turin (Italy); United Arab Emirates and Kuwait.

Professor Galal served as member of Strategic Planning and Technical Support Centre, a member of Board of Research, Development and Innovation Program, a Program funded by the European Union of the Ministry of Higher Education of Egypt.

Professor Galal also served as Dean of Faculty of Science, Cairo University and as Interim Head of Center of Hazardous and Environmental Mitigation of the University.

Professor Galal earned several honors: Graduate Fellowship Award, Quantum Chemical Corporation (USA), Fellowship award of the "Alexander von Humboldt Stiftung,"

He also acts as member in National Committee on New and Advanced Materials, the committee of Basic Sciences, of the Academy of Science and Technology of Egypt.

Professor Galal was the Egyptian delegate to: International Science Technology and Innovation Centre for South-south Cooperation under the Auspices of UNESCO, (Malaysia); The inauguration of the Euro-Mediterranean University (EMUNI) (Slovenia); The Centre for Science and Technology of the Non-Aligned and other Developing Countries (NAM S&T), Cochin – (India); The RussNano Forum (Russia); COMEST meeting on Ethics of Science and Technology under the Auspices of UNESCO (Qatar); COMEST meeting on Ethics of Science and Technology under the Auspices of UNESCO (Qatar).

Professor Galal is a member of the Advisory Board for International Institute for Multifunctional Materials for Energy Conversion (IIMEC), Texas A&M University (USA).

Professor Galal earned Cairo University Prize in Excellence in Research in the area of Advanced Materials for Technological Applications. He authored and co-authored over 140 published and presented papers in international journals and conferences. He supervised several theses that led to both Master and PhD degrees.

His research covers a wide spectrum of interests including surface tailoring with nano-structured materials for energy conversion, storage and sensing applications. Synthesis and modification of carbon based nano-structures with emphasis on graphene and carbon nanotubes, and perovskites. Another area of research interest is corrosion prevention and control using smart coating and studies of newly developed super-alloys for advanced applications.

Professor Galal is member of the editorial board of "Talanta" published by Elsevier and is reviewer for several international journals.

ENERGY AND MATERIALS FOR SUSTAINABLE ENVIRONMENT

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Electrical energy storage varies from low to high capacity and slow to fast charge/discharge cycles. The last decade witnessed a paramount up-scaling of high-tech portable products that inferred the need for investigating alternatives for electrical energy storage means that realize both fast charge/discharge cycles and high storage capacities. Metal oxides have been used extensively in the merge of semi-conducting capacitors in the mid of last century. New materials merged with the rise of nanotechnology as a promising science and technology application. In this work, we are introducing the combination of graphene and nickel oxide/hydroxide hybrid as a new candidate material for electrical "super-capacitor" for energy storage. On one hand, we are anticipating that graphene being one of the environmentally benign nanostructured materials with unique properties interfaced with Ni-oxides/hydroxides. On the other hand, we will investigate this interfacing using electrochemical and surface techniques. The main objective will be mainly to characterize the charge transfer characteristics between graphene films and Ni-oxides/hydroxides and their influence on the charge storage capacity. Other examples will be presented including surface tailored structures for enhanced electrocatalytic conversion for green production of fuels.

Key words: Graphene; Nickel Oxide/Hydroxide; Supercapacitors; Green Chemistry; Electrocatalysis; Hydrogen Production.

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Hafedh Kochkar is a Professor of chemistry at the National Research Center of Materials Science (NRCMS) in Tunisia; in the field of materials engineering and nanomaterials for catalysis applications. He obtained his Ph.D., in Chemistry from Claude Bernard University, France (1997) and pursuded worldwide post doctoral research positions at: Institute of Chemical Technology and Engineering, Aachen, Germany (1998-2000) and IRCELyon of CNRS in collaboration with Renault Research Center, France (2000-2001). He worked as scientist at ExxonMobil Research and Engineering Corporation in Annandale, New Jersey, USA (2001-2004). He developed continuous flow measurements using the Tapered Element Oscillating Microbalance for MTO process. Prof. Kochkar has excellent skills in kinetic and catalysis. He is a Fellow of the American Sigma Xi Research Society. Prof. Kochkar leads a group of research scholars working in the field of materials engineering and catalysis. Prof. Kochkar has 6 patents (European, US and International); two of them are now used in industry. He is leading many projects with Laboratories and Institutes from the European Union.

ADVANCED NANOMATERIALS AN IDEAL ‘’NANOTOOLS’’. THE MAJOR CHALLENGES FOR FURTHER DEVELOPMENTS

Hafedh KOCHKAR

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Nanotechnology has been receiving increasing attention in the past decade due to its applications in the field of catalysis, electronics, high-density magnetic recording media, sensors, nanofiltration and biotechnology. However, the control of the size, the size distribution, and the shape of nanoparticles remains a challenging task. In this respect, shape control provides ideal “nanotools” since providing well defined nanoparticles with precise proportion of crystallographic facets and fraction of surface atoms on corners and edges [1-2]. This precise control leads to simplified catalytic systems on which mechanistic information of various organic and inorganic reactions can be easily retrieved. Indeed, the formation of nanocrystals is found to be a highly kinetics-driven process. Colloidal techniques to synthesize shape-controlled metallic nanoparticles (NPs) have been tremendously developed in the last 10 years due to a great interest in materials science for the unusual optical properties of anisotropic nanocrystals. The synthesis of shape-controlled metallic nanoparticles has also recently opened the way to new physical and chemical applications.
In this talk I would be discussing our investigation on the development of nanomaterials and its potential applications in various structure-sensitive reactions and in Advanced Oxidation Processes (AOPs)[3].

Key words: nanotools, size, morphology, oxidation and hydrogenation

References

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Abdulrahman AL-WARTHAN

Abdulrahman Abdullah Alwarthan is a professor of Analytical Chemistry. He obtained his Ph.D from Hull University in United Kingdom, in 1988.

His research interest is in :-

- Synthesis of Nanoparticles
- Nanocatalysis
- Green Chemistry
- Forensic Chemistry
- Chromatographic Techniques

Currently he is the :-

- Editor-in-Chief of the Arabian Journal of Chemistry
- Associate editor-in-Chief for Arabian Journal of Forensic Sciences and Forensic Medicine
- Associate editor-in-Chief for Journal of King Saud University-Science branch
- A member of the International Advisory Board for the Jordan Journal of Chemistry

He has chaired several international chemistry conferences. He is a member of the Saudi Chemical Society Council and a member of the College of Forensic Sciences Council at Naif Arab University for Security Sciences.

Prof. Alwarthan has published more than 100 research articles in international journals. He has authored several books and has three patents. He has supervised more than 6 Ph.D. students and more than 35 M.Sc. students

Biogenic synthesis of metallic nanoparticles and prospects toward green chemistry

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The immense importance of nanoparticles and their applications is a strong motivation for exploring new synthetic techniques. However, due to strict regulations to manage potential environmental impacts, greener alternatives to conventional synthesis are the focus of intense research. In the scope of this perspective, a concise discussion about the use of green reducing and stabilizing agents toward the preparation of metal nanoparticles is presented. Reports on the synthesis of noble metal nanoparticles using plant extracts, ascorbic acid and sodium citrate as green reagents are summarized and discussed, pointing toward an urgent need of understanding the mechanistic aspects of the involved reactions.

Zouhaier KSIBI

Personal

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Nationality	Tunisian

Awards and Distinctions

Presidentiel Price of Chemistry in Tunisia : June 1982.

University degrees

- Faculty of Sciences of Tunis (Tunisia)

June - 1982	Maitrise de Chimie (four year university degree in chemistry)
June - 1984	Post graduate studies in chemistry D.E.A. (Diplôme d'Etudes Approfondies) Equivalent to MASTER
June - 1988	PHD in chemistry
May – 2002	second PHD (Doctorat d'Etat) in Chemistry

Professional career

1982-1989	Assistant
1989-2002	Assistant Professor
2002-2007	Associate Professor
Since 2007	Professor

Position

Since December 2011 Director of Laboratoire de Chimie des Matériaux et Catalyse (70 researchers) – Faculty of Sciences of Tunis – University of Tunis El Manar

Field of research

Catalytic oxidation of biorecalcitrant aqueous effluents from Agro-Food and Industrial Productions.
Catalytic valorisation of bioressources and phenolic compounds.
Kinetic studies of catalytic wet air oxidation.
Environment Chemistry and Green Chemistry.
Participation in CAT-MED European Research project (2003-2006)
Supervision of 7 PhD and 6 Master students

Recent Publication

1- Asma Turki, Chantal Guillard, Frederic Dappozze, Zouhaier Ksibi, Gilles Berhault, Hafedh Kochkar. Phenol photocatalytic degradation over anisotropic TiO_2 nanomaterials: Kinetic study adsorption isotherms and formal mechanisms. Applied Catalysis B: Environmental 163 (2015) 404-414.

2- Jihène Jemal, Carolina Petitto, Gérard Delahay, Zouhaier Ksibi and Hassib Tounsi. Selective catalytic reduction of NO by NH_3 over copper-hydroxyapatite catalysts: effect of the increase of the specific surface area of the support. Reac Kinet Mech Cat DOI 10.1007/s11144- (2014) - 0762-7.

3- Asma Turki, Chantal Guillard, Frederic Dappozze, Gilles Berhault, Zouhaier Ksibi, Hafedh Kochkar. Design of TiO_2 nanomaterials for the photodegration of formic acid-Adsorption isotherms and kinetics study. Journal of Photochemistry and Photobiology A: Chemistry 279 (2014)08-16.

4- M.Triki, Z. Ksibi, A. Ghorbel, F. Medina: "Preparation and characterization of CeO_2-Al_2O_3 aerogels supported ruthenium for catalytic wet air oxidation of p-hydroxybenzoic acid", Journal of Sol - Gel Science and Technology, 59 (2011)1-6.

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Effect of Chemicals on the Environment and on health.

KSIBI Zouhaier

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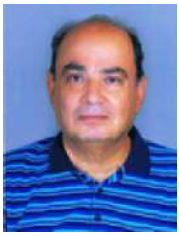
Faculté des Sciences de Tunis – Université de Tunis El Manar - Tunisie

Chemicals invading our world and are part of our daily life , they cause significant damage to the environment (pollution of soil , water and air , climate change , storms , natural disasters , the greenhouse effect, acid rain, ozone hole , deterioration of air quality and drinking water, several accidents in factories , ...) and cause various diseases to mankind and animals (increased cancer rates , kidney and liver damage, significant levels of pesticides in food and drinking water, appearance of new unknown diseases , extinction of animal species , ...) . Unfortunately the lack of user awareness and lack of will of one part of industry and governments cause growth problems caused by chemicals. The extent of this damage requires civil society and the political world to respond and to take urgent steps to improve the environment and protect the health of living beings in order to apply the concepts of healthy sustainable development for present and future generations . It is for the attention of chemists and chemical users to the dangers they represent and to push them to being more Vigilant and more cautious that I address this conference entitled: Effect of Chemicals on the Environment and on health.

Adel M'NIF



Prof. Dr. Adel M'NIF, professor of inorganic chemistry and chemical engineering industry, he graduated in chemical engineering from the National School of Engineers of Tunis (ENIT) in June, 1976, was awarded the Diploma of Advanced Studies in Inorganic Chemistry in September 1978 at the Faculty of Tunis and got the PhD doctorate in inorganic chemistry in 1984 from the Faculty of Sciences of Tunis and had his HDR in chemistry in 2001 from the Faculty of Sciences of Sfax.

Prof. Adel M'NIF held the following positions:

- Senior Engineer and Chief Engineer, Head of minerals Division in the national Office of Mines,
- Assistant Director of Research Mining at the Ministry of National Economy (Directorate of Mines and Geology).
- Assistant Professor: Naval Academy Menzel Bourguiba (December 1989 – October 1995);
- Assistant Professor (October 1995-October 2003): National Institute of Scientific and Technical Research;
- Lecturer Professor (October 2003-2008): National Institute of Scientific and Technical Research/ National Centre for Research in Materials Sciences;
- Professor (2008-present) : National Centre for Research in Materials Science;
- Laboratory Director of natural resources Development and recycled materials 2007-2009;
- Director of the Valorization Laboratory of useful materials (2010-present);
- Member of Scientific Council of CNRSM.

Prof. Dr. Adel M'NIF participated in several research projects: potassium salts and magnesia extraction from natural brines, sodium sulfate extraction from natural brines, lithium characterization and extraction from natural brines, environmental impact of phosphogypsum released solution on natural medium, valorization of siliceous sand and silica studies. He is the author of over 50 papers published in international journals, participated in many national and international scientific events. Supervise a number of masters and thesis in the field of specialization.

Silica Sand Techno-Scientific and Economic Issues

Adel M'NIF

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After air and water, sand is the most used resource in the world. It represents a volume of international trade of $ 70 billion per year[1]. The most important uses are construction and industries. In the last case, the fields of use of silica are wide and varied; its main applications include foundries, glass industry, metal alloys, ceramics, abrasives, insulation, silicones, telecommunications or chemistry. World production of silica products, applications and all forms combined, amounts to more than 141 Mt in 2013 (USGS, 2013), of which about 90% for foundry and glass, 3% for metals and silicon alloys and 1% for silicon carbide and abrasives industry. The United States is the largest producer and consumer of industrial sand in the world with 52.5[2] Mt in 2013 with an average price value of about 49.6 dollar per ton.
In the construction field, USA consumes around 864 Mt in 2013 for a price average of 7.8 dollar per ton (15.73% of industrial sand price). This last remark shows the huge difference between the prices of two types of silica sand. Accordingly our attention will be given to industrial sand. Nevertheless the sands used in the industry are purer than those used as a filler of concrete. The fields of use of industrial sand depend on the nature and concentrations of impurities contained therein. For example, the coloration of a glass depends on the purity of the sand used as a raw material, and especially metallic oxides as Fe_2O_3. Silica for optical glass should have the following composition: SiO_2 = 99.8%, Al_2O_3= 0.1% and Fe_2O_3 = 0.02%. A uniform particle size is also an important criterion. Given these few observations, we present in this work, industrial silica sand in terms of world production, prices and physicochemical characteristics in relation to industrial applications. After that, we will provide search results on Tunisian silica sand. In this respect we present results of silica purification followed by the preparation of sodium silicate and its applications for waste water purification and for porous hollow silica particles preparation. We present finally the use of silicate for the transformation of phosphogypsum into sodium sulfate and calcium silicate.

[1] Denis Delestrac, « Le sable, enquête sur une disparition »
[2] USGS: United States Geological Survey

Hanifa EASA TAHER

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Hanifa Taher earned his PhD in Chemical Engineering from UAE University, in 2014. Dr. Taher research works were on the enzymatic production of biodiesel from lipids extracted from different feedstocks using supercritical technology, and developing and integrated production process. These resulted in 1 patent in publication, total 10 peer-reviewed review and research papers and 5 international conferences in UAE and Canada. Her PhD research work was examined by Prof. Richard Smith, who is the Regional Editor, of Supercritical Fluids journal. During her M.Sc, She held a teaching assistantship at UAEU, and a visiting scholar in VCU, Virginia, USA. Dr.Taher reviewed some of research articles submitted for publications to Biomass and Bioenergy, Fuel journals, and Journal of Supercritical Fluids. In addition, Hanifa was member in the organizing committees of several events organized by UAEU; including Renewable Energy Conference in 2010 and 2012, and Future UAE Energy and Environment leaders' Competitions in 2013 and 2014. Her fields of interest are in the biofuel production, supercritical technology and enzymes catalysis. Recently, she received a research fund from National Research Foundation for a proposal titled; Green technologies for biofuel production, aiming to utilize some of industrial waste.

Utilization of Green technologies in the Biofuel production from lipid-rich feedstocks

Hanifa Easa Taher

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Currently, the world energy demand is mainly met by fossil fuels, which are being depleted, resulting in an increase in their prices. At the same time, the global environment is threatened by the emissions of harmful gases. Reducing the dependency on fossil fuels is crucial. The most sufficient way to achieve this goal is to find alternative fuels that are sustainable, cost-effective and environmentally-friendly. Recently, biodiesel has received much more attention, where soybean, rapeseed, palm and corn oils are the primary feedstocks. However, production from these crops is limited to many factors. Wastes and biomass can be used as feedstock. The commercial production processes have several environmental issues and require feedstock pretreatment, which otherwise would cause saponification. In addition, such processes require complex biodiesel purification, which further increases the production cost. The overall process also involves many steps, of which the lipids extraction is the most important and costly. Therefore, searching for an effective process is critical.

Various approaches were considered, and we had proposed an integrated process using green solvents. We had developed an environmentally-friendly process for production from waste fats and microalgae lipids. In addition, the leftover after extraction can possibly be used in other applications.

Oral Communications' List

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Name	Ref
K. Abderrazek, N. Frini Srasra *CNRSM - Borj Cédria* Effect of operating parameters on the photocatalytic activity of [Zn-Al] LDH	**OC19B**
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R. Abu-Zurayk *University of Jordan* Factors affecting interfacial interaction between polyethylene matrix and clay layers in polyethylene/clay nanocomposites	**OC15A**
A. Al Bawab *University of Jordan - Jordan* Spontaneous emulsification in the system water, ethanol and benzene	**OC4C**
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A.A. Azzaz, S. Jellali, L. Bousselmi *CERTE - Borj Cédria* Optimization of methylene blue adsorption onto chemically modified orange sawdust using the response surface methodology approach	**OC20A**
R. Banani, Y. Snoussi, M. Bezzarga, M. Abderrabba *IPEST - La Marsa* Waste Frying Oil with High Levels of Free Fatty Acids as one of the prominent sources of Biodiesel Production	**OC6C**
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S. Ben Mahmoud, W. ESSAFI, F. BOUE *INSAT - Tunis* Structure variation of polyelectrolyte with hydrophobicity: Confirmation of the pearl-necklace model	**OC9C**
N. Ben Salah, H. Casabianca, I. Essaidi, S. Chenavas, C. Sanglar, A. Fildier, N. Bouzouita *FSM - Monastir* New flavonoid from *Diplotaxis virgata*, structural elucidation by HRMS and NMR, and antioxidant ACTIVITIY	**OC4B**
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H. Benaissa, Z. Senouci - Berekci *University of Tlemcen, Algeria* Equilibrium and kinetics studies of methylen blue sorption from synthetic aqueous solutions by thistle stalks: Effect of pH and attempts of desorption	**OC18C**
H. Benmoussa, S. Chamali, A. Farhat, J. Bouajila, M. Romdhane *ENIG - Gabès* Green and eco-friendly process for extraction of essential oil from dry anisum seeds: Microwave hydrodififfusion and gravity	**OC10C**

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N. Bouazizi, R. Ouargli, S. Nousir, R. Benslama, A. Azzouz *ENIG - Gabès* Elaboration and characterization in iron nanoparticles incorporated SBA-15 mesoporous materials via the in-situ dispersion method	**OC4A**
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C. Elazizi Riahi, H. Hammi, H. Majdoub, A. M'nif *CNRSM - Borj Cédria* The impact of the substitution of the portland cement by the '*Opuntia ficus indica* 'and kernels of dates on the properties of mortar	**OC15B**

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M. Ghazouani, H. Akrout, L. Bousselmi *CERTE - Borj Cédria* Anodic oxidation of tetracycline on BDD electrodes: Kinetic study and effect of the nature of the supporting electrolyte	**OC13C**
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A. Midouni, M.I. Houchati, A.H. Hamzaoui, A. M'nif *CNRSM - Borj Cédria* Calcium hydride as a powerful reducing agent for topotactic oxide deintercalation: Synthesis, characterization and structural properties of topotactically reduced $La_{2-x}Ca_xCuO_4$ (0£ x< 0.2).	**OC10B**
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M.F. Nsib, N. Naffati, A. Rayes, N. Moussa, A. Houas *ENIG - Gabès* The hybrid Ni-TiO_2/PANI as a catalyst for hydrogen photoproduction under visible light: Effect of some operational parameters	**OC11B**
F. Odeh *Univeristy of Jordan* Nanoflora: How nanotechnology enhanced the use of active phytochemicals	**OC1A**

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M. Stouri, E. Srassra, L. Billon *CNRSM - Borj Cédria* Synthetic methodology effect on the microstructure and thermal properties of poly(MMA-stat-sty)-b-PSSNa synthesized by nitroxide mediated polymerization	**OC17A**
S. Teka, A. Gaied, N. Jaballah, M. Majdoub *FSM - Monastir* Electrochemical detection of Cu^{2+}, Pb^{2+} and Hg^{2+} ions using new β-cyclodextrin-based semi-conducting rotaxane	**OC1C**
I. Tlili, G. Caria, S. Net, I. Ghorbel-Abid, B. Ouddane, R. Ternane, M. Ayadi-Trabelsi *FSB - Bizerte* Quantification of 26 pharmaceutical residues in wastewater and river waters using on-line SPE-LC-MS/MS	**OC19A**

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F. Walha, M. Khemakhem, A. Maazouz, M. Jaziri *ENIS - Sfax* Toughening modification of poly(lactic acid)/polyamide11 blends via in situ reactive compatibilization	**OC2C**
A. Youssef, Y.M. Issa, Ola R. Shehab, H. Sherief *University of Cairo - Egypt* Determination of microgram amounts of copper in real samples by using chemically modified carbon paste electrode	**OC20C**
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Oral Communications' Abstracts

Program of Monday, March 16th 2015

Nanoflora: How Nanotechnology Enhanced the Use of Active Phytochemicals

Fadwa Odeh

Associate Prof. Physical Chemistry
Department of Chemistry/The University of Jordan
Amman-JORDAN

This chapter will try to shed light on the various advancements that were made in tackling issues of stability, solubility, toxicity and formulations in addition to other problems that usually accompany active chemicals extracted from natural resources with nanotechnology. This chapter will include:

- Introduction: An overview of nanotechnology, nanoparticles, drug delivery systems, drugs and their two main resources – natural and synthetic-, and importance of the continuous efforts to design and discover new medications.
- Difficulties encountered in bio-active phytochemicals: Such problems include limited bioavailability due to solubility issues, instability of such compounds, instability of their various formulations, and toxicity of most of these phytochemicals.
- Role of nanoparticles in overcoming such problems: An overview on advantages of nanoparticles and the various types that can be used depending on the active compounds need to be delivered. The several parameters that need to be taken into consideration in designing nanoparticles for phytochemical encapsulation.
- Successful stories: Several examples on drugs reached the markets in which nanotechnology was capable of solving the various issues accompanying active compounds originated from natural resources.
- Conclusions: Highlights of the most important factors affecting this field and its future impact on the quality of life.

Setbacks in using phytochemicals as new medicines. Even though natural resources are considered a major source for new drugs used to improve quality of life throughout history, active phytochemicals suffer from different disadvantages which, in turn, make them difficult to use in drug formulations.

THERMAL, STRUCTURAL AND OPTICAL PROPERTIES OF Er^{3+} DOPED PHOSPHATE GLASSES CONTAINING SILVER NANOPARTICLES

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A series of silver nanoparticles (AgNPs) embedded phosphate glasses belonging to (42.25-x) P_2O_5-(42.25-x) Na_2O-15ZnO - 0.5Er_2O_3 - xAg_2CO_3 (x = 0; 0.5; 1 and 1.5 %) are prepared by the conventional melt-quenching technique. The purpose of this work is to investigate the structural changes that appear in the phosphate glasses matrix with the addition and increasing of silver content. From the differential thermal analysis (DTA) measurements, it was found that ΔT increases with the increase of Ag NPs concentration. The transmission electron microscopic images confirm the presence of spherical silver NPs having average diameter in the range of 20-40 nm. The EDX analysis shows the presence of Ag nanoparticles. The glass sample containing AgNPs exhibits the surface Plasmon resonance (SPR) band of Ag NPs centered at 403 nm. The FTIR and Raman spectra reveal a band shift due to the incorporation of Ag NPs with different concentrations that consequently affects the bonding between ligands in the glass matrix and induced a depolymerisation of phosphate groups. Optical band gap is found to decrease with increase of Ag NPs concentration. Our results suggest that the proposed glasses can be nominated as potential materials suitable for solid state lasers and optical devices.

Keywords: phosphate glasses, silver nanoparticles (Ag NPs), Thermal stability, Raman spectroscopy.

Preparation of nanocomposite by adding of modified clay in unsaturated polyester: Thermal and mechanical study

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Unsaturated polyester (PE) resin is widely used for the matrix of composites such as fiber reinforced plastic (FRP) and polymer composites. Consequently, inexpensive and high performance resins are important for the future of polymer composites.
One recent method for enhancing the performance of polymer composites is the manufacture of PE-MMT nanocomposite synthesized by dispersing the silicate layers of MMT into the PE resin.

The chemical modifications of the clay cause a desired effect on its final properties improving the performance of the nanocomposites.

In our study we are used four types of clay: pure montmorillonite (MMT), purified montmorillonite (MMTb), complexed montmorillonite (MMTc) and activated montmorillonite with ammonium quaternary (MMTa). All these clays were mixed separately with the polyester resin and tested with mechanical analysis, X-ray diffraction, thermal analysis (DSC and TGA) and transmission electron microscopy. The results of compression strength given in Fig 1 and table 1 of all types of nanocomposite, indicate that the mechanical properties of PE-MMT nanocomposites are better than those of pure PE. The 2 wt % added clay showed the highest result.

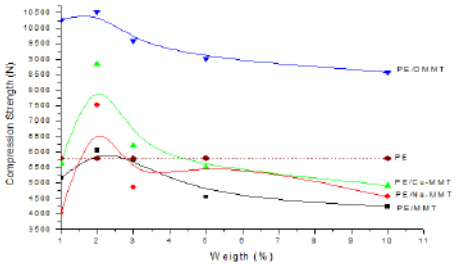

Fig 1.Compression Strength of PE PE/MMTb, PE/MMTp, PE/MMTc and PE/MMTa with wt% of clay.

Table 1

	1%	2%	3%	5%	10%
PE (N)	5797.1	**5797.1**	5797.1	5797.1	5797.1
PE/MMTb (N)	5156.1	**6050.6**	5751.6	4545.2	4243.5
PE/MMTp(N)	4017	**7533**	4843.5	5814.5	4551.6
PE/MMTc(N)	5650	**8840**	6200.3	5514.1	4912.6
PE/MMTa	10275	**10528.1**	9600	9015.8	8582.2

Key words: Polyester, nanocmposite, montmorillonite.

Elaboration and characterization in iron nanoparticles incorporated SBA-15 mesoporous materials via the in-situ dispersion method

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SBA-15-Fe were synthesized via the in-situ dispersion of Fe (0) nanoparticles (M-NPs) in SBA-15, is an interesting mesoporous silica material having highly ordered nanopores, which is widely employed as absorbents supports. M-NPs incorporated by the in-situ dispersion methods is in order to modify their functionality, which makes change structural and absorb the total hydroxyl groups (peak around 954 cm^{-1}).

Key words: SBA-15, XRD, Iron nanoparticles, hydroxyl groups.

Tunisian Air Quality Monitoring Network Presentation

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It is among the basic missions of the National Agency for Environmental Protection (NAEP) to preserve and improve the quality of our nation's air. In order to accomplish this mission, the Agency must be able to evaluate the status of the atmosphere compared to clean air standards and historical informations. That's why the NAEP established since 1996 an Air Quality Monitoring Network (AQMN) composed by thirteen permanent monitoring stations, two mobiles laboratories and a data elaboration centre with the aim of characterizing human health exposure and public impacts of conventional pollutants.
In Tunisia, that extends over 163900 km² and includes 24 metropolitans towns, there are several permanent monitoring stations located in many towns like Tunis, Sfax, Sousse, Gabès Tozeur, Kef.
In this paper, some stations of Tunisia and its monitoring networks have been selected as representative sites of the investigated area. In Tunis, the selected monitoring stations were located in Traffic area (Bab Saadoun), in urban area (El Mourouj) and in a suburban area (Nahli).
Each station is provided with automatic analyzers of O_3, NOx, SO_2, PM10, CO and with meteorological sensors such as temperature, barometric pressure, relative humidity, solar radiation, wind speed and direction.
Nitrogen oxides, NO and NO_2, were analysed using the chemiluminescence method. The measurement of ozone is based on the capacity of such gases to absorb ultraviolet rays with opportune wavelengths, generated by built-in lamp. Carbon monoxide CO is analyzed infrared rays absorption (IR). The measurement of PM10 is based on the beta ray attenuation method while SO_2 is analysed using a fluorescence method.
For all parameters, the data are collected by the monitoring network every hour. The data were validated according to this protocol: first, a preliminary validation was carried out by the software, which invalidated all the data occurred in calibration hours and the data identified as artifacts then a manual calibration was carried out by the operators, considering the relations among the parameters. This way it is possible to verify that eventual critical data are related to real pollution situations. Moreover, meteorological data (wind speed and direction) were used to investigate about the influence of natural events on high or low concentration situations.

Key words: Air Monitoring, Tunisia, automatic analyzers, Permanent stations, air pollutants.

CHARACTERISATION AND PHOTOLUMINESCENT PROPERTIES OF NEW PYROCHLORE SOLID SOLUTION $GdEuTi_2O_7$

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A wide range of pyrochlore-type dititanate compounds ($RE_2Ti_2O_7$) have been synthesized and studied from a structural, magnetic and electrical point of view, they could be also a suitable system for understanding relationships between luminescence properties and crystal structure. Therefore, the present research focuses on the photoluminescence properties as well as structural characterization of new photoluminescent pyrochlore $GdEuTi_2O_7$ synthesized using solid-state reaction.

X-ray diffraction (XRD) combined to the Raman spectroscopy were employed to study the structure evolution of the sample and to determine the crystalline phases present after heat treatment. A well-crystallized single-phased was obtained after calcination at 1200°C. FTIR spectra of $GdEuTi_2O_7$ powder shows the absorption bands corresponding to the pyrochlore structure $A_2B_2O_7$ and give information about the distribution of ions between the A and B sites. The X-ray Rietveld analysis indicated an overall $A_2B_2O_7$ cubic pyrochlore structure (Fd3m) with a = 10.197 Å and confirmed that Eu^{3+} ions are located in the A^{3+} site with inversion symmetry.

When the Eu^{3+} ions locate in the sites with the inversion center, the orange emissions become relatively strong, while the red emissions become very weak because electronic dipole transitions are parity forbidden. This is consistent with the observed results in PL emission spectra in which the $^5D_0$–$^7F_1$ magnetic dipole transitions of the Eu^{3+} ions were split into two lines A and B (λ_1=589, λ_2=597 nm). The excitation measurements obtained by monitoring the emission of Eu^{3+} $^5D_0$–$^7F_1$ transition at 590 nm yield a weak and broad band with a maximum at about 300 nm attributed to the $Gd_2Ti_2O_7$ host excitation band. The presence of the $Gd_2Ti_2O_7$ host band in the excitation spectrum of Eu^{3+} indicates that there exists an energy transfer from the host to the doped Eu^{3+}.

However, a number of sharp peaks centered from 360 to 470 nm are ascribed to the intra-$4f^6$ transitions of Eu^{3+}.

The effects of reprocessing cycles, residence time, and specific mechanical energy in high shear twin-screw extrusion on the structure and properties of silica reinforced polypropylene/ ethylene-propylene-rubber nanocomposites

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This work focuses on the effect of repeated processing cycles on the structure and properties of nanosilica-thermoplastic olefin elastomer (TPO) nanocomposite through a series of three extrusion cycles using a high shear twin screw extruder by varying the speed screw rotation (300; 800 and 1200 rpm). The content of silica nanoparticles was set to 3wt% and the nanocomposites were compatibilized with polyethylene modified with maleic anhydride MAPE. Rheological, morphological, thermal, and mechanical Analyses were carried out on as-produced and reproduced samples in order to explore different parameters such as number of extrusions, shear rate level, SME (specific mechanical energy) and residence time, on properties of the recycled systems.
The analysis of the different results illustrate that the mechanical and the rheological properties were affected by the multiple extrusion. In particular, this process provokes a decrease of the molecular weight and the melt flow index. The first recycling procedure induced an increase of the Young's modulus and tensile yield stress, while for higher recycling numbers, these two parameters dropped. The MAPE copolymer stabilized the tensile elongation at break at 3 recycling procedures due to a decrease of the size of EPR nodules and a homogenization of their shape, while that of neat (PP/EPR) continuously decreased with recycling numbers.

Key words: residence time, SEM, nanocomposites, rheology.

FERRITES BASED NANOMATERIALS FOR BIOMEDICIAL AND MODERN TECHNOLOGY PURPOSES

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Spinel–type ferrites based nanomaterials have attracted increasing interest in recent years due to their unusual physical and chemical properties. Mixed Zn nanoferrites (MZnNFs) are promising for several applications in the areas of biomedicine and technology [see for instance 1,2]. We have focused our research work on the MZnNFs based materials (i) $Co_xNi_{0.5-x}Zn_{0.5}Fe_2O_4$ and (ii) colloids of $NaYF4:Ln^{3+}$ (Ln = Eu; Tb; Eu/Yb;…)@$Co_{0.6Z}n_{0.4}Gd_xFe_{2-x}O_4$ for high frequency and diagnosis/therapy applications, respectively. For the core@shell part of (ii), controlling the chemical composition and the microstructure is crucial to adjust the magnetic and the optical properties. We have succeeded to obtain highly crystalline and stoichiometric powders of MZnNFs and n anoparticles of the core, $NaYF4:Eu^{3+}$, utilizing the polyol method. Nanoparticles of (i) were subsequently sintered at different conditions in order to optimize their magnetic and electrical characteristics. Compared to bulk ferrites counterparts, improved characteristics were obtained [3]. For (ii), the magnetic properties of the shell were discussed in relation to their structure and microstructure [4]. Finally, attempts to design the core@shell part of (ii) and to functionalize its surface with appropriate organic molecules are in progress.

Key words: Nanoparticle, Ferrite, Magnetic, Electric

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PHOTOCATALYTIC ACTIVITY OF Ag DOPED ZnO AND THE INVESTIGATION OF ITS ANTIBACTERIAL ACTIVITY

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Zinc oxide, a typical kind of II–IV compound semiconductor with a wide band gap (3.37 eV) and a large excitation binding energy of 60 meV, has been widely used as a photocatalyst, owing to its high activity, low cost, and environmentally friendly featured in gas and liquid phase pollution control [1]. However, the efficiency of photocatalytic degradation by ZnO should be further improved in order to meet the requirements of environmental protection. Many approaches have been pursued to promote the photocatalytic performance, such as modification of ZnO by noble metals like platine, silver and gold [2].

In this work, we have successfully prepared ZnO modified catalysts with various contents of silver by the hydrothermal method. The samples characterization was investigated using X-ray diffraction (XRD), Nitrogen physisorption at 77K; Fourier transformed infrared spectroscopy (FTIR), UV-Visible spectroscopy and Photoluminescence spectra (PL).

The textural, structural and optical properties of pure ZnO and Ag-doped ZnO photocatalysts with different Ag loading are illustrated in Table 1. It reveals that the average crystallite size does not change with low content of Ag (0.5 - 1%) then it decreases with high content of Ag (2 - 4%). The specific surface area (S_{BET}) increases with the increase of the Ag content.

The values of the band gap gradually decrease with the increase of the Ag doping level, and change in a small range.

Table.1. Textural properties, crystallite size and gap energy of the as-synthesized catalysts.

Sample	Specific surface area (m^2/g)	Cristal size (nm)	Eg (eV)
0% Ag-ZnO	14.93	59.5	3.31
0.5% Ag-ZnO	15.30	59.5	3.28
1% Ag-ZnO	15.66	58.4	3.23
2% Ag-ZnO	16.77	51.6	3.22
4% Ag-ZnO	18.18	50.7	3.21

To evaluate the photocatalytic activity of the catalysts, the degradation of bisphenol A (BPA) and nonylphenol (NP) in the presence of Ag-doped ZnO with different Ag content was studied.

The results of the photocatalytic degradation showed that silver ions doping greatly improved the photocatalytic efficiency of ZnO. The TOC conversion of BPA and NP are 72.1% and 81.08% respectively obtained using 1% Ag-doped ZnO. The modification of ZnO with an appropriate amount of Ag can increase the separation efficiency of photogenerated electrons and holes in ZnO. The antibacterial activity of the catalysts using Escherichia coli as a model for Gram-negative bacteria confirmed that Ag-doped ZnO possessed more antibacterial activity than the pure ZnO.

Key words: Ag-doped ZnO, endocrine disrupting, photocatalytic activity, antibacterial activity.

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NANO FIBROUS CERIUM(IV) HYDROGEN PHOSPHATE MEMBRANE SELF SUPPORTED ANILINE POLYMERIZATION AGENT

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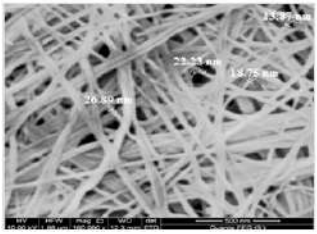
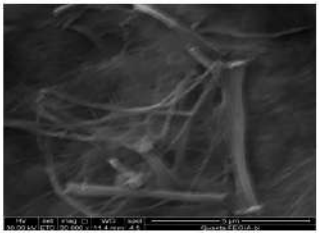
Conducting polymers polyaniline, polypyrrole and polyindole have received increasing attention in various fields, electronics,and others. Nanosized fibrous cerium(iv) hydrogen phosphate membrane, $Ce(HPO_4)_2 2.9H_2O$ ($nCeP_f$), was prepared and characterized. Its average size found to be ~20.5 nm. Nanofibrous cerium phosphate / polyaniline nanocomposite membranes were prepared via in-situ chemical oxidation of the monomer that was promoted by the reduction of Ce(iv) ions present in the inorganic matrix.. The resultant materials were characterized by TGA, elemental (C,H,N) analysis, FT-IR and scanning electron microscopy(SEM). SEM image of $nCeP_f$ is shown in Figure(1). Typical SEM images of the resultant nanocomposites are shown in Figure (2) reveals a uniform distribution of the polymer on the inorganic matrix, coating the $nCeP_f$ and filling the empty spaces between the fibers. Inspite of the likely conversion some of $nCeP_f$ to $CePO_4$, nanocomposites present fibrous morphology. A possible explanation is $nCeP_f$ present on the surface of the fiber is attacked by the aniline and converted to cerium(III) orthophosphate. The rest of the $nCeP_f$ was preserved. The degree of polymer loading and its type can be controlled by varying method of the prepartion. Accordingly two types of green emeraldine salts (in form of chloride and sulfate salts) were obtained. Amount of polyaniline present in the resultant composites found to be in the range of(4.5- 16.74 %wt.).





Figures (1, 2)

OPTIMIZED PREPARATION OF MONOLITH BARS AS PASSIVE SAMPLERS OF POLLUTANTS AND STUDY OF THEIR ADSORPTION CAPABILITY

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Pollution of surface water by chemicals has always been a threat to the aquatic ecosystems and to biodiversity. For this reason, new directive was adopted in order to protect surface waters from pollution. Most conventional screening methods for pollutants in water involve grab sampling providing only the level of pollutants at the moment of sampling. However, the concentrations of chemicals can fluctuate very rapidly and often a frequent sampling is required. In order to overcome these limitations, passive sampling technology was proposed as a new alternative. The operating principle of passive sampling is based on the accumulation of contaminants by passive diffusion into the absorption/adsorption devices over long durations.
In the current work, we have synthesized monolith bars via sol-gel method as solid supports and performed their surface modification to provide them with an affinity to organic pollutants. The morphology of the solid supports that control the accessibility to adsorption sites and the kinetics of adsorption have been firstly investigated. The variation of several synthesis parameters allowed the production of three monolithic materials with different macro porosities.

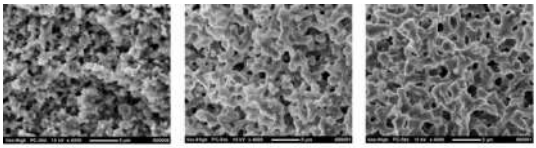

Figure 1: Effect of increasing porogen concentrations in the synthesis recipe on macroporosity (a: 3.2%; b: 3.6%; c:4.0%)

The monolith bars was hydrophobized by grafting of alkyl chains. The grafting density was estimated to 1.3µmol.m^{-2}. Comparison study for the evaluation of performance of modified and unmodified monoliths was conducted.
Modified monoliths showed significantly high adsorptivity compared to unmodified ones. The adsorption kinetics were best described by a pseudo-second order model. Combination of morphology characterizations in terms of macro- and mesoporosity from BET measurements and modeling of kinetic data provide additional insights to better understand the mechanisms involved in the passive sampling by such devices.

DEXAMETHASONE LOADED CORE-SHELL SF/PEO NANOFIBERS VIA GREEN ELECTROSPINNING

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Green electrospinning' is defined that electrospinning of polymers from aqueous solution avoiding harmful organic solvents [1], it's a significant step forwards safe and clean electrospinning. SF has been widely investigated as a biomaterial for biomedical applications, such as tissue engineering and drug carrier due to its perfect biodegradability, biocompatibility and mechanical properties [2]. To increase the viscosity of the solution and obtain a suitable surface tension for generating stable continuous spinning, PEO was commonly added into SF solution. Moreover, the PEO phase could be extracted from SF and the porosity and surface roughness of nanofibers would be enhanced [3]. Silk fibroin (SF)/PEO nanofibers prepared by green electrospinning are safe, non-toxic and environment friendly, with potential as drug delivery carrier for tissue engineering. In this study, Dex@SF/PEO core-shell nanofibers were obtained via green electrospinning with SF/PEO as the shell and dexamethasone (Dex) in the core. The morphology and core-shell structure of nanofibers were studied by Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM). The Dex release behavior from nanofibers was tested by high performance liquid (HPLC) method. TEM results indicated the distinct core-shell structure of nanofibers. *In vitro* drug release studies demonstrated that dexamethasone could keep sustained release over 192 hrs. The core-shell nanofibers showed slower release of Dex compared with the blending electrospinning nanofibers.

Key words: Electrospinning, Nanofibers, Controlled release, Dexamethasone

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IN SEARCH OF NEW MAGNETOCALORIC EFFECT MATERIALS

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Intermetallic compounds based on rare earth (R) and transition metals (M) have received considerable attention due to their interesting magnetic properties (permanent magnets, high density magnetic recording, magnetic refrigeration, etc) needed for several technology applications. In this paper, we are interested in the development and study of new magnetic materials based on rare earth and transition metal, i.e., RFeAl compounds. Several compounds have been elaborated by arc furnace melting followed by annealing in silica bulbs sealed under secondary vacuum. At the end of the elaboration, we conducted a systematic crystallographic study by X-ray diffraction in order to define the nature of the phases present in the elaborated samples and their relative proportions. This was made possible with an analysis of the experimental data by Rietveld refinement. The results obtained show that single phase compounds can be obtained by simple fusion arc furnace. The elaborated compounds are characterized by Curie temperature varying between 410 K and 440 K depending on the amount of the added aluminum. The magnetic measurements have allowed us to highlight a magnetocaloric effect in these compounds. However, this effect is limited and could be improved by the substitution of iron by suitable metals.

Key words: magnetic materials, crystallographic study, X-ray diffraction, magnetocaloric effect.

SYNTHESIS AND CHARACTERIZATION OF BIO-INSPIRED HETEROGENEOUS CATALYSTS

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As part of the green chemistry approach, there is an increasing need of designing original materials. In bio-inspired catalysis we can exploit the basic principles of the enzyme active site functionality for creating new catalysts analogues. Carbon dioxide molecule is a highly stable nonpolar compound which activation requires the use of heterogeneous catalysts distinguished by their high stability and yet high aptitude to stimulate the CO_2 adsorption and then the electron transfer toward it. In this work, bio-inspired materials based on silica were synthesized following the structural model of carbonic anhydrase, a metalloenzyme that intervene in the breathing mechanism. An analogy between the enzyme active site and the molecular vicinity inside the synthesized catalyst are proposed using mesoporous silica and the molecular stencil patterning method to isolate the polyamine ligand before its complexation by divalent metals. The molecular vicinity was monitored in a way to mimic the enzymatic cavity. The structural and textural properties of the different materials were investigated using various techniques. Also, carbon dioxide adsorption behaviors on different catalysts were investigated. Indeed, its adsoption-desoption isotherms were measured to get a better understanding of the phenomena that occur on the surface of silica.

Key words: MCM-41, bio-inspired catalyst, carbon dioxide.

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Effect of electrochemical deposition methods of PbO_2 anodic layer on SS/SiO_x for the Amaranth dye degradation.

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Textile wastewaters are characterized by strong color, biotoxicity and its recalcitrant behavior against conventional treatments. It causes serious damage for the receiving water environment [1]. In this work, the electrochemical performance of SS (Stainless Steel)/SiO_x/PbO_2 as anode for the degradation of textile dye (Amaranth) was investigated. SiO_x film was deposited using Plasma-Enhanced Chemical Vapor Deposition (PECVD), by applying different percentages of gas (oxygen and argon), on (SS) substrate. PbO2 anodic layers were electrodeposited using galvanostatic (G) and Pulsed Current (PC) methods. Obtained layers were examined by different techniques. The morphology of SiO_x interlayer examined by Field emission scanning electrons microscope (FESEM) shows relatively homogeneous coatings. At larger scale, it is uniformly distributed on stainless steel substrate. PbO_2 composition formed on different anodic layers was examined using X-Ray diffraction (XRD). Results demonstrated that higher oxygen amount used for plasma SiOx deposition involves a faster nucleation of β-PbO_2 crystallites [2]. Color and COD removals at acidic pH reached after 300 min, 100% and 83% COD respectively for SS/SiO_x/PbO_2 (G), compared to 95% and 80%, respectively, using SS/SiO_x/PbO_2 (PC) anode. It was concluded that preparation methods could controllably affect the structure of PbO_2 films, which further played important role in the performance on dye degradation.

Key words: SiO_x, Electrochemical treatment, PbO_2 anodic layer, Amaranth.

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NEW FLAVONOID FROM *DIPLOTAXIS VIRGATA*, STRUCTURAL ELUCIDATION BY HRMS AND NMR, AND ANTIOXIDANT ACTIVITIY

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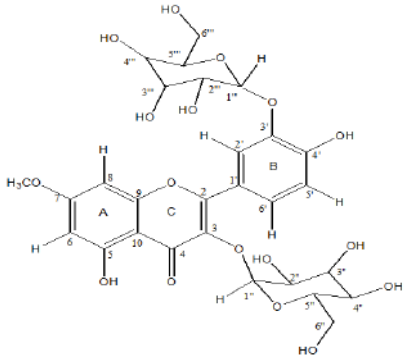
D.virgata is a perennial plant distributed in many sandy and calcareous areas of Mediterranean basin. In Tunisia the concentration of distribution of these species is especially high in South. This plant is economically important; it is used as alternative food and for medical purposes. The present work describes the guided fractionation of the ethanolic extract of *D. virgata* flowers which is concentrated under reduced pressure and partitioned successively with hexane, chloroform, ethyl acetate, n-butanol and water.

n-butanol extract of flowers were separately subjected to purification on C18 SPE cartridge to yield new compound **Rhamnetine-3,3'-di-O-β-glucopyranoside**. Structural elucidation was carried out using conventional 1D and 2D NMR and high resolution mass spectrometry. The antioxidant activity of butanol extract and isolated compound was evaluated using $DPPH^{\cdot}$ and $ABTS^{\cdot+}$ scavenging assays. Results showed that the purified compound had a better activity than the whole extract in both tests.



Rhamnetine-3,3'-di-O-β-glucopyranoside

Key words: *D. virgata*; Flavonoids; NMR; Antioxidant activitiy.

STUDIES OF PHOSPHORUS REMOVAL FROM AQUEOUS SOLUTIONS BY BIOCHARS DERIVED FROM CHEMICALLY PRETREATED LIGNOCELLULOSIC BIOMASS

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Biochars, which are carbon-enriched and microporous materials resulting from thermal treatment of natural biomasses in oxygen limited environment, have been identified this last decade as promising materials for the removal of various organic and mineral pollutants from wastewater. The ability of biochars (prepared from pyrolysis performed at 400°C; 500°C and 600°C of pretreated cypress sawdust impregnated with $MgCl_2$) in removing phosphorus from synthetic solutions was investigated in batch reactor. The effects of several experimental parameters including contact time, initial phosphorus concentration, pH and biochar loads on the sorption of phosphorus were examined. The experimental results indicated that the cypress sawdust pretreatment with $MgCl_2$ induced important modifications of the physical and chemical biochars' properties favoring phosphorus removal from the aqueous solutions. For all the tested biochars, the adsorption kinetics data were best described by the pseudo-second-order model, and equilibrium was achieved after 120 min of contact time. Furthermore, the higher is the pyrolysis temperature, the more phosphorus removal efficiencies are. Indeed the adsorbed phosphorus amounts increased from 19.2 mg/g to 33.8 mg/g when the pyrolysis temperature increased from 400°C to 600°C. Moreover, the phosphorus removal efficiencies increased with increasing the initial concentration and adsorbent dosages. These results suggest that the biochars derived from $MgCl_2$ pretreated cypress sawdust has potential use in remediation of solution contaminated by phosphorus.

Key words: Cypress sawdust, pyrolysis, biochar, phosphorus, adsorption, batch reactor.

Preparation of macro-porous membrane using natural Kaolin, Alumina and Tunisian lignite as a pore forming agent

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In this study the objective has been to prepare a low cost macroporous ceramic membrane using natural Tunisian Kaolin, Alumina and natural lignite as porosity agent. The characteristics of this membrane have been defined to be used on filtration processes and as a support for multilayer ceramic membranes. The study includes the characterization of the raw materials in order to define optimal processing parameters to obtain the membranes. A study of the effect of the percentages of lignite and of Alumina have been made and a percentage of 20% of lignite, 15% of Alumina and a temperature of 1200°C were chosen considering the porosity, density and mechanical strength as parameters to be optimized. The membranes show good porosity above 36% but with a low mechanical strength that doesn't exceed 34 MPa. These membranes can be considered as efficient regarding the good results shown in the gas permeation tests.

ELABORATION AND CHARACTERIZED OF NANO-FOAM POLYMERS WITH GIANT PERMITTIVITY

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Polymer materials with giant permittivity stand for an important position. Therefore, these materials can be considered as a solution to the request for both for alternative source of energy and for better energy storage solution.
The objective of the present work is to combine the nano-foam properties to those of the constituent polymer and ionic liquid system in order to create an attractive material from the viewpoint of the dielectric properties.
Adding ionic liquid to Acrylonitrile Butadiene Styrene (ABS) causes a significant decreasing in the glass transition temperature (Tg). Thermal behavior is characterizated by using differential scanning calorimetry (DSC). Same analysis technique is used to prove existence of ionic liquid after foaming. An increasing of permittivity values versus frequency after foaming is shown with dielectric measurement using Broadband Dielectric Spectroscopy (BDS).
Scanning Electron Microscopy (SEM) is used to complete that study by estimating porosity size and shape. We conclude that the more we increase ionic liquid proportion the more the foaming is heterogeneous and the less nanometric size is dominant.

Key words: Nano-foam, giant permittivity, ionic liquid.

(ETHYLENE-PROPYLENE) COPOLYMER/OLIVE SOLID WASTE COMPOSITES: EFFECT OF FILLER LOADING AND COMPATIBILIZER CONTENT ON RHEOLOGICAL AND PHYSICAL PROPERTIES

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In the olive oil-producing countries, large quantities of olive solid waste are rejected to nature causing by the way several environmental problems. Therefore, the use of this by-product as filler in (Ethylene-Propylene) matrix could be an effective way to reduce the significant quantities of the disposed biomass and to elaborate a cost-effective material. However, the hydrophilic nature of this natural filler decreases the compatibility with hydrophobic matrix. To improve the interfacial adhesion, maleated polypropylene was added as compatibilizing agent.

In a first step, the influence of filler loading on rheological, thermal and mechanical properties was investigated. Composites with various filler contents were prepared by melt blending using a twin-screw extruder and injection molding. The filler addition led to an increase of the viscoelastic properties and the degree of crystallinity. Then, the coupling agent (MAPP) was reported to enhance the rheological, thermal and mechanical properties of the composites up to a critical amount beyond which the platicizing effect becomes more predominant than the reinforcing effect, a fact that could be responsible for the decrease of viscoelastic properties, crystallinity and mechanical properties.

Key words: Olive solid waste, (Ethylene-Propylene) copolymer, Rheology, physical properties.

Removal of U(VI) from Simulated Liquid Waste using Synthetic Organic Resin

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A novel resin, poly(acrylic acid-dimethylaminoethylmethacrylate) was prepared using gamma irradiation technique at radiation dose 60KGy with dose rate 1.25kGy/min. The obtained organic resin used to removal of simulated solution of U(VI) that produced from the waste solution of Egyptian Fuel Manufacturing Pilot Plant (FMPP). Preliminary test of adsorption of U(VI) onto organic resin using X-ray fluorescence (XRF) confirmed high capacity of obtained organic resin towards U(VI). The relevant factors affecting uranium adsorption onto the adsorbent involved; pH values, contact time, temperature, and initial uranium concentration were studied. It was found that the optimum pH value at 5.00 for removal of U(VI) From the waste solution, the adsorption capacity was 105.7 mg/g and the uranium uptake $t_{1/2}$ at 5 min. The adsorption isotherm of uranium onto the resin was studied to Langmuir, and Freundlich adsorption models. The results showed that the obtained equilibrium data fitted well to the Langmuir isotherm. The obtained organic resin was promising to apply in removal of U(VI) from different liquid waste streams of the Egyptian Fuel Manufacturing Pilot Plant (FMPP).

Keywords: Organic Resin; FMPP liquid waste; Removal of Uranium.

Calcium Hydride as a Powerful Reducing Agent for Topotactic Oxide Deintercalation: Synthesis, Characterization and Structural Properties of Topotactically Reduced $La_{2-x}Ca_xCuO_4$ ($0 \leq x < 0.2$).

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The capability of calcium hydride as a reducing agent in oxide deintercalation reactions is explored. Reaction of the n = 1 Ruddlesden-Popper phase $La_{2-x}Ca_xCuO_4$ ($0 \leq x < 0.2$) with CaH_2 in a sealed evacuated tube at $200 \leq T/°C \leq 300$ yields the Topotactically Reduced phase $La_{2-x}Ca_xCuO_{3.5}$ (a = 8.6071 (4) Å, b = 3.8378 (8) Å, c = 12.9964 (4) Å). The variation in the anion vacancy distribution of these reduced phases as a function of stoichiometry is discussed in relation to the coordination polyhedra of the metal cations. Structural characterization indicates the coexistence of a new oxygen-vacancy-ordered arrangement of cooperatively- distorted Cu_2O_3 planes containing 4-coordinate Cu^+/Cu^{3+} sites.

Key words: nonstoichiometric oxides, topotactic reaction mechanism, Oxygen mobility.

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The hybrid Ni-TiO_2/PANI as a catalyst for hydrogen photoproduction under visible light: Effect of some operational parameters

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In order to improve the efficient use of solar energy to hydrogen production from water-splitting, Ni-TiO_2/PANI hybrid photocatalysts are prepared by direct and simple impregnation method. The prepared hybrid materials are characterized using X-Ray Diffraction (XRD), Scanning Electronic Microscopy (SEM), UV/Vis Diffuse Reflexion Spectroscopy (UV/Vis DRS), Thermogravimetric Analysis (TGA) and Fourier Transform Infra-Red (FTIR) techniques. It is mainly found that there is no change in the wurtzite structure of the prepared ZnO after Ni doping and no evidence of any secondary phases up to 10 mol% of the Ni content. Besides, the increase of the Ni content to 5 mol % changes drastically the Ni-ZnO particles from spherical to nanorod-like morphology. Moreover, it is shown that PANI extends the light absorption to the visible region, and thus is able of sensitizing ZnO efficiently.

The prepared hybrid photocatalysts are experimented for the hydrogen photoproduction from water-splitting under visible irradiations. The hydrogen production efficiency is measured in relation to some operational parameters such as the Ni^{2+} content, the amount of PANI, the mass of the catalyst and the nature of the added sacrificial electron donor. The obtained results showed that the hybrid Ni-ZnO/PANI has high photocatalytic efficiency in sustainable hydrogen production under visible irradiation.

Exploring the Nature of clopidogrel-sulfonephthalein dyes-Interaction via Spectrophotometric Measurements and Quantum Chemical Calculations

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Clopidogrel is an oral, thienopyridine class antiplatelet agent used to inhibit blood clots in coronary artery, peripheral vascular and cerebrovascular diseases. A spectrophotometric method was developed for clopidogrel bisulfate (CLPB) determination using the sulfonephthalein dyes, bromocressol green (BCG) and bromoxylenol blue (BXB), as ion-pairing agents. The quantitative analysis is based on extracting the yellow-formed ion-pair into chloroform from aqueous medium. They exhibit absorption maxima at 413 and 407 nm with BCG and BXB, respectively. The optimum conditions of the reactions were studied experimentally and optimized. The calibration graphs show that CLPB can be determined up to 115.0 µg mL^{-1}. The detection limit (LOD) is 0.57 and 1.14 µg mL^{-1} and the corresponding quantitation limits (LOQ) are 1.86 and 3.79 µg mL^{-1} for BCG and BXB, respectively. The low relative standard deviation values, 0.16-1.16, indicate good precision. The results were compared to other published and were treated statistically using F and t-tests. Moreover, to explore the interaction mechanism of CLPB with BCG as well as with BXB at molecular level scale, DFT calculations were performed. Full geometry optimization at B3LYP/6-311++G(d,p) was taken place for the individual molecules (CLPB, BCG, and BXB) as well as for CLPB reaction product with BCG and BXB. Furthermore, the vibrational frequencies and UV-visible spectra for the individual systems and their ion-pairs were calculated at the same level of theory.

Keywords: Clopidogrel bisulfate, Bromocresol green, Bromoxylenol blue, Molecular modeling, DFT calculations.

Electrochemical detection of Cu^{2+}, Pb^{2+} and Hg^{2+} ions using new β-cyclodextrin-based semi-conducting rotaxane

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An impedimetric sensor based on a new semi-conducting rotaxane (BPAAn/β-CD) has been described for detection of various metallic cations. The rotaxane consists of a π-conjugated semi-conducting material (BPAAn) encapsulated into β-cyclodextrin (β-CD). A thin rotaxane film was deposited by spin coating to develop a new electrochemical sensor. The wettability of the rotaxane was evaluated using contact angle measurements. The gold/rotaxane/solution interface was investigated by electrochemical impedance spectroscopy and the obtained data were fitted using an equivalent electrical circuit. The response of the gold/rotaxane membrane towards Cu^{2+}, Pb^{2+} and Hg^{2+} cations was studied and the results showed a good sensitivity to these ionic species.

Keywords: Rotaxane, β-Cyclodextrin, Electrochemical impedance spectroscopy (EIS).

Toughening Modification of poly(lactic acid)/polyamide11 blends via In Situ Reactive Compatibilization

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This work deals with the study of the reactive extrusion process of Poly (lactic acid)/Polyamide11 bio-sourced polymer blends. The main focus of this blend is to overcome the shortcomings of PLA including its thermal instability, poor shear and low elongation viscosities in order to enhance its processability and to expand its application area. However, the immiscibility between PLA and PA11 required the incorporation of a multi-functionalized epoxide named Joncryl as a reactive compatibilizer to improve the interfacial adhesion. The crystalline structure and the thermo-mechanical properties of blends were characterized thanks respectively to the Differential Scanning Calorimeter (DSC) and the Rheometrics Dynamic Analyzer (RDA). The phase morphology of blends was observed by scanning electronic microscopy (SEM). Miscibility and homogeneity of blends have been examined via Han plots and relaxation spectrum. Experiments results indicated that the compatibility between the polymers was significantly improved by *in situ* formation of PLA-*co*-PA11 copolymers in the presence of the GMA functions. Tensile test highlights an enhancement of the ductility of the compatibilized blends which was evidenced by an important increase of the elongation at break. SEM micrographs showed a decrease of dispersed particles size and a disappearance of co-continuous phase for blends containing epoxy-functionalized chains. The differential scanning calorimeter (DSC) results reveal that thermal properties of PLA were enhanced due to homogeneous nucleation effect of the *in situ* compatibilized PA11domains.

Key words: Bio-sourced polymer-Compatibilization-Thermo mechanical properties

PANI AS CORROSION PROTECTION COATINGS ON MODERN TIN BRONZE

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The conservation of the bronze artifacts from the damages requires the application of a barrier layers on the metal surface. However, few investigations have tested the implementation of coatings on Cu-Sn alloys [1-2].
The objective of this work is to explore the corrosion protection behaviour of electrochemically synthetized PANI on Cu-Sn alloy in NaCl 0.5M electrolyte.
The cyclic voltammograms shows that the introduction of monomer generates a significant change in the interfacial behaviour of the bronze electrode. The structure and the property of the formed films were characterized by FTIR spectroscopy, RAMAN spectroscopy and AFM microscopy. The corrosion performances of the bare and coated electrode without and with monomer were tested in a chloride electrolyte. The percent of protection efficiency (%PE) for the coated surface without and with monomer were 51% and 57% respectively. The influence of the immersion time on the protection performance of the coatings was considered. It was remarked that the %PE decreases to reach 12% after one hour of contact in the chloride electrolyte. The evolution of the corrosion rate with the rise of temperature was also evaluated. It was found that the %PE increase with temperature even at 323K (%PE = 74%) indicating the stability of the PANI coatings. Two behaviours were evidenced in the temperature range 300-323K. The activation parameters (Ea, ΔH^*, ΔS^*) were calculated in two temperature regions.

Key words: PANI, Corrosion protection, Tin bronze alloy, Activation parameters.

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SPONTANEOUS EMULSIFICATION IN THE SYSTEM WATER, ETHANOL AND BENZENE

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Spontaneous emulsification was introduced by Gad in 1878[1], who related the spontaneity of the process to the energy release at the interface chemical reaction between hydroxide groups in the aqueous phase and fatty acid molecules in the oil phase. This focus on interfacial phenomena in spontaneous emulsification for a long time[2] was the only mechanism under consideration, until Miller introduced the phase diagram/diffusion approach[3]. Miller calculated the diffusion path in the diagram of water, alcohol and oil and showed experimentally that, when the diffusion path entered a two-phase region, spontaneous emulsification occurred.

In the first experiment in this lecture two mutually insoluble aqueous solutions along the solubility limit in the water, benzene and ethanol system were brought into contact[4], resulting in spontaneous emulsification between the two aqueous solutions with a final outcome of an oil phase separated.

The emulsification took place at the interface between the solutions and the emulsion advanced into both of them, albeit slower into the top solution. The process was slow and equilibrium reached first after 24hrs, leaving two layers; an aqueous transparent solution and a corresponding oil layer on top. The initial changes showed the dimension of the lighter aqueous transparent layer to be reduced linearly as the square of time, indicating the main mechanism is governed by a diffusion process within the layer and transfer of ethanol to the heavier aqueous solution. The information from the phase diagram confirmed the process to be a transfer of ethanol into the heavier of the two aqueous phases. The dimensions of the aqueous and oil layers at the final equilibrium agree with the features of the phase diagram.

In the second experiment[5], emulsions; one O/W and one W/O were layered on a layer of water. For the O/W emulsion the emulsification now became highly complex. One aqueous emulsion immediately formed on top of the water layer, with a semitransparent aqueous layer on top of it and, finally, a much less concentrated W/O emulsion at the highest level of the original emulsion. With time (15 min) the transparent water layer on top of the dense O/W emulsion was linearly reduced to zero and the top W/O emulsion layer became transparent. These complex changes are examined with the phase diagram as basis. For the W/O emulsion the mechanism was simpler. Also in this case, a dense emulsion was initially formed on top of the water layer, but with time a separation took place to clear oil and water layers.

Finally[6,7], the diffusion approach is analyzed within the two-phase region in the system water, ethanol and benzene, when water is added to a composition at the plait point. The predicted changes in the volumes of the two phases with added water by the phase diagram is compared to the experimental results and the transfer is described of the individual compounds between the two phases. High speed optical recording was made of the interface process showing the gradual development of the emulsions formed.

INVESTIGATION OF SPECTRAL PROPERTIES, THERMAL STABILITY, AND MORPHOLOGY IN PANI-MODIFIED BIOBR

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According to the hybrid approach that aims to the improvement in performance of new useful materials depending on their areas of application, a series of PANI-BiOBr composites were successfully synthesized by solid state route with different content of BiOBr which was elaborated via hydrothermal method. These hybrid structures were characterized by Fourier transform-infrared spectroscopy (FT-IR), X-ray diffraction (XRD), Scanning electron microscopy (SEM), transmission electron microscopy (TEM). The decomposition and degradation were visualized by thermogravimetric analysis (TGA). The PANI modified bismuth oxybromide is a promising material for environmental application.

Key words: Polyaniline; Bismuth oxybromide, Hydrothermal.

Waste Frying Oil with High Levels of Free Fatty Acids as one of the prominent sources of Biodiesel Production

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The decrease of fossil fuel resources and the awareness of the impact of greenhouse gases emissions on the environment have created a need to find alternative energy sources to replace traditional ones. Thus, recent years have seen the development of several sectors of renewable energy production with the government incentives. The biodiesel production is an alternative of non-toxic, clean, renewable and biodegradable fuels production [1-4]. The aim of this work was to develop a procedure for the biodiesel production from waste frying oil having an acid value of 32.82 mg KOH/g-oil. The optimum conditions of the biodiesel production from waste frying oil in two-step catalyzed process were investigated. In the first step, the sulfuric acid was used as a catalyst for the esterification reaction to reduce the free fatty acids content in the oil under 2%. The next base catalyzed transesterification process converted the pretreated frying oil into biodiesel and glycerol in the optimum and non-optimum FFA conditions. In the optimum FFA condition the highest biodiesel yield was 98% with 0.214% of free fatty acid.

Key words: Biodiesel, tow-step catalyzed process, free fatty acid, waste frying oil, optimization

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NEW BIO-SOURCED SURFACTANT: SYNTHESIS AND EMULSION PROPERTIES

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Nowadays, the increasing environmental concern and the demand for petroleum alternative stimulates the search for eco-friendly and renewable starting materials. Thus, numerous synthons are actually provided for the design of new sustainable compounds. Among them, isosorbide (1,4:3,6-dianhydro-D-glucitol), obtained by the double dehydration of sorbitol, is now available at the scale of several thousand tons a year. Recently, several researchers are working on its evaluation for use as a hydrophilic synthon to design various derivatives, ranging from solvents to surfactants or as a sustainable alternatives to bisphenol A [1,2]. Inspired from the literature [3], a facile and effective method for the preparation of a new cationic surfactant derived from isosorbide is introduced. The 8-dodecylamino diglycidyl ether of isosorbide (8-DODGEDAS) structure was characterized by means of size exclusion chromatography (SEC) and electro spray mass spectrometry (ESI-MS). Thermal behavior was studied using thermogravimetric analysis (TG) and showed excellent properties of 8-DODGEDAS. A very low critical micelle concentration (CMC) was determined by mean of conductivity. A formulation of (O/W) emulsion using 8-DODGEDAS was investigated. Then the stability of the optimized formula was studied using small-angle light scattering and zeta potential. Thereby, synthesized product could be used as a surfactant for fabric softeners or hair conditioners. They can also be used in the elaboration of bio-based materials with high performance.

Key words: Surfactant; isosorbide; emulsion; textile softener.

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Ions transfer modeling through monopolar and bipolar membranes: Treatment of wastewater containing ammonium nitrate by electrodialysis

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In this study, a mathematical model was proposed to illustrate the different transport modes contributing in transfer of all involved species through anion, cation and bipolar membranes. This study was led on a wastewater containing ammonium nitrate treated by bipolar membrane electrodialysis (BPMED). In this electrodialysis, different species are involved, in particular, ammonium ions NH_4^+, nitrates NO_3^-, ammoniac NH_3, water H_2O and protons H^+. Calculations led from the material balance sheets equations obtained in bath mode, allowed to validate the proposed transfer model and to check balance sheets material on all involved species during electrodialysis.

Key words: Bipolar membrane electrodialysis, material balance sheets, ions and water transfer

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STRUCTURE VARIATION OF POLYELECTROLYTE WITH HYDROPHOBICITY: CONFIRMATION OF THE PEARL-NECKLACE MODEL

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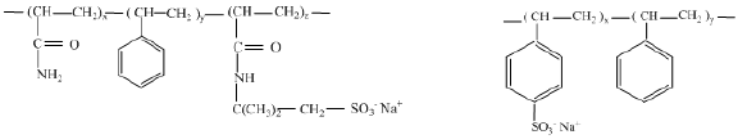
Polyelectrolytes combine ionizable groups, which dissociate in water into electrostatically charged parts and counterions. This combination opens a huge range of opportunities for properties, exploited in industrial. In fact, the structure of the PE is described as a pearl-necklace model [1]. It is considered that a single chain is formed by compact beads, the pearls, joined by narrow elongated strings. The aim of this study is to investigate the conformation of PE chain in water as a function of hydrophobicity. The used PE are poly (styrene-co-styrene sodium sulfonate) noted PSSNa and poly (acrylamide - styrene - acrylamido methyl propane sodium sulfonate) noted $P(AM_x\text{-}ST_y\text{-}AMPS_z)$, (see figure 1). For the PSSNa, the electrostatic charge and hydrophobicity are correlated, however for $P(AM_x\text{-}ST_y\text{-}AMPS_z)$ the electrostatic charge and hydrophobicity are decorrelated.



(a) (b)

Figure 1: The chemical structure of $P(AM_x\text{-}ST_y\text{-}AMPS_z)$(a) and PSSNA (b)

The Small Angle Neutron Scattering (SANS) data were fitted according to the theoretical model[2] and it was shown that the conformation of the hydrophobic PE is in agreement with the pearl necklace mode: the chain length of the PE decreases while the pearl radius forming the chain increases, with hydropobicity.

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Keywords: polyectrolytes, Small Angle Neutron Scattering

GREEN AND ECO-FRIENDLY PROCESS FOR EXTRACTION OF ESSENTIAL OIL FROM DRY ANISUM SEEDS: MICROWAVE HYDRODIFIFFUSION AND GRAVITY.

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With the development of the green chemistry, and starting from the idea on how to eliminate the pollution and hazards at the source, there has been an increasing demand of researchers to develop an effective and a cleaner process to extract the natural products from the dried plant materials, especially the essential oils, with shorten extraction time, simplifying manipulation, reducing reagent consumption, lower cost, energy saving, and preventing pollution.

Green microwave extraction technique leads to high-quality for the extraction of the essential oil from the aromatic plant because the microwave energy is a superior alternative to other thermal extraction techniques.

Microwave hydrodiffusion and gravity (MHG) is an original combination of microwave irradiations and earth gravity performed at atmospheric pressure without adding any solvent or water.

MHG has been compared with conventional method hydrodistillation (HD) in terms of extraction time, yield, energy, cost and aromatic composition of the essential oil obtained from dried anise seeds.

Essential oils isolated by MHG were quantitatively (yield) and qualitatively (aromatic profile) similar to those obtained by HD, but MHG was better than HD in terms of rapidity (15 min against 3h for HD), lower operation cost, reduced energy consumption (0.15 kWh/g of essential oil versus 2.5 kWh/g of essential oil) and cleanliness.

MHG is a green, fast, economical and environmentally friendly approach for the extraction of essential oils from dry spices.

Key words: Green process, Microwave hydrodiffusion and gravity, Hydrodistillation, Essential oil, Anisum seed.

NEW DISTYRYLANTHRACENE-BASED POLYMERS: SYNTHESIS AND CYANO-GROUP EFFECT ON THE OPTICAL PROPERTIES

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Anthracene was one of the first aromatic materials used in organic light emitting diodes (OLEDs). The first experiments were carried out by Pope in the early 1960s. [1] However, the anthracene tends to recrystallize with time, which led to a degradation of device performance.
In this communication we report the synthesis and characterization of soluble biphenylvinylanthracene based semi-conducting polymer (Figure 1).

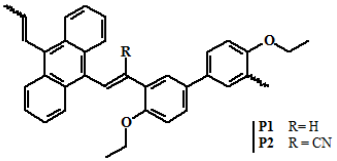

Figure 1: molecular structures of the polymers P1 and P2

The polymer **P1** has been synthesized via the Wittig reaction and **P2** by Knoevenagel polycondensations. Their molecular structures were confirmed by ^{1}H NMR and FT-IR spectroscopies. The polymers are soluble in common organic solvents and have number-average molecular weights of 11397 and 15273 g.mol^{-1} for **P1** and **P2**, respectively. The thermal properties were investigated by DSC analysis. The **P1** and **P2** thermogramm exhibit a good thermal stability and an amorphous morphology for both materials.
The optical properties of the polymers were investigated by UV-visible absorption and photoluminescence spectroscopies. The optical gaps for **P1-2** were estimated from the absorption-onsets of the polymer films and showed the same values at 2.69 eV. It make possible to classify these polymers among the semi-conducting materials. The HOMO and LUMO levels were estimated using cyclic voltammetry analysis; the values show a significantly effect of the cyano groups on the electronic structure of these semi-conducting polymers. Single-layer devices of the configuration [indium-tin oxide/polymer/aluminium] have been fabricated and have a relatively low turn-on voltage.

Key words: semi-conducting polymers, anthracene, Wittig reaction, Knoevenagel reaction, photoluminescence.

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The role of tetraethylphosphonic acid on corrosion inhibition performance of Zn^{2+} in carbon steel

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Phosphonates are molecules that contain one or more groups $R\text{-}PO(OH)_2$. Their good chemical stability and solubility in water make possible their use in the inhibition of corrosion and scale formation. Among all these properties, their ability to form strong complexes with metal plays a very important role in the rate of transport of these compounds in the environment [1, 2]. Phosphonates are a group of chelating agents that are used in cooling water and desalination systems to inhibit scale formation and corrosion. Different phosphonic acids are used in cooling water technology over decades and the corrosion inhibition is decided by the ability to form a protective film on steel. Studies have been carried out with different phosphonic acids in different corrosive environment. The results showed the addition of these molecules decreased the corrosion current density and the corrosion rate. The presence of Ca^{2+} and Zn^{2+} ions enhances the inhibition efficiency by synergism.

In the present study Corrosion inhibitory effects of new synthesized compound namely tetraethylphosphonic acid (TEPA) in the presence of Zn^{2+} ions on carbon steel in 3%NaCl was investigated at different temperatures using open circuit potential (OCP), potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS). Results showed that TEPA-Zn^{2+} inhibited carbon steel corrosion in acid solution and indicated that the inhibition efficiencies increased with the concentration of inhibitor, but decreased proportionally with temperature. Changes in impedance parameters suggested the adsorption of TEPA-Zn^{2+} on the carbon steel surface, leading to the formation of protective films. EDX spectroscopic analysis (XPS) of the protective film exhibited the presence of the elements viz., iron, phosphorus, oxygen, carbon and zinc.

The chemical shifts in the binding energies of these elements inferred that the surface film is composed of oxides/hydroxides of iron(III), $Zn(OH)_2$ and [Fe(II)/(III)–Zn(II)–TEPA] complex. Further, the surface analysis techniques viz., DRX and SEM studies confirm the formation of an adsorbed protective film on the carbon steel surface. Based on all these results, a plausible mechanism of corrosion inhibition is proposed.

Key words: carbon steel; phosphonic acid; OCP; EIS; corrosion inhibition

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Program of Tuesday, March 17th 2015

Fabrication and Characterization of Nano Silicon Carbide from Some Waste Materials (Rice Husk) Coming from Egyptian Fields

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This work has been carried out to investigate, the preparation of nano SiC from waste material (rice husk). The results revealed that by firing the ash at 500^{o}C, it maintained its amorphousity; while the firing at 800^{o}C led to staring crystallization and decomposition of the amorphous rice husk ash into silica and carbon. By increasing the firing temperature up to 1550^{o}C, well crystalline silicon carbide was formed. The particle sizes of the obtained SiC powders were in the range of 30nm. Three rice-husk ash samples having different features were used. The first was fine (hand-ground) rice-husk ash, the second was coarse (as is) rice-husk ash while the third was ball milled one. Effect of ball milling of the starting ashes for 6 hours on the formation of nano SiC was investigated and compared with those prepared without milling. Phase composition and particle sizes as well as the morphology of the formed SiC powders were characterized by X-ray diffraction (XRD) and transmission electron microscope (TEM). The particle sizes of the prepared nano SiC materials were affected by the milling process. The particle sizes of the obtained nano SiC from ball milled staring materials were smaller than those prepared without milling. The pyrolysis conditions i.e. the temperature and atmosphere was optimized. The effect of pyrolysis atmosphere i.e. argon, vacuum and nitrogen was also demonstrated. The pyrolysis in nitrogen atmosphere led to formation of SiC/Si_3N_4 nano-composite. The temperature that required for sintering nano powder SiC had been optimized then after, the microstructure of the produced sintered SiC compared of three different features sources.

Key words: Nano SiC; Rice husk ash; Pyrolysis; SiC/Si_3N_4 nano composite, Environmental Applications

SYNTHESIS OF ZINC OXIDE NANOPARTICLES AND THEIR APPLICATION IN PENICILLINS PHOTODEGRAFATION

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Zinc oxide nanoparticles were synthesized using a simple precipitation method with zinc sulfate and sodium hydroxide as starting materials; the product was calcinated at different temperatures for 2 h and then tested for the photodegradation of penicillins.
The average crystallite sizes of the samples were calculated from the full width at half maximum of XRD peaks by using Debye-Scherrer's formula and were found to be in the nanorange.
Time resolved UV-Vis spectroscopy has been used to follow the growth of particles in situ in solution. An empirical relation between the optical band gap given from absorption measurements, and particle size given from XRD measurements has been developed.

Key words: Semiconductors, Chemical synthesis, Nanoparticles, Photodegradation, penicillins

Factors affecting interfacial interaction between polyethylene matrix and clay layers in polyethylene/clay nanocomposites

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Project funded by Scientific Research Support Fund (SRF)- Jordan

It was aimed in this study to check factors affecting interfacial interaction between polymer matrix and clay layers. This was achieved by preparing Polyethylene/clay nanocomposites as blown films using different formulas (clay contents (4 and 6 wt %) and compatibilizer/clay ratio (1/2, 1.0, 2.0)). Structure, thermal and mechanical behavior were tested. It was found that blown film extrusion process decreased the tactoids size and consequently enhanced the exfoliation degree of the clay layers inside the polymer matrix, which is due to the elongational stress during extrusion. Addition of clay had some effect on crystallization behavior. It caused change in crystalline thickness, in addition to some delay in melting and crystallization process due to confinement of polymer chains by clay layers. Regarding their effects on mechanical behavior, there was an increase of yield strength (around 30%). Yield strength is related to the interfacial interaction between the polymer and the clay layers in the nanocomposites, which would be enhanced by enhanceing the compatibility between polymer and clay layers. At 90% confidence interval, the yield strength was positively correlated with compatibilizer/clay ratio, aspect ratio and exfolition number and negatively correlated with interparticle distance between clay tactoids. Modeling of yield strength showed that the interfacial stress transfer parameter (τ) is 32 at clay loading of 4wt%, while it was found to be 15 at clay loading of 6wt%. This means that the examined nanocomposite systems have some strong interactions specially at low clay content (4wt%).

Key words: Nanocomposites, compatibilizer/clay ratio, interfacial interactions.

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GENERATION OF BASIC SITES IN Y ZEOLITE AND APPLICATION TO KNOEVENAGEL CONDENSATION REACTION

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In order to extend the range of zeolite applications, modifications of the surface properties that control interactions with various guest species have been performed. Tuning these properties by replacing small parts of the zeolite structure by selected groups could create new catalytically active sites, allowing these materials to promote a broad range of reactions with potentially improved selectivity. In this context it is well established that the substitution of nitrogen for oxygen in the zeolite framework leads to an increase of the Lewis basicity, as a result of the lower electronegativity of nitrogen with respect to oxygen. The present study describes structural, textural and catalytic properties alterations upon nitrogen incorporation into Y zeolite framework. The incorporation of nitrogen was performed by thermal treatment of the parent zeolite under an ammonia flow at temperature between 500 and 900°C for a prolonged time: hereafter referred to as ‘‘nitridation’’.

Nitrided samples were analyzed by chemical analysis for the determination of the amount of nitrogen incorporated, X-ray photoelectron spectroscopy (XPS) to identify the chemical nature of incorporated nitrogenous species, nitrogen adsorption/desorption isotherms and X-ray diffraction (XRD) to follow the evolution of textural and structural properties upon the nitridation treatment. Finally, the Knoevenagel condensation reaction between benzaldehyde and malononitrile was performed to evaluate the impact of the nitridation on the basicity. The Knoevenagel condensation is one of the reactions used for carbon-carbon bond formation and is often used for the commercial preparation of fine chemistry intermediates. This reaction, which is a key step in the commercial production of antimalarial drug Lumefantrine, is also largely employed as probe reaction for the characterization of catalyst basicity.

According to XPS and chemical results an increase of the nitridation temperature induces an increase of the nitrogen content and the appearance of nitrogenous species in the following order of increasing temperature: NH_4^+, adsorbed NH_3, $-NH_2$,-NH- and >N- [1]. From the catalytic results it can be concluded that nitridation has a beneficial effect on the basic catalytic activity as long as the zeolitic framework was maintained [2].

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EFFECT OF SURFACTANT TAIL STRUCTURE ON PHASE BEHAVIOR OF BRANCHED AND LINEAR ALKYLBENZENE SULFONATE IN WATER AND OIL TERNARY SYSTEMS

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Systematic investigations into the phase behavior of a ternary system of linear alkyl benzene sulfonate (LAS) or branched alkyl benzene sulfonate (BAS) in oil (corn or olive oil) and water were carried out. The study showed that there is a relationship between the surfactants chemical structure, aggregation properties, and the formation of micelles. The aggregation properties were dramatically affected by small structural variations of the hydrophobic group at constant carbon number. Micelles formation was found to be correlated with the extent of chain branching by affecting oil-water interfacial tension, where the branched alkyl tail has significantly more ability to form micelles than the linear one. It was found possible to distinguish between LAS and BAS based on their phase behavior.

Key words: Linear alkyl benzene sulfonate; Branched alkyl benzene sulfonate; Phase diagram; Micelles; Aggregation

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COUNTERCURRENT PACKED COLUMN FOR SUPERCRITICAL CO_2 SEPARATION OF WATER-ISOPROPANOL MIXTURES

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Current research in chemical engineering is mainly focussed on development of cleaner and less energy-intensive processes, with a low environmental impact. Research in bio-fuels led to propose production of alcohol by fermentation, which yields dilute solutions of these alcohols. However, use of conventional processes for the downstream purification steps diminishes the interest of such bioprocesses. Indeed, conventional purification operations present significant energy costs. An alternative, using supercritical CO_2 as the separating agent, can be proposed and has been studied for long by the team of G. Brunner [1]. Nevertheless, this alternative process is relatively new to the industrial world. It is an unfortunate situation because of the high potential of this technology which, due to the very high selectivity of the process, can lead in some cases to very competitive costs.

The aim of this study was to evaluate the separation performances of a counter-current packed column for the purification of water-isopropanol mixtures using supercriticalCO_2. Experiments were performed on a 2m height and 17 mm diameter packed column. Variation of pressure, solvent to feed ratio and composition of the feed were investigated. For instance, for a diluted aqueous feed mixture (5%w/w of IPA), at 10 MPa and 40°C, increase of the solvent ratio (S/F) from 4.5 to 11 has shown beneficial effect on the water purification. For a S/F ratio of 11, a 99. 8% water purity at the column bottom was achieved with a theoretical 98.1% IPA recovery at the column heading. Experimental results were compared to simulations done using the commercial software ASPEN PLUS where modeling the column is based on the concept of mass transfer coefficient. This study has provided understanding and quantitative results allowing proposing the design of a process for producing high purity isopropanol from a dilute aqueous solution with supercritical CO_2 using counter-current packed columns.

Key words: Supercritical CO_2, isopropanol, Countercurrent column, Process simulation.

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THE IMPACT OF THE SUBSTITUTION OF THE PORTLAND CEMENT BY THE '*OPUNTIA FICUS INDICA* 'AND KERNELS OF DATES ON THE PROPERTIES OF MORTAR

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Potential use of vegetable biomass from the '*Opuntia Ficus Indica*' (OFI) and kernels of dates as partial cement replacement material in production of mortar was investigated.

A total of twelve mortar mixtures varying level of cement replacement using OFI and kernels of dates in addition to a normal mortar (containing only neat cement as binder) were produced according to a plan of experience of factorial matrix 2^2.

Mechanical strength, durability by acid attack and the setting time of the cement paste of mortar mixtures produced by partial replacement of cement with 1, 2.5 and 4% of OFI and kernels of dates by weight were investigated.

Test results indicated improvement, in the compressive strength properties and the durability of mortar samples by the inclusion of the 'OFI' and the kernels of dates as a partial replacement of cement at a replacement level of 1% and with particle sizes less than 212 µm. The setting test showed retarding effect on the hydration of cement for the kernels of dates admixtures and an accelerator's one for the OFI admixtures.

These results put forward the influence of these admixtures, on the properties of cementing materials, thus showing the economic and environmental interest of the partial substitution of the Portland cement by the OFI and the kernels of dates.

Keywords: mortar, admixtures, compressive strength, durability, setting time.

Modified Co exchanged zeolite catalysts for ethane ammoxidation into acetonitrile

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Ammoxidation of ethane in particular to acetonitrile (AN) is of special significance due to its wide use as a solvent in the purification of butadiene in refineries and for the manufacture of pharmaceuticals, agrochemicals and photographic films. Several catalyst systems have been tested in ethane ammoxidation, e. g. cobalt exchanged zeolites [1–3] and the Co/ZSM-5 catalysts exhibited the high catalytic performances and AN activity of 4.2 μmol.g^{-1}.s^{-1} at 425°C [3]. An effective way for improving the catalytic performance of Co/BEA catalyst at low temperature is to add small amounts of transition metals as promoters. *M*–Co/BEA catalysts (*M*=Cu, Cr, Mn, Zn, Fe and La) were prepared by solid-state ion exchange and tested in ethane ammoxidation (10% C_2H_6, 10%O_2, 10%NH_3 and 70% He). Fe–Co/BEA and La–Co/BEA solids exhibited AN activities of 4.2 and 5 μmol.g^{-1}.s^{-1} at 400°C, respectively. Thermal analyses coupled with mass spectrometry revealed that melting $CuCl_2$, $CrCl_3$ and $MnCl_2$ salts inhibited the exchange of $CoCl_2$ with NH_4^+–BEA, while $ZnCl_2$ does not contribute in the solid-state exchange. However, H_2–TPR analysis revealed that La_2O_3 specie reduced the excessive formation of Co_3O_4 agglomerates, inadequate for ammoxidation, while iron promoter increases the amount of $Co(OH)^+$, which seems to be the active species, and store the catalytic activity as the reaction temperature increases.

Keywords: BEA zeolite, Ethane, Ammoxidation, H_2–TPR, Mass spectrometry.

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Anodic oxidation of tetracycline on BDD electrodes: Kinetic study and effect of the nature of the supporting electrolyte

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Pharmaceutical wastewaters are characterized by high oxygen demand and non-biodegradability. Their release threatens on the environment; increases the antibiotic-resistant pathogens and can cause serious alterations in aquatic ecosystems [1]. Therefore, suitable wastewater treatment of effluents containing antibiotic, known as emergent compounds, becomes a major issue. In this present work, Anodic Oxidation (AO) using monopolar and bipolar Boron Doped Diamond (Si/BDD) electrodes has been performed on synthetic solutions containing tetracycline as pharmaceutical pollutant model which is widely used in human and animal therapy for the prevention and control of infectious disease. The degradation of tetracycline is investigated with three different supporting electrolyte (sodium chloride, sodium nitrate and sodium sulfate) at fixed conditions (j=40mA/cm², Ctetracycline=0.1g/L). At these experimental conditions tested, an appreciable degradation of tetracycline was observed (more than 90% abatement of COD) after 3 h essay using bipolar cell whatever the supporting electrolyte used. Best degradation rate is obtained in the case of chloride medium due to the oxidizing powerful of electro-generated hypochlorite. However, a less significant mineralization is obtained (60% of COD removal) using monopolar BDD electrodes. The effect of nature of the supporting electrolyte on the kinetic of degradation was studied in this work. The current efficiency and the energy consumption were also evaluated.

Key words: wastewater, tetracycline, BDD, oxidation, electrolyte support, kinetics

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INFLUENCE OF MINERALISERS ON THE CLINKER BURNING TEMPERATURE AND ITS MORPHOLOGY

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In a laboratory process of cement fabrication, the influence of mineralizes addition to three industrial raw materials on the clinkering process as well as on the morphology of the produced clinker is examined.
The industrial raw material used in this study comes from the Cement Society of Gabes. These raw that have been prepared in accordance with the production procedures followed by the cement's plant of Gabes, were named raw 1, raw 2 and raw 3. These later were variable at fineness. The mineraliser added to these mixtures, with varying percentages (0%, 1% and 2%) is fluorspar. It is added to the raw meal to prepare 5 g of a homogeneous mixture. Clinkers were synthesized in a kiln type Nabertherm. The burning temperatures varied between 1300 and 1450°C. The basis of assessment of raw materials was the estimation of free lime and C3S content. Chemical, physical and mineralogical characterizations were made by using XRF, X-ray diffraction (XRD), thermogravimetric and differential thermal analysis (TG- DTA) and optical microscopy techniques.
The experimental results can show that the addition of small amounts of fluorspar (2%) to cement raw material accelerates the formation of C3S phase and intensifies the process of the free lime combining during the clinkerisation.

Key words: fluorspar, mineraliser, clinkers, X-ray diffraction, optical microscopy.

Synthesis, structural and electrical properties of $[C_7H_{10}NO]_2\ SnCl_6.\ 2H_2O$

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Synthesis, structural and electrical properties are given for a new organic stannous halide material. The structure of the $[C_7H_{10}NO]_2\ SnCl_6.\ 2H_2O$ reveals that the $[SnCl_6]^{2-}$ dianion, located on an inversion center, exhibits a highly distorted octahedral coordination environment. These chains are themselves interconnected by means of O—H…Cl, N—H…Cl and N—H…O hydrogen bonds consolidate the packing, which can be described as consisting of alternating anionic (containing dianions and lattice water molecules) and cationic layers parallel to ab plane. [1,2]. The AC electrical conductivity and the dielectric relaxation properties of the $[C_7H_{10}NO]_2\ SnCl_6.\ 2H_2O$ compound have been investigated by means of impedance spectroscopy measurements over a wide range of frequencies and temperatures, 20 kHz–2MHz and 275–330K, respectively. Besides, a detailed analysis of the impedance spectrum suggested that the electrical properties of the material are strongly temperature dependent. Nyquist plots (Z' versus Z") are well fitted to an equivalent circuit model which consists of a parallel combination of a bulk resistance Rb and constant phase elements CPE. The frequency-dependent conductivity data were fitted in the modified power law [3]: $\sigma(\omega) = \sigma_{dc} + A\omega^n$
The nature of variation of DC conductivity suggests Arrhenius type of electrical conductivity. Furthermore, the modulus plots can be characterized by full width at half height or in terms of a non-experiential decay function $\Phi(t) = \exp(-t/\tau)^{\beta}$. Besides, the activation energy responsible for relaxation has been evaluated and found to be close to the DC conductivity.

Key words: Synthesis, halide, AC electrical conductivity, DC conductivity

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S235JR TANK STEEL CORROSION BEHAVIOR IN CHLORIDE MEDIA_TEMPERATURE AND SCAN RATE EFFECTS

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Carbon steel is used as a building material for the hydrocarbon storage tanks due to its excellent mechanical properties and its low cost. These tanks are subjected to severe corrosion which represents a major hazard concern for the petroleum refinery industry. To find reliable methods for materials preservation, several studies have been concerned on understanding the corrosion mechanism of carbon steel in neutral media containing chloride ions [1,2].

The present work is devoted to compare the corrosion behavior of S235JR non alloyed tank steel in aerated NaCl 0,5M solution to the behavior of carbon steel used in concrete.

Cyclic voltammetry was used to evaluate the pitting corrosion of the two studied steels at 25°C in NaCl 0.5M solution. The results showed that S235JR steel was less subjected to pitting corrosion. The comparison of the anodic and cathodic polarization curves obtained for the two steels revealed that S235JR is less corroded in chloride media. The temperature effect on the interfacial behavior of the steels was evaluated. For both steels, with increasing temperature from 298K to 318K, Epit (pitting potential) shifted to more negative potentials denoting a decrease in the pitting corrosion resistance. Epit was linearly correlated with the temperature but the slope values differ for the two steels. Besides, Ecorr, βa, βc and Jcorr were determined for each temperature using Tafel domain. Ecorr were found to decrease as temperature increased. Thermodynamic parameters of activation (Energy activation Ea, enthalpy of activation ΔH^* and the entropy of activation ΔS^*) were calculated and discussed for the two steels. The effect of potential scan rate ν on the polarization curves of the studied steels in NaCl 0,5M at 25°C was investigated. It was noticed that Epit and Erep (repassivation potential) was influenced by increasing the scan rate from 1 to 50 mVs^{-1} and both shifted to more positive values. Some linear relationships between ν and Epit, Erepa were found. The effect of immersion time (5 min to 1 hour) was also studied for the two steels. The alteration of S235JR steel sheets sampled from a storage tank of a Refining Industry was characterized by FTIR and XRD.

Key words: steel, pitting, temperature, scan rate, polarization measurements, FTIR, XRD.

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Program of Wednesday, March 18th 2015

Synthetic methodology effect on the microstructure and thermal properties of poly(MMA-stat-sty)-b-PSSNa synthesized by nitroxide mediated polymerization

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In this work, we report the synthesis of poly (methyl methacrylate-statistic-sulfonated styrene)-b-sulfonatedpolystyrene **poly(MMA-stat-sty)-b-PSSNa**copolymers by the nitroxide mediated polymerization (NMP) technique, and we describe the ''in situ'' synthesis of ''all-acrylic'' copolymer **poly(MMA-stat-sty)-b-PSSNa/**clay materials at different low contents of raw and modified Montmorillonite (5–15 wt % versus monomer),using N-tert-butyl-N-(1-diethylphosphono-2,2-dimethylpropyl)nitroxide (SG1) as a control agent and 2-methylaminoxypropionic-SG1 alkoxyamine (BlocBuilder) as the initiator.

The copolymers are synthesized either by batch or semibatch processes via the determination of the instantaneous fraction of monomer incorporated in the copolymer. A control of the molar mass together with low molar mass distribution (Mw/Mn< 1.4) is observed.PMMA-b-PS was sulfonated with acetyl sulfate, and the sulfonation was confirmed by FTIR, ^{1}H NMR,and TGA. Then, the sulfonated PMMA-b-PS was casted as membranes. The dependence of the copolymer glass transition temperature with conversion was followed by differential scanning calorimetry.

The copolymers are investigated by carbon nuclear magnetic resonance NMR sequences to study the effect of the monomer addition mode on the microstructure of copolymers. The morphology of microphase separation of block copolymer thin films was investigated by AFM, and long-range order lamellar morphology was observed after vapor-annealing.

The copolymerization of methyl methacrylate with a low molar proportion of styrene could be performed at temperatures below 80 °C, NMP was also chosen to elaborate in situ poly (MMA-grad-SSNa)-b-PSSNa/clay materials by performing polymerization, the sulfonated PMMA-b-PS was casted as membranes(proton exchange membranes).

KEYWORDS: clay nanocomposite; In situ polymerizationl; nitroxide mediated polymerization; Block copolymer; thermal stability; Sulfonation of PMMA-b-PS

DIELECTRIC PROPERTIES OF RARE EARTH OXIDES TYPE PEROVSKITE

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The electrical properties of inorganic materials has attracted great interest of researches since decades. Thanks to the successful control of the electrical property of an inorganic material, they offer a wide range of energy-related applications covering energy storage, energy conversion and energy utilization.

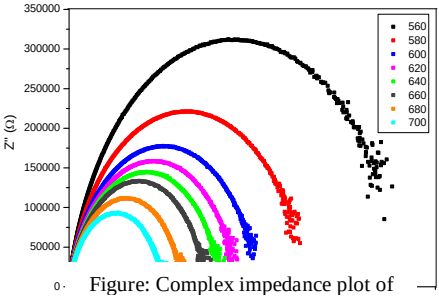

Figure: Complex impedance plot of LaErO3 at different temperatures

Today, rare earth oxides are gaining new interest as a promising alternative dielectric because of their large band gap (4 eV-6 eV) and their good thermal stability [1,2]. According to some previous works, only theoretical investigation of dielectrical properties of some rare earth oxides type perovskite like $LaErO_3$ and $LaHoO_3$ were reported [3,4]. In this work we suggest to study the electrical and dielectical properties of $LaErO_3$ and $LaHoO_3$ which are studied in the range of 560 °C and 700 °C and a frequency of $0.6x10^{-3}$ MHz to 8 MHz.

Keywords: perovskite, rare earth, dielectric.

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QUANTIFICATION OF 26 PHARMACEUTICAL RESIDUES IN WASTEWATER AND RIVER WATERS USING ON-LINE SPE-LC-MS/MS

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Drug residues can be easily introduced into the aquatic environment directly through the sewage discharges or indirectly from agricultural activities (intensive livestock effluents, farms). Given their ubiquity and the fact that these products are designed to be biologically active at low doses, chronic exposure of aquatic organisms and men therefore raises many questions. Their presence in the environment, particularly in the aquatic environment has become a growing concern worldwide. Today, the scientific community is very interested in this issue. The relationship between the wastewater treatment processes, fate and impact of pharmaceutical residues is not yet well known. To date, these residues are the subject of several scientific research programs. This underlines the urgent need to improve the knowledge on the behavior, fate, impacts of these residues.

The objective of this work is to determine the contamination levels of 26 drug residues (human and veterinary products) obtained from four sampling sites in France and in Tunisia using on-line SPE-LC-MS/MS. Among the 26 drug residues includes three classes of human and animal antibiotics the most currently used: sulfonamides (SAs), fluoroquinolones (FQs) and tetracycline (TCs). The concentration of the 26 drug residues were determined in rivers water, input and output of aerated lagoon, and input and output of Wastewater Treatment Plant (WWTP) collected from four sites in Northern France and Tunisia. The results show that drug residues were ubiquitous in aquatic environment. Their concentrations were detected all the samples obtained from rivers water, effluent and influent of WWTP and lagoon. Individual concentration can be up to more than 100 ng L^{-1}.

Keywords: Antibiotic, drug residue, wastewater, river, lagoon, on-line SPE-LC-MS/MS

Optimization of methylene blue adsorption onto chemically modified orange sawdust using the response surface methodology approach

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The presence of dyes in water bodies generally cause serious public concern regarding human and aquatic organisms' health, fauna and flora growth due to their toxic properties. This last decade, high attention has been paid to the study of the adsorption onto raw and modified organic and mineral materials as an effective and low cost method for dyes removal. In this study, methylene blue (MB) adsorption onto chemically modified orange sawdust was investigated in batch system. The effects of operating parameters namely, NaOH treatment concentration, adsorbent dose, contact time and initial MB concentration were followed. The process optimization was performed through the response surface methodology approach (RSM) by using Minitab16 software. ANOVA test was performed in order to determine the quadratic polynomial coefficients and allowed the classification of the parameters importance as follows: NaOH treatment concentration > Contact time > Initial MB concentration > adsorbent dose. Student's and Fischer tests showed that mostly linear and quadratic parameters do affect significantly the MB removal yield. The optimal experimental conditions ensuring maximal MB removal efficiency were found as follows: NaOH treatment concentration of 0.14 M, adsorbent dose of 50 g/L, 1 hour as optimal contact time and an initial dye concentration of 69.5 mg/L. Kinetic and isotherm studies were carried out for the modified sawdust under the optimal conditions determined above. The pseudo-second-order model was found to explain the kinetics of MB adsorption most effectively. The Langmuir and Freundlich isotherm models were applied to the equilibrium conditions, showing that Langmuir model fits best the experimental data, and suggesting that a monolayer adsorption process is mostly probable. Further investigations are currently undertaken in order to study the optimization of the MB desorption process.

Key words: Adsorption, Methylene blue, modified sawdust, Response surface methodology.

POLARIZATION PHENOMENA IN DCMD PROCESS FOR HYPERSALINE SOLUTION TREATMENT

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In the present work, NaCl concentrated solution treatment was studied by direct contact membrane distillation (DCMD) using polyvinylidene fluoride (PVDF) flat-sheet membrane. The effect of relevant operating parameters including feed ionic strength, temperatures and flow rates, was investigated. Among them, the most important factors, that influence greatly the permeate flux, were the feed temperature and the inlet ionic strength. Jp decreased, linearly, from 8.4 to 3.2 kg m-2 h-1 as the feed ionic strength was increased from 0.5 to 5 M, and rose, exponentially, from 1.5 to 17.5 kg m^{-2} h^{-1}, due to the feed temperature increase from 35 to 75°C. A complete analysis of polarization phenomena in DCMD was carried out. All Experiments run reveal that permeate fluxes were dependent upon the temperature and concentration polarization coefficient (TPC, CPC). The noticeable modification on the permeate flux due to the operation conditions changes was accompanied by variations in TPC and CPC trends. DCMD performances might be significantly improved by an optimal choice of the experimental conditions. In our case, the hot and cold streams temperatures were, respectively, set at 59 and 20°C, and the hot and cold flow rates were, both, fixed to 20 L h^{-1}.

Key words: NaCl concentrated; DCMD; PVDF; Operating conditions; TPC; CPC

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FUNCTIONALIZATION OF [60]FULLERENE BY [3+2]CYCLOADDITIONS: SYNTHESIS AND CHARACTERIZATION OF THE ADDITION OF THE TRYPTOPHAN-METHYL ESTERS ON THE [60]FULLERENE

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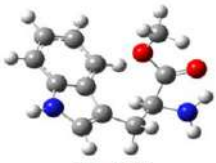
Fullerenes continues to be a current focus of research leading to potential applications of fullerene and its derivatives in biological and material science [1]. However, while the chemistry of fullerenes is nowadays considered as a well-established matter with a high level of understanding of their chemical reactivity [2] a wide variety of important reactions have never been carried out on the fullerene surface [3]. [60]Fullerene (C_{60}) reactivity with organic compounds has been so far dominated mainly with investigation of mono-addition reactions. The synthesis of organic poly-adducts of C_{60} has been reported in only a few cases, mainly by successive cyclopropanation or by 2,3-dipolar cyclo-addition of azomethine ylides (AMY) to C_{60}.
On either its hydrophobic nature and its exceptional geometry, the fullerene C_{60} could be used as a multifunctional nanovector, both to facilitate membrane passage of hydrophilic active ingredients, than for selectively achieve a therapeutic target.
In this context, we synthesized the carbon-based nanoparticle for vectoring active ingredients, by grafting the tryptophan methyl esters (TrpME) on the C_{60} by a photo-addition reaction involving the oxygen to the condition singlet.

[60]Fullerene



TrpME

We have proposed a mechanism of formation of the monoadduct fulleropyrrolidine, and identified the reaction intermediates by high resolution mass spectrometry coupled to the liquid chromatography (MS, HPLC-ESI-MS and HPLC-APCI-MS).

Keywords: [60]Fullerene, Photo-addition, Tryptophan-methyl-esters, Fulleropyrrolidine, azomethine ylides (AMY).

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BEHAVIOR OF SODIUM POLYSTYRENESULFONATE/ POLYDIALLYLDIMETHYLAMMONIUM CHLORIDE COMPLEXES

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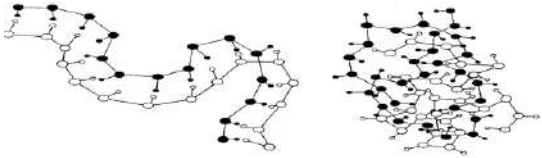
Polyelectrolytes complexes (PEC_s) result on strong Coulomb interaction between oppositely charged polyelectrolytes and lead to spontaneous aggregation. They are widely used in various fields such as water treatments, paper industry and gene therapy. They have been studied since years ago [1]; no clear picture has yet emerged. Precise knowledge and control of polyelectrolyte complex formation is however highly desirable. In its most simple form, complex formation is observed when the two oppositely charged polyelectrolytes are mixed in aqueous solution. PECs formation leads to quite different structures defined by two models: the ladder-like structure where complex formation takes place on a molecular level via conformational adaptation and the scrambled egg structure, where a high number of chains are incorporated into a particle, depending on the characteristics of the components used and the external conditions of the media. [2]



Ladder-like model Scrambled egg model

Our main objective is to gain knowledge about the behavior of PEC_S formed by two strong polyelectrolytes: sodium polystyrenesulfonate (PSS) and polydiallyldimethylammonium chloride (PDADMAC) in aqueous solutions, as a function of different physical chemical parameters. The viscosimetric and turbidity measurements showed an evolution of the macroscopic and microscopic structure of the formed complexes, with molar ratio, chain size, charge ratio and the solvent composition.

Key words: Polyelectrolyte complexes, aqueous solution, Viscosity, Turbidity.

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Effect of operating parameters on the photocatalytic activity of [Zn-Al] LDH

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[Zn-Al] LDH with a molar ratio R = n_{Zn}/n_{Al} = 3 (Zn-Al-3) was prepared by direct coprecipitation method. Parts of R = 3 were calcined at 500 °C and 900 °C and denoted c-Zn-Al-3-500 and c-Zn-Al-3-900 respectively. The obtained samples were characterized by different techniques such as XRD, FT-IR, UV-DRS, TEM and nitrogen physisorption at 196 °C.

XRD patterns of Zn-Al-3 showed the characteristic structure of LDH. Calcination at 500 °C leads to the destruction of the lamellar structure and only peaks of ZnO are present. At 900 °C, well crystallized oxides ZnO and $ZnAl_2O_4$ are obtained.

The three samples were tested for the photocatalytic degradation of methylene blue (MB). The effect of operating parameters (solution pH, photocatalyst dose, MB concentration, the presence of different anions...) has been studied.

It was found that the photocatalytic process depends on several factors. For example, working with an alkaline pH contributes to better degradation of the dye. However, a high concentration of the dye leads to a decrease in the activity of the three studied photocatalysts.

Kinetic studies showed that the degradation of MB follows a pseudo-first order.

Correlation between the electronic properties of the molecules of *Capparis Spinoza* extracts and their corrosion inhibition capabilities

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Corrosion process plays an important role in the field of economics and safety [1]. Many researchers report that the inhibition effect mainly depends on some physic-chemical and electronic properties of the organic inhibitor which relate to its functional groups, steric effects, electronic density of donor atoms, and orbital character of donating electrons, and so on [2,3]. However, some of these corrosion inhibitors are toxic to the environment [4,5] which has prompted the search for eco-friendly corrosion inhibitors for metals in acid solutions. Several plant extracts such as *Gossipium hirsutum* [6], *Cocos nucifera* [7], *lawsonia* [8] were tested for material preservation.

In this work we used Quantum chemical calculation in order to tentatively correlate the electronic properties of some molecules of *Capparis Spinoza* methanolic extract to the corrosion inhibition capabilities. The calculated parameters linked to the inhibition efficiency were the dipole moment (μ), the energy of the highest occupied molecular orbital (E_{HOMO}) and the energy of the lowest unoccupied molecular orbital (E_{LUMO}). Furthermore, the separation energy (ΔE) electronegativity (χ), hardness (η) and electrophilicity (w), softness (σ) were calculated. In addition, the electron fraction transferred molecules (ΔN) was determined in order to understand the interaction-extracted surface. The Fukui function and local softness indices were performed to illustrate the process of adsorption of some specific components of the extract.

Key words: plant extract, Corrosion Inhibitors, Adsorption, DFT Calculations; Fukui Function, Iron, Copper

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DEGRADATION OF ACID RED 18 BY ELECTROCHEMICAL PROCESS USING A GRAPHITE-FELT CATHODE

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Synthetic azo-dyes are widely used in textile, printing, cosmetic, food colorants and pharmaceutical industries. Their strong electron withdrawing character stabilizes their aromatic substances against conversion by oxygenizes. Indeed, azo-dyes and their products are toxic against aquatic organisms and suspicious of being carcinogenic and mutagenic for human [1]. Consequently, their discharge in environment as effluent causes pollution of surface water, soil and underground water.

The present study was undertaken to investigate the degradation removal of Acid Red 18 azo-dye by the environmentally friendly indirect electro-Fenton method. The electrochemical cell contains a carbon felt cathode and a platinum anode. The great oxidation ability of this process is due to the large production of hydroxyl radicals (OH**.**) by electrochemically induced Fenton reagents [2], which react on organics up to complete mineralization.

This work was devoted to optimizing the degradation parameters of Acid Red18 synthetic aqueous solution by electro-Fenton process. Accordingly, the influence of some experimental parameters such as initial concentration, current intensity, catalyst concentration, temperature and processing time on the degradation rate of Acid Red18 has been investigated. The evolution of its concentration with processing time shows a pseudo first order kinetics (kapp = 0.521 min-1).

Key words: Acid Red 18**,** advanced oxidation processes, electro-Fenton, hydroxyl radicals, degradation

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FLUORIDE ADSORPTION STUDIES OF MONTMORILLONITE TUNISIAN CLAY

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The removal of F^- ions with clays in aqueous solution has been studied. Batch adsorption experiments were carried out by considering various solution pH, interaction time, and temperature. The adsorption equilibrium follow Langmuir adsorption isotherm model. The maximum adsorption capacity values were 40,22mg/g and 73mg/g for natural and activated clay respectively.

Key words: Fluoride, adsorption, Montmorillonite, activated clay.

EQUILIBRIUM AND KINETICS STUDIES OF METHYLENE BLUE SORPTION FROM SYNTHETIC AQUEOUS SOLUTIONS BY THISTLE STALKS: EFFECT OF pH AND ATTEMPTS OF DESORPTION.

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Among the various types of dyes, various cationic dyes, including methylene blue, are used in dye, paint production and in wool dyeing [1]. Though methylene blue is not strongly hazardous, it can cause some harmful effects (eye burns, nausea, vomiting, etc [2]. Thus, the treatment of effluent containing such dye is of major scientific interest, decolourization of dye-containing effluent becoming an obligation both environmentally and for water re-use. In the perspective of application of sorption in the depollution of wastewaters contaminated by dyes, it is necessary to know the performances of the process whatever the pH of the medium, and, to regenerate and re-use the sorbent after it has become loaded with dye in order to make the sorption process economical.
In this context and as a continuation to our previous works [3], the effect of initial pH of dye solution on the methylene blue sorption kinetics by a natural plant material: thistle stalks was studied. This sorbent material is abundantly available through our country and the world. Dye sorption/desorption kinetics and equilibrium tests were carried out in batch conditions. The experimental data obtained from the sorption equilibrium and kinetics of methylene blue were fitted by different models available in the literature. Furthermore, attempts of desorption of methylene blue from dye-laden- thistle stalks were also performed by utilizing various desorption agents.

Key words: sorption, methylene blue, thistle stalks, initial pH effect, kinetics, equilibrium desorption

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NANOSTRUCTURED METAL-ORGANIC FARMEWORKS BASED ON Tl(TCNQ)

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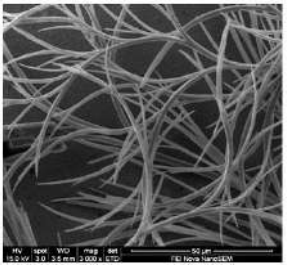
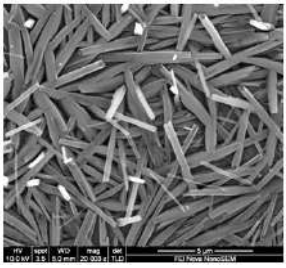
The synthesis and fabrication of nanostructured materials having fascinating optical, electrical, and magnetic properties is essential motive for developing new generation of electronic and memory devices. TCNQ-based molecular materials have been implemented in such applications, and in recent years considerable efforts have been directed towards the design and fabrication of tunable nanoscale materials. In this presentation, facile electrochemical approaches for the synthesis, characterization, and fabrication of morphology-tunable semiconducting/insulating Tl(TCNQ) nanostructures onto conducting (Pt, Au, GC) and semiconducting (ITO) electrode surfaces will be discussed. These approaches involve solid-solid phase transformations of TCNQ microcrystals, attached to an electrode surface, into the corresponding Tl(TCNQ) when immersed in an aqueous solution of $Tl^{+}_{(aq)}$ electrolytes, and subjected to one-electron reduction to form the $TCNQ^{-}$ radical. The overall TCNQ/Tl(TCNQ) conversion process requires the transfer of one-electron at the triple phase, electrode| $TCNQ/TCNQ^{-}|Tl^{+}_{(aq)}$ junction, with a nucleation-growth rate-determining step. Characterization of the electrochemically generated Tl(TCNQ) based materials via wide range of spectroscopic (IR, Raman), microscopic (optical, SEM, EDAX), as well as conventional and synchrotron-based XRD techniques will be highlighted. Compare to other synthetic methods, this electrochemical approach provides an easy access for tuning the morphology and crystals size of Tl(TCNQ) materials as well as facile fabrication onto conducting and semiconducting substrates to suit the desired applications.





Keywords: TCNQ, Nanostructured materials, Fabrication, electrochemistry

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DETERMINATION OF MICROGRAM AMOUNTS OF COPPER IN REAL SAMPLES BY USING CHEMICALLY MODIFIED CARBON PASTE ELECTRODE

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A new chemically modified carbon paste electrode (CMCPE) based on copper-Cefazoline (Cu-CFZ) ion-associate has been developed. The electrode exhibits a Nernstian slope of 29.44 mV per decade for Cu(II) ion over a concentration range of $4.01x10^{-6}$-$7.52x10^{-3}$ mol L^{-1}. The electrode has low limit of detection, 6.28×10^{-7} mol L^{-1}, fast response time of $\leq$10 s, highly selective, thermally stable and its potential response is pH independent in the pH range 1.5-5.8. This modified electrode was successfully used in determination of copper (II) in different water samples, milk powder, brass powder, soft drink and tea.

Key-words: Carbon paste; Copper; ion-associate; potentiometry

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Posters Communications' Abstracts

UV-Patterning of Ion Conducting Negative Tone Photoresists Using Azide-Functionalized Poly(Ionic Liquid)s

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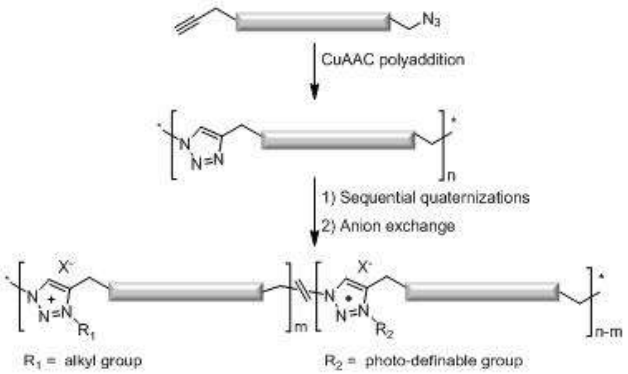
Poly(ionic liquid)s (PILs) have recently attracted a lot of attention in the fields of materials science and alternative energies since they combine the unique properties of ionic liquids with the tunable macromolecular design of polymer materials.[1] Their tremendous potential in a broad range of applications including dye sensitized solar cells, batteries, electrochromic devices or catalysis has been widely demonstrated.[2] 1,2,3-Triazolium-based poly(ionic liquid)s (TPILs) constitute the most recent addition to the pool of PIL materials.[3-5] Their synthesis benefits from the versatile, robust and orthogonal nature of CuAAC in combination with efficient quaternization and anion metathesis reactions. It thus affords developing new synthetic approaches and tackeling structural limitations of existing PILs such as lack of functionality. Herein we present the synthesis of functionalized TPILS having photo-definable reactive groups able to perform the unprecedented soft lithography of PIL materials.



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SYNTHESIS OF ZEOLITE LTA FROM ALUMINUM SCRAP AND TECHNICAL GRADE OF SODIUM METASILICATE

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Zeolites are crystalline, microporous aluminosilicates with 3-dimensional network of $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ tetrahedra linked at their corners to form channels and cages. Electrical neutrality is achieved by the inclusion of alkali or alkaline earth ions such as Na^+, K^+, Ca^{2+}, or Ba^{2+} and interstitial voids contain water molecules which may be removed reversibly or replaced by other species. Out of the various low silica zeolites, Na-LTA zeolite with chemical formula $Na_{12}[(AlO_2)_{12}(SiO_2)_{12}]\cdot 27H_2O$, has been widely used in industry as catalysts, adsorbents, ion exchangers (builder in laundry detergents) and zeolite membranes. In this work, we carry out a detailed study of the synthesis of zeolite Na-LTA using inexpensive starting materials: aluminum scrap (99.8% purity) and technical grade of sodium metasilicate ($Na_2SiO_3.5H_2O$, PQ CORPORATION). Other types of aluminum source materials have been also used for comparison: aluminum nitrate ($Al(NO_3).9H_2O$, MERCK 95% purity), aluminum chloride ($AlCl_3.6H_2O$, FLUKA 99 % purity) and potassium aluminum sulfate ($KAl(SO_4)_2.12H_2O$, MERCK 99.5 % purity). The starting aluminosilicate hydrogel with final molar composition: **3,741 Na_2O: Al_2O_3:1,450 SiO_2:175,520 H_2O** has been prepared by pouring sodium aluminate's solution into silicate solution at room temperature. As the amorphous hydrogel forms, it was agitated to produce homogeneous mixture, then aged at room temperature for 14 hours and finally heated to 80°C for 6 hours. From this study, we demonstrated that only aluminum scrap source leads to a well crystallized Na-LTA zeolite, while, the other aluminum sources provide amorphous solids (T=80°C and t=6h). It seems that the presence of inorganic anions (NO_3^-, SO_4^{2-}, Cl^-) slow down the crystallization process by increasing the steric and electrostatic interactions between the inorganic anion and the aluminosilicate anions and thereafter slow the germination and growth processes. The influence of hydrothermal synthesis temperature on zeolite crystallization process was examined using aluminum scrap. The crystallization time is reduced with increasing temperature. Pure Na-LTA zeolite was obtained after 10 hours at 60 ° C, while it is only 6 hours at 80 ° C. For the low temperatures synthesis: 40 ° C and 25 ° C, the times were 4 days and 8 days, respectively. This result suggests that increasing reaction temperature accelerates the partial dissolution of the silica and alumina components from the gel into the aqueous phase and the formation of crystal nuclei within the gel framework.

Key words: Na-LTA zeolite, aluminum scrap, hydrothermal synthesis.

PHOTOCATALYTIC DEGRADATION OF A MIXTURE OF ANTIBIOTICS: SULFAMETHOXAZOLE AND TYLOSIN

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Photocatalytic oxidation process based on UV irradiated TiO_2 is a promising technology for the degradation and mineralization of many environmental pollutants like pesticides, dyes and pharmaceutical compounds.

In this work, the photocatalytic degradation of a mixture of two antibiotics Sulfamethoxazole (SMX) and Tylosin using supported TiO_2 was investigated under UV irradiation in a recirculating reactor. A High performance liquid chromatography (HPLC) was used to determine the amount of SMX and Tylosin in the mixture and to compare the degradation of both molecules individually and mixed. The measurement was conducted at two wavelengths 262 and 290 nm, respectively for SMX and tylosin.

The kinetic model of Langmuir-Hinshlewood was applied to the experimental data and the apparent reaction rate constants were determined. Results show that the degradation efficiency of one antibiotic may be enhanced by the presence of the other.

The evolution of the total organic carbon (TOC) of two molecules during their photocatalytic degradation in single and mixed solution was also studied.

Key words: SMX; Tylosin; Mixture; Photocatalysis; TiO_2.

LEAD TRANSPORT IN AN UNSATURATED SOIL: COLUMN EXPERIMENTS AT CONSTANT WATER CONTENT

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The contamination of soil with heavy metals is a problem of current interest. The migration of heavy metals has been the topic of experimental researches, mainly at laboratory scale, using batch experiments or saturated column tests. Although the results found under saturated conditions are encouraging, they remain insufficient to evaluate a real pollution often developed under unsaturated conditions. The aim of this study is a better understanding of heavy metals transport in unsaturated soils under controlled suction conditions. Very little data of this experimental conditions case have been reported in the literature.

In this study, lead is chosen as a model pollutant as it is frequently found in contaminated soils. The porous media used in the laboratory experiments is a soil constituted from the mixture of Hostun sand (96,7%), clay (3%) and compost used as a source of organic matter (0,3%). In a first step, batch and saturated column experiments were carried out to identify sorption isotherms**.** In all cases, Langmuir isotherm was found. Results of batch experiments indicate that the maximum sorption capacity of the model soil is 1666 mg / kg, in accordance with saturated column experiments.

In a second step, and to better understand lead transport mechanisms, soil column experiments in unsaturated regime under suction-controlled conditions were carried out. The experimental device consists of a Plexiglas column with a height of 20 cm and a diameter of 7,022 cm. The column is provided with a lateral outlet for measuring the suction and therefore the soil water content. This device, given the infiltration flow and the column geometric conditions, has been designed to obtain nearly constant water content. After equilibrium, lead solution at a concentration of 200 mg/L is introduced using Mariotte device. Gravity flow is established. pH as well as the concentration of lead are monitored at the column outlet. The breakthrough curve of lead shows that lead concentration at the column outlet is zero up 4 porous volumes and the curve can be modeled and identified according with Langmuir kinetics. It was also noticed that the mass of sorbed lead is 22% higher in saturated tests than unsaturated experiments.

Our study allowed the establishment of a non-saturated column device for evaluating the transport of lead in unsaturated model soil at constant water content. The results have identified the adsorption kinetics.

Key words: unsaturated soil-column experiments-constant water content.

ELABORATION AND CHARACTERIZATION OF ULTRAFILTRATION MEMBRANES MADE FROM NATURAL AND SYNTHETIC PHOSPHATES

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This study is related to the development of ceramic membranes using natural and synthetic phosphate. The preparation and characterization of macroporous support [1, 2], microfiltration and ultrafiltration layer were described in this work. Microfiltration membrane of natural Moroccan phosphate [3] and ultrafiltration of potassium titanyl phosphate (KTP) were performed respectively by the slip casting process and sol-gel route. The characterization of this membrane was obtained by mercury porosimetry (for microfiltration layer), by adsorption d'azote (for ultrafiltration layer) and SE. The support fired at 1000 °C showed pore diameters centered near 10 µm and porosity about 43 %. The pore diameters are 0,35 µm and 10 nm respectively for microfiltration and ultrafiltration membrane fired at 550 °C . The water permeability measured for the microfiltration membrane is 700 $l.h^{-1}.m^{-2}.bar^{-1}$ and for ultrafiltration is 80 $l.h^{-1}.m^{-2}.bar^{-1}$.

Key words : Ceramic ; Phosphate ; Sol – gel ; Microfiltration ; Ultrafiltration ; Potassium titanyl phosphate.

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Eco-Friendly chemical routes for the transformation of terpenoids in green solvents to effective additive materials by using ruthenium complexes

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New terpenoids were prepared by ruthenium catalyzed cross-metathesis of terpenoids **1a-c** with methyl acrylate **2**. Because of side reactions, the transformation of these terpenoids was more difficult. The cross-metathesis reactions were carried out under environmentally friendly conditions in glycerol. The new terpenoids compounds were fully characterized by FT-IR, ^{1}H, ^{13}C spectroscopies, and by HRMS analysis.

Keywords: terpenoids, ruthenium, cross-metathesis

Valorization of fine and big wastes of Oum El Khechab's phosphate by the method of floatation

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Phosphates' reserves in Tunisia decrease constantly as a result of their intense exploitation. Thus, at present all researches focused on the fine and big rejects' recovery. Indeed, analyses by X- ray Diffraction reveal mineral phases of these releases. The most common one is the Carbonate Fluor Apatite $\mathbf{Ca_{9.35}(PO_4)_{4.72}F_{1.98}(CO_3)_{1.483}}$ which is considered as the noble mineral of phosphates rocks and which can be recycled. Further, exploitation of these rejections solves the problem of soil and waters pollution of Metlaoui area in southern Tunisia. Therefore, this research is undertaken to valorize rejects in the Oum El Khechab's future factory.

The work is devoted to find the convenient process then to optimize floatation parameters. The enhancement of the treatment was confirmed by chemical analyses. In fact, weight yields of fine rejections decreased significantly at about 76,33% of the fraction between 100µm and 40µm which recycled and those of big rejections decreased significantly at about 30,63%. Moreover, recuperation of P_2O_5 reached respectively about 88,19% and 45,22% of P_2O_5.

Keywords: phosphate, big rejections, fine rejections, floatation.

Analytical Study of Electrode Cadmium Oxide Prepared by Nanoic method

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In this research, preparation of Cadmium Oxide nanoparticles (NPs) that has been prepared from materials primary using microwave-assisted method. CdO NPs were characterized by X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FT-IR). That the size NPs of CdO were about (17.44nm) as calculated from the Scherrer formula from the most intense XRD peak. In addition to comparison between CdO NPs and CdO Microparticles (MPs) by using the thermal analysis (TG,DTG,DTA).

A Cd^{2+}-selective electrode based on poly vinyl chloride (PVC) was prepared by using CdO NPs powders synthesized and di-butyl phthalate (DBP), In addition to a comparison between the specification analytical for CdO NPs electrode and CdO MPs electrode. CdO NPs electrode and CdO MPs electrode give linear rang of (10^{-2}-10^{-5})M and (10^{-2}-10^{-4})M, Nernstain slope of (30.18 mV/decade) and (31.77 mV/decade), correlation coefficient of (0.999) and (0.998), detection limit of ($1.0044x10^{-7}$) M and ($2.8458x10^{-6}$)M, quantitative limit of ($3.348x10^{-7}$)M and ($9.486x10^{-6}$)M, the response time of (6-35) second and (10-33) second, respectively. The lifetime for each of electrode was 13 days. The optimal conditions for each of CdO NPs electrode and CdO MPs electrode were (5.5-7),(20-30° C), (10^{-4})M for pH, temperature and concentration of filling solution respectively.

In addition to a measurement selectivity of these electrodes using mixed solutions method, the results showed that the selectivity coefficient values for all interferences ions are less than one. And make some applications of these electrodes using direct method and multiple standard addition method.

Key words: Cadmium Oxide, nanoic method, analytical study, NPs, CdO NPs electrode.

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Natural antioxidants (polyphenolics and flavonoids) of olive leaves from olive trees grown in Palestine

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Different olive leaf samples were collected from different geographical regions of West Bank (north, middle, and south) at different maturation stages (June 2013, October 2013, and January 2014). The olive leaves samples were dried at 30 °C, grinded with a blender, and the dried powder was extracted with water or with acidified water (pH 3.0), at room temperature for two hours. The crude extracts were then analyzed for their total phenolic content (TPC), total flavonoid content (TFC), and antioxidant activity (AA) using standard assay methods (Folin-Ciocalteau method for TPC, ferric ion reducing antioxidant power (FRAP) for AA, and colorimetric assay method for TFC). Three samples of olive leaves of each treatment were independently analyzed in each sampling, and all of the determinations were carried out in triplicate. The results revealed that maturation stage affected the composition of the olive leaves under investigation in terms of total phenolic, total flavonoid, and antioxidant activity. Additionally it was found that there is a significant difference in TPC, TFC, and AA of the olive leaves samples collected from north, middle, and south West Bank. Comparing both extraction media (water and acidified water) in terms of TPC, TFC, and AA, there was no clear trend because the differences were significant in all samples, but the higher values alternate between different extraction methods in different samples. Pearson correlation between TPC, TFC, and AA of samples collected from the three areas in West Bank showed that antioxidant activity is significantly correlated with total phenolic content in both extraction treatments, and weakly correlated with total flavonoids content. But when all data was pooled, both TPC and TFC were highly and significantly correlated with AA. It was also found no significant correlation between TPC and TFC in any sample under investigation.

During different maturation stages (from June 2013 to January 2014), total phenolic content varied between 26.14 - 47.52, 25.42 - 46.13, and 21.56 - 44.13 mg gallic acid equivalents (GAE)/ g dry of olive leaves (extracted with water) collected from north, middle, and south West Bank, respectively. Total flavonoid content, also varied during consecutive maturation stages and ranged between 19.4 - 26.5, 19.3 - 21.6, and 21.6 - 32.6 mg catechin equivalents/g dry of olive leaves (extracted with water) collected from north, middle, and south West Bank respectively. The antioxidant activity (FRAP assay) measured during different maturation stages was found to be in the range of 422.20 - 936.67, 337.53 -747.70, and 318.53 – 1106.43 µmol FRAP equivalents/g dry of olive leaves (extracted with water) collected from north, middle, and south West Bank, respectively.

Key words: Olive leaves, antioxidant activity, phenolic content, flavonoid content, Palestine.

Hydrodesulfurization of thiophene using Phosphomolybdic acid and nickel substituted Phosphomolybdic as catalyst

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Phosphomolybdic acid (PMA) supported on Silica and substituted by Ni was prepared in different loading and characterized by several techniques including FT-IR, powder X-ray diffraction, Thermogravimetric analysis, BET surface area and acid strength. The results show that the Keggin structure is intact in all the catalysts even after calcination at 300 ^{o}C. The surface areas of the catalysts are slightly lower than the surface area of the silica used in making these catalysts. All catalysts were tested for their reactivity in the hydrodesulfurization of thiophene. The catalysts studied showed very good activity under the reaction conditions. The results indicate that 25% PMA catalysts are more active in hydrodesulfurization compared with 15% PMA on silica. The results of nickel substituted heteropoly acids clearly indicate that although the catalytic activity of nickel-substituted HPA is higher than the unsubstituted HPA, but complete substitution lowers the activity, indicating that the presence of acid and nickel both play a role in the hydrodesulfurization of thiophene.

The promoting effect of drying under supercritical conditions on the characteristics and catalytic performance of palladium supported on alumina modified with zirconia

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This work constitutes a new trial to enhance the properties of palladium supported on alumina modified with zirconium. As already mentioned in previous works [1,2], the above catalysts were prepared using sol-gel process or sol-gel combined with impregnation. The second route of preparation is seen to be better [2]. In fact, it contributes to more active catalysts with better palladium dispersion but mediocre specific surface area. In order to enhance the textural properties of these catalysts which may improves the palladium dispersion and hence the catalytic properties, we have tried the drying of Al_2O_3-ZrO_2used as support under supercritical conditions of the solvent. For this aim, a mixture of tri-sec butoxide of Aluminum and Sec-Butanol was stirred for 30 min. Zirconium acetylacetonate is then added to have a zirconium loading of 2, 5 and 10 %wt. The addition of acetic acid to this mixture with a molar ratio $\frac{n(alkoxide)}{n(CH_3COOH)}$ equals to 4, allows the formation of wet gels that we dried in an autoclave at a temperature T above the critical temperature T_c of the solvent. Palladium is finally deposed on the obtained aerogels by wet impregnation using Palladium acetylacetonate as precursor to have a weight loading of 0.5%.

Surface area, crystalline phase, palladium dispersion and reducibility were determined for the various catalysts. Catalytic activities were measured in methane combustion. The composition of the reaction gas was 1% CH_4, 4% O_2 and balance He. The catalytic tests were performed in a dynamic microreactor over the oxidized sample (100 mg).

From the obtained results, we can confirm the promoting effect of this mode of drying on the textural properties and even the metal dispersion and reducibility. The catalytic performance is also seen to be better than that of sol-gel derived catalysts.

Key words: Palladium, Aerogel, methane, Zirconium

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CONVENIENT FIRST SYNTHESIS OF TERTIARY HYDROXY-γ,γ'-BISPHOSPHONATES AND EVALUATION OF THEIR ANTI-INFLAMMATORY ACTIVITY

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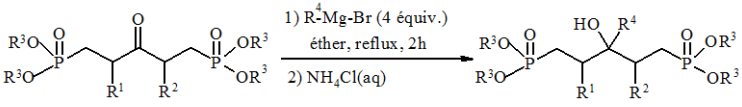
Recently, the development of organophosphorus chemistry has been characterized by a great interest in bisphosphonates and bisphosphonic acids owing to their many useful properties ranging from pharmacological activities to their heavy-metal-complexing ability. To the best of our knowledge, although hydroxy-*gem*-bisphosphonates are well described in the literature, their symmetric γ,γ'-bisphosphonate analogues with a tertiary alcohol functionality, have never been synthesized and evaluated. Therefore, we have been interested in this work in developing a synthetic pathway to these novel hydroxybisphosphonates of type **2**, from the reaction of γ,γ'-diphosphonyl ketones **1** with Grignard reagents, followed by hydrolysis (Scheme 1).



1 **2**

Scheme 1

In the second part of this work, we focused our efforts to evaluate the anti-inflammatory activity of the synthesized compounds **2**. Anti-inflammatory activity tests were carried out by hind-paw edema's method, using *yeast beer* as a phlogistic agent, on Wistar rats. The results showed that tertiary hydroxy-γ,γ'-bisphosphonates **2** were effective as anti-inflammatory agents with a paw-inhibition toward 20% compared to the standart drug (77 %) after 24 hours.

Key words: hydroxy-γ,γ'-bisphosphonates, γ,γ'-diphosphonyl ketones, anti-inflammatory activity.

MOLECULARLY IMPRINTED POLYANILINE SORBENT OF SUNSCREENS IN WATER

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Sunscreens, including chemical UV filters, are a threat to the environment due to their endocrine potential in contributing, among others, to the feminization of fish species and coral bleaching [1]. These filters pollute the aquatic environment, rivers lakes and seawater, either directly by swimming or water sports, or by indirect contributions related to discharge of water from wastewater treatment units.

The analysis of such pollutants is conducted by chromatography. In general, these pollutants exist in low concentrations in complicated matrices, so their determination requires extraction and concentration steps prior to instrumental analysis. Solid phase extraction (SPE) are now coming mostly used for the extraction of analytes and are substituting liquid-liquid extraction (LLE) protocols.

Molecularly Imprinted Polymers (MIPs) have been used as SPE sorbents and seem tobecome the promising development to circumvent the drawbacksof traditional SPE sorbents owing to the recognition ability of MIPsfor given molecules.

Acrylic polymers weremostly used for the production of MIPs. However, the common synthetic route (free radical polymerization) requires specific and non simple conditions (organic and oxygen free medium, thermo- or photolabile initiators). Polyaniline (PANI) could be a good alternative for the production of MIPs due to its easy synthesis by oxidative polycondensation of aniline in aqueous medium andits excellent environmental stability.

The aim of the present work was to develop a polyaniline-based MIP for sunscreen at the surface of silica particles. Thin layers of MIP at the surface of silica particles provideeasily accessible sites related to fast association/dissociation kinetics.

Polymerisable entities were first grafted at the surface of silica particles. The grafting degree was determined from elemental analysis. Polymerisation was conducted from surface attached groups in the presence or absence of sunscreen for the production of MIP or NIP coating respectively. The adsorption sites were revealed by extraction of sunscreen from coated silica particles. A good extraction was attained by use of soxhlet method.

Comparison study between imprinted (MIP) and non imprinted (NIP) materials was conducted by means of kinetics and adsorption isotherms measurements.

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EFFECT OF Co ADDITION ON THE MARTENSITIC TRANSFORMATION OF $Ni_{50-x}Co_xMn_{38}Sn_{12}$ (x = 1, 2 and 3 at.%) MELT SPUN ALLOYS RIBBONS

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In this work, ribbons of composition $Ni_{50-x}Co_xMn_{38}Sn_{12}$ (x = 1, 2, and 3 at%.) were produced by the melt-spinning technique. Martensitic transformation shifts to lower temperatures with increasing: a) concentration of Co or b) the external magnetic field. Structure at room temperature of alloys with x=1 and x=2 is martensite (Monoclinic with a 10M modulation), whereas for the alloy with x=3 is austenitic. The changes in temperatures of martensitic and magnetic transformations are confirmed to directly report to the (e/a) ratio of the matrix. The minimum (e/a) ratio value for the occurrence of the martensitic transformation are estimated at 8.11 for the alloy system studied.

Keywords: A. Shape memory alloys; B. Martensitic transformation; C. Rapid solidification; D. Microstructure.

INVESTIGATION ON THE TiO_2-MODIFIED MONTMORILLONITES WITH IMPROVEMENT ACTIVITY

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The decreasing availability of fresh water all over the world is a great worry. That's why many efforts have been made to deal with this problem. This study presents a new method for wastewater treatment. In fact, a series of TiO_2-modified montmorillonites, as photocatalysts, was successfully prepared with different ways. The photocatalytic activity of this porous material was evaluated by 4-Nitrophenol degradation in wastewater.

The effect of photocatalysts preparation methods on the textural, structural and catalytic properties of the samples was examined. To understand the relationship between the structure and performance of the photocatalysts, sample materials were characterized by X-ray diffraction (XRD), UV-spectroscopy, Scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/MEB), Fourier transform infrared spectroscopy (FTIR), and specific surface area and porosity measurement, respectively.

Finally, to improve the 4 –Nitrophenol degradation, various parameters effect were studied, such as photocatalyst loading, type of catalyst, 4–Nitrophenol concentration, pH of aqueous solution, and addition of H_2O_2.

TiO_2-modified montmorillonites with high specific surface, mesoporous structure and high stability were obtained.

The degradation degree was tested by UV spectral analysis and HPLC indicating that 4–Nitrophenol was completely mineralized, so photocatalysts used are able to perform efficiently degradation of 4-nitrophenol using UV irradiation.

Key words: wastewater treatment, TiO_2-modified montmorillonites, porous material, photocatalytic degradation.

EFFICIENT CONTINOUS FLOW SYNTHESIS OF 2-MERCAPTOTHIAZOLE DERIVATIVES AS INHIBITORS OF INDOLEAMINE 2,3-DIOXYGENASE

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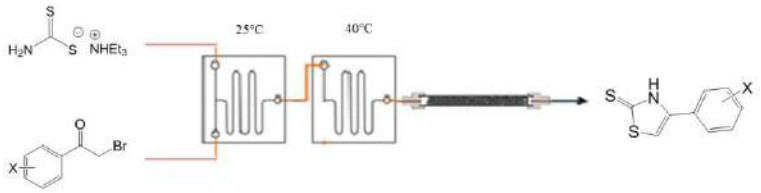
Common industrial processes such as production of petrochemicals are mostly carried out in continuous flow systems. However, production of most fine chemicals and pharmaceuticals is carried out in batch reactors. Moreover, synthetic methodologies and tactics for making complex organic molecules have been traditionally developed based on batch technology in laboratories. Recently, the introduction of continuous-flow synthesis platform to laboratory synthesis and production of fine chemicals and pharmaceuticals has become increasingly popular [1]. Global research activities have clearly demonstrated that the application of micro-structured reactors, mixers and other microfluidic components offers numerous technical advantages [2-4] for reactions in chemical processes. Such methodology has obvious potential for the reduction of waste and decrease in the direct handling of potentially hazardous materials and less polluting processes is attractive to sectors that face a range of regulatory and commercial pressures.
In this presentation we describe an efficient continuous-flow procedure for the synthesis of 2-mercaptothiazole derivatives starting from substituted phenacyl bromide and ammonium dithiocarbamate.



Key words: Flow chemistry, 2-mercaptothiazole, indoléamine 2,3-dioxygénase

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Biodiesel production from unrefined and refined olive pomace oil: Comparative study

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Tunisia is occupying the fourth ranking in the world olive oil producers [1]. Despite its economical benefits, Olive oil production is generating a huge amount of olive pomace which is containing a highly toxic compound. In Tunisia, the valorization of this waste into renewable energy constitutes new trends. The valorization has many environmental contributions in addition to the economical assets [2-5]. The aim of this study was to determine the optimum conditions of the biodiesel production from refined and unrefined olive pomace oil. The refined olive pomace oil had an acid value of 2.34 mg KOH / g-oil. Therefore, transesterification reaction could be directly applicable due to the low free fatty acids content. The biodiesel yield reached from this oil was 100%. The unrefined olive pomace oil had an acid value of 44 mg KOH / g-oil. Due to its high free fatty acids content, a pretreatment step, an esterification reaction, was required to decrease the percentage of free fatty acids to the limit necessary to achieve the transesterification reaction. Conditions already optimized with the refined olive pomace oil for the transesterification process were preserved for the pretreated one. The yield of the produced biodiesel in that case reached 99 %.

Key words: Biodiesel, free fatty acids, transesterification, esterification, unrefined olive pomace oil.

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CARACTERISATION DE LA CHARGE POLLUANTE DES DEBLAIS DE FORAGES PETROLIERS DU SUD TUNISIEN ET ESSAI DE DEPOLLUTION PAR VOIE THERMIQUE

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Dans l'industrie pétrolière et suite à l'identification d'un gisement potentiel, on procède à un forage d'exploration. Cette étape constitue à creuser le sol avec un trépan, jusqu'à parfois plus de 4000 m de profondeur, tout en injectant une boue de forage.

Il s'agit d'un système à base d'eau, d'huile et/ou de gaz contenant en suspension d'autres additifs minéraux et organiques.

Ce mélange de composition variable, assure le bon déroulement du forage et ramène à la surface les fragments de roches détruites, appelés déblais. Ces derniers, séparés des boues sont rejetés dans un bourbier, et peuvent garder en retention des quantités importantes des fluide de forage. Ainsi, ces déblais constituent un rejet polluant et une source potentielle de contamination du sol.

Afin d'évaluer la charge polluante des déblais de deux forages pétroliers du sud tunisien, on a déterminé la teneur en hydrocarbures (Total petroleum hydrocarbons) par pyrolyse Rock-Eval. La caractérisation des polluants des bourbiers échantillonnés a été réalisée par chromatographie en phase gazeuse et on a pu confirmer la présence de métaux lourds par ICP-AES; suite à une digestion assistée par micro-ondes.

Les résultats trouvés confirment une contamination d'origine pétrogénique des déblais par les composés hydrocarbonés

Enfin, la technique de dépollution des échantillons par voie thermique s'est avérée efficace, puisqu'elle a permis d'atteindre des taux d'abattement intéressants, pour les essais réalisés.

Mots clés : Déblais, forage pé trolier, hydrocarbures, TPH, métaux lourds, traitement thermique, ICP-AES, Rock-Eval, CPG.

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SYNTHESIS AND PHOTOCATALYTIC ACTIVITY OF C-DOPED ZNO FOR THE BISPHENOL A DEGRADATION UNDER UV IRRADIATION

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Semiconductor photocatalysis shows importance over the last few years due to its ability to destroy a wide range of organic and inorganic pollutants. The basic process of photocatalysis consists of ejecting an electron from the valence band (VB) to the conduction band (CB) of the semiconductor creating a hole in the valence band [1]. Oxide semiconductors such as TiO_2, ZnO, SnO_2, WO3, etc. have been used for the photocatalytic degradation of a wide variety of environmental contaminants. Zinc oxide is a wide band gap semiconductor with an energy gap of 3.37 ev. It has been proven to be active in the photocatalytic degradation of environmental pollutants [2].

In this work C-doped ZnO with different doping contents of carbon were successfully synthesized by a hydrothermal method. The samples were characterized by X-ray diffraction (XRD), Nitrogen physisorption at 77 K, Raman spectroscopy, Fourier transformed infrared (FTIR), UV–vis diffuse reflectance spectroscopy, and photoluminescence spectra (PL). The results show that the as-prepared catalysts have high crystallinity and the undoped catalyst presents the smallest surface area (14.93 m^2/g), indicating that the carbon doping increases the catalyst's surface area. Furthermore, it can be seen that the pore size distribution strongly depended on the carbon content. The presence of carbon could improve the separation extent and restrain the recombination of the photo-induced electron and hole carriers. The bisphenol A degradation and mineralization were carried out to evaluate the photocatalytic activity of the already prepared catalysts under UV irradiation and in the presence of hydrogen peroxide.

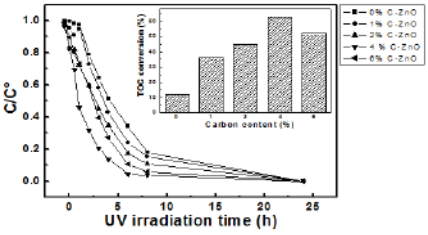

Fig.1. Effect of the carbon doping content on the BPA photocatalytic degradation, inset shows the corresponding TOC conversion

It is clearly seen that in the presence of doped catalysts with different carbon molar ratios, the BPA photocatalytic degradation is much higher than that of pure ZnO (Fig. 1). Kinetic study shows that 4% C-doped ZnO K_{app} value reached the highest one and was about twice the pure ZnO K_{app} value.

Key words: C-doped ZnO, Semiconductor, Bisphenol A, Photocatalytic degradation.

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STUDY OF THE BEHAVIOR OF SOME TYPES OF REFRACTORY BRICKS IN KILN CEMENT

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Refractory bricks are exposed in a cement kiln to thermo –chemical stresses that can accelerate the infiltration of material and volatile elements in the bricks [1]. In this context, three types of post -mortem bricks were studied: two magnesia – spinel bricks (burning area) and aluminosilicate brick (72 m out of the kiln). One of the two bricks magnesia –spinel has retained its original color. The two others have been divided into different parts in the ferrule –material direction: the second magnesia –spinel brick has three parts which has the colors: light brown, white and brown. The aluminosilicate brick has four parts which has the colors: black, gray, red and yellow. To understand the chemical changes that bricks have undergone, analysis by ICP, electron microscopy and XRD has been used. The results demonstrated the variation of contents of some minor elements in the different samples. The treatment of diffractograms, by the X'Pert Highscore software, revealed that these elements have attributed to the formation of new phases such as K_2MgSiO_4 in magnesia –spinel brick and leucite K ($AlSi_2O_6$) in the yellow area of the aluminosilicate brick. In addition, the iron could be incorporate in the spinel $MgAl_2O_4$ forming $MgFe_{0,2}\ Al_{1,8}\ O_4$.

Key words: magnesia – spinel and aluminosilicate bricks, chemical and thermal stresses, kiln, volatile elements.

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PREPARATION AND CHARACTERIZATION OF β-CYCLODEXTRIN BASED MICROCAPSULES

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Nowadays, the synthesis of polymeric materials based on monomers from renewable feedstock is a steadily growing area of interest, stimulated by the rising concern for the environment and the fast/prompt exhaustion of petroleum reserves [1]. On the other hand, several (such) bio-based polymers are exploited in microencapsulation field as a shell surrounding a functional compound. In this context, we study the development of polyurethane bio-sourced microparticles based on β-cyclodextrin for further applications in cosmetotextile field. A novel kind of microparticles containing 2-ethoxynaphtalen (neroline) as a perfume was successfully synthesized. The biodegradable polyurethane shell was synthesized by interfacial polycondensation of β-cyclodextrin (β-CD) with hexamethylene diisocyanate (HDI) [2]. The success of polymerization reaction leading to the formation of the polyurethane shell was checked by ATR-FTIR. Optical microscopy and scanning electron microscopy (SEM) were used to study the microparticles morphology and shape. Particle size distribution was studied by small-angle light scattering using a particle size analyzer. Thermal behavior studied by thermogravimetric analysis (TG) and Differential Scanning Calorimetry (DSC) showed excellent properties of synthesized microcapsules. Their stability and isoelectric point (IEP) in aqueous solution were studied using small-angle light scattering in order to have an idea about their storage and their fixation onto textile substrate.

Key words: Polyurethane; β-cyclodextrin; renewable feedstock; neroline; interfacial polycondensation; microcapsule.

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Structure determination and optical properties of $CsSm(PO_3)_4$

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The $CsSm(PO_3)_4$ polyphosphate was synthesized by flux method and characterized by XRD, IR and Raman spectroscopies. It crystallizes in the monoclinic system with space group $P2_1/n$ and adopts the following unit cell parameters: a = 10.382(2), b = 8.978(6), c = 11.205(4) Å, β = 106.398(3)° and Z = 4. Its crystal structure was consisted of a three-dimensional framework in which the zig-zag $[(PO_3)]^{n-}$ chains are connected by SmO_8 and CsO_{11} polyhedra. The spectroscopic properties of Sm^{3+} in this new compound including excitation, emission and lifetime measurements was performed at room temperature. The emission spectrum shows four photoluminescence emissions at 560, 600, 650 and 700 nm corresponding to the $^4G_{5/2} \rightarrow {}^6H_J$ (J = 5/2, 7/2, 9/2 and 11/2) transitions of the Sm^{3+} ion, respectively. The lifetime of the strongest emission at 600 nm and the chromatic coordinates of the emitted light indicate the usefulness of this phosphorus as a potential orange- reddish emitting material.

Key words: Samarium; polyphosphate; excitation; emission and chromatic coordinates.

Advanced Pd/$Ce_xZr_{(1-x)}O_2$/MCM-41 catalysts for methane combustion: Effect of precursors addition step

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Catalytic methane combustion has received considerable attention in the last decades due to its practical applications in both power generation and pollutant abatement such as NOx, SOx, unburned hydrocarbons,… [1, 2]. This reaction has been shown to be effective in producing energy in gas turbine combustors. Recently, the supported palladium catalysts doped with transition metals such as Ce, Zr are attracting considerable attention due to their high activity [3]. Thanks to palladium oxide which is considered the most effective catalyst used in methane oxidation and to the $Ce_xZr_{(1-x)}O_2$ solid solution which has drawn great attention as an outstanding oxygen storage material. In this work, The MCM-41 is prepared by sol-gel method and the addition step of cerium and zirconium are changed in order to modify the Pd-support interaction and consequently the structure of the active phase.

The Pd/$Ce_xZr_{(1-x)}O_2$/MCM-41catalysts are prepared by sol-gel method with palladium acetylacetonate, cerium nitrate, zirconium acetylacetonate, CTAB and tetraethyl orthosilicate (TEOS) as precursors. The cerium and zirconium introduction step during the catalyst synthesis is changed. In effect, cerium nitrate and zirconium acetylacetonate are introduced in the TEOS solution (M1), on the dried MCM-41gel (M2) or on the calcined MCM-41 (M3). 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 M3 compared to M2 and M1 catalysts. The XRD results show that only M3 catalyst contain a $Ce_xZr_{(1-x)}O_2$ solid solution. The MET-EDX results show a considerable growth of the CeO_2 and ZrO_2 particle size according to the following order: M3 > M1 > M2. Furthermore, the different reduction temperatures of the PdO phase obtained from TPR characterisation shows that the catalyst reducibility depends greatly on the ceria-zirconia structure and probably to the Pd-ceria-zirconia.

The catalytic activity results show that the ceria-zirconia introduction step modifies remarkably the activity of the catalytic sites. Globally, the improvement of palladium dispersion and PdO reducibility increases the activity in methane combustion.

Key words: methane combustion, palladium, ceria-zirconia.

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FATTY ACID COMPOSITION OF *DIPLOTAXIS ERUCOIDES* FLOWERS AND ANTIOXIDANT AND ANTIMICROBIAL ACTIVITIES

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The genus *Diplotaxis* (Brassicaceae family) includes more than 30 species which are native to the Mediterranean region and currently distributed from Central Europe. It is also widespread in Tunisia. Many original studies refer to *Diplotaxis* as a traditionally used plant with therapeutic properties and as an alimentary plant usually consumed in salads. The present work describes the guided fractionation of the ethanol extract of *D. erucoides* flowers which is partitioned successively with hexane, chloroform, ethyl acetate, n-butanol and water. Fatty acid composition of hexane extract was determined by gas chromatography (GC-MS).The antioxidant activity of the different extracts was evaluated using DPPH and ABTS radical scavenging and reducing power assays. The antibacterial potential was tested against several human pathogenic bacteria. Results showed that the fatty acid composition of *D. erucoides* flowers is predominated by the polyunsaturated linoleic acid (23.65 %). The butanol extract had the highest antioxidant activity with an IC_{50}value of 0.024 ± 0.001 mg/mL. This extract had also an interesting antimicrobial activity against *Staphylococcus aureus* and *Pseudomonas aeruginosa monocytogene* which were the most sensitive bacteria with a 21 mm of inhibition diameter at an extract concentration of 250 µg / ml.

Key words: Diplotaxis erucoides, Fatty acids, antioxidant and antimicrobial activities.

ASSESS OF PHYSICAL ANTISCALE-TREATMENTS AS ALTERNATIVE OF THE CHEMICAL ONES DURING BRACKISH WATER DESALINATION BY CONVENTIONAL ELECTRODIALYSIS

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In electrodialysis (ED) desalination plants, calcium carbonate is the main component of meted scales. To prevent its formation several treatments were proposed. For more efficiency, treatments must be assessed at experimental conditions close to real ones. Thus, this work is a contribution to understand and evaluate three anti-calcareous physical treatments, as alternatives to the chemical ones, for ED desalination systems simulating real conditions. Magnetic field (MF) and ultrasonic field (UF) were applied to concentrate solution, compartment where scaling is imminent in the used ED pilot unit. The third treatment was a pulsed electric field (PEF) application. Tested solution was a synthetic brackish water. Results show that magnetic and ultrasonic treatments accelerate the precipitation of $CaCO_3$ by reducing the nucleation time and the metastable domain. It is also shown that pulsed electric treatment accelerates $CaCO_3$ precipitation resulting from desalination improvement comparing to stationary mode. However, all these treatments favor the homogeneous precipitation which prevents scale formation on membrane surfaces. It seems that MF improves the desalination only by preventing membrane scaling. However, UF and PEF application improve desalination by preventing membrane scaling and by improving the ions transfer during desalination; UF application acts on ions mobility or diffusion, while PEF application reduces the concentration polarization layer.

Key words: Electrodialysis, Desalination, Calcium carbonate, Antiscaling treatment

Kinetic and thermodynamic study of iron adsorption on different sources of calcite.

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This paper presents study of iron removal in aqueous solutions using different sources of calcite. Five limestone samples were used as adsorbents, including three from natural rocks. The effects of various factors (Chemical composition of the limestone samples, temperature and pH) were studied. Chemical composition of these samples can affect the removal reaction between Fe (II) and calcareous material. Therefore, the impurities containing in the naturals rocks were found to foster Fe (II) uptake by calcite and increase the removal effectiveness by complexing Fe (II), most likely through the minerals hydroxides such as SiO_2, FeO_3 and AlO_2, thereby maintaining Fe (II) fixation in the calcareous surface. Moreover, various kinetic and equilibrium parameters for iron removal from water using calcite were determined using kinetic pseudo-second-order, Langmuir and Freundlich models. Our experimental results demonstrate that 0.4 gram of calcite 'RC' can fix up to 71.65 and 24.3 mg/g of iron as per Langmuir and Freundlich models, respectively. A thermodynamic study was carried, showing an endothermic ($\Delta H>0$) and spontaneous ($\Delta G<0$) iron adsorption reaction into calcite 'RC'.

Key words: Iron removal, Oxidation, Adsorption, Calcite.

SYNTHESIS AND CHARACTERIZATION OF NEW ANTHRACENE-BASED SEMI-CONDUCTING POLYMERS

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Two polymers containing alternated phenylene-vinylene and anthrylene-vinylene moieties (figure 1) have been synthesized via the Wittig polycondensations according to a previously reported procedure using the *t*-BuOK/THF system. [1] Their molecular structures were confirmed by ^{1}H NMR and FT-IR spectroscopies. The polymers are soluble in common organic solvents and have number-average molecular weights of 4000 and 13750 $g.mol^{-1}$ for **P1** and **P2**, respectively. The DSC analysis shows a good thermal stability and a Tg at 120 °C for **P1** and 110°C for **P2** were obtained.

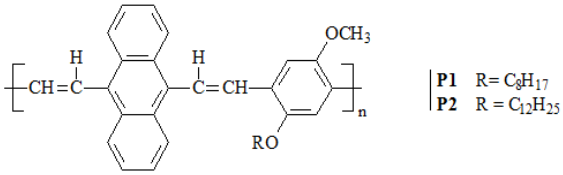

Figure 1: Molecular structures of the polymers

The optical properties of the polymers were investigated by UV-visible absorption and florescence spectroscopies. The optical gaps were estimated from the absorption-onsets of the polymer films; the obtained values were 2.75 eV and 2.60 eV for **P1** and **P2**, respectively. In dilute solution, the **P1** exhibits a blue emission; whereas, a yellow florescence was observed in the case of **P2**. The HOMO and LUMO levels were estimated using cyclic voltammetry analysis; the values show a significantly effect of the side chain on the electronic structure of these semi-conducting polymers.

Key Words: semi-conducting polymers, anthracene, Wittig reaction, photoluminescence.

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DETECTION OF DRUG RESIDUES IN THE ENVIRONMENT USING GC-MS TECHNIQUE: METHOD DEVELOPMENT, DISTRIBUTION IN THE DISSOLVED, PARTICULATE AND SEDIMENTARY PHASES FROM TRANSBOUNDARY RIVERS.

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Because of their large and widespread application, drug residues, are ubiquitous in the environment. Their presence in the environment has attracted considerable attention due to their potential impact on ecosystem functioning and on public health. In this work 14 drug residues have been quantified in three matrices (in dissolved phase, associated to suspended solid matter, and sediment) collected from fifteen watercourses and Rivers located in a highly industrialized zone at the cross-border area in Northern France and Belgium. Extraction using ASE for solid matrixes (suspended solid matter, sediment) and SPE for liquid matrix followed by GC-MS analysis, allowed the determination of individual and total drug residues concentration in the three matrices from the fifteen stations. While the Σ_{14}drug residues were lower than1.3 µg L^{-1} in dissolved phase, lower than30 ng L^{-1}associated to SSM (lower than 1.4 µg g^{-1}dw of SSM) and n.d.-60.7±9.1 ngg^{-1}dw in sediment.

Keywords: drug residues, water, sediment, ASE, SPE, SSM, GC/MS.

ELECTRODIALYTIC REMOVAL OF IRON FROM BRACKISH WATER

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The aim of this work is to study the removal of iron from brackish water using electrodialysis. Experiments were carried out on synthetic brackish water solutions using a laboratory scale electrodialysis cell.
The influence of several parameters on process efficiency was studied. This efficiency is expressed by the removal rate, transport flux, current efficiency and power consumption.
The applied voltage, the feed flow rate, the pH and iron initial concentration of the feed solution have a significant effect on the process efficiency and mainly on the iron transfer from dilute to concentrate compartment. Nevertheless feed ionic strength hasn't an effect on iron removal. But, the effect is only noted on the specific power consumption.

Key words: Electrodialysis; Brackish water; iron removal; Process efficiency**.**

Electrochemical and spectral studies of auto-assembled arrays of calix[4]arenequinhydrone charge-transfer complex on indium–tin oxide (ITO) glass

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A sensing materiel based on calix[4]arene molecules is electrochemically deposited on ITO electrode coated. A brown film was electrodeposited at a potential E_{imp} = –1.00 V versus SCE in acetonitrile solvent, however in dichloromethane solvent, a bluish film autoassembled on the ITO electrode coated at a potential E_{imp} = -0.65 V versus SCE. Both films are subsequently analyzed by cyclic voltammetry and UV–Vis spectroscopy.

This investigation shows that in acetonitrile solvent, the charge-transfer complex, calix[4]arenequinhydrone was formed in electrolytic solution and it was not self-assembled on the ITO electrode. The related UV–Vis spectrum shows a single absorption band towards a wavelength about 350 nm. The optical behaviour of the blue film shows two absorption bands: the first one appears on the first absorption band of the acceptor at 305 nm and the second one in the visible range at 502 nm. The band situated in the visible range correspond to a well-defined charge-transfer band indicating the presence of the charge-transfer complex, the calix[4]arenequinhydrone.

EVALUATION OF THE USE OF SUNFLOWER LEAVES AS POTENTIAL SORBENT FOR ACID DYE REMOVAL FROM SYNTHETIC AQUEOUS SOLUTION

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Synthetic dyes are extensively used for dyeing and printing in industries. Their presence in watercourses is aesthetically unacceptable and may be visible at concentration as low as 1 ppm [1]. Among the various types of dyes, various acidic dyes are used to color nylon, wool, sole in textile industries, paper and leather. This class of dyes is the most problematic, because they tend to pass through conventional treatment systems unaffected [2]. Although different physical and / or chemical treatment methods are efficient in treating a dye wastewater, they are quite expensive and have operational problems [3]. As a result, there is a search for low-cost, naturally occurring, abundant sorbent materials that can serve as viable alternatives to activated carbon used in adsorption process.

This work explores the possibility of using an agricultural waste namely: sunflower leaves, as an inexpensive sorbent for the removal of two acid dyes: bezanyl blue and bezanyl red, as models of acid dyes, from synthetic aqueous solutions. This material is abundantly available in Algeria, hence cost effective sorbent. Dyes sorption equilibrium and kinetics tests were performed at natural pH of solutions in batch conditions. The effect of some parameters such as: contact time, initial dye concentration and dye type on dye sorption kinetics, has been studied. Two simplified kinetic models including a pseudo first-order and pseudo second-order models were selected to analyse the dye sorption kinetics. The equilibrium data were analysed using Langmuir and Freundlich models.

Key words: removal, acid dyes, sorption, sunflower leaves, kinetics, equilibrium, modelling.

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COMPARISON OF PHYSICOCHEMICAL AND SORPTION PROPERTIES OF ACTIVATED CARBONS PREPARED BY PHYSICAL AND CHEMICAL ACTIVATION OF *JUJUBA SEEDS* (NEBKA)

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Jujuba seeds (NEBKA) were used as a precursor for the preparation of activated carbons by physical and chemical activation with CO_2, $ZnCl_2$, KOH and mixed activation $ZnCl_2/CO_2$ respectively. The effect of pyrolysis temperature (500 and 800 °C) and activation method on the acid–base character of the surface, textural parameters as well as sorption properties of adsorbents prepared toward gas and liquid impurities was tested. Depending on the method of preparation, the final products were microporous activated carbons of surface area ranging from 35 to 1277 m^2/g and pore volume from 0.015 to 0.61 cm^3/g, with very diverse acid–base character of the surface. The results obtained in our study proved that a proper choice of Jujube seeds (NEBKA) pyrolysis and activation procedure can produce adsorbents with high capacity for different contaminants from the liquid phase.

Key: Jujuba seeds; Physical/chemical activation; Activated carbons; Adsorption, phenols.

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THE ADSORPTION MECHANISM OF ANIONIC AND CATIONIC DYES BY COCONUT SHELL-BASED MICROPOROUS ACTIVATED CARBON

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The preparation of activated carbon from coconut shell with potassium hydroxide activation (CAC) and its ability to remove dyes (methylene blue MB, methyl orange MO and red congo RC), from aqueous solutions were reported in this study. The adsorbent was characterized with Fourier transform infrared spectrophotometer (FT-IR), and scanning electron microscope (SEM). Various physiochemical parameters such as, contact time, initial dye concentration, pH of dye solution and temperature were investigated in a batch-adsorption technique. Result showed that the adsorption of both MB, MO and RC dyes was favorable at basic pH. The adsorption uptake was found to increase with increase in initial dye concentration, and contact and temperature of the system. The chemisorption, intra-particle diffuse, pseudo-first-order and pseudo-second-order kinetic models were applied to test the experimental data. The pseudo-second order exhibited the best fit for the kinetic studies, which indicates that adsorption of (MB, MO and RC) is limited by chemisorption process. The equilibrium data were evaluated using Langmuir, Freundlich, Temkin and Fritz–Schlunder isotherms. The adsorption of dyes in this study onto coconut shell activated carbon is heterogeneous with multi-layers. Thermodynamic parameters such as Gibbs free energy, enthalpy and entropy were determined. It was found that (MB and MO) dye adsorption was spontaneous and endothermic.

Keywords: Activated carbon; Adsorption; dye, Isotherm; Kinetics; Thermodynamics

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Structure and properties of Biodegradable PLA/PCL Blends

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In this study, blends of biodegradable Poly (lactic acid) (PLA) and Polycaprolactone (PCL) were prepared using a twin-screw extruder with a film die to evaluate their structural, thermal and mechanical properties. Morphology of tensile fractured surfaces was examined through Transmission electron microscopy (TEM). PLA/PCL blends exhibited an increase in percentage of elongation and a decrease of strength and modulus, when compared to those of neat PLA. Among the various blends proportions considered in this work, 95/5% and 75/25 PLA/PCL blends exhibited the highest elongation and tensile strength. The Effect of PCL content on glass transition temperature (Tg), melting temperature and degree of crystallinity of PLA/PCL blends was investigated with Differential scanning calorimetry (DSC).

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ELECTROCATALYTIC OXIDATION OF ASCORBIC ACID AT Cu_2O THIN FILM

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Ascorbic acid (AA) or vitamin C is distributed widely in both the plant and animal kingdoms. In vegetable cells, it occurs free from, and also often bound to protein as ascorbigen. Among animal organs, the liver, leukocytes and anterior pituitary lobe show the highest concentrations of ascorbic acid. Vitamin C is also present in many other biological systems and multivitamin preparations, which are commonly used to supplement inadequate dietary intake. Nevertheless, it is widely used in foods as an antioxidant for the stabilization of color and aroma with subsequent extension of the storage time of the products [1].

Due to the important roles of AA in biological bodies [2], extensive research has been underway to develop voltammetric methods for determining AA in biological systems for many years. However, because of its high overpotential, poor reproducibility, low selectivity and poor sensitivity on the conventional electrodes, it is nearly impossible to determine AA by direct oxidation of it on these electrodes. Thus, much interest has been focused on the use of mediators and modified electrodes to catalyse the oxidation of AA.

Therefore, this work aims to study the electrochemical oxidation of ascorbic acid on a Cu_2O thin film obtained by electrodeposition method. The resulted products have been characterized by various microscopic techniques as scanning electron microscopy (SEM), X-ray diffraction (XRD) and electrochemical (chronoamperometry, voltammetry).

It has been found that, the electrodeposited Cu_2O film show higher catalytic activity and stability towards electro oxidation of ascorbic acid.

Key words: Cu_2O; electrocatalytic oxidation; Electrodeposition; Ascorbic acid.

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Preparation of a new chemically bonded phosphate ceramics (CBPC) derived from magnesium silicate

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Chemically bonded phosphate ceramics (CBPCs) are formed like cements, but their structure and properties are similar to ceramics. They are highly crystalline materials and can thus be considered as room temperature-setting ceramics developed for use in structural materials. The hardening is produced through chemical reaction between magnesium oxide and acid phosphates. This material has several characteristics over conventional Portland cements; witch includes very rapid setting, high early strength and hight bonding strength very good durability including frost resistance and wear resistance.

The key to obtain a chemically stable material with super high performance is to control the acid-base neutralization rate during the initial setting and hardening period. The exothermic of this reaction is affected mainly by the reactivity of magnesium oxide witch can be controlled by a prior calcination at 1500°C. The cost arising from the calcination process makes us thinking to substitute the base resource of magnesium by magnesium silicate.

Synthetic magnesium silicate was obtained by precipitation using solutions of $MgSO_4$, $7H_2O$ and Na_2SiO_3. The chemical compositions of the product and their crystalline structures were determined by X-ray diffraction (XRD) and infrared spectroscopy (IR).

The study was undertaken to check the possibility of using the synthetic magnesium silicate as base compound in the preparation of phospho-silicate magnesium cement and to establish the effect of some parameters of these materials on the chemical and mechanical properties of CBPCs in term of microstructure (SEM), mechanical strength and setting time. It was found that the cement prepared with a magnesium silicate calcined at 650°C and with a ratio MgO/SiO_2= 1 showed good compressive strength and the presence of K-struvite ($MgKPO_4$,$6H_2O$) as principal reaction product.

Controlled synthesis and thermal conductivity of TiO_2 nanoparticles via the polyol method

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Titanium dioxide nanoparticles (TiO_2-NPs) with a pure rutile and anatase phase were successfully prepared using a polyol synthesis process using diethylene glycol (DEG) as solvent. The preparation state is controlled via the precursor concentration, the hydrolysis rate and the synthesis time. The powders thus prepared were investigated using X-ray diffractometry, FTIR spectra and the complex impedance spectroscopy. XRD patterns show that TiO_2-NPs were in rutile and anatase phases at 500°C and 800°C, respectively. FT-IR spectra exhibit broad peaks whereas both phases of TiO_2 demonstrate very sharp peaks. Besides, a detailed analysis of the impedance spectrum suggested that the electrical property of the titanium dioxide with a rutile structure was strongly temperature dependent. As results, the conductivity was improved thermally, and the Nyquist plots (Z' versus Z") are well fitted.

Key words: Polyol, TiO_2-NPs, synthesis process, impedance spectroscopy.

NEW ROUTE FOR PLA/PHBV BLENDS' COMPATIBILISATION: EVOLUTION OF THE INTERFACIAL TENSION AND THE MORPHOLOGICAL PROPERTIES

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The environmental concerns related to conventional polymer materials has motivated researchers to work on the development of polymers from renewable resources. Poly(lactic acid) (PLA) properties make it a potential candidate to be used in various markets, such as packaging, textile, and automotive industries. However, despite its high strength and modulus, the inherent brittleness and high permeability significantly limit its applications [1]. Recently, Boufarguine [2] showed that blending PLA with a small amount of poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) using a custom multilayer co-extrusion process, increases both ductility and gas barrier properties of co-extruded films compared with neat PLA. On the other hand, we found in the literature that PLA low molecular weight (Mw <20 000 g / mol) is miscible with molten PHBV over the entire range of composition tested [3]. Hence the idea of adding a small amount of PLA oligomers to play the role of compatibilizer and consequently improves the properties of PLA / PHBV blends. So various blends of PHBV / PLA Low molar mass (PLA Mn = 5000 g/mol) were prepared by solvent casting method to found the interfacial tension and see the impact of the addition of PLA oligomers on the morphology of the extruded mixtures.
Rheological studies were conducted in Anton Paar MCR rheometer using parallel plate geometry. The experiments were performed within the linear viscoelastic regime under the dynamic oscillation mode from 0.01 to 100 Hz at 185°C at a constant strain of 1%. Interfacial tension was measured using retraction of a deformed drop method DDRM [4]. The technique consists of deforming a spherical drop of PHBV and then recording, as a function of time, the relaxation to the equilibrium spherical shape. The interfacial tension is then determined from the viscosities of the two polymers and the dimensions of drops.
A decrease of the interfacial tension between PLA and PHBV after the addition of the compatibilizing agent was observed. In fact, when PLA with a molar mass of 5000 g.mol-1 (1wt% in the PHBV phase) was added to the blend, the interfacial tension decreases from 0.5 mN/m to 0.3 mN/m. SEM observations of the extruded films using single screw extruder have shown that the lamella PHBV are dispersed in the PLA matrix. In fact, the length of PHBV lamellas decreases from 44 µm (for PLA / PHBV film) to 15 µm (for PLA / PHBV / PLA 5000 89/10/1 extruded film). Also, SEM observations of rods using twin screw extruder have shown that PHBV nodules are dispersed in PLA matrix and the size of PHBV nodules decreases from 300 nm to 100 nm by adding 1% of PLA oligomers. Therefore, the compatibilizing effect of PLA oligomers on PLA/PHBV blends has been confirmed.

Key words: PLA,PHBV, oligomers, compatibilization, interfacial tension, morphology

Agriculture crop residues as a source for the production of nanofibrillated cellulose with low energy demand

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Nanofibrillated cellulose (NFC) from three agricultural crop (rice straw, corn and rapeseed stalk) residues was isolated with high-yield production using either high pressure homogenisation or a high speed blender. The fibres were extracted from the neat biomass via an $NaClO_2$/acetic acid and alkali pulping process. TEMPO-mediated oxidation pretreatment at pH 7 and 10 was accomplished to facilitate the release of the cellulose microfibrils. The fibrillation yield, transparency degree and morphological characteristics of the ensuing NFC were analysed using the centrifugation method, transmittance measurement and SEM observation. The energy consumption during the disintegration process was also accessed. It was shown that the mode of lignin removal and the fibre pretreatment notably affected the nanofibrillation efficiency and energy demand. A successful production of NFC with yield exceeding 90 %, using a simple Waring blender, was achieved when the $NaClO_2$/acetic acid delignification followed by a TEMPONaBr–NaClO oxidation at pH 10 was adopted.

Key words: Nanofibrillated cellulose _ Homogenisation _ Agricultural crops _ Rice _ Rapeseed _ Corn stalk

STUDY AND COMPATIBILISATION OF BIOPOLYMERS MIXTURES (PLA / PHBV)

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Recently, the use of renewable materials has seen increasing attention in the academic and industrial world. Among a few commercially available bio-based thermoplastic polymers, poly(lactic acid) has undergone the most investigation. In fact, PLA can be used in various markets, such as packaging, textile, and automotive industries. Despite its numerous advantages such as high strength and high modulus, the inherent brittleness significantly impedes its wide applications in many fields [1]. Therefore, PLA is often toughened by blending with other polymers.

The purpose of this study was to explore a new way of compatibilization of PLA/PHBV blends in order to improve mechanical properties and gas permeability. A PLA oligomers (Mn=5000 g.mol-1) were used as a compatibilizer. In fact, PLA oligomers (Mn<15000 g.mol-1) were known to be miscible with PHBV [2]. The compatibilizer was obtained using a hydrolysis method of commercial PLA as described by Aressy [3]. Various blends of PLA/PHBV with different rate of compatibilizer, obtained by single screw extrusion, were characterized.

The interfacial tension, measured by deformed drop method DDRM [4], decrease after the addition of PLA oligomers (PLA5000) to PHBV. Indeed, the interfacial tension between PLA and PHBV decreased from 0.5 mN / m to 0.3 mN /m with the addition of 1% of PLA5000. Tensile tests have shown an improvement in the elongation at break. It increased from 3% for the PLA/PHBV films to 15% for the PLA/PHBV film with 1% of compatibilizer. SEM observations of the extruded PLA/PHBV films have shown that the PHBV lamellas are dispersed in the PLA matrix. After the addition of 1% of compatibilizer, the size of the lamellas of PHBV decreases. Finally, after the addition of 1% of compatibilizer, the helium permeability of PLA/ PHBV films showed a decrease of about 33% compared to PLA film.

Different results confirmed that the PLA oligomers act as compatibilizer for PLA/PHBV blends and the addition of 1% of compatibilizer to two brittle polymers gives a ductile film with interesting barrier properties.

Key words: PLA,PHBV, biopolymer, polymer blend, compatibilization, interfacial tension, morphology, permeability

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EFFECT OF SEVERE PLASTIC DEFORMATION ON THE MICROSTRUCTURE AND THERMAL STABILITY OF A NANOCRYSTALLINE ALUMINUM ALLOY

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Nanostructured metals and alloys, with average grain sizes typically smaller than 100 nm, have been the subject of a considerable interest in recent years. The refinement of the grain size and accumulation of crystalline defects following severe plastic deformation (SPD) allow improving the mechanical properties of the deformed metallic materials and alloys. For instance there has been a significant development of SPD techniques used for obtaining ultrafine grain (UFG) metals and alloys. Take, for example, mechanical milling, torsion under high pressure and equal channel angular pressing (ECAP). Coupling different SPD techniques, results to refining the grain size to a higher extent and improving the temperature range wherein the grain size remains in the nano- or submicron domain. In the present work, investigation on the microstructure and thermal stability of SPD powders obtained from the undeformed and ECAP'ed low purity aluminum samples was carried out using XRD, SEM and TEM. The crystallite size, lattice distortions and dislocation density achieved after SPD were determined using XRD line profile analysis. SPD leads to an intensive refinement of the grains to the nanometer domain and enhanced dislocation density. Using ECAP as a previous deformation process to high energy ball milling did not significantly affect the crystallite size obtained after ball milling. However, lattice distortions and dislocation density were highly improved. Accordingly, the crystallite size of the investigated powders remained in the nanometer domain for temperatures as high as 300 °C. This could be attractive for powder metallurgy processes and applications.

Key words: Aluminum, Nanocrystalline, SPD, ECAP, mechanical milling, microstructure, refinement, dislocations, X-ray

STRUCTURAL AND THERMOCHEMICAL STUDY OF STRONTIUM SODIUM PHOSPHATE GLASS

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The physical and structural peculiarities of phosphate glass as well as their low optical dispersion, high refractive index and low glass transition temperature have attracted significant interest at a fundamental level. Their similarities with the mineral part of the bones allow their use as bioactive materials. Moreover strontium oxide is a component of many of those materials, its presence increases the biological activity and provides a radio-opacity needed for the implants.

The present work focuses on the synthesis, the structural and the thermochemical study of glass compositions in the (50-x) Na_2O -x SrO- 50 P_2O_5 system with x ranging from 5 to 50. In order to understand the role of strontium oxide on the structure and the thermochemical properties of metaphosphate glasses. The structural study was conducted by X ray diffraction, the density of the glass was measured with Archimedes' method, glass transition and crystallization temperatures were determined using differential scanning calorimetry, infrared spectroscopy and Raman spectroscopy was used to show the potential changes in the vitreous network and the NMR MAS ^{31}P measurements were conducted to see the variation in isotropic chemical shift of metaphosphate (Q^2) species. The thermochemical study was carried out in a Tian-Calvet microcalorimeter, the Setaram C80.

The glasses were synthesized with the meld-quenching technique using the stoechiometric amount of strontium carbonate, sodium hydrogenophosphate monohydrate and phosphoric acid. The batch was then poured into preheated graphite mold and kept for a night at 150°C.

The X-ray diffraction patterns of samples confirm the amorphous states of all the studied compositions. The density increases when sodium is replaced by strontium. The DSC measurements show the rise of glass transition and crystallization temperatures revealing the increase in the rigidity of the vitreous network. The infrared and Raman spectra shows the constancy of the metaphosphate chains for all the compositions and the NMR MAS ^{31}P results reveal the shielding of Q^2 phosphate species when the amount of strontium oxide increases.

The dissolution enthalpy varies from endothermic to exothermic values when the amount of strontium exceeds 20%. This unexpected variation could be reported to the coordination of strontium and sodium in the glassy matrix.

Key words: Strontium phosphate glass, Microcalorimetric analysis, Structural investigations.

SYNTHESIS AND ANTIBACTERIAL ACTIVITIES OF NEW PERFLUOROALKYL *N,N*-DISUBSTITUTED THIOUREAS

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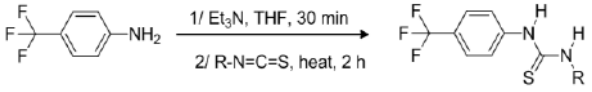
Thioureas are valuable functional groups in organic chemistry, and have found a wide use in bioconjugate [1] and heterocyclic chemistry [2]. They are used in agriculture as pesticides and herbicides with well established insecticidal and herbicidal properties [3]. In addition, thiourea derivatives have high importance in medicinal chemistry as many of them are known for their interesting pharmacological properties as antibacterial, anti-fungal, anti-tubercular, anti-HIV, anti-thyroid, anti-helmintic and rodenticidal agents [4,5]. This has inspired much effort to develop efficient routes for the synthesis of this class of compounds and the most common methods involve the condensation of amines with isothiocyanates. As a continuation of our interest in the reactivity of the latter compounds [6], we describe in the present work the synthesis of new fluorinated thioureas from isothiocyanates and *4*-trifluoromethylphenylamine. These trifluomethylated thioureas were obtained in good yields and characterized using spectroscopic IR, NMR and HRMS techniques. The antibacterial activity of some of these compounds against various Gram-positive and Gram-negative bacterial strains is also presented. The possible use of such compounds as bioactive molecules as well as thermal energy storage materials will be discussed.



1-9

R= Ph; NO_2-C_6H_5; CF_3O-C_6H_5; Pyridyl; Bn; Et; Bu; Allyl; Cyclohexyl

83%-92%

Keywords: isothiocyanates, fluorinated thioureas, antibacterial activities, NMR.

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THE ADSORPTION BEHAVIOR OF POLLUANT CD (II) FROM AQUEOUS SOLUTION USING SMECTITE CLAY FRACTION

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The aim of this study is to assess the effects of some operating factors on the cadmium (II) retention by a smectite clay fraction. For this purpose, adsorption experiments were carried out using batch process. The smectite clay fraction was characterized by different methods infrared spectroscopy, XRD, x -ray fluorescence, measure of Cation Exchange Capacity (CEC) and specific area. The adsorption of the ions of cadmium shows that the equilibrium was attained only after 240min with a rate of 79,4 % adsorption. The results show that the kinetics obeys a pseudo-first order reaction. On the other hand, it is found that cadmium (II) maximum uptake depends on the contact time, temperature, agitation speed and pH, which was regarded as a principal factor in the analysis of the adsorption process. The uptake of Cd (II) increases with the pH increasing. The raise of the temperature influences negatively the process of the adsorption, what shows the phenomenon of the physisorption of cadmium ion on the material tested. The studies of the retention of cadmium ion showed that it is mainly influenced by the physico-chemical properties of used clay.

Key words: clay, bentonite, cadmium (II), metals, Adsorption,

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ANALYTICAL METHOD FOR THE DETERMINATION OF THE SPECIFIC MIGRATION OF PHTHALATES PLASTICIZERS PRESENT IN PACKAGING MATERIALS FOR FATTY FOOD

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The Regulation (EU) N°10/2011 limits the migration of four phthalates: dibutyl phthalate (DBP), di-2-ehtylhexyl phthalate (DEHP), butyl benzyl phthalate (BBP) and di-isononyl phthalate (DNP) to a level of less than 0.3, 1.5, 30 and 60 $mg.Kg^{-1}$ respectively in plastic food contact materials [1].

As a result of this regulation, and in order to verify the conformity of packaging materials, it's necessary to monitor the presence and the amount of these specific compounds. To evaluate the migration of phthalates into fatty and oily foods, vegetable oil was used as a simulant. The different plastic materials (flexible, film …) have been into contact with a vegetable oil (according to EN13130-01 standard). After contact period, the analytes were extracted by acetonitrile. The extraction was conducted using a SPE with C18 cartridge. After evaporation, 1 mL of acetone was added. The analysis of phthalate was performed by GC/MS chromatography equipped with HP-5MS column. The analysis was conducted in SIM mode. The parameters of analysis were evaluated according to NF V 03-110 standard. The limit of detection and quantification (LD, LQ) and linearity were determined. The LQ was 0.1 $mg.Kg^{-1}$ (DBP, DMP), 0.2 $mg.Kg^{-1}$ (DEHP) and 0.4 $mg.Kg^{-1}$ (BBP). The detection levels are considerably lower than the regulations. A good accuracy and linearity were obtained. The analysis of the samples resulted in detection of phthalate in each of the different plastic materials. However, the phthalate level of all analysis was well below the regulatory limits.

Key words: Phthalates; fatty foods; food-packaging, migration.

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SYNTHESIS AND PHYSICO-CHEMICAL CHARACTERIZATION OF $Na_4VO(PO_4)_2$ COMPOUND: APPLICATION AS A POSITIVE ELECTRODE IN Na-ION BATTERIES

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In search of new electrode for sodium ion battery materials, we therefore focused on the study of new polyanionic materials, particularly the $Na_4VO(PO_4)_2$ material.

Indeed the $Na_4VO(PO_4)_2$ material was synthesized in two steps by the solid route in sealed tubes [1]. This phase crystallizes in the orthorhombic system (space group $Pbc2_1$) and has a one-dimensional structure. The structural refinement by the Rietveld method shows good crystallinity of the material, confirming the adequacy of the structural model to describe the structure $Na_4VO(PO_4)_2$ with the lattice parameters: a = 15.449Å, b=14.827Å, c = 7.0180Å. Sodium assay by atomic absorption spectroscopy confirmed the stoichiometry and sodium in the experimental formula is $Na_{4,027}VO(PO_4)_2$. The study conducted between magnetism 0 - 350K showed a paramagnetic character of vanadium with μeff = 1.74 corresponding to spin V4+. According to thermal analysis under inert atmosphere (N2), this phase is stable up to 700°C. The electrochemical properties of $Na_4VO(PO_4)_2$ was studied as a positive electrode in a battery sodium. The first results show the possibility of extracting a sodium by a two-phase process in redox potential of 3.4V. However, a large irreversibility is observed due to the large size of the particles.

Key words: Solid state, $Na_4VO(PO_4)_2$, Sodium-ion battery, Cathode.

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TOWARDS ELECTROCHEMICAL SENSORS BASED ON CONDUCTING POLYMERS AND CARBON NANOFIBER COMPOSITES MODIFIED CARBON PASTE ELECTRODE

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During the last decades, different approaches have been used to develop high-performance electrochemical sensors based on conducting polymers [1] and carbon structure, such as carbon paste, carbon nanotube, carbon nanofibers (CNF). The combination of carbon materials with polymers is expected to improve the properties of these materials for different applications [2]. The oxidative electropolymerization of diaminonaphthalene derivatives in a simple way and reproducible one-step procedure into carbon paste electrode modified with carbon nanofiber lead to a conducting polymer that can be applied for chemicals sensing. The aim of this work is to report some applications related to polydiaminonaphthalene [poly (DAN)] prepared on carbon paste electrode and carbon nanofiber in the presence of Nickel Nanoparticles (NiNPs). The strategies applied to improve the property of conducting polymer obtained are discussed. The dispersion, morphology, conductivity and structure of the modified electrode are characterized by scanning electron microscopy, cyclic voltammetry, and electrochemical impedance spectra. The electrochemical characterization of the film modified carbon paste electrode (CPE/CNF/Poly(1,5-DAN)/NiNPs) exhibits redox behavior of Nickel Nanoparticles (NiNPs) in alkaline medium. It has been shown that the modified electrode improves efficiency of the electrocatalytic oxidation of methanol and has a good selectivity toward different ions like Mercury or Lead.

Keywords: Carbon Paste Electrode, Poly(1,5-Diaminonaphthalene), Electropolymerization, Carbon Nanofiber, Nickel Nanoparticles, Methanol

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Synthesis and characterization of CdS nanoparticles prepared by precipitation in the presence of Span 20 as surfactant

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Cadmium sulfide (CdS) nanoparticles of size 2.8- 56.3 nm have been synthesized using chemical precipitation reaction of $CdCl_2$ and Na_2S in the presence of nonionic surfactant stabilized reverse emulsions. Sorbian monolaurate (Span 20) was used for the stabilization of emulsions. The influence of Span 20 on controlling the size and properties of CdS nanoparticles was studied. The obtained CdS nanoparticles were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), Energy-dispersive x-ray (EDX), UV- visible absorption spectroscopy and X-ray diffraction (XRD).

Keywords: Chemical precipitation, CdS nanoparticles, Span 20

Spectroscopic study of the Iron(III) and Ni(II) azo Schiff base chelates and their corrosion inhibition charactsitics for mild steel in 0.5 M hydrochloric acid

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Two metal chelates with azo Schiff base with general formula $[M(L)(OH)_x(H_2O)_y]H_2O$,where L represents the azo Schiff base and M = Fe(III) and Ni(II) ions were synthesized and investigated on the basis of CHNS elemental analyses, molar conductivity, IR, electronic, mass and EPR spectroscopies. The used investigations exhibited the presence of non-electrolytic nature for both chelates and confirmed the existence of an octahedral geometry for Fe(III) chelate and a square planar structure for Ni(II) chelate. The effect of the azo Schiff base chelates of Ni(II) and Fe(III) ions on the corrosion of mild steel in 0.5M, HCl has been investigated at 30 °C using weight loss method. The obtained results revealed that although these compounds already chelated, they are good inhibitors and that their inhibition efficiency as high as 88% at low concentration 3×10^{-3}M.

Key words: Schiff base, azo Schiff base, corrosion inhibitor, mild steel, hydrochloric acid azo Schiff base chelates.

Application of *Acacia modesta* and *Dalbergia sissoo* gums as green matrix for silver nanoparticles binding.

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A low cost, efficient and eco-friendly method for the synthesis of silver nanoparticles (AgNPs) using gums as reducing agent was performed. The nanoparticles obtained are characterized by UV–visible spectroscopy. The antibacterial activity of prepared nanoparticles was tested against Gram negative and Gram positive bacteria. The *in vitro* toxicity was evaluated by performing hemolytic analysis. The mutagenic activity was evaluated by using Ames test. The data suggests that the prepared nanoparticles posses high antibacterial potential and also inhibited the biofilm formation. Under the conditions of this study, it is concluded that the prepared silver nanoparticles were non-toxic and non-mutagenic and posses pharmaceutical applications.

Key words: silver nanoparticles, mutagenic activity, Antibacterial

ELECTROCHEMICAL DEGRADATION OF FERULIC ACID ON A BDD ANODE

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The electrochemical oxidation of ferulic acid (FA) (4-hydroxy-3-methoxy-cinnamic acid), one of the most representative polyphenolic compounds present in olive oil mill wastewater, was studied by galvanostatic electrolysis using boron-doped diamond (BDD) as anode. The influence of several operating parameters, such as applied current density, initial ferulic acid concentration, temperature, flow rate, effect of the NaCl concentration and initial pH value, was investigated. UV spectroscopy and chemical oxygen demand measurements were conducted to study the reaction kinetics of ferulic acid mineralization in the presence of Na_2SO_4 (0.05 M) as conductive electrolyte.

The experimental results showed that the electrochemical process was suitable for almost completely removing COD, due to the production of hydroxyl radicals on the diamond surface. In particular, the COD removal follows a pseudo first-order kinetics and the apparent rate constant increased with flow rate and temperature, while it is almost unaffected by applied current and pH value. The influence of chloride ions on the oxidation kinetics was also evaluated and it was found that the electrooxidation kinetics, current efficiency and energy consumption are much more sensitive to the presence of chloride. Under optimal experimental conditions of flow-rate (300 L h^{-1}), temperature (20 °C), current (0.2A) and supporting electrolyte Na_2SO_4 50 mM and 2g/L of NaCl, 99.99% of COD was removed during 3 h electrolysis, with 7.62 $kWhm^{-3}$ energy consumption.

Keywords: Ferulic acid, Polyphenol, Wastewater, Electrochemical degradation, Boron-doped diamond.

Microwave-Assisted Palladium-Catalyzed *C*-Allylation of β-Enaminones

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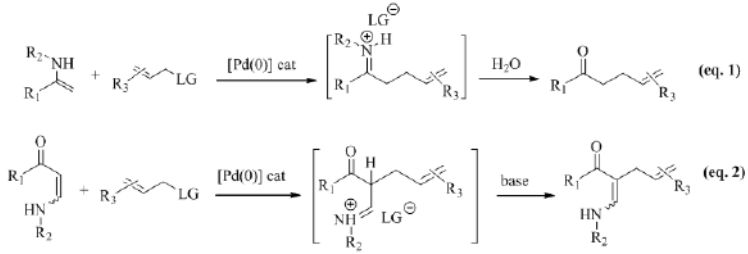
Palladium-catalyzed transformations have gained an important place in the toolbox of synthetic chemists.[1] During the last decades, the Tsuji-Trost allylation arose as a versatile method for the functionalization of a wide array of scaffolds. In particular, enamines have been amongst the first nucleophiles shown to smoothly react with a η^3-allylpalladium complex, as reports by Tsuji and co-workers in their seminal paper.[2] Indeed, such structures can be efficiently allylated at carbon, leading to the corresponding γ,δ-unsaturated carbonyls after hydrolysis of the iminium ion intermediate (Eq. 1).[3] On the other hand, *C*-allylation of enamines with conservation of the nitrogen atom is a hitherto unsolved and challenging transformation,[4] of high potential interest for the synthesis of nitrogen-containing frameworks. To reach this goal, and in keeping with our ongoing interest in η^3-allylpalladium chemistry, we report, in the present study, a new protocol of allylation of β-enaminones, wherein the enamine function is preserved (Eq. 2).



Scheme: Pd-mediated allylation of enamine derivatives.

Key words: enaminone, palladium, allylation, allyl acetate, microwave-irradiation.

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Judd–Ofelt analysis of spectroscopic properties of Eu^{3+} doped $KLa(PO_3)_4$

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Based on Judd-Ofelt (J-O) theory, the intensity parameters (Ω_2 and Ω_4) have been evaluated from the emission spectra of $KLa_{(1-x)}Eu_x(PO_3)_4$ (where x = 2; 5; 10; 15; 20 and 30 mol%) polycrystalline powders as a function of Eu^{3+} ion concentration. The registered trend ($\Omega_2 < \Omega_4$) reveal the ionic character of the La(Eu)–O bonds as well as a relatively high symmetry environment around the rare earth ion (RE) site in $KLa(PO_3)_4$ as compared to other host matrix. From these intensity parameters various radiative properties like spontaneous emission probability (A), branching ratio (β_R), stimulated emission cross-section (σ) and the radiative lifetime (τ_R) have been calculated. The decay profiles for the $^5D_0$ excited level of Eu^{3+} in $KLa(PO_3)_4$ have been found to be single exponential and independent of Eu^{3+} ion concentration even at 30 mol%. In comparison with other Eu^{3+}-doped materials, $KLa(PO_3)_4$: Eu^{3+} has a long lifetime, low multiphonon relaxation rates (W_{NR}), high quantum efficiency (η) and better optical gain parameter ($\sigma \times \tau_{exp}$). The determined radiative properties reveal the usefulness of $KLa_{(1-x)}Eu_x(PO_3)_4$ in developing visible red lasers as well as optical display devices at around 613 nm. Our results were discussed as a function of Eu^{3+} concentration, crystal structure of $KLa(PO_3)_4$ and particle-size distributions.

Key words: $KLa(PO_3)_4$, Eu^{3+} ions, Photoluminescence, Judd-Ofelt analysis, Optical properties, Radiative lifetime.

Preparation and characterization of porous catalysts: WO_3-Ti-PILC

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Nowadays, pillared clays (PILCs) are described as a class of porous materials like zeolites used for the catalysis of several chemical reactions. The use of TiO_2, as a catalyst support of metal transition, is limited by its small surface area, low porosity and modest resistance to sintering. It is well known that WO_3 supported on TiO_2 is one of the successful industrial catalysts. Unlike TiO_2, Titanium pillared clay (Ti-PILC) offer a better alternative in term of surface area, porosity, thermal stability, an adequate acidity as well as its abundance with low cost. In this context, considerable efforts have been made to investigate the substitution of conventional TiO_2 by Ti-PILC support in order to design a brand-new highly-efficient pillared clays catalysts. So far, there has not been any published work describing the preparation of W-Ti-PILC or studying the physicochemical properties of these solids.

In this work, we present attempts for a new preparation method of tungsten supported on Titanium pillared clay issued from different Ti-precursors, characterization of these solids by different technique and investigation of their catalytic behavior in chemical reaction requiring surface acidity of the catalysts. The Ti-PILC support of tungsten was prepared by hydrolysis of mineral and organic Titanium precursors: $TiCl_4$, $Ti(OCH_3)_4$ and $Ti(OC_2H_5)_4$ by HCl. The effect of tungsten amount and the Ti-sources on the physicochemical properties of the catalysts was investigated. The textural characterization indicates very large surface areas and mono modal pore size distribution with pore sizes in the range 27-50 Å. The results show that the specific surface areas, the micro- and meso- porosity as well as the pillar length are mainly influenced by the Ti-sources: organic or mineral precursor and the tungsten amount. Upon calcinations at high temperature, the hydrated polycations dehydrate and dehydroxylate then react to form rigid metal oxide pillars therefore an increase of surface area and microporosity was obtained. The very well Ti-precursor allows the synthesis of W-Ti-PILC with a homogeneous distribution of pillars and good stability at high temperature 400-500°C.

Keywords: WO_3, organic and mineral Ti-precursors, pillared clay, porous catalysts.

Photocatalytic degradation of 4-Nitrophénol with Cu doped Au-ZnO nanoparticles

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Bifunctional Nanoparticles Au-ZnO has several advantages (toxicity low biocompatibility, high chemical stability) and much interest not only for biological and photocataytic applications but also recently for optoelectronic applications**.** Several works reported the results of aromatic compounds degradation by zinc oxide ZnO under UV-visible irradiation.
In this study we reported the synthesis in a polyol medium of Cu doped Au-ZnO nanoparticles ($Au\text{-}Zn_{1-x}Cu_{2x}O$; where x = 0.00, 0.01 and 0.10). The as-prepred products where characterized by X-ray diffraction (XRD), energy spectroscopy (EDAX), transmission electron microscopy (TEM) and Ultraviolet-visible spectroscopy. The X- ray diffraction patterns revealed that the samples are composed by hexagonal wurzite and CFC gold phases. Transmission electron microscopy studies show that the size of ZnO crystallites is in the range of 8-12 nm.The absorption spectra exhibit a sharp absorption edge ≈ 350 nm corresponding to band gap of≈3.1 eV. The photocatalytic activity of the prepared Au-ZnO nanoparticles has been investigated for the degradation of 4-Nitrophénol under solar light. The effect of various parameters was investigated.

Keywords: Nanoparticules, Au-ZnO, polyolmedium, 4-Nitrophénol, photocatalyst, degradation.

The removal of indigo dye from textile waste water by adsorption on natural clay

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ISET - Ksar Hellal

The aim of this work is the removal of indigo dye from textile waste water by adsorption on natural clay. The elimination of indigo was studied as a function of pH, contact time, temperature operation, indigo and clay concentration. The adsorption dynamic modeling shows a good agreement with the first order kinetic model, and that the whole process is controlled by diffusion through the film and inside particular. The thermodynamic study shows the indigo adsorption is dependent on pH and the mass of clay used. Langmuir and Freundlich models fitted well the adsorption of indigo on natural clay. Finally this work contains a comparative study dealing with activated carbon.

Key words: effluents, indigo, adsorption, natural clay.

Synthesis and physicochemical characterization of substituted bioactive glasses

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Bioactive glasses present different rates of biodegradation and mechanical properties depending on their chemical compositions. Thus, our study lies within the scope of a synthesis of pure and substituted bioactive glass, with physicochemical characterization and biological evaluation.
The first studied composition was a pure 46S6 whose wt % composition is 46%SiO_2-24% Na_2O-24%CaO-6%P_2O_5. This sample was used as a reference composition to validate our experimental procedure [1]. In this work, 46S6 was substituted by, 1; 10 and 20 wt % of boron according to the melting method. This chemical element has been chosen because of its high physiological interest and also for improving the mechanical properties. Using DTA, the decrease of the melting temperature of glass is observed; Tf=1250 °C for 46S6, and Tf = 1100 °C for the 20 wt % B_2O_3- substituted BG-B_{20}.
The goal of this study is the control of the bioactivity before and after the substitution of glass by boron with different content. By XRD, the amorphous structure of glasses borosilicate before immersion, and their crystalline structure at the time of the contact bioglass-SBF are detected. By SEM, a change in the pores size and shape of the crystals are observed, which gave us an idea on the kinetics of formation of the layer of silicone gel on the surface of glasses. By FTIR, many modifications in position and intensities of the boron and silicon bands were detected, which explain the destruction of the borosilicate network during the immersion. The perspectives of this work are the biological evolution, and mechanical studies of these borosilicate glasses. Study of the ionic exchanges between the SBF solution and the bioglass will be carried out by the ICP-OES technique. The goal is to follow the evolution of the concentrations of silicon, calcium, and phosphorus released in SBF solution.

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NEW WAY OF SYNTHESIS PYRAZOLOTRIAZINES PHOSPHONATES

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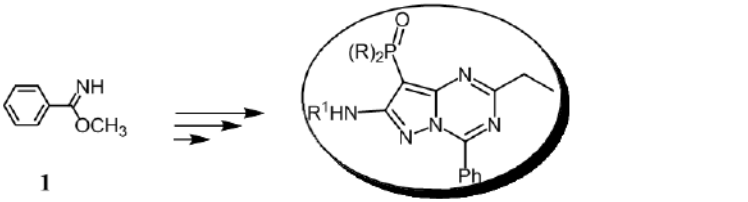
The synthesis of nitrogen-based heterocycles holders of phosphorylated groups is one of the objectives of our laboratory [1]. These compounds have been extensively studied because of their broad spectrum of chemotherapeutic activity and anti-inflammatory, anticancer, antimicrobial and antifungals. The pyrazolotriazines are found in some biologically actives molecules as chemotherapeutic agents against cancer and [2] inhibitors of protein kinase CK2 [3].
In this context to extend the range of heterocyclic synthesized in our laboratory we propose a new strategy for the synthesis of phosphonated pyrazolotriazines from the imidate **1**.
The structures of all the newly prepared compounds were confirmed by ^{1}H, ^{13}C NMR spectral data, and elemental analysis.



Key words: Heterocyclic compounds, Pyrazolotriazines, imidate.

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Eco-Friendly chemical routes for the transformation of terpenoids in green solvents to effective additive materials by using ruthenium complexes

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New terpenoids were prepared by ruthenium catalysed cross-metathesis of terpenes **1a-c** with methyl acrylate **2** under environmentally friendly conditions in glycerol. The new terpenoids compounds were fully characterized by FT-IR, ^{1}H, ^{13}C spectroscopies, and by HRMS analysis. Furthmore, the synthesis of some diazecin-3-ones is described via reaction of compound **4** with hydrazine hydrate or phenyl hydrazine. The new compounds **5** were characterized by FT-IR, HRMS analysis and NMR spectral data.

Keywords: terpenoids, ruthenium, cross-metathesis, diazecin-3-ones

Synthesis, characterization, antibacterial, antioxidant and anti-inflammatory activities of new 4-Aryl-2-amino-6-(4-hydroxy coumarin-3-yl) pyridine-3-carbonitriles derivatives

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This study aimed for the synthesis of 4-Aryl-2-amino-6-(4-hydroxy coumarin-3-yl) pyridine-3-carbonitriles **4** suitable for use as antibacterial, anti-oxidant and anti-inflammatory activities via a versatile, readily accessible 3-((2E)-3(aryl)prop-2-enoyl)-2H-chromen-2-one **3** which were prepared by refluxing 3-acetyl-4-hydroxy coumarin with aromatic aldehydes in chloroform in the presence of a catalytic amount of piperidine**.** Then the direct reaction of the obtained chalcones **3** with malonitrile in the presence of ammonium acetate in one step gave products **5**.The structures of the new compounds **3-4** were confirmed by elemental analysis, IR, and multinuclear/multidimensional NMR spectroscopy (^{1}H, ^{13}C-NMR, NOESY, HMBC) which allowed assignment of the complete network of proton and carbon atoms. Most of the new compounds **4** exhibit moderate antibacterial activity against Gram Negative bacterial strains (E. coli, K. pneumonia, S. typhi and S. flexneri). In addition, the compounds **4** were investigated for anti-oxidant activities by super oxide radical, DPPH (2,2-Diphenyl-1-picrylhydrazyl), and hydroxyl radical scavenging assays, where most of the compounds displayed significant antioxidant activities. Furthermore, these 4-Aryl-2-amino-6-(4-hydroxy coumarin-3-yl) pyridine-3-carbonitriles **4** were evaluated for anti-inflammatory activity by indirect haemolytic and lipoxygenase inhibition assays where compounds revealed good activity.

Key words: 4-Aryl-2-amino-6-(4-hydroxy coumarin-3-yl) pyridine-3-carbonitriles, Antibacterial, Antioxidant, Anti-inflammatory, Synthesis

ELECTRODEPOSITION OF NiO THIN FILMS FOR ELECTROCATALYTIC OXIDATION OF METHANOL

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In recent years, electro-oxidation of small organic molecules has attracted much attention due to the development of direct liquid fuel cells, which require highly reactive fuels with low toxicity and high energy density. Among the several small organic molecules, methanol is one of the most promising fuel because of its low toxicity, abundant availability, low permeability across proton exchange membrane and higher energy density. In the electrochemical oxidation of methanol, the electrode material is an important factor where a highly efficient electrocatalyst is needed. Platinum is the most used metal during the anode construction for direct methanol fuel cells (DMFCs). However, Pt is expensive and its resources are limited, which would affect the future commercialization of DMFCs. Finding a low-cost electrocatalyst for methanol oxidation reaction is an important issue in view of its practical application in clean energy generation systems.
Recently, electrodeposition of semiconductors emerged as a competitive technique for the fabrication of semiconducting thin films [1-2]. In this study NiO thin films are prepared by electrochemical deposition method from aqueous nickel nitrate solution at 55°C on titanium substrate. The effect of several parameters is investigated including the concentration of electrolyte, the electrodeposition time and the electrodeposition potential aiming at maximizing the electrocatalytic activity. The obtained films are systematically tested towards the oxidation of methanol in 1.0 M NaOH solution. It has been found that, the electrochemical oxidation of methanol is greatly increased at the electrode obtained under optimal experimental parameters, indicating that the oxidation of methanol was catalyzed at the Ti/NiO electrode. Scanning electron microscopy images show that the NiO thin films grown under optimal experimental parameters are uniformly and fully covered the substrate. The corresponding X-ray patterns of the formed films exhibit the dominant peaks corresponding to the NiO structures.

Keywords: NiO, Electrodeposition, thin films, electrocatalytic activity, methanol.

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SYNTHESIS AND TRANSITION METAL IONS COMPLEXATION STUDIES OF CALIX[4]ARENES MULTI-FUNCTIONALIZED BY HYDRAZONE AND ACYLHYDRAZONE GROUPS

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In recent years, the importance of monitoring the levels of environmental pollutants in waters and soils, and the need for the recycling of resources and for waste water treatment[1] has generated increasing interest in the designing of new extractants for toxic metals. Toxic heavy metals like copper, mercury, chromium, lead, nickel, and cadmium can have a serious impact on the environment, animals, and humans.[2] Among macrocyclic ligands capable of decontaminating industrial effluents water and nuclear wastes by complexing pollutants, the calixarenes are good candidates. [3, 4, 5, 6]

Calix[n]arenes are an important class of molecules in macrocyclic chemistry. The chapters and the large number of references cited in a recent monograph[7] attest to the wide range of work that has been directed at understanding their properties, especially the interactions in which the calixarene plays the role of ligands (hosts) for metal ions (guests).

Binding capacity is determined by the nature of intermolecular interactions, host pre-organization, solvation effects etc. In this context we interested to calix[4]arene-hydrazone and acylhydrazone derivatives as ligand for transition cations binding. The synthesized ligands were firstly characterized by multinuclear NMR, IR spectroscopy and UV-Vis spectrophotometry. In a second step, the complexation efficiency and selectivity of the synthetized calix[4]arenes derivatives towards the metal cations have been investigated by UV–Vis titration in MeOH to determine both stoichiometries and stability constant values of the complex species formed by using software package that make use of a multiwavelength and multivariate treatment of spectral data.

Key words: calix[4]arene, hydrazone, acylhydrazone, complex, transition metal ions.

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Study of Metal Separation from Wet Process Phosphoric Acid Using Liquid-Liquid Extraction

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The Wet Process for Phosphoric Acid (WPA) production yields an acid that contains several organic as well as mineral impurities. The presence of such impurities is harmful to the quality of the wet process phosphoric acid for its end uses in the fertilizers or food industries. Some metals, contained in the acid such as rare earths, uranium, and vanadium…, could be beneficiated for other uses. The valorization of such metals has become more important in recent years. Many techniques were developed for the separation of these impurities from the WPA. Techniques such as precipitation, adsorption, ion exchange and solvent extraction were used for this purpose. Of these metal separation techniques, liquid-liquid extraction is the most widely used.

The aim of this work is to develop models to simulate metal separation from WPA using liquid-liquid extraction and alternative processes. These models will be used to unfold optimal operating parameters affecting separation processes.

Key words: Wet process phosphoric acid, impurities, separation process, liquid-liquid extraction, optimization

Comparative study of CdS/PS and CdS/PMMA composite films

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Nanocrystalline CdS particles have been dispersed in both polystyrene (PS) and poly-methyl methacrylate matrices in the form of thin composites films by ex situ methodology. These films were successfully prepared by spin coating technical. The structural characterization was performed by X-ray diffraction and Raman spectroscopy. The UV–VIS measurements showed blue shift of the absorption threshold, proving the presence of nanoparticles, but the shift in CdS/PS samples is more than that those of CdS/PMMA composites films. Raman spectra exhibit the longitudinal optical phonon mode (LO) characteristic peak of CdS.

PL spectrum showed that the elaborate samples present bands of luminescence in the visible field from where the possibility of regarding these samples as optically active mediums. We can also show that the photoluminescence bands of CdS/PS films are blue shifted than those of CdS/PMMA composites films, this difference is due to the effect matrix.

Key words: CdS/PS and CdS/PMMA composites films; XRD; Raman spectroscopy; UV-Visible absorption; PL spectroscopy.

Structural and magnetic properties of nano-sized NiCuZn ferrites synthesized by co-precipitation method with ultrasound irradiation

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Sonochemically assisted co-precipitation has been used to prepare nano-sized Ni-Cu-Zn-ferrite powders. One of the motivations of this study is to show if a minimum energy and minimum synthesis duration exists with method assisted by ultrasound in comparison to classical methods.

A suspension of constituent hydroxides was ultrasonically irradiated for various times at different temperatures with high intensity ultrasound radiation using a direct immersion titanium horn. The effects of continued and pulsed ultrasound on obtained powders have been studied in this work. The influences of different parameters (time, temperature, dissipated power) on the structural and magnetic properties are being studied. XRD, FT-IR, BET, TEM and VSM techniques are the toolkits of this work.

The experimental results have shown that the $Ni_{0.4}Cu_{0.2}Zn_{0.4}Fe_2O_4$ nanopowders were composed of a pure spinel phase with the grain size of 10 – 25 nm. The maximum saturation magnetization was observed for the ferrite powder which was synthesized at 90 °C for a synthesis duration of 2 hours with the continued ultrasound mode.

Key words: Sonochemistry; Ferrite; Spinel structure; Magnetic nanoparticle

ANTIOXIDANT ACTIVITY OF DERMATAN SULFATE ISOLATED FROM THE SKIN OF THE RAY: RAJA MONTAGUI

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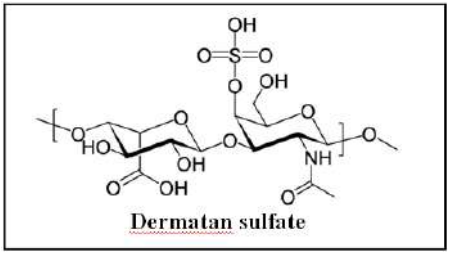
Waste is one of the most alarming problems the planet is currently experiencing. In order to valorize fish waste and find new sources of biomolecules, the dermatan sulfate was isolated from the skin of the ray: *Raja montagui*. Indeed, dermatan sulfate was extracted by papain digestion followed by purification with anion exchange chromatography[1].



The characterization was carried by Fourier-transform infrared spectroscopy (FTIR) and size exclusion chromatography (SEC). Its average molecular weight (Mw) was 39KDa and its sulfate content was 25% w/w.
Preliminary antioxidant tests showed that this dermatan sulfate has the ability to scavenge free radicals and thus act in living organisms as antioxidant.

Key words: fish waste valorization, polysaccharide, dermatan sulfate, SEC, antioxidant activity

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Synthesis, Structure and Photocatalytic Activity of Calcined Mg-Al-Ti- layered double hydroxides

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Mg–Al–Ti layered double hydroxides (LDH) consisting of di-, tri- and tetra-valent cations, with different Al^{3+}/Ti^{4+} ratio have been synthesized by co-precipitation method which were demonstrated as efficient visible-light photocatalysts. The structure and chemical composition of the compound have been characterized by PXRD, FT-IR, SAA, and N_2 adsorption–desorption isotherms were determined. It is found that no hydrotalcites structure have been formed for $Ti^{4+}/(Ti^{4+}+Al^{3+})>0.5$. The calcined sample containing titanium showed relatively high adsorption capacity for methylene blue (MB) as compared to that without titanium. Results show that the pseudo second-order kinetic model and the Langmuir were found to correlate the experimental data well. The photocatalytic activity was evaluated for the degradation of the methylene blue. The photocatalytic activity increases with the increase of the Al/Ti cationic ratio. 71% of the dye could be removed by the Mg/Al/Ti-LDH with the cationic ratio Al/Ti=0:1 and calcined at 500 °C.

Keywords: LDH; Photocatalysis; Methylene blue; Titanium; hydrotalcite; DRX

CATALYTIC OXIDATION OF METHANE OVER SILVER SUPPORTED CATALYSTS

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The catalytic combustion of methane is an important process in industry as it is an alternative route to produce energy without air pollution. In general, palladium based catalysts are regarded as the most active for methane combustion [1-2]. These have been extensively studied for both high and low temperature methane oxidation. In this study, we reported the synthesis and the characterization of silver supported zirconia aerogel catalysts (Ag-ZrO_2) for the complete oxidation of methane.

The Ag-ZrO_2 materials containing various amounts of silver (2, 4 and 6 wt%) were prepared by sol gel method then dried under the super-critical conditions of the solvent and calcined at 550 °C. The physicochemical properties of the obtained catalysts were examined by N_2 physisorption at 77 K, XRD, H_2-TPR and infrared spectroscopy. The results showed that all the Ag-ZrO_2 catalysts are classified as mesoporous materials and exhibit high surface area and pore volume. On the other hand, these materials develop the diffraction peaks of tetragonal and monoclinic phase of ZrO_2. It was also shown that the Ag/ZrO_2 catalysts are active in the total oxidation of methane, the CH_4 conversion increases with the increase of the amount of silver. The high catalytic activity is obtained in the case of the sample containing 6 % Ag.

Key words: Silver, Zirconia, CH_4 oxidation

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Production of activated carbon from peach stones by physical activation

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In this work activated carbon was prepared from peach stones by physical activation with steam. AC was prepared using factorial design as a statistical tool to facilitate the process and to reach results that would not be possible changing one variable at a time. The retained factors are the activation temperature, time and the temperature of steam. For each labored AC, the experiments of methylene blue adsorption are performed in batch mode.
According to the planification results the most performed AC is which prepared under steam, at activation temperature equal to 800◦C for 4h and with steam temperature equal to 50◦C.

Key words: Peach stones, Activated carbon, physical activation, adsorption.

CADMIUM AND ZINC REMOVAL FROM WATER BY POLYELECTROLYTE ENHANCED ULTRAFILTRATION

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The efficiency of two metal ions (cadmium, zinc) removal from aqueous solutions by ultrafiltration (UF) and Polymer Enhanced Ultrafiltration (PEUF) processes were investigated in this work.
The UF and PEUF studies were carried out using an ultrafiltration tangential cell system equipped with 5.000 MWCO regenerated cellulose. A water-soluble polymer: the polyacrylic acid (PAA) was used as complexant for PEUF experiments. The effects of transmembrane pressure, pH, metal ions and loading ratio on permeate fluxes and metal ions removals were evaluated. In UF process, permeate fluxes increase linearly with increasing pH for different transmembrane pressure, which may be the consequence of the formation of soluble metal hydroxyl complexes in the aqueous phase. In PEUF process, above pH 5.0, the Cd(II) retention reaches a plateau at 90% and Zn(II) at 80% for L = 5. Also, cadmium retention at different L is greater than zinc retention at pH varying from 5.0 to 9.0. In a mixture solution, cadmium retention is higher than zinc for different loading ratio; this is due to interactions between carboxylic groups of PAA and metal ions and more important with cadmium ions.

Key words: cadmium; zinc; polymer enhanced ultrafiltration; removal efficiency; ultrafiltration.

PREPARATION AND CRYSTAL STRUCTURE OF DI-BENZYL-AMMONIUM SELENITE MONOHYDRATE †

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This study focuses on the di-benzyl-ammonium selenite monohydrate and their crystal structures. The solution obtained to a slow evaporation at laboratory temperature for a few days until the formation of transportation single crystals of parallelepiped shape.

The X-ray structural analysis of this compound has been carried in Faculty of Sciences of Sfax. The substance crystallizes at room temperature in the triclinic space group P-1, a=6.7851(5)Å, b=10.1401(5)Å, ***c***=12.5334(5)Å; V=823.6(8) $Å^3$, Z=2, R=0.0381 for 14694 observed reflections. Examination of the structure of the compound shows (Fig.1.) an arrangement perpendicular to the direction [001] layers. Plans minerals groups alternate with organic groups. These plans are linked together by hydrogen bonds bridge type N - H ... O. The water molecules are linked to selenite groups by hydrogen bonds weak O – H ... O

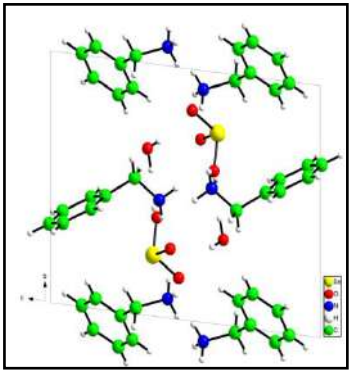

Fig.1. *Projection to bc plane of* $(C_6H_5CH_2NH_3)_2SeO_3H_2O$.

Key Words: di-benzyl-ammonium; selenite; monohydrate; crystal structure.

† : article soumis à "**Journal of Advances in Chemistry (JAC) ".**

VALORIZATION OF ALUMINUM SCRAP INTO GAMMA ALUMINA

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Gamma alumina (γ-Al_2O_3) has been widely recognized as adsorbent and catalyst or catalytic support for many chemical reactions due to its high surface area and mesoporous properties. Many methods for the synthesis of γ-Al_2O_3 are reported in the literature, mainly involving expensive raw materials and harsh synthetic conditions that include high temperatures and pressures. Taking these into consideration, it is still a challenge to develop a facile and environmentally friendly method for obtaining γ-Al_2O_3 [1]. Thus, the aim of this study was to valorize aluminum scrap (99.8% purity) collected from aluminum workshop into γ-Al_2O_3 by acidic precipitation of sodium aluminate ($NaAlO_2$) obtained via an alkaline attack of the scrap. The $NaAlO_2$ solution was neutralized with 2M HNO_3 at room temperature to pH 6, 7 or 8, resulting in the precipitation of a white gel. Some aliquots of the synthesized gel were aged for different times (24h, 48h, 72h and 92 h) in the mother liquor at room temperature. Following the aging step, the gels were washed until no more nitrate ions were detected in the washings. Finally, they were dried overnight at 80°C and then ground in porcelain mortar. The samples were then calcined for 3h in a muffle furnace at different temperatures 300°C, 400°C, 500°C and 600 °C.

This study showed that there is an alternative way, besides the recycling, to valorize aluminum scrap by its facile transformation to γ-Al_2O_3 using soft synthetic conditions. At room temperature, we proved that the neutralization pH is an important parameter that controls the nature of the precipitated phase. At pH=6, the XRD pattern showed the characteristic peaks of well crystallized bayerite ($Al(OH)_3$). The increase of the neutralization pH to 7 and 8 promote only the precipitation of boehmite (AlOOH). Whatever the neutralization pH, the calcination of the gel at 500 °C for 3h transformed it to γ-Al_2O_3. The spectra ^{27}Al NMR of non-calcined gels exhibit a single peak at 6 ppm attributed to Al nuclei in octahedral coordination. The calcination of the gels at 500 °C reveals a second signal around 68 ppm corresponding to Al nuclei in tetrahedral coordination.

Key words: aluminum scrap, boehmite, bayerite, **γ-**alumina.

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Structure and biodegradation studies of PLA/PCL and PLA/PEG biofilms

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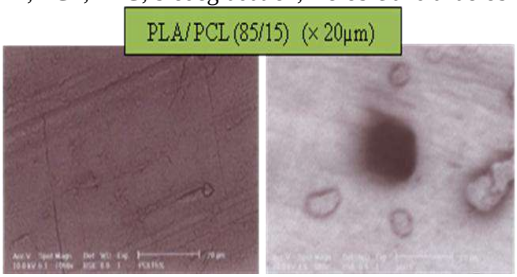
PLA is one of many typical biodegradable and compostable thermoplastic polymers with good mechanical, optical and barrier properties (Wu *et al*.; 2011). However, because of its inherent brittle nature and low thermal stability, PLA needs to be modified to be suitable for use in various applications where mechanical properties are important. There have been a considerable number of studies to toughen PLA with the goal of balancing and increasing tensile strength, impact strength and modulus while retaining their biocompatible and biodegradable nature (Hashima *et al*.; 2010). Several attempts to toughen PLA have included blending with other flexible polymers, such as polycaprolactone (PCL) (Wu *et al*.; 2010). PEG is also an attractive polymer which shows great promise as a plasticizing agent for PLA as it gives a large increase in ultimate elongation with the smallest decrease in tensile strength (Rathi *et al*.; 2011).The objective of the present work is to study the effect of PCL and PEG on the biodegradation properties of PLA. Thus, a comparative study of aerobic and anaerobic biodegradation of PLA, PLA/PCL and PLA/PEG blends has been conducted in the solid state under aerobic conditions and in the liquid phase under both aerobic and anaerobic conditions. To determine the efficiency of the biodegradation of polymers, quantitative (mass variations, oxygen uptake, pressure variations, biogas generation…) and qualitative (variation of $\mathbf{T}_g$ and $\mathbf{T}_f$, SEM studies (Figure 1)) analyses have been made. Otherwise, various fungi grown on solid state used (for their growth) different biopolymers as sole carbon source. The fungi were isolated. In addition, the presence of lipase activity was demonstrated.

Keywords: PLA, PCL, PEG, biodegradation, Aerobic and anaerobic conditions.



Before biodegradation After biodegradation

Figure 1: Figure illustrating the morphological changes of PLA/PCL (85/15) after biodegradation studied in the solid state under aerobic conditions

Synthesis, crystal structure and conductivity studies analysis of semi-conductor $[C_2H_{10}N_2][SnCl(NCS)_2]_2$ compound

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The synthesis and crystal structure for the dichloro diisothiocyanato stannate (II) organic slat. An X-ray has shown that the new compound crystallizes at 298K. in monoclinic, space group $P2_1/n$, Z=2, a= 5.694(10) Å, b=19.886(7) Å, c=8.2591(10) Å and β=108.758(10)°. The structural determination reveals that the adjacent Sn (II) centres are bridged by a pair of SCN^- anions to form a 1-D array giving rise to the anionic chains $\left(SnCl(NCS)_2 \right)_{2n}^{2n-}$. These chains are hemselves interconnected by means of N-H…Cl and N-H…S hydrogen bonds originating from the organic cation $[(NH_3)_2(CH_2)_2]^{2+}$. The complex impedance of compound $[C_2H_{10}N_2][SnCl(NCS)_2]_2$ have been investigated in temperature 293K. The AC conductivity
σ (ω) is governed by the following dependence organic $=A\omega^S$. The value of the exposent S was S=1. This behavor assumes charge carrier happing between the localised states over potential barriers.

Keywords: organic dichloro diisothiocyanato stannate (II), AC conductivity.

Comparative study of heavy metals accumulation in muscle and head of four fish species from the Tunisian coast

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Bioaccumulation of heavy metals in muscle and head of marine organisms has been identified as an indirect measure of the abundance and availability of metals in the marine environment. Monitoring fish muscle contamination therefore serves an important function as an early warning indicator of sediment contamination or related water quality problems and enables appropriate action to be taken in order to protect public health and the environment. In the present work, trace element contents in the muscles and the heads for four fish species, *Diplodus annularis*, *Zosterisessor ophiocephalus*, *Liza aurata* and *Caranx rhonchus*, were determined using atomic absorption spectrometry. Zinc, copper, iron, lead, mercury and cadmium elements exhibited different patterns of distribution among the species and the sex factors. It was evident from our study that, head was the site of maximum accumulation for the elements while muscle was the overall site of least metal accumulation. The element contents were identified to have the following decreasing sequence: Zn>Fe >Cu>Pb>Cd>Hg in both muscles and heads. These values were found to be higher than WHO/FAO maximum recommended limits in food fish in the head but to be lower in all fish muscle. Therefore, consumption of head fish from this area poses toxicological risk public health, but muscle fish were safe.

Keywords: Heavy metals, bioaccumulation, fish muscle, head.

Synthesis, characterization, energy band and optical properties of $YbPO_4$

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Singles crystals of $YbPO_4$ have been synthesized by high temperature solid-state reaction method and characterized by single crystal X-ray diffraction, infrared and Raman spectroscopy. The energy bands structure, density of states and chemical bonds for $YbPO_4$ have been calculated with the density functional theory (DFT). Since there is a strong Coulomb repulsion between the highly localized 4f electrons of rare earth atoms, we present a combination of the GGA and the LDA + U approaches in order to obtain the appropriate results. Furthermore, the linear photon-energy-dependent dielectric functions, conductivity and some optical constants such as refractive index, reflectivity and absorption coefficients have been calculated. The calculated total and partial densities of states indicate that the top of valance band is mainly built upon O-2p states with P-3p states via σ (P-O) interactions, and the low conduction bands mostly originate from Yb-5d states. The results indicated that the energy gap and optical properties are in good agreement with the experimental values.

Keywords: ytterbium monophosphate, Density Functional Theory, Coulomb correction, Electronic structure, Optical properties

LEAD FREE MORPHOTROPIC BOUNDARY SOLID SOLUTION $0.0925KNbO_3$-$0.075K_{0.5}Bi_{0.5}TiO_3$ SYNTHESIZED BY HYDROTHERMAL PROCESS

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The lead zirconate titanate PZT is an excellent piezoelectric compound; it is used in the most common systems. However, because of this toxicity, it is necessary to replace it with lead-free materials to called "environmental friendly". Our laboratory is interested in compounds with perovskite structure and having interesting piezoelectric properties. $KNbO_3$ and $K_{0.5}Bi_{0.5}TiO_3$ are among the most interesting compounds. In previous work, we have studied the solid solutions between these two compounds. Solid solutions of (1-x) $KNbO_3$-$xK_{0.5}Bi_{0.5}TiO_3$ system have an orthorhombic structure for low values of x and tetragonal for x above 0.1. In the morphotropic zone separating the two areas, we achieved a solid solution contains both orthorhombic and tetragonal symmetries. It corresponds to $0.0925KNbO_3$-$0.075K_{0.5}Bi_{0.5}TiO_3$. The morphotropic solution shows excellent piezoelectric and dielectric properties. This solid solution could be a candidate to replace PZT. Our work is to synthesize this solid solution by hydrothermal process. The MPB phase with $0.0925KNbO_3$-$0.075K_{0.5}Bi_{0.5}TiO_3$ solid solution was confirmed by XRD and Raman analysis. Ceramics sintered at 980 ° C has an excellent densification reaching 97%. Morphological study was performed by SEM. Dielectric measurements show that the morphotropic phase exhibits the best dielectric properties relative to all others solid solutions of KN-KBT system. The dielectric permittivity ε_r is about 980 and loss *tanδ* do not exceed 3% at room temperature and 1 kHz.

Key words: MPB solid solution, KN-KBT system, dielectric properties.

SYNTHESIS AND MICROSTRUCTURAL PROPERTIES OF THE MECHANICALLY ALLOYED $Fe_{75}Si_{10}B_{10}Nb_5$ (at.%) POWDER MIXTURE

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Nanostructured $Fe_{75}Si_{10}B_{10}Nb_5$ powders were prepared by mechanical alloying of Fe, Si, B and Nb elements in a high-energy planetary ball mill. The structural properties, morphology changes and local iron environment variations were investigated as a function of milling time (in the 0–100 h range) by means of X-ray diffraction and scanning electron microscopy. At early stage, the dissolution of B and Si atoms into the Fe lattice leads to the formation of bcc Fe(Si,B) solid solution. After milling until 100 h, a mixture of Fe(Si,B) (~ 20 nm), Fe_3B boride phase (~19 nm) and Nb(B) (~10 nm) is obtained. On the other hand, an interesting phenomenon called mechanical recrystallization, related to the formation of a crystalline phase of a partial amorphous powder (obtained at ~ 56 h), was investigated.

Keywords: A. Nanostructures; B. Powder metallurgy; C. X-ray diffraction; D. Recrystallisation.

Nanofibrillated cellulose/poly(methacylic acid-co-maleic acid) nanocomposite aerogel as a novel heavy metal ions adsorbent

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Nanocomposite aerogel based on nanofibrillated cellulose and poly (methacrylic acid-co-maleic acid) (NFC-MAA-MA) was prepared *via* radical polymerization in aqueous solution using Fenton's reagent. The presence of NFC was shown to improve the mechanical resistance of the aerogel and prevent its rapid disintegration. The aerogel was characterized by FTIR and NMR and used as an adsorbent for the removal of heavy metal from aqueous solution. The nanocomposite aerogel showed efficient adsorption exceeding 95% toward Pb^{2+}, Cd^{2+}, Zn^{2+} and Ni^{2+} when their concentration was lower than 10 ppm and ranged from 90-60% for metal concentration higher than 20 ppm. Over 98% of the adsorbed metal ion was recovered using EDTA as a desorbing solution, and the subsequent washing allowed the aerogel to be reused repeatedly without noticeable loss of adsorption capacity. It was concluded that the NFC-MAA-MA nanocomposites aerogel could be used as a high capacity and reusable sorbent materials in heavy metal removing processes.

SYNTHESIS OF FAUJASITE X ZEOLITE FROM WASTE SANITARY PORCELAIN USING ALKALINE FUSION

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Zeolites are aluminosilicate microporous materials with large surface areas containing exchangeable cations within their structure. The zeolite chemical formula is expressed as: $M_m[Al_mSi_nO_{2(m+n)}].xH_2O$, where M is alkali cations electrostatically incorporated into the extra-framework. Zeolites account of the majority of industrial applications such as adsorption, ion-exchange, separation and catalysis. In conventional zeolite synthesis, hydrated silicates, precipitated silica powders and aluminum salts, aluminum wire, metal aluminates are suitable silica and aluminum sources, respectively. Alternatively, zeolites can be also prepared using rather inexpensive starting materials: natural products such as clays, volcanic glasses or low-cost waste product such as ashes (coal fly ash, oil ash and husk ash). Therefore, the aim of this study was to investigate the zeolitization of waste sanitary porcelain. The starting material contains mainly SiO_2 and Al_2O_3 (> 88%) with SiO_2/Al_2O_3 molar ratio of 4.8, which form crystalline phases (quartz and mullite) and amorphous glass. A preliminary study showed the difficulty to dissolve the waste sanitary porcelain into alkaline solution by hydrothermal reaction since most of SiO_2 and Al_2O_3 are in crystalline phases. Accordingly, the alkaline fusion using NaOH followed by a hydrothermal reaction was chosen as technique to convert the porcelain waste into zeolite products. Ten grams of powdered waste (< 150 μm diameter) was grounded with appropriate amounts of NaOH pellets in the weight ratios of 1.0, 1.2, 1.4 1.6 and 2.0. The resultant mixture was then heated in a nickel crucible in air at 550°C for 1 h. The fused material was ground again, dissolved in 85 mL of distilled water and crystallized at 60°C for 24 h. The XRD patterns of the recovered solids after crystallization using NaOH weight ratio of 1.0 showed that no zeolite product was formed and the persistence of the intensities of quartz peaks. However, the increasing the weight ratio of NaOH from 1.2 to 2.0 reduced the intensities of quartz peaks and appeared the new reflexions of faujasite zeolite. FTIR spectroscopy confirms the synthesis of faujasite type X zeolite by the presence of the bands near 1100 and 700 cm^{-1} which are absent in the faujasite type Y.

Key words: waste sanitary porcelain, alkaline fusion, zeolite, faujasite X

REMOVAL OF COPPER IN WASTEWATER IN A FIXED BED REACTOR OF ZINC PARTICLES BY USING CEMENTATION PROCESS

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Cementation is the recovery of an ionized metal from solution by spontaneous electrochemical reduction to its elemental metallic state, with consequent oxidation of a sacrificial metal. The process has been largely used in industry for a long time, not only in hydrometallurgy but also in the purification process of stream and wastewaters [1-3]. The cementation process has several advantages, such as recovery of metals in essentially pure metallic form, simple control requirements and low energy consumption.
The aim of this work is to study the influence of some operating parameters (the initial concentration of copper ions C°, the pH of the solution, the flow of the solution and the initial weight of the sacrificial zinc metal).
The temporal evolution of the concentration at the reactor's outlet has revealed the existence of two kinetic functioning regimes of the reactor, simply after the transition period that is approximately 30 min. the kinetic curve is preceded by a transition step characterized by a sharp concentration diminution (rate enhancement), followed by a linear variations corresponding to the slowed rate. This negative change in the slope can be explained by the blocking effect caused by the deposit, i.e. during cementation, a solid layer of metallic copper is formed at the surface of zinc particles which causes the inhibition of the zinc dissolution and this might result in reducing or blocking the cementation process.
The cementation rate constant, deduced from the slope of straight lines given by the variation of ln C°/C in function of time increases with increasing C° of copper ions. These results are not in conformity with a first-order reaction which normally implies that the rate constant should be independent of the initial concentration C°. These changes in the slope can be related to variations in the electrode surface area due to the copper deposit irregularities and the zinc electrode corrosion.

Keywords: Cementation; Copper; Fixed bed; zinc particles

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The effects of the preparation methods on the physicochemical and catalytic properties of Mo/ZSM-5

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Molybdenum containing zeolites have been widely studied and were often used as redox or even as bifunctional catalysts. Direct incorporation of well-defined MoOx oxo-species inside the microporous network is difficult to achieve while using classical aqueous routes because of the polarity and size issues. In this work we describe other less common preparation methods. These include solid-solid ion exchange (i), organic impregnation (ii) and sublimation (iii) using $MoCl_5$ and NH_4^+-ZSM-5(Si/Al=26) zeolite as starting materials. The issued solids were characterized by means of several techniques (XRD, elemental analysis, NH_3-TPD, H_2-TPR, DRS UV-Vis and DRIFT spectroscopy) and evaluated in the selective ammoxidation of light hydrocarbons into acetonitrile. Results show that molybdenum speciation (localization and geometry) and catalytic properties were governed by the preparation method which seems to be a crucial parameter. Elemental analysis shows that Mo was retained by the zeolite whereas a pronounced weight loss is noted with solid prepared by method (iii). XRD results do not reveal any damage to the zeolite structure or dealumination. NH_3-TPD and DRIFT spectroscopy reveal that Mo loading affects strongly Si-OH-Al sites for solids issued from methods (i) and (iii) leading to the formation of Mo moieties (like $[MoO_4]^{2-}$, $[Mo_2O_7]^{2-}$ and $[Mo_7O_{24}]^{6-}$) in cationic positions having either tetrahedral or octahedral coordination as demonstrated by H_2-TPR and DRS UV-Vis spectroscopy. However in the method (ii), Mo was preferentially grafted on Si-OH groups. From catalytic performances, it was concluded that the solid prepared by method (i) is the most effective catalyst in terms of acetonitrile selectivity and yield.

Key words: Molybdennum, Zeolite, preparation method

Influence of Surfactants on the Physical Properties of Silica Nanoparticles Synthesis via Sol-Gel Method

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Silica nanoparticles (n-SiO_2) have been synthesized using hydrolysis of tetraethoxysilane as starting material and ethanol as solvent by sol-gel method. The effect of different surfactants such as non ionic surfactants sorbitan tristearate (Span 65) and sorbitan trioleate (Span 85) and anionic surfactants Sodium dodecyl sulfate (SDS) and emulsifier T-80 (Tween-80) on morphology and size of the particles was investigated. The role of these surfactants in controlling the size and properties of silica nanoparticles has been discussed. The particles size of silica nanoparticles is controlled also using Ammonia as base catalyst The obtained silica nanoparticles were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), Energy-dispersive x-ray (EDX), UV- visible absorption spectroscopy and X-ray diffraction (XRD). The obtained silica nanoparticles have particles size in the range of 20 nm.

Keywords: Silica nanoparticles, Sol-gel method

PREPARATION AND CHARACTERIZATION OF THE COMPOSITE FILMS BASED ON DATE PITS AND LDPE

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This work is devoted to investigate of an available agricultural Tunisian waste: the date pits as reinforcing filler for thermoplastic matrix. First, the chemical composition of this reinforcing filler was established, and found that when compared to non-wood plants, they contain greater amounts of extractives, lignin, and comparable holocellulose content [1]. Then, they were used to prepare different composites films using brabender mixing device. A series of composite film was established by different loadings of the date pits waste with 10 to 50% of the filler in 10% as an interval. The ensuing composites materials were then characterized by several techniques such as the morphology of the composites, which was investigated using scanning electron microscopy (SEM). The thermal properties of prepared materials were studied using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Finally the mechanical and water absorption properties were involved. The obtained results indicated that date pits based particles enhanced the thermo-mechanical properties of the thermoplastic matrix and demonstrated that this available lignocellulosic biomass can be considered to be a promising filler material.

Keywords: Date pits, Particle-reinforced composites, Extrusion, Dynamic mechanical thermal analysis

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APPLICATION DES NOUVELLES FONCTIONNELLE DFT (MPWB1K, BMK ET M05) DANS LE CALCUL DES CONSTANTES DE VITESSES REACTIONNELLES

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Dans la quête permanente d'une méthodologie efficace de calcul des paramètres thermodynamiques et cinétiques, l'approche basée sur la théorie de la fonctionnelle densité (DFT) est d'une importance capitale.

Dans le cadre de cette étude nous avons évalué à quel niveau de qualité se situe les nouvelles fonctionnelles DFT : MPWB1K, BMK et M05, pour le calcul des constantes de vitesse en considérant la réaction d'abstraction d'atome d'hydrogène $H+NH_3 \rightarrow NH_2 + H_2$ en phase gazeuse. Les réactions d'abstraction d'atomes d'hydrogène sont très importantes dans les processus de combustion, d'oxydation et de pyrolyse. Le calcul des constantes de vitesse k(T) de la réaction $H + NH_3 \rightarrow NH_2 + H_2$ a été effectué en utilisant la théorie de l'état de transition (Transition State Theory (TST)) pour un domaine de température allant de 298 K jusqu'à 2000 K. Pour prendre en compte la correction à l'effet tunnel, nous avons utilisé la théorie de Wigner.

Mots clés: réaction d'abstraction d'hydrogène; DFT ; Etat de transition; constante de vitesse.

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W, N, S-tri- doped TiO_2 for enhanced photocatalytic degradation of Congo Red under visible light irradiation

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A novel high-performance W-S-N tri-doped anatase-TiO_2 was successfully prepared by a simple sol-gel method using tungstic acid, sodium sulfate and urea were as tungsten, sulfur and nitrogen sources, respectively. The physicochemical properties of the photocatalysts were characterized by XRD, BET, TEM, SEM and UV–Vis DRS techniques. The XRD results show that the main crystal phase of samples is anatase. It was also found that the tri-doping of TiO_2 increases its BET specific surface area from 95 to 121 $m^2. g^{-1}$. Besides, it was shown that tri-doping narrows the band gap of TiO_2 effectively, which has greatly improved the photocatalytic activity in the visible light region. The photocatalytic activities of the as prepared photocatalyts were evaluated by degradation of Congo Red (CR) under visible irradiation. Thus, the as-prepared 0.5% W-N-S-TiO_2 heat-treated at 450°C exhibited the highest photoactivity among as-prepared undoped TiO_2, single-doped $WTiO_2$, codoped samples ($WNTiO_2$ and $WSTiO_2$) and the commercial P25 TiO_2, implying that W, N and S tri-doping produced a synergetic effect. The tri-doped catalyst achieves an excellent photocatalytic performance with k_{app} of 0.089 min^{-1}, which is 4.75 times that of the commercial P25 TiO_2 (k_{app} = 0.0125 min^{-1}). Total organic carbon (TOC) removal of CR was up to 72 % and confirmed its significant mineralization in the presence of 0.5 % $WNSTiO_2$ photocatalyst. This research provides a very effective photocatalyst with visible light response for environmental application.

Keywords: Sol–gel; W-N-S tri-doped TiO_2; Congo Red; Visible light photocatalysis.

TiO_2 nanorods for gas phase photocatalytic applications

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Anatase TiO_2 nanorod photocatalysts were prepared following a two-step procedure, consisting first in the alkaline hydrothermal treatment at 150°C for 24 h of commercial Aeroxide TiO_2 P25, and a subsequent calcination at a temperature between 350°C and 700°C. The final calcination led to the transformation of dried TiO_2 (B) nanotubes into pure anatase TiO_2 nanorods, together with a decrease in surface area as well as with the maintain of both one-dimensional morphology and anatase phase with the temperature increase. In the case of the methanol degradation, an optimum in terms of performances was obtained for TiO_2 nanorods calcined at 500°C, with a surface area of 122 m^2/g. This resulted from balanced physicochemical properties with increasing the temperature, with the increase in TiO_2 crystallinity, beneficial for lowering the recombination rate, and the decrease in surface area, associated to the increase in the anatase crystallite size, that lower both adsorption capacity of TiO_2 nanorods and ability to pro-duce $OH^{\bullet}$ radicals. By contrast, the optimum in terms of conversion or sulfur removal rate was observed for nanorods calcined at 380°C in the more rarely studied photocatalytic oxidation of H_2S, for which the accumulation at the surface of sulfates as ultimate reaction products deactivates the photocatalyst. Indeed, TiO_2 nanorods calcined at a lower temperature of 380°C suffered from a strongly less marked deactivation than the Aeroxide TiO_2 P25 reference and nanorods calcined at lower as well as at higher temperatures. They might take advantage of a higher surface area of 219 m^2/g for overcoming their detrimental lower crystallinity and thus improving their resistance to deactivation, by allowing the storage of larger amounts of poisoning sulfates.

Keywords: TiO_2 nanorods, Gas-phase photocatalysis, Hydrogen sulfide degradation, Methanol degradation

Preparation of porous plates carbonated hydroxyapatite and treatment by acoustic waves

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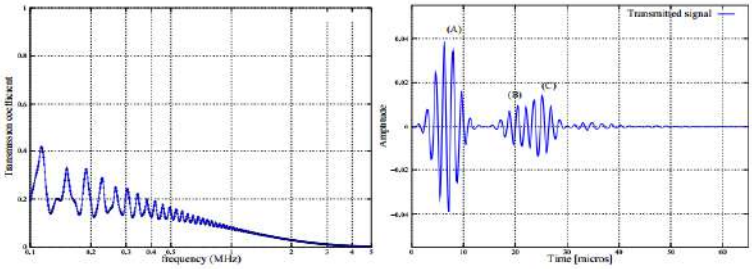
Osteoporosis is a bone disease caused by a biochemical change and which affects the hormonal balance between resorption and deposition of bone. It affects older people and particularly postmenopausal women. It causes micro-architectural deterioration (mainly cancellous bone) leading a weakening of the bones and increases the risk of fractures. The aim of this study is therefore the synthesis of biomaterials such as the carbonate hydroxyapatite (HAC) in order to obtain a synthetic bioactive, biocompatible and biodegradable bone substitute. After having physico-chemical characterization the carbonated hydroxyapatite, we prepared spongy models playing on two parameters: the reinforcement and the porosity. For this purpose, in this work we interested in the detection of the physical properties in (HAC) using ultrasound to study the propagation of the latter in a porous medium. Our results showed that the measuring speed of propagation of sound waves in a porous material at the end to determine the variations of the physical parameters of the medium. For this, Biot theory is used which is the most general model to study the propagation of ultrasound in a porous medium.



Structural, thermodynamic, electric conductivity PEG (300) ratio effect in MgO prepared by chemical precipitation

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In this work, we investigate the properties of magnesium oxide powders synthesized *via* simple chemical precipitation (SPC). The effect of polyethylene glycol 300 (PEG 300) ratio in electrical behavior of MgO is the main part of this study. Crystalline structure and particle size were investigated through X-ray diffraction (XRD). Crystallite size of obtained MgO powders was calculated using Scherer's formula. Fourier transforms infrared spectroscopy (FTIR), Differential Thermal Analysis (DTA) and Gravimetric Analysis (TGA) were used to investigate PEG (300) effect, in fact the mass loss decreases when the PEG (300) ratio increases. Two type of OH group in the MgO srface sites: Type A and Type B with different coordinations, one for the first type and more (2, 3, 4) for the second, were highlighted. The number of OH group decreases when the ratio of MgO/PEG (300) increases. Ratio effect, on dielectric phenomena and electrical conductivity properties of MgO powders, was studied to improve the conductivity. XRD revealed that obtained MgO powders are crystalline and of high purity. Complex impedance spectroscopy was used to examine electrical properties. The activation energies of the MgO powders in the temperature range of study were correlated with PEG (300) ratio.

Keywords : MgO, PEG (300), chemical precipitation, electrical conductivity.

DEVELOPMENT OF HPLC ANALYTICAL METHOD FOR DETERMINATION OF ACETALDEHYDE MIGRATION FROM IN WATER STORED IN POLYETHYLENE TEREPHTALATE (PET) BOTTLES

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For consumer health protection, PET (polyethylene terephthalate) packaging must comply with all European and national legal requirements. The European Regulation N°10/2011 specifically limits migration of acetaldehyde to a level of less than 6 $mg.Kg^{-1}$ [1].

As a result of these regulations, and in order to verify the conformity of packaging water materials, it's necessary to monitor the presence and amount of specific compound. A chromatographic method according to standard NF XP 90-210 are developed and validated using HPLC-DAD instrument. The parameters of analysis were evaluated according to V03-110. The limit of detection, quantification (LD, LQ) and linearity were determined. The level of concentration of acetaldehyde has been determined for the different bottles (0.5L, 1.5L and 2L).

The results show an increase in the acetaldehyde content as a function of storage time at room temperature, 40°C and 60°C. But we obtained a same value for the bottles of 1.5L. These results can be explained by the volume/area ratio.

Migration of acetaldehyde from a PET bottle to bottled drinking water grows in time and temperature. In all the cases the concentrations of acetaldehyde are always considerably lower than the permitted level of migration from PET packaging (according to the European Regulation N°10/2011).

Key words: Migration, PET, acetaldehyde, analytical method

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Effect of ionic liquid anion and cation on the physical properties of ply(vinylidene fluoride)/ ionic liquid

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Poly(vinylidene fluoride), PVDF, has been blended with different ionic liquids (IL) in order to evaluate the effect of the different IL anions and cations on the electroative phase, thermal, mechanical and electrical properties [1], [2] of the polymer blend. C_2MIMCl, C_6MIMCl, $C_{10}MIMCl$, $C_2MIMNTF_2$, $C_6MIMNTF_2$, $C_{10}MIMNTF_2$ have been selected and were introduced in the polymer at a wt% of 40%. It was found that the incorporation of ILs into the PVDF matrix leads to an increase of the β-phase content due to the strong electrostatic interactions between the dipolar moments of PVDF and the ILs [3]. Further, the incorporation of ILs into PVDF strongly decrease the elastic modulus and increases the electrical conductivity of the blend with respect the pure polymer matrix, all these effects being accompanied by a modification of the crystallization kinetics, as indicated by the modified spherulitic microstructure. Therefore, novel PVDF/IL blends films with high transparency, excellent antistatic properties, and highly polar crystal form fraction were successfully achieved.

Key words: PVDF, Ionic Liquid, Electroactive polymers, EAP.

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STATISTICAL CORRELATION ANALYSIS TO MODELING THE VISCOSITY ARRHENIUS TYPE-EQUATION FOR SOME NEWTONIAN FLUIDS

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In transport phenomena, precise knowledge or estimation of fluids properties are necessary, for mass flow and heat transfer computations. Viscosity is one of the important properties which are affected by pressure and temperature. In the present work, based on statistical techniques for non linear regression analysis, we propose a novel equation modeling the relationship between the two parameters of viscosity Arrhenius-type equation, such as the energy (*Ea*) and the pre-exponential factor (*As*). Then, we introduce a third parameter, the Arrhenius temperature (T_A), to enrich the model and the discussion. Empirical validations, using about 90 data set of viscosity of pure solvents studied at different temperature ranges are provided from literature, give excellent statistical correlations, thus allowing us to rewrite the Arrhenius equation using a single parameter instead of two. Then, taking some mathematical considerations, we try in the present work to suggest an empirical exponential law-type equation valid even for the very viscous fluids ($Ea < 40$ kJ·mol^{-1}) and ($-20 < \ln(As$ /Pa·s)). In addition, we have tried to give some physical meaning of the proposed equation parameters. Also, the suggested model is very beneficial for engineering data since it would permit to estimate the missing parameter value, if a well established estimate of the other is readily available.

Key words: Pure liquid; viscosity; Arrhenius activation energy; Correlation; Statistics; Modeling.

SONOGASHIRA COUPLING REACTION OF ARYL HALIDES WITH TERMINAL ALKYNES CATALYZED BY PALLADIUM UNDER ULTRASONIC IRRADIATION UNDER OPTIMIZED CONDITIONS

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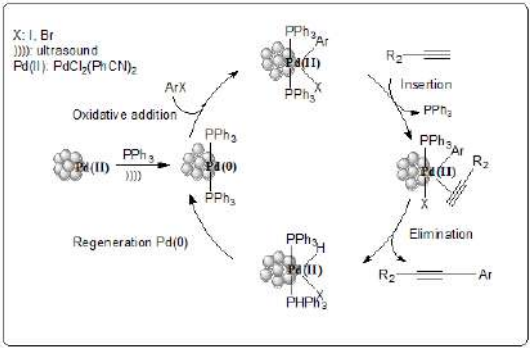
Although much progress against the Sonogashira coupling reaction has been made in recent years, there is still a long way to go to reach the ideal procedure. Many improvements are still needed to fully develop the general coupling procedures that enable good results. In this regard, we studied various terminal alkynes with aryl halides in the presence of palladium complexes under ultrasonic irradiation under optimized conditions at room temperature and in the absence of copper [1]. It has been demonstrated that ultrasonic irradiation increases the reaction rate and improves yields. In this regard, we propose the reaction mechanism of the Sonogashira reaction.



Scheme. Proposed mechanism for the Sonogashira coupling reaction catalysed by Pd (II) under ultrasonic irradiation.

Key words: Sonogashira, terminal alkynes, Palladium, Ultrasonic irradiation.

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THE EFFECT OF ULTRASOUND ON THE YIELDS OF HENRY REACTION UNDER PHASE TRANSFER CATALYSIS

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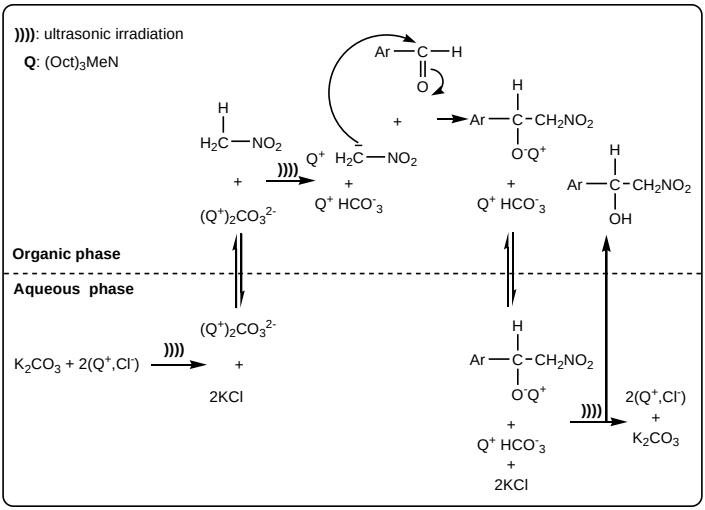
The Henry reaction was successfully performed out by various aldehydes with nitromethane. The biactivation by a catalyst of phase transfert type Aliquat-336 under ultrasonic irradiation improves the reactivity in N, N-Dimethylformamide / Water mixture. Different reaction conditions, such as base,solvent, catalyst of phase transfert and sonochemical activation were studied. It has been demonstrated that ultrasonic irradiation combined with Aliquat-336 increases the reaction rate and improves yields. In this regard, we propose the reaction mechanism of the Henry reaction.

Keywords: Henry, ultrasonic irradiation, Phase Transfer Catalysis, biactivation.



Scheme. Proposed mechanism for the Henry reaction of nitromethane with carbonyl compound under ultrasonic irradiation in the presence of PTC.

Adsorption-desorption of metals onto Tunisian clay through a membrane

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A new method of adsorption-desorption using membrane filtration was used to study the removal of few toxic metals onto the local clay extracted from Oued Tfal south west Tunisia (Gafsa Basin). Indeed, the adsorption and desorption prove, first, the capacity of a clay retain Pb, Zn, Cd and, second, the effectiveness of the amendments to this type of clay, taking into account the variability of factors such as the concentration of metals, pH, temperature, the solution / solid ie the contact surface available (amount of adsorbent or mixture of adsorbents with respect to that of the solution). The local clay, has been characterized. The mineralogical and chemical compositions of the materials have been determined. Heavy metals adsorption kinetics is relatively slow, with a high percentage of total adsorption taking place in less than eighty hours. The equilibrium adsorption data were correlated reasonably well by Langmuir and Freundlich isotherm. The desorption rate of the metal is lower than that of adsorption rate.

Keywords: Adsorption, Desorption, Isotherm, Competitive, Heavy metals.

PREPARATION AND PROPERTIES OF POLYMER BLENDS CHARGED WITH EVA AND PU STEMMING FROM REJECTS OF THE SHOE INDUSTRY

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Plastics are inexpensive, lightweight and durable materials that can be readily moulded into a variety of products and used in a wide range of applications. The production of plastics has increased markedly over the last few decades, which has led to several waste-disposal environmental problems.

Recent research has been interested in the search for effective strategies for the alleviation of these problems. The literature indicates that the best way to limit this plastic waste prevent rubbish from going to landfill is to recycle.

Accordingly, our project aims to study the properties of the polymer blends matrix poly(ethylene-co-vinyl acetate) [EVA], which is an elastomeric material, and the polyurethane [PU] and (ethylene-co-vinyl acetate) [EVA] particle loads stemming from rejects of the shoe industry. The blends were prepared using different concentrations of EVA and PU by a single-screw extrusion.

These polymer mixtures were analyzed by Differential Scanning Calorimerty (DSC), Scanning Electronic Microscopy (SEM), Melt Flow Index (MFI) and tensile test machines.

The results were interpreted in terms of the interfacial tension changes and adhesion at the interface.

PHOTO-OXIDATION BEHAVIOR OF POLYPROPYLENE/ POLYAMIDE-6 BLENDS COMPATIBILIZED WITH PP-G-MA

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Polymer blend degradation is a very complex phenomenon due to the variety of interactions between the blend components. These may generate antagonistic or synergistic effects, depending on the composition, the degradation conditions and the blend miscibility [1]. The processes are more complicated in the presence of compatibilizers [2,3]. In this study, the photo-oxidation behavior of uncompatibilized and compatibilized PP/PA-6 (70/30 wt %) with PP-*g*-MA was investigated using FT-IR spectroscopy, melt flow index (MFI) tester, tensile test, differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). FT-IR analysis of the structure of the compatibilized and uncompatibilized blends after exposure to UV light showed the formation of photo-products corresponding to both components. The MFI and mechanical results obtained revealed that photo-oxidation started primarily in PA-6 rather than PP. In addition, the uncompatibilized blends exhibited a higher degradation rate compared to neat polymers for long exposure time, and the addition of PP-g-MA increased slightly their ageing rate in accordance with TGA data. Further, DSC analysis showed an increase in the crystallinity index and a decrease in the melting temperature of PP and PA-6 after UV exposure either as neat polymers or as blend components. SEM micrographs of the cryo-fractured surfaces of the samples illustrated the formation of cracks and fractures after UV irradiation.

Key words: Artificial weathering; Polymer blend; Compatibilization; Infrared analysis.

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SYNTHESIS OF FUNCTIONALIZED MESOSTRUCTURED SILICA WITH METALLIC NANOPARTICLES

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In this work, functionalized meso-structured silica with metallic nanoparticles (Pt, Pd and Au) was synthesized in a one-step synthetic pathway. The mesoporous silica is obtained by the co-condensation of tetraethoxysilane and hydrophobic organoalkoxysilane using surfactant as pore template. The morphology of mesostructured silica can be controlled by simple varying the amount of added organoalkoxysilane. The metal precursor was incorporated in the porous matrix of MCM-41 in situ, using a hydrophobic solvent as carrier. Metal particles could be detected using transmission electron microscopy which confirming the absence of large metal particles outside the mesoporous silica. The calcined material had a high surface area (800-1200 $m^2.g^{-1}$).

Key words: MCM-41, Au/ MCM-41, Pt/MCM41, Pd/ MCM-41

HETEROGENEOUS SYNTHETIS AND CARACTERISATION OF CELLULOSE TRIACETATE FROM DATE SEEDS CELLULOSE

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Cellulose triacetate was synthesized by the acetylation of microcrystalline cellulose from Deglet Nour date seeds, using acetic anhydride as the as the acetyl donor and acetic acid as the solvent in the presence of sulfuric acid as catalyst. A ripening stage was conducted to have a uniform intermolecular degree of acetyl substitution. The product obtained was characterized through nuclear magnetic resonance spectroscopy (NMR), Fourier transform infrared spectroscopy (FTIR) and differential scanning calorimetry (DSC). FTIR spectrum indicated the presence of acetyl groups and the esterification process. DSC curves showed that the cellulose triacetate obtained had 217 °C as melting point. The frequency of acetyl group substitution in the cellulose was received from the ^{1}H-NMR and ^{13}C-NMR spectra and confirmed the uniform intermolecular acetylation with be 2.8 as degree of acetyl substitution.

Key words: Cellulose triacetate, acetylation, melting point, degree of acetyl substitution.

NEW ROUTE USING GREEN CHEMISTRY FOR A CATALYTIC SYNTHESIS OF SOME OXAZOLINES IN TUNISIAN MATERIALS MIDDLE: SPECTROSCOPIC AND THEORETICAL STUDIES

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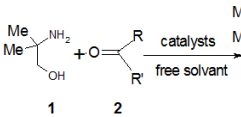
In this work, we describe our finding on reaction of 2-amino-2-methylpropanol **1** with benzaldehyde **2a** and various ketones **2b-g** in the presence Tunisian acid activated clay $\mathbf{H_2}$ without solvent and under autogeneous pressure at different temperature to yield oxazolidines **3a-g**.



2,3	R	R'
a	H	Ph
b	CH_3	Ph
c	CH_3	CH_3
d	CH_3	CH_3CH_2
e	CH_3	$PhCH_2$
f	$PhCH_2$	$PhCH_2$
g	CH_3	$ClCH_2$

The structure of heterocycles **3** were determined par ^{1}H and ^{13}C NMR Spectroscopectrum data and on the other hand the reagents **1** and **2** and products **3** were fully optimized at DFT level of theory with the functional B3LYP and 6-31G (d,p) basis set as minimum on the potential surfaces at the same level of theory. The more stable geometries of these compounds **1-3** have been determined and characterised.

We can conclude that a good agreement was then obtained between the experimental and theoretical results and on the other hand we have advanced a new route of synthesis of oxazolidines using heterogeneous catalysts have been advanced to responds to environmental demands.

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PHOTOCATALYTIC DEGRADATION OF NONYLPHENOL IN THE PRESCENCE OF CE-DOPED ZNO UNDER UV IRRADIATION

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Endocrine disrupting compounds (EDCs) are a diverse group of "chemicals of emerging concern" which have attracted much interest from the research community since the 1990's. They influence processes associated with the endocrine system and alter its functioning [1]. Nonylphenols (NP), a kind of endocrine disruption substances [2], are used widely as emulsifiers, detergents, surface modifiers, flotation and dispersing agents in industry.

The present study reports on the synthesis, the characterisation and the photocatalytic activity of Ce-doped ZnO photocatalysts. The as-synthesized products were characterized by XRD, Nitrogen physisorption (77K), UV-Vis spectroscopy, FTIR spectroscopy, Raman and photoluminescence spectra.

The Ce-doped ZnO catalyst exhibited excellent photocatalytic performance for both NP degradation and mineralization. NP photodegradation obeyed the first-order kinetics according to the Langmuir–Hinshelwood model; the kinetic constants were influenced by the catalyst concentration, the initial NP concentration, H_2O_2 concentration and the solution pH. The possible photodegradation mechanism based on the identification of intermediate products was discussed.

Key words: Nonylphenol, Ce-doped ZnO, Photocatalysis, Photodegradation mechanism.

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Synthesis and characterization of geopolymer cement based on Tunisian kaolinite clay: Alkaline activation effect on calcined kaolin at different temperatures

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In the present work we sought to determine the most convenient calcination temperature of Tunisian kaolin in order to produce a more reactive metakaolin and to determine the most suitable potassium hydroxide (KOH) and sodium hydroxide (NaOH) concentration to produce geopolymer cements with good mechanical performance, less energy consumption and low cabon dioxide emission. This product would play the role of a construction material able to replace the Portland cement. The raw material used in this research is kaolin collected from Tabarka (Tunisia). It was characterized by X-ray diffraction, infrared spectroscopy and thermal analysis. Then kaolin fractions were first calcined at different temperatures ranging from 550 to 1100°C. Then calcined metakaolin were treated by potassium hydroxide and sodium hydroxide. The mechanical and physical properties of hardened samples were characterized respectively by compressive strength, porosity and water absorption. The results obtained showed that the temperature to get a most reactive metakaolin is about 700°C. The various physical properties reached optimum values at this temperature and for base concentration equal to 13 M. These values decreased at higher temperature and concentration.

Key words: Kaolin, Calcination, Metakaolin, Alkaline solution, Geopolymer, Compressive strength

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LE TRYPTOPHANE-METHYLE-ESTER: INVESTIGATION SUR LA STRUCTURE MOLECULAIRE, LE SPECTRE UV, ET LES PROPRIETES THERMODYNAMIQUES

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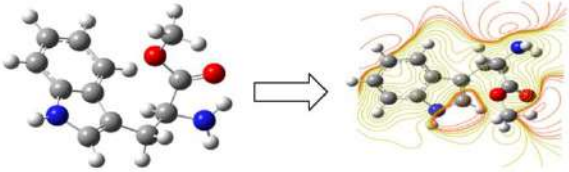
Quelle que soit l'origine des acides aminés [1], ils forment sans aucun doute l'une des plus importantes classes de molécules d'intérêt biologique en tant que molécules libres comme en tant que maillons élémentaires des protéines [2].
En dehors de leur rôle protéinogène, les acides aminés participent à la régulation de nombreuses voies biochimiques intracellulaires. Ce rôle signal a été étudié, surtout, au niveau musculaire où certains acides aminés sont actif sur la promotion de la synthèse protéique [3].



Le tryptophane-méthyle-ester (TrpME) est parmi les acides aminés naturels, apolaire et hydrophobe contenant un hétérocycle indole qui lui confère des propriétés spectroscopiques d'absorption et de fluorescence, cet acide aminé est devenu un objet de curiosité scientifique dans des nombreux domaines de recherche dont la médicine est le plus important. Pour prévoir leur structure moléculaire la plus stable, nous avons effectué une étude théorique, en se basant sur les deux méthodes de calculs ab-initio (Hartree-Fock) et la théorie fonctionnelle de la densité (DFT), avec la base standard 6-31G(d,p) et les trois fonctionnelles B3LYP, BPW91 et SVWN5. Sa géométrie et ses paramètres optimisés sont calculées par la fonctionnelle B3LYP, qui a donnée la structure la plus stable thermodynamiquement, et les trois bases 6-31G(d), 6-31G(d,p) et 6-31+G(d,p).
Nous comparons aussi les valeurs des paramètres géométriques optimisés de différentes formes moléculaire, zwittérionique et cationique de cette molécule, afin que nos simulations soient les plus proches possibles des valeurs expérimentales.
Cette étude est étendue à calculer l'énergie de gap, la polarisabilité, les charges de Mulliken et les propriétés thermodynamiques du TrpME.
Le spectre UV-Vis a été enregistré aussi, et montre un bon accord avec le spectre retrouvé expérimentalement.

Mots clés: Tryptophane-méthyle-ester, ab-initio, DFT, spectre d'absorption, structure moléculaire.

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New method synthesis of O,O-bis-(2,2,2-trifluoroethyl) Alkyl and Arylcarbamoylphosphonothioates and bis-(2,2,2-trifluoroethyl) alkyl and arylcarbamothioylphosphonates

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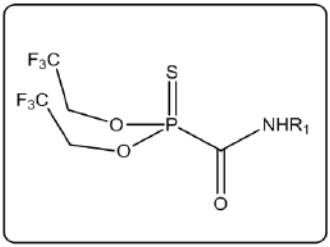
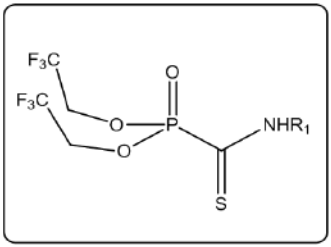
Many studies were reported on the convenient synthesis of carbamoylphosphonothioates [1], [2], [3]. In recent year, we focused our research on the extension of these novel reaction paths for the following reasons: should possess biological activities [2] and their applicability, as conveniently accessible precursors for the preparation of a wide variety deserve significant interest. In the present work, we report the synthesis of a new series of O,O-bis-(2,2,2-trifluoroethyl) alkyl, aryl carbamoylphosphonothioates and bis-(2,2,2-trifluoroethyl) alkyl and aryl carbamothioylphosphonates.
All compounds were characterized on the basis of their IR, NMR Spectroscopy (^{1}H, ^{13}C, ^{19}F and ^{31}P) and in some cases by elemental analysis, HRMS.





Key words: O,O-bis-(2,2,2-trifluoroethyl)carbamoylphosphonothioate, O,O-bis-(2,2,2-trifluoroethyl)carbamoylphosphonothioate.

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ELECTROCHEMICAL STUDIES AND VOLTAMMETRIC DETERMINATION OF TRACE LEAD AT 1,8-DIAMINONAPHTHALENE AND BISMUTH FILM MODIFIED CARBON PASTE ELECTRODE.

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In the last few years, many papers have been devoted to electrochemical sensors based on composite mataerial with conducting polymers for environmental monitoring. The development of electrochemical sensors hold important place compared to other techniques for the benefits of electrochemical techniques in terms of cost, speed and reliability. The aim of this work is the development of a composite material based on carbon paste a electrode doped with 1,8-Diaminonaphthalene (1,8-DAN) and a thin film of Bismuth. The 1,8-DAN mixed with a carbon paste electrode leads to a conducting polymer in acidic medium, the electropolymerization of poly(1,8-DAN) was carried out by using cyclic voltametry in an acidic solution, the modified electrode was characterized by cyclic voltammetry in the presence of the ferri-ferrocyanide and electrochemical impedance. In addition an in situ bismuth film was deposited on the polymer formed on the surface.

A carbon paste electrode (CPE) modified by poly(1,8-DAN) and a bismuth film was then used to determine Pb^{2+} in aqueous solutions using square wave voltammetry (SWV). The analytical application of this electrode towards the detection of metal ions in a real sample such as waste water has been demonstrated.

Key words: Composite material, Polymer, Bismuth Film, Electropolymerization

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MATERIALS AND BIODEGRADABLE BIOSORBENTS BASED LOCAL RESOURCE APPLICATION TO FIXING DYE AQUEOUS SOLUTION.

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Pomegranate peels studied within the frame work constitute a locally abundant and law const resources. The aim of study is to illustrate the valorization of these fruits peels and their use for processing of waste chaste changed in textiles dyes. The purpose of our study is the adsorption of the textile dye BS (Blue Solophenyl GL, CI Direct Blue). The experiments were performed in batch under contact agitation after determining the parameters influencing the phenomena such as the equilibration time, the PH, the mass of adsorbent, the initial concentration of dye.

Key words: Pomegranate peels, biosorption, textile dye, wastewater.

SYNTHESE OF 2-HYDROXYMETHYLAZIRIDINES AS PRECURSORS OF 1,3-OXAZOLIDIN-2-ONES

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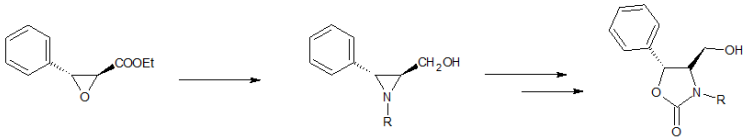
Aziridines have emerged as versatile synthons with numerous applications in the preparation of a wide variety of β-functionalized amino compounds owing to the combination of their easy preparation and their propensity to undergo ring opening reactions with various nucleophiles[1]. In this context and in connection of our interest in the preparation and application of aziridine-2-carboxylates[2] as precursors of azaheterocycles, we describe herein a multi-step strategy for the synthesis of new substituted 1,3-oxazolidin-2-ones. The approach involved a sequence of acylation-cyclization of 3-phenyl-2-hydroxymethylaziridines upon treatment with methyl chloroformate in acetonitrile. These aziridines were prepared in good yields from aziridine-2-carboxylates through reduction with $LiAlH_4$. The structures of all these cyclic carbamates were confirmed by ^1H and ^{13}C-NMR and by ^1H- C HMBC, showing that C_4 and C_5 substituents are situated in trans-position.



trans- β-phenylglycidate *2-Hydroxymethylaziridines* *1,3-oxazolidin-2-ones*

Key words: 2-Hydroxymethylaziridines, 1,3-Oxazolidin-2-ones

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A NEW NANOCOMPOSITE OF PPY/CNFS FOR DETECTION OF TRACE HEAVY METALS

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Room Temperature Ionic Liquids (RTILs) are an exciting class of solvent, receiving much attention in recent years as a replacement for conventional solvents in a wide range of applications. ILs have some unique properties, such as a low vapor pressure, good thermal stability, high polarity, tunable viscosity and an ability to dissolve many compounds, a wide electrochemical window, the ability to dissolve many compounds high conductivity, high heat capacity and solvents available to control reactions.

Nanocomposites based on conducting polymers and carbon nanomaterials have attracted considerable attention and a unique place in nanoscience owing to their exceptional electrical, thermal, chemical and mechanical properties. Polypyrrole (PPy) is the one of most investigated conducting polymers, because of its facile synthesis, good conductivity, redox properties, long-term environmental stability and biocompatibility as polymer in vivo, and has shown promising applications including sensors, photovoltaic/solar cells and corrosion protection etc. Among various carbon materials, one-dimensional carbon nanofibers (CNFs) have attracted more and more attention from researchers as a class of chemically stable supports to enhance the electrical conductivity of metallic oxides as the efficient electrode materials due to their one-dimensional properties, high specific surface area and good electrical conductivities.

In the present work a new nanocomposite of Polypyrrole/ Carbon nanofibers (PPy/CNFs) were synthesized for the first time on the modified gold electrode and exactly employed as an electrochemical sensor to selective detection of Pb(II). The room-temperature ionic liquid 1-ethyl-3-methylimidazolium bis(trifluoromethyl-sulfonyl)imide, [EMIM][NTf_2], was employed as electrolyte in the electrosynthesis of PPy/CNFs. several parameters were studied and optimized, the different morphologies were characterized by scanning electron microscopy.

Keywords: Polypyrrole, Carbon nanofibers, Room Temperature Ionic Liquids, Electrochemical sensor, Lead.

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THERMAL STABILITY OF 99.1% ALUMINUM PROCESSED BY EQUAL CHANNEL ANGULAR PRESSING

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Microstructure evolution of 99.1% aluminum after equal-channel angular extrusion (ECAE) and subsequent heat-treatment was investigated. After deformation the samples were annealed at different temperatures. The deformed and annealed states were characterized by Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), and microhardness tests. The microstructure of ECAE deformed material show the presence of almost parallel and long grains which contain a very high density of dislocations. At this stage, ECAE leads to the formation of sub-grains of about 400 nm in size with many dislocation walls. In addition, dislocation loops were observed within the deformed grains. The microstructural changes observed during annealing were supported by the evolution of microhardness. Continuous grain coarsening at lower temperatures led to a gradual hardness decrease and resembled softening due to recovery prior recrystallization. Thereafter, at annealing temperatures above 200°C, the softening was more rapid. The microstructure obtained after annealing for 1 h at 100°C, contains some arrangement of the dislocations into sub-grain boundaries within 0.5 lm-wide grains. Annealing at 300°C led to the appearance of a duplex microstructure consisting of bands of slightly coarsened grains associated with refined grains. At 400°C, a fully recrystallized structure was observed and no growth of dislocations cells was observed. In XRD measurements, the observed decrease of the peak broadening is attributed to the decrease of the lattice distortions and the increase of the material grain size. In fact, when increasing annealing temperature, an important increase of D is observed parallel to a substantial decrease of ε.

Key words: Aluminum, Equal-channel angular extrusion (ECAE), TEM, XRD.

ELECTROCHEMICAL ANALYSIS OF LEVOFLOXACIN ANTIBIOTIC USING A GLASSY CARBON ELECTRODE

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A simple, rapid and applicable electrochemical method was developed for the detection and quantification of Levofloxacin (LEV) in aqueous solutions. LEV is a broad spectrum antibiotic of the fluoroquinolone drug class. The study was carried out by the cyclic voltammetry method using a glassy carbon disk as working electrode (GCE). Platinum wire and sodium sulfate were used counter electrode and as supporting electrolyte, respectively. Voltammograms obtained showed a well defined irreversible anodic peak which current intensity and potential depend on LEV concentration, potential scan rate, pH and temperature. The peak current increased linearly with the concentration of LEV in the range from 11.25 to 134.99 µmol L^{-1}, the temperature between 20 and 60°C and the potential scan rate from 20 to 200 mV s^{-1}. The maximum current intensity was obtained at pH 5.5. Under optimized conditions, limits of detection and quantification were 2.64 10^{-5} and 8.8 10^{-7} mol.L^{-1}, respectively.

Key words: Antibiotic; Levofloxacin; Cyclic voltammetry; Glassy carbon; Detection; Quantification.

Synthesis and characterisation of mesoporous PAAM-NH_2-MCM-41 adsorbent

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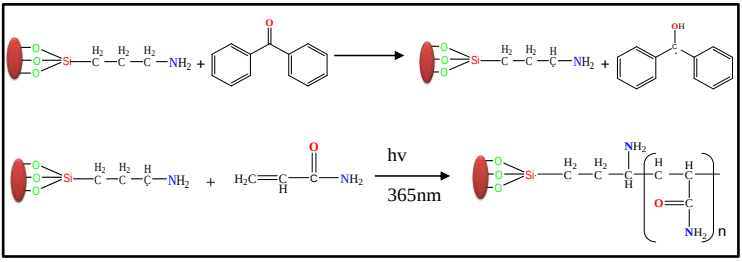
MCM-41 molecular sieves have first been by researchers at Mobil Oil [1]. The preparation of inorganic mesoporous MCM-41 has attracted considerable attention because of its potential application in catalysis and sorption for large molecules [2]. MCM-41 was used to remove organic and mineral effluents. In addition, modified mesoporous silicas were verified to be good adsorbent for the removal of heavy metals from solutions [3]. Modified MCM-41 materials were applied to adsorb anionic dyes, mercury [4].
A hybrid mesoporous PAAM-NH_2-MCM-41(scheme 1) was prepared by method of solvent dispersion in methanol by UV irradiation grafting polymerization and characterized using XRD, XPS, FTIR and TGA techniques.

Scheme 1: Preparation of PAAM-NH_2-MCM-41



Key words: Mésoporous adsorbent, Surface-initiated; Atom transfer radical polymerization; Polyacrylamide.

CampusGLVT

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ADVANCED $Pd/Ce_xZr_{1-x}O_2/SiO_2$ CATALYSTS FOR METHANE COMBUSTION: EFFECT OF PALLADIUM ADDITION STEP

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For applications in gas turbines and boilers, there is an urgent need for the development of new and thermostable catalysts for the combustion of natural gas. Palladium has been reported to be the most active species for methane combustion when operating in excess of oxygen [1]. It is generally accepted that palladium dispersion and palladium-support interaction in the supported Pd catalysts play an important role on their catalytic performance [2]. In addition, it is well known that cerium and zirconium are effective promoters for noble-based combustion catalysts because they can enhance dispersion and stability of the metallic active phase [3]. In this work, silica is used as a support due to its relative high surface area and the addition step of palladium precursor is changed in order to modify the Pd-support interaction and consequently the structure of the active phase.

The $Pd/Ce_xZr_{1-x}O_2/SiO_2$ catalysts (0.5 wt. % Pd) are prepared by sol-gel method with palladium acetate, cerium nitrate, zirconium oxynitrate and tetraethylorthosilicate (TEOS) as precursors. The palladium introduction step during the catalyst synthesis is changed. In effect, palladium acetate is introduced in the sol-gel solution (Pd1), on the dried gel (Pd2) or on the calcined catalyst (Pd3). N_2-physisorption, 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 and UV-Visible results show that all samples contain a $Ce_xZr_{1-x}O_2$ solid solution. Nevertheless, the crystallinity is better on the Pd1 solid. The MET-EDX results show a considerable growth of the $Ce_xZr_{1-x}O_2$ particle size according to the following order: Pd1 > Pd2 > Pd3. Furthermore, the different reduction temperatures of the PdO phase obtained from TPR characterisation shows that the catalyst reducibility depends greatly on the palladium introduction step and probably to the Pd-support interaction. The catalytic activity results show that the palladium introduction step modifies remarkably the activity of the catalytic sites. Globally, the improvement of palladium dispersion and PdO reducibility increases the activity in methane combustion.

The obtained results show that palladium introduction step affect considerably the texture, the structure and the activity of the $Pd/Ce_xZr_{1-x}O_2/SiO_2$ catalysts.

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INVESTIGATIONS OF THE MOLAR GIBBS FREE ENERGY OF ACTIVATION OF VISCOUS FLOW AND DERIVED PROPERTIES IN AQUEOUS 1,2-DIMETHOXYETHANE SOLUTIONS AT DIFFERENT TEMPERATURES

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Physical and chemical properties of solvent mixtures are important for understanding their thermodynamic behavior. One of the most important considerations is that these properties may provide information about molecular interactions [1]. Excess molar volumes, viscosity deviations and excess molar Gibbs energy of activation of viscous flow in 1,2-dimethoxyethane + water binary mixtures at (303.15, 308.15, 313.15, 318.15 and 323.15) K. were calculated from experimental density and viscosity data presented in previous work [2]. Here these experimental values were used to test the applicability of the correlative reduced Redlich-Kister equation and the Herráez and Belda equations as well as their corresponding relative properties. Their correlation ability at different temperatures, and the use of appropriate numbers of parameters, is discussed for the case of limited experimental data. The relative functions are important to reduce the effect of temperature and, consequently, to reveal the effects of different factors and types of interactions. Values of limiting excess partial molar thermodynamic properties at infinite dilution were deduced from different methods.

Key words: Binary liquid mixture, viscosity, Reduced Redlich-Kister equation, Molecular interaction, 1,2-dimethoxyethane.

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MOLECULAR CHARACTERIZATION OF PARAFFIN ISOLATED FROM DIFFERENT TUNISIAN CRUDE OILS BY NMR

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Wax deposition is one of the serious problems in oil industry. Indeed, paraffinic waxes present in petroleum crude can precipitate when temperature decreases during oil production, transport or storage.

These deposits are mainly composed by n-paraffins (linear alkanes) and branched paraffins. The precipitation of these paraffins is responsible for changes in the waxy crude oil properties, including gelation of oil and an increase in viscosity.

In this context we are interested in the characterization of paraffins, after isolation from different crude oils using precipitation method and purification by column chromatography.

Important informations about the structural composition of these waxes were obtained by Nuclear Magnetic Resonance Spectroscopy: 1H NMR, 13C NMR, DEPT 135 and Heteronuclear Single-Quantum Correlation (HSQC).

Key words: paraffin, precipitation, characterization, NMR

AROMATIC NATURAL PRODUCTS FROM PALESTINIAN FLORA AND INDUSTRIAL APPLICATIONS

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Palestine enjoys natural recourses including biodiversity with over 2700 plant species. These plants are found mainly in wild habitats of diverse ecosystems. Among these plants are aromatic and medicinal plants which produce secondary metabolites of volatile oils. Essential oils are aromatic, volatile, and natural products. These compounds are synthesized by plants through secondary metabolism. The essential oils are accumulated in storage organs within plant body including: petals of flowers, trichomes and/or glandular hairs of leaves. The essential oils are characterized as bioactive metabolites increasingly attracting the attention of various industries due to their multiple advantages, especially antioxidant and antimicrobial activities [1]. Essential oils are recognized as raw material for various commercial applications in perfumery, cosmetics, foods, and pharmaceutical medicines. Naturally, some secondary metabolites are involved in defense mechanisms in plant body against abiotic stress (i.e. radiation) and are important in the interaction of plants with other organisms (e.g., attraction of pollinators). There are three major groups of secondary metabolites, including terpenes (i.e. essential oils), phenylpropenoids and N- and S-containing compounds (i.e. alkaloids) [2]. The essential oils are originated from primary metabolites leading to production of valuable compounds used in many applications: herbal medicine which become more attractive to research and alternative medicine. Although, essential oils are produced by traditional methods like maceration but historic technologies like distillation are applied to extract essential oils for industry. However, biotechnology approaches are used to develop the harvest and production of essential oils, like plant tissue culture technique and conserve the producing plants at the same time [3]. This will widen the production of essential oils despite the environmental variation in nature.

Key words: Biodiversity. Natural products. Plant secondary metabolism. Essential oils. Bioactive compounds.

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Synthesis, characterization and study of swelling behavior of nanocomposites polymers

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This work show the growing importance of the chemical modification of polymer hydrogels to obtain a material sensitive to external stimuli, with a high capacity to absorb liquids. Thereafter, the structural stability of these hydrogels was also improved by introduction of inorganic fillers (silicates) in a nanometer level. In this work, we used natural clay (Montmorillonite) to reinforce the structure sensitive hydrogels stimulus temperature. Characterization was done by FTIR, XRD and by transmission electron microscopy (TEM); the thermal stability was studied by thermogravimetric analysis (TGA), the swelling was measured in distilled water and in saline solution at different temperature. Characterization confirmed the new structures of hydrogels nanocomposites. The thermal stability has been greatly improved.

Keywords: sensitive hydrogels, poly (N-isopropylacrylamide), nanocomposite, Montmorillonite, crosslinking, swelling**.**

AB INITIO INVESTIGATION OF OPTICAL PROPPERTIES OF RARE EARTH OXIDES USING DFT CALCULATIONS

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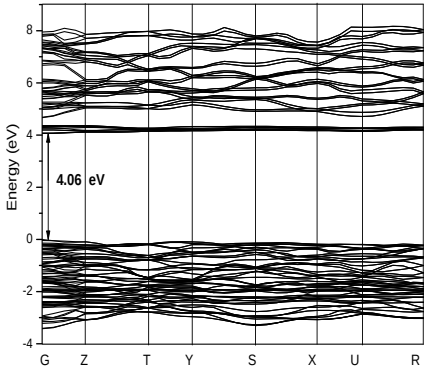
We computationally investigate rare earth oxides type perovskite $LaMO_3$ (M = Er, Ho, Y, Tm), which are key materials in different fields like optical, electrical and magnetic applications. The investigation of optical properties such us optical band gap was experimentally realized for the different rare earth sesquioxides [1,2].

The combination of two rare earth oxides may introduce reduction in optical band gap as well as deformation in the band structure. Since there is few works reported about band gap optical properties of combined rare earth oxides, we intend in this work to realize DFT calculation using GGA and LDA functionals in order to predict band structure, gap energy calculation and electronic properties such as density of states. The theoretical results were in good agreement with experimental results.



Band structure calculated using LDA/CA-PZ+U functionals

Keywords: rare earth, DFT, oxide, band gap.

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THERMAL, VIBRATIONAL AND OPTICAL PROPERTIES OF Er^{3+} /Ag CO-DOPED PHOSPHATE GLASSES: THE EFFECT OF HEAT TREATMENT

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The melt quenching technique was used to synthesize the phosphate glasses doped Er^{3+} ions and containing silver nanoparticles (Ag NPs). The glasses were characterized by X-ray diffraction (XRD), differential scanning calorimetry (DSC), transmission electron microscopy (TEM), Raman spectroscopy and UV–VIS-IR absorption. The nucleation and growth of Ag NPs were controlled by a thermal annealing process. The X-ray diffraction pattern shows no sharp peak indicating an amorphous nature of the glasses. DSC curves data indicate that the glass considered as a glass with relatively good thermal stability. The presence of Ag NPs is confirmed from transmission electron microscopy. The Raman spectra reveal a band shift due to heat annealing process that consequently change the bonding between ligands in the glass matrix and induced a depolymerisation of phosphate groups. Absorption spectra show typical surface plasmon resonance (SPR) band of Ag NPs centered at 403 nm in addition to the characteristic absorption peaks of Er^{3+} ions. It was found that the presence of silver NPs nucleated and grown during the heat annealing process improves the intensity of the plasmon band (SPR). The proposed glasses can be used as potential candidates for the development of solid state lasers.

Keywords: Phosphate glasses, Silver nanoparticles (Ag NPs), Surface plasmon resonance (SPR), Raman spectroscopy.

THE EMISSION INVENTORY IN TUNISIA: METHODOLOGY AND RESULTS

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In order to have a clearer idea about air quality in Tunisia and to identify the best countermeasures against air pollution, a national emission inventory was set up.
The emission inventory is in fact a comprehensive listing by source of air pollutant emissions in a geographic area during a specific time period.
Emission inventories are one of the fundamental components of air quality management plans to measure progress or changes over time to achieve cleaner air and to determine compliance with environmental regulations.
At the Tunisian emission inventory, all the sources were taken into consideration: large point sources (fixed sources) such as industrial sources, airport…etc, non-point sources and mobile sources…
Particulate matters (PM_{10} and $PM_{2.5}$) and gaseous pollutants such as NOx, SOx, NMCOV, CH_4, CO are estimated.
In the absence of national emission factors for all the activities, international ones were used for the large point and non-point sources. As for mobile sources, the Copert IV software is used.
A combination of bottom -up and top-down methodologies is used for the calculation.
The inventory shows that industrial and mobile sources are representing the most important sources of air pollution in the big cities of Tunisia such as Greater Tunis, Gabès, Sfax and Bizerte.
The results obtained combined with the air quality measurement are used as an essential input for the air quality modelling aiming essentially for air quality forecast.

Key words: Air quality, pollutant emissions, emission inventory, emission factors.

ELECTROCHEMICAL INDUSTRY : ECOFRIENDLY TECHNOLOGIES FOR CLEANER PRODUCTS, POWER GENERATION AND INDUSTRIAL WASTE TREATMENT

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The electrochemistry is a science with great range and applications in most industries especially chemical industries. The present work consists of an overview of the electrochemical industrial processes.
The great progress of electrochemistry, directed to technology and called Industrial Electrochemistry, has provided a series of developments, reaching various segments and industrial applications, such as batteries, fuel cells, metal coatings, electrolytic refining of new metals, electrochemical synthesis of organic products, sensors, monitoring, controllers, analysers, anti-corrosion protection techniques, power generation, effluent treatment and recycling.
The application of various products of electrochemical industry converges in the formation of the critical technical conscience in electrochemistry, in the development of clean technologies and consequently on environmental preservation. Finally, it is concluded that the electrochemistry is the technique of the next millennium in both the production of metals, inorganic and organic products as in the treatment of industrial effluents.

Key words: Industrial Electrochemical, energy, environment, recent technologies

Synthesis and characterization of a new benzylated rotaxane based on β-cyclodextrin

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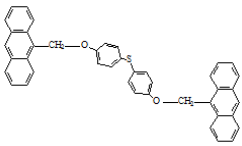
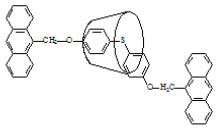
Semiconducting rotaxane (TDPAn/β-CD) based on β-cyclodextrin and anthracene moieties was synthesized via the Williamson reaction in solvent-free conditions. The supramolecular was chemically modified to obtain a new benzylated rotaxane. The chemical structures of the materials were confirmed by ^{1}H-NMR and FT-IR spectroscopic analysis. The optical properties of this organic material were investigated by UV-visible absorption and photoluminescence spectroscopy. A bleu emission was observed in TDPAn/β-CD solid thin film and a green one in the benzylated rotaxane. The optical band gap values were 3.10 and 3.06 eV for both TDPAn/β-CD modified and non-modified, respectively. The HOMO and LUMO levels were estimated using cyclic voltammetry analysis. The surface properties of the perbenzylated rotaxane, were investigated and compared with TDPAn/β-CD and non-rotaxane (TDPAn) materials.

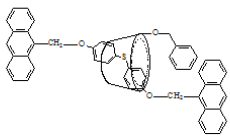
Keywords: Rotaxane; β-Cyclodextin; Semi-conducting material; Photoluminescence; Thin film morphology.



TDPAn



TDPAnβ-CD



RTDPBn

UNDERSTANDING THE REACTIVITY OF TRI-ISOBUTYL ALUMINUM WITH SBA15700 °C SURFACE: B-H ELIMINATION VS. ALKYL TRANSFER

Baraa Werghi, Anissa Bendjeriou-Sedjerari, Julien Sofack-Kreutzer, Edy Abou-Hamad, Jean-Marie Basset.

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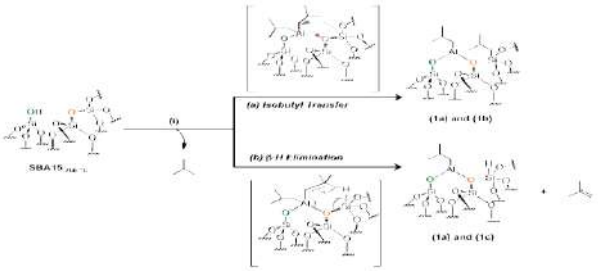
The main objective of this research work is to design a new highly electrophilic Lewis acid single site: aluminum hydroxide supported on mesoporous SBA15: [(≡Si-O)2-Al-OH]. This type of single site can be used as an anchoring site for organometallic complexes via SOMC [(≡Si-O)2-Al-OMLn, with M = W or Ta, Ln= carbyne, carbene or hydride). By this way, we expected to improve the actual turnover number of alkane metathesis (TON = 120).1

Herein, we present the first step of this investigation. It concerns the understanding of the reaction mechanism of TIBA with SBA-15 surface. Thus, the reaction of 1 equivalent of triisobutylaluminum (TIBA, 1M/hexane) at 0 °C with a partially dehydroxylated mesoporous SBA15 700 °C leads to the formation of a well-defined bipodal [(≡SiO)2-Al-CH2CH(CH3)2] along with silicon hydride [≡Si-H] and silicon alkyl [≡Si-C4H9] species. Their structural identities were characterized by FT-IR and solid-state NMR spectroscopies (1H, 13C, 29Si and 27Al), EFTEM microscopy, elemental and gas phase analysis.



After reaction of TIBA with the single silanol site (protonolysis with evolution of one equivalent of isobutane per aluminum), two competitive mechanisms explain the formation of the different surface species.

(1) An alkyl transfer from the aluminum to a siloxane bridges of the SBA15700 C surface;

(2) β-H elimination via the formation of six-membered ring transition state, with evolution of one equivalent of isobutene per aluminum.

The results are reinforces with those obtained after reaction of the surface with 5 and 2.5 equivalent of TIBA.

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Synthesis, characterization and photoluminescence properties of $Zn_xCd_{1-x}S$ quantum dots with high photocatalytic activity

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Over the last decades, many fundamental and applied research works have focused on semiconductor nanoparticles, also called quantum dots (QDs), due to their exceptional optical properties originating from quantum confinement [1]. Photoluminescence (PL) of ternary QDs can be tuned not only by controling the particles size but also by changing their chemical composition. Among these materials, $Zn_xCd_{1-x}S$ QDs are promising materials in different fields such as high density optical recording devices, photovoltaic cells or photocatalysis[2].
This study focuses on the optical properties of aqueous phase-prepared $Zn_xCd_{1-x}S$ ($0.2 < x < 0.9$) QDs using 3-mercaptopropionic acid (MPA) as capping ligand. The effects of the synthesis parameters (pH, time of reflux, Zn/Cd ratio) on the QDs structure and PL properties have been investigated.
The X-ray diffraction patterns of the $Zn_xCd_{1-x}S$ samples exhibit the cubic zinc blende phase characteristics. The diffraction peaks positions shifted to low (2θ) diffraction angles with increasing the cadmium content in accordance with the formation of the ternary $Zn_xCd_{1-x}S$ materials. Transmission Electronic Microscopy analysis reveals spherical monodispersed particles with an average diameter of ca. 4.5 nm. PL spectra show that the emission wavelength maximum λ_{max} is redshifted with increasing the Cd content (492 nm for $Zn_{0.8}Cd_{0.2}S$ and 600 nm for $Zn_{0.3}Cd_{0.7}S$), which further confirmed the formation of the ternary QDs (Figure 1).
This redshift is enhanced when the QDs are doped with 0.5% of copper (noted $Zn_xCd_{1-x}S$:Cu). Adding a ZnS shell around the $Zn_xCd_{1-x}S$:Cu nanoparticles strongly improved their PL quantum yield (QY) without modifying the wavelength emission. An increase of the PL QY of $Zn_{0.5}Cd_{0.5}S$:Cu QDs from 9% to 40% was observed upon ZnS shell introduction. Photocatalytic activities of ZnO/ZnCdS:Cu@ZnS nanocomposite were finally examined by considering the degradation of the water soluble Orange II dye under solar light irradiation. It was found that the association of small amounts of ZnCdS:Cu@ZnS QDs (less than 5 weight %) with ZnO markedly increased the photocatalytic efficiency due to the enhanced absorption in the visible light region.

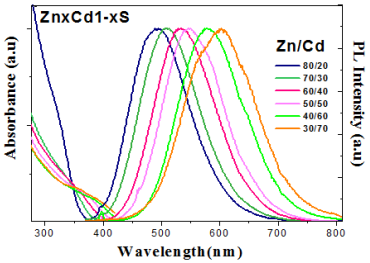

Figure 2: UV-visible absorption and PL emission spectra of $Zn_xCd_{1-x}S$ QDs prepared in aqueous solution with 3-MPA as capping ligand. The excitation wavelength is 350 nm.

Keywords: ternary semiconductors, aqueous phase, core/shell, photoluminescence, photocatalysis.

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Synthesis of a new materials derivate of 8-hydroxyquinoline. Application in wastewater treatment

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A New materials derivatives of 8-hydroxyquinoline was synthesized by the Mannich reaction from secondary amines. Various analytical techniques (^{1}H & ^{13}C NMR, FTIR, Mass spectroscopy) were used to characterize this material. The use of new material for the uptake and removal of heavy metal in aqueous solution was investigated. Conditions for an effective sorption were optimized with respect to different experimental parameters in batch process.

Key words: 8-hydroxyquinoline- secondary amines- Mannich- heavy metal extraction

VALORISATION DES PHOSPHOGYPSES PAR LEUR INTEGRATION DANS LES MATERIAUX DE CONSTRUCTION

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This work deals with the valorization of phosphogypsum by its integration in building materials. Clayey raw materials used in this study originate from Agareb (Sfax) and phosphogypsum from Tunisian Chemical Group (Sfax).
The raw materials were first characterize chemical, mineralogical, granulometric and textual analyses were carried out. Them, the effect of incorporating different percentages of clay and phosphogypsum on the cylindric experiments was investigated and physico-mechanical characteristics were analyzed. The resultats showed that sun-dried phosphogypsum integration in clay to make cylindrical eprouvette would give the material a better resistance to compression in comparison to others phosphogysum treatments. The optimization of the composion exhibited that the mixture of 25% phosphogypsum improves compression resistance **(2,55 MPa)**, porosity **(19,52 %)** and density **(1,88 g/cm3)**.

Keywords : Phosphogypsum, building materials, compression resistance.

Sintering Behaviour of Some Clay-Degreaser Mixtures

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Numerous ceramics are elaborated from clay based raw materials compacted and strengthened by sintering at high temperatures. Their application domain is very vast; it spreads from the traditional ceramic for buildings to the more technical products, for electronics notably. For this reason, the study of clay minerals has found interest for many years and the literature is very rich. Unfortunately, no survey involving the firing behaviour of clay-degreaser mixtures has been undertaken. This study reports the results of an investigation dealing with the sintering behaviour up to 1150°C of some clay-degreaser mixtures (from Tunisia) by studying the apparent density and the mechanical properties evolutions as a function of the sintering temperature and/or the mixture composition. In fact, it was shown that whatever the mixture composition, the clay and the degreaser behave independently of one another and the system seems governed by the major element during sintering.

Key words: clay, degreaser, sintering behaviour, apparent density and mechanical properties.

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