8th International Solid State Chemistry Conference

ISSCC 2017

Organized by the

Tunisian Chemical Society

17-20 December 2017

Royal Thalassa Hotel, Skanes Monastir - Tunisia

Abstracts of Lectures and Communications
Participants' List

TUNISIAN CHEMICAL SOCIETY - Short Program of the ISSCC 2017

Sunday 17 December 2017	
14.00	**Welcoming participants, distribution of documents and check in**
16.30	**ISSCC 2017 Opening Ceremony**
16.45 - 17.20	**Plenary Lecture 1** **Rabah BOUKHERROUB** *Université de Lille, Avenue Poincaré, CS 60069, 59652 Villeneuve d'Ascq, France*
17.25 - 18.00	**Plenary Lecture 2** **Jean-Marc GRENECHE** *Univ. du Maine, Institut des Molécules et Matériaux du Mans, Le Mans, France*
19.00	***Dinner***
Monday 18 December 2017	
09.00 - 09:35	**Plenary Lecture 3** **Marco SCAVINI** *Università di Milano, via C. Golgi, I-19 20133 Milano, Italia*
09.35 - 10:10	**Plenary Lecture 4** **Marco GEPPI** *University of Pisa, via Moruzzi 13, 56126 Pisa, Italy*
10.15 - 11.00	**Poster Session 1 (P 1 - P 36) Alphabetical Order**
11.00 - 12.30	**Oral Communications - Session 1: OC1-OC6**
13.00	***Lunch***
14.40 - 15.15	**Plenary Lecture 5** **Aziz AMINE** *Faculty of Sciences and Techniques, Hassan II University of Casablanca, Morocco*
15.15 - 16.30	**Oral Communications - Session 2: OC7-OC11**
16.30 - 17.30	**Coffee break + Poster Session 2 (P 37 - P 72) Alphabetical Order**
17.30 - 18.30	**Oral Communications - Session 3: OC12-OC15**
19.00	***Dinner***
Tuesday 19 December 2017	
09.00 - 09:35	**Plenary Lecture 6** **Pavel POSTNIKOV** *Tomsk Polytechnic University, Tomsk, Russia*
09.35 - 10:10	**Plenary Lecture 7** **Jean-Marie NEDELEC** *Institut de Chimie de Clermont-Ferrand, F-63000 Clermont-Ferrand, France*
10.15 - 11.00	**Poster Session 3 (P 73 - P 108) Alphabetical Order**
11.00 - 12.30	**Oral Communications - Session 4: OC16-OC21**
13.00	***Lunch***
14.40 - 15.15	**Plenary Lecture 8** **Ayhan ORAL** *Çanakkale Onsekiz Mart University, Çanakkale, Turkey*
15.15 - 16.30	**Oral Communications - Session 5: OC22-OC26**
16.30 - 17.30	**Coffee break + Poster Session 4 (P 109 - P 141) Alphabetical Order**
17.30 - 18.30	**Oral Communications - Session 6: OC27-OC30**
20.00	***Dinner Buffet***
Wednesday 20 December 2017	
09.00 - 09.35	**Plenary Lecture 9** **Cécile AUTRET** *Université François Rabelais, Parc de Grandmont, 37200 Tours, France*
09.45 - 11.00	**Oral Communications - Session 7: OC31-OC35**
11.00 - 11.35	**Plenary Lecture 10** **Noureddine RAOUAFI** *University of Tunis El Manar, Tunis, Tunisia*
11.45 - 12.00	**Closing Remarks, Awards Distribution and 9th ISSCC (in 2019) Announcement**
12.30	***Lunch and check out***

Being on time is a key factor for the success of the conference

FOREWORD

Dear Participants,

On behalf of the Tunisian Chemical Society, we would like to welcome you to the 8th International Solid State Chemistry Conference.

The scientific program will provide an opportunity for researchers and scientists in the chemistry fields to exchange new ideas, information and share knowledge and experience with distinguished scientists.

The program will include outstanding 10 plenary lectures, 100 oral presentations and around 141 posters provided by leading scientists invited both from academia and industry, as well as promising young researchers.

Many thanks for all invited speakers who accepted to contribute lectures at this event despite this period of festive days, and for all our participants. We wish all our guests a memorable scientific conference and a pleasant stay in this nice area of Tunisia

Adel MEGRICHE

President of the Tunisian Chemical Society

Chair, Scientific Committee, ISSCC 2017.

Scientific Committee

Adel MEGRICHE (Chairman**)**	FST - Tunis, University of Tunis El Manar
Mohamed KHITOUNI (Co-chair**)**	FSS - Sfax, University of Sfax
Mohamed Mehdi CHEHIMI	University Paris Diderot, Sorbonne Paris Cité, ITODYS, France
Halim HAMMI	CNRSM - Borj Cédria, University of Carthage
Ezzeddine SRASRA	CNRSM - Borj Cédria, University of Carthage
Amor HADDAD	ISSAT - Mahdia, University of Monastir
Dalila BEN HASSEN-CHEHIMI	FSB - Bizerte, University of Carthage
Moheddine ABDELLAOUI	INRAP - Sidi Thabet
Noureddine AMDOUNI	FST - Tunis, University of Tunis El Manar
Cécile AUTRET	University of Tours , Tours, France
Marco GEPPI	University of Pisa, Italy
Noureddine RAOUAFI	FST - Tunis, University of Tunis El Manar
Rached BEN HASSEN	ISSBAT - Tunis, University of Tunis El Manar

Organizing Committee

Hatem MEJDOUB (Chairman**)**	FSM - Monastir, University of Monastir
Halim HAMMI (Co-chair**)**	CNRSM - Borj Cédria, University of Carthage
Adel MEGRICHE	FST - Tunis, University of Tunis El Manar
Amine MNIF	FST - Tunis, University of Tunis El Manar
Latifa LATROUS	IPEIM - Tunis, University of Tunis El Manar
Imed LAAJIMI	Tunisian Chemical Society
Mohamed Mehdi CHEHIMI	University Paris Diderot, Sorbonne Paris Cité, ITODYS, France
Béchir CHAOUACHI	ENIG - Gabès, University of Gabès
Mohamed BELHOUCHET	FSS - Sfax, University of Sfax
Younes MOUSSAOUI	FSG - Gafsa, University of Gafsa
Taoufik BOUBAKER	FSM - Monastir, University of Monastir
Rym ABIDI	FSB - Bizerte, University of Carthage

Program of the ISSCC 2017

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Sunday 17 December 2017		
14.00	**Welcoming participants, distribution of documents and check in**	
16.30	**ISSCC 2017 Opening Ceremony**	
16.45 - 17.20 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 1** **Rabah BOUKHERROUB** *Institut d'Electronique, de Microélectronique et de Nanotechnologie (IEMN), UMR CNRS 8520, Université de Lille, Avenue Poincaré, CS 60069, 59652 Villeneuve d'Ascq, France* **Carbon-based nanocomposites for energy, environmental and biomedical applications**	*Chairman:* Adel Megriche
17.25 - 18.00 *(30 min speech + 5 min discussion)*	**Plenary Lecture 2** **Jean-Marc GRENECHE** *Université du Maine, Institut des Molécules et Matériaux du Mans, UMR CNRS 6283, 72085 Le Mans Cedex, France* **Solid State Physics: What can we learn from Mössbauer spectrometry**	*Chairman* Mohamed Khitouni
19.00	***Dinner***	

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Monday 18 December 2017 (morning)						
09.00 - 09:35 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 3** **Marco SCAVINI** *Università di Milano, via C. Golgi, I-19 20133 Milano, Italia* **Applications of pair distribution function analysis to material science**				*Chairman:* Hatem Majdoub	
09.35 - 10:10 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 4** **Marco GEPPI** *University of Pisa, via Moruzzi 13, 56126 Pisa, Italy* **Solid state NMR spectroscopy: applications to the study of structural and dynamic properties of drugs and materials**				*Chairman:* Halim Hammi	
10.15 - 11.00	**Poster Session 1 (P 1 - P 36) Alphabetical Order**					
Oral Communications - Session 1 -						
	Room A – *Halim Hammi*		**Room B –** *Dalila Helali*		**Room C –** *Latifa Latrous*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
11.00 - 11.15	**OC-01A**	ABID Dhouha	**OC-01B**	AJMI Jihen	**OC-01C**	BEN HADJ SADOK Ines
11.15 - 11.30	**OC-02A**	ABID Marwa	**OC-02B**	ALOUI Zouhaier	**OC-02C**	BEN HAJ SALAH Maroua
11.30 - 11.45	**OC-03A**	ABID Rahma	**OC-03B**	ASKRI Amira	**OC-03C**	BEN MBAREK Wael
11.45 - 12.00	**OC-04A**	ABID DERBEL Marwa	**OC-04B**	BASDOURI Zeineb	**OC-04C**	BEN MOUSSA Oumaima
12.00 - 12.15	**OC-05A**	ABIDI Maali	**OC-05B**	BELAM Wahid	**OC-05C**	BEN MOUSSA Sana
12.15 - 12.30	**OC-06A**	AK Metin	**OC-06B**	BEN ALI Saida	**OC-06C**	BENAHSENE Amani Hend
13.00	***Lunch***					

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Monday 18 December 2017 (afternoon)						
14.40 - 15.15 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 5** **Aziz AMINE** *Laboratory of Chemical Engineering and Environment, Faculty of Sciences and Techniques, Hassan II University of Casablanca, Morocco* **Recent advances in application of molecularly imprinting polymers in electrochemical sensing**				*Chairman:* Mohamed Lotfi Efrit	
Oral Communications - Session 2 -						
	Room A - *Med Lotfi Efrit*		**Room B -** *Mohamed Dammak*		**Room C -** *Nabil Zouari*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
15.15 - 15.30	**OC-07A**	BOUALLEGUI Thamer	**OC-07B**	ZRELLI-ANNABI Rahma	**OC-07C**	CHEIKH Dorsaf
15.30 - 15.45	**OC-08A**	BOUAZIZ Emna	**OC-08B**	BOUTAR Marwa	**OC-08C**	DACHRAOUI Olfa
15.45 - 16.00	**OC-09A**	BOUCHOUICHA Hamed	**OC-09B**	BRAIEK Faycel	**OC-09C**	DARDOURI Mokhtar
16.00 - 16.15	**OC-10A**	BOUGHDIRI Seif Eddine	**OC-10B**	CHAHBANI Amna	**OC-10C**	DGACHI Souad
16.15 - 16.30	**OC-11A**	BOUHIDEL Zakaria	**OC-11B**	CHAKER Manel	**OC-11C**	COSKUN Yalcin
16.30 - 17.30	**Coffee break + Poster Session 2 (P 37 - P 72) Alphabetical Order**					
Oral Communications - Session 3 -						
	Room A - *Fathi Touati*		**Room B -** *Salah Kouas*		**Room C -** *Rym Abidi*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
17.30 - 17.45	**OC-12A**	DHIFI Marwa	**OC-12B**	ELMHAMDI Abdelhakim	**OC-12C**	FREDJ Donia
17.45 - 18.00	**OC-13A**	DHOUIB IKram	**OC-13B**	FADHLAOUI Amor	**OC-13C**	GALAI Haykel
18.00 - 18.15	**OC-14A**	DISSEM Nour	**OC-14B**	FADHLI Marwa	**OC-14C**	GHARBI Amina
18.15 - 18.30	**OC-15A**	DKHILI Samiha	**OC-15B**	FEZAI Ramzi	**OC-15C**	GUSELNIKOVA Olga
19.00	***Dinner***					

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Tuesday 19 December 2017 (Morning)						
09.00 - 09:35 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 6** **Pavel POSTNIKOV** *Tomsk Polytechnic University, Tomsk, Russia* **Diazonium chemistry: The universal way for the design of functional materials**					*Chairman:* Mehrez Romdhane
09.35 - 10:10 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 7** **Jean-Marie NEDELEC** *Institut de Chimie de Clermont-Ferrand, F-63000 Clermont-Ferrand, France* **Cationic substitutions in sol-gel derived calcium phosphates bioceramics: A fine tuning of biological properties**					*Chairman:* Béchir Chaouachi
10.15 - 11.00	**Poster Session 3 (P 73 - P 108) Alphabetical Order**					
Oral Communications - Session 4 -						
	Room A - *Béchir Chaouachi*		**Room B -** *Haykel Galai*		**Room C -** *Nizar Bellakhal*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
11.00 - 11.15	**OC-16A**	GUESMI Afef	**OC-16B**	HFIDHI Nabil	**OC-16C**	JAMMAZI Donia
11.15 - 11.30	**OC-17A**	HAMDI Intissar	**OC-17B**	ISMAIL Rimeh	**OC-17C**	JEDIDI Wiem
11.30 - 11.45	**OC-18A**	HARBAOUI Dhouha	**OC-18B**	JABALLI Rim	**OC-18C**	JEMMALI Mosbah
11.45 - 12.00	**OC-19A**	HASSEN Sabri	**OC-19B**	JABEUR Wiem	**OC-19C**	JENDOUBI Imen
12.00 - 12.15	**OC-20A**	HCHICHA Khouloud	**OC-20B**	JALEL Bouthaina	**OC-20C**	JLASSI Raja
12.15 - 12.30	**OC-21A**	HAMACHA Rachida	**OC-21B**	JAMAI Nabil	**OC-21C**	JRABA Nawel
13.00	***Lunch***					

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Tuesday 19 December 2017 (afternoon)						
14.40 - 15.15 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 8** **Ayhan ORAL** *Çanakkale Onsekiz Mart University, Çanakkale, Turkey* **Effects of Temperature on the Crystallinity and Morphology of Poly(Lactic)Acid - Phenylethyl alcohol**					*Chairman:* Farhat Rezgui
Oral Communications - Session 5 -						
	Room A - *Farhat Rezgui*		**Room B -** *Mohamed Khitouni*		**Room C -** *Ridha Ben Salem*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
15.15 - 15.30	**OC-22A**	KADRI Ines	**OC-22B**	KLAI Kacem	**OC-22C**	LTAIEF Hasna
15.30 - 15.45	**OC-23A**	KEFI Sana	**OC-23B**	LABIDI Khaled	**OC-23C**	MAALAOUI Ahlem
15.45 - 16.00	**OC-24A**	KHEDHIRI Lamia	**OC-24B**	LACHHEB Rabeb	**OC-24C**	MATHLOUTHI Maha
16.00 - 16.15	**OC-25A**	KHELFA Tarek	**OC-25B**	LASSOUED Abdelmajid	**OC-25C**	MCHIRI Chadlia
16.15 - 16.30	**OC-26A**	KHITOUNI Nawel	**OC-26B**	LASSOUED Mohamed Saber	**OC-26C**	MEJRI Rania
16.30 - 17.30	**Coffee break + Poster Session 4 (P 109 - P 141) Alphabetical Order**					
Oral Communications - Session 6 -						
	Room A - *Mohamed Toumi*		**Room B -** Mohamed *Belhouchet*		**Room C -** *Wahid Belam*	
	Com.	*communicating*	*Com.*	*communicating*	*Com.*	*communicating*
17.30 - 17.45	**OC-27A**	MHADHBI Mohsen	**OC-27B**	NEFZI TOUIL Hadhemi	**OC-27C**	SAIDANI Sofien
17.45 - 18.00	**OC-28A**	MOGHDED Intissar	**OC-28B**	RHOUMA Salem	**OC-28C**	SEHIMI Hiba
18.00 - 18.15	**OC-29A**	MRABET Hounaida	**OC-29B**	SAID Senda	**OC-29C**	SHILI Afef
18.15 - 18.30	**OC-30A**	Nasri Rawia	**OC-30B**	SAIDI Marwa	**OC-30C**	SONNI Manel
20.00	***Dinner Buffet***					

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Wednesday 20 December 2017 (Morning)				
09.00 - 09.35 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 9** **<u>Cécile AUTRET</u>** *Université François Rabelais, Parc de Grandmont, 37200 Tours, France* **Synthesis, structural characterization and dielectric properties of $CaCu_3Ti_4O_{12}$ core-shell structure**			*Chairman:* Adel Megriche
Oral Communications - Session 7 -				
	Room A - *Nizar Bellakhal*		**Room B -** *Taoufik Boubaker*	
	Com.	*communicating*	*Com.*	*communicating*
09.45 - 10.00	**OC-31A**	SOUISSI Fatma Zahra	**OC-31B**	WEDERNI Asma
10.00 - 10.15	**OC-32A**	TARHOUNI Mohamed	**OC-32B**	ZAARA Kaouther
10.15 - 10.30	**OC-33A**	TLILI Hafsia	**OC-33B**	ZAGHDOUDI Malika
10.30 - 10.45	**OC-34A**	TOUMI Sirine	**OC-34B**	ZANNEN Moneim
10.45 - 11.00	**OC-35A**	TRIMECHE Marwa	**OC-35B**	ZIDANI Chafika
11.00 - 11.35 *(30 mn speech + 5 mn discussion)*	**Plenary Lecture 10** **<u>Noureddine RAOUAFI</u>** *University of Tunis El Manar, Tunis, Tunisia* **Nanomaterials-Based Electrochemical Biosensors**			*Chairman:* Hatem Ben Romdhane
11.45 - 12.00	**Closing Remarks, Awards Distribution and 9th ISSCC (in 2019) Announcement**			
12.30	***Lunch and check out***			

Speakers' Abstracts

Rabah BOUKHERROUB

Institut d'Electronique, de Microélectronique et de Nanotechnologie
Université de Lille, Villeneuve d'Ascq, France

Dr. Rabah Boukherroub received a PhD in chemistry from the University Paul Sabatier in Toulouse, France. He is currently a CNRS research director and a group leader at the Institute of Electronics, Microelectronics and Nanotechnology (IEMN), University of Lille, France. He is Associate Editor for ACS Applied Materials & Interfaces. He is also a guest Professor, China University of Petroleum, Qingdao, China. His research interests are in the area of nanostructured functional materials, surface chemistry, and photophysics of semiconductor/metal nanostructures with emphasis on biosensors, nanomedicine, photocatalysis and energy-related applications. He is a co-author of 440+ research publications and wrote 31 book chapters in subjects related to nanotechnology, materials chemistry, and biosensors. He has 11 patents or patents pending.

Carbon-based nanocomposites for energy, environmental and biomedical applications

Rabah Boukherroub

Institut d'Electronique, de Microélectronique et de Nanotechnologie (IEMN), UMR CNRS 8520, Université de Lille, Avenue Poincaré, CS 60069, 59652 Villeneuve d'Ascq, France
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Recent developments in materials science and nanotechnology have propelled the development of a plethora of materials with unique chemical and physical properties for various applications. Graphitic nanomaterials such as carbon nanotubes, fullerenes and, more recently, graphene oxide (GO)/reduced graphene oxide (rGO) and carbon quantum dots, have gained a great deal of interest for their potential applications in various aspects of science and technology.

Graphene, the name specified to a one atom-thick two-dimensional (2D) single layer of sp^2 hybridized carbon atoms arranged in a honeycombed lattice with large surface area, exceptional thermal, mechanical, optical and structural properties. This wonder material is a "hot topic" of research in interdisciplinary sciences with potential applications in several fields such as nano-electronics, organic catalysis, environmental remediation, drug delivery, etc.

The last decade has also witnessed a huge interest in the synthesis of carbon quantum dots (CQDs) and their applications in various fields due to their ease of synthesis and functionalization, their outstanding properties such as size- and wavelength-dependent luminescence emission, resistance to photobleaching and good biocompatibility.

Due to their low cost of production, large specific surface area and abundant surface chemistry, rGO and CQDs have shown great promise in the development of novel composites, biosensors, photocatalysts, electrocatalysts, and drug delivery systems. These hybrid nanomaterials offer unusual combinations of electrical, thermal, mechanical, catalytic, electrocatalytic, optical and magnetic performances that are difficult to attain separately from the individual components.

In this presentation, I will focus on the different strategies for the preparation of rGO- and CQDs-based hybrid materials and the various applications of these nanohybrids in energy, biomedicine, and environmental remediation.

Jean-Marc GRENECHE

Institut des Molécules et Matériaux du Mans
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Director of UMR CNRS 6283 501/01/2012 - 31/12/2016)
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Alexandre von Humboldt Fellow in Karlsruhe (Germany) 1988-1989
Invited Professor at Santiago de Compostela (Spain) 1992
Key-words: amorphous metallic alloys, nanocrystalline alloys, magnetic nanoparticles, amorphous and crystalline fluorides, nanostructured milled and alloyed powders, functionalized nanoparticles, mesoporous structures magnetic nanostructures, magnetic frustration, non colinear magnetism, surface phenomena, interface phenomena, grain boundaries, core shell architectures, MOF, redox phenomena, physical biogeochemistry 57Fe Mössbauer spectrometry, numeric simulation
National Collaborations: Versailles Institut Lavoisier (C. Serre, F. Millange, P. Horcajada), Paris VII (ITODYS S. Ammar), Grenoble (LGIT L. Charlet), Orléans (BRGM C. Tournassat), Strasbourg (IPCMS G. Pourroy, S. Begin-Colin), Caen (V. Caignaert, D. Pelloquin), Cachan (F. Mazaleyrat)
International Collaborations: Spain (Santiago de Compostela, Barcelona, Sevilla, Bilbao, Oviedo), Germany (Erlangen), Colombia (Cali, Medellin, Ibague, Bogota), Ireland (Dublin), Poland (Warsaw, Poznan, Katowice), Slovak Republic (Kosice, Bratislava), Great Brittany (London, Milton Keynes), India (Bengalore, Madras), Argentine (Buenos Aires, La Plata, Bariloche), Brazil (Rio de Janeiro, Bello Horizonte), Algeria (Annaba, Tizi-Ouzou), Morocco (Rabat, Casablanca), Tunisia (Sfax), South Korea (Deajon), Hungary (Budapest), Romania (Bucharest, Iasi), Italy (Roma, Cagliari), Peru (Lima), Portugal (Coimbra, Porto), Mexico (UNAM Mexico City)
Publications: ~ 520 papers (~ 10000 citations and H = 46 WoS, H=53 Google Scholar, H=49 Scopus)
105 Invited conferences at International Conferences and Schools and National Events
French Representative at the International Board on Applications of Mössbauer Effect since 1998
Secretary of the International Board on Applications of Mössbauer Effect 2002-2007
Vice Chairman of the International Board on Applications of Mössbauer Effect since 2007
Referee: Phys. Rev. B, Phys. Rev. Letters, J. Appl. Physics, J. Appl. Lett., J. Phys.: Condensed Matter, J. Phys. D: Applied Physics, J. Magn. Magn. Mater., Hyper. Inter., Material Science and Engineering, Materiala Scripta, Eur. J. Phys. AP, Solid State Science, Corrosion Science, Physica B, European Journal of Mineralogy, J. Alloys Compds, Chem. Mater., ...
French Responsible of Bilateral Agreements: Picasso (2) Spain; Procope (1) Germany; Stefanik (1) Slovak Republik; CNRSPAS (6) Poland; CNRS-SAS (3) Slovak Republic; CNRS-Ireland (2); CNRS-RAS (2) Romania; ECOS NORTE (3) Colombia; CNRSKOCEF (1) South Korea; CMEP Algeria (1); CMEP Morocco (1)
French Responsible of NATO projects (2) and partner of FUNMIG (EU 6thPCRDT)
Partner of 5 ANR projects (Research French National Agency) and 2 Regional Projects
Member of scientific organizing committees of national and international conferences
Organizer of 2 Maghrebin Schools on Nanomagnetism and NanoMaterials
Organizer of 2 French Speaking Mössbauer Group Workshops
Organizer of 4 Schools devoted to Mössbauer spectrometry and its applications
Member of the Editorial Board of Hyperfine Interactions, Mössbauer Effect Reference Data Journal
Editor of Journal of Alloys and Compounds, Nanomagnetism, Hyperfine Interactions

Apport de la spectrométrie Mössbauer à l'étude des matériaux

J.M. Greneche

Université du Maine, Institut des Molécules et Matériaux du Mans, UMR CNRS 6283, 72085 Le Mans Cedex, France

Après un bref rappel des principes de l'effet Mössbauer et de la nature des interactions hyperfines observées, on donnera quelques aspects instrumentaux, expérimentaux et numériques nécessaires à l'approfondissement de la technique et de ses applications. Ensuite, nous illustrerons à partir de différents exemples comment la spectrométrie Mössbauer contribue à l'investigation des propriétés structurales et magnétiques des matériaux en raison de caractère de sonde locale et son aspect non invasif.

Les exemples permettront d'apprécier l'apport de la technique à l'étude de matériaux cristallins (oxydes, fluorures, métalliques, structures microporeuses), puis amorphes et de nanoarchitectures (nanoparticules, poudres nanostructurées, alliages nanocristallins, nanoparticules creuses). Les mesures en fonction de la température et en présence d'un champ magnétique extérieur montreront par ailleurs leur complémentarité nécessaire pour mieux résoudre les structures hyperfines dans certaines situations et mieux comprendre les propriétés magnétiques des matériaux à base de Fe.

Marco SCAVINI

Chemistry Department, University of Milano
Milano, Italy

- Marco Scavini is born in Vigevano (Italy) in 1965. He received his MSc degree in Chemistry in 1992 from the University of Pavia (Italy) and, in 1996, his PhD in Chemical Sciences from the "Consorzio Interuniversitario di Genova, Pavia e Torino", discussing a thesis on the study of the defect equilibria and of the physical properties of neodymium cuprate high TC superconductors.
- In 1997-1998 he worked as a quality assurance assistant at the pharmaceutical plant "Farmabios srl". Finally, in 1998, he joined the Chemistry Department. of the University of Milan (Italy) as a Faculty member.
- Marco Scavini's research activity deals with the study of relationship among structure, defect thermodynamics and physical properties in disordered materials such as perovskites oxides and fluorides, fluorite structure oxides, nanoparticles and nanocomposites.
- In recent years, he focused his interests in the fields of local structure determination with the use of the pair distribution function analysis of diffraction data collected at synchrotron radiation and neutron sources. To this purpose he was main proposer and co-proposer of more than seventy experiments accepted at European Large Facilities.
- He authored about seventy scientific articles on international journals, books and book chapters.

Five selected papers:

"Percolating Hierarchical Defect Structures drive Phase Transformation in $Ce_{1-x}Gd_xO_{2-x/2}$: a Total Scattering study" M. Scavini*, M. Allieta, M. Coduri, P. Masala, M. Brunelli, S. Cappelli, C. Oliva, F. Orsini, C. Ferrero, **IUCrJ 2** (2015) 511-522.

"Charge ordering transition in $GdBaCo_2O_5$: evidence of reentrant behavior" M. Allieta*, M. Scavini*, L. Lo Presti, M. Coduri, L. Loconte, S. Cappelli, C. Oliva, Phyl Pattison, Paolo Ghigna, Valerio Scagnoli, **Phys. Rev. B 88** (2013) 214104.

"Role of intrinsic disorder in the structural phase transition of magnetoelectric $EuTiO_3$", M. Allieta, M. Scavini*, L. Spalek, V. Scagnoli, H. Walker, C. Panagopoulos, S. Saxena, T. Katsufuji, C. Mazzoli. **Physical Review B85**, (2012) 184107.

"Probing complex disorder in $Ce_{1-x}Gd_xO_{2-x/2}$ using the Pair Distribution Function Analysis", M. Scavini*, M. Coduri, M. Allieta, M. Brunelli, C. Ferrero, **Chem. Mater. 24** (2012) 1338-1345.

"Effect of local disorder on the transport properties of Al doped $SmBa_2Cu_3O_{6+d}$ superconductor", M. Scavini*, M. Coduri, M. Allieta, L. Mollica, M. Brunelli, L. Malavasi, A. Lascialfari, C. Ferrero, **The Journal of Physical Chemistry C, 114** (2010) 19509-20.

Applications of pair distribution function analysis to material science

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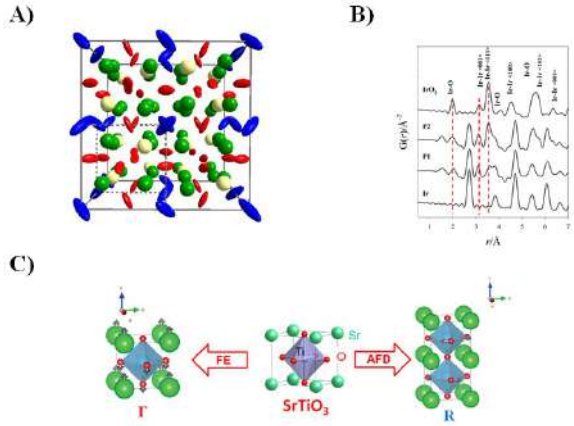
The fascinating physical properties of functional crystalline materials are strictly linked to symmetry and/or compositional inhomogeneities at the nanoscale and sometimes at even larger length scales, caused by high defect concentrations or strong electron correlation. Therefore, a meaningful structural characterisation should encompass investigations at all the available length scales.
The so-called Pair Distribution Function (PDF), that is the real space analysis of total scattering (diffraction) data, is emerging as a powerful technique to achieve a complete and accurate structural picture at all the length scales. In fact, interatomic distances detected by PDF may extend up to tens or even hundreds of Angstroms, allowing revealing both the nature and the coherence length of local distortions present in materials and how they merge into the average structure.
IAfter a brief introduction to the technique, some applications of PDF to material science are presented, which highlight the role of high *Q* resolution data for jointed reciprocal space and real space investigation and the interplay between X-ray and neutron diffraction for a detailed structural description of disorder.
Examples will include i) highly defective oxides, where defect clusters hierarchically self-organise into nano-domains; ii) nanoparticles and composites; iii) perovskite oxides with electronically driven symmetry breaks.



A) Structure of $Ce_{0.5}Gd_{0.5}O_{1.75}$. The amplitude and the anisotropy of thermal parameters (at 99% probability) highlight the presence of large compositional disorder in this solid solution.
B) Local structure of Ir/IrO_2 nanocomposites (P1 and P2 samples) compared to the ones of the microcrystalline pure compounds.
C) Ferroelectric (FE) and Antiferrodistorsive (AFD) structural instabilities of $SrTiO_3$ at the Γ and R points, respectively.

Marco GEPPI

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Born in Rosignano M.mo (LI, Italy) on 01/01/1966.

- M. sc. in Chemistry in 1991 at the University of Pisa in 1991, with mark 110/110 cum laude, defending an experimental thesis concerning studies of molecular dynamics and orientational order of mesogens in nematic and smectic liquid crystalline phases by means of 2H-NMR, supervisor prof. C.A. Veracini.
- Ph.D. in Chemistry in 1997 at the Scuola Normale Superiore of Pisa, with mark 70/70 cum laude, defending the thesis “Study of polymeric materials by means of solid state NMR”, supervisors prof. C.A. Veracini and prof. F. Ciardelli.
- Research activities as Ph.D. and post-doc student at:
  - group of prof. R.K. Harris, University of Durham (UK); CNR of Pisa; University of Pisa; Scuola Normale Superiore of Pisa.
- **2001-2014** assistant professor in Physical Chemistry at the Department of Chemistry and Industrial Chemistry, University of Pisa.
- **Since 2014** associate professor in Physical Chemistry at the same Department.
- **2001-now:** Teacher in courses of Physical Chemistry, Physical Chemistry of Materials, Solid State NMR Spectroscopy, Advanced Spectroscopic Methods, Errors and Analysis of Data.
- Leader of the solid-state NMR group at the University of Pisa.
- **2015** Gold medal award given by GIDRM (Italian Group of Discussions in Magnetic Resonances) and GIRM (Interdivisional Magnetic Resonances Group of the Italian Society of Chemistry) for “the important contributions given to the development and the application of solid state NMR spectroscopy for the study of the structural and dynamic properties of materials in condensed phases and for the great involvment in the diffusion of NMR and the formation of young researchers”.
- Supervisor of several Master and PhD theses at the University of Pisa.
- Scientific responsible of several contracts with chemical industries for solid-state NMR studies of materials and pharmaceuticals, as well as of national and international scientific projects.
- Organizer, member of the scientific board and chairman at tens of national and international meetings.
- President of GIDRM (Italian Group of Discussions in Magnetic Resonances) since January 2017.
- Research activities: NMR of liquid crystals, solid-state NMR of materials (polymers, composites, biomaterials, pharmaceuticals), nuclear spin-relaxation and molecular dynamics.
- Author of 120 papers on international journals and books (3 reviews and 6 papers on books and encyclopedias), about 50 invited lectures at meetings, institutions, and schools, and about 300 communications to conferences.

Solid state NMR spectroscopy: applications to the study of structural and dynamic properties of drugs and materials

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Solid state Nuclear Magnetic Resonance Spectroscopy is able to give detailed structural and dynamic information on solid systems and soft matter, representing one of the most powerful characterization techniques in several fields, ranging from pharmaceuticals to polymeric materials, from inorganic compound to hybrids, from liquid crystals to biological systems.
Structural information covers a space range from 0.1 to about 100 nm, and it concerns bondings, conformations, intra- and inter-molecular interactions, supramolecular spatial arrangements, molecular organization in crystalline and amorphous phases, interphases and interfaces, arrangement and dimensions of the different domains in heterophasic systems.
Furthermore, solid state NMR allows one to investigate molecular dynamics in a very detailed and quantitative way, especially for what concerns rotational and interconformational motions with characteristic correlation times ranging from seconds to picoseconds.
In this lecture, these features will be discussed showing in particular applications to drugs and polymeric/composite materials, mostly coming from the research activity of my group in Pisa.[1-5]

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Aziz AMINE

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Hassan II University of Casablanca, Morocco

Professor Aziz AMINE, has been Head of Department of Chemical Engineering and Environment of the Hassan II University of Casablanca during the period 1999-2003.

Professor Amine's research over the last 25 years has focused on sensors and biosensors and there use in Analytical Chemistry. He is author of more than 120 papers, 5500 citations and an h-index of 40. He has served as coordinator of several national and international research projects. He is a reviewer for several scientific international journals.

He is one of the Editors of the International Journal "Biosensors and Bioelectronics" published by Elsevier with Impact Factor 7.8.

Chairman of the International Workshop "Biosensors for Food Safety and Environmental Monitoring" organized every two years in Morocco (www.biocap.ma).

Chairman of the international conference "Journées Pratiques Francophones des Sciences Analytiques" organized every year in Marrakech (www.jpfsa.org).

He was invited speaker in several international congresses.

Recent Advances in Application of Molecularly Imprinting Polymers in Electrochemical Sensing

Aziz Amine

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Molecularly imprinted polymers (MIPs) are polymers that are fabricated via chemical, photochemical or electrochemical methods in the presence of a target template material of interest. This process results in the fabrication, following removal of the template, of polymeric structures containing cavities, complementary in shape and chemical properties with the template itself. Consequently, they can selectively recognize and bind specific target molecules through non-covalent strength interactions between the host matrix and the guest molecule, including van der Waals force, hydrogen bonding, electrostatic features, hydrophobic phenomena and metal-ion coordination. MIPs have found wide application in several areas including in separation, purification processes, chemical sensors, catalysis and drug delivery. Furthermore, molecularly imprinted polymers based electrochemical sensors were applied for the detection of various kinds of analytes from small such as metals ions, amino acids and emerging pollutants such as drugs and food additives to much larger proteins, bacteriophage or microbial cells. This technique have attracted more attention recently due to the various advantages that offers such as low cost preparation, great specific recognition ability, high stability and good sensitivity toward the target analyte.

Recently, we reported a novel electrochemical sensor for the selective detection of 17-β-Estradiol that considered as an endocrine disruptor. The developed sensing strategy was based on magnetic molecularly imprinted polymers (Mag-MIP) and showed a high specificity and sensitivity for 17-β-Estradiol. The developed Mag-MIP sensing system was applied successfully for the detection of 17-β-Estradiol in Mohammedia river water samples showing a satisfactory recovery. Moreover, another application of MIPs for detection of emerging pollutants such sulfonamides was reported recently. This MIP sensor was based on a hybrid nanomaterial sensing system based on magnetic molecularly imprinted polymer specific for sulfonamide detection. Magnetite nanoparticles (Fe_3O_4 NPs) were used due to their separation and pre-concentration properties combined with the molecularly imprinted polymer, which can capture and bind selectively sulfonamide. The molecularly imprinted polymer decorated magnetite nanoparticles sensing system operates via electrochemical impedance spectroscopy and exhibits a very high sensitivity towards sulfamethoxazole detection. The developed sensor was applied successfully for sulfamethoxazole determination in seawater samples. Ongoing work consist on development of a simple, low cost and selective electrochemical sensor for bacillus cereus spores detection. The proposed method is based on spore-imprinted polymer and exhibits a high specifity to bacillus cereus spores.

Ongoing work by our group involves the design and development of a novel artificial receptors based on imprinting technology for bisphenol A and heavy metal ions. A general overview on electrochemical methods based on MIPs for will be presented and discussed.

Pavel POSTNIKOV

Tomsk Polytechnic University,
Tomsk, Russia

Actual Position:

Assoc. Prof. Department of Technology of Organic Substances and Polymer Materials, Institute of Natural Resources.

Biography, education, and employment:

Pavel Postnikov studied Chemistry and Chemical Technology at Tomsk Polytechnic University and made Ph.D. in organic chemistry in 2011. He built up the group of Highly reactive organic substances in 2015 at the Department of Technology of Organic Substances and Polymer Materials.

Research Interests:

The main scientific interests are the chemistry of diazonium and iodonium compounds, their structure, reactivity and application for the design of new functional materials. A special focus of research is the transfer of synthetic methodologies of organic chemistry to the surface chemistry.

Awards and Honours:

- Laureate of the competition for the award of the Tomsk region in the field of education, science, health, and culture, 2012
- Engineer of the year, Russia, 2013
- Best Faculty Teacher of the year, 2013
- Scholarship of President of Russian Federation for young scientist, 2016

Diazonium chemistry: the universal way for the design of functional materials

Pavel Postnikov

Tomsk Polytechnic University, Tomsk, Russia

The application of diazonium chemistry and the design of the new functional materials will be discussed in a lecture. The great attention will be devoted to arenediazonium tosylates as a green, safe and highly reactive alternative to traditional arenediazonium tetrafluoroborates. The examples of the application of arenediazonium tosylates for the modification of thin metal films as well as polymer surfaces will be provided. The short inside to the synthesis of functional nanoparticles and its application in medicine will be presented.
In conclusion, the design of new functional materials with modified surfaces will be discussed. The secondary functionalization of surface functional groups provides a unique tool-box for the creation of highly-sensitive sensor system based on surface-enhanced Raman spectroscopy or allow to design the stimuli-responsive materials with controlled wettability governing by electric field or temperature.

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Vie Maritale, deux enfants

Jean-Marie Nedelec est Professeur de Chimie à SIGMA Clermont et développe ses activités de recherche au sein de l'Institut de Chimie de Clermont-Ferrand UMR CNRS 6296. Au sein de cet institut, il est responsable de l'équipe Matériaux Pour la Santé qui regroupe une vingtaine de personnes et a la particularité de mêler des chimistes des matériaux, des physico-chimistes, des biologistes, physiciens et des praticiens hospitaliers, chirurgiens orthopédiques et pharmaciens cliniciens.

Jean-Marie Nedelec est spécialiste de la chimie douce qui regroupe les méthodes alternatives pour l'élaboration de matériaux fonctionnels, ces méthodes s'inspirant largement du monde vivant et offrant des conditions d'élaboration originales qui les rendent compatible avec les constituants du vivant, molécules organiques, protéines, cellules, bactéries,… Popularisées dans les années 90 ces techniques et notamment le procédé sol-gel, ont conduit au développement des matériaux hybrides associant une composante minérale et une composante organique. Tous les champs applicatifs sont concernés. Au sein de l'ICCF, l'équipe du Pr Nedelec s'intéresse particulièrement au contrôle de la morphologie et de la porosité au sein de matériaux inorganiques et hybrides et à leurs applications dans le domaine médical en tant que substituts osseux, revêtements de prothèse, supports pour l'ingénierie tissulaire ou systèmes de relargage contrôlé de substances actives.

Ses travaux ont été reconnus internationalement à plusieurs reprises. En 2006 il participe à la finale de la « Young European chemist competition » au 1er congrès européen de Chimie où il est le seul représentant français. L'année suivante, il est reconnu par ses pairs en recevant l'International Ulrich Award 2007, récompense délivrée tous les deux ans à un chercheur ayant contribué de façon exceptionnelle au domaine du Sol-Gel. En 2010, l'année de sa nomination en tant que professeur à l'ENSCCF, il est nommé membre du prestigieux Institut Universitaire de France et devient le premier chimiste clermontois lauréat depuis la création de l'Institut en 1991.

Le Pr Nedelec est l'auteur de plus de 140 publications dans des revues internationales. Il est membre de nombreuses sociétés savantes où il exerce ou a exercé différentes responsabilités, président de la Division Chimie du Solide et membre du conseil d'administration de la Société Chimique de France et président sortant de l'International Sol-Gel Society.

Cationic substitutions in sol-gel derived calcium phosphates bioceramics: a fine tuning of biological properties

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Introduction

Human allograft is by far the best solution for repairing and reconstructing human skeleton. Upon trauma or as a consequence of a disease, clinical situations where the surgeon need bone defect filling materials are very frequent. When autograft or allograft is possible, this will always be the preferred solution. Unfortunately, in many cases, the surgeon has to use synthetic bone filling materials, either because the volume to be filled is too important or the co-morbidity an issue. Among all potential materials, bioceramics are the most interesting and commonly used materials. Behind the word bioceramics, mainly calcium phosphates are found but also some compositions of silicate glasses known as bioactive glasses[1]. The common feature of these two classes of materials is a kinetic modification of the material surface upon interaction with biological medium resulting in the integration of the implanted material (osteointegration). Some particular compositions are even bioactive meaning that they stimulate the body self-repairing process (osteoinduction).

Results and Discussion

Beyond these remarkable properties, it is highly desirable to bring multifunctionality to the bioceramic. Chemical doping though ionic substitution is a very efficient and convenient way to do it. In this lecture, we will focus on cationic substitution in hydroxyapatite and tricalcium phosphate. We will try to demonstrate how fine knowledge of crystallo-chemistry taking place in these materials is necessary to fully understand the material behaviour. Using the example of zinc, iron and copper doping in biphasic calcium phosphates[2,3], we will show that unsuspected doping mechanisms can be put forward. In this example, bioceramics with anti-inflammatory property can then be produced with a fine-tuning of the material. Other examples concerning materials with luminescent properties for diagnostic[4], antibacterial activity, anti-osteoporosis property and others will be presented

Conclusions

The take home message of this lecture is that upon thorough knowledge of materials chemistry taking place upon doping, it is now possible to reach on demand design of multifunctional biomaterials with improved properties.

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Dr. Ayhan ORAL is a faculty member in the Department of Chemistry at Çanakkale Onsekiz Mart University. He had a Master Science Degree from Çanakkale Onsekiz Mart University and earned his Ph.D. From Ege University. He conducted post-doctoral research with Prof. Yusuf Yagci at Istanbul Technical University (Turkey) and with Prof. Judit Puskas at The University of Akron (USA). Among his research interests are biopolymers, nanocomposites, electrospining, electrospraying, photopolymerization and ring opening polymerisation. He works as the director of Science and Technology Research and Application Center at Çanakkale Onsekiz Mart University.

Effects of Temperature on the Crystallinity and Morphology of Poly(Lactic)Acid - Phenylethyl alcohol

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Lately, biodegradable and biocompatible polymers have become a major concern of research studies and sectoral stakeholders, especially from an ecological perspective. Poly(Lactic acid) (PLA) is a bio-based, compostable, biodegradable, synthetic, and thermoplastic polymer. PLA has rapidly evolved into a competitive commodity material over the last decade. A considerable number of scientists have been carrying out studies to proliferate application areas of PLA. The control of crystallinity is among the ways to increase the number of application areas. The crystallization kinetic is affected by several factors, such as nucleating agents, temperature, molecular weight, etc. It is important to control the crystallinity grade in PLA and for this purpose different kinds of nucleating agents, such as organic and inorganic chemicals, were used. Phenylethyl alcohol (PEA) is a natural volatile oil which is isolated from rose. In the present study, the effects of temperature on crystallinity of PLA - PEA were investigated. The polymer films produced for the purpose of the study were prepared by solution casting method. PLA - PEA films, containing 20% of PEA (w/w), were then subjected to room temperature, 30^0C, 35^0C, 40^0C, and 45^0C to produce structural and morphological changes in crystals. The changes in crystallinity were observed via Optic Microscopy and Scanning Electron Microcopy.
Keywords: Poly(Lactic acid), Crystallinity, Phenylethyl alcohol, Temperature, Structural and morphological changes

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Education and Professional experience

· **2012 January : Assistant Professor-HDR section 33ème** UFR sciences et techniques**,** Tours University GREMAN Laboratory (UMR7347)
· **December 2011 : degree to lead research project (HDR)**
· **December 2002 : 1999-2002 Candidate for the PhD in Materials Sciences**

Scientific responsibilities
My scientific activity connected to the synthesis and to the structural and microstructural characterization as well as the study of the magnetic and electric properties allowed me :
Person in charge of the studies in transmission electronic microscopy of the pole CERTeM, based to the CEA (ATOMIC ENERGY COMMISSION) Ripault
Person in charge of the technique of electronic paramagnetic resonance of the pole CERTeM based in the EMA to the Faculty of Science of Tours
Person in charge of X-ray diffraction and of chemical division in the EMA to the Faculty of Science of Tours
Person in charge of dilatometer in the EMA to the Faculty of Science of Tours
Coordinator and local contact of the Federation MATV2L Centre region **-** Limousin
Member : Member of the 28-33-62nd committee (2008-2020)
Member of council of laboratory (2009-2012 et 2016-2020)
National committee CNU 33ème (2016-2020)
Purchase, specifications and implementation of experimental instruments**,** such as the dilatometer (2008), X-ray diffraction (2007 et 2012), electronic paramagnetic resonance (2005)
Member of Editorial board of "Pure and Applied Chemical Sciences" of the publishing Hindawi since March, 2012
Member of Editorial board of E-journal of Chemistry of the publishing by Hikari Ltd since January, 2013

Reporter : of project of the program JCJC SIMI 9 " Sciences of the engineering, the materials, the processes, the energy " publishing 2013 of the National Agency of the Research (ANR)

Scientific Projects :
2017-2019 CEA-Région **HYTEMPE** Mécanisme de vieillissement et dégradation des céramiques dans les piles électrochimiques (**coordinator**)
2016-2019 **Melusine** - Mécanisme de vieillissement et dégradation des céramiques utilisées pour les piles à combustible (**coordinator**)
2016-2019 **SIBERSITE** - Sodium Ion Batteries : Energy for the Strategy of Innovation in the Center Region (**responsable scientifique** pour le GREMAN- gestion finance et recherche)
2016- 2018 **microbag** - Micro-batteries tout-solide à base de graphène et de silicium budget
2015-2016 CEA-Région PROH2-**FIABIPERO** Mécanisme de vieillissement et dégradation des céramiques dans les piles électrochimiques (**coordinator**)
2014 2017 **COCONUT** Core-Shell" structure materials for multilayer ceramic capacitors with a high capacitance (**coordinator**)
2014 2016 **ALBATTROS** Alternatives en électrodes pour batteries ion sodium
2012 2015 **INTIM** Interfaces in thin films – Capacitors 2015
2012 2015 **BLADES** Batterie lithium durable avec électrodes silicium nanostructurée
2012 2014 **GRANITE** Guarinoites pour l'abattement des nitrates dans les eaux d'AEP
2011 2013 **FEDER/CONCERTO** Matériaux à permittivité colossale dans des condensateurs céramiques multicouches (**coordinator**)
2011 2013 **APR/OXYMORE** Optimisation d'oxydes multifonctionnels par ingénierie de zones de phases morphotropiques
2010-2013 **ANR/PNANO/3DCAP** Procédé d'enduction capillaire d'oxydes mixtes pour la réalisation de condensateurs 3D à très forte capacité spécifique intéressant l'électronique nomade

with several Scientific supervisions and Publications.

Synthesis, structural characterization and dielectric properties of $CaCu_3Ti_4O_{12}$ core-shell structure

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The $CaCu_3Ti_4O_{12}$ (CCTO) compound, a complex cubic perovskite-like oxide, has attracted considerable attention due to its colossal dielectric constant in the order of magnitude close to 10^5 with weak temperature dependence (100 K – 600 K) in the frequency range from 100 Hz to 100 kHz [1-2]. But the problem of this material is on one hand the dielectric loss ($\tan \delta \approx 0.1$) and on the other hand the low resistivity. If capacitors are made of CCTO, $\tan \delta$ should remain below 0.05 over a wide range of frequencies. It has been established that the origin of this high permittivity values comes from extrinsic polarization induced from internal barrier layer capacitance (IBLC). The IBLC model is described by a structure composed of semi-conducting grains and insulating grain boundaries. The grain boundaries play an important role in the dielectric behavior [3]. But, the nature of the barriers, boundaries grains still has not been fully characterized. So, to improve the weak points of the materials CCTO, we tried to obtain a new microstructure core-shell of CCTO particles coated by an insulating material (SiO_2, TiO_2, Al_2O_3…). The structure of these materials was characterized by XRD, Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and the dielectric properties by impedance spectroscopy.

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[3] S De Almeida-Didry, C Autret, C Honstettre, A Lucas, M Zaghrioui, Solid State Sciences 61 (2016) 102-105

Noureddine RAOUAFI

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Prof. Noureddine Raouafi obtained a Bachelor degree in Chemistry in 1999, then a research master degree and a PhD in Chemistry respectively in 2001 and 2004. Between 2005 and 2009, during the preparation of his habilitation, he worked a post-doctoral fellow with Prof. Christian Amatore at the Ecole Normale Supérieure d'Ulm (Paris, France) on the development of new redox molecules capable of chelating and triggered releasing of alkali and alkaline earth metals. He gained a Senior Lecturer position in 2011, and since then has been interested in the functionalization of nanomaterials for their use to design modified electrodes and electrochemical biosensors for sensing and biosensing purposes. Since 2016, he is a Professor of Analytical Chemistry and leading his own research group in the Laboratory of Analytical Chemistry and Electrochemistry at the Department of Chemistry of the Faculty of Science of Tunis (University of Tunis El Manar). In addition, he is also the head of the aforementioned laboratory. With his team, he has published more than 30 papers on the design of electrochemical biosensors and has patented three tools for detecting a biomarker of Alzheimer's disease based on modified gold nanoparticles, non-invasive detection of glucose in saliva by an ultra-sensitive glucose bioelectrode and the detection of ochratoxin A in coffee and wine samples.

Nanomaterials-Based Electrochemical Biosensors

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Nanomaterials are a pivotal part in the design of electrochemical biosensors since they allow: i) increasing the active surface area of the electrodes, ii) amplifying the electrochemical signal, ii) helping the adsorption of biomolecules to the electrode surface through covalent or non-covalent bond formation. In this talk, I will present some recent achievements during our investigations on the functionalization of nanomaterials and their uses to build electrochemical biosensors (Figure). In our group, we mainly focused on the development of tools for detection and quantification based on nanomaterials such as metallic nanoparticles, quantum dots, silica nanoparticles, graphene and carbon nanotubes, etc. conjugated to biological macromolecules such as DNA, enzymes, antibodies, ligands, etc.[1] We target applications in healthcare (detection of biomarkers of diseases such as Alzheimer's disease, cancer, ...), food security (mycotoxins, bacteria, etc.) and the environment monitoring (metals heavy, toxins, ...) [2]

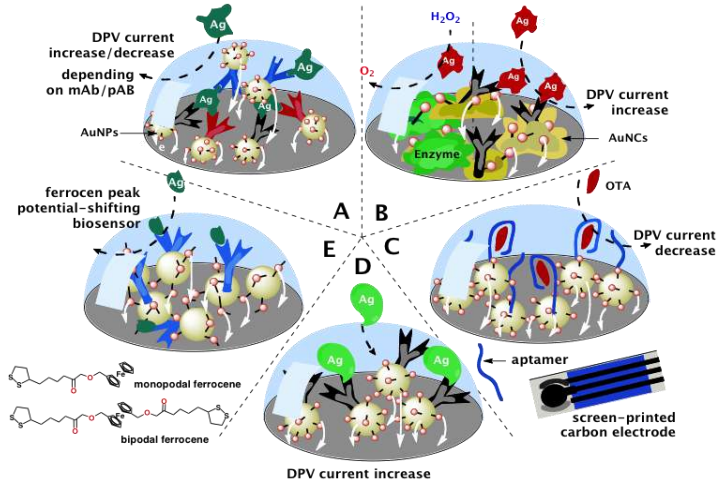

Figure : Some developed methods and their applications in biodetection

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[2] a) M. Saadaoui, I. Fernández, G. Luna, P. Díez, S. Campuzano, N. Raouafi, J. M. Pingarrón and R. Villalonga, *Anal. Bioanal. Chem*., **2016**, 408, 7321; b) A. Rabti, W. Argoubi and N. Raouafi, *Microchim. Acta*, **2016**, 183, 1227.

Oral Communications' List

(alphabetically authors' name)

Communications	Ref
D. Abid, I. Dhouib, Z. Elaoud *FSS - Sfax* Crystal structure, thermal behavior and spectroscopic studies of $[(C_2H_5)_4N]H_3Se_2O_8$	**OC1A**
M. Abid, S. Boufi, S. Bouattour *FSS - Sfax* Facile functionalization of cotton with nanostructured silver/titania for visible-light plasmonic photocatalysis	**OC2A**
R. Abid, G. Delahay, H. Tounsi *ENIS - Sfax* Influence of hydrothermal treatment on the activity of Ce-Cu-Y zeolite catalyst in the selective catalytic reduction of nitrogen oxides by ammonia	**OC3A**
M. Abid Derbel, H. Naïli, W. Rekik *FSS - Sfax* Crystal structure and thermal behaviour of $[\mu\text{-}(Br/Cl)_2\text{-}Cu_2(Br/Cl)_2(C_2H_8N_2)_2]$	**OC4A**
M. Abidi, S. López-Bernabeu, F. Huerta, F. Montilla, S. Besbes-Hentati, E. Morallón *FSB - Bizerte* Spectroelectrochemical study on the copolymerization of *o*-aminophenol and aminoterephthalic acid	**OC5A**
J. Ajmi, I. Nagazi,A. Harchani, A. Haddad *FSM - Monastir* Crystal structure of a New Strandberg-type selenomolybdate $(C_7N_2\ H_7)_4[Se_2Mo_5O_{21}].4H_2O$	**OC1B**
M. Ak *Pamukkale University, Turkey* Conducting polymers for electrochromic and biosensing applications	**OC6A**
Z. Aloui, M. Rzaigui, C. Ben Nasr *FSB - Bizerte* Synthesis, crystal structure, and spectroscopic studies of a new Bi(III) halide complex: $[C_7H_{10}NO]_2BiBr_5$	**OC2B**
A. Askri, K. Nahdi, A. Martínez-Arias *FSB - Bizerte* Copper promotion by $BaMO_3$ (M=Zr,Ce) perovskites applied for CO-PROX	**OC3B**

Communications	Ref
Z. Basdouri, B. Trojette, T. Milagros Tomas, M. Graia Mohsen, Larry R Falvello *FST - Tunis* Synthesis, crystal structure, Hirschfield surface analysis and thermal studies of a new hybrid compound $Zn(H_2O)_6$,2$[C_6H_{12}N_4Cl_3Zn]$,0.5H_2O	**OC4B**
W. Belam *FSB - Bizerte* Synthesis and physico-chemical characterizations of the nanomaterial $Li_{2.8}Hf_{1.755}Si_{0.9}P_{2.1}O_{12}$	**OC5B**
S. Ben Ali, M. Belhouchet *FSS - Sfax* Structural, vibrational and thermal properties of a new hybrid material based on bismuth	**OC6B**
I. Ben Hadj Sadok, F. Hajlaoui, N. Zouari *FSS - Sfax* Crystal packing, high-temperature phase transition and second-order nonlinear optical in an hybrid material: [(S)-$C_7H_{16}N_2$][$CUBR_4$]	**OC1C**
M. Ben Haj Salah, N. Mercier, N. Zouari *FSS - Sfax* Hybrid perovskites for optoelectronics	**OC2C**
W. Ben Mbarek, E. Pineda, L. Escoda, J.J. Suñol, M. Khitouni *FSS - Sfax* High efficiency decolorization of azo dyes by Ca-Al particles	**OC3C**
O. Ben Moussa, H. Chebbi, M.F. Zid *FST - Tunis* Synthesis, crystal Structure and Hirshfeld surface analysis of bis(2,6-diaminopyridinium)tetrachloridocobaltate(II)	**OC4C**
S. Ben Moussa, K. Kaaroud, M. Gruselle, B. Badraoui *FSM - Monastir* Synthesis and characterization of calcium hydroxyapatite functionalized with tyramine	**OC5C**
A.H. Benahsene, N. Ennaceur L. Bendjeddou *University Frères Mentouri - Constantine1, Algeria* Synthesis, structural study and NLO properties of a new hybrid compound of 4, 4'-diaminodiphenyl sulfone ligand	**OC6C**
T. Bouallegui, B. Ayed, A. Haddad *FSM - Monastir* Synthesis and characterization of a novel inorganic-organic hybrid material based on polyoxotungstate clusters: $(C_7H_{10}N_2)_4[H_4\beta - SbMo_6O_{24}].4H_2O$	**OC7A**

Communications	Ref
E. Bouaziz, S. Boujelben, N. Chniba-Boudjada, T. Mhiri, M. Boujelbene *FSS - Sfax* Crystal structure, Hirshfeld surfaces analysis, vibrational and thermal behavior of (2,6-Diaminopyridinium)perchlorate	**OC8A**
H. Bouchouicha, G. Panczer, Y. Guyot, D. De Ligny, R. Ternane *FSB - Bizerte* Co-doped Eu^{3+}/ Eu^{2+} calcium aluminosilicate glass-ceramics : Elaboration and luminescent properties	**OC9A**
S.E. Boughdiri, K. Ferchichi, N. Amdouni *FST - Tunis* Preparation of polypyrrole-polyaniline/$LiCoO_2$ nanocomposites as an electrode for Li-ion batteries	**OC10A**
Z. Bouhidel, S. Maza, A. Cherouana, S. Dahaoui *University Frères Mentouri - Constantine1, Algeria* Synthesis and structural study of a new triazole Schiff base	**OC11A**
M. Boutar, M.H. Noamane, N. Mattoussi, C. Desroches, R. Abidi, D. Luneau *FSB - Bizerte* Synthesis of new complexes of calix[4]arene and its characterization	**OC8B**
F. Braiek, M. Graia *FSS - Sfax* Structural study of $(C_5H_6N_2Cl)_2\,SiF_6$	**OC9B**
A. Chahbani, N. Zouari, N. Elkechaw *ENIG - Gabès* Microwave drying of green peas (*Pisumsativum L.):* Drying kinetics and physical quality	**OC10B**
M. Chaker, K. Horchani-Naifer, M. Ferid *CNRSM - Borj Cédria* Synthesis, characterization and physical properties of $KErP_2O_7$	**OC11B**
D. Cheikh, F. Garcia Villen, H. Majdoub, M. Benna Zayani, C. Viseras *FSB - Bizerte* Tunisian montmorillonite clay as drug carrier	**OC7C**
Y. Coskun, I. Tas, A. Oral, *Canakkale Onsekiz Mart University, Çanakkale, Turkey* Investigation of the allelopatic effects of thymol, carvacrol and alpha-terpinene as natural Chemicals	**OC11C**

Communications	Ref
O. Dachraoui, A. Atyaoui, F. Manaii, H. Maghraoui Meherzi, L. Chokri *FST - Tunis* Validation and uncertainty estimation of an ICP-AES method for the determination of bismuth in bismuth sulfide thin films prepared by chemical bath deposition	**OC8C**
M. Dardouri, F. Ammari, F. Meganem *FSB - Bizerte* Adsorption of cadmium (II) and lead (II) from water by new cross-linked reusable polystyrene adsorbents	**OC9C**
S. Dgachi, A. Maatar Ben Salah, M.M. Turnbull, T. Bataille, H. Naïli *FSS - Sfax* *In situ* X-Ray diffraction studies, thermal behavior and magnetic properties of organic-inorganic copper (II) complexs	**OC10C**
M. Dhifi, S. Thabet, A. Haddad *FST - Tunis* A Novel organic-inorganic hybrid based on [PW12O40] polyanion $[C_4H_7N_2]2[HPW12O40]$	**OC12A**
I. Dhouib, P. Guionneau, S. Pechev, Z. Elaoud *FSS - Sfax* Investigations on $[(CH_2CH_2CH_3)_4N]_2[M^{II}Cl_4]$ halogenometallate complexes with M^{II} = Co and Mn: Crystal structure, spectroscopy studies and thermal behavior	**OC13A**
N. Dissem, B. Artetxe, J.M. Gutiérrez-Zorrilla, L. San Felices, O. Castillo, L. Lezama, A. Haddad *FST - Tunis* Cluster transformation triggered by evacuation of solvent molecules in a new hybrid arsenomolybdate-metalorganic framework with permanent microporosity	**OC14A**
S. Dkhili, S. López-Bernabeu, F. Huerta, F. Montilla, S. Besbes-Hentati, E. Morallón *FSB - Bizerte* Electrochemical synthesis of Poly(2-Aminoterephthalic acid-co-Aniline) on Pt electrodes. Ascorbic Acid and dopamine oxidation	**OC15A**
A. Elmhamdi, L. Pascual, R. Castañeda, A. Kubacka, K. Nahdi, A. Martínez-Arias *FSB - Bizerte* Effect of the presence of CO_2 in the reactant mixture for $CuO / CeO_2 / MgAl_2O_4$ catalysts in CO-PROX	**OC12B**

Communications	Ref
A. Fadhlaoui, S. Kouas, H. Dhaouadi *FSB - Bizerte* $Bi_xCe_{1-x}PO_4$ (x = 0.00, 0.02, and 0.08) nanorods: structural, electrical, optical, and electrochemical properties	**OC13B**
M. Fadhli, I. Khedher, J.M. Fraile, Z. Ksibi *FST - Tunis* Asymmetric epoxidation of styrene with TBHP catalyzed by bis(oxazoline)-vanadyl-laponite materials	**OC14B**
R. Fezai, M. Rzaigui *FSB - Bizerte* Synthesis, crystal structures and physicochemical study of two hybrid cyclohexaphosphates	**OC15B**
D. Fredj, C. Ben Hassen, N. Chniba Boudjada, T. Mhiri, N. Zouari, M. Boujelbene *FSS - Sfax* Fabrication and characterization of two new hybrid materials	**OC12C**
H. Galai, R. Kalfat *INRAP - Sidi Thabet* Preparation and characterization of amorphous solid dispersion of poorly soluble drug (Ibuprofen, Atorvatatine and Celecoxib)	**OC13C**
A. Gharbi, H. Oudadesse, B. Lefeuvre, N. Jouini, H. El Feki *FSS-Sfax* Bioactive glasses development:Mechanical study and *"In vitro"* simulation	**OC14C**
A. Guesmi, H. Marouani *FSB - Bizerte* Synthesis, crystal structure and Hirshfeld surface analysis of a novel organic-inorganic hybrid material $C_8H_{14}N_2(HSO_4)_2$	**OC16A**
O. Guselnikova, P.S. Postnikov, O. Lyutakov, V. Svorcik *Tomsk Polytechnic University - Russia* Diazonium chemistry for modification of polymeric materials	**OC15C**
R. Hamacha, A. Argoub, C. Bachir, B. Boukoussa, A. Bengueddach *Université d'Oran1 - Algeria* Synthesis of MIL-101@g-C_3N_4 nanocomposite for enhanced adsorption capacity towards CO_2	**OC21A**
I. Hamdi, T. Roisnel, H. Naïli *FSS - Sfax* Synthesis, crystal structure and characterization of a new organic-inorganic hybrid $(C_6H_{10}N_2)_2[MNCL_6]\cdot 2H_2O$	**OC17A**

Communications	Ref
D. Harbaoui, M.M.S. Sanad, C. Rossignol, E.K. Hlil, S. Obbade, N. Amdouni *FST - Tunis* Magnetic and electrochemical properties effects of iron–aluminum substitution in new sodium nickel polyphosphate cathode materials $Na_2Ni_2(Fe,Al)(PO4)_3$	**OC18A**
S. Hassen, H. Chebbi, M.F. Zid, Y. Arfaoui *FST - Tunis* Synthesis of 2-guanidinobenzimidazolium dibromide monohydrate salt and investigation of its structure via x-ray diffraction, spectroscopic measurements and DFT calculations	**OC19A**
K. Hchicha, N. Mhadhbi, H. Naïli *FSS - Sfax* Growth and structural characterizations of $La_2Mo_{2-y}S_yO_9$ $(0.1 \leq y \leq 0.6)$, a new solid solution on sulfur-stabilized $La_2Mo_2O_9$	**OC20A**
N. Hfidhi, O. Kammoun, R. Pelka, M. Fitta, H. Naïli *FSS - Sfax* Structure features, thermal analysis, model and experiment magnetic behavior of tetranuclear oxo and sulfato bridged copper (II) aggregate	**OC16B**
R. Ismail, J. Arfaoui, Z. Ksibi, G. Delahay, A. Ghorbel *FST - Tunis* Novel Ag/Ce-ZrO2 catalyst for the low temperature total oxidation of toluene	**OC17B**
R. Jaballi, W. Maalej, P. Horcajada, Z. Elaoud *FSS - Sfax* Experimental study on the structure, vibrational and thermal properties of crystal structure of tetraaqua-(5,5'-Dimethyl-2,2'-Bipyridyl)nickel(II) selenate	**OC18B**
W. Jabeur, N. Mhadhbi, H. Naïli *FSS - Sfax* Innovative solid oxide fuel cells based on La_2MoSO_9 electrolyte: synthesis and characterization	**OC19B**
B. Jalel, N. Ennaceur, R. Henchiri *FSG - Gafsa* Synthesis, structure and characterization of a hybrid centrosymmetric material (4-Dimethylaminopyridinium nitrate gallic acid monohydrate)	**OC20B**
N. Jamai, M. Rzaigui, S. Akriche Toumi *FSB - Bizerte* Synthesis and biophysicochemical characterization of a new organically templated cobaltoborate $(C_4H_{12}N_2)[CoB_{12}O_{14}(OH)_{12}].6H_2O$	**OC21B**

Communications	Ref
D. Jammazi, S. Akriche *FSB - Bizerte* Synthesis, structure supported with Hirshfeld surface analysis and characterization of 3D-supramolecular based decavanadate compound	**OC16C**
W. Jedidi, J. Arfaoui, Z. Ksibi, A. Ghorbel *FST - Tunis* Novel Co/ZrO_2 aerogel catalysts for the total oxidation of methane: Effect of Co content	**OC17C**
M. Jemmali, N. Bouchaala, S.Walha, K. Zehani, A. Ben Salah, L. Bessais *FSS - Sfax* Isothermal Cross-Sections of Nd-Fe-Ge System at 800°C	**OC18C**
I. Jendoubi, R. Ben Smail, M. Miroslaw, M.F. Zid *FST - Tunis* Synthesis, characterization and visible-light-driven photocatalytic performance of $Na_2Mn_2(MoO_4)_3$	**OC19C**
R. Jlassi, H. Naïli, T. Rüffer, H. Lang, A. J. L. Pombeiro, W. Rekik *FSS - Sfax* Synthesis, crystal structure, and catalytic properties in diastereoselective nitroaldol (Henry) reaction of New Zinc(II) and Cadmium(II) compounds	**OC20C**
N. Jraba, T. Makhlouf, H. Tounsi *FSS - Sfax* Valorization of aluminum Chips into γ-Al_2O_3 and η-Al_2O_3 with high surface areas via the precipitation Route	**OC21C**
I. Kadri, W. Rekik, H. Naïli *FSS - Sfax* (C6H14N2)[CO(H2O)6](S0.83SE0.17O4)2: IR spectroscopy, 3D H-bonded structure and thermal behaviour	**OC22A**
S. Kefi, D. Boa, Dalila Hellali, H. Zamali *FST - Tunis* Thermodynamic modeling of the ($LiNO_3$+$NaNO_3$+$TlNO_3$) ternary system	**OC23A**
L. Khedhiri, M. Rzaigui, C. Ben Nasr *FSB - Bizerte* A Hirshfeld surface analysis, crystal structure and infrared characterization of a new organic cyclohexaphosphate, $(C_8H_{12}N)_4(Li)_2(P_6O_{18})(H_2O)_6$	**OC24A**

Communications	Ref
T. Khelfa, M. A. Rekik, J.A. Muñoz-Bolaños, J.M. Cabrera-Marrero, M. Khitouni *FSS - Sfax* Influence of grain boundary misorientations on the mechanical behavior of a AA6060 alloy processed by ECAP	**OC25A**
N. Khitouni, R. Daly, L. Escoda, N. Llorca-Isern, J.J. Suñol, M. Dammak *FSS - Sfax* The magnetic and structural properties of nanostructured $Fe_{50}Co_{25}Ni_{25}$ alloy prepared by mechanical alloying	**OC26A**
K. Klai, K. Kaabi, C. Jelsch, E. Winger, C. Ben Nasr *FSB - Bizerte* A Hirshfeld surface analysis, synthesis, structure and characterization of a new Ni(II) complex with the bidentate ligand homopiperazine	**OC22B**
K. Labidi, M. Zrida, Z. Cao, A. Murphy, A.H. Hamzaoui, D.M. Devine *CNRSM - Borj Cédria* Alfa fiber polypropylene composites: effect of fiber alkaline extracted and steam-exploded fibers content on Mechanical properties	**OC23B**
R. Lachhab, M.A. Rekik, H. Azzedine, T. Baudin, A.L. Helbert, F. Brisset, M. Khitouni *FSS - Sfax* Microstructure evolution of Fe-48wt%Ni alloy processed by equal channel angular pressing process	**OC24B**
A. Lassoued, B. Dkhil, S. Ammar, A. Gadri *FSG - Gabès* Synthesis, structural, morphological, optical and magnetic Characterization of Spinel ferrites ($Cu_xCo_{0.5-x}Ni_{0.5}Fe_2O_4$) nanoparticles by co-precipitation method	**OC25B**
M.S. Lassoued, A. Gadri, S. Ammar, A. Ben Salah, S. García-Granda *FSG - Gabès* Synthesis, crystal structure, spectroscopic characterization and magnetic properties of new hybrid compound aquatrichlorocuprate 2-methyl imidazolium	**OC26B**
H. Ltaief, A. Mahroug, M. Belhouchet *FSS - Sfax* Chemical synthesis, crystal structure, vibrational spectroscopy and thermal analysis of new hybrid compound $C_{12}H_{14}N_2O_2S(Hg_4Cl_{12})_{0.5}.H_2O$	**OC22C**
A. Maalaoui, S. Toumi Akriche *FSB - Bizerte* Synthesis, bio-physic-chemical properties of a di-V-substituted type Lindqvist polyoxotungstate	**OC23C**

Communications	Ref
M. Mathlouthi, A.C. Dhieb, M. Rzaigui, W. Smirani *FSB - Bizerte* Crystal structure, dielectric characteristics and conduction mechanism of a new hybrid material, tetra(phenylpiperazinium) decachlorotriplumbate(II)	**OC24C**
C. Mchiri, S. Acherar, C. Frochot, P. Arnoux, H. Nasri *FSM - Monastir* Synthesis, photophysics characterization and X-ray molecular structure of the 2-methylimidazole tetrakis(tert-butylphenyl)porphyrinato)cadmium(II). Complex	**OC25C**
R. Mejri, J.C. Dias, S.B. Hentati, S. Lanceros Mendez *FSB - Bizerte* The effect of the imidazolium-based ionic liquid type in the performance of polymer actuators	**OC26C**
M. Mhadhbi *INRAP - Sidi Thabet* Microstructural changes in nanocrystalline $(Ti_{0.9}Cr_{0.1})C$ powders obtained by mechanical alloying	**OC27A**
I. Moghded, A. Mbarek, H. Naïli *FSS - Sfax* A new polymeric copper (I) complex containing hexafluorophosphate groups. Synthesis and crystal structure determination of $\{[Cu(PF_6)(H_2O)_4]\}_n$	**OC28A**
H. Mrabet, M. A. Cherbib, I. Khattech *FST - Tunis* Effect of the addition of BaO on the structural, thermochemistry properties of phosphate glasses	**OC29A**
R. Nasri, M.F. Zid *FST - Tunis* Electrical and vibrational studies of $K_{0.4}Na_{3.6}Co(MoO_4)_3$	**OC30A**
H. Nefzi Touil, F. Sediri *IPEIT - Tunis* Electrochemical properties of vanadium oxide nanourchins as cathode material	**OC27B**
S. Rhouma, S. Said, A. Megriche, C. Autret-Lambert *FST - Tunis* Enhancement of dielectric properties of $CaCu_3Ti_4O_{12}$ ceramics by Sr and Ni doping ions	**OC28B**

Communications	Ref
S. Said, C. Autret, M. El Amrani, A. Megriche *FST - Tunis* Influence of sintering conditions on microstructure and electrical properties of $CaCu_{2.9}Ni_{0.1}Ti_{3.9}Ni_{0.1}O_{12}$ (CCNTNO) ceramics	**OC29B**
S. Saidani, L. Ben Tahar, A. Smith *Université de Limoges, France / FSB/FST* A new route for active Belite P-Ti-Rich clinker	**OC30B**
M. Saidi, M. Jemmali, S. Walha, A. Kabadou, L. Bessais *FSS - Sfax* Phase equilibria of the Gd-Fe-Cu system at 800°C	**OC27C**
H. Sehimi, M.F. Zid *FSG - Gabès* A novel honeycomb-like Mn(II) μ-oxalato-bridged 2D coordination polymer: Crystal structure, spectroscopic, thermal and magnetic properties.	**OC28C**
A. Shili, F. Hajlaoui, N. Zouari *FSS - Sfax* Synthesis, crystal packing and second-order nonlinear optical in an hybrid material: $[(R)\text{-}C_7H_{16}N_2]_2[ZnBr_4]_2{\cdot}2H_2O$	**OC29C**
M. Sonni, R. Marzouki, M.F. Zid *FST - Tunis* Elaboration, structural study and validation (CHARDI and BVS) of a new lyonsite $(Na_{0.4}Li_{0.6})(Li_2Cr)(MoO_4)_3$	**OC30C**
F.Z. Souissi, H. Ettoumi, M. Barré, M. Toumi *FSS - Sfax* Preparation and caracterisation of potassium-aluminium phosphate glasses	**OC31A**
M. Tarhouni, B. Jamoussi, V. Sol, F. Dumoulin *FSB - Bizerte* Designing of a novel synthetic mitochondrion-targeting tri phosphonium-functionalized photosensitizing phthalocyanine	**OC32A**
H. Tlili, S. Walha Dammak, S. Elleuch, B. Fares Ali, M. Awad Hassan, H. Naili *FSS - Sfax* Structural, biological activities, DFT and optical studies of a new non-centrosymmetric hybrid material $(C_4H_{12}N_2)[COBr_4]$	**OC33A**
S. Toumi, S. Akriche *FSB - Bizerte* A new synthesized based decavanadate compound: Structural, Hirshfeld surface, spectroscopic and antibacterial analyzes	**OC34A**

Communications	Ref
M. Trimeche, N. Etteyeb, M. Romdhane, A. Romane, X.R. Nóvoa *FSG - Gabès* Anticorrosive action of some trees cultivated in Tunisian arid zones on steel rebar's in alkaline chloride solution	**OC35A**
A. Wederni, W. Ben.Mbarek, L. Escoda, J.J. Sunol, M. Khitouni *FSS - Sfax* Effects of Cu additions on the martensitic transformation of Ni–Mn–Sn shape memory alloys	**OC31B**
K. Zaara, M. Chemingui, J.J. Suñol, M. Khitouni *FSS - Sfax* Fe-Ni-Mn alloy prepared by mechanical alloying and arc melting process	**OC32B**
M. Zaghdoudi, F. Fourcade, D. Floner, H. Maghraoui Meherzi, A. Amrane, F. Geneste *FST - Tunis* Preparation of stable Gold Nanoparticles-Modified graphite felt electrode	**OC33B**
M. Zannen, M. Benyoussef, A. Lahmar, J. Belhadi, H. Khemakhem, M. El Marssi *FSM - Monastir* Electrostatic energy storage in antiferroelectric like perovskite	**OC34B**
C. Zidani, B. Benyoucef *University of Tlemcen, Algeria* Design and investigation of a conical solar furnace powered by optical fibers	**OC35B**
R. Zrelli-Annabi, F. Chehimi-Moumen, H. Moutaabbid, O. Kurakevych, Y. Le Godec, D. Ben Hassen-Chehimi, M.Trabelsi-Ayadi *FSB - Bizerte* Synthesis, crystal Structure and thermal decomposition of $[HGdP_2O_7 \cdot 2H_2O] \cdot NH_3$. Electric properties of $HGdP_2O_7$ and $Gd_2P_4O_{13}$	**OC7B**

Oral Communications' Abstracts

Program of
Monday
18 December 2017

Crystal structure, thermal behavior and spectroscopic studies of $[(C_2H_5)_4N]H_3Se_2O_8$

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Ongoing studies aimed at developing a novel proton conducting solids, resulted in the new organic-inorganic compound $[(C_2H_5)_4N]HSeO_4(H_2SeO_4)$. The crystals have been prepared by slow evaporation at room temperature. Crystal structure, thermal characterization, FTIR, Raman and electrical measurements are given to characterize this compound. X-ray study showed that the crystal belongs to the $P2_1/c$ space group of monoclinic system with lattice parameters: a = 19,705 (1) Å; b = 7,5420 (3) Å; c = 19,199(1) Å, β=115° and Z=4. The structure consists of infinite two-dimensional planes built by $[HSeO_4]^-$ and H_2SeO_4 tetrahedra inter-linked via strong hydrogen bonds (O-H…O) giving birth to trimmers. The complex impedance of the compound has been investigated in the temperature range of 290–373 K and in the frequency range 1 KHz and 13.10^6 Hz. The impedance plots show semicircle arcs at different temperatures, and an electrical equivalent circuit has been proposed to explain the impedance results. Conductivity measurements show that this material is a good protonic conductor.

Graphical abstract

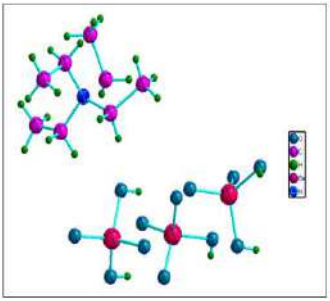

Fig1: The asymmetric unit of [(C2H5)4N]HSeO4(H2SeO4)

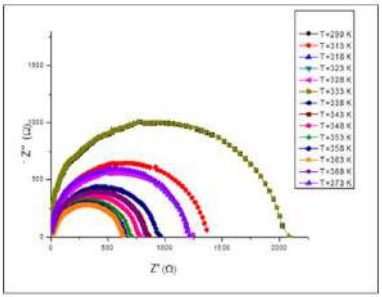

Fig 2: Complex impedance diagrams - Z''(Ω) versus Z'(Ω) over the temperature range 290– 373 K.

Keywords: Organic-inorganic compound, Crystal structure, spectroscopic measurements.

Facile Functionalization of Cotton with nanostructured Silver/Titania for Visible-Light Plasmonic Photocatalysis

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In recent years, many attentions have been focused on the surface functionalization of cotton with nanoparticles which is an area of growing interest to create new fabrics properties such as antibacterial, anti-smell, self-cleaning, UV protection, photocatalytic activity.
In the present work, a simple, reliable and cost-effective approach to functionalize cotton fabrics with Ag-TiO_2 nanoparticles strongly bound to the fibres and with visible-light responsive photo-activity is presented. This new class of photocatalytic materials, named plasmonic photocatalysts, is one of the most important breakthrough in photocatalysis using semiconductors and offers an alternative to enhance the photocatalytic activity under visible light irradiation.
The hybrid cotton-Ag-TiO_2 fabrics were characterized by Raman, AFM, XPS, GSDR, and LIL to confirm the generation of metallic Ag nanoparticles and crystalline TiO_2 and investigate how the concentration of Ag and TiO_2 precursors affected the morphology and the luminescence properties of the nanostructured layer grafted on the cotton fibres.
The photocatalytic activity of the cotton-Ag-TiO_2 hybrid systems was evaluated by the discoloration of Remazol Brilliant Blue R in water under visible light irradiation.
The extended photocatalytic activity to the visible is here explained by a synergistic effect of both the excitation of the Ag NPs plasmon resonance by visible light and a delayed electron-hole recombination rate caused by Ag NPs, as it can be observed by UV absorption.

Keywords: Plasmonic photocatalyst; silver-TiO_2; cotton; visible light; surface functionalization.

Influence of hydrothermal treatment on the activity of Ce-Cu-Y zeolite catalyst in the selective catalytic reduction of nitrogen oxides by ammonia

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This work was devoted to the study of the hydrothermal stability and structural changes accompanying deactivation of bimetallic zeolitic catalyst Ce-Cu-Y tested in the selective catalytic reduction of nitrogen oxides by ammonia (NH_3-SCR). The parent zeolite Na-Y (Si/Al = 1.38 and S_{BET} = 649 m^2/g) used as support for the preparation of catalysts was obtained from industrial sodium metasilicate and aluminum scraps. Firstly, Na-Y zeolite was exchanged with ammonium chloride NH_4-Y (S_{BET} = 576 m^2 / g) and then was impregnated by copper and cerium chlorides to prepare the Ce-Cu-Y catalyst. Hydrothermal treatment of catalysts was carried out in a quartz fixed-bed microreactor for 4 hours at 700 °C and 800°C in flowing air. The fresh and steamed catalysts were tested in the NH_3-SCR of NOx ($NO+NO_2$) in the temperature range of 200-550°C. The reaction gas composition was: [$NO+NO_2$] = 1000 ppm (NO/NO_2=1), [NH_3] =1000 ppm, [O_2] =8% and [H_2O] =3.5%. The results of the catalytic tests before and after hydrothermal treatment are summarized in **figure 1**. The fresh catalyst Ce-Cu-Y presented a NOx conversion over 80% in the whole temperature range (200-550°C). After the hydrothermal treatment at 700°C, there is a decrease of NOx conversion at temperatures below 500°C. By increasing the temperature of the hydrothermal treatment to 800°C, NOx conversion strongly decreased to 45% in comparison with the fresh sample and hydrotreated at 700°C (Ce-Cu-YHT700). Characterization of the hydrotreated solids showed that the deactivation in the NH_3-SCR of NOx was attributed either to the changes in copper and cerium species and to the collapse of the zeolite framework.

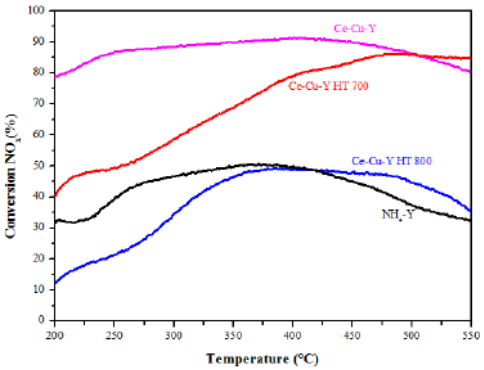

Figure 1: NOx conversion of the carrier (NH_4-Y), the fresh catalyst (Ce-Cu-Y); the hydrotreated at 700°C (Ce-Cu-YHT700), and the hydrotreated at 800°C (Ce-Cu-YHT800)

Key Words: Zeolite, Ce-Cu-Y, hydrothermal treatment, NH_3-SCR, aluminum scraps.

CRYSTAL STRUCTURE AND THERMAL BEHAVIOUR OF $[\mu\text{-}(Br/Cl)_2\text{-}Cu_2(Br/Cl)_2(C_2H_8N_2)_2]$

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A new organic-inorganic hybrid material has been synthesized and crystallographically characterized by single-crystal X-ray diffraction. At room temperature, this new compound crystallizes in the monoclinic symmetry (space group $P2_1/m$) with the following unit-cell parameters: a = 6.927(3) Å, b = 5.884(3) Å, c = 8.399(4) Å, β = 94.856(13)°, V = 341.1(2) Å^3 and Z = 2. The 1D structure of the title compound consists of infinite dimeric chains running along the crystallographic b axis. These chains are constructed from dimeric units $[Cu_2(Br/Cl)_2(C_2H_8N_2)_2]$ linked together by sharing (Br/Cl) atoms. N-H…Br/Cl hydrogen bonds ensure the connection between the dimeric chains and the cohesion of the structure. The thermal decomposition of the precursor proceeds through two stages giving rise to the copper oxyde.

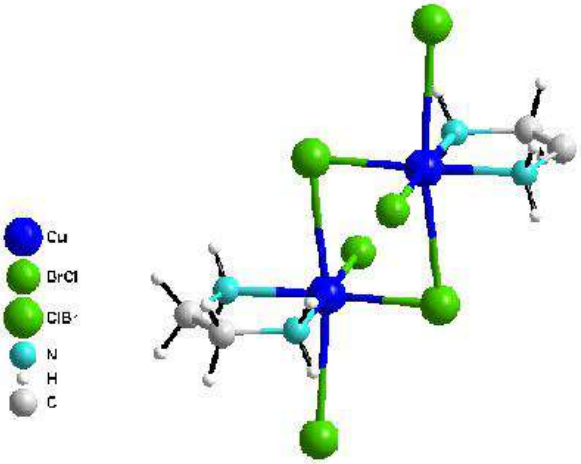

Figure: dimeric unit.

Key words: Hybrid material, X-ray diffraction, IR spectroscopy, thermogravimetric measurement.

Spectroelectrochemical study on the copolymerization of *o*-aminophenol and aminoterephthalic acid

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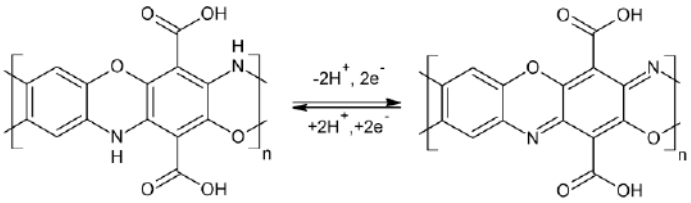
The electrochemical copolymerization of aminoterephthalic acid (ATA) and *o*-aminophenol (OAP) was carried out on platinum electrodes in acidic medium. The obtained material, poly(ATA-*co*-OAP), can be deposited by cyclic scanning of the potential in a wide potential range and shows significant electroactivity. However, it was observed that a copolymer with less over oxidation defects is obtained at inversion potentials where only OAP monomer (and not ATA monomer) is oxidized. The successful incorporation of unoxidized ATA monomers to the growing copolymer chain was confirmed by *in situ* FTIR spectroscopy and XPS, which suggested a monomer ratio close to 1:2 (ATA:POAP) within the obtained copolymer material. The redox switching of poly(ATA-*co*-OAP) in acidic medium involves reversible benzenoid-quinoid and phenoxazine-phenoxazone conversions but carboxylic groups do not participate actively on these transformations.



Scheme1. Chemical structure of poly(ATA-*co*-OAP).

Conducting Polymers for Electrochromic and Biosensing Applications

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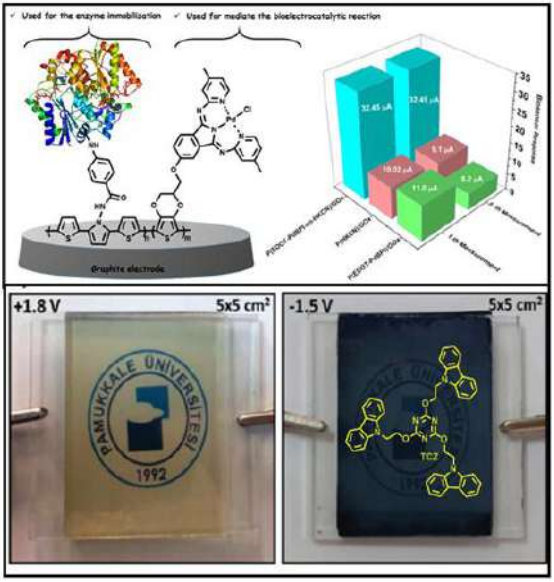
Thanks to its interesting optical and electrical properties, conducting polymers is one of the best materials to use in potential technological applications. Over the past two decades, a wide variety of conjugated polymers with novel properties have been synthesized. Many of these newly synthesized conjugated polymers are used in the development of thin film devices such as OLEDs, photovoltaics, cells, electrochromic devices, as well as in the fields of biosensors, metal sensors and chemical separation.

In this work, we have designed and synthesized new electroactive functional monomers. Optical and electrical properties of conducting polymers obtained by electropolymerization of these monomers have been characterized. Potential usages of these functional conducting polymers in electrochromic, biosensors and metal sensor platforms have been investigated.



Usage of the newly synthesized conductive polymers in biosensors and electrochromic applications

Keywords: conducting polymer, electrochromic, biosensor, electrochemistry, spectroelectrochemistry

Synthesis and characterization of a novel inorganic-organic hybrid material based on polyoxotungstate clusters: $(C_7H_{10}N_2)_4[H_4\beta - SbMo_6O_{24}].4H_2O$

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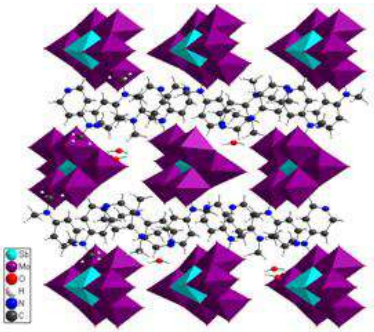
Polyoxometalates (POMs) as discrete anionic metal-oxygen clusters are ubiquitous inorganic building blocks possessing a wide range of the composition, highly alterable sizes, shapes, and charge densities [1]. The inorganic compounds have attracted great attention in the past years because of their potential applications in catalysis, photochemistry and magnetism [2]. In addition, they can be tuned at molecular level and exhibit fascinating structures and properties [3]
A new organic–inorganic hybrid compound based on β-Anderson polyoxotungstate, $(C_7H_{10}N_2)_4[H_4\ \beta\text{-}SbMo_6O_{24}].4H_2O$ have been isolated and characterized by single-crystal X-ray diffraction, infrared, ultraviolet spectroscopy, cyclic voltammetry and Thermogravimetric analysis (TGA). This compound crystallized in the monoclinic system, space group $P2_1/c$, with a = 12.4806(4) Å, b = 22.9845(6) Å, c = 17.2853(5) Å, β = 110.6(°) and Z=2. The crystal structures of the compounds exhibit three-dimensional supramolecular assembly based on the extensive hydrogen bonding interactions between organic cations, water molecules and β-Anderson polyoxoanions. The infrared spectrum fully confirms the X-ray crystal structure. Furthermore, the Uv spectrum and the electrochemical behavior were investigated.



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Crystal structure, Hirshfeld surfaces analysis, vibrational and thermal behavior of (2,6-Diaminopyridinium)perchlorate

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One novel organic-inorganic hybrid supramolecular assemblies $(C_5H_8N_3)ClO_4$ has been synthesized by slow evaporation method at room temperature and characterized by X-ray single crystal diffraction. The new hybrid compound crystallizes in the monoclinic system with the non-centrosymmetric space group $P2_1$ and the following parameters are a = 5.017(5)Å, b = 10.363(5)Å, c = 8.238(5)Å, β = 105.532° with Z = 2 and V=901.4(9)Å^3. In the structure, the supramolecular organic cations $(C_5H_8N_3)^+$ and inorganic ClO_4^- anions are arranged alternately and linked by N-H…O hydrogen bonds. It is worthy to note that the ClO_4^- are linked to form zero-dimensional inorganic chain through N-H…O hydrogen bonds along b-axis. Hirshfeld surface analysis was used to visualize the fidelity of the crystal structure. This method permitted the identification of individual types of intermolecular contacts and their impact on crystal packing. Thermal studies by thermogravimetric analysis (TGA) established that the compound undergoes two mass loss ending with total degradation of the title compound. The existence of vibrational modes correspond to the organic and inorganic groups are identified by the Raman and infrared spectroscopy in the frequency ranges 500-0 cm^{-1}, 4000-0 cm^{-1} and 4000-500 cm^{-1}, respectively.

Keywords: Hybrid material; X-ray diffraction; Hirshfeld surfaces; Vibrational spectroscopy

Co-doped Eu^{3+}/ Eu^{2+} calcium aluminosilicate glass-ceramics : Elaboration and luminescent properties

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The potentiality of Eu^{2+}-doped calcium aluminosilicate (CAS) glass showed that this material is a promising phosphor toward the developments of a smart lighting. However, the weak luminescence of phosphor glass can limit their application. Glass-ceramics doped with REE (rare-earth element) may improve luminescence intensity and also be a good alternative for white light emitting diodes (W-LEDs) because such materials combine the excellent luminescent environment of crystal and the good chemical stability of glass matrix.
Eu^{3+} and Eu^{2+} doped calcium aluminosilicate glass-ceramics have been prepared by heat-treating a glass of the SiO_2-Al_2O_3-CaO-MgO system. The control of crystallization by X-ray diffraction (XRD) and Raman spectroscopy showed that melilite, $Ca_2Mg_{0.25}Al_{1.25}Si_{1.25}O_7$ crystallized as the major phase and anorthite, $CaAl_2Si_2O_8$ as the minor phase at 900°C. Luminescent properties were investigated by emission, lifetime and the color coordinates were calculated. The results showed that, Eu^{3+} emission spectra in these glass-ceramics were strongly modified compared to that of the glass because Eu^{3+} was incorporated into the crystalline phase. On the other hand, the $Eu^{3+}$was reduced to Eu^{2+} during heating of the glass and therefore, emission intensity of Eu^{2+} was enhanced and increased with increasing heat-treatment duration. The color coordinates shift after annealing suggesting that crystallization process appears to be a useful technique to obtain phosphors with a tunable emission.

Preparation of polypyrrole-polyaniline/$LiCoO_2$ nanocomposites as an Electrode for Li-ion Batteries

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In the last decade, conducting polymers has been the subject of intense research [1], due to their conjugated double bond, they possess electronic properties similar to metals [2]. One of those polymers was Polypyrrole (PPy) and polyaniline (Pani). Because of their excellent properties such as high biocompatibility [3], good adhesion to substrate [2], excellent electrical and mechanical and thermal properties (PPy) and (Pani) can be used as electrode material for energy storage devices [2]. The present study aims at investigating the preparation process and properties of materials made of $LiCoO_2$ particles dispersed in a Polypyrrole-Polyaniline matrix. Thus, composite materials with favorable encapsulation of the bare oxide by Pani and PPy are expected providing electrical contact to electrodes, high conductivity, and retaining the lithium ion exchange capacity of the bare oxide.

Therefore, PPy-Pani additives can be used as conductive agents and cathode materials. Reports pointed out that the PPy coating on $LiCoO_2$[4], or Pani on $LiCoO_2$[5] had a benefit to its electrochemical performance. However, up until now, using (PPy and Pani) powder as additives for $LiCoO_2$ cathode has not been reported.

A nanocomposite PPy-Pani/$LiCoO_2$ was prepared with different amount of pyrrole and pani (25, 5 and 2 wt. %). The materials were characterized by IR spectroscopy, ultraviolet-visible spectroscopy (UV-Vis), X-ray diffraction (XRD) and scanning electron microscopy (SEM).

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SYNTHESIS AND STRUCTURAL STUDY OF A NEW TRIAZOLE SCHIFF BASE

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Schiff bases *i.e.* R2C=NR' (R and R'≠H), are an important class of organic compounds reported for the first time in 1864 by Hugo Schiff [1]. This family of compounds is used as starting materials in the synthesis of important drugs, such as antibiotics and antitumor substances [2-4]. A number of these bases have been reported to possess antioxidant, anticonvulscent, analgesic, antituberclotic, and diuretic activities [5]. Our interests have been in the synthesis, the characterization and the biological properties of heterocyclic Schiff bases.
The compound : N-[(E)-(2,4-dihydroxyphenyl)methylidene]-4H-1-2-4 triazol-4-amine was synthesized under reflux.

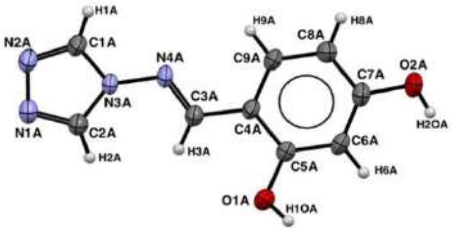

Figure 1: ORTEP of N-[(E)-(2,4-dihydroxyphenyl)methylidene]-4H-1-2-4 triazol-4-amine with thermal displacement ellipsoids drawn at the 50% probability level.

Key words: X ray diffraction, Triazole, Schiff base

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A Novel Organic-Inorganic Hybrid Based On [PW12O40] Polyanion $[C_4H_7N_2]2[HPW12O40]$

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Polyoxometalate anions have been of interest for many years not only from a structural point of view, but also because they are potentially interesting for applications in different areas such as medicine, biology, catalysis, material sciences and chemical analysis [1]. In this context a novel organic–inorganic hybrid compound (C4H6N2)2 [HPW12], was obtained by solution method using sodium tungsten dihydrate, phosphoric acid 85%and 2-methylimidazole reagents in aqueous solution. This compound was characterized by Fourier-transform infrared spectra (FTIR) UV-visible spectroscopy and single-crystal X-ray diffraction analysis. A single-crystal X-ray diffraction study shows that the compound crystallizes in monoclinic crystal system with space group C2/m and cell constants of a = 20.1753 Å, b = 18.1300 Å, c = 12.7663 Å, β= 104.929°.

Keyword: polyoxometalate, Synthesis

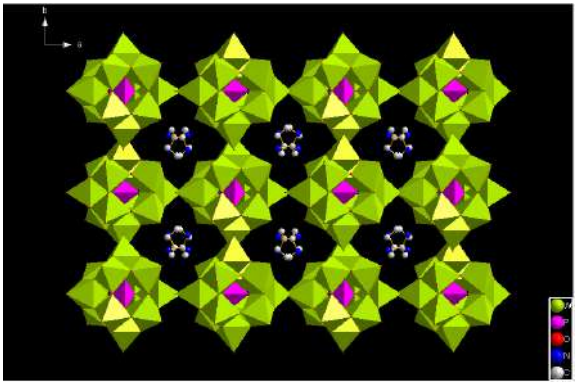

Fig. A Projection along the [001] direction of the title compound.

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Investigations on $[(CH_2CH_2CH_3)_4N]_2[M^{II}Cl_4]$ halogenometallate complexes with M^{II} = Co and Mn: Crystal structure, Spectroscopy studies and Thermal behavior

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Single crystals of two new compounds with the general formula $[(CH_2CH_2CH_3)_4N]_2[M^{II}Cl_4]$ with M^{II} = Co(II) (1) and Mn(II) (2). The compounds (1) and (2) have been grown by the slow evaporation method at room temperature. These compounds were subjected to the following characterization techniques: single crystal X-ray diffraction, thermal analysis (TG-TDA), FT-IR and FT-Raman spectroscopy. The crystals of $[(C_3H_7)_4N]_2[CoCl_4]$ belong to the monoclinic $P2_1/c$ space group whereas $[(C_3H_7)_4N]_2[MnCl_4]$ crystallize with the C_2/c space group. Molecular structures of the two coordination compounds consist of $[MCl_4]^{2-}$ anions and $[(C_3H_7)_4N]_2^+$ cations. In both crystals, each M^{II} atom center is four-coordinate, being surrounded by four chloride ligands forming a slightly distorted tetrahedral geometry. The $[CoCl_4]^{2-}$ anion is ordered however the $[MnCl_4]^{2-}$ anion is disordered over three positions with refined site occupancies of 0.25, 0.5 and 0.25. Additionally, in the structure of compounds (1) and (2), the tetrapropylammoniun $[(C_3H_7)_4N]_2^+$ is disordered over two positions with refined site occupancies of 0.5 and 0.5. Moreover, in both compounds structure, there are neither hydrogen bonds nor any unusual short-range intermolecular interactions. The structure consists of isolated MX_4 and $[(C_3H_7)_4N$ tetrahedra. Furthermore, the room temperature IR and Raman spectra of the title compounds were recorded and analyzed. On the basis of detailed vibrational studies, the detailed assignment confirms the presence of the organic groups and the anionic entities. Besides, the thermal analysis studies have been performed, but no phase transition was found in the temperature range between 298 and 430 K. Results from X-ray crystallography, Raman, IR spectroscopy and thermal analysis are combined to provide a description of the new compounds $[(C_3H_7)_4N]_2[CoCl_4]$ and $[(C_3H_7)_4N]_2[MnCl_4]$.

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Cluster transformation triggered by evacuation of solvent molecules in a new hybrid arsenomolybdate-metalorganic framework with permanent microporosity

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The hybrid compound $[Cu(cyclam)]_2[B\text{-}H_2As_2Mo_6O_{26}(H_2O)].12H_2O$ (**1**) (cyclam =1,4,8,11-tetraaza-cyclotetradecane) has been synthesized by reacting the $\{Cu(cyclam)\}^{2+}$ complex with *in situ* generated $[B\text{-}H_4As_2Mo_6O_{27}]^{4-}$ anions. The crystal packing of **1** exhibits a POMOF-like supramolecular hybrid framework built of dimeric covalent arsenomolybdate/metalorganic units and $\{Cu(cyclam)\}^{2+}$ cations, the stacking of which generates square-like channels parallel to the *z* axis with approximate cross section of 10 x 11 $Å^2$ where the hydration water molecules are hosted. Compound **1** do not preserve its crystallinity upon thermal evacuation of hydration water molecules. However, when crystals are dehydrated under vacuum, they undergo structural transitions that proceed *via* single-crystal to single-crystal transformations [1] leading to the anhydrous phase $[Cu(cyclam)]_2(A\text{-}H_2As_2Mo_6O_{26})$ (**1a**). Total dehydration results in important modifications in the inorganic cluster skeleton. This transition is also associated with modifications in the Cu^{II} coordination spheres that lead to covalent cluster/metalorganic layers that retain the supramolecular open-framework nature of **1**. This permanent microporosity provides interesting functionalities to **1a** such as selective adsorption of CO_2 gas over N_2. Structural variations between **1** and **1a** are also reflected in the electron paramagnetic resonance spectra (EPR).

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Electrochemical synthesis of poly(2-Aminoterephthalic acid-co-Aniline) on Pt electrodes. Ascorbic Acid and dopamine oxidation

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Poly(2-aminterephtalic acid-co-aniline): (poly(2ATA-co-ANI)), has been synthesized using aniline (ANI) and 2-aminoterphtalic acid (2ATA) solution in $HClO_4$, using the electrochemical oxidation procedures. Several characterization techniques were employed to gain information on the chemical structure and redox behavior of the obtained polymer, such as XPS, in situ IR and SEM. The successful incorporation of an oxidized 2 ATA monomers to the growing copolymer chain was confirmed by in situ FTIR spectroscopy and XPS. The redox switching of poly(2ATA-co-ANI) in acidic medium involves reversible benzenoid-quinoid and phenoxazine-phenoxazone conversions but carboxylic groups are only spectators and do not participate actively on these transformations. The copolymer exhibited good electrocatalytic properties towards ascorbic acid and dopamine oxidation.

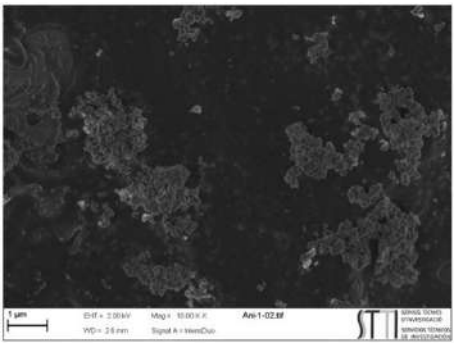

Fig. SEM image of the poly(2ATA-co-ANI)

Key words: Electrochemical oxidation, Poly(2ATA-co-ANI), Electrocatalytic properties.

Acknowledgements: Financial support from the Spanish Ministerio de Economía y Competitividad and FEDER funds (MAT2016-76595-R) is gratefully acknowledged. S. Dkhili thanks the Ministry of Higher Education and Scientific Research of Tunisia for funding her stay at the University of Alicante.

Crystal structure of a New Strandberg-type selenomolybdate $(C_7N_2H_7)_4[Se_2Mo_5O_{21}].4H_2O$

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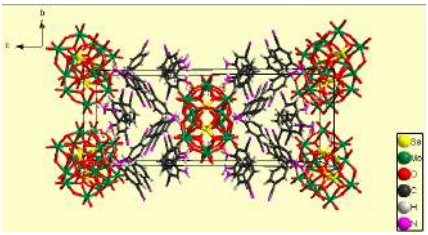
Polyoxometalates (POMs) are clusters of metal oxide units, possessing a vast array of physicochemical properties which have been studied in various fields such as medicine, energy storage, catalysis, magnetism, and electrochromic applications[1-3]. The Strandberg family of compounds represents a limit variety of structures compared to others .

The title compound $(C_7N_2H_7)_4[Se_2Mo_5O_{21}].4H_2O$ (1) was obtained by aqueous solution. A single-crystal X-ray diffraction study shows that the compound crystallizes in monoclinic system with space group I2/a and cell constants of a=12.179(1) Å, b=12.146(1) Å, c=31.664(4) Å, β=96.8402(11)°, Z=4. The arrangement of the Strandberg type selenomolybdate clusters is decribed as a pillar, 3-Aminobenzonitrile and the water molecules housed between the cluster column . The crystal structure of compound is stabilized by an extensive network of hydrogen bonds .Theoretical and experimental studies was reported in this work; thermal, electrochemical and spectral analysis also Hirshfeld surface study.

Keywords : Hybrides. Polyoxomolybdates. Crystal structure. Hirschfield-surfaces



View along axis a of the compound (1)

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Synthesis, crystal structure, and spectroscopic studies of a new Bi(III) halide complex: $[C_7H_{10}NO]_2BiBr_5$

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A new organic-inorganic hybrid compound, 2-methoxyanilinium pentabromobismuthate(III), $[C_7H_{10}NO]_2BiBr_5$, was synthesized and its structure determined by means of single crystal X-ray diffraction studies at room temperature. This compound crystallizes in the orthorhombic $C222_1$ space group with cell parameters a = 11.8870(4) Å, b = 23.4775(8) Å, c = 8.1232(3) Å, V = 2267.0(1) $Å^3$ and four molecules in the unit cell. The structure of the title compound is built up from one-dimensional $[BiBr_5]^{2n-}_n$ polyanionic zig-zag chains composed of deformed $BiBr_6$ octahedra share Br(2) and 2-methoxyanilinium cations. The assignment of the vibrational bands was based on comparison with vibrational mode frequencies of homologous compounds. Theoretical calculations were performed using density functional theory (DFT) for studying the vibrational spectrum of the investigated molecule in its ground state. The ^{13}C CP-MAS NMR spectrum is in agreement with the X-ray structure.

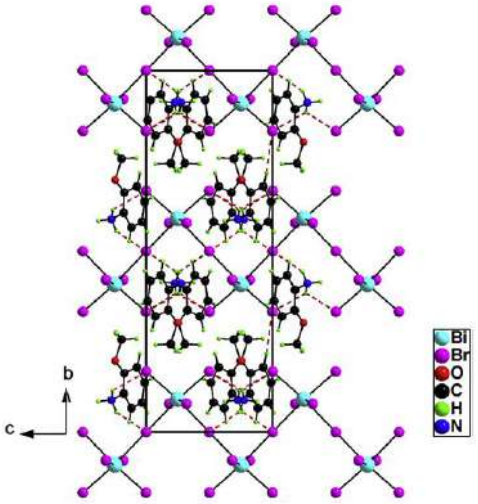

Figure Projection of the structure of $[C_7H_{10}NO]_2BiBr_5$ along the c-axis.

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

Copper promotion by $BaMO_3$ (M=Zr,Ce) perovskites applied for CO-PROX

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Power consumption requirement in nowadays moved researchers towards the implementation of fuel cells systems. The polymer Electrolyte Membrane Fuel Cells (PEMFC) are preferred for reduced scale equipment. The Hydrogen used in PEMEC is usually produced from hydrocarbons or biomass-derived molecules. The gas produced by such process usually has a high carbon monoxide concentration that impedes efficient handing of the fuel by the Pt alloy anode normally employed in the PEMFC[1, 2].
For this propose, the preferential oxidation of CO in a H_2–rich stream is recognized as one of the most straightforward and cost-effective method to reach such goal[3]. Basically three types of catalysts have displayed activity for such process: those based on platinum group metals, those based on gold and those based on base metals. Within the latter, catalysts based on copper can apparently compete with noble metal ones while they are more interesting from an economical point of view. Since copper (or copper oxide) does not exhibit on its own a significant performance for the process, it is required its combination with a promoter (within bi-functional mechanistic schemes in which both catalyst components participate) in order to acquire activity levels able to compete with noble metal catalysts. Typically, CeO_2 has been employed as a promoter of CuO_x for CO-PROX. On the other hand, $BaMO_3$ perovskite materials with M = Ce or Zr are well known because of their capability for conduction of both protons and oxygen which has made them most interesting material for intermediate temperature fuel cells. It is also known that catalysts based on such type of perovskite present improved resistance towards the presence of SO_2 impurities in the reactant mixture in comparison with CeO_2-based systems. Within this context, the aim of this work is the study of the catalytic activity of copper supported on $BaMO_3$ (M = Zr, Ce) and how the promotion of copper by such type of perovskite could affect to oxidation reactions like those involved in the CO-PROX process. For this purpose, analysis by TGA and XRD has been dedicated to explore the preparation of $BaCeO_3$ and $BaZrO_3$ by two methods, micro-emulsion and sol-gel. In turn, catalysts of copper supported on the perovskites, as well as an additional sample in which copper is intended to be integrated in the perovskite structure as M substituent, have been characterized by XRD, S_{BET} measurement, XPS and TPR (Fig.1a), and their catalytic activity has been tested under CO-PROX conditions (Fig.1.b) in combination with *operando*-DRIFTS (Fig.1c) in order to establish main structural and catalytic properties for this type of materials..

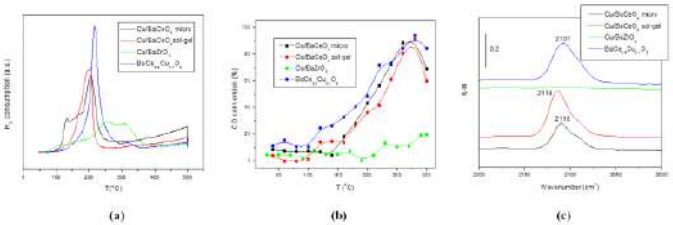

Figure1.TPR profiles (a), Catalytic activity under CO-PROX mixture (1%CO+1.25%O2 +50%H2 and He balance) (b) and DRIFTS under the same reactant mixture at 100°C (c) observed for the indicated samples.

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Synthesis, crystal structure, Hirschfield surface analysis and thermal studies of a new hybrid compound $Zn(H_2O)_6,2[C_6H_{12}N_4Cl_3Zn],0.5H_2O$

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In the course of recent years, organic-inorganic hybrids have attracted wide attention because they have the advantages of both organic and inorganic materials [1]. In the present work, we report our investigations of a new organic-inorganic hybrid compound $Zn(H_2O)_6,2[C_6H_{12}N_4Cl_3Zn],0.5H_2O$. This compound crystallizes in trigonal system, space group P-3c1 with the following unit cell parameters a = 9.8303(2) Å, c = 17.5460(2) Å, Z=2, V= 1468.38(4) $Å^3$. Crystal structure was solved with a final R (F) = 2.25% and wR (F^2) = 5.93%.
The compound was synthesized by the slow evaporation method at room temperature and characterized by X-ray diffraction, FTIR and Hirshfield surface analysis.
Thermogravemetric analysis (TGA) and differential thermal analysis (DTA) measurements have been carried out on $Zn(H_2O)_6,2[C_6H_{12}N_4Cl_3Zn],0.5H_2O$ crystal in the temperature range between 50 and 500 ºC.

Key words: X-Ray Diffraction, Hybrid Compound, Zinc Complex, Hirschfield surface, FTIR, TGA, DTA

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Synthesis and Physico-Chemical Characterizations of the Nanomaterial $Li_{2.8}Hf_{1.755}Si_{0.9}P_{2.1}O_{12}$

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The Nanomaterial $Li_{2.8}Hf_{1.755}Si_{0.9}P_{2.1}O_{12}$ was synthesized by sol-gel process. The formed white gel was dried at 100 °C in order to obtain the preliminary precursor which was then analyzed by coupled DTA-TGA (Figure1) to determine its crystallization temperature and as a result leading to the estimation of its calcined temperature. Furthermore, the calcined powder, obtained by heating the dehydrated precursor at temperature higher than its crystallization temperature, has been characterized by other physicochemical techniques: XRD, CIS and IR-FT. The XRD analysis (Figure 2) shows the obtaining of only the single phase without any detection of other phases and confirm the nanostructure of the studied material with crystallite size of 10.96 nm, whereas the complex impedance spectroscopy (Figure 3) results have highlighted the best total electric conductivity, 1.14 10^{-4} $S.cm^{-1}$ at 303 K. However concerning the IR-FT study (Figure 4) we can conclude that the results of vibration mode frequency assignment tentatives tend to be compatibles with bibliographic studies.

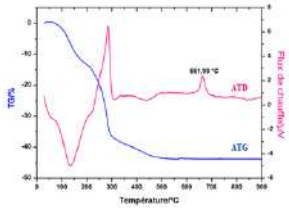

Figure 1: The thermogram DTA-TGA coupled of the Nanomaterial $Li_{2.8}Hf_{1.755}Si_{0.9}P_{2.1}O_{12}$

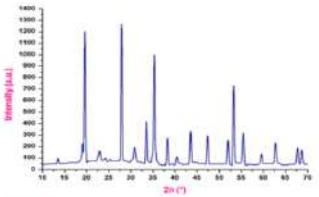

Figure 2: The X-Ray Powder diffratogram of the nanomaterial $Li_{2.8}Hf_{1.752}Si_{0.9}P_{2.1}O_{12}$

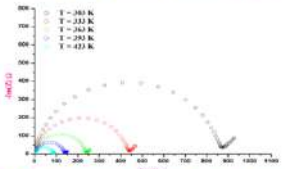

Figure 3 : The impedance diagram of the nanomaterial $Li_{2.8}Hf_{1.755}Si_{0.9}P_{2.1}O_{12}$

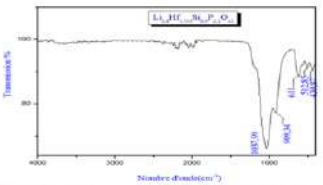

Figure 4 : The infrared absoption spectrum of the nanomaterial $Li_{2.8}Hf_{1.755}Si_{0.9}P_{2.1}O_{12}$

STRUCTURAL, VIBRATIONAL AND THERMAL PROPERTIES OF A NEW HYBRID MATERIAL BASED ON BISMUTH

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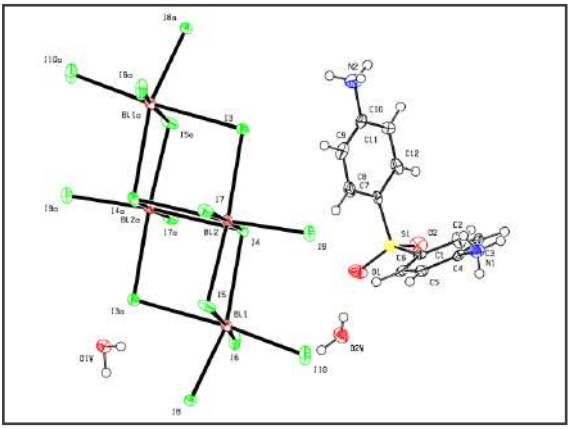
$[C_{12}H_{14}N_2O_2S]Bi_2I_8.2H_2O$ is a bismuth-iodide hybrid compound. This product was synthesized and characterized by single-crystal X-ray diffraction. It crystallizes in the monoclinic space group $P2_1/n$, with a = 12.0453 (14) Å, b = 20.437(2) Å, c = 13.1306 (16) Å, β = 96.880 (2)° and Z = 2. The crystal structure can be described as a stack of organic–inorganic layers along the [010] direction. The organic part consists of one organic cation type which orients its two amine groups towards the neighboring anions. The inorganic entity is made up of $[Bi_2I_8]^{2-}$ dimers. The water molecules are sandwiched between the $[Bi_2I_8]^{2-}$ anion and its neighboring $[C_{12}H_{14}N_2O_2S]^{2+}$ cations.. The cohesion between these entities is performed via N–H…I, O–H…I and N–H…O hydrogen bonds. The infrared spectrum was recorded in the 4000–400 cm^{-1} frequency region and the Raman spectrum was recorded in the external region of the anionic sublattice vibration 500–50 cm^{-1}. Further thermal characterization was performed by means of ATG and DSC techniques.



Asymmetric unit of $[C_{12}H_{14}N_2O_2S]Bi_2I_8.2H_2O$

Keywords: Hybrid compound, Crystal structure, Vibrational studies, thermal analysis.

Synthesis, Crystal Structure and Thermal Decomposition of $[HGdP_2O_7 \cdot 2H_2O] \cdot NH_3$.Electric properties of $HGdP_2O_7$ and $Gd_2P_4O_{13}$

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A new gadolinium diphosphate $[HGdP_2O_7 \cdot 2H_2O] \cdot NH_3$ was synthesized via a soft chemistry route from evaporation of aqueous solution . It crystallizes in the space group $P\bar{1}$ with Z = 2 and the following unit–cell dimensions: a = 6.4609; b = 6.9810; c = 9.8125 Å; α = 81.345; β = 80.547; γ = 88.430°. IR spectroscopy confirmed the presence of PO-H group, and both ammonia and water molecule in the studied material.

The thermal decomposition of $[HGdP_2O_7 \cdot 2H_2O] \cdot NH_3$, investigated by Controlled Rate Thermal Analysis, took place in three stages between -23°C and 600°C leading to obtain the anhydrous salt $HGdP_2O_7$. The calcination of the synthetized product at 850°C in a static air furnace, led to form obtain the tetraphosphate $Gd_2P_4O_{13}$ product-ray powder diffraction and infrared spectroscopy were used to identify these materials.

The electrical properties of $HGdP_2O_7$ and $Gd_2P_4O_{13}$ were investigated by impedance complex analysis. Modest conductivity was observed in both materials at relatively medium temperature range. Activation energies of 0.62 and 1.21 eV were deduced from the Arrhenius plots for $HGdP_2O_7$ and $Gd_2P_4O_{13}$ respectively.

Synthesis of new complexes of calix[4]arene and its characterization

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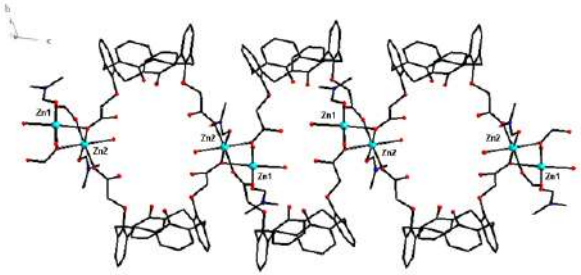
As part of our interest in coordination chemistry of calixarene[1]and thiacalixarene[2] macrocycles, we investigated their ability to form metal-organic frameworks (MOFs). Indeed, as well as others, we believe this should allow the building of materials with large pore by combining additively the ability of the calixarene macrocycle to make cavities together with the porosity induced by the coordination network[3]. Further as they may be easily functionalized this give an opportunity for easy post-synthesis modifications of the MOFs.
Calix[4]arenes with carboxylic functions [4,] and specially 25,27-bis(methoxy-carboxylicacid)-26,28 dihydroxycalix[4]areneso called herein Calix[4]arenedi-methoxycarboxylicacid (calixH2) has been developed for many years[25] for their extraction capacity of alkalineand alkaline earth metals.
Here we report the result of our tentative to synthesis MOFs using this calix[4]arenedimethoxycarboxylic with manganese(II) and zinc(II).



View of the infinite ruban running along the c axis direction for (Zn).

Key words: diacid calix[4]arene, synthesis, complexation, MOF.

Acknowledgement: we thank the PHC Utique program for the financial support.

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Structural study of $(C_5H_6N_2Cl)_2\,SiF_6$

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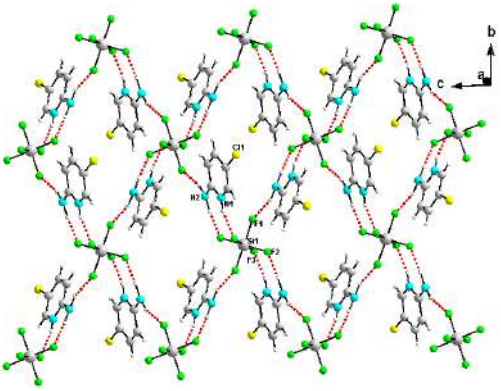
Crystalline inorganic–organic hybrid solids, are of growing interests because of their potential applications in various fields including gas adsorption or separation, catalysis, fluorescence, etc.
In the present work, the synthesis and crystal structure of the inorganic–organic hybrid compound $(C_5H_6N_2Cl)_2SiF_6$, is reported.
The compound has been prepared by slow evaporation at room temperature and its structure was elucidated using single-crystal X-ray diffraction. It crystallizes in the monoclinic system, $P2_1/c$ space group with the cell parameters: a = 6.296(3) Å, b =12.894(3) Å, c = 9.340(1) Å, β =100.94(2) ° and volume V = 888.73(4) $Å^3$.
The asymmetric unit consists of one half SiF_6 octahedron anion of Ci symmetry and one $C_5H_6N_2Cl$ organic cation. In the studied compound, intermolecular forces, as hydrogen bonds or Π-Π stacking, stabilise the crystal networks.



Structure of inorganic–organic hybrid $(C_5H_6N_2Cl)_2SiF_6$ compound

Key words: Organic - inorganic hybrid, SiF_6 octahedron, crystal structure.

Microwave Drying of Green Peas (*Pisumsativum L.):* Drying Kinetics and Physical Quality

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Several microwave power settings were applied to green peas in order to determine the effect on the drying kinetics along with colour values. In the experiments, several microwave power settings were studied as 100 w; 300 w and 450 w from the results, it is seen that the level of microwave power affected the drying kinetics and colour of the green peas. The six best known thin-layer drying models that were used in literature were applied to the experimental data and the results showed that weibull model best fits the experimental data for the drying kinetics of green peas. Effective moisture diffusivity was found to be between 2.599×10^{-8} and 8.3041×10^{-8} m^2/s. Using modified Arrhenius type equation the activation energy was found as 1.39 kW/kg. It was observed that an increase in the microwave power increased the brightness and yellowness; on the contrary, it decreased the greenness of the dried grains.

Synthesis, characterization and physical properties of $KErP_2O_7$

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$KErP_2O_7$ diphosphate has been synthesized by solid-state reaction and characterized by X-ray diffraction. This compound crystallizes in the orthorhombic system with space group Cmcm and cell parameters: a= 5.703(2) Å, b= 9.197(2) Å, c = 12.216(2) Å, Z = 4.The structure of $KErP_2O_7$ consists of a three-dimensional framework of ErO_6 octahedra, linked by P_2O_7 diphosphate units, forming tunnels running parallel to [100] which are occupied by K atoms. $KErP_2O_7$ powder was characterized by XRD, FTIR and Raman spectroscopy.
The close value of the activation energies calculated through the analysis of conductivity data found to be 0.9 eV. The activation energies obtained from impedance spectra were analyzed in order to explain the mechanism of conduction.

Effect of the presence of CO_2 in the reactant mixture for CuO/CeO2/$MgAl_2O_4$ catalysts in CO-PROX

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Catalysts of copper (1 wt. %) supported on $CeO_2/MgAl_2O_4$ have been prepared using impregnation method. Different pH values (4, 8 and 10) of the copper impregnation solution were used in order to favor different situations of interaction between copper and the two support components. The prepared catalysts have been applied to the CO-PROX process in the presence of CO_2. The effect of CO_2 on the catalytic activity was studied. Important differences are revealed in their catalytic properties which are rationalized on the basis of characterization results performed by XRD, TEM (and associated techniques, particularly XEDS mapping), EPR, XPS and TPR. Generally speaking, the catalysts supported on $CeO_2/MgAl_2O_4$ exhibit promising properties in terms of resistance towards CO_2. The data are analyzed on the basis of joint analysis of activity and operando-DRIFTS results. The trend towards stabilization of carbonate-type species, responsible of the deactivation of active interfacial sites, over the $MgAl_2O_4$ support component is pointed out as most relevant factor to explain the obtained results.

Keywords: Cu/$CeO_2/MgAl_2O_4$ catalysts, CO-PROX, Operando-DRIFTS, Hydrogen.

$Bi_xCe_{1-x}PO_4$ (x = 0.00, 0.02, and 0.08) nanorods: Structural, electrical, optical, and electrochemical properties.

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$Bi_xCe_{1-x}PO_4$ (**x** = 0.00–0.08) nanorods were synthesized using the solvothermal method at 160 °C. The successful incorporation of Bi^{3+} dopant ions into the hexagonal structure of $CePO_4$ was verified by XRD, EDX, FTIR, and UV-Vis spectroscopic techniques. The XRD and IR spectroscopy results have demonstrated that low bismuth doping does not alter the $CePO_4$ structure and good nanocrystalline materials are synthesized. The temperature dependence of conductivity, for both the pure and Bi^{3+}-doped samples, obeys the Arrhenius law of conduction. The Bi doping process has been successful in effectively tuning the optical band gap energy and electronic band structure of the cerium phosphate materials. Electrochemical tests showed an improved capacity when increasing the Bi content and a stable cycling performance of the $Bi_xCe_{1-x}PO_4$ (**x** = 0.00, 0.02, and 0.08) nanorods, making them interesting for potential further investigations.

Keywords : Nanostructures; Optical properties ; X-ray diffraction ; Electrical properties; Electrochemical performances.

Asymmetric epoxidation of styrene with TBHP catalyzed by bis(oxazoline)-vanadyl-laponite materials

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Bis(oxazoline)-vanadyl complexes have been prepared from two different precursors, $VO(acac)_2$ and $VOSO_4$, and they have been immobilized on laponite by cation exchange. The nature of the precursor conditions the vanadium loading of the final catalyst, always much higher in the case of $VO(acac)_2$. Both the homogeenous and heterogeneous complexes have been used as catalysts for the asymmetric epoxidation of styrene with tert-butyl hydroperoxide (TBHP) as an oxidant. The ligand improves the activity of the vanadyl precursor itself, with similar or slightly better selectivity to styrene oxide (up to 42%) and up to 63% in ee solution. The presence of the solid support is also beneficial in most cases, with selectivities to styrene oxide in the 30-60% range. Better enantioselectivities are obtained with the solids prepared from $VOSO_4$, up to 72% ee, although in general the highest enantiomeric excess is linked with a lower selectivity to epoxide. The heterogeneous catalysts are recoverable in part, with a slight drop in activity and enantioselectivity, although the selectivity to epoxide increases in most cases.

Synthesis, Crystal Structures and Physicochemical Study of Two Hybrid Cyclohexaphosphates

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Crystal structures of hybrid cyclohexaphosphates depend of many experimental factors such as pH value, solvent, temperature, etc. In this work, we show that the isomerism of reagents is also a determining factor in the structural design. Thus, the use of two isomeric amines allowed us to synthesize tow diferent hybrid cyclohexaphosphates. The first one, with general formula $[4\text{-Cl-2-}(CH_3)C_6H_3NH_3]_6P_6O_{18}.2H_2O$ **(1),** crystallizes in the triclinic $P\bar{1}$ space group with V = 1501(2) $Å^3$ and Z = 1. Whereas, the second one is an acidic hybrid compound $[5\text{-Cl-2-}(CH_3)C_6H_3NH_3]_4H_2P_6O_{18}$ **(2)** which crystallizes in the monoclinic $C_{2/c}$ space group with V = 4244,9(19) $Å^3$ and Z = 4. The atomic arrangements of both compounds are compared and their symmetries are confirmed by ^{31}P MAS NMR. Furthermore, spectroscopic and thermal stability of these two compounds are investigated.

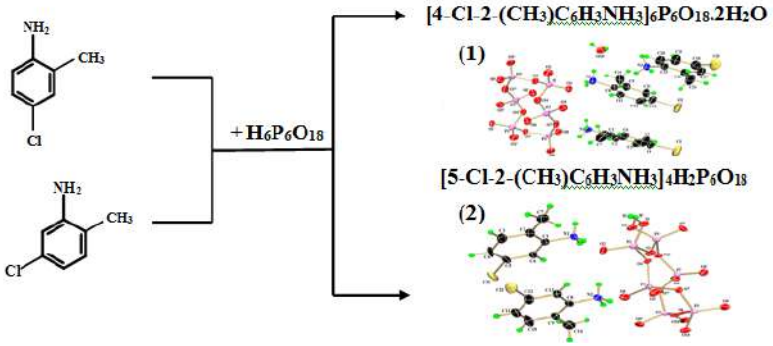

Fig. Formula and Ortep plot of both hybrid cyclohexaphosphates

Key words: Hybrid material, Crystal structure, Cyclohexaphosphate, Spectroscopic study

CRYSTAL PACKING, HIGH-TEMPERATURE PHASE TRANSITION AND SECOND-ORDER NONLINEAR OPTICAL IN AN HYBRID MATERIAL: [(S)-$C_7H_{16}N_2$][$CUBR_4$]

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The directed synthesis of noncentrosymmetric copper (II) bromo-complex has been achieved through the use of homochiral organic molecule. Reaction containing (S)-(–)-3-aminoquinuclidine, $CuBr_2$, HBr and H_2O were subjected to mild hydrothermal conditions, resulting in the growth of single crystals of [(S)-$C_7H_{16}N_2$][$CuBr_4$]. The compound crystallizes in the non polar space group $P2_12_12_1$(No. 19), which exhibits the enantiomorphic crystal class 222 (D_2). In the crystal structure, the tetrabromocuprate(II) anion is connected to three organic cations through N–H**…**Br hydrogen bonds to form cation-anion-cation molecular units, which are held together by means of offset face-to-face interactions to give one-dimensional chains. DSC measurements indicated that the compound [(S)-$C_7H_{16}N_2$][$CuBr_4$] underwent a reversible phase transition at 353 K. [(S)-$C_7H_{16}N_2$][$CuBr_4$] is more than 1.2 times as efficient as KDP in second harmonic generation; making is a potentially attractive material for non-linear optical applications.

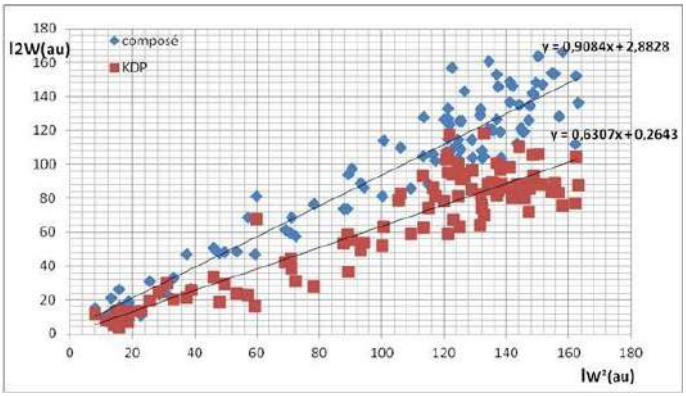

Figure. Power dependence of $I_2\omega$ vs. $I\omega^2$ for (S-$C_7H_{16}N_2$)[$CuBr_4$] and KDP.

Keywords: Tetrabromocuprate (II)-complexe; Noncentrosymmetric, Chiral amine, SHG activities

Hybrid perovskites for optoelectronics

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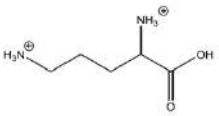
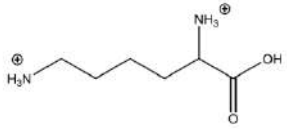
Owing to the high potential of producing novel materials with intriguing properties and unusual structures, organic–inorganic hybrid systems have attracted more and more the attention of material chemists in the last decade. In the wide field of hybrids, iodometalate salts of Sn(II), Pb(II), Sb(III), and Bi(III) ions are attractive for device applications due to their electronicor optical properties as well as their room-temperature processability.The approach of a ‘‘building block’’ being adopted to guide the design and synthesis was proven to be successful in many other fields.

Certainly, the hybrid perovskites based on SnII and PbII have caught much more attention owing to their electronic properties and device applications. For instance, the FET mobility of $(C_6H_5C_2H_5NH_3)_2SnI_4$, as a semiconducting channel in a thin-film field effect transistor FET, their 2D perovskite networks with suitable flexiblility can be easily stabilized by specific cations, especially non-bulky primary ammonium. Depending on characteristics of cationic entities as well as the stereoactivity of lone pair, perovskite sheets are more or less distorted. And it has been proven that undistorted sheets would dramatically reduce the band gap of the corresponding hybrid. In this aspect, we have recently proposed the use of suitable bifunctional cations which can impact the bonding features of inorganic layers due to weak interactions at the organic–inorganic interface.

We have synthesized a new two-dimensional HPs(n=1, n=2) based on Bromoplombate with different ligands like





High efficiency decolorization of azo dyes by Ca-Al particles

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The present study reports on the degradation of azo dye Reactive Black 5 by Ca-Al metallic particles prepared by melt-spinning and ball-milling. The morphology and the phase structure of the metallic powders were characterized and the decolorization efficiency of reactive black 5 solutions was assessed by monitoring the dye degradation by ultraviolet-visible absorption spectrophotometry. The decolorization process using the Ca-Al powders shows an exceptional fast kinetics and high efficiency. 40 mg L-1 dye solution was successfully decolorized in 1 min using 0.1 g / 100 mL of $Ca_{65}Al_{35}$ powders, suggesting it as an effective, low-cost mean for degradation of azo-compounds.

Keywords: Ball milling; Rapid solidification; Reactive Black 5; Decolorization.

Synthesis, crystal Structure and Hirshfeld surface analysis of bis(2,6-diaminopyridinium)tetrachloridocobaltate(II)

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Crystals of bis(2,6-diaminopyridinium)tetrachloridocobaltate**(1)** were grown by slow evaporation method. The single crystal X-ray diffraction results reveal that the newly-prepared compound crystallizes in the monoclinic system with $P2_1/n$ space group with the following parameters: a=7.389(1)Å, b=15.372(2)Å, c=15.387(3)Å, β=98.20°. The crystal structure of (1) can be described as an alternation of inorganic layers, built from tetrachloridocobaltate anions $[CoCl_4]^{2-}$ and organic one formed by protonated cations of 2,6-diaminopyridinium. The crystal packing is governed by the N–H···Cl and C–H···Cl hydrogen bonding interactions between the organic and the inorganic ions, in which they may be effective in the stabilization of the crystal structure **(Fig.1)**. Moreover, the cations show a π-π interaction adding extra stability to the three-dimensional architecture. The prevalence of these interactions is illustrated by an analysis of the three-dimensional Hirshfeld surface (3D-HS) and by two-dimensional fingerprint plots (2D-FP). The relative contribution of different interactions to the 3D-HS indicates that the H…H account for 30,8 % of the total 3D-HS area **(Fig.2)**.

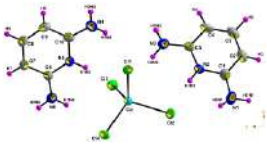

Fig1. The asymmetric unit of the crystal structure of(1) showing the atom numbering scheme.

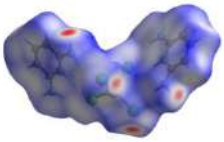

Fig.2 View of the 3D-HS of (1) mapped with d_{norm}

Key words: Bis(2,6-Diaminopyridinium)tetrachloridocobaltate, Crystalstructure,Hirshfeld surface, Fingerprint plots.

Synthesis and characterization of calcium hydroxyapatite functionalized with tyramine

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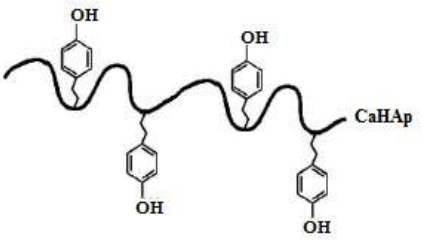
Calcium hydroxyapatite Ca10(PO4)6(OH)2, (CaHAp) is an important inorganic compound widely used in biology and chemistry [1]. The compound is specifically known for its particular properties of ionic exchange, affinity of adsorption, and its capacity to establish links with organic molecules of different sizes. The inorganic–organic hybrids obtained have interesting applications such in chromatography [1], catalysis [2] and biomedical domain [3]. The aim of this work is to prepare hybrid inorganic–organic compounds, at different amounts of tyramine (TYR). These materials were prepared via the hydrothermal method. The obtained powders were characterized through chemical analysis, XRD, IR, ^{31}P MAS-NMR, TEM, and TG-TDA. The XRD showed that the TYR did not affect the apatite composition. Indeed, only a reduction of the crystallite size was noted. The presence of tyramine fragment bounded to hydroxyapatite was confirmed by IR and solid-state NMR spectroscopy, TEM technique indicate that the presence of tyramine changes the morphology of the particles.



Scheme. Functionalization of tyramine on hydroxyapatite surface

Keywords: Apatitic surface, Grafting tyrosine, Hybrid compound

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SYNTHESIS, STRUCTURAL STUDY AND NLO PROPERTIES OF A NEW HYBRID COMPOUND OF 4, 4'-DIAMINODIPHENYL SULFONE LIGAND

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Hybrid organic-inorganic compounds are receiving considerable attention in recent years due to the possibility of combining the different characteristics of the components to get unusual and enormous variety of interesting structural topologies and wide potential applications in the fields of catalysis, non-linear optics, sensors, magnetism and molecular recognition [1]. During our investigation we discovered a new hybrid organic-inorganic compound presenting second-order Nonlinear optical properties [2]. The hybrid compound crystallises in the non centrosymetric space group $Pna2_1$ of the orthorhombic system, with the following cell parameters: a= 7.7434(5) Å b= 9.1241(5)Å c= 22.0914(5)Å. Asymmetric unit of the title compound (Fig.1) consists of one diprotonated4,4' daps ligand and two nitrate anions

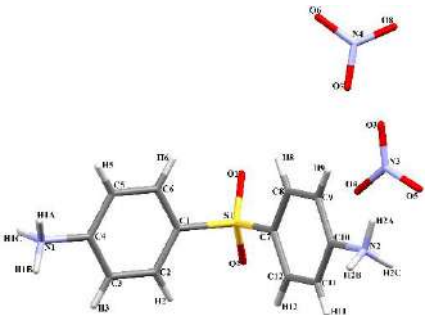

Fig.1: Asymmetric unit of 4,4'-diammoniodiphenyl sulfone di(nitrate)

Key words: NOL, Hydrogen bonds, Daps ligand.

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TUNISIAN MONTMORILLONITE CLAY AS DRUG CARRIER

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Dicolofenac sodium (DS) is a widely used non-steroidal anti-inflammatory drug (NSAID) to control pain and inflammation recognized to have gastrointestinal side effects [1]. In the present study, a low cost formulation made up from Tunisian purified montmorillonite (MMT) and chitosan (CS) as a drug carrier of DS was prepared via intercalation solution technique for drug delivery purposes as an attempt to increase therapeutic effectiveness and reduce side effects. X-ray diffraction, thermal analysis, Fourier transformed infrared spectroscopy, High resolution transmission electron microscopy coupled with energy dispersion were employed for the characterization. Images obtained by transmission electron microscopy and XRD results confirmed that the anionic drug was successfully loaded in the nanocomposite structure .The thermal analysis results were in agreement with XRD data revealing an intercalation of DS and CS in the MMT interlayer gallery suggesting a higher thermal stability of the organic matter when intercalated into the clay mineral and proving the compatibility of diclofenac sodium with CS and the clay used to prepare the DS loaded CS nanocomposite. Thus our study can be fruitful for biomedical applications by using the simple approach described in our study for the synthesis of clay loaded nanocomposites as carriers for different therapeutic molecules .

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Validation and uncertainty estimation of an ICP-AES method for the determination of bismuth in bismuth sulfide thin films prepared by chemical bath deposition

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Validation and measurement uncertainty evaluation of a quantitative method for the determination of bismuth (Bi) in bismuth sulfide thin films using an inductively coupled plasma atomic emission spectrometry are presented. RF power of 1000 watts, plasma gas flows of 13 L min^{-1}, nebulizer flow of 0.02 L min^{-1} were used. Plasma view at radial mode and the wavelength was monitored for bismuth at 223.061 nm. According to AFNOR Norm NFT 90-210 (2009), the regression equation was calculated from standards in the same matrix of the prepared samples and the linear range was 0.2-2.0 mg L^{-1}. Limits of detection (LOD) and quantification (LOQ) were 0.03 and 0.12 mg L^{-1}, respectively. The recovery percentages (%) obtained ranged between 91 and 109 %. Accuracy data obtained were also adequate. According to GUM and EURACHEM guides, the estimated value of the expanded uncertainty was estimated to 1.7%.

Keywords: Method validation; Uncertainty estimation; Bismuth; Bismuth Sulphide; Thin films; Chemical bath deposition

Adsorption of cadmium (II) and lead (II) from water by new cross-linked reusable polystyrene adsorbents

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In this study, the commercial polystyrene (PS, M= 350000) was grafted with chelating group via amino linkage. PS with amino groups was initially prepared by nitration and reduction reactions, as described in literature **[1**, **2].** Then, the aminated PS was cross-linked by 2, 2'-dichlorodiethylether (DCDEE) to give a polymer named PS-NH-DCDEE (fig .1). The obtained products were characterized by Infrared Spectroscopy, Elemental Analysis and TGA, DTA analyses. The evaluation of metallic cations extraction was carried out by flame atomic absorption spectroscopy measurements of the remaining solutions. The new cross-linked polymer was tested for its ability to extract some metallic cations such as Pb^{2+}, Cd^{2+} from aqueous phase (pH=5-6) at room temperature.

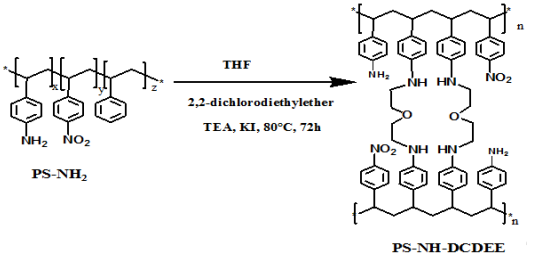

Fig. 1. Proposed cross-linked structure of PS-NH-DCDEE.

Keywords: Polystyrene adsorbents, Cross-linking, Adsorption.

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IN SITU X-RAY DIFFRACTION STUDIES, THERMAL BEHAVIOR AND MAGNETIC PROPERTIES OF ORGANIC-INORGANIC COPPER (II) COMPLEXS.

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Copper (II) systems appear to be a very affective and original family to elucidate the relationship between structural parameters and magnetic properties. As a part of our ongoing research, two coordination complexes were prepared and studied. Molecular structures of the compounds consist of anions and cations entities linked together via non-covalent interactions essentially hydrogen bonding and π···π stacking which lead to three-dimensional supramolecular architecture. Thermal analysis TGA-DTA, DSC measurements and *in situ* X-ray powder diffraction were carried out to highlight the possible phase transitions and the thermal stability of the compounds. Both compounds exhibit weak antiferromagnetic interactions.

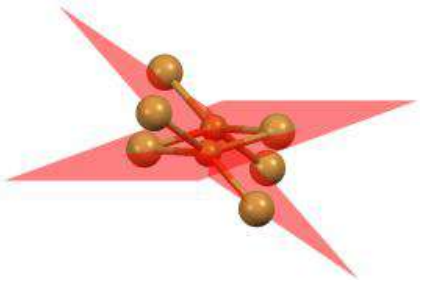

Fig. twisted distortion in anionic dimmers.

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

Investigation of the Allelopatic Effects of Thymol, Carvacrol and Alpha-terpinene as Natural Chemicals

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Weeds cause the loss of crops and quality at least as much as insects and disease agents in agricultural production. Most of the herbicides used to weed control are derived from synthetic chemicals. Many of these chemicals are toxic to humans and other living things due to their aliments and ground water. On the other hand, chemical inputs used before and after sowing/planting processes, as well as toxic substances that have accumulated in the seed bed before can cause problems in germination and emergence of plants. In a sustainable agricultural production, reliable food, reliable water, the highest yield and quality are targeted by satisfying all the needs of the plants in the period from sowing to harvest from natural sources. Increasing and evolving technology is increasing research on the use of chemical preparations derived from naturally occurring plants instead of the use of chemicals that are harmful to plants discovered many years ago and which are also toxic to living organisms. In this study, the allelopathic effects of thyme essential oil components on the germination and root length of some wild oak obtained from wheat fields were investigated. For this purpose, α-terpinene, carvacrol, thymol were added to each petri dish at different doses (0, 2, 5, 10 and 20 µl/petri dish). At the end of third week, the effects of bio-chemicals on the germination rate of root length of wild oak were investigated. As a result of the comparison, the negative effects of carvacrol, thymol on germination of weed seed and root length were statistically significant at a significance level of 0.01. But α-terpinene was not statistically significant. Increased dose compared to control application, reduced germination rate and root length. This result suggests that bio-chemicals used in the research could be used as bio-herbicides instead of chemical herbicides to control of wild oat in winter plants. However, in order to use these bio-chemicals as bio-herbicides, the effects of winter plants should be investigated.

Keywords: Wild oat, Germination, allelopathy, carvacrol, thymol, α-terpinene

Fabrication and Characterization of two new hybrid materials

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Organic-inorganic hybrid materials have been broadly investigated owing to their various properties used in optoelectronics and importanly due to their potential to utilize high throughput, solution phase processing, which will lead to low cost production.
Here, we report synthesis of two new organic-inorganic hybrid materials which are obtained by slow evaporation at room temperature using the same organic cation as well as the same metal for two different molar ratio. These two compounds are characterized by X-ray diffraction, infrared and Raman spectroscopy, optical absorption and photoluminescence measurements.
Additionally, we report the morphological properties of these new materials.
It was found that the two compounds crystallize in the same space group but their structures are organized differently. The Raman and IR spectra recorded at room temperature confirm the presence of the organic and inorganic groups.

Preparation and Characterization of Amorphous Solid Dispersion of Poorly Soluble Drug (Ibuprofen, Atorvatatine and Celecoxib)

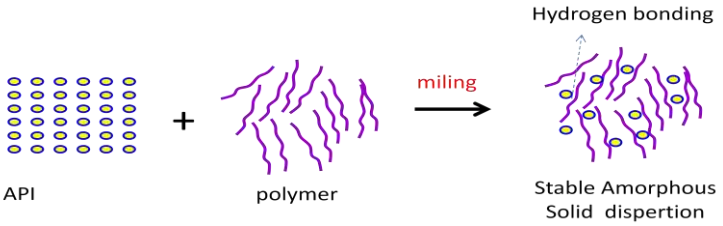
Haykel Galai and Rafik Kalfat

Institut National de Recherche et Analyses Physico-chimiques, Biotechnopôle Sidi-Thabet 2020.

Poor water solubility of many drugs has emerged as one of the majors challenges in the pharmaceutical word. Amorphous solid has been considered as the major advancement in overcoming limited aqueous solubility and oral absorption issues; however this form can easily revert to crystalline one on process and storage. The incorporation of Active Principe Ingredient (API) in hydrophilic polymer, leading to Amorphous Solid Dispersion (ASD), has been found to be very useful in inhibition of drug crystallization and enhancement of dissolution [1].

In this work, ASD have been prepared by co-milling [2] of three API, atorvastatin, celecoxib and ibuprofen with Polyvinylpyrrolidone (PVP) and hydroxypropyl methylcellulose (HPMC). The solid-state characterization for such systems was studied by combining the infrared spectroscopy (FTIR) and X-ray diffraction (XRD) data with the results of thermal analysis, differential scanning calorimetry (DSC).

The evaluation of cristallinity, glass transition (Tg) and physical–chemical interactions in the prepared ASD, have been highlighted the role of drug-polymer hydrogen bonding interaction on the molecular mobility and crystallization inhibition.



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Bioactive Glasses development: Mechanical study and *"In vitro"* simulation

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Two new glasses with compositions based on the SiO_2-Na_2O-CaO-P_2O_5 system; B_2O_3 and CaF_2 were added separately in increasing amounts (from 0 to 20 wt. %) were produced according to the melting process. These elements (boron, fluorine) present the chemical, mechanical and biological interests. The present study allows us to understand the properties of these glasses which could have an influence on their chemical reactivity and their bioactivity. Fluorine released from implant materials might result in the formation of fluorapatite, and this reduces both solubility and acid produced by bacteria. Addition of B_2O_3 generally increases glass materials bioactivity. In addition, borate based glasses were also used as orthopedic implants thanks to their solubility. This study allows the tailoring of novel fluoride and borate-containing bioactive glasses to address orthopedic needs.

Key words: Bioactive Glasses, Boron, Fluorine, Physico-chemical characteristics, Mechanical proprieties.

Diazonium chemistry for modification of polymeric materials

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1. University of Chemical Technology, Prague, Czech Republic
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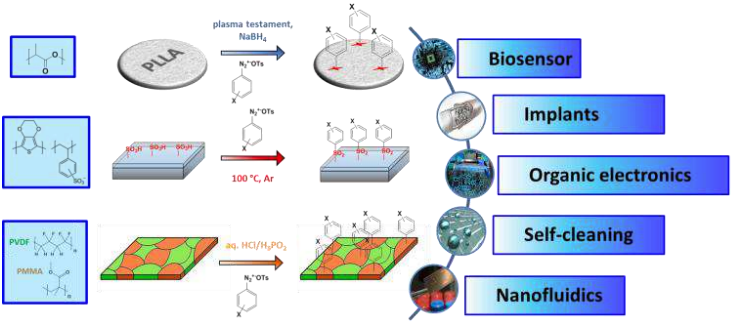
Polymers are represent one of the most commonly used materials in our daily living due to their numerous attractive and specific properties: mechanical, optical, transport, chemical and biological, and low cost [1]. Therefore, there is a strong need for altering and addition of specific properties to their surface; especially appealing to tailor the surface properties of a polymer independent of its structure and bulk properties. Aromatic diazonium salts (ADS) are one of the most effective modification agents for a wide range of surfaces, including polymers. Powerful class of ADS - arenediazonium tosylates (ADTs) demonstrate high stability, explosion safety and excellent solubility on water and organic solvents [2]. We also found that ADTs are also able to covalently modify the surface of biomedically significant PLLA, conductive polymer PEDOT:PSS and polymer blend with piezo-electric properties PVDF-PMMA [3[a,b,c]].



Modification by ADTs with different substituents (X= NO_2, NH_2, COOH, SO_3H, C_8F_{17}, $(CF_3)_2$, $C_{18}H_{37}$) enable to alter wettability of surface, biorepellence/bioadhesion, biodegradation, nanomechanical, electrical and optical properties. As a result, developed approaches for functionalization give the opportunity to adjust surface properties of polymer for targeted application.

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Synthesis, crystal structure and Hirshfeld surface analysis of a novel organic-inorganic hybrid material $C_8H_{14}N_2(HSO_4)_2$

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A novel organic-inorganic hybrid material $C_8H_{14}N_2(HSO_4)_2$ formed by p-xylylenediaminium cation and two hydrogen sulfate anions has been synthesized and structurally determined by X-ray diffraction method, Hirshfeld surface analysis, IR, UV visible and Thermal analysis. This compound crystallizes in the monoclinic system, space group $P2_1/_C$, with a=5.5406 (7) Å, b= 14.5547 (17) Å, c = 8.3864 (9) Å, β = 93.842 (5) °, V = 674.77(14) $Å^3$ and Z = 2. The crystal structure of the title salt consists of infinite sheets of alternating organic and inorganic entities. The p-xylylenediaminium cations are linked to the hydrogen sulfate anions by N–H…O, O–H…O and C–H…O hydrogen bonds, forming a three-dimensional network.
The Hirshfeld surface analysis and the two-dimensional fingerprint maps indicate that the packing is dominated by H…O/O…H and H…H contacts.

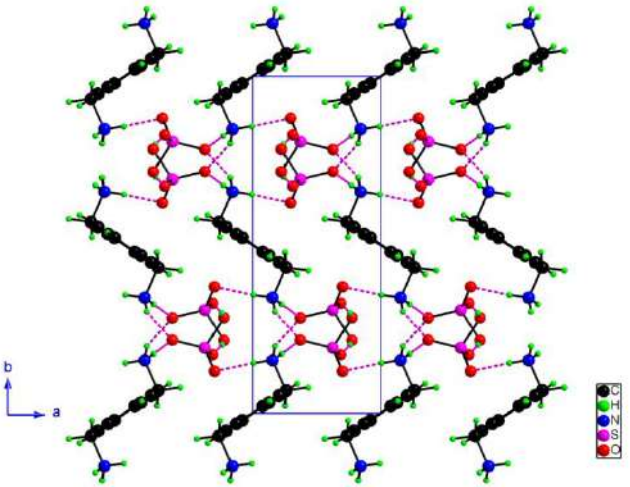

Fig. Projection of $C_8H_{14}N_2(HSO_4)_2$ along the c-axis.

Keywords: crystal structure, p-xylylenediaminium, sulfate, Hirshfeld surface analysis, fingerprint maps.

SYNTHESIS, CRYSTAL STRUCTURE AND CHARACTERIZATION OF A NEW ORGANIC-INORGANIC HYBRID $(C_6H_{10}N_2)_2[MNCL_6]\cdot 2H_2O$

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Single crystals of the novel Bis (2-aminomethyl pyridindium) hexachloridomangante (II) bi-hydrate $(C_6H_{10}N_2)_2[MnCl_6]\cdot 2H_2O$ were grown by slow evaporation solution technique at room temperature. The compound was characterized by IR, Raman spectroscopy and single X-ray diffraction. The title compound crystallizes in the triclinic system, space group P-1, with a=7.5214(8) Å, b=7.6973(7) Å, c=10.1789(11) Å α= 89.159(3), β= 78.686(3), γ= 64.775(3), V= 521.17(9) $Å^3$ and Z=1. The asymmetric unit contains one half anion, one cation and one water molecule. The whole $(C_6H_{10}N_2)_2[MnCl_6]\cdot H_2O$ structure geometry is generated through an inversion center with Mn being at the crystallographic postion (0, ½, ½). These entities: $[MnCl_6]^{4-}$,$(C_6H_{10}N_2)^{2+}$ and H_2O are held together by hydrogen bonds and π-π stacking interaction which lead to three-dimensional supramolecular architecture.

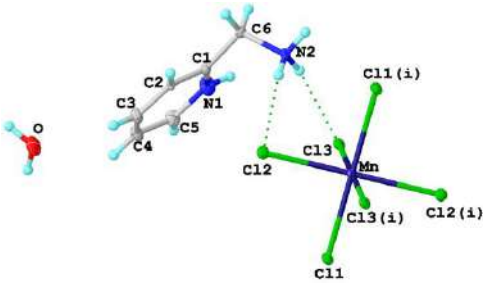

Fig. 1. The asymmetric unit of $(C_6H_{10}N_2)_2[MnCl_6]\cdot 2H_2O$ showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Keywords: IR, Raman spectroscopy, π-π and O/N-H…Cl interactions, supramolecular architecture.

Magnetic and electrochemical properties effects of iron–aluminum substitution in new sodium nickel polyphosphate cathode materials $Na_2Ni_2(Fe,Al)(PO4)_3$

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[4] *Université El Manar, U.R. Physico-Chimie des Matériaux Solides,1060 Tunis, Tunisia*

Herein we report the studies of different physical properties (structural, magnetic, thermal, morphologic, electrical and electrochemical) of two new allotropic β-$Na_2Ni_2M(PO_4)_3$ (NNMP) phosphates, with M = Fe and Al. Pure orthorhombic single-phase powders were prepared under air, using autocombustion synthesis method, Crystal structures of both compounds were determined using X-ray powder diffraction and Rietveld method refinements, which indicate the occurrence of Ni^{2+} in the 8g site, and of M^{3+} in the 4a site of the structure. The structure consists of three-dimensional anionic framework obtained by the association on MO_6, NiO_6 and PO_4 polyhedrons, sharing edges and corners. The resulting three-dimensional structure creates mono-dimensional channels along the [100] and [010] directions, formed by face-shared oxygen polyhedra and occupied by Na^+ cations. This non-disordered cationic distribution in confirmed by a significant change of magnetic properties. Thus, both NNAP and NNFP samples show paramagnetic to ferromagnetic transition at 14 and 19 K, respectively. For the two compounds, thermal stability, electrical conductivity and electrochemical properties have been also studied. The interaction/desintercalation properties of NNMP compounds as positive electrode were tested in sodium-ion batteries. The cycling curves exhibit a significant polarization even for both prepared samples.

Keywords: Na-ion battery; Crystal structure; Surface morphology; Curie temperature, Discharge capacity.

Synthesis of 2-guanidinobenzimidazolium dibromide monohydrate salt and investigation of its structure via x-ray diffraction, spectroscopic measurements and DFT calculations

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The 2-guanidinobenzimidazolium dibromide monohydrate salt **(I)** was prepared and characterized through single-crystal X-ray diffraction, photoluminescence, IR and UV-Vis diffuse reflectance spectroscopy. The structural unit of **(I)** consists of a discrete guanidinobenzimidazolium, two Br^- and one water molecule of crystallization **(Fig.)**. The crystal structure of the title salt is stabilized by N-H…Br, O-H…Br, N-H…O and C-H…Br hydrogen bonds. The structure of the title compound was computed with the Gaussian 09 program using the DFT hybrid B3LYP functional and the 6-311+G(d,p) basis set. The electronic transitions were calculated using the time dependent TD-DFT method. The Theoretical results of the title compound were in good agreement with the experimental one.

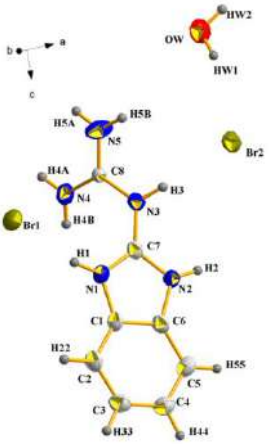

Fig. The asymmetric unit of the crystal structure of (I) showing the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level

Key words: crystal structure, photoluminescence, IR, UV-Vis diffuse reflectance spectroscopy, DFT calculations.

Growth and structural characterizations of $La_2Mo_{2-v}S_vO_9$ ($0.1 \leq y \leq 0.6$), a new solid solution on sulfur-stabilized $La_2Mo_2O_9$

Khouloud Hchicha*, Noureddine Mhadhbi and Houcine Naïli

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A new fast oxide ion conductor $La_2Mo_2O_9$ was reported by Lacorre et al. **[1]** in 2000, which exhibits higher oxide ion conductivity (at temperatures above 580°C) compared to that of standard 8 mol % yttrium stabilized zirconia (YSZ8%). This compound undergoes a phase transition around 580°C from a low temperature α-form to high temperature β-form. It is worth noting that this phase transition leads to a significant increase of the cell volume by 0.46% which can induce mechanical stress at the electrode interfaces and in extreme cases could lead to fracture of the membrane. Our strategy was to suppress this phase transition through solid solution formation with the aim to stabilize the high temperature β-form to lower temperatures. Numerous works **[2-4]** were then devoted to aliovalently or isovalently substituted derivatives of $La_2Mo_2O_9$, the so-called LAMOX family.

In this work, this solid solution $La_2Mo_{2-y}S_yO_9$ was examined to obtain a deeper insight into the influence of sulfur on the stabilization of the phase β. These oxides were synthesized by conventional solid state route. Their purity was certified by X-ray diffraction. Above a sulfur content y greater than 0.1, the high temperature, disordered and highly conducting β form is stabilized at the expense of the low temperature α form. Along the series, the linear reduction of the unit cell volume with increasing the sulfur content y reflects that S^{6+} is much smaller than Mo^{6+} cation. Thermogravimetric analyses showed that the loss of sulfur start above 900°C which tends to increase linearly as y increases. The electrical conductivity studies showed that none of the tested compositions allows exceeding the conductivity of the compound $La_2Mo_2O_9$. Studies by spectroscopy Infrared and Raman were realized to supply information on the vibrations of atoms within our materials. The perusal of the Infrared and Raman spectrum allowed confirming the presence of the characteristic peaks of SO_4^{2-} and MoO_4^{2-}.

Key words: LAMOX, Ionic conductivity, SOFC, IR and Raman spectroscopy.

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Synthesis of MIL-101@g-C_3N_4 nanocomposite for enhanced adsorption capacity towards CO_2

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3 Département de Génie des Matériaux, Faculté de Chimie, Université des Sciences et de la Technologie Mohamed Boudiaf, BP 1505 El Mnaouer, 3100 Oran, Algeria.

MIL-101@g-C_3N_4 nanocomposite was prepared by solvothermal synthesis and used for CO_2 adsorption. The parent materials (MIL-101 and g- C_3N_4) and the MIL-101@g- C_3N_4 were characterized by X-ray diffraction, argon adsorption/desorption, Fourier transform infrared spectroscopy, thermal analysis (TG/DTA), transmission electronic microscopy, and Energy-dispersive X-ray spectroscopy. The results confirmed the formation of well defined MIL-101@g-C3N4 with interesting surface area and pore volume. Furthermore, both MIL-101 and MIL-101@g-C3N4 were accomplished in carbon dioxide capture at different temperatures (280, 288, 273 and 298 K) at lower pressure. The adsorption isotherms show that the nanocomposite has a good CO_2 adsorption affinity compared to MIL-101. The best adsorption capacity is about 1.6 mmol g^{-1} obtained for the nanocomposite material which is two times higher than that of MIL-101, indicating strong interactions between CO_2 and MIL-101@g- C_3N_4. This difference in efficacy is mainly due to the presence of the amine groups dispersed in the nanocomposite. Finally, we have developed a simple route for the preparation of an effective and new adsorbent for the removal of CO_2, which can be used as an excellent candidate for gas storage, catalysis, and adsorption.

$(C_6H_{14}N_2)[CO(H_2O)_6](S_{0.83}SE_{0.17}O_4)_2$: IR SPECTROSCOPY, 3D H-BONDED STRUCTURE AND THERMAL BEHAVIOUR

Ines Kadri, Walid Rekik, Houcine Naïli

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

An organically templated sulfate/selenate based compound, $(C_6H_{14}N_2)[Co(H_2O)_6](S_{0.83}Se_{0.17}O_4)_2$, has been synthesized by slow evaporation and crystallographically characterized. The IR spectroscopy confirms not only the partial substitution of sulfur by selenium atom but also the amine protonation. The new complex crystallizes at room temperature in the monoclinic centrosymmetric space group $P2_1/c$, with the following unit cell parameters: a = 12.0669(13) Å; b = 12.1768(11) Å; c = 12.1158(12) Å; □ = 104.023(4)° and Z = 4. Its crystal structure is built of cobalt(II) cations coordinated by six water molecules in octahedral geometry, dabcodiium cations and sulfate/selenate anions, resulting in an extensive hydrogen-bonding network. The thermal decomposition of this compound proceeds through several stages giving rise to the cobalt oxide as final product at 900°C .

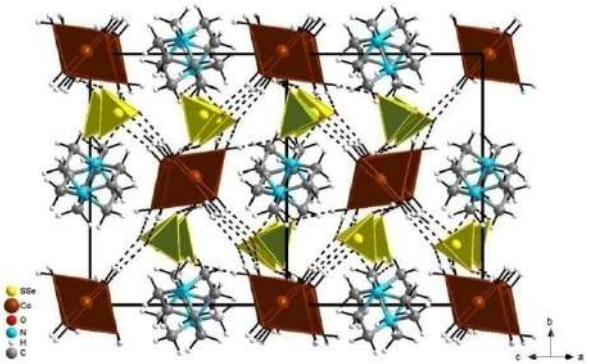

Fig.1: Projection of the structure along the [101] direction.

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

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

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The experimental determination of phase diagrams of ternary systems requires a very high number of experimental measurements and the use of several techniques. To target the necessary experiments to study these systems, we choose a method which based on thermodynamic calculation. Indeed, the compilation of all the experimental data relative to the three pure components and to their binary mixtures allows studying the coherence between the thermodynamic data and the phase diagram.

A thermodynamic assessment of the ($LiNO_3$+$NaNO_3$+$TlNO_3$) ternary system is carried out, in this work, for the first time. The ($LiNO_3$+$NaNO_3$), ($NaNO_3$+$TlNO_3$) and ($LiNO_3$+$TlNO_3$) binary systems, have been optimized earlier in previous works [1-3]. The parameters describing the Gibbs energies of all the binary phases are then used to estimate the phase diagram of the ($LiNO_3$+$NaNO_3$+$TlNO_3$) ternary system.

In order to verify the results of our assessment, a vertical section, X_{NaNO3} = 0.20, is studied experimentally by simultaneous direct and differential thermal analysis technique (STA/DTA). The experimental results are then compared to the calculation. A good agreement is obtained.

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

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A Hirshfeld Surface Analysis, Crystal Structure and Infrared Characterization of a New Organic Cyclohexaphosphate, $(C_8H_{12}N)_4(Li)_2(P_6O_{18})(H_2O)_6$

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Single crystals of a new organic cyclohexaphosphate $(C_8H_{12}N)_4(Li)_2(P_6O_{18})(H_2O)_6$ prepared by an acid/base reaction between cyclohexaphosphoric acid $H_6P_6O_{18}$ and 2,6-trimethylaniline, have been successfully grown by slow evaporation technique at room temperature. This compound was characterized using X-ray diffraction (XRD) and FT-IR spectroscopy. The results show that the title compound was phased with a triclinic structure and P-1 space group. The crystal structure was solved by using 5203 independent reflections with a final R value of 0.031. The P_6O_{18} ring is centrosymmetrical. Its main geometrical features are those commonly observed in others cyclohexaphosphates. The atomic arrangement of this compound can be described by an organization in a three-dimensional framework, built up from alternated organic and inorganic species. Adjacent P_6O_{18} rings are linked via corner-sharing by LiO_4 tetrahedra, generating anionic $[Li_2(P_6O_{18})(H_2O)]^{4-}$ layers parallel to (110) **(Fig. 1)**. Charge balance is achieved by the protonated amine which is trapped in the interlayer space and interacts with the organic framework through strong and medium hydrogen bonding **(Fig. 2)**. An extensive network of N-H...O and O-H...O hydrogen-bonding interactions link the components into a three-dimensional network and additional stabilization is provided by weak C-H...O hydrogen bonds. Intermolecular interactions were investigated by Hirshfeld surfaces. The vibrational absorption bands were identified by infrared spectroscopy. The stretching modes of P_6O_{18} ring and band assignments are described.

Keywords: Cyclohexaphosphate; X-ray diffraction; Hirshfeld surface; Enrichment ratio; Infrared spectroscopy.

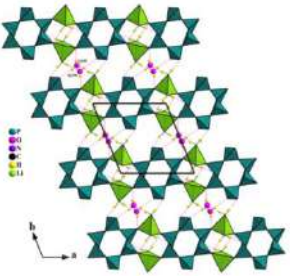

Fig. 1: Projection along the *c*-axis, of an inorganic layer in the structure of $(C_8H_{12}N)_4(Li)_2(P_6O_{18})(H_2O)_6$

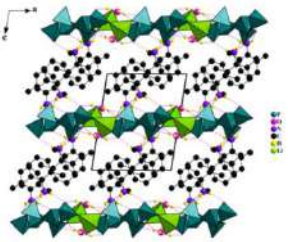

Fig. 2: Atomic arrangement of $(C_8H_{12}N)_4(Li)_2(P_6O_{18})(H_2O)_6$, projected along the *b*-axis,

Influence of grain boundary misorientations on the mechanical behavior of a AA6060 alloy processed by ECAP

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An AA6060 alloy was processed by equal-channel angular pressing (ECAP) and the features of microstructural transformation were studied using EBSD analysis. The results show a non-monotonic variation in the density of low-angle and high-angle boundaries of the phase grains with increasing strain during ECAP. The relationship between the misorientations of the grain boundaries and the mechanical properties of the alloy were examined.

Keywords: AA6060 alloy, Ultrafine-grained structure, Grain boundaries, Strength, Ductility

The magnetic and structural properties of nanostructured $Fe_{50}Co_{25}Ni_{25}$ alloy prepared by mechanical alloying

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Magnetic nanocrystalline $Fe_{50}Co_{25}Ni_{25}$ powder mixture was prepared by mechanical alloying. Morphological, microstructural properties and thermal stability changes during the milling process were followed up by scanning electron microscopy, X-ray diffraction, and differential scanning calorimetry, respectively. The magnetic characterizations of the milled powders obtained at different milling time were investigated by SQUID magnetometer. The results show that for milling time up to 100 h, two nanocrystallines bcc-Fe(Co,Ni) and fcc-Co phases were identified. The crystallite size decreases to the nanoscale and the level of microstrains increases, as a function of milling time. These behaviors are related to the increase of dislocations and accumulation of stacking faults. Moreover, a decrease in coercivity (Hc) with increasing the milling time was also observed. This variation is explained based on the crystallite size and strain variations in the samples during milling. Depending on the structural state of the milled powders, the DSC analyses demonstrate several overlapping exothermic peaks over the temperature range of 100 to 700°C.

Keywords: FeCoNi alloy; High energy ball milling; X-ray diffraction; nanostructure; Magnetic properties; thermal stability.

MICROSTRUCTURAL CHANGES IN NANOCRYSTALLINE ($Ti_{0.9}Cr_{0.1}$)C POWDERS OBTAINED BY MECHANICAL ALLOYING

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Nanostructured ($Ti_{0.9}Cr_{0.1}$)C powders were obtained by mechanical alloying (MA) of elemental Ti, Cr and C powders, using a planetary ball mill. The effect of ball milling on microstructural change of nanocrystalline ($Ti_{0.9}Cr_{0.1}$)C powders was investigated. X-ray diffraction was used to evaluate the microstructural parameters, including lattice parameter, mean crystallite size and mean microstrain. The defects and surface stress generated during milling are deemed to be responsible for the increasing of lattice parameter. The crystallite size displays a diminishing trend with increasing milling time, which is consistent with the SEM observation. However, the mean microstrain reduces with milling time due to the strain relaxation by the formation of dislocations.

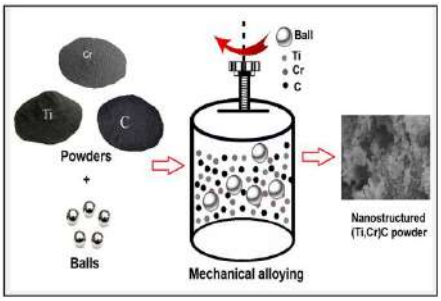

Fig. Schematic diagram of mechanical alloying.

Key words: (Ti,Cr)C; Powders; Mechanical alloying; Microstructure; X-ray diffraction; Nanocrystalline.

A new polymeric copper (I) complex containing hexafluorophosphate groups Synthesis and crystal structure determination of $\{[Cu(PF_6)(H_2O)_4]\}_n$

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A new one-dimensional (1D) copper (I) polymer with the formula $\{[Cu(PF_6)(H_2O)_4]\}_n$ was synthesized by slow evaporation at ambient temperature and structurally characterized by infrared, UV-visible and raman spectroscopy. The title compound cristallografically characterized by the single-crystal X-ray diffraction which crystallizes in the monoclinic system with the space group $P2_1/c$ and the unit cell dimensions: a= 5.3261(3) Å, b= 9.6090(5) Å, c= 7.2172(4) Å, β= 105.263(6) ° and Z=2. The original metal-inorganic crystals stack along the c crystal direction which is constituted by a distorted octahedral of $CuF_2(H_2O)_4$ contains four different Cu-O bond lengths that range between 1.9511 and 1.9542 Å and the Cu-F bond length is 2.3276 Å which the fluorine atoms makes this distortion. The octahedral (PF_6) is somewhat distorted too. Furthermore, the atomic arrangement revealed that the metal octahedral and the hexafluorophosphate octahedral shares a fluorine atom. These layers are bonded by establishing hydrogen bonds O-H…F. The use of a new Cu(I) in polymeric composite appears as a novel approach in catalysis. Vigorous interest continues towards the synthesis and structural elucidation of metal-inorganic polymer crystalline solids.

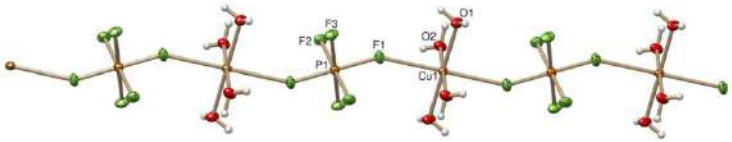

Figure: Representation of the crystal packing, projected along c.

Keywords: Single-Crystal/X-Ray study/Spectroscopic investigation.

Effect of the addition of BaO on the structural, thermochemistry properties of phosphate glasses

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Phosphate glasses are largely investigated owning to their interesting physical properties such as high coefficient of thermal expansion, low softening and melting temperatures and high transparency in UV region compared to silica based glasses. These properties make them candidates for solid state batteries, radiation shielding materials and dosimeters. Phosphate glasses and glass ceramics are already used for as sealant for nuclear waste, in tissue engineering, bone graft scaffold and amplifiers in solid state lasers. Nevertheless, the low chemical durability of these glasses limits their use in many applications.
The present work is devoted to study of the structure properties relationship of barium polyphosphate glasses with the formula $(100\text{-}x)NaPO_3\text{-}xBaO$ with x ranging from 5 to 25 molar%. The glasses were synthesized with the melt quenching technique and show a limit of vitrification of 25% of BaO. The FTIR and Solid State NMR of ^{31}P show the depolymerization of the metaphosphate network. The density and thermal analysis of the glasses show the shrinking and the progressive reticulation of the phosphate network. The chemical durability analysis shows the strengthening of the polyphosphate chains towards water attack. The thermochemical study carried out in the Setaram C80 Tian-Calvet microcalorimeter, shows the decrease of the dissolution enthalpy as the content of BaO increases in the glasses.

Key words: Glass structure, barium phosphate glass, Glass thermochemistry, Chemical durability

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Electrical and Vibrational Studies of $K_{0.4}Na_{3.6}Co(MoO_4)_3$

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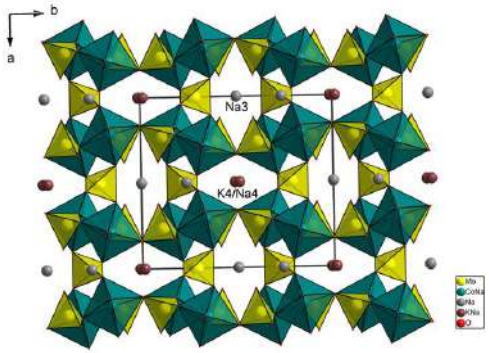
$K_{0.4}Na_{3.6}Co(MoO_4)_3$ compound was obtained by solid state reaction. It crystallizes in the monoclinic system, space group C2/c with cell parameters a = 12.8054 (8) Å, b = 13.532 (9) Å, c = 7.188 (6) Å, β = 112.24 (8) °, V = 1153.10 (1) $Å^3$. The three-dimensional framework consists of a network of infinite chains of edge-sharing $Co(2)O_{10}$ dimers which are linked together by MoO_4 tetrahedra. They form a framework enclosing two types of hexagonal tunnels where Na^+ and K^+ cations reside (Fig. 1).
The material was characterized by Scanning electron microscope (SEM) and infrared spectroscopy. The electrical properties were measured using impedance spectroscopy. The structural model was confirmed by charge distribution (CD) and bond valence sum (BVS) methods.



Projection of $K_{0.4}Na_{3.6}Co(MoO_4)_3$ structure, along c axis, showing the hexagonal channels.

Key words: crystal structure; X-ray diffraction; BVS; electrical properties.

Structure features, thermal analysis, model and experiment magnetic behavior of tetranuclear oxo and sulfato bridged copper (II) aggregate

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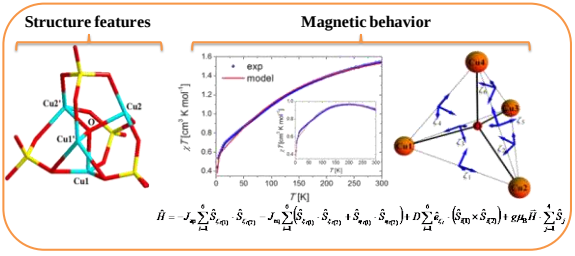
The novel tetranuclear copper (II) cluster namely $(C_5H_7N_2)_2[Cu_4(\mu_4\text{-}O)(\mu_3\text{-}SO_4)_4(C_5H_6N_2)_4]$ (1) was obtained by the hydrothermal reaction of copper (II) sulfate and the monodentate 4-aminopyridine ligand. Single-crystal X-ray diffraction indicated that compound 1 crystallizes in the monoclinic system, space group C2/c, a=25.827(5) Å, b=10.657(5) Å, c=19.838(5) Å, β=121.195(5)°, V=4671(3) $Å^3$ and Z=4. The discrete $[Cu_4(\mu_4\text{-}O)(\mu_3\text{-}SO_4)_4(C_5H_6N_2)_4]^{2-}$ anion present in (1) contains $Cu_4(\mu_4\text{-}O)$ tetrahedra with the copper centers held together by four sulfate groups in two different coordinated modes: $\eta^1.\eta^1.\eta^1.\mu_3$:3.1110 and $\eta^2.\eta^1.\mu_3$:3.2100 (Harris notation). Again, the structural arrangement and the intermolecular interactions such as hydrogen-bonding are discussed from which supramolecular assembly was formed. The thermal behavior of the compound, studied by TG-DSC, showed that the successive decomposition stages of precursor lead to the formation of intermediate phases during heating giving rise to the copper oxide as a final product at 700 °C. The magnetic properties of (1) have been studied by variable temperature dc magnetic susceptibility techniques. Using an idealized geometry of the tetranuclear cluster the magnitude of exchange coupling constants is examined. The calculations within the simplified model reveal a relatively strong superexchange interaction between the Cu(II) ions (on the order of a 100 cm^{-1}) indicating a substantial anisotropy with the apical coupling being ferromagnetic while the equatorial coupling favoring an antiferromagnetic arrangement of the spin components.



KEYWORDS: Crystal structure, Cluster compound, Supramolecular, Magnetic properties.

Novel Ag/Ce-ZrO_2 catalyst for the low temperature total oxidation of toluene

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[b]*Institut Charles Gerhardt Montpellier UMR 5253, CNRS–UM–ENSCM, Matériaux Avancés pour la Catalyse et la Santé, ENSCM, 240 avenue du Professeur Emile Jeanbrau, CS 60297, 34296 Montpellier Cedex 5, France*

New Ag/ZrO_2 and $Ag/Ce\text{-}ZrO_2$ catalysts have been developed for the low temperature total oxidation of toluene ($C_6H_5\text{-}CH_3$). The ZrO_2 and $Ce\text{-}ZrO_2$ aerogel supports were synthesized via the one-step sol-gel procedure. While, silver (Ag) was deposited onto the supports by the impregnation method. N_2-adsorption-desorption at 77 K, XRD, H_2-TPR and NH_3-TPD have been used to examine the physico-chemical properties of the samples. The results show that the aerogel supports (ZrO_2; $Ce\text{-}ZrO_2$) and the catalysts (Ag/ZrO_2 ; $Ag/Ce\text{-}ZrO_2$), calcined at 550 °C, are classified as mesoporous materials and develop essentially the diffraction peaks of the tetragonal phase of ZrO_2 (Table 1 and Figure 1). The addition of cerium modifies the acidic and the redox properties of Ag/ZrO_2 catalyst and affects its activity in the total oxidation of $C_6H_5\text{-}CH_3$. The Ag/ZrO_2 catalyst exhibits high activity in the total oxidation of toluene. The addition of cerium enhanced the total oxidation of toluene, especially at low temperature.

Table 1. Textural properties of the samples

Catalyst	S_{BET} (m^2/g)	Vp (cm^3/g)	Dp (Å)
ZrO_2	107	0,33	88
10Ce-ZrO_2	119	0,35	87
2Ag/ZrO_2	76	0.24	97
2Ag/10Ce-ZrO_2	73	0,31	127

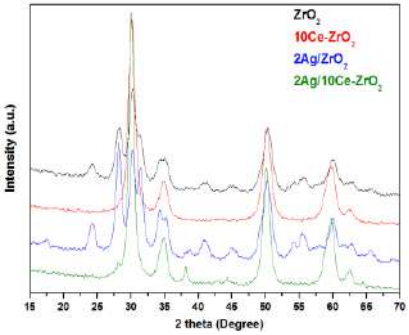

Figure.1. XRD patterns of samples

Experimental study on the structure, vibrational and thermal properties of crystal structure of tetraaqua-(5,5'-Dimethyl-2,2'-Bipyridyl)nickel(II) selenate

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HORCAJADA Patricia[2], and ELAOUD Zakaria[1]

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[2] *Advanced Porous Materials Unit, University of Madrid, IMDEA Energy, Madrid, Spain.*
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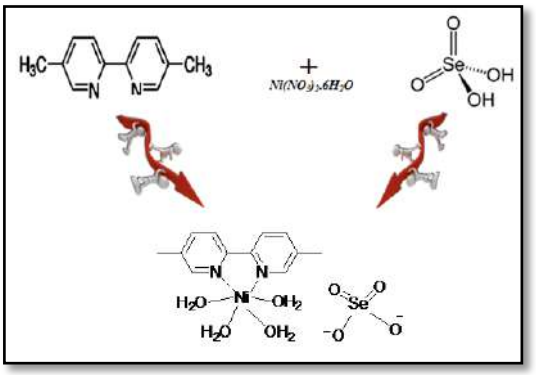
We particularly investigate a new compound $[Ni(C_{12}H_{12}N_2)(H_2O)_4]SeO_4$, that it was hydrothermally synthesized by reaction of 5,5'-dimethyl-2,2'-bipyridne, denoted (dmbpy), metal salt and selenic acid. The blue crystals are characterized by X-ray single crystal diffraction, infrared and Raman spectroscopy. The thermogravimetric analysis indicated that the dehydration occurs in one step leading to an anhydrous compound. The title compound belongs to the monoclinic system with the centrosymmetric space group $P2_1/c$ and the following parameters a = 9.6916 (2) Å, b = 9.7534 (2) Å, c = 17.8240 (4) Å, β = 93.20°, V = 1682.20 (6) $Å^3$ and Z = 4 . The asymmetric unit contains one free SeO_4^{2-} counter-ion and $[Ni(dmbpy)(H_2O)_4]^{2+}$ complex cation. The crystal structure of the title compound is built up from infinite parallel two-dimensional planes, containing all the components of the structure and perpendicular to the axis b. The aqua ligands are connected to the sulfate anion via O-H…O hydrogen bonds that stabilize the three-dimensional network.

Keywords: hybrid compound, RXD, ATG-DSC.

Graphical Abstract:



Innovative solid oxide fuel cells based on La_2MoSO_9 electrolyte: Synthesis and characterization

Wiem Jabeur*, Noureddine Mhadhbi and Houcine Naïli

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Solid oxide fuel cell (SOFC) technology has been under development for a broad range of power generation applications, thanks to their high yields and their ability to cogenerate electricity and heat by operating at temperatures between 500 and 1000 °C. Operating at high temperature to assure conduction of oxide ions, current challenge is to decrease this temperature to reduce degradation reactions at interfaces and manufacturing costs. This can be achieved by the search for new materials that perform better than yttriated zirconia (YSZ: ZrO_2-Y_2O_3), with an operating temperature is 800-1000°C, and/or by the optimization of the shapes or devices. $La_2Mo_2O_9$ derived from the LAMOX material has attracted great interest due to its high oxide-ion conductivity. This material retains a monoclinic α-polymorph (sp.gr. $P2_1$) **[1]** phase at low temperature **[2]** and goes through a reversible phase transition at 580°C to form cubic β-polymorph **[3, 4]**. The β-polymorph has a conductivity of ~6×10^{-2} S/cm at 800°C **[5]**, which is higher than those of the stabilized zirconia in an intermediate temperature range **[6]**. While the conductivity of α-polymorph is two orders of magnitude lower than that of β-polymorph **[3]**. In order to obtain ceramic electrolytes for anionic and also protonic conductive fuel of the $La_{2-2x}(Mo, S)_2O_{9-3x}\cdot xH_2O$ type, isovalent substitution for the molybdenum with sulfur has been carried. The result is unexpected. Another pure and homogeneous composition appeared and it is responsible for the stabilization of the disordered form β at room temperature. The experimental study will include the following aspects: The preparation techniques of material that is derived from LAMOX, the structural characterization by DRX at room temperature and in temperature, thermal analyzes by ATD/ATG, the conductivity measurements and the vibrational study by IR and Raman of La_2MoSO_9. All these analyzes show finally that an original and important phase derived from LAMOX family material is obtained.

Key words: LAMOX, oxide ion conductor, X-ray powder diffraction, thermal studies, conductivity, Infrared and Raman spectroscopy.

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Synthesis, structure and characterization of a hybrid centrosymmetric material (4-Dimethylaminopyridinium nitrate gallic acid monohydrate).

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New hybrid material: 4-Dimethylaminopyridinium nitrate gallic acid monohydrate abbreviated DNGA monohydrate was successfully synthesized by slow evaporation method at room temperature. X-ray diffraction (XRD) on a single crystal showed that the latter was crystallized in P-1 space group. Likewise, thermal analyses demonstrated the stability of our crystal up to 80°C. Besides, the analysis of the infrared spectrum (FTIR), allowed us to confirm the presence of the different groups present in the structure. Furthermore, by studying the UV-Visible spectrum, the transparency of our crystal was proven. The stability and cohesion of the structure is ensured via hydrogen bonds.

Synthesis and Biophysicochemical Characterization of a New Organically Templated Cobaltoborate $(C_4H_{12}N_2)[CoB_{12}O_{14}(OH)_{12}].6H_2O$

Nabil Jamai, Mohamed Rzaigui and Samah Akriche Toumi

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A new organically templated cobaltoborate $(C_4H_{12}N_2)[CoB_{12}O_{14}(OH)_{12}].6H_2O$ (*Abbrev. PCoB6*) is synthesized and structurally determined by X-ray diffraction and characterized by experimental IR, UV-Vis, fluorescence spectroscopy measurements and TDA-TG thermal analysis. PCoB6 crystallizes in the triclinic space group P-1 with a = 8.226 (3) Å, b = 10.157 (2) Å, c = 11.298 (4) Å, α = 65.98 (2)°, β = 74.20 (3)°, γ = 69.03 (5)°, Z = 1, and V = 796.2 (4) $Å^3$. Its structure exhibits anionic porous *3D*-hydrogen bonded framework of $[CoB_{12}O_{14}(OH)_{12}]^{2-}$ monomers filled with piperazinium dications. It thermally decomposed in multiple steps involving dehydration and release of piperazinium group leading to a novel unknown phase which undergoes crystallization. Moreover, the fluorescent and biological analyses reveal red photoluminescence and promising enhancement of antibacterial activity of reported material in comparison with its parent reactants.

Key words: Cobaltoborate, Synthesis, X-ray diffraction, Thermal analysis, Fluorescence, Biological activity.

A Hirshfeld Surface Analysis, Synthesis, structure and characterization of a new Ni(II) complex with the bidentate ligand homopiperazine

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Nitrogen heterocyclic ligands can coordinate a variety of metal ions yielding different geometries based on the metal ion and its oxidation state [1], moreover, their transition-metal complexes show interesting structural, chemical, and physical properties [2-5]. A new Ni(II) complex with the bidentate ligand homopiperazine, $[Ni(C_5H_{12}N_2)_2](CH_3COO)_2.4H_2O$, has been prepared and characterized by single crystal X-ray diffraction, elemental analysis, solid state NMR and IR spectroscopy. The title compound crystallizes in the monoclinic space group $P2_1/n$ with a = 7.4445 (6) Å; b =8.7471 (7) Å; c = 16.1529 (13) Å; β = 93.281° (3) and Z = 2. The basic coordination patterns of the homopiperazine coordinated metal cations are slightly distorted square planar. In the atomic arrangement, the cationic complexes $[Ni(C_5H_{12}N_2)_2]^{2+}$ are interconnected by the CH_3COO^- anions via N–H…O and C–H…O hydrogen bonds to form layers parallel to the (a, b) plane. These layers are connected by hydrogen bonds generated with water molecules to build a three dimensional network. Intermolecular interactions were investigated by Hirshfeld surfaces. The ^{13}C and ^{15}N CP–MAS NMR spectra are discussed and the vibrational absorption bands were identified by infrared spectroscopy. Electronic properties such as HOMO and LUMO energies were derived.

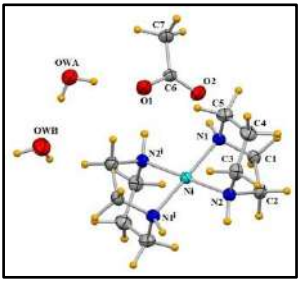

Fig. The molecular structure of the title compound showing the atom-numbering scheme and displacement ellipsoids drawn at the 40% probability level. (i : −x, −y+1, −z+1)

Keywords: Square planar; Ni(II) complex; X-ray diffraction; Coordination compound ; Hirshfeld surface; contact enrichment ratio; DFT calculations.

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Alfa fiber polypropylene composites: Effect of fiber alkaline extracted and steam-exploded fibers content on Mechanical properties

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Eco-composites made from Alfa fibers and polypropylene matrix were manufactured using twin-screw extrusion. The fibers were extracted from stems using sodium hydroxide solution and steam explosion. The effect of fiber extraction method and of fiber concentration inside the composites was studied in terms of tensile properties. It has been shown that sodium hydroxide treatment was stronger than stem explosion concerning the removal of non-cellulosic components. Moreover, the optimum fiber concentrations were 15% for tensile strength and 30% for Young's modulus.

Microstructure evolution of Fe-48wt%Ni alloy processed by Equal channel angular pressing process

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The evolution of the microstructure of a Fe-48wt%Ni alloy submitted to severe plastic deformation using equal channel angular pressing (ECAP) was investigated using electron backscatter diffraction (EBSD). A stacking of 9 sheets was introduced and pressed into die with an inner angles of Φ=90º and an outer arc of curvature ψ= 17° from 1 to 2 passes at room temperature following route A. Another test of ECAP (1 pass) was also done using a parallelepipedic sample of the same material. The present study will be concentrated on analyzing the initial state (N=0), the peripheral plates and the central plate of the stack after 1 and 2 passes and 3 sections of the parallelepipedic sample after 1 pass. Results obtained after each pass show a heterogeneous grain structure. Significant change in grain misorientation were also noted (Fig.). Different parameters can affect the ECAP process and as a consequence the heterogeneity. One of these parameters is the crystallographic orientation of grains, which is in relation with slip systems and/or deriving from the geometry and the conception of the die.

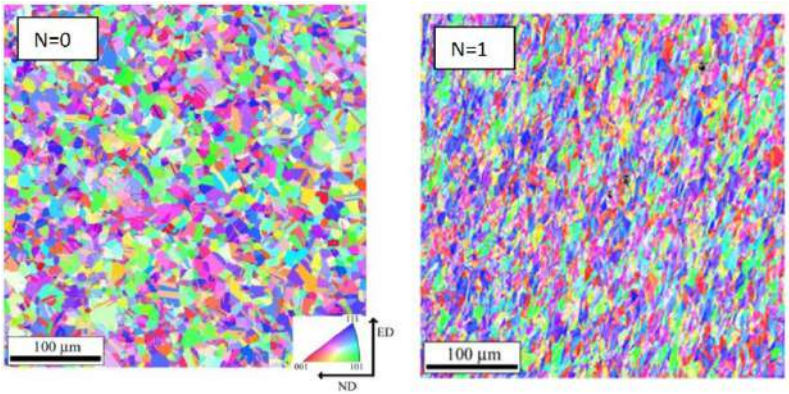

Fig. IPF maps showing the microstructure of Fe-48Ni alloy before and after ECAP process.

Keywords: ECAP, texture, Fe-Ni alloy, microhardness, EBSD, DRX.

Synthesis, structural, morphological, optical and magnetic Characterization of Spinel ferrites ($Cu_xCo_{0.5-x}Ni_{0.5}Fe_2O_4$) nanoparticles by co-precipitation method

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We report the synthesis of $Cu_xCo_{0.5-x}Ni_{0.5}Fe_2O_4$ with x= (0.1, 0.2, 0.3, 0.4) nanoparticles using the co-precipitation method in the presence of oleic acid as a surfactant and coating material. The X-ray diffraction analysis confirmed the formation of cubic phase with Fd-3m space group of all the prepared nano-ferrites. The average crystallite size varies in the range of 21–38 nm with varying concentration of copper. The lattice parameter was found to increase with increase in copper substitution. This may be attributed to larger ionic radius of copper as compared to that of cobalt. TEM and SEM analysis showed the monodispersion and cubic-like nanostructure. Two prominent stretching bands were observed in FT-IR spectra around 400–600 cm^{-1}. These two bands confirmed the spinel structure of the prepared nanoparticles. Raman spectroscopy is used to verify that we have synthesized ferrite spinels and determines their phonon modes. The thermal decomposition of $Cu_xCo_{0.5-x}Ni_{0.5}Fe_2O_4$ was investigated by TGA\DTA. The optical study UV-visible is used to calculate the optical band gap energies. The products exhibited the attractive magnetic properties with high saturation magnetization, which were examined by a vibrating sample magnetometer (VSM).

Synthesis, crystal structure, spectroscopic characterization and magnetic properties of new hybrid compound aquatrichlorocuprate 2-methyl imidazolium

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A new organic-inorganic complex, aquatrichlorocuprate 2-methyl imidazolium was synthesized and characterized by single crystal and powder X-ray diffraction, Hirshfeld surface, scanning electronic microscopy, vibrational spectra, thermal analyses (TGA and DTA) and magnetic properties. At room temperature, it crystallizes in the monoclinic $P2_1/c$ space group with the following unit-cell parameters: a= 9.0805(3), b= 14.0304(5), c= 7.3884(2) Å, β= 97.358(3) °, V= 933.55(6) A^3 and Z= 4. The asymmetric unit consists of $[CuCl_3 (H_2O)]$ anions and the 2-methyl imidazolium cations $(C_4H_7N_2)^+$, which are interlinked by three varieties of hydrogen bonding interactions, namely N–H···O, N–H···Cl and O–H···Cl, forming a chain network. Hirshfeld surface analysis was used to investigate intermolecular interactions, as well 2D finger plots were conducted to reveal the contribution of these interactions in the crystal structure quantitatively. The X-ray powder is in agreement with the X-ray structure. Scanning electronic microscopy (SEM) was carried out. Furthermore, the room temperature Infra Red (IR) spectrum of the title compound was analyzed on the basis of data found in the literature. Solid state ^{13}C NMR spectrum shows four signals, confirming the solid state structure determined by X-ray diffraction. Thermal analysis shows two anomalies at 257 and 485 °C. Photoluminescence measurements showed a strong emission line at 2.81 eV associated with radiative recombinations of excitons confined within the $[CuCl_3 (H_2O)]$ which were investigated at room temperature. The vibrating sample magnetometer (VSM) was used to obtain the hysteresis parameters.

ELECTROCHEMICAL PROPERTIES OF VANADIUM OXIDE NANOURCHINS AS CATHODE MATERIAL

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Recently, many studies have shown that nanostructured materials can provide a new opportunity to improve their electrochemical performance thanks to their nanoscale size, which could increase the electroactive surface area [1]. Among them, 3D hierarchical nanostructures could improve the rate capability; ensure shorter path length for Li^+ and electron transport, leading to an improved capacity retention. [2].

The present communication deals with the electrochemical performances of vanadium oxide nanourchin-like morphology. Electrochemical measurements were carried out on thin films deposited on to ITO modified glass electrodes. The obtained results revealed that despite the decrease of the current densities with repeated cycles, the insertion/extraction process of Li^+ cations remains reversible from the 1st to the 50th cycle. Furthermore, in order to investigate the kinetics of reactions as electrode materials, cyclic voltammetry curves were carried out at various scan rates from 5 to 50 $mV.s^{-1}$. It was found that peak currents (Ip) follow a power law relationship with the sweep rate. The results indicate that the reaction is controlled by a mixture of two contributions: charge transfer process and diffusion process.

Key words: Nanostructures; Electrochemical properties.

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Enhancement of dielectric properties of $CaCu_3Ti_4O_{12}$ ceramics by Sr and Ni doping ions

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The perovskite-based ferroelectric ceramics such as $(Pb,La)(Zr,Ti)O_3$(PLZT) or the relaxor ferroelectrics like $SrBi_2Nb_2O_9$ (SBN), are considered as the famous materials possessing high dielectric constant. However, their dielectric constant is closely related to the temperature and phase transition, which is undesirable for most industrial and electronics applications. In recent years, a new material called $CaCu_3Ti_4O_{12}$ (CCTO) has been discovered by Subramanian et al. in 2000, which has attracted enormous research attention due to its giant dielectric constant ($\sim 10^4$) and it remains constant in the temperature range of 100 to 400K.[1,2].
The aim of this work is to improve the dielectric properties and the stability of the CCTO ceramic by co-doping with Sr-Ni doping ions. All the ceramics were synthesized by solid state reaction method. In the first part of this work, we attempt to find the optimum conditions for synthesis. In the second part, the effect of Sr doping ratio on microstructure as well as the dielectric and electric properties of un-doped and Ni-doped CCTO were investigated.
X-Ray diffraction data revealed the formation of $CaCu_3Ti_4O_{12}$ as a major phase for all the ceramics samples ($Ca_{1-x}Sr_xCu_{1-y}Ni_yTi_4O_{12}$ with x=0.1/0.5/0.2 and y=0.1). SEM micrographs revealed that the grain growth of Ni-doped CCTO was strongly influenced by Sr doping ions at different ratio of Sr doping ions. EDS results revealed the presence of TiO_2 phase at grain boundaries and is maximum for $Ca_{0.8}Sr_{0.2}Cu_{2.9}Ni_{0.1}Ti_4O_{12}$. The $Ca_{0.9}Sr_{0.1}Cu_{2.9}Ni_{0.1}Ti_4O_{12}$ shows the excellent dielectric constant and the lowest dielectric losses at 10 KHz ($\varepsilon \approx 43890$ and $\tan\delta \approx 0.05$) with good frequency stability. Additionally, the results revealed that the permittivity and losses values are in perfect correlation with the grain boundaries capacitance and resistance, respectively.

Key words: co-doped CCTO, SEM, EDS, dielectric properties

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Influence of sintering conditions on microstructure and electrical properties of $CaCu_{2.9}Ni_{0.1}Ti_{3.9}Ni_{0.1}O_{12}$ (CCNTNO) ceramics

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Undoped $CaCu_3Ti_4O_{12}$ is well known for its colossal dielectric constant in a broad temperature and frequency range. A multi mechanism was proposed to explain the origin of the colossal dielectric constant. According to the bibliography, dielectric properties of the interesting material was slightly changed by doping with many elements such as Mg, Co, Sr, ... In the context, new Ni-doped CCTO solid solution were prepared by the solid–state process, and sintered at 1100 °C for different time. Their crystalline structure, microstructural and electrical properties were studied. The results exhibit that the grain size is affected by the sintering time. We note that increasing the sintering time, most grains grow up firstly, and then some diminish for higher times. It should be noted that prolonging sintering time from 6 to 12 hours promotes grain growth, microstructural densification, and improves the dielectric properties. Particularly ceramics sintered at 1100°C for 12 h exhibit giant dielectric constant of 10^5 and dielectric loss. Whereas if the sintered time is about 24h, the impedance spectrum measurement show that not only does the dielectric loss decrease but also the dielectric constant remains sufficiently high. Sample presents low dielectric loss (<0.1). The most suitable sintering time is 24 h.

Key words: $CaCu_3Ti_4O_{12}$ ceramics; dielectric constant; dielectric loss; sintering time

Phase equilibria of the Gd-Fe-Cu system at 800°C

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Phase relations in the Gd-Fe-Cu system were derived from X-ray diffraction data and, optionally, from scanning electron microscopy energy dispersive X-ray spectroscopy (SEM/EDS) of arc-melted samples which have been annealed at 800°C for 7 days and quenched to room temperature. The binary compounds Gd_2Fe_{17}, $GdFe_3$, $GdFe_2$, GdCu, $GdCu_2$, $GdCu_5$ and $Gd_{0.8}Cu_{5.4}$ have been confirmed. While, the binary compound Gd_6Fe_{23} was not observed up to 800°C, but it became stable at 900°C. The isothermal section of the phase diagram at 800°C consists of twelve single-phase regions, eleven two-phase regions and ten three-phase regions. The limiting compositions of the solid solution ranges of the binary compounds have been determined. The solubility range of $GdFe_{2-x}Cu_x$ extended from about 0 to 6.6 at% Cu. The compound $GdFe_{17-x}Cu_x$ with the Th_2Zn_{17}-type structure was found to have a broad solubility ranging from 0 to 7.89 at% Cu. The maximum solubility of Fe in $GdCu_{1-x}Fe_x$, $GdCu_{2-x}Fe_x$, $GdCu_{5-x}Fe_x$ and $Gd_{0.8}Cu_{5.4-x}Fe_x$ are about 25, 20, 3.33 and 3.22 at% Fe, respectively. One liquid area was present at the Gd-rich corners of this isothermal section.

Synthesis, Structure supported with Hirshfeld Surface Analysis and Characterization of 3D-Supramolecular Based Decavanadate Compound

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A new organic–inorganic hybrid compound, 2-amino-1,3-diazinium decavanadate dihydrate, (*Abbrev. 2AV10*), is synthesized and characterized by means X-Ray diffraction, IR, UV-vis and fluorescence measurements. X-Ray diffraction shows that it crystallizes in the monoclinic system with space group $P2_1/c$ and cell parameters: a = 9.8177(10) Å, b = 18.1521(17) Å, c = 14.2793(14) Å and β = 97.217(6)°. Crystal structure supported with Hirshfeld surface analysis employing 3D molecular surface contours and 2D fingerprint, reveal that the intermolecular interactions are associated with O…H/H…O (56.4%) and H…H (18.2%) inter-contacts between $(C_4H_6N_3)^+$ cations, lattice water molecules and $[V_{10}O_{28}]^{6-}$ polyanions. Moreover, the compound was evaluated for its optical properties and microbial behavior in both bacterial and fungal pathogens of clinical importance and was found to exhibit blue luminescence and promising antimicrobial effects on both strains system.

Key words: Decavanadate, X-ray diffraction, Hirshfeld surface analysis, Optical properties, Bacterial and fungal pathogens.

Novel Co/ZrO_2 aerogel catalysts for the total oxidation of methane: Effect of Co content

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New Co/ZrO_2 aerogel catalysts with variable quantities of Co (5; 7.5 and 10 wt.%) were prepared for the total oxidation of methane (CH_4). The physico-chemical properties of the obtained catalysts were examined by means of N_2-adsorption-desorption at 77 K, XRD, H_2-TPR and DRUV-vis spectroscopy. The N_2 adsorption-desorption results (Table 1) show that all the solids calcined at 550 °C are classified as mesoporous materials and exhibit high surface area and large porosity. The XRD patterns reveal that ZrO_2 and Co/ZrO_2 are well structured materials and develop essentially the diffraction peaks of the tetragonal phase of ZrO_2. The H_2-TPR analysis demonstrates that the increase of the Co quantity modifies the redox properties of the catalysts suggesting that the nature of Cobalt surface species and their interaction with zirconia are affected by the Co content.

Table 1. Textural properties of the samples.

Catalyst	**S_{BET} (m^2/g)**	**Vp (cm^3/g)**	**Dp (Å)**
ZrO_2	115	0.42	108
5% Co/ZrO_2	106	0.47	121
7.5% Co/ZrO_2	103	0.51	152
10% Co/ZrO_2	99	0.63	188

The results of CH_4 oxidation show that ZrO_2 support is inactive in this reaction, while, the addition of cobalt leads to Co/ZrO_2 active catalysts for this reaction. The activity of Co/ZrO_2 aerogel catalysts is influenced by the amount.

Isothermal Cross-Sections of Nd-Fe-Ge System at 800°C

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The phase equilibrium of the ternary Nd–Fe–Co system at 800°C was investigated by means of powder X-ray diffraction (XRD) and scanning electron microscopy–energy dispersive X- ray spectroscopy(EDS). Eight binary compounds, ie, Nd_2Co_{17}, $NdCo_5$, Nd_5Co_{19}, Nd_2Co_7, $NdCo_3$, $NdCo_2$, Nd_3Co, Nd_2Fe_{17} were identified to exist at this isothermal section. This isothermal section consist seventeen two phases and eleven three phases regions. All measured compositions and unit-cell refinements were performed at room temperature from quenched samples annealed at 800 °C for one week. The maximum solubility at 800°C of Fe in $NdCo_{2-x}$ Fe_x ($MgCu_2$-type structure,$Fd\bar{3}m$), $NdCo_{3-x}Fe_x$ ($PuNi_3$-type structure, $R\bar{3}m$ space group), $Nd_2Co_{7-x}Fe_x$ (Ce_2Ni_7-type structure, $R\bar{3}m$),Nd_5Co_{19-x} Fe_x($CeCo_{19}$-type structure, $R\bar{3}m$ space group) and $NdCo_{5-x}Fe_x$($CaCu_5$-type structure,P6/mmm) and $Nd_2Co_{17-x}Fe_x$(Th_2Zn_{17} type structure, $R\bar{3}m$) are about 32.5 at.% Fe, 52,5 at.% Fe, 12,5 at.% Fe, 10 at.% Fe and 11,25 at.% Fe respectively. The solid solubility range of Co in Nd_2Fe_{17} form discontinuous series of 3 ranges is about 0-27,5%at Co, 32,5 -45%at Co and 63,75 – 80at%Co. The solid solubility range of Fe in Nd_2Co_{17} is about 0- 11,25 at% Fe. There is no solubility of Fe in Nd_3Co.

Synthesis, characterization and visible-light-driven photocatalytic performance of $Na_2Mn_2(MoO_4)_3$

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Single crystals of a new ternary molybdate, disodium dimanganese trimolybdate, $Na_2Mn_2(MoO_4)_3$ were synthesized using the flux method and the crystal structure was determined from X-ray diffraction data. Polycrystalline sample of the molybdate was prepared by conventional solid-state reaction method. The sample obtained was characterized by X-ray powder diffraction, infrared spectroscopy and Raman scattering. Thermal stability of the studied material was analyzed by differential scanning calorimetry (DSC). Optical properties were analyzed by UV–Vis diffuse reflectance spectroscopy and photoluminescence. The photocatalytic activity of the sample was evaluated for the degradation of methylene blue (MB) under visible light. The sample demonstrated an efficient photocatalytic activity and this result indicate that $Na_2Mn_2(MoO_4)$ could be a potential photocatalyst driven by visible-light.

Key words: Gap, photocatalytic, X-ray diffraction, methylene blue, visible-light.

Synthesis, Crystal Structure, and Catalytic Properties in diastereoselective nitroaldol (Henry) reaction of New Zinc(II) and Cadmium(II) compounds

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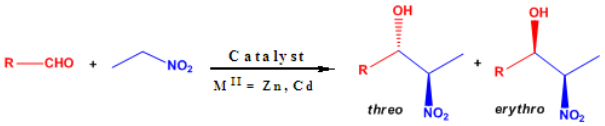
New organically complexes $[Zn(im)_4](NO_3)_2$ **(1)** and $[CdNa_2(\mu\text{-}L^2)]_n.6.34H_2O$ polymer **(2)**, were synthesized and their crystallografically characterized. The structure of the compound (1) contains [Zn(C3H4N2)]2+ cations and the NO^{3-} anions interlinked by N-H···O hydrogen bonds. Conversely, the polymeric structure of (2) is stabilized by the C–H···O intermolecular hydrogen bonds as well as by the interactions of ligands with sodium and cadmium cations. Additionally, sodium cations are involved in the interactions with water molecules which ensure O–H···O hydrogen bonds and participate in the structure cohesion. Both complexes were used as catalysts in an application to the Henry reaction **[1–3]**, and high yields were obtained under the optimum conditions **[4–6]**. They act as diastereoselective catalysts for the nitroaldol (Henry) reaction. The threo/erythro diastereoselectivity of the β-nitroalkanol from nitroethane and aliphatic or aromatic aldehydes with typical yields of 89% and threo/erythro ratio up to 89:11, depends on the used catalyst and substrate.



Scheme 1: Formation of nitroaldols by the Henry reaction.

Key words: Metal-organic frameworks, Crystal structure, Henry reaction Diastereoselectivity.

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Valorization of aluminum Chips into γ-Al_2O_3 and η-Al_2O_3 with high surface areas via the precipitation Route

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The aim of this study was to valorize aluminum chips collected from metal manufacturing industry in the region of Sfax into γ-Al_2O_3 and η-Al_2O_3 by acidic precipitation of sodium aluminate obtained via an alkaline attack of the scraps. The sodium aluminate solution was neutralized at RT with 2M HNO_3 solution to pH values of 6, 7, 8, 9 and 10. The neutralization pH is an important parameter that controls the nature of the precipitated phases. At pH 6, the XRD pattern showed the characteristic peaks of well crystallized bayerite. The further increase of pH to 7, 8 or 9 promotes only the precipitation of pseudo-boehmite. At pH 10, we obtained a mixture of bayerite and pseudo-boehmite. The aging time of the gel in the mother liquor is in important parameter that determines the nature of obtained phases. Without any aging time, pseudo-boehmite is the only phase present at pH 6. Whereas, when the gel was aged during 24 h and 48 h, well crystallized bayerite was formed which transforms to pseudo-boehimte if maintained in the mother liquor for 72 and 96 h. This result illustrates Oswald's rule of successive transformations. The nucleation and crystallization of the new phase occur in the supersaturated solution throughout the dissolution of the former phase. Through the calcination of bayerite at 400 °C, η-Al_2O_3 was obtained with specific surface area of 455 m^2/g, whereas the calcination of pseudoboehmite leads to globular aggregate made by fibrous crystals of γ-Al_2O_3. During the structural transformation by heat treatment of pseudo-boehmite into γ-Al_2O_3 and bayerite into η-Al_2O_3 a portion of the aluminium ions migrate from octahedral sites to tetrahedral sites in the alumina lattice. The tetrahedral to octahedral Al ratios are about 1 to 3 and 1 to 4 for γ-Al_2O_3 and η-Al_2O_3, respectively.

CHEMICAL SYNTHESIS, CRYSTAL STRUCTURE, VIBRATIONAL SPECTROSCOPY AND THERMAL ANALYSIS OF NEW HYBRID COMPOUND $C_{12}H_{14}N_2O_2S(Hg_4Cl_{12})_{0,5}.H_2O$

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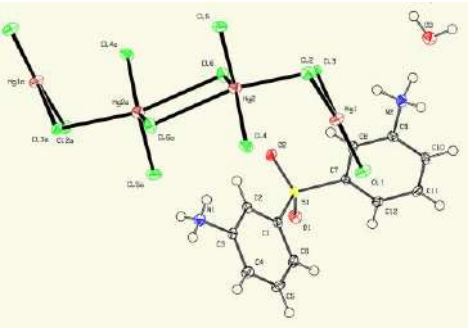
Single crystals of a new organic inorganic hybrid compound $C_{12}H_{14}N_2O_2S(Hg_4Cl_{12})_{0,5}.H_2O$ were synthesized by slow evaporation method at room temperature. The prepared compound was studied by X-ray diffraction, infrared absorption, Raman spectroscopy scatting, thermogravimetric analysis (TGA) and differential scanning calorimetric (DSC). This compound crystallizes in the monoclinic system, and $P2_1/_C$ space group. The cell parameters are: a =12.4741(5) Å, b = 15.0020(6) Å, c = 11.6397(4) Å and β = 101.860(1) °. The atomic arrangement can be described as inorganic chains parallel to the (b, c) plane at x = 0 and x = 1 connected with the organic groups by means of N-H…O and N-H…Cl hydrogen bonds to form 3D network. In the anionic entity, the mercury environment presented the distorted trigonal bipyramidal geometry on the one hand and a trigonal geometry on the other hand. The thermal analysis for the title compound shows two weight losses. The first loss corresponds to the departure of water molecule and the second one corresponds to the decomposition of the compound. The spectroscopic study by infrared absorption and Raman diffusion confirms the existence of the different modes of vibration of the groups observed in the structural study.



Asymmetric unit of $C_{12}H_{14}N_2O_2S(Hg_4Cl_{12})_{0,5}.H_2O$

Keywords: Hybrid material, Mercury, Spectroscopic study, thermal analysis, X-ray diffraction**.**

Synthesis, bio-physic-chemical properties of a di-V-substituted type Lindqvist polyoxotungstate

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A new V-substituted polytungstates of 1-ethylhydroxy-piperazin-1,4-dium (H_2pipEtOH), (H_2pipEtOH)$_2$[$V_2W_4O_{19}$].4H_2O (compound (1)), was synthesized in aqueous solution and characterized by ICP and elemental analysis, X-ray diffraction, IR, Raman, UV-Visible, luminescence and conductivity measurements. X-ray structural displays polymeric-3D-hydrogen bonded networks of porous extended polyanionic framework filled by H_2pipEtOH dications. The conductivity and dielectric behavior of compound (1) was studied as a function of temperature and frequency. The electrical analysis shows that the Nyquist plots (Z" versus Z') are well fitted to an equivalent circuit model which consists of a parallel combination of a bulk resistance R and constant phase elements CPE. Besides, the dielectric constants ε', ε″ and loss tangent (tan(δ)) versus frequency, at several temperatures, show a distribution of relaxation times. Furthermore, the activation energy responsible for relaxation has been evaluated and found to be close to the conductivity. Moreover, the biological properties were investigated showing promising antibacterial and antifungal activities.

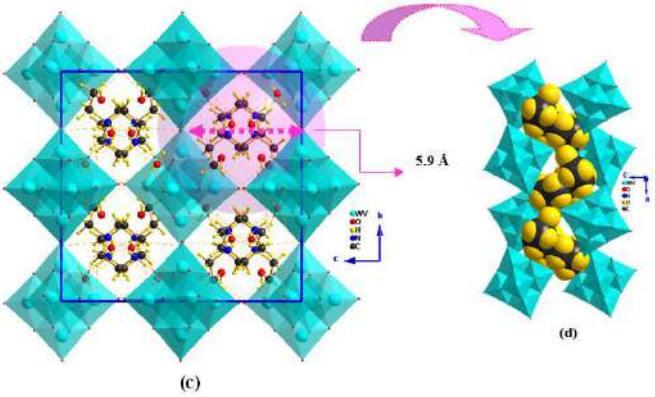

Fig. (a) Crystal packing of structure **1** down *a* -axis evidencing the porous architechture filled by organic moities. **(b)** Perspective view of channel voids extending along a - direction in structure **1**.

CRYSTAL STRUCTURE, DIELECTRIC CHARACTERISTICS AND CONDUCTION MECHANISM OF A NEW HYBRID MATERIAL, TETRA(PHENYLPIPERAZINIUM) DECACHLOROTRIPLUMBATE(II)

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Mohamed RZAIGUI, Wajda SMIRANI

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Monocrystalline $(C_{10}H_{15}N_2)_4Pb_3Cl_{10}$ was obtained via an aqueous solution reaction and characterized by single-crystal X-ray diffraction technique, elemental analysis, IR spectroscopy, UV–Vis spectrum, photoluminescence properties and thermal analysis. Structural characterization reveals that the title compound is built from $[Pb_3Cl_{10}]^{4-}$ units sharing Cl2 atom to form 2-D $[Pb_3Cl_{10}]_n^{4n-}$ layers, these anions are linked to the organic cations via N-H···Cl and C-H···Cl hydrogen bonding. Moreover, dielectric relaxation spectroscopy has also been determined to show different molecular motions. Measurements of AC conductivity as a function of frequency at different temperatures indicated the hopping conduction mechanism.

Keywords: Crystal structure, UV/Vis spectroscopy, Order-Desorder, AC conductivity spectra, Relaxation time.

Synthesis, photophysics characterization and X-ray molecular structure of the 2-methylimidazole tetrakis(tertbutylphenyl)porphyrinato)cadmium(II) complex

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Porphyrin macrocycle is an aromatic heterocyclic system .The size of the macrocycle is perfectly suited to bind almost all metal ions and indeed a large number of metals (Fe, Zn, Cu, Ni, Hg, Co, Cd etc.) can be inserted in the center of the macrocycle forming metalloporphyrins. The Cd(II) ion is an important transition metal element and many cadmium(II) porphrin complexes have been studied. However, till now, the Cd(II) porphyrin complexes having N-donor axial ligands are little. In this work we present the new 2-methylimidazole Tetrakis(tert-butylphenyl)porphyrinato)cadmium(II).coordination compound with the Formula [Cd^{II}(TBPP)(2-MeIm)]. The molecular structure of this new cadmium (II) porphyrin derivative has been established by single-crystal X-ray diffraction analysis. The compound crystallizes in the monoclinic crystal system with space group $P2_1/c$ and unit cell parameters: a=13.509(5) Å b=41.253(8) Å, c=11.221(5) Å, β=108.413(4)^o and Z=4 The asymmetric unit of the structure consists of one molecule of [Cd^{II}(TBPP)(2-MeIm)]. The supramolecular structure is constructed by C-H---π intermolecular interactions involving pyrrole and phenyl rings of the porphyrin.
The aim of the present work concerns the synthesis, the photophysics and characterization as well as the X-ray molecular structure of our new complex.

The effect of the imidazolium-based ionic liquid type in the performance of polymer actuators

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Actuators based on polymer blends of poly(vinylidene fluoride) (PVDF) with 40% of different ionic liquids (IL) are prepared by solvent casting. [C2mim][Cl], [C6mim][Cl], [C10mim] [Cl], [C2mim][NTf2], [C6mim][NTf2] and [C10mim][NTf2] were selected in order to evaluate the effect of anion and cation sizes in the bending properties [1]. The addition of ionic liquid into the polymer matrix decreases significantly its degree of crystallinity and the elastic modulus. It is also confirmed the good miscibility between PVDF and IL, determined by the interaction of the CF2 groups from the PVDF chains with the imidazolium ring in the ionic liquid (IL). The mechanical properties are independent on the IL type but the AC conductivity of the composites depend more on the anion type than on the size of the alkyl chain connected to the imidazolium based cation.
The bending movement of the IL/PVDF composites is correlated to with the anion and cation sizes and the best value of bending response (0.53%) being found for IL/PVDF composite with 40 wt% of [C6mim][Cl] at an applied voltage of 10 V square signal [2].

Key words: PVDF, Ionic liquid, Actuator, Electrical conductivity, Bending

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A NEW ROUTE FOR ACTIVE BELITE P-Ti-RICH CLINKER

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The main concerns of cement industry and cement researchers continue to be energy saving, environmental protection, pollution control while improving the cement properties. To reach these goals, cement industry and relative sectors are looking for using either alternative fuels or alternative raw materials in the production of clinker and cement. Industrial wastes are an alternative to fossil fuel energy; they are used essentially for their calorific values and also because of the similarity between the chemical composition of their ashes and usual raw materials, as well as for the purpose of reducing CO_2 emission. That kind of wastes can have high content of foreign ions, such as phosphates **[1]**, chromates **[2]**, borate **[3]** Etc. This ions can affect C_3S formation which is the main phase responsible for the hydraulic activity of clinker **[4]**. The phosphate contents of Portland cement clinkers are normally low (around 0.2 Wt.% as P_2O_5) although significant levels (up to 1 Wt.%) may be experienced when phosphate is present in the raw materials. In the present study, different clinkers with a small amount (varying between 1%Wt and 5%Wt) of phosphorus on the form of hydroxyapatite (HAP) were prepared in the laboratory with procedure similar to the industrial process. The formation of high temperature silicate phases (polymorphs of belite) was noticed. Crystallographic and microstructural studies were carried out on these phases to understand the mechanism of their formation and to predict their hydraulic properties. As a foreign ion in Portland cement clinker, titanium dioxide has a positive effect on the formation of clinker phases. This presence promotes the hydraulic properties by increasing the hydraulic activity of silicate phases, because of the crystal lattice distortion, as result of substitution of the Si^{4+} by the Ti^{4+} ions **[5]**. Based on last results, we attempted to combine titanium and phosphorus elements as additives for the clinker formation aiming to improve clinker properties.

Different polymorphs of calcium silicate were formed by changing P_2O_5 and TiO_2 contents in the raw mix. Minerals were analyzed by X-ray diffraction and IR spectroscopy. Rietveld method was adopted to carry out quantitative phase analyses. In addition, qualitative and quantitative mapping using analytical electron microscopes (STEM-EDX) of produced clinkers was carried out. STEM-EDX cartography showed the expected major elements (Ca, Si, Al and Fe) and minor additives (P, Ti). High solubility of the minor elements into silicate phases was observed according to STEM-EDX results. The incorporation of these foreign ions into silicate phases leads to think about a new belite clinker with an enhanced hydraulic activity.

The presence of P and Ti as foreign ions can impede the formation of tricalcium silicate (C_3S) phases and enhance the formation of belite (C_2S) type phases. The formation of belite-cement as an alternative to alite-cement could improve hydraulic and mechanical properties of new produced.

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A novel honeycomb-like Mn(II) μ-oxalato-bridged 2D coordination polymer: Crystal structure, spectroscopic, thermal and magnetic properties.

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Among the wide variety of organic ligands used to build up high-dimensional coordination polymers, the oxalate ligand has proved to be one of the most useful and played prominent role through conferring electronic [1], photochemical [2], magnetic [3] or optical [4] properties on those materials. The 2D honeycomb structure is the most common for those oxalates [5,6], hence, we have synthesized and characterized a new manganese(II) coordination polymer which exhibits a supramolecular assembled 3D network constructed by N—H…O and O—H…O hydrogen bonds and π–π interactions between adjacent 2D $\{[Mn_2(C_2O_4)_3]^{2-}\}_n$ layers. $\{(C_5H_8N_3)_2[Mn_2(C_2O_4)_3].3H_2O\}_n$ (I) is monoclinic, $P2_1/n$, a=9.756(3) Å, b=16.743(5) Å, c=15.420 (1) Å, β= 97.53(5)°, V=2497.1(11) $Å^3$ and Z=4. The complex has been fully characterized by elemental analysis, IR, single-crystal X-ray diffraction, UV-spectrum and optical band gap investigation, TGA-DSC, Rietveld refinement and magnetic susceptibility.

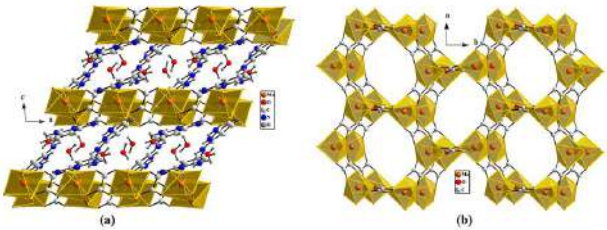

Fig 1. Sector from the title layered compound: (a) [010] projection and (b) [001] projection for anionic layers only.

Key words: 2D coordination polymer, manganese, oxalate ligand.

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Synthesis, crystal packing and second-order nonlinear optical in a hybrid material: $[(R)\text{-}C_7H_{16}N_2]_2[ZnBr_4]_2{\cdot}2H_2O$

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Chiral or noncentrosymmetric hybrid materials have drawn great interest due to their intriguing structures, specific physical, chemical and biological properties such as nonlinear optics (SHG), ferroelectricity, magnetochiral dichroism, switchable dielectric devices and enantioselective adsorption chiral separation activities. Such hybrid materials have been synthesized using homochiral components, either through direct incorporation of amino acids or organic amines. In this work, we report the use of homochiral organic molecule to direct crystallographic noncentrosymmetric in novel tetrahalogenozincate (II)-complex $[(R)\text{-}C_7H_{16}N_2]_2[ZnBr_4]_2{\cdot}2H_2O$. The crystal belongs to *P*1 space group with a good optical quality and high transmission in the visible and near infra-red spectral regions. $[(R)\text{-}C_7H_{16}N_2]_2[ZnBr_4]_2{\cdot}2H_2O$ is more than 0.81 times as efficient as KDP in second harmonic generation; making it a potentially attractive material for non-linear optical applications.

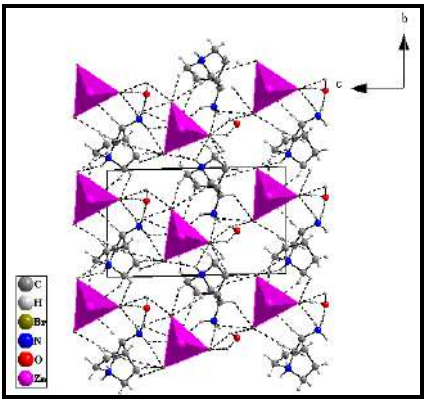

Figure: Projection of the crystal structure of $[(R)\text{-}C_7H_{16}N_2]_2[ZnBr_4]_2{\cdot}2H_2O$ along the a-axis.

Keywords: X-ray diffraction, Noncentrosymmetric, Homochiral amine and SHG activities

Elaboration, structural study and validation (CHARDI and BVS) of a new lyonsite $(Na_{0.4}Li_{0.6})(Li_2Cr)(MoO_4)_3$.

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The title compound, $(Na_{0.4}Li_{0.6})(Li_2Cr)(MoO_4)_3$, was prepared by solid-state reaction and characterized by X-ray diffraction. The compound crystallizes in the orthorhombic space group Pnma with a = 5.100(2) Å, b = 10.483(2) Å, c = 17.458(3) Å, V = 933.3(4) $Å^3$, Z= 2, R (F^2) = 3.53%, Rw (F^2) = 7.96%, S= 1.042. It's structure is isotypic to $Li_3Cr(MoO_4)_3$ whish has interesting magnetic and conductive proprieties [1,2]. We are interesting in this work to its structure investigation and CHARDI and BVS validation.

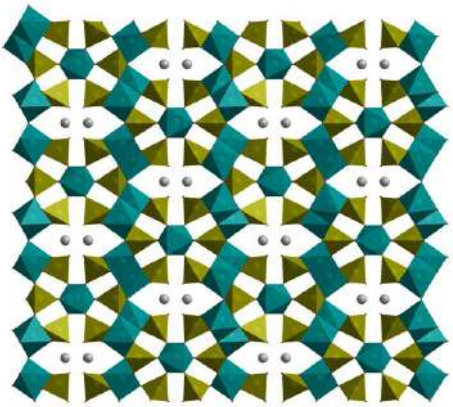

Fig. Projection of $(Na_{0.4}Li_{0.6})(Li_2Cr)(MoO_4)_3$ along the c axis.

Key words: lyonsite structure, framework, crystal structure, BVS, CHARDI.

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Preparation and Caracterisation of potassium-Aluminium phosphate glasses

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The effect of Al_2O_3 content on electrical conductivity and glass stability against crystallization in the system KH_2PO_4–Al_2O_3–P_2O_5 was investigated using Raman, IR, and impedance spectroscopies.
DSC observations were carried out on glasses from a series of KH_2PO_4/(1-x) P_2O_5/x Al_2O_3, with x = 0, 0.5, 0.1, 0.2 and 0.3 mol, to determine the relative glass stability. The DSC observations indicate that from x = 0.1 the glasses become more stable. For this composition (x = 0.1) the conductivity, which was obtained by AC-impedance spectroscopic studies, was 7.0×10^{-6} $S.cm^{-1}$ at room temperature.
Spectroscopic investigations reveal the depolymerization of the phosphate glass network by systematic conversion of Q^2 structural units into Q^1 and finally into Q^0 structural units. Even though Q^2 to Q^1 conversion is taking place due to breaking of P-O-P linkages, formation of P-O-Al linkages provide cross linking between short P-structural units, which make the glass network more rigid.

DESIGNING OF A NOVEL SYNTHETIC MITOCHONDRION-TARGETING TRI PHOSPHONIUM-FUNCTIONALIZED PHOTOSENSITIZING PHTHALOCYANINE

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Photodynamic therapy (PDT) is based on the association of photosensitizing molecules (PS) able to concentrate in tumor cells and of a focused light of appropriate wavelength (dependent on PS), to generate singlet oxygen which is highly reactive and toxic, thus induces cell death locally, without damaging healthy tissues. Among the different ways to optimize the efficiency of a photosensitizer, the targeting of mitochondria remains scarcely explored, especially for phthalocyanine-based photosensitizer, despite their advantages due to the maximum absorption at NIR wavelengths allowing deeper penetration of the light and excitation into the phototherapeutic window. Triphenylphosphonium moieties are known to have an excellent mitochondrion-targeting effect, hence triphosphonium-functionalized Zn(II) phthalocyanine was designed. The synthetic strategy will be presented and discussed together with its so-far completed synthetic and photo characterization.

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STRUCTURAL, BIOLOGICAL ACTIVITIES, DFT AND OPTICAL STUDIES OF A NEW NON-CENTROSYMMETRIC HYBRID MATERIAL $(C_4H_{12}N_2)[COBr_4]$

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The novel inorganic–organic hybrid $(C_4H_{12}N_2)[CoBr_4]$ was synthesized by slow evaporation and characterized by various physicochemical techniques. It crystallizes at room temperature in the non-centrosymmetric space group $P2_12_12_1$. The crystal structure of this compound was solved by direct methods and refined by full-matrix least squares on F2. The tetrahedral $(ZnBr_4)^{2-}$ anions are connected to the piperazinediium $(C_4H_{12}N_2)^{2+}$ cation by hydrogen bonding (N–H···Br). Theoretical calculations were performed using density functional theory with the B3LYP/LanL2DZ level for studying the molecular structure and vibrational spectra of the title compound. TDDFT calculations at the same level of theory was undertaken to investigate the electronic properties. The optical study reveals that the title compound undergoes three optical absorption bands at 641, 666 and 698 nm, respectively with an energy gap estimated to 2.23 eV. The synthesized product was also screened for in vitro antioxidant and antimicrobial activities.

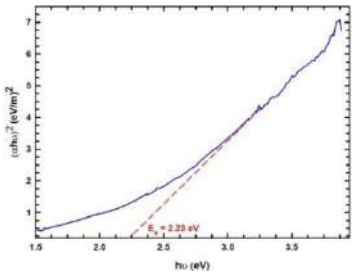

Fig. 1: The energy gap of the title compound $(C_4H_{12}N_2)[CoBr_4]$.

Keywords: Organic template, Metal halides, DFT calculations, thermal studies and Biological activities.

A New Synthesized Based Decavanadate Compound: Structural, Hirshfeld Surface, Spectroscopic and Antibacterial Analyzes

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A synthesis between V_2O_5 and 4-methoxybenzylamine in acidic aqueous solution resulted in a novel decavanadate compound. Its single crystal X-ray diffraction analysis revealed the decavanadate cage-like structure $[V_{10}O_{28}]^{6-}$ which constitutes the discrete anionic cluster associated to organic 4-methoxybenzylammonium cation into the hydrated neutral $(C_8H_{12}NO)_6V_{10}O_{28}.2H_2O$ compound. The 3D structure supramolecularity is non-covalent weak interactions mainly hydrogen bonding inter-contacts also elucidated by a detailed Hirshfeld surface analysis and two-dimensional fingerprint maps. The optical properties are also reported showing dielectric behavior and photoluminescent possession of reported material. Furthermore, its antibacterial activity against *G+ Enterococcus feacium* is tested and approved.

Key words: Decavanadate, Single crystal X-ray diffraction, Hirshfeld surface analysis, optical properties, Antibacterial activity.

Anticorrosive action of some trees cultivated in Tunisian arid zones on steel rebar's in alkaline chloride solution

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The aim objective of this work is to study the anticorrosive action of aqueous extracts of some trees cultivated in Tunisian arid zones on steel rebar's immersed in alkaline chloride medium simulating the interstitial solution of concrete contaminated with chlorides. The study was carried out by electrochemical (Polarisation and Electrochemical Impedance Spectroscopy: EIS) and analytical (SEM and EDS) techniques. According to polarisation results, the investigated extracts act as mixed type inhibitor. The EIS results showed that the charge transfer resistance (R_t) value increases while both the capacitance of the double layer (C_{dl}) and corrosion current density (j_{corr}) values decrease with the presence of aqueous extracts. The SEM and EDS analyses of samples in presence of aqueous extracts revealed the presence of protective inhibitor layer at the metallic surface, resulting in protection of steel rebar's from corrosion.

Keywords: Anticorrosive, alkaline solution, aqueous extract, trees.

Effects of Cu Additions on the Martensitic Transformation of Ni–Mn–Sn Shape Memory Alloys

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The nickel based ferromagnetic shape memory alloys are becoming an important element of sensor and actuator materials due to their large magnetic-field-induced strain and shape memory effects in recent years. The $Ni_{50-x}\,Mn_{36}\,Sn_{14}\,Cu_{x}$ (x=0, 1, 2) and $Ni_{50}\,Mn_{36}\,Sn_{13}Cu$ alloys were prepared by arc melting and then produced in a ribbon shape by rapid solidification using melt spinning technique. The present study reports on the analysis of microstructure and austenitic-martensitic transformation. The crystal structures of the fabricated alloys were determined by X-ray diffraction technique. In addition, the characteristic transformation temperatures and the thermodynamic parameters of the samples were determined by differential scanning calorimetry measurements. Moreover, this study investigated the effect of the substitution of Ni by Cu in $Ni_{50-x}\,Mn_{36}\,Sn_{14}\,Cu_{x}$ and the Sn by Cu in $Ni_{50}\,Mn_{36}\,Sn_{14-x}\,Cu_{x}$ systems.
The martensitic transformation temperatures, enthalpy and entropy changes were found to depend on the copper content.

Keywords: Shape memory alloys, Martensitic transformation, Cu additions, X-ray diffraction.

Fe-Ni-Mn alloy prepared by mechanical alloying and arc melting process

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A Fe-Ni-Mn alloy was synthesized by two different ways: arc melting and through the mechanical alloying process. Scanning electron microscopy and X-ray diffraction were carried out to monitor morphology and investigate changes in structural and microstructural properties during the milling and the melting and quenching processes. The diffusion of Mn and Ni to the Fe matrix, during milling, leads to a heterogeneous solid solution with FCC-rich and BCC-rich environments after 25 h of milling. The end product, obtained by milling, is a mixture of a disordered structure FCC and BCC phases having different structure and microstructure parameters. Through the X-ray diffraction analyses the formation of a single BCC phase was identified in the melted sample.

Preparation of stable Gold Nanoparticles-Modified graphite felt electrode

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The immobilization of gold nanoparticles (Au-NP) on a graphite felt was investigated in this work [1]. The graphite felt electrode was functionalized by electrochemical reduction of diazonium salts. Two methods were examined: electrostatic interactions and covalent S-Au bonds. The concentration of Au-NP solution was optimized and characterized using electronic scanning microscopy.

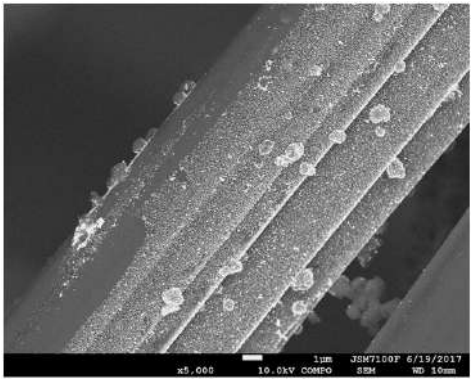

Fig. SEM images of GF-Ph-SH-AuNP

Key words: Gold Nanoparticles- graphite felt- modified electrode

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Electrostatic energy storage in antiferroelectric like perovskite

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Lead-free ferroelectric $Na_{0.5}Bi_{0.5}TiO_3$ (NBT) and NBT-doped Ln (Ln = Er and Ho) in both bulk and thin films forms we processed via solid state reaction and solution deposition respectively. SEM investigations showed dense samples with fine grained and smooth microstructure for thin films. A pure perovskite structure was observed by the X-ray diffraction for all specimens. The frequency dependences of the dielectric constant and loss tangent are investigated at room temperature and revealed a decrease in the dielectric losses with the introduction of the lanthanide element. The energy storage density (*W*) was calculated using the obtained **P**–**E** loops. The stored and delivered energies were found depending on the form and composition of the sample.

Keywords: Sodium Bismuth Titanate system; Ceramics; thin films; energy storage property.

Design and investigation of a conical solar furnace powered by optical fibers

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A novel solar furnace used in photothermal system has been designed. This study deals with the thermal study and performance assessment of the system based on the idea of transmission concentrated solar energy via optical fibres (TCSEvOF). The design can be used in the biotechnology and the biochemistry fields.
The aim of this work is the study and the modelling of a new design of photothermal device with reduced losses and high efficiency. We optimize the coupling of a paraboloidal dish, which concentrates direct solar irradiance with dual axes tracking component, and the optical fibre, which transmits concentrated solar energy; this study is aimed for the site of Tlemcen in Algeria.
We present the daily power obtained at the output of the optical fiber. The power supply is estimated to be 39 W at the end of a 4 m long of optical fiber. We show that the transported energy is diffused until the top of the cone then disperse inside them. The energy is absorbed by the inner surface of the cone, which is made by a selective surface. Temperatures of the solar furnace higher than 1000°C may be reached then maintaining very good efficiency.

Key words: solar lighting, concentrated solar energy, optical fibre, solar furnace.

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Posters Communications' Abstracts

Mechanism of Olefin Hydrogenation Catalyzed by $RuH(PMe_3)_2(CO)Cl$ Complexes : A DFT Study

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Using a DFT method, we studied the reaction mechanism of butene hydrogenation catalyzed by a $RuH(PMe_3)_2(CO)Cl$ complex by two different pathways. The first one with a monophosphine complex (the catalytic cycle begins with the dissociation of a phosphine from the metal) and the second with a biphosphine complex (the phosphine remain in all the mechanism steps) according to **fig. 1**. We have found that the activation energy of the monophosphine pathway ($\Delta G^{\#}$ = 10.3 kcal.mol^{-1}) is lower than that of the diphosphine pathway ($\Delta G^{\#}$ = 13.2 kcal.mol^{-1}). This is in agreement with experience [1].

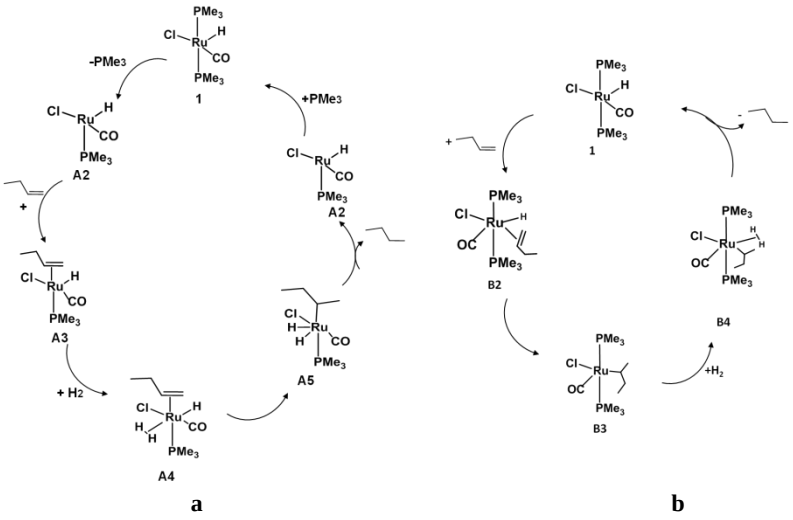

Fig.1: Proposed catalytic cycles for the hydrogenation of butene by the complex $RuH(CO)(Cl)(PMe_3)_2$ with the mono[a] and biphosphine[b]

Key words: Catalyse, olefin hydrogenation, energy profiles.

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Synthesis, structure and bonding of 4-dimethylaminopyridine heptamolybdate heptahydrate

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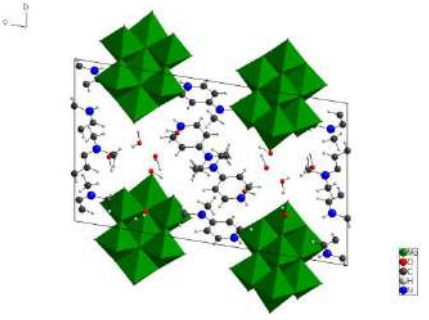
Heptametalate polyoxomolybdates and polyoxotungstates [1.4] of general formula $[M_7O_{24}]^{6-}$ (M = Mo^{VI} or W^{VI}, can both be isolated in the solid state. The heptamolybdate (paramolybdate) anions can be crystallized following acidification of aqueous molybdate solution to within the pH range of approximately 3 to 5.5. The structure of the heptamolybdate ion was solved by crystal-structure analysis, by Lindqvist,[5] who established the peculiar butterfly-shaped configuration adopted by the molecular ion.

The 4-dimethylaminopyridine heptamolybdate heptahydrate crystallizes in the triclinic system with space group P-1 and Z = 4 (R = 0.0314). The unit cell dimensions are a = 8.955(8)Å, b = 13.570(4)Å, c = 22.667(3)Å, α=80.9990°,β=89.9697°,γ=76.6751° and V =2645.93 (2) $Å^3$. The planar cations and the water molecules are positioned so as to be able to form hydrogen bonds with either molybdate oxygen atoms or water oxygen atoms. Four different types of hydrogen bonds have been found-: N-H... O, N-H... Ow, Ow-Hw... O and Ow-Hw...Ow .

Theoretical and experimental studies was reported in this work; thermal, electrochemical and spectral analysis also Hirshfeld surface study



View along axis a of the compound

Keywords: Hybrides. Heptamolybdate. Crystal structure. Hirschfield-surfaces

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A new Ru(II)-complex for the degradation of azoic textile dyes in water

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Many searches have been carried out aiming at developing novel technologies for the removal of synthetic dyes from water, in particular azoic ones which are considered more toxic than others.[1,2] Indeed, catalytic oxidation has been recognized as an effective technique for the removal of dyes from aqueous medium.[3,4]

In this work, a new helical ruthenium-complex has been prepared, fully characterized and used as an heterogeneous catalyst for the degradation of *Eriochrome Blue Black* B (one of the azoic dyes) in water in the presence of an environmental friendly oxidant. The effect of the main operating conditions in the dye degradation was analyzed. It was found that the increase in temperature leads to a higher removal of the dye, when using a fixed dose of the oxidant. The energetic parameters have been also determined, confirming that the synthetic catalyst was efficient for the degradation of anionic dyes using an ecological oxidant. The Ru-complex has the advantage of an easy separation from the reaction medium and the same performance was reached in five consecutive cycles.

Keywords: Azoic dyes; Dye degradation; Eriochrome Blue Black B; Catalysis; Helical complexes.

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New metallated complexes for the decolouration of wastewaters

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Dyes represent an important class of organic compounds that are used in textile, cosmetic, paper, drug, and food-processing industries in large quantities. They are designed to be adherent, long-lasting, and resistant to chemical processes.
It is known that wastewaters containing dyes are very difficult to treat, because the dyes are recalcitrant molecules, resistant to aerobic digestion and stable to light, heat, and oxidizing agents.[1,2] It has also been demonstrated that the oxidation processes could provide a promising treatment option for colour removal using an oxidizing agent, such as hydrogen peroxide.[3]
In this work, we report the synthesis and characterization of new palladium (II) and ruthenium (II) complexes. The catalytic activity of such metallated complexes was evaluated in the decolouration of *Calmagite* (azoic dye) solutions, in the presence of hydrogen peroxide. Experiments revealed that the degradation process is affected by many parameters such as the oxidant dose, the initial dye concentration and the temperature. The efficiency of the dye removal was evaluated through the calculation of activation energy. Such property including easy separation and recycling tests would bring a bright perspective for the application of these new compounds for the treatment of coloured waters.

Keywords: Azoic dyes; calmagite; decolouration; catalysis; helical complexes.

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Bifunctional mixed oxides catalyzed phosgene-free-halogen-free synthesis of carbamate

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Mixed oxides derived from hydrotalcites [1,2] were prepared over coprecipitation and followed by calcination at 723 K. Structural and textural features of materials were investigated by XRD, physisorption of nitrogen at 77 K, TEM and SEM-EDX. The influence of Zr loading on acid–base properties of mixed oxides was studied by TPD-CO_2 and TPD-NH_3.

Mixed oxides were evaluated in the phosgene-free-halogen-free synthesis of carbamate [3]. Noteworthy, bifunctional catalysts were active and exhibited a good selectivity towards carbamate as well as an appreciable amine conversion at high pressure.

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Synthesis, crystal structure and characterization of a new organic–inorganic hybrid material: $[C_6H_{16}N_2O]SbCl_5$

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The present paper undertakes the study of $[C_6H_{16}N_2O]SbCl_5$ which is a new hybrid compound. It is synthesized and characterized by single-crystal X-ray diffraction, thermal analysis, IR and solid state NMR spectroscopy. The centrosymmetric compound crystallizes in the monoclinic space group $P2_1/n$, with the following unit cell parameters: a = 9.8519(2), b = 8.8345(2), c = 17.3087(4) Å, ß = 102.3(1)°, V = 1457.90 (6) $Å^3$ and Z = 4. The atomic arrangement shows an alternation of organic and inorganic entities. The cohesion between these entities is performed via N–H…Cl and O–H…Cl hydrogen bonding to form a three-dimensional network. The ^{13}C CP-MAS NMR spectrum is in agreement with the X-ray structure. Infrared spectrum is recorded in the 4000–400 cm^{-1} frequency region. This study confirms the presence of the organic cation $[C_6H_{16}N_2O]^{2+}$. DFT calculations allow the attribution of the carbon peaks to the different atoms.

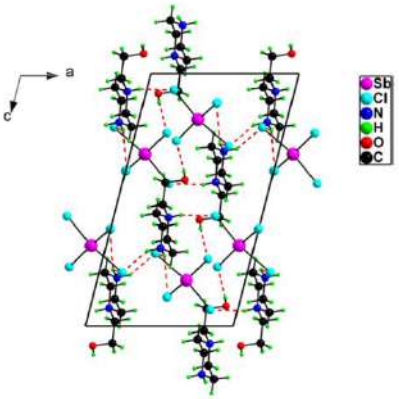

Figure: Projection of the structure of $[C_6H_{16}N_2O]SbCl_5$ along the b-axis.

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

2-Aminothiazole-functionalized triazine-modified polystyrene decorated with gold nanoparticles as composite catalyst for the reduction of 4-nitrophenol

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Herein, polystyrene (PS) bearing di(2-aminothiazole)-substituted triazine as chelation unit for both Au(III) ions and Au(0) nanoclusters was prepared through successive chemical modification of native polystyrene including nitration (treatment with HNO3/H2SO4), reductive amination (treatment with SnCl2/HCl), and successive grafting of 2,4,6-trichloro- 1,3,5-triazine and 2-aminothiazole. The derived gold nanoparticles- (AuNPs-) containing composite material was obtained through chelation of the nanogold precursor, namely hydrogen tetrachloroaurate, and subsequent *in situ* hydride-assisted reduction providing gold nanoparticles with low size (about 14 nm) and spherical shape. It is shown that the presence of 2-aminothiazole-grafted triazine units on the phenyl side groups of PS enables the stable anchoring of the AuNPs while preventing agglomeration. Chemical structure of the PS derivatives was confirmed after each synthesis step by a combination of experimental methods such as infrared and energy dispersive X-ray spectroscopies, elemental analysis, Xray diffraction and scanning electron microscopy. To highlight the catalytic potential of the so-designed nanostructured catalyst, the reduction of 4-nitrophenol was considered in the presence of a large excess of sodium borohydride as reductant specie. UV-visible spectroscopic monitoring of the progress of the reaction confirmed the pseudo-first order kinetic profile associated with a rate constant as high as 3.07 × 10-2 s-1.

Synthesis, crystal structure and spectroscopic characterizations of porphyrin-based Mg(II) complexes Potential application as antibacterial agent

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Metalloporphyrins play a key role in nature for oxygen transport in hemoglobin or photosynthesis in chlorophyll for example. Indeed, they have attracted a lot of attention over the past decades because of their remarkable abilities in number of applications such as materials [1], used as an electronic system for storing information [2], solar photovoltaic cells [3], and catalysis [4]. Several derivatives of porphyrin have been developed as photosensibilizating agents (PS) for photodynamic therapy (PDT) [5]. In addition to these important roles, the porphyrinic nucleus presents an efficient source for the synthesis of a large number of active agents in the antibacterial and antimicrobial activities [6].

In this work, we combined the porphyrin framework with imidazole in order to design and study a novel complex [Mg(TBrPP)(HIm)] (**I**). Spectroscopic and structural characterization of compound **I** will be given together with preliminary biological studies in the antibacterial domain of application.

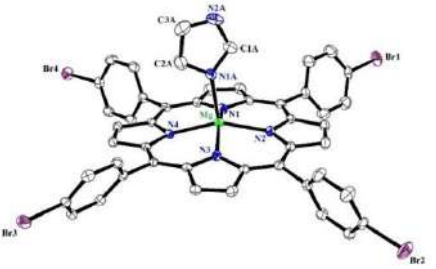

Figure 1. ORTEP diagram of [Mg^{II}(TBrPP)(HIm)].

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A Merrifield resin functionalized by 4-amino benzo-15-crown-5. Inductively coupled plasma study of the extraction of Li(I) and Cd(II) by modified polymer.

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Recently, the solid phase extraction has been shown to be an excellent separation technique due to immiscibility of resins with aqueous phase and minimum release of toxic organic solvents [1, 2]. In this context, grafted resins have been used in separation of hazardous metallic ions in industrial effluents [3, 4]. The aim of this study was to evaluate the efficiency of a new polymer obtained by functionalization of a Merrifield resin (MR reticulated by 2% divinylbenzene, 200-400 mesh, 2.1 mmol $Cl \cdot g^{-1}$) by grafting 4-amino benzo-15-crown-5 (Fig.1) groups to extract some metal cations from aqueous solutions. The structural properties of the polymer were systematically investigated by different analytical methods; namely elemental analysis (EA), differential scanning calorimetry (DSC) and infrared spectroscopy (FT-IR). The percentage of extraction was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The obtained polymer gave an extraction ratio of Cd^{2+} = 56.9 % which highlight the importance of the substitution of chlorine atoms by amino groups.

Keywords: Merrifield resin; Functionalization; 4-amino benzo-15-crown-5; Metal cations; Extraction; Inductively coupled plasma- atomic emission spectrometry.

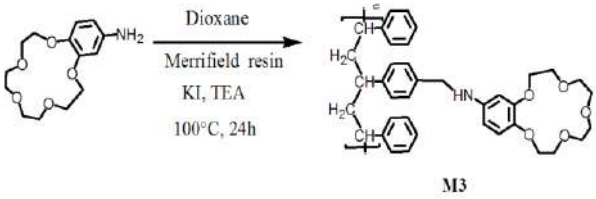

Fig. 1. Synthetic scheme of aminobenzocrown-Mer (M_3).

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Synthesis, structure and physical properties of a new inorganic complex based on Strandberg cluster

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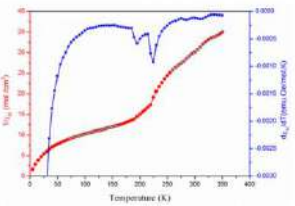
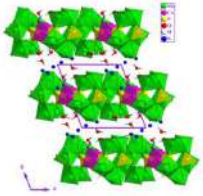
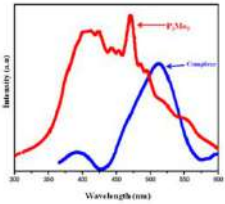
Polyoxometalates (POMs) have attracted a great deal of attention in recent years owing to their attractive electronic and molecular properties that give rise to a variety of applications in diverse fields such as catalysis, medicine, molecular magnetism or materials science [1-2]. In particular, heteropolyoxometalates (HPOMs) are of special interest for the engineering of novel framework structures since they combine high stability with extensive structural variety and application. The work described herein reports the synthesis and characterization of novel POMs based on monohydrogendiphosphopentamolybdate anion. Single-crystal X-ray diffraction analysis reveals that this novel compound is composed of monomeric anion $[Co(H_2O)_4HP_2Mo_5O_{23}]_2^{8-}$, ammonium cations and water molecules. The molecular Hirshfeld surface indicates that the crystal packing is stabilized by H-bonds interactions to generate a 3D supramolecular framework. Furthermore, the optical constants such as band gap energy, refractive index and dielectric constant of the compound were determined. Magnetic measurements show that this compound exhibits an antiferromagnetic (AFM) to paramagnetic (PM) phase transition at a temperature (T_N) lower than 2K.

Keywords: Optical constant; Magnetic properties; Structural data; Band gap energy; Fluorescence.







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Additive effect of coupling photocatalysis with non-thermal plasma for ethanol decomposition on TiO_2-nanotubes

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Titanate nanotubes were synthesized by hydrothermal treatment of TiO_2 (P25) in an alkaline aqueous solution followed by acid washing and calcination at 400°C [1]. The prepared nanotubes were characterized by XRD, N_2-physisorption and photoluminescence. The initial titanium dioxide and titanate nanotubes were both used to remove the ethanol, at low concentration, under UV light and under non-thermal plasma. The combination of those both technologies was also tested. Obtained results show that the ethanol conversion is higher by using HNTc rather than P25 whatever the technology (UV alone, NTP alone or UV/NTP). Also, the CO_2 selectivity is higher with HNTc as compared to P25 except under UV alone.
When photocatalysis and non-thermal plasma are coupled, a low concentration of ethanol (30 – 50 ppmv) can be totally eliminated from an air flow with a very low specific energy (7.5 – 14.3 J L^{-1}) and with a good CO_2 selectivity [2].
In order to determine whether or not a synergy effect occurs by coupling plasma and photocatalysis, a kinetic simulation of the two contributions of plasma and photocatalysis was proposed. This kinetic simulation of ethanol conversion has shown that the behavior observed when photocatalysis and non-thermal plasma are coupled could result from additive ethanol elimination by photocatalysis alone and plasma-catalysis alone.

Key words: Titanate nanotubes, Non-Thermal Plasma, Photocatalysis.

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Synthesis, physicochemical characterization, crystal structure and Hirshfeld surface analysis of a novel copper (II) complex $CuC_{12}H_8N_4O_6$

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A novel organic Copper (II) complex was synthesized and characterized by FT-IR, UV and single-crystal XRD. The crystal structure has been determined by the X-Ray diffraction method. The compound crystallizes in the monoclinic system, space group P21/n. The unit cell dimensions are a=8.7760(8), b=9.1000(17), c=17.1590 (17) with β= 101.760(9). The complex contains three copper (II) centers which are bridged from nitrate group to form tri-nuclear units. Crystallographically independent cations in this crystalline phase have also been investigated in terms of their corresponding Hirshfeld surface. Normal and bifurcated C-H…O hydrogen bonds between the cations and anions lead to the formation of a three-dimensional network structure. The three-dimensional Hirshfeld surfaces (3D-HS) [1] analysis shows closely similar Hirshfeld surface shapes for cations in the compound, reflecting similar intermolecular contacts and similar conformations. The two-dimensional fingerprint plots (2D-FP) [2] are quite asymmetric, due to the presence of more than one component (cation and anion).

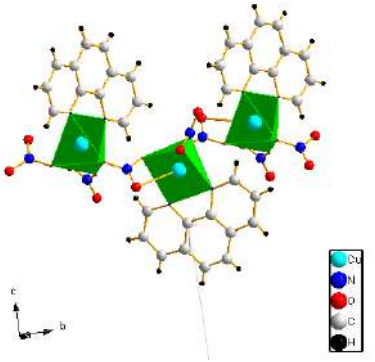

Fig. 1. View along the [100] direction of the complex $CuC_{12}H_8N_4O_6$

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Crystal Structure, Quantum Mechanical Investigation, IR and NMR Spectroscopy of a New Organic Sulfate $(C_6H_{22}N_4)_2\ (SO_4)_2\ (HSO_4)_4$

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Crystals of the new organic sulfate: $(C_6H_{22}N_4)_2\ (SO_4)_2\ (HSO_4)_4$ were prepared and grown at room temperature by slow evaporation method. The grown crystal has been subjected to X-ray diffraction, TG-DTA, IR and NMR spectroscopy. The results of these investigations are discussed. The coordination of N,N'-Bis(2-aminoethyl)ethane-1,2-diamine with sulphuric acid was confirmed by FT-IR studies in 4000-400 cm^{-1} range. The crystal data were collected by subjecting the grown samples to single crystal XRD and the study reveals that the sulfate is triclinic and it belongs to P-1 space group. The lattice parameter values are a = 9.385(2), b = 11.596(3), c = 16.229(4) Å, α = 102.06(1), β = 101.48(1), γ = 98.82(1) °, Z = 2 and V = 1657.39(7) $Å^3$. From the structural investigations, it is found that the studied compound exhibits complex three-dimensional network of H-bonds connecting all its components via several N-H…O and C-H…O interactions **(Fig. 1)**.

Fig. 2 presents the results of TGA and DTA measurements of $(C_6H_{22}N_4)_2\ (SO_4)_2\ (HSO_4)_4$ in the temperature range from 293 to 900 K

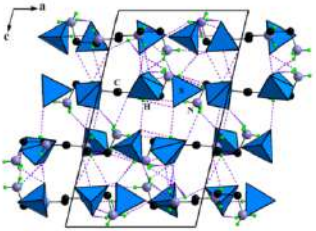

Fig. 1: The structure of $[C_{12}H_{20}N_2][HSO_4]_2$ projected along the b axis

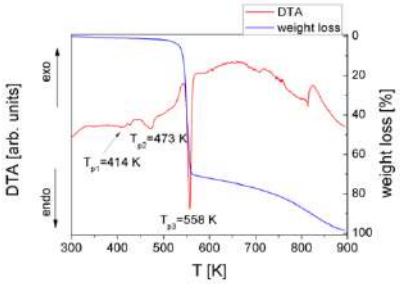

Fig. 2: Thermal analysis of $(C_6H_{22}N_4)_2\ (SO_4)_2\ (HSO_4)_4$

Synthesis and Characterization of a New Organic Sulfate: 1-(2,3-dimethylphenyl)piperazine-1,4-diium bis(hydrogensulfate)

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Chemical preparation, crystal structure, calorimetric and spectroscopic investigations (IR and RMN) are given for a new organic-cation hydrogen sulphate $[C_{12}H_{20}N_2][HSO_4]_2$. This compound is monoclinic $P2_1/c$ with the following unit-cell parameters: a = 14.310(3), b = 7.4982(1), c = 15.555(3) Å, β = 104.521 (2) °, Z = 4 and V = 1615.77(6) $Å^3$. The HSO_4^- anions are interconnected via O-H…O hydrogen bonds to form corrugated chains extending along the *b*-axis direction at x = 1/3 and x = 2/3 **(Fig. 1).** The 1-(2,3-dimethylphenyl)piperazine-1,4-diium cations connect these chains via N-H…O and C-H…O hydrogen bonds to form layers parallel to the (a, c) plane **(Fig. 2).** Solid-state ^{13}C and ^{15}N CP-MAS NMR spectroscopy is in full agreement with the X-ray structure. The vibrational absorption bands were identified by infrared spectroscopy. DFT calculations allowed the attribution of the NMR peaks. The electronic properties such as HOMO and LUMO energies were carried out.

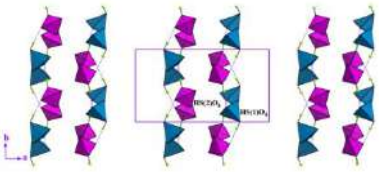

Fig. 1: Corrugated $[HSO_4]_n$ chains in $[C_{12}H_{20}N_2][HSO_4]_2$

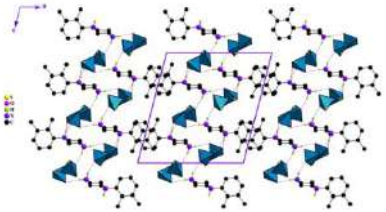

Fig. 2: The structure of $[C_{12}H_{20}N_2][HSO_4]_2$ projected along the b axis

HYDROTHERMAL SYNTHESIS, OPTICAL AND ADSORPTION PROPRIETIES OF NI DOPED CUO NANOPARTICULES

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Nanomaterials have numerous potential applications in many areas such as electronics, optoelectronics, catalysis and composite materials. Particularly, the one-dimensional (1D) and bi-dimensional (2D) nanostructures have been regarded as the most promising materials for electronic and optoelectronic devices and like a system for the selective detection of metal ions. The synthesis and characterization of transition-metal oxides with a size and shape controlled that present uniform dimension have attracted considerable interest due to their unique physicochemical properties. Among these, we can cite the copper oxide (CuO) that presents high stability and non-toxicity. It is a p-type semiconductor with a narrow band gap 1.2 eV. The physical and chemical properties of CuO nanostructures can be affected by doping in terms of the resulting optical, structural and microstructural properties. CuO has been studied intensely because of its numerous applications in photocatalysis, gas sensors, solar energy transformation, photovoltaic devices and magnetic storage media.

This work describes the synthesis of undoped and Ni-doped CuO nanostructures by hydrothermal route at different doping concentrations (undoped, 5, 10 and 20 %). The physico-chemical proprieties were investigated by X-ray diffraction [XRD], Energy dispersive X-ray [EDX], Fourier Transform Infrared Spectroscopy [IR], Scanning Electron Microscopy [SEM], Brunauer Emmett-Teller N_2 adsorption-desorption analysis [BET] and UV-Vis spectroscopy [UV-Vis]. The ability and analytical efficiency of the CuO nanostructure adsorbents on the adsorption capacity of silver Ag (I) was studied.

The adsorption capacity of all adsorbents was studied. The impact of the contact time and adsorbent concentrations were analyzed and the obtained results were evaluated and fitted using adsorbent equilibrium isotherms and kinetic models.

Supercritical fluid CO_2 extraction of essential oil from seeds and cones of *Cupressus sempervirens*: Comparison by hydrodistillation method to determinate global yields and chemical composition

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Essential oil of Tunisian Cupressus sempervirens was obtained by two different methods. Supercritical fluid extraction (SFE) with CO_2 and conventional hydrodistillation (HD) are achieved in order to carry out a comparative study. Both of them were performed from various parts of the plants (leaves, cones and seed). The analysis by chromatography/mass spectrometric detection GC-SM shows that the chemical composition of the Cupressus sempervirens essential oil extracted by SFE and HD from cones is mainly composed of α- pinene (the major compound). In the other side, the extracted oil from seeds by SFE reveals that the α-pinene wasn't the major compound. The comparison between HD and SEF extractions presented many distinctions about colour, odour, yield and chemical composition.

More insight on the solid-state ion exchange of solid $MoCl_5$ into ZSM-5 zeolite

Faouzi Ayari[a,b], Emna Mannei[a], Esther Asedegbega–Nieto[c], Mourad Mhamdi[a,d], Antonio Ricardo Guerrero–Ruiz[c], Gérard Delahay[e], Abdelhamid Ghorbel[a]

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Molybdenum exchanged zeolites have attracted widespread attention due to their unique catalytic performance in hydrotreatment and hydrogenation of oil fractions [1], partial oxidation of methane into C_1 oxygenates [2], methane dehydrogenation and aromatization [3] and light hydrocarbons ammoxidation into acetonitrile [4]. The methods used for loading molybdenum onto zeolites include essentially impregnation from inorganic salt solutions (*e.g.* ammonium heptamolybdate tetrahydrate $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$), grafting or vapour phase adsorption of organometallic salts (*e.g.* molybdenum carbonyl $Mo(C{\equiv}O)_6$, molybdenum (*VI*) acetylacetonate $MoO_2(C_5H_7O_2)_2$ and molybdenum carbide Mo_2C) [5]. However, solid–state ion exchange is still promising [6] since this easy method consists on grinding both zeolite and Mo precursor and then spreading the salt inside the zeolite channels by thermal activation.

In this work, we focus on the elucidation of the solid–state ion exchange of molybdenum (V) chloride into NH_4^+–ZSM-5 (Si/Al = 26) zeolite under helium stream using thermal analysis coupled to mass spectrometry. The results show that the highly hygroscopic $Mo^{V}Cl_{5(s)}$ retained water molecules before being oxidized into $Mo^{VI}OCl_{4\,(s)}$ at low temperatures. Above 77 °C, solid $MoOCl_4$ evaporates and transforms (simultaneously) into $Mo^{V}OCl_{3(s)}$ and $Mo^{VI}O_{3(s)}$ with the exit of hydrogen, hydrogen chloride and chlorine. At 313 °C, solid $MoOCl_3$ evaporates and transforms into gaseous $MoOCl_4$. Besides the exit of chlorine, both $MoOCl_3$ and $MoOCl_4$ compounds were lost by evaporation above 367 °C, evidencing the vanishing of $MoCl_5$, while MoO_3 sublimates at 625 °C.

In the presence of NH_4^+–ZSM-5 zeolite, $MoCl_5$ is oxidized into $MoOCl_4$, and the formation of chlorine is inhibited. The formation of $MoOCl_3$ is, therefore, excluded and $MoOCl_4$ transforms into the volatile $Mo^{VI}O_2(OH)_2$ at 342 °C. The non–isothermal solid–state exchange of $MoO_2(OH)_{2\,(g)}$ into H^+–ZSM-5 takes place between 342 and 523 °C, following the formation of gaseous hydrogen chloride and the zeolite reversible dealumination. However, the isothermal–exchange of $MoO_2(OH)_{2\,(g)}$ is recommended between 342 °C and 523 °C, while the isothermal–exchange of volatile MoO_3 oxide is recommended at 650 °C, but neither the zeolite dealumination nor dehydroxylation will be avoided.

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Investigation of the solid–state ion exchange of Molybdenum (VI) acetylacetonate into ZSM-5 zeolite

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Zeolites exchanged with *d*-block cations, in particular Mo(VI), were used in several catalytic applications such as ethylene carbonylation [1], conversion of methanol into hydrocarbons [2] and C_2H_x (x = 4 and 6) ammoxidation [3]. In aqueous medium, Mo (VI) forms the stable tetraoxidomolybdate (2–) complexe, $[MoO_4]^{2-}$, which is easily protonated and polymerized, giving rise to very complex systems [4]. However, in the case of the solid–state ion exchange, a dry mixture of the zeolite and Mo salt is manually mixed and then treated at the desired temperature.

To our knowledge, the thermal decomposition of Mo(VI) acetylacetonate, $MoO_2(C_5H_7O_2)_2$, under the solid–state reaction condition is not yet investigated despite the effectiveness of this salt in the synthesis of olefins oxidation catalysts [3,5].

In this work we investigate the solid–state ion exchange reaction of Mo acetylacetonate salt into NH_4^+–ZSM-5 (Si/Al =26) zeolite under helium stream (30 mL min^{-1}) between room temperature and 700 °C (2 °C min^{-1}) by meaning of thermogravimetric analysis coupled to mass spectrometry. The viscous $MoO_2(C_5H_7O_2)_2$ salt evolves two *acetylacetonato* ligands between room temperature and 325 °C, which decompose into methane and acetic acid. Molybdenum dioxide $Mo^{IV}O_{2(s)}$ is the final product of decomposition as confirmed by the thermal behaviour of pure $Mo^{VI}O_{3(s)}$ salt and thermodynamic calculations.

In the presence of NH_4^+–ZSM-5 zeolite (6 *wt*. % of Mo), the departure of the two ligands was accelerated and the salt transformed into $MoO_{2(s)}$ at 285 °C. The former oxide transforms into gaseous $Mo^{VI}O_2(OH)_2$ (280–320 °C) in the presence of molecular oxygen and water. The non–isothermal exchange occurs between protonated H^+–ZSM-5 zeolite and volatile $MoO_2(OH)_2$ specie between 320 and 570 °C. At higher temperatures (T > 570 °C), residual methane and carbon were oxidized into CO_2.

Taking into account the partial zeolite dehydroxylation at 520 °C, we believe that the exchange Mo(VI) acetylacetonate into zeolite would be more efficient between 320 and 520 °C. However, residual organic compounds would be eliminated by performing an isothermal exchange under helium stream at a selected temperature T (320 << T < 520 °C) for several hours.

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Synthesis, Structural Study and Physico-Chemical Characterization of a New Perchlorate $[C_6H_7ClN]ClO_4$

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Hybrid materials are of great interest in solid state chemistry due to their enormous variety of intriguing structural topologies and their fascinating properties as well as great potential applications in many fields **[1, 2]**. In the present contribution, we report on the synthesis and characterization of a new organic perchlorate $[C_6H_7ClN]ClO_4$. Molecular structure is determined by single crystal X-ray diffraction analysis. The asymmetric part of the unit cell contains a 3-chloroanilinium cation and a perchlorate anion **(Fig. 1)**. The title compound form inorganic layers made up of ClO_4^- anions and NH_3^+ ammonium groups, developed parallel to the (b, a+c) plane. The bodies of the organic entities are located between these layers and connect each other via C-H…O hydrogen bonds to perform an infinite three-dimensional network **(Fig. 2)**. This compound is also characterized by IR and solid state NMR spectroscopy. Electronic properties such as HOMO and LUMO energies were studied by quantum mechanical evaluation by using the B3LYP/6-31+G* method.

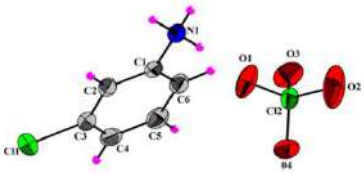

Fig. 1: Asymmetric unit of $[C_6H_7ClN]ClO_4$

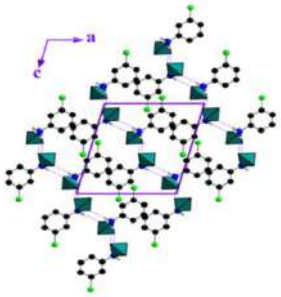

Fig. 2: The crystal packing of $[C_6H_7ClN]ClO_4$ viewed along the *b* axis.

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Synthesis, Structural Study and Physico-Chemical Characterization of a New Perchlorate $(C_6H_{18}N_3)\cdot(ClO_4)_3.H_2O$

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Recently, much attention has been devoted to simple molecular-ionic crystals containing organic cations and acid radicals due to the tunability of their special structural features and their interesting physical properties. In particular, organic perchlorates resulting from the interaction between perchloric acid and organic molecules, such as amines, amino alcohols and amino acids represent a class of high-performance materials due to their interesting physicochemical properties**.** The structure is determined by X-ray diffraction method. A perspective view of the asymmetric unit of $(C_6H_{18}N_3)\cdot(ClO_4)_3.H_2O$, built of one 1-(2-ammoniumethyl)piperazin-1-ium cation, three perchlorate anions (ClO_4^-) and one water molecule, is depicted in **Fig. 1**. The crystal structure of 1-(2-ammoniumethyl)piperazin-1-ium tri(perchlorate) hydrate exhibits complex three-dimensional network of H-bonds connecting all their components. In the atomic arrangement the ClO_4^- anions form corrugated chains. The lattice water serves as a link between pairs of cations and pairs of anions via several O–H…O and N–H…O interactions **(Fig. 2)**. This compound is also investigated by FT-IR and solid-state ^{13}C and ^{15}N CP-MAS NMR spectroscopy.

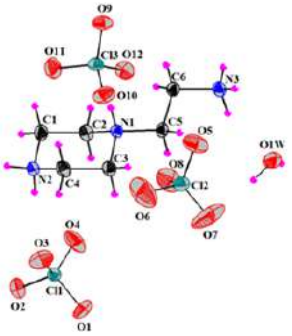

Fig. 1: Asymmetric unit of $(C_6H_{18}N_3)\cdot(ClO_4)_3.H_2O$

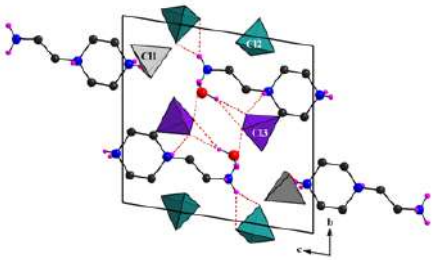

Fig. 2: The crystal packing of $(C_6H_{18}N_3)\cdot(ClO_4)_3.H_2O$ viewed along the *a* axis.

Synthesis, structural study and validation (CHARDI and BVS) of a new compound $NaCu_2As_3O_{10}$.

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The title compound $NaCu_2As_3O_{10}$ has been synthesized by solid-state reaction. Crystal structure was studied by X-ray diffraction. The obtained structural model is supported by charge- distribution (CHARDI) analysis and bond-valence-sum (BVS) validations tools. It crystallizes in the triclinic space group P-1 with a= 7.367 (1) Å, b= 7.645 (1) Å, c= 8.378 (1) Å, V = 398 (2) $Å^3$, Z= 2, R (F^2) = 0.025, Rw (F^2) = 0.066.The structure can be described as a three-dimensional framework resulted from corner-connection between copper metallic chains running along [-110] and As_3O_{10} groups; the negative charge is balanced by Na^+ ions which house the two tunnels parallel to a and b axes.

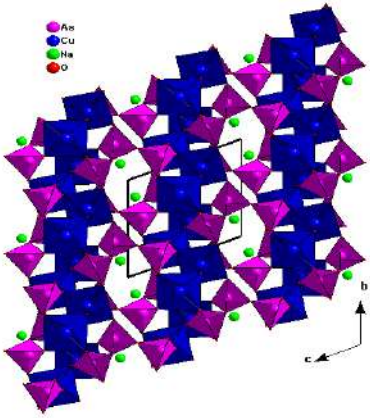

Fig. Projection of the structure along direction near [100].

Key words: Solid state reaction, framework, crystal structure, BVS, CHARDI.

SYNTHESIS, CRYSTAL STRUCTURE, VIBRATIONAL SPECTROSCOPY AND THEORETICAL STUDIES OF A SILVER COMPLEXE CONTAINING HETEROCYCLIC NITROGEN DONOR LIGANDS

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Crystal engineering of metal complexes, especially coordination polymers or metal-organic frameworks (MOFs) has become the focus of intense interest over the past decade not only because of their diverse and beautiful structures, but also for their wide potential applications as luminescence electrochemical, nanoparticle precursors, magnetic and gas storage properties [1]. Herein, we report the synthesis, structure and characterization of a silver complexe. The title compound crystallizes in the monoclinic space group $P2_1/c$ with the following parameters: a= 8.6621(11), b= 8.6621(15), c= 13.0931(2)Å, β= 128.7(15)° and V= 766.7 (2) $Å^3$. Its crystal structure consists of silver atom bridged to two N atoms from two adjacent piperazine ligands. The N–Ag–N skeleton gives rise to an infinite 1-D polymeric zigzag chain of formula $\{[Ag(C_4N_2H_{10})]^+\}_\infty$. Theoretical calculations were performed to study the molecular structure and the vibrational spectrum. All calculations were carried out at the DFT/B3LYP/LanL2DZ level of theory using Gaussian 09 program [2]. The calculated vibrational wavenumbers were compared to the experimental counterparts.

Keywords: Metal-organic framework, Coordination Polymer, Crystal Structure, DFT Calculation.

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Synthesis, crystal structure, physico-chemical characterization of a new hybrid material (2-hydroxyethyl)piperazine-1,4-diium hexachlorostannate(IV) monohydrate

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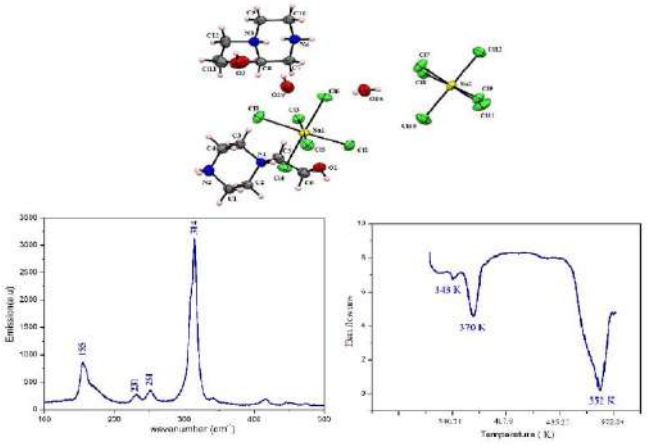
The new organic-inorganic compound $[C_6H_{16}N_2O](SnCl_6).H_2O$ was synthesized and single crystals were grown from an aqueous solution through these slow evaporation technique. This compound crystallizes in the $P2_1/c$ monoclinic space group with the following unit cell parameters: a=10.3644(3) Å, b=11.7188(3) Å, c=27.4621(8) Å, Z=4 and V=3285.43(16) $Å^3$. The atomic arrangement can be described by organic-inorganic layers parallel to the (a, c) plane. These layers are connected via hydrogen bonds (O-H··O and C-H··Cl) to build a three dimensional arrangement. The ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray structure. The vibrational absorption bands were identified by infrared spectroscopy and Raman scattering. The DSC profile shows endothermic peaks centered at approximately 343, 370 and 552 K.

Keywords: Organic-inorganic hybrid; X-ray diffraction; IR and Raman spectroscopy; NMR spectra; differential scanning calorimetric study.

Graphical abstract:



Crystal structure, Hirshfeld surfaces computational study and physicochemical characterization of the hybrid material $(C_7H_{10}N)_2[SnCl_6]\cdot H_2O$

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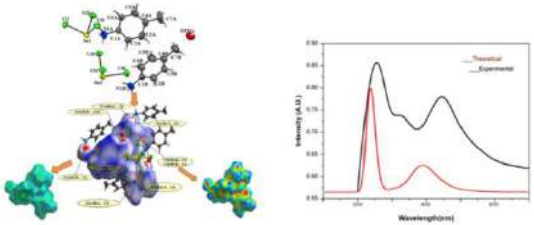
(UR14ES10), Institut préparatoire aux études d'ingénieurs d'El Manar, B.P.244 El Manar II, 2092, Tunisia

Hybrid compounds based aniline and substituted anilines are widely used as starting materials in a vast amount of pharmaceutical, electro-optical and many other industrial processes [1-2]. So, extensive experimental and theoretical investigations have focused on elucidating physicochemical properties of hybrid compounds based on aniline and its methyl derivatives and on tin metal. As a part of our continued involvement of organic tin complexes, we report here the synthesis, structure, spectroscopic measurement, optical study and thermal analysis of a new compound $(C_7H_{10}N)_2[SnCl_6]\cdot H_2O$ organic halogenostannate(IV) material. The title compound crystallizes in the monoclinic space group $P2_1/c$ with a = 13.093(1) Å, b = 7.093(6)Å, c = 24.152(2)Å, β = 98.536(4)° and V = 2218.4(4) $Å^3$. Their crystal structure exhibits alternating inorganic layers parallel to the (ab) plane at z = n/2. The different entities, $[SnCl6]^{2-}$, organic cations and water molecules, are connected via hydrogen bonds to form a three-dimensional network. The powder XRD data confirms the phase purity of the crystalline sample. The intermolecular interactions were investigated by Hirshfeld surfaces. The vibrational absorption bands were identified by IR spectroscopy and have been discussed. The optical properties of the crystal were studied by UV–visible absorption studies. The compound was also characterized by DTA to determine its thermal behavior with respect to the temperature.

Keywords: Crystal structure; Powder X-ray diffraction; Hirshfeld surface analysis; IR spectroscopy optical study; differential thermal analysis; X-ray photoelectron spectroscopy analysis.

Graphical abstract:



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A NANOCOMPOSITE BASED ON POLYPYRROLE/ $Ni_{0.3}Co_{2.7}O_4$ FOR REFRACTORY ORGANIC POLLUTANTS DEGRADATION

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The hybrid electrode polypyrrole (Ppy)/ $Ni_{0.3}Co_{2.7}O_4$ electrocatalytic activity towards the oxygen reduction reaction (ORR) was checked and its ability to degrade refractory organic pollutants was examined. The $Ni_{0.3}Co_{2.7}O_4$ oxide nanoparticles (NPs) were previously prepared by sol-gel route and characterized. The composites Ppy / $Ni_{0.3}Co_{2.7}O_4$ with different oxide NPs concentrations were then fabricated by pyrrole (Py) electropolymerization in the oxide presence. The ORR electo-catalysis and kinetic parameters were determined by LSV method and Koutchy Levich (K-L) equation. The results show that (Ppy)/ $Ni_{0.3}Co_{2.7}O_4$ electrode exhibits a very good electrocatalytic activity towards the O_2 reduction and involves two electrons at low overpotential providing peroxide ions, with a cathodic transfer coefficients (β) of 0.33 and an exchange current density (j_0) of about 8×10^{-3} mA/cm^2. Afterwards, the elaborated material performance for organics electochemical treatment was tested using ethylene glycol (EG) solution with an initial concentration of 0.2 mol/L, taken as a no biodegradable molecules model. The oxide concentration effect against the degradation rate was studied. UV and Fourier transmittance infra red (FTIR) spectrometer were used to determinate the EG degradation kinetics and products, at different electrolysis time intervals. The optical spectra at λ_{max} = 227 nm reveal that the EG concentration decreases by the time, confirming the (Ppy)/ $Ni_{0.3}Co_{2.7}O_4$ electrode aptitude for EG degradation. By increasing the oxide concentration from 2.5 g/l to 7.5 g/l, the degradation rate increases significantly from 60% to reach 86.5% and the reaction mechanism is modeled by zero order kinetics. From the FTIR spectra, oxalic and formic acid are the EG degradation principle products. Therefore, the elaborated nano-material electrocatalytical properties make it promising for many environmental applications.

Keywords: indirect oxidation, O_2 reduction, hybrid electrode, oxide nanoparticles, environmental applications.

STRUCTURAL CHARACTERIZATION, VIBRATIONAL STUDIES, THERMAL ANALYSIS AND OPTICAL PROPERTIES OF A NEW LUMINESCENT ORGANIC-INORGANIC MATERIAL $[C_{12}H_{14}N_2O_2S]_2ClBiCl_6.5H_2O$

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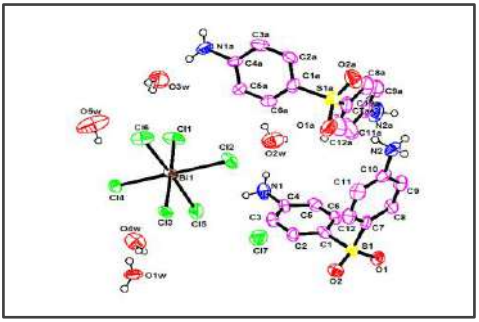
The $[C_{12}H_{14}N_2O_2S]_2ClBiCl_6.5H_2O$ is a new compound belonging to the large hybrid organic-inorganic compounds family based on bismuth, which has been synthesized by slow evaporation method at room temperature and characterized by single crystal X-ray diffraction. It crystallizes in the orthorhombic system with the $P2_12_12_1$ space group. The cell dimensions are: a = 12.3174 (7) Å, b = 16.1741 (9) Å, c = 19.466 (12) Å, V = 3878.1 (4) $Å^3$ and Z = 4.The crystal structure consists of discrete ionic layers of $[C_{12}H_{14}N_2O_2S]^{2+}$ cations, $[BiCl_6]^{3-}$ anions, Cl^- anions and five water molecules linked via N-H…Cl and O-H…Cl hydrogen bonds. The octahedral $[BiCl_6]^{3-}$ are linked together through water molecules via O-H ... Cl hydrogen bonds giving rise to zigzag chains along the a-axis.. The cations are interconnected via the N-H ... O hydrogen bonds leading to chains along the c axis. The structure of the prepared compound has been investigated using the IR and Raman spectroscopic techniques. The detailed interpretation of the vibrational modes was carried out. The thermal analysis for the title compound shows two weight losses. The optical absorption measurements on thin films of $[C_{12}H_{14}N_2O_2S]_2ClBiCl_6.5H_2O$ revealed one absorption band at 2.7 eV. Photoluminescence measurement shows a strong emission line attributed to the excitonic transition in the $[BiCl_6]^{3-}$ anions.



Asymmetric unit of $[C_{12}H_{14}N_2O_2S]_2ClBiCl_6.5H_2O$

Keywords: Hybrid compound, Crystal structure, Vibrational studies, thermal analysis, Absorption, Photoluminescence

SYNTHESIS, STRUCTURE AND NON-LINEAR OPTICAL PROPERTIES OF TETRAHALOGENOCUPRATE (II)-COMPLEX [(R)-$C_7H_{16}N_2$][$CUBR_4$]

Ines Ben Hadj Sadok [a], Fadhel Hajlaoui [a] and Nabil Zouari[a]

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Exploring new noncentrosymmetric (NCS) materials is certainly of topical interest in inorganic solid-state materials chemistry field, because these materials can exhibit extremely important physical properties such as ferroelectricity, piezoelectricity, photochromic, pyroelectricity, and second-harmonic generation (SHG), i.e., nonlinear optical (NLO) properties. In this context, the synthesis of noncentrosymmetric tetrabromocuprate (II)-complex has been achieved through the use of homochiral organic amine. [(R)-$C_7H_{16}N_2$][$CuBr_4$] has been synthesized by the slow evaporation method and crystallizes in the noncentrosymetric space group $P2_12_12_1$. The structural arrangement can be described as an alternation of the cationic and anionic layers in the plane (b, c) which are connected by a 3-D hydrogen-bond network. We report also the effects from inclusion of homochiral organic amines into systems containing second order Jahn-Teller ions and comment upon this technique as an approach toward the synthesis of noncentrosymmetric materials. The structure and SHG activities of [(R)-$C_7H_{16}N_2$][$CuBr_4$] are discussed.

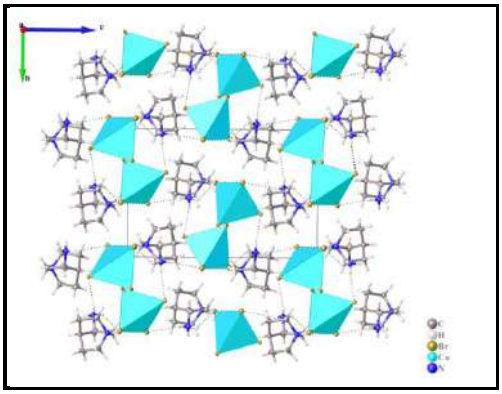

Figure: Projection of the crystal structure of [(R)-$C_7H_{16}N_2$][$CuBr_4$] along the a-axis.

Keywords: Chemical Preparation; X-ray diffraction, Noncentrosymmetric, homochiral molecule and SHG activities

Crystal structure and spectroscopic studies on a new organic-inorganic compound

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The crystal structure of a new organic nitrate (imidazoliuim nitrate) was determined from single crystals obtained by slow evaporation methods ($P4_12_12$ space group; a =7, 2827 (4) Å, b=7, 2827 (4) Å, c=22,0090 (1) Å; Z= 8). A projection of the structure along [110] (Fig.1) direction shows that the crystal structure is built up from a hydrogen bonding network linking the isolate nitrate groupments to the imidazoluim cations. The FT-IR spectrum of this organic nitrate has been recorded in the region between 4000 and 400 cm-1. The observed frequencies are assigned on the basis of other similar compound. Furthermore, the UV-vis and Fluorescence spectra were recorded in the range between 200 – 800 nm.

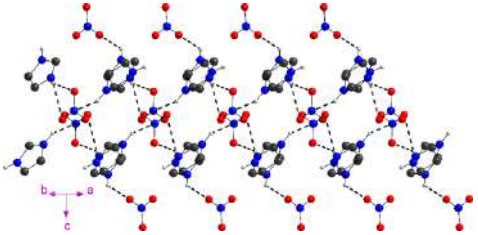

Fig.1: Crystal packing of the Imidazoliuim nitrate along [110] direction

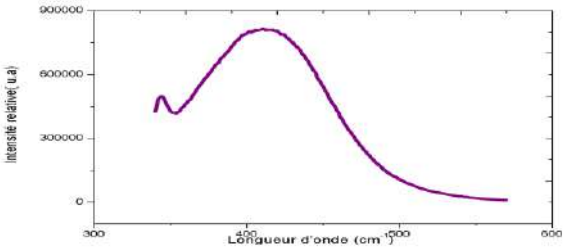

Fig.2: Spectre d'emission d'Imidazoluim nitrate.

Key words: organic nitrate, X-ray single-crystal determination, Hydrogen bond, Vibrational analysis, UV-Vis.

Crystal structure, DFT study and Hirshfeld surface analysis of a new organic perchlorate

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A new *p*-toluidinium perchlorate, $(C_7H_{10}N)[ClO_4]$ **(I)**, has been synthesized by slow evaporation at room temperature and characterized by single crystal X-ray diffraction. Compound **(I)** crystallizes in the monoclinic system with the space group $P2_1/c$, Z=4, and unit cell parameters a=9.513(1) Å, b=7.196(5) Å, c=14.297(4) Å, β=90.41(2)°. In the crystal structure, all the amine group H atoms are involved in N-H…O hydrogen bonds with O atoms of ClO_4^- anion. These hydrogen bonds link the anionic and the cationic units in two-dimensional network parallel to the *bc* plane. The density functional theory (DFT) optimized structure at the B3LYP/6-311++G(d,p) level is compared with the experimentally determined molecular structure. Comprehensive experimental and theoretical structural studies on the studied compound are carried out by IR and UV-visible spectroscopies. The three-dimensional Hirshfeld surface (3D-HS) **[1]** and the two-dimensional fingerprint plots (2D-FP) **[2]** reveal that the structure is dominated by H…O/O…H (54.2 %) and H…H (26.9 %) contacts **(fig.)**.

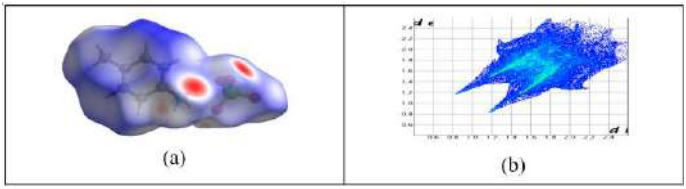

Fig. View of the 3D-HS mapped with d_{norm} (a) and the 2D-FP of (I) showing all contacts (b).

Key words: Hirshfeld surface, *p*-toluidinium perchlorate, crystal structure, fingerprint plots.

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Fabrication of nanoporous copper by dealloying of amorphous Cu–Mg–Ca alloys

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Before uniform nanoporous structure can be achieved through chemical dealloying of an amorphous Cu_{75}-x$Mg_{25}Ca_x$ (x = 30,35,40,45 at. %) ribbon alloy in a deaerated 0.04 M H_2SO_4 aqueous solution under free corrosion conditions, microstructure evolution of the alloy during dealloying process. The microstructure of as-dealloyed samples was characterized using X-ray diffraction and scanning electron microscopy. The experimental results show that the alloy presents remarkably different microstructure evolution characteristics during dealloying process in the acidic.
Our findings are not only relevant to understanding the dealloying mechanism of metallic glasses, but also provide promising materials for multiple applications.

Keywords: Amorphous materials; Microstructure; Corrosion; Dealloying; Nanoporous copper.

Vibrational spectral studies, non-linear optical properties and HOMO-LUMO analysis of tetrakis(2,6-dimethylpiperazinium) tetrakis(sulfate) dihydrate: A Density Functional Theory approach

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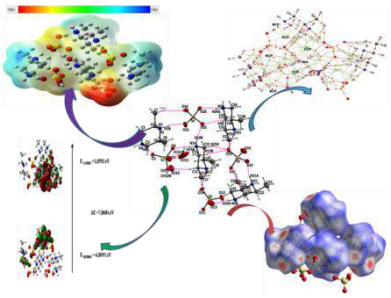
In this work, we report a combined experimental and theoretical study of a new compound tetrakis(2,6-dimethylpiperazinium) tetrakis(sulfate) dehydrate using DFT/B3LYP method with 6-311++G(d,p) as basis set. Single crystals were grown from aqueous solution by slow evaporation at room temperature. The compound crystallizes in the non-centro symmetric space group Cc of the monoclinic system. The FT-infrared spectrum of the compound was recorded in the region 4000– 400 cm^{-1}. The theoretical wavenumbers were scaled and compared with experimental FT-IR. Complete vibrational assignments and analysis of the fundamental modes of structure were performed on the basis of the potential energy distribution (PED) calculations. The lowering in the HOMO and LUMO energy gap explains the eventual charge transfer interactions that take place within the molecules. The Hirshfeld surface analysis and the two-dimensional fingerprint maps indicate that the packing is dominated by H…O/O…H and H…H contacts.

Topological parameters such an electron density and its Laplacian at bond critical points (BCP) of O-H and O…H contact bonds were analyzed in details with the help of the atoms in molecules (AIM) approach in order to study the intermolecular hydrogen bonding.

In addition, a molecular electrostatic potential map (MEP) of the title compound has been analyzed for predicting the reactive sites. The nonlinear optical (NLO) properties were reported and compared with that of the urea. The thermodynamic properties namely heat capacity, entropy, and enthalpy have been calculated for the molecule at different temperatures.



Key words: DFT ; FT-IR ; HOMO-LUMO ; Hirshfeld surface ; AIM analysis ; MEP ; NLO; thermodynamic properties

Synthesis, structural, spectral (FTIR, UV, NMR), HOMO-LUMO and Hirshfeld surface analysis of $2,6\text{-}(CH_3)_2C_4H_{10}N_2(C_2O_4)(H_2C_2O_4)_{0,5}$ by density functional theory

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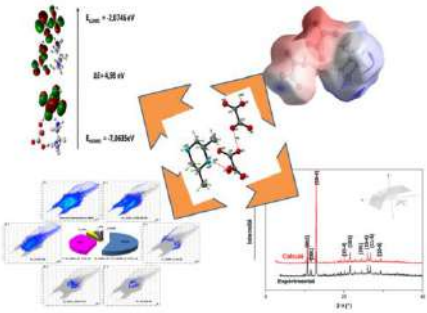
The crystal structure of a new organic-inorganic compound of stoichiometry $2,6\text{-}(CH_3)_2C_4H_{10}N_2(C_2O_4)(H_2C_2O_4)_{0,5}$ was determined. The new compound has been synthesized and single crystals were grown from aqueous solution by slow evaporation and were characterized by X-ray powder diffraction study to confirm the crystalline nature of synthesized compound. Single crystal X-ray diffraction study on grown crystal reveals that they belong to monoclinic crystal system with space group $P2_1/c$. The unit cell dimensions are: a = 7.9598(16) Å, b = 8.4457(15)Å, c = 18.080(3) Å, β = 106,349(9)° and Z = 4. The optimized molecular structure, vibrational spectra and HOMO-LUMO were calculated by the density functional theory (DFT) method using the B3LYP function with the 6-31G(d,p) basis set. Simulation of infrared spectra led to excellent overall agreement with the observed spectral patterns. Three-dimensional Hirshfeld surface analysis and two-dimensional fingerprint maps reveal that the structure is dominated by H…O/O…H and H…H contacts. The ^{13}C CP-MASNMR spectrum is in agreement with the X-ray structure. UV–Vis spectrum indicates that the crystal is transparent in UV and visible region with a lower cutoff wavelength of 279 nm. In addition, a molecular electrostatic potential map (MEP) of the title compound has been analyzed for predicting the reactive sites. Mulliken charges were also calculated and interpreted.



Key words: DFT **;** X-ray powder ; MEP- Mulliken charges ; UV–Vis spectrum; Hirshfeld surface analysis ; HOMO-LUMO ; ^{13}C CP-MAS NMR; Vibrational spectra

Synthesis, crystal structure, physico-chemical characterization and Hirshfeld surface analysis of bis(2,6-diaminopyridinium)tetrachloridocobaltate (II) monohydrate

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The titleorganic–inorganic hybrid compound $(C_5H_8N_3)_2[CoCl_4]\cdot H_2O$ **(1)** was synthesized by slow evaporation at room temperature and characterized through IR spectroscopy, UV-vis-NIR and single-crystal X-Ray diffraction. The compound crystallizes in the monoclinic system with *P*-1 space group with the following parameters: *a*=7.000(2)Å, *b*=8.842(2)Å, *c*=16.283(2)Å, *α*=82.65(2)°, *β*=78.56(2)°,γ=68.49(2)°. The asymmetric unit consists of one tetrahedral geometry $[CoCl_4]^{2-}$, two protonated organic cations $(C_5H_8N_3)^+$ and one uncoordinated water molecule of crystallization **(Fig)**. The crystal structure of (1) can be described as a succession of inorganic layers, built from tetrachloridocobaltate anions $[CoCl_4]^{2-}$ and free water molecule, linked together by O—H…Cl hydrogen bonds, and organic columns parallel to the (010) plane. Crystal cohesion is achieved through, N—H…Cl, O—H…Cl, N—H…O, C—H…Cl hydrogen bonds building up a three-dimensional network. The three-dimensional Hirshfeld surface (3D-HS) analysis and the two-dimensional fingerprint plots (2D-FP) reveal that the structure is dominated by H…Cl (47.9 %) and H…H (27,0 %) contacts.

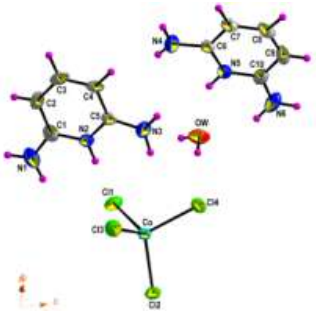

Fig. The asymmetric unit of the crystal structure of (1) showing the atom numbering scheme.

Key words: cobalt complex, crystal structure, Hirshfeld surface analysis.

Synthesis, characterization of britholite structure based $Ca_7La_3(PO_4)_3(SiO_4)_3F_2$ and its catalytic activity for one-pot Biginelli condensation

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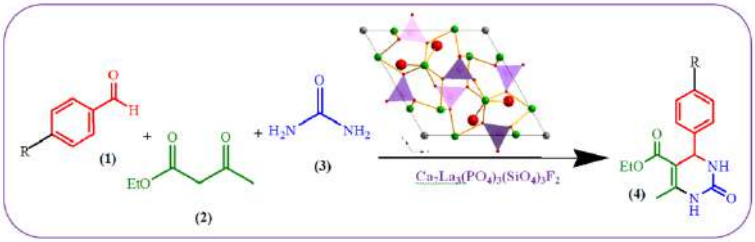
Several studies demonstrated the ability of britholites to retain radionuclides such as the caesium and actinides [1,2]. Therefore, a compound with formula $Ca_7La_3(PO_4)_3(SiO_4)_3F_2$, has been prepared by solid state reaction. The resulting material have been characterised by X-ray powder diffraction, chemical analysis and IR spectroscopy. X-ray diffraction analysis shows that the obtained material consists of a single phase having an apatitic structure. The catalytic activity of the fluorobritholite synthesized was tested on one pot Biginelli reaction for the synthesis of 3,4-dihydropyrimidin-2(1H)-one [3]. We observed that this britholite has interesting catalytic properties.



Scheme. One-pot synthesis of 3,4-dihydropyrimidinones

Keywords: Fluorobritholite, Biginelli reaction, 3,4-dihydropyrimidin-2(1H)-one

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

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

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

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SYNTHESIS, STRUCTURAL STUDY BY XR DIFFRACTION ON SINGLE CRYSTAL OF A NEW COORDINATION COMPLEX BASED ON COPPER AND 4, 4'-DIAMINODIPHENYL SULFONE LIGAND

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The rational design, synthesis and characterization of new metal-organic architectures arouse great interest in coordination and supramolecular chemistry, not only for their intriguing variety of structures, but also for their potential applications as functional materials [1]. During our investigation we successfully synthesized a new coordination copper complexes using 4,4'-Diaminodiophenyl sulfone ligand well known for his multiple biological properties[2]. The Diaquadichlorobis(4,4'-diaminodiphenylsulfone) copper(II) crystallises in the monoclinic system, $P2_1/n$ space group with the following cell parameters: a= 5.7379(5)Å b= 7.6381(5)Å c= 31.6814(5)Å with β = 92.7741(5)°. Asymmetric unit of the coordination complexes (Fig.1) consists of one Cu^{II} ion, one coordinated neutral ligand (4,4'-daps), one chloride ion and one coordinated water molecule.

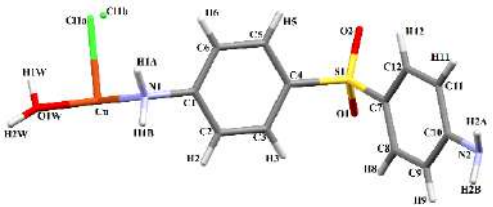

Fig.1: Asymmetric unit of *[Cu^{II}(4,4'-daps)(Cl)(H_2O)]*

Key words: Copper complexes, Hydrogen Bonds, Daps ligand.

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Encapsulation of anticancer drug doxorubicin inside dendritic macromolecular cavities: A theoretical study

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The drug molecule vectorization is a very promising approach in terms of both medical and economical factors for the delivery of active substances with low bioavailability[1]. Symmetrical and branched macromolecules such as star polymers and dendrimers, seem to be more attractive solutions. Indeed, these structures can effectively combine a high stability in biological media and the ability to encapsulate active ingredients. Thanks to the well-defined architecture, they can achieve a high level of reproducibility, while avoiding the problem of polydispersity[2]. In recent years, many dendritic structures have been proposed; however, the design of new effective dendritic nanocarriers is still relevant. This is due to many reasons such as related to biocompatibility, encapsulation efficiency of therapeutic agents, as well as economic reasons. Considering this, we present theoretical results based on density functional theory to study the encapsulation drugs by dendrimers .

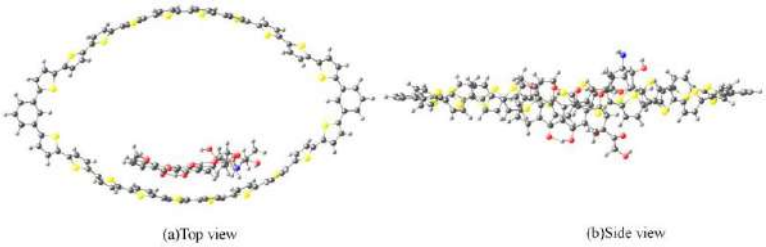

Figure: Encapsulation of Doxorubicine inside thiophene (phenyl-cored) dendrimer (C, O, H, N and S atoms are represented as: gray, red, white, blue, yellow).

Keywords : Dendrimers, DFT, anticancer drugs..

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Effect of soil parameters on the corrosivity of buried-steel

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The aim objective of study is to investigate soil parameters such as pH, resistivity, salinity and sulphate those affect the corrosive nature of soils toward the buried-steel used in the Ecumed petroleum society in Zarzis, Tunisia. The corrosion rate of the buried-steel was evaluated by the weight loss and electrochemical methods. The test shows that presence of chlorides or sulphate ions in the soil create very severe environment, in fat they increases the corrosion rate values.

Keywords: Buried-steel, Soil, Weight loss, Electrochemical methods.

Synthesis, Characterization and vibrational studies of a new compound $Na_6(TeMo_6O_{24})[H_2SO_4].12H_2O$

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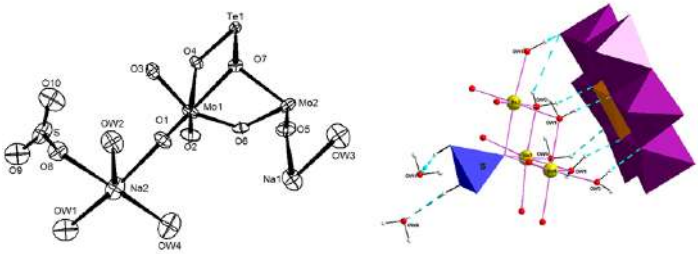
The remarkable chemical and physical properties of polyoxometalate anions (POMs) have been of interest for many years due to their large variety of applications such as medicine, biology, catalysis, material sciences and chemical analysis [1-5]
Single crystals of a new molybdo-tellurate compound $Na_6(TeMo_6O_{24}).(H_2SO_4)_2.12H_2O$ (I) were grown by the low evaporation technique and characterized by X-ray diffraction, infrared absorption and the optical study was also investigated by UV-Vis absorption spectrum. The compound crystallizes in the monoclinic system and C2/m space group with a = 16,431(1)Å, b = 12,493(1)Å, c =10,329(1)Å, β = 109,55(1) **V** = 1998.1(2) $Å^3$, Z = 2, R = 0. 0.0395 and wR (F^2) = 0.090 for 1890 unique observed reflexions [I > 2s(I)].
The crystal structures of the compounds exhibit three-dimensional supramolecular assembly based on the extensive hydrogen bonding interactions between, H_2SO_4 molecule, sodium cations, water molecules and Anderson polyoxoanions $(TeMo_6O_{24})^{6-}$. The infrared spectrum fully confirms the X-ray crystal structure and the UV spectrum of the title compound exhibits an absorption peak at 220 nm.

Keyword : Polyoxometalates, clusters, X-ray diffraction, Three-dimensional structures, Infrared



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CHEMICAL SYNTHESIS, CRYSTAL STRUCTURE, VIBRATIONAL AND UV-VIS SPECTROSCOPY OF NEW HYBRID COMPOUND BASED ON SULFATE

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Organic-inorganic hybrid materials are endowed with the advantages of both of their building components. In fact, they exhibit a manifold of intermolecular interactions such as hydrogen bonds that contribute significantly in the building and the stabilization of such structures. Hence, new single crystals of organic sulfate were synthesized using slow evaporation solution growth technique and were later characterized by single crystal X-ray diffraction.

The title compound crystallizes in the monoclinic system with the non centro-symmetric space group $P2_1$. The unit cell dimensions are: a = 10.6218 (4) Å, b = 9.6697 (4) Å, c = 14.1442 (5) Å, β = 106.322 (4) °, V = 1394.20 (10) $Å^3$ and Z = 2. The atomic arrangement consists of inorganic layers built by $[SO_4]^{2-}$ isolated tetrahedron between which organic cations $[C_{12}H_{14}N_2O_2S]^{2+}$ are inserted. It is worth noting that the organic layers are built up by two crystallographic independent cations. The packing of these basic units in the crystal structure was found to be ensured by means of N-H... O hydrogen bonds.

The present work also outlines a vibrational study of the different entities present in the synthesized compound lead by IR and Raman techniques and identified by a specific comparison with similar compounds.

Moreover, UV-Vis spectroscopy investigation was performed and the different pics were assigned.

SYNTHESIS, STRUCTURAL STUDY AND BIOLOGICAL ACTIVITY OF A NEW SILVER COMPLEXE

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The heterocyclic compounds with nitrogen atoms in the ring system have frequently been used in the synthesis of biologically active complexes. It is, however, noteworthy that the biological activity is enhanced on undergoing complexation with metals [1-2]. The antimicrobial properties of silver complexes are due to their peculiar mechanisms of action, different from that of other metal compounds. Silver can kill bacteria by entering cells through the impairment of essential enzymes that can bind Ag^+ on the surface of the cell wall or weakening the cell wall integrity by removal of an electron from these cellular components [3-6].

The compound: [Ag (L)] (NO3) (H2O) was synthesized under reflux (**L** = **[$C_9H_9N_4O_2$]**).

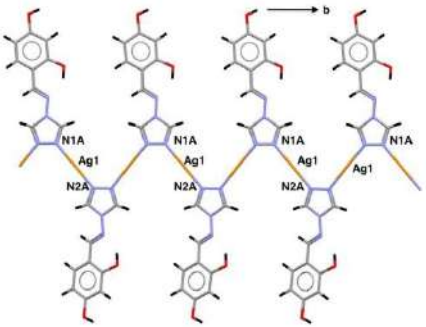

Figure: Polymeric chains of [Ag(L)]n(NO3)n(H2O) developing along b axis.

Key words: Silver complexes, X ray diffraction, biological activity.

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Crystalline structures and magnetic properties of water soluble calixarenes complexes

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Initial work with calixarenes involved the *p-tert-butyl* derivative which has a very low water solubility[1] Substitution at either the 'upper rim', R, or the 'lower rim', Y, of the calixarene, can lead to water-soluble species. These substances are of particular importance with regard to cation complexation[2].
Our interest are the species which contain transition metal ions or complexes. The pillaring of clays by transition metal complexes is a popular form of activation of the clays for organic reaction purposes[3]. For transition metal ions three possibilities present themselves: the metal ion could bridge the layers, the metal ion could be bound to one of the layers only, or the metal ion could be intercalated in the form of a complex between layers.

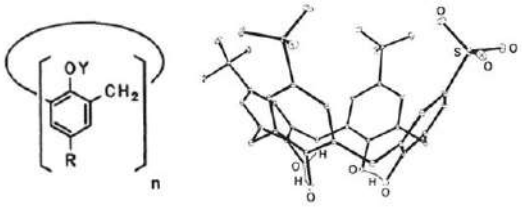

Figure: Cone conformation for the [calix[4]arene sulfonate]- anion stabilized by intramolecular hydrogen bonding

Key words:water soluble calixarene, complexes,crystalline structure, magnetic properties.

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Effective heterogeneous electro-Fenton process for the removal of polyvinylamine sulfonate anthrapyridone, dye, using pyrite as a reusable catalyst

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The release of dye-containing effluents into water environment leads severe pollution problems worldwide owing to the presence of toxic, carcinogenic or mutagenic products such as heavy metals and aromatic compounds with the concomitant aesthetic effect. Electrochemical Advanced oxidation processes (EAOPs) are considered an suitable alternative to remove these recalcitrant compounds from wastewater. One of the most popular EAOPs is electro-Fenton process; however, the use of soluble iron instead of heterogeneous catalyst presents numerous operational problems.

Pyrite, the most abundant sulphide mineral in the Earth's crust, has been successfully used as heterogeneous catalyst for several processes based on Fenton's reaction. Its capacity to regulate the iron ions into the solution in the presence of O_2 enhances the degradation and mineralization effectiveness compared to classical Fenton process [1]. Therefore, the goal of this study was to evaluate the applicability of electro-Fenton-pyrite process fordecolourization and degradation of a polymeric dyesuch as Poly R-478 and optimize different operational parameters such as pyrite dosage and applied current intensity.

Keywords: heterogeneous electro-Fenton, polymeric dye, pyrite, reusability

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SOLID PHASE EXTRACTION OF SOME HEAVY METALS FROM AQUEOUS SOLUTIONS USING POLY (VINYL CHLORIDE) FUNCTIONALIZED BY POLYALKYLAMINES

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Three polyamine-functionalized poly (vinyl chloride) prepared by ethylenediamine (EDA), diethylenetriamine (DETA) and triethylenetetramine (TETA) called respectively: PVC-EDA, PVC-DETA and PVC-TETA. The synthesized modified polymers have been investigated as high-capacity chelating agents for trivalent cations Bi(III) and Cr(III) from aqueous solutions. The structural properties of the polymers were characterized by elemental analysis and infrared spectroscopy (FT-IR). These new polymers were found to be highly effective for the extraction of Bi(III) and Cr(III) ions. Kinetic data revealed that the extraction process was achieved within 24 hours. The functionalized polymer by TETA: PVC-TETA had a high selectivity of extraction for the metals Bi(III) and Cr (III) with percentages of extraction respectively about 97 % and 90 %.

Keywords: Poly (vinyl chloride), Polyalkylamines, Functionalization, Metal cations, Extraction.

REMOVAL OF Cr(III) AND Al(III) IONS FROM AQUEOUS SOLUTIONS USING POLY (VINYL CHLORIDE) FUNCTIONALIZED BY ALIPHATIC AND AROMATIC AMINES

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The aim of this study was to evaluate the efficiency of two chelating polymers obtained by functionalization of a commercial poly (vinyl chloride) by grafting diethylenetriamine (DETA) and aniline (ANI) groups to extract some metal cations during depollution applications. The obtained polymers were characterized via attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR), elemental analysis (EA) and differential scanning calorimetry (DSC). The percentage of extraction was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The synthesized polymers were then assayed to evaluate their efficiency to extract metallic ions such as Cr^{3+} and Al^{3+} from aqueous solutions. These chelating resins were found to extract these ion at high level (> 90 %) with a clear selectivity and seemed to have an affinity following the order Cr^{3+}> Al^{3+}.

Keywords: Poly (vinyl chloride), Functionalization, Amines, Metal cations, Extraction.

Synthesis and structural study of the diphosphate $KHoP_2O_7$

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The diphosphate $KHoP_2O_7$ was synthesized, by the solid method, in the presence of holmium. The obtained powder was characterized by XRD diffraction and infrared spectroscopy. Results reveal the formation of a unique crystalline phase of $KHoP_2O_7$. This phase crystallizes in the orthorhombic system with Cmcm space group with cell parameters:
a = 5.718 (2) Å, b = 9.217 (1) Å, c = 12.248 (2) Å, V = 645.49 (1) $Å^3$, Z = 4.
This structure is characterized by a three-dimensional framework formed by HoO_6 octahedra and $P_2O_7^{4-}$diphosphates which adopts an eclipsed confirmation, connected by Ho-O-P bridges and arranged in alternating layers (Figure 1).
This figure represents the projection of the structure according to [110]. We chose this direction for a better presentation of the connections between polyhedra as well as channels containing the K^+ ions.

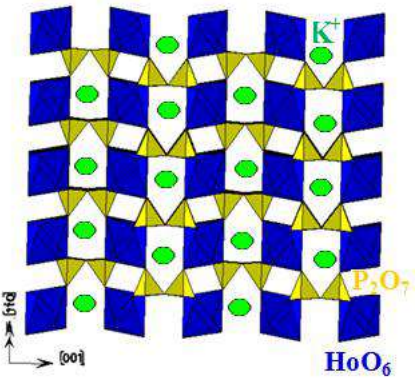

Figure 1: View Projection of the $KHoP_2O_7$ structure according to the direction [110].

REMOVAL OF ANTI - INFLAMMATORY DRUG IBUPROFEN FROM ELUENT BY USING A NATURAL CLAY OF ALGERIA

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The presence of emerging contaminant in water has become a serious concern in recent times. There are no standard treatment process designed to remove pharmaceuticals, as ibuprofen from water. Clay is ubiquitous material not expensive, which makes competitive with other adsorbents such as activated carbon better known. This study concerns the removal of ibuprofen by natural clay from El Milia (Jijel, Algeria). The study of the structure of this clay shows that its composition is kaolinite, calcite and quartz .The elimination of ibuprofen by this clay is around 90%. This elimination is affected by the increase temperature, the increase pH and the agitation speed . This elimination decreases with the temperature variation and by chemical treatment with nitric acid and phosphoric acid. The concentration of ibuprofen after treatment was quantified using a Ultra-Violet – Visible spectro-photometer. The test results describe the adsorption process as a pseudo second order kinetics and that the isotherm is well described by the Langmuir model.

Key words : adsorption, clay, ibuprofen, isotherm.

ELECTRIC MODULUS ANALYSIS AND DIELECTRIC BEHAVIOR OF $Na_3Nb_4As_3O_{19}$ COMPOUND

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The synthesis of compounds containing monovalent cations with tunnel or layer structures is of potential interest due to their remarkable ionic conductivity **[1-4]**. In this context, we have investigated A–Nb–As–O system (A = monovalent cation) and isolated a few novel mixed oxides **[5-8]** such as $Na_3Nb_4As_3O_{19}$.
In fact, the $Na_3Nb_4As_3O_{19}$ compound was synthesized via conventional solid state technique using Na_2CO_3, Nb_2O_5 and $NH_4H_2AsO_4$ as starting materials. Its structure can be described as a three-dimensional anionic framework containing two kinds of tunnels parallel to [001] direction where Na^+ cations are located.
Dielectric relaxation study is very important for understanding the transport behavior of ions in solid state ionic conductors. The conductivity mechanism of our material has been investigated using combined complex impedance, dielectric and modulus formalisms over a temperature interval from 573 to 893 K and in the frequency range between 0.01 and 13000 kHz.
The purpose of the present work is to report the electrical and dielectric behavior of $Na_3Nb_4As_3O_{19}$ compound and to establish correlation between its structure and its physical properties.

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Involvement of hybrid materials based on natural clay and poly(vinyl chloride) into extraction of metal cations

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The solid-liquid extraction is one of the most used methods for protection of the environment against organic and inorganic pollutants. The hybrid materials have been synthesized by a simple meth- od from natural clay and modified poly(vinyl chloride) (PVC). The obtained products were character- ized by various techniques, infrared spectroscopy (FT-IR), thermogravimetric analysis (TGA) and *differ- ential thermal analysis* (DTA). The final materials were successfully used for entrapping heavy metal ions (Fe, Cu, Cd, Zn, Pb, Mn and Co). The quantities of adsorbed metal cations were determined by atomic absorption analysis. It has been revealed that the metals extraction was governed essentially by the af- finity between the functional groups of the hybrid material and the investigated metal ion. The cations are extracted in the following decreasing order of effectiveness: $Fe^{3+} > Cu^{2+} > Co^{2+} \approx Mn^{2+}$ Pb^{2+} $Cd^{2+} \approx Zn^{2+}$.

Keywords: modified poly(vinyl chloride), clay, hybrid material, metal cations, extraction.

Electrolyte materials for Solid Oxide Fuel Cell: Complex influence of isomorphous partial substitution of Mo^{6+} by S^{6+} in the novel $La_2Mo_2O_9$ – based oxide ion conductors

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The oxide-ion conductor is a kind of solid electrolyte, in which the majority of charge carriers are oxygen ions and the ionic conductivity σ_i is much higher than the electronic conductivity σ_e. In fact, the conductivity of LAMOX can be considered as purely ionic because the ionic conductivity transport number is approximately 0.999 at 750 °C **[1]**. In order to examine the effect of isovalent Molybdenum site substitution by chalcogen element (Sulfur) on the performance of fast oxide ion conductor β-$La_2Mo_2O_9$ doped with the nominal compositions $La_2Mo_{2-y}S_yO_9$, the classical solid state reaction is the synthetic approach that was used for this solid solution at ambient atmosphere and temperature. Unit cell parameters have been obtained from the diffraction pattern fitting by the le Bail method **[2]** using the program FullProf **[3]**. Crystallization behavior and ionic conductivity of these electrolytes were investigated from temperature controlled X-ray powder diffraction including the Rietveld algorithm), thermal analyses, complex impedance spectroscopy and vibrational analyses. The main objective is to obtain a better insight into the structure, thermal stability, oxide-ion conducting properties and spectroscopic vibrations of these doped series.

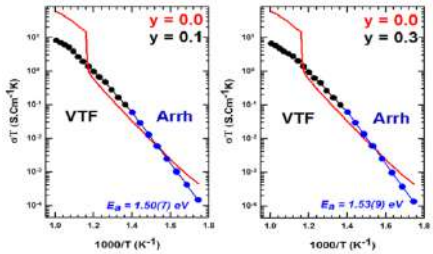

Fig.1: Thermal evolution of the conductivity of Arrhenius and VTF regimes for the solid-solution $La_2Mo_{2-y}S_yO_9$ as function of y. The curve of $La_2Mo_2O_9$ denoted by solid red line is presented at each graph for comparison

Key words: oxide-ion conductor, $La_2Mo_{2-y}S_yO_9$, substitution, Rietveld, electrolytes, FullProf.

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Comparison of the kinetics of photo catalytic degradation of methylene blue on a suspended and immobilized TiO_2

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The purpose of this work is to study the kinetics of photocatalytic degradation of methylene blue by pseudo homogeneous (TiO 2 in suspension in the solution) or heterogeneous (TiO2 films fixed on glass plates).
For several decades, photocatalysis has been widely studied and has found various applications for the degradation of phenolic compounds, dyes or emerging pollutants such as pesticides and medicines. The speed of the photocatalytic reaction depends on several factors: the nature, the shape, the particle size of the catalyst and to the light source. In this work, experiments are carried out under three operating conditions: agitation, pH and concentration Co varying on five levels and under solar irradiation for the two forms of catalyst. The response surface method was applied to find a reliable relation between these parameters and the catalytic efficiency for the two categories of catalysis. Pseudo first order and second order kinetics were applied to study the transient behavior of the reaction. Second-order polynomials with interaction were found on the basis of a series of tests carried out for the two catalysts. The analysis of the variance for the coefficients of the regression equations was carried out by Minitab 17. The exploitation of the surfaces responses and iso response curves has allowed to find the areas of optimal variation of these parameters ensuring the best removal yield.

Key words: degradation, kinetics, homogeneous photocatalyst, heterogeneous, TiO2, response surface

ORGANIC TEMPLATED METAL HALIDE: THERMAL BEHAVIOR AND MAGNETO-STRUCTURAL CORRELATIONS.

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A molecular copper **(II)** complex has been grown by the slow evaporation method at room temperature. The grown crystals were subjected to multiple characterization techniques. The crystal packing is guided by N-H···Br hydrogen bonds and π···π interactions. The thermal decompositions of the complexes studied by simultaneous TGA-TD analysis and DSC show endothermic phenomena that may mark interesting structural phase transitions. The *In situ* Xray powder diffraction measurements show amorphization of the material at the first endothermic peak's temperature, which may correspond to a melting. Interestingly, $Cu^{I}Br$ crystallizes slowly from 130 °C and it seems to be a material of importance for chemical sensors. The compound exhibits weak antiferromagnetic interactions.

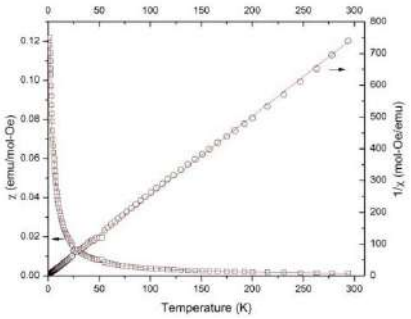

Fig. Susceptibility data for compound plotted as χ(T) and χ-1(T).

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

STRUCTURAL STUDY, HIRSHFELD SURFACE ANALYSIS, SPECTROSCOPIC PROPERTIES AND ELECTRICAL STUDIES OF A NEW HYBRID COMPOUND $(C_{10}H_{16}N_2)CDCL_4$

DHIEB Abdelhamid Chiheb, WACHERINE Intissar,
RZAIGUI Mohamed, SMIRANI Wajda

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A new organic-inorganic hybrid material of formula $(C_{10}H_{16}N_2)CdCl_4$ was synthesized and characterized by X-Ray diffraction analysis. It crystallizes in the $P2_1/c$ monoclinic space group with the following unit cell parameters: a=7.7978(2) Å, b=20.4170(4) Å, c=9.7703(2) Å, Z=4 and V=1498.03(6) $Å^3$. The X-ray structural analysis supported by a detailed Hirshfeld surface analysis has been elucidated the different inter-contacts in the crystal structure mainly associated with N-H···Cl and C-H···Cl contacts between $[CdCl_4^{2-}]$ anions and 1-Phenylpiperazine-1,4-diium cations (Fig 1). IR, Raman, NMR, UV-Visible and fluorescent spectroscopies were also used to characterize this complex. Differential scanning calorimetry (DSC) has revealed a structural phase transition of the order-disorder type around 390 K, and dielectric measurements were performed to discuss the mechanism of this phase transition (Fig. 2). The evolution of dielectric constant as a function of temperature of the sample has been investigated in order to determine some related parameters.

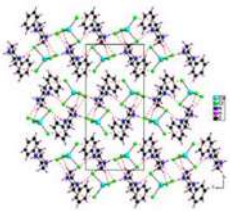

Fig 1. View of the atomic arrangement of $(C_{10}H_{16}N_2)CdCl_4$

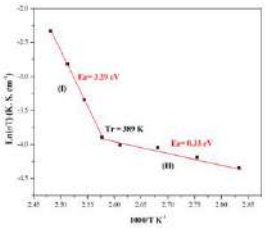

Fig 2. Temperature dependence of the conductivity

Keywords: Crystal structure; Hirshfeld surface analysis; IR-Raman spectroscopy; photoluminescence; dielectric study

Synthesis and Characterization of a novel (C7H10N2) [HW11O39]. 4H2O

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[b)] *Faculty of Sciences of Monastir, University of Monastir*
[c)] *Superior Institute of Applied Sciences and Technologies, Mahdia, University of Monastir*

The current increasing attention and powerful driving force in the polyoxometalate (POM) chemistry, originated from their fascinating properties in material science, medicine [1], catalysis, etc In this context a new compound (C7H10N2) [HW11O39]. 4H2O have been isolated by conventional solution method and characterized by single-crystal diffraction, IR and UV–visible spectroscopies.

Compound 1 crystallizes in monoclinic system, space group P 21/n with a=11.555Å, b=30.371Å, c= 22.255Å, β= 94.36°

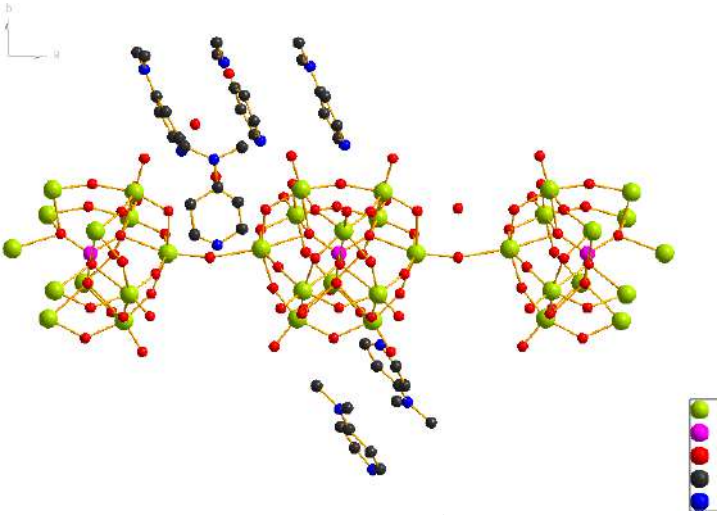

Fig. A Projection along the [001] direction of the title compound.

Reference

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Synthesis and crystal structure of a new hybrid Wells-Dawson-type arsenomolybdate

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The reaction involving the combination of *in situ* generated classical α-Wells-Dawson-type arsenomolybdate [1-2] with 3-aminotriazol in a very acidic aqueous medium (pH =1) leads to the formation of a new organic-inorganic hybrid salt based on polyoxometalate clusters, namely $(C_2N_4H_5)_6[As_2Mo_{18}O_{62}]\cdot 12H_2O$ ($C_2N_4H_5$= 3-amino-1,2,4-triazolium) (**1**). The elaborated compound has been chemically (elemental analysis, TGA-DTA) and spectroscopically (FT-IR, UV-Vis) characterized and its electrochemical behavior studied by cyclic voltammetry (CV). The structural analysis by single-crystal X-ray diffraction reveals that the crystal packing of **1** consists of discrete α-Wells-Dawson-type $[As_2Mo_{18}O_{62}]^{6-}$ polyoxoanions linked by organic 3-amino-triazolium cations and hydration water molecules through an extensive hydrogen bonding network. The formation of N–H···O_{POM}, C–H···O_{POM} and Ow–H...Ow –type contacts results in supramolecular zig-zagging chains running along the [100] direction (Fig. 1.).

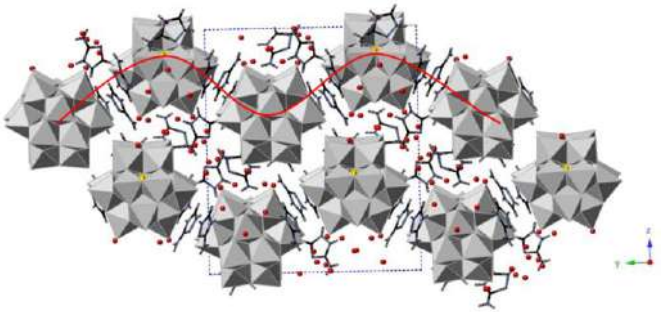

Fig. 1. View along the [100] direction of hybrid **1** highlighting the supramolecular zig-zagging chains

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Electrocatalytic Oxidation of Ascorbic Acid and Dopamine Using a Poly(aniline-co-4-aminobenzyl phosphoric acid) Modified Platinum Electrode

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A poly(aniline-co-4aminobenzyl phosphoric acid) (ANI-co-4ABPA) was successfully synthesized on a platinum electrode (PtE) by electrochemical copolymerization using a scan potential range from 0.05 to +1.4 V (vs. RHE) in acidic medium.
The copolymer confers electrochemical response at pH 7 improving those for the un modified polyaniline (PANI).
The poly(ANI-co-4ABPA) was developed and applied for the determination of ascorbic acid and dopamine in perchloric acid solution. The oxidation of ascorbate anion at the poly(ANI -co-4ABPA)/Pt-modified electrode occurred at 1.2V of less positive potential with an enormous increase in the peak current compared to that of the bare platinum electrode.

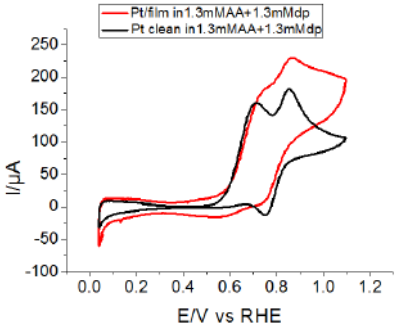

Fig. Electrochemical response of the modified electrode: Pt/(ANI -co-4ABPS) in $HClO_4$ solution after the dosage of dopamine and ascorbic acid. Scan rate 50 mV s^{-1}

Key words: Film, modified electrodes, Poly(aniline-co-4aminobenzyl phosphonic acid); Electrocatalysis; Dopamine; Ascorbic acid; pH effect.

Synthesis and Physico-chemical studies of a new Bi complex $[C_5H_{14}N_2]_2Bi_2Br_{10}.H_2O$

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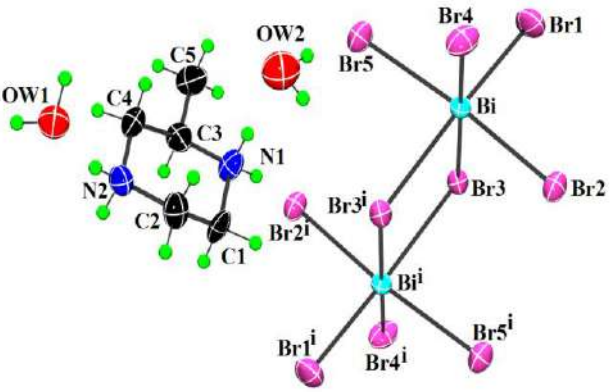
A new Bi complex, $[C_5H_{14}N_2]_2Bi_2Br_{10}.4H_2O$, has been elaborated by slow evaporation at room temperature. This complex crystallizes in the $P2_1/c$ monoclinic unit cell with the following parameters: a=10.368(4), b= 14.012(7), c= 14.360(9) Å, β= 126.25(4)°, V=1682.4(15)$Å^3$ and Z = 2. The crystal structure consists of discrete binuclear $[Bi_2Br_{10}]^{4-}$ anions, $[C_5H_{14}N_2]^{2+}$cations and water molecules. The inorganic entity is made up of $[Bi_2X_{10}]^{4-}$dimers composed of two equivalent irregular octahedra sharing one edge.The diprotonated piperazine ring adopts a chair conformation, with the methyl group occupying an equatorial position. The crystal packing is governed by a three dimensional network of N-H . . . O and N-H . . . Br hydrogen bonds. This material was characterized too by IR spectroscopy, UV–Vis spectrum, photoluminescence and thermal analysis.

Key words: Bi complex; Crystal structure; X-ray diffraction; Optical properties.



Représentation Ortep de la structure de $[C_5H_{14}N_2]_2Bi_2Br_{10}.4H_2O$

Crystal and geometry-optimized structure, Hirshfeld surface analysis and spectroscopic studies of tetrachlorocuprate and nitrate salts of 1-(2-fluorophenyl)piperazine cations $(C_{10}H_{15}FN_2)[CuCl_4]$ (I) and $(C_{10}H_{14}FN_2)[NO_3]$ (II)

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Two new organic-inorganic hybrid materials, 1-(2-fluorophenyl)piperazine-1,4-diium tetrachlorocuprate, $(C_{10}H_{15}FN_2)[CuCl_4]$ **(I)** and 1-(2-fluorophenyl)piperazin-4-ium nitrate, $(C_{10}H_{14}FN_2)[NO_3]$ **(II)**, have been synthesized by an acid/base reaction at room temperature in the presence of 1-(2-fluorophenyl)piperazine as an organic-structure directing agent and their structures were determined by single crystal X-ray diffraction. Compound **(I)**, $(C_{10}H_{15}FN_2)[CuCl_4]$, crystallizes in the monoclinic system and $P2_1/c$ space group with a = 7.5253 (2), b = 20.6070 (7), c =9.7281 (3) Å, β = 103.6730 (17)°, V = 1465.82 (8) Å^3 with Z = 4. Full-matrix least-squares refinement converged at R = 0.037 and $wR(F^2)$ = 0.088. Compound **(II)**, $(C_{10}H_{14}FN_2)[NO_3]$, belongs to the monoclinic system, space group $P2_1/c$ with the following parameters: a =10.8034 (2), b = 7.5775 (1), c =14.4670 (3) Å, β = 111.761 (2)°, V = 1099.91 (4) Å^3 and Z = 4. The structure was refined to R = 0.044, $wR(F^2)$ = 0.136.

In the structures of **(I)** and **(II)**, the anionic and cationic entities are interconnected by means of set of hydrogen bonding contacts forming three-dimensional networks. Intermolecular interactions were investigated by Hirshfeld surface analysis. The results of the optimized molecular structure are presented and compared with the experimental one. The Molecular Electrostatic Potential (MEP) maps and the HOMO and LUMO energy gap of both compounds were computed. The vibrational absorption bands were identified by infrared spectroscopy. Theory (DFT) calculations of normal mode frequencies are compared with experimental ones.

Keywords: X-ray diffraction; Hirshfeld surface; hybrid compounds; IR; CP-MAS NMR; DFT.

Synthesis, crystal structure and physico-chemical characterization of bis(2-methoxybenzylammonium) diaqua-bis(dihydrogen diphosphato)-manganate(II) dihydrate

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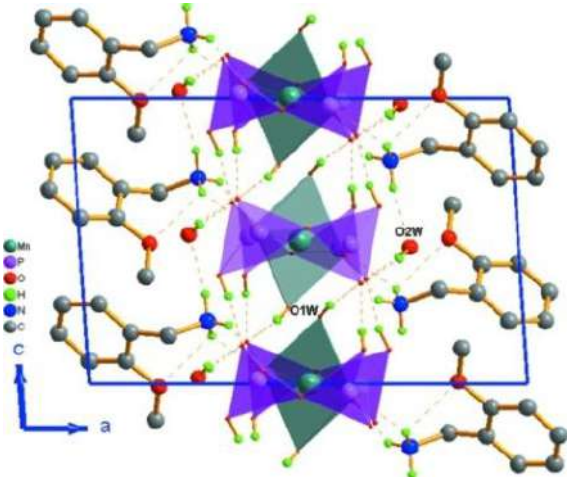
The asymmetric unit of the title compound, $(C_8H_{12}NO)_2$-$[Mn(H_2P_2O_7)_2(H_2O)_2]$ $2H_2O$, consists of half an Mn^{II} complex anion, a 2-methoxybenylammonium cation and a solvent water molecule. The Mn^{II} complex anion lies across an inversion center, and has a slightly distorted octahedral coordination environment for the Mn^{II} ion, formed by two bidentate dihydrogendiphosphate ligands and two water molecules. In the crystal, the components are linked by O- H O and N—H O hydrogen bonds, forming layers parallel to (100). An intramolecular N-H O hydrogen bond is also observed.



Projection of the $[2\text{-}(CH_3O)C_6H_4CH_2NH_3]_2[Co(H_2P_2O_7)_2(H_2O)_2].2H_2O$ along the *b*-axis.

Crystal structure, vibrational and optical properties of a new organic-inorganic material: $(C_5H_7N_2)_2[SbCl_4]Cl$.

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the new organic antimonate(III) $(C_5H_7N_2)_2[SbCl_4]Cl$, was synthesized by slow evaporation method, which was characterized by single-crystal X-ray diffraction, infrared absorption, Raman spectroscopy scattering, optical absorption and photoluminescence measurements.
The centrosymetric compound crystallizes in the triclinic system of space group $P\bar{1}$ with two formula units cell(Z=2).
The unit cell dimensions are : a= 9.5018(5)Å, b= 10.4618(5)Å, c= 12.2616(5)Å, α= 115.2500(5)°, β= 109.3762(5)°, γ= 90.2513(5)° and V = 1024.6(8) $Å^3$.
the crystal structure is composed of discrete $[SbCl_4]$ anions with two Cl atoms isoleted which carried the negative charge to balance the total charge of this compound surrounded by 4 pyridinethioamide cations which are interconnected by means of hydrogen bonding contacts N-H...Cl to form a Zero-dimensional network.
The infrared and Raman study at room temperature charge in the 4000-400 and 500-100 cm^{-1} frenquency regions respectively, shows the existence of the organic cation $[C_5H_7N_2]^+$ and of the $[SbCl_4]^-$ anion.
The Photoluminescence spectrum shows a broad and strong band of luminescence located at 1.95 eV (635nm), which can be even observed with naked eye at room temperature and is due to exciton emission.

The electrochemical degradation of RG 19 with combination of Electro-Fenton and anodic oxidation by BDD anode using the definitive-screening design and optimal augmentation

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In this work, a novel three-level definitive screening design (DSD) was initially employed to investigate the electrochemical degradation of Reactive Green 19 (RG19) by combination of Electro-Fenton(EF) using reticulated vitreous carbon (RVC) cathode coupled with anodic oxidation using boron-doped diamond (BDD) anode . This method allows a dramatic reduction in the number of experiments needed to investigate those systems characterized by a large number of variables. The effects of nine quantitative parameters on RG19 removal rate: initial dye concentration (50-150 mg L^{-1}), current density (200-400 A m^{-2}), NaCl concentration (5-15 mM), Na_2SO_4 concentration (35-65 mM), pH (2-4), temperature (10–40°C), inter electrode distance (0.5-3.5 cm), stirring rate (250-750 rpm) and Fe^{2+} concentration (0.05-0.15 mg/L). The model obtained was validated and used to select four out of the nine factors wich are initial dye concentration, current density, pH and temperature; were statistically significant. These factors were retained for process characterization using a D-optimal augmentation design (OAD). Overall, the number of experiments was reduced from over 500 to only 31, thus confirming the validity of the proposed approach as a time saving and efficient method. This was confirmed with UV-vis and TOC assessments during the EF/AO treatment.

Keywords: Reactive Green 19, combination EF-AO, Definitive screening design, D-optimal augmentation.

Synthesis, characterization of a new organic-inorganic hybrid material trans-2,5-dimethylpiperazine-1,4-diium pentachlorobismuthate(III): $[C_6H_{16}N_2]BiCl_5$

Manel Essid, Mohamed Rzaigui

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A new organic-inorganic hybrid compound, trans-2,5-dimethylpiperazine-1,4-diium pentachlorobismuthate(III), $[C_6H_{16}N_2]BiCl_5$, was synthesized and its structure determined by means of single crystal X-ray diffraction studies. It crystallizes in the monoclinic space group C2/c, with the following parameters: a = 15.365(5), b = 12.634(3), c = 8.313(3) Å, ß = 120.59(3)°, V = 1389.1(7) $Å^3$ and Z = 4. Its crystal structure consists of $[BiCl_5]^{2-}$ anions surrounded by $[C_6H_{16}N_2]^{2+}$ cations. Complex hydrogen bonding interactions between $[BiCl_5]^{2-}$ and organic cations through N(C)-H…Cl hydrogen bonds to form a three-dimensional network (Figure). The crystal packing is stabilized by Cl…Cl interactions. The structure reveals that the piperazine ring is in a chair conformation. The vibrational spectrum has been measured at room temperature by FT-infrared spectroscopy (4000-400 cm^{-1}) on polycrystalline sample. The number of the NMR components in the ^{13}C CP-MAS NMR spectrum proves the presence of three crystallographically independent carbons as revealed by X-ray structure determination.

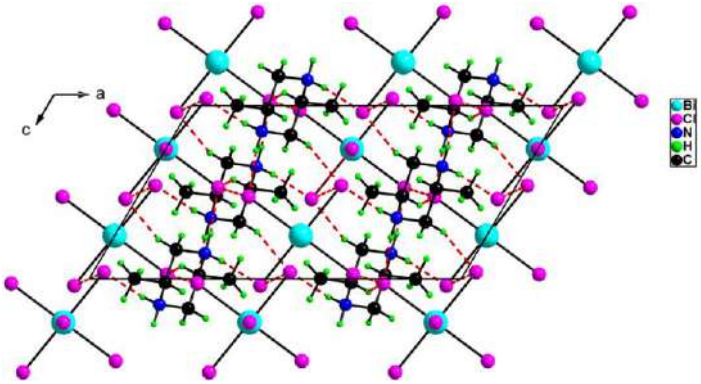

Figure: Projection of crystal structure of $[C_6H_{16}N_2]BiCl_5$ along the *b*-axis.

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

Synthesis, crystal structure, and characterization of a new non-centrosymmetric Bi(III) halide complex: $[C_6H_{16}N_2]_2BiBr_6NO_3$

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A new organic–inorganic hybrid material, $[C_6H_{16}N_2]_2BiBr_6NO_3$, was synthesized, and its structure was determined by means of single crystal X-ray diffraction studies at room temperature in the non-centrosymmetric orthorhombic space group $P2_12_12$ with the following parameters: a = 17.629(5), b = 20.279(5), c = 7.319(5) Å, V = 2617(2) Å^3 and Z = 4. The crystal lattice is composed of discrete hexabromobismuthates and nitrate anions surrounded by trans-2,5-dimethylpiperazine-1,4-diium cations linked via simple and bifurcated N–H…Br(O) and weak C–H…Br(O) hydrogen bonds to form three-dimensional network (Figure). The vibrational spectrum has been measured at room temperature by FT-IR spectroscopy (4000-400 cm^{-1}) on polycrystalline sample, shows the presence of organic cation and nitrate anion. The number of ^{13}C CP-MAS NMR lines is in full agreement with the crystallographic data.

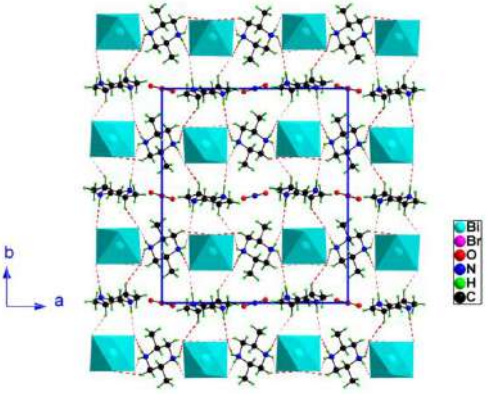

Figure: Projection of the atomic arrangement of $[C_6H_{16}N_2]_2(BiBr_6)NO_3$ along the c-axis.

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

Comparison of Ta/MCM-41 and Ti/MCM-41 as Catalysts for the Enantioselective Epoxidation of Styrene with TBHP

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Enantiomerically pure epoxides are very useful synthetic intermediates in the synthesis of different biologically active compounds [1]. The most important methods for enantioselective epoxidation of alkenes include the use of homogeneous chiral catalysts, such as Ti-tartrates used for epoxidation of allylic alcohols with alkyl hydroperoxides [2].
Chiral Ti- and Ta-MCM41 catalysts have been prepared by grafting of $Ti(O^iPr)_4$ and $Ta(OEt)_5$and modification with R-(+)-diethyl L-tartrate or R-(+)-diisopropylL-tartrate.The solid catalysts are, in general,more active andselective than their homogeneous counterparts in the epoxidation of styrene with*tert*-butylhydroperoxide. The enantioselectivities depend on both the nature of the chiral ligand and the calcination temperature of the support, as it is supposed that controls the type of formed surface species. The best result, 71% ee, is obtained with DIPT-Ta-MCM_{550}, and it is the first example of use of Ta catalyst for enantioselective epoxidation of unfunctionalized alkenes. Unfortunately, recoveredTacatalysts are less active and selective.

References

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2. T. Katsuki in Comprehensive Asymmetric Catalysis, Vol. II; E. N. Jacobsen, A. Pfaltz, H. Yamamoto, Eds.; Springer: Heidelberg, 2000, pp. 621 – 648.

Characterization of Tunisian clay suitable for pharmaceutical and cosmetic applications

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The aim of this study is to assess the suitability of purified Tunisian clay for its possible pharmaceutical and cosmetic uses. Mineralogical, chemical and physicochemical characterization was performed with the aid of tools like X-ray diffraction (XRD), Chemical composition, DTA/TGA analysis, Cation exchange capacity (CEC), particle size distribution by laser dispersion (PSD), surface area (BET method), Point of zero charge (PZC) and Surface charge density σ_H. Pharmacopeia tests were also carried: Swelling capacity, Sedimentation volume and pH measurement. For Microbiological tests, the absence of *Salmonella species*, *Escherichia coli*, *Staphylococcus aureus* and *Pseudomonas aeruginosa* was tested.

The X-ray diffraction data revealed that the Tunisian sample is an interstratified illite-smectite. The percentage of smectitic fraction is estimated to be 78%. The chemical, textural and porosimetric results show important values suggesting that this mineral clay can function in creams, powders and emulsions.

In view of the fundamentals of major pharmacopeias for use of bentonite in pharmacy and considering the swelling capacity and sedimentation volume values, and microbiological results we could designate a pharmaceutical acceptable denomination for Tunisian purified clay.

Keywords: illite-smectite, chemical and textural properties, pharmaceutical and microbiological tests.

Synthesis, theoretical and experimental studies of p-xylylenediaminium bis(nitrate).

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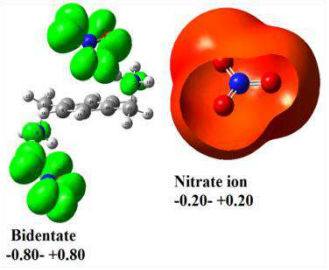
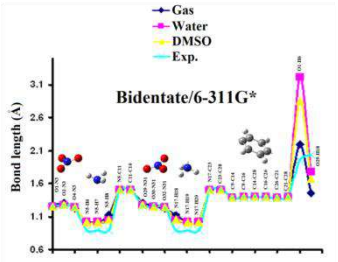
b Quantum Physics Laboratory, Faculty of Sciences, University of Monastir, Monastir, 5079, Tunisia.

The p-xylylenediaminium bis(nitrate) compound have been synthesized and then, it was characterized by using Fourier Transform infrared (FT-IR) in the solid phase and, by using the Ultraviolet-Visible (UV-Visible) and Hydrogen and Carbon Nuclear Magnetic Resonance (^{1}H- and ^{13}C-NMR) spectra in aqueous solution and in dimethylsulfoxide solvent. In this work, two monodentate and bidentate coordination modes were considered for the nitrate ligands in both media in order to study the structural and vibrational properties of that salt. Hence, the natural bond orbital (NBO), atoms in molecules (AIM), Merz-Kollman (MK) charges, molecular electrostatic potentials (MEP) and frontier orbitals studies were performed for p-xylylenediaminium bis(nitrate) and their cation and anion species taking into account for the salt those two coordination modes for the nitrate ligands. The intermolecular interactions of this salt were also evaluated by Hirshfeld surface analysis. The B3LYP calculations performed by using the hybrid method and the 6-311G* and 6-311++G** basis sets generate monodentate and bidentate structures with C_i and C_2 symmetries, respectively. The force fields and the force constants values for these two structures were also computed and their complete vibrational assignments were performed by using those both levels of theory. The strong band at 1536 cm^{-1}, the bands between 2754 and 2547 cm^{-1} and the bands between 1779 and 1704 cm^{-1} support clearly the presence of the dimeric species while the IR bands at 1986/1856 cm^{-1} could justify the presence of the bidentate species in the solid phase.



Calculated electrostatic potential surfaces on the molecular surface of p-xylylenediaminium bis(nitrate) with bidentate coordination modes for the nitrate groups and for the nitrate ion in gas phase.



Bond lengths variations corresponding to bidentate (6-311G*) structures of p-xylylenediaminium bis(nitrate) calculated in gas phase and in aqueous and DMSO solutions at different levels of theory.

Keywords: Monodentate, bidentate, NBO, AIM, MEP, Hirshfeld surface analysis, Force fields.

Synthesis, structure and physico-chemical studies of a novel oxalate material p-xylylenediaminium bis(hydrogen oxalate) hydrate, $[C_8H_{14}N_2](HC_2O_4)_2.H_2O$

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A new material with the general formula $[C_8H_{14}N_2](HC_2O_4)_2.H_2O$, has been synthesized by slow evaporation at room temperature and the structure was determined by single crystal X-Ray diffraction analysis, Hirshfeld surface analysis, IR, UV visible and Thermal analysis. This compound has been crystallized in the monoclinic system, space group $P2/_C$, with a = 20.9359 (11) Å, b = 5.6375 (3) Å, c = 13.8789 (7) Å, β = 101.910 (2) °, V = 1602.81(14) $Å^3$and Z=4.

The asymmetric unit of the title compound contains two halves of the organic cation p-xylylenediaminium cation, two oxalate anions and one water molecule. The structure is built up from anionic chains parallel to the b-axis and interconnected by the organic cations and water molecules via N–H…O, C–H…O, O(W)–H…O hydrogen bonds so as to build a three dimensional arrangement. The intermolecular interactions were further evaluated by using the three-dimensional Hirshfeld surfaces and two-dimensional fingerprint plots. The hydrogen bonding interactions associated to O–H…O, N–H…O and C–H…O represent the top fraction of 52.9 % followed by those of the H…H type contributing 29.5 %.

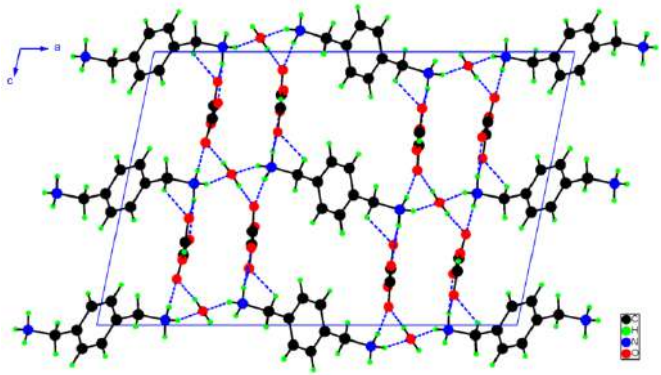

Fig. Projection of $[C_8H_{14}N_2](HC_2O_4)_2.H_2O$ along the b-axis.

Keywords: crystal structure, p-xylylenediaminium, oxalate, Hirshfeld surface analysis, fingerprint maps.

Structural characterization, thermal and dielectric study of organic–inorganic compound $[C_7H_{12}N_2]_2Bi_2Br_{10}\cdot 4H_2O$

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A novel organic-inorganic hybrid compound, 2,4-diammonium toluene decabromodibismuthate(III) tetra-hydrate, was synthesized and its structure was determined by single crystal X-ray diffraction at room temperature. It crystallizes in the monoclinic system with the $P2_1/c$ space group. The structure consists of isolated bioctahedra $[Bi_2Br_{10}]$ sharing one edge, one type of diprotonated $[C_7H_{12}N_2]^{2+}$ cations and waters molecules. The cohesion between these entities is ensured by N-H…Br, N-H…Ow and Ow-H…Br hydrogen bonds that build a three-dimensional network. The atomic arrangement can be described as organic–inorganic layers stacked in [010] direction. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) measurements have been carried out on $[C_7H_{12}N_2]_2Bi_2Br_{10}\cdot 4H_2O$ crystal in the temperature range of 298-700 K and 200-500 K, respectively. The Infrared and ^{13}C NMR spectra of the compound are reported. This study shows the presence of one organic group $C_7H_{12}N_2$ which is in agreement with the X-ray structure. DSC study indicates a presence of one phase transition located at 360 K. Furthermore, the evolution of the dielectric constant as a function of temperature at several frequencies of this compound has been investigated in order to discuss the mechanism of the phase transition. The AC conductivity spectrum was found to obey Jonscher's universal power law, whereas DC conductivity shows a typical Arrhenius. The variation of frequency exponent, n, suggests that the correlated barrier hopping (CBH) is the most probable conduction mechanisms in this material.

Keywords: Bromobismuthate(III), Cristal structure, Infrared and NMR spectroscopy, dielectric proprieties, AC conductivity.

Synthesis and Crystal Structure of a New Acidic Organic Cyclohexaphosphate

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In the crystal structure of the title compound, $4C_5H_6ClN_2^+.H_2P_6O_{18}^{4-}$, the $[H_2P_6O_{18}]^{4-}$ anions are interconnected by strong O-H…O hydrogen bonds characterized by short distances ($d_{O\cdots O}$ = 2.418 (3) Å), leading to the formation of infinite ribbons extending along the *a*-axis direction **(Fig. 1)**. These ribbons are linked to the organic cations, via N-H…O and C-H…O hydrogen bonds, into a three-dimensional network **(Fig. 2)**. It is worth noting that the strong H-bond between phosphoric rings ($d_{O\cdots O}$ < 2.73 Å) is rather observed in cyclohexaphosphates. The six P atoms of the $[H_2P_6O_{18}]^{4-}$ anion form chair conformation. The complete cyclohexaphosphate anion is generated by inversion symmetry. Despite the limited number of organic cation cyclohexaphosphates (about forty related structures), we can distinguish only few acidic cyclohexaphosphates such as the title compound. This compound is also characterized by thermal analysis and IR spectroscopy.

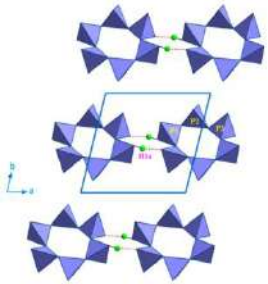

Fig. 1 : Projection of the structure of $(C_5H_6ClN_2)_4\ H_2P_6O_{18}^{4-}$ along the c axis

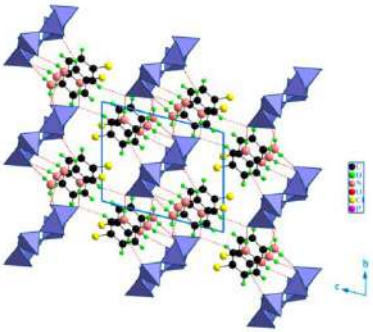

Fig. 2 : The structure of $(C_5H_6ClN_2)_4\ H_2P_6O_{18}^{4-}$ projected along the a axis

SYNTHESIS, STRUCTURE FEATURES AND THERMAL ANALYSIS OF THE NEW COMPLEX $(C_6H_{10}N_2)_4[FEBR_4]_2 \cdot BR_6$

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A new organic–inorganic hybrid material has been synthesized by the slow evaporation method. The grown crystals were subjected to the following characterization techniques: single crystals X-ray diffraction, thermal analysis (ATG-TD), IR and Raman spectroscopy. The title compound crystallizes in the monoclinic system (space group $P2_1/c$) with the following unit-cell parameters: a=12.3579(13) Å, b=28.572(3) Å, c=13.5442(14) Å, β=92.01° and Z=4.This structure is made up of isolated $[FeBr_4]^-$anions, bromide ions (Br^-) and $(C_6H_{10}N_2)^{2+}$ cations . All these entities are linked together by a 3D hydrogen bond network. Both IR and Raman spectroscopies show the protonation of the amine whereas the thermal analysis curves reveal the possibility of a structural phase transition that can lead to interesting physical properties.

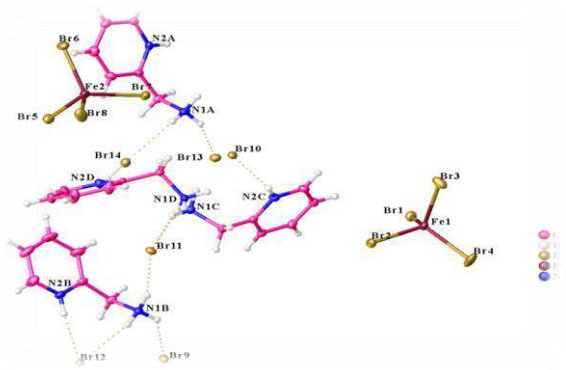

Fig.1. The asymmetric unit of $(C_6H_{10}N_2)_4[FeBr_4]_2 \cdot Br_6$, showing the atom-labelling scheme. the dashed lines represents the hydrogen bonds.

Keywords: X-Ray diffraction, thermal analysis (ATD, TG), 3D network, phase transition.

Synthesis by the Sol-Gel Autocombustion Method and Physicochemical Characterizations of Ferrimagnetic Nanoferrites of Co Ni Cu and Zn.

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The nanomaterial $Ni_{0.2}Cu_{0.2}Co_{0.6}Fe_2O_4$ was synthesized by sol-gel auto combustion process. The formed black gel was dried at 100 °C in order to obtain the preliminary precursor. Furthermore, the calcined powder, obtained by heating the dehydrated precursor at 300 K, has been characterized by other physicochemical techniques: XRD, IR-FT and magnetic measurement ZFC-FC.
The XRD analysis displayed in **Fig.1.** shows the obtaining of only the single phase without any detection of other phases and confirm the nanostructure of the studied material with crystallite size of 84 nm.
However, concerning the IR-FT study, from **Fig. 2.** we can conclude that the results of vibration mode frequency assignment tentative tend to be compatible with bibliographic studies.
Moreover, magnetic measurements in ZFC-FC method shown in **Fig.3.** performed in the temperature range 10-300 K under a magnetic field H = 500 Oe, helped determined the superparamagnetic behavior of this nanomaterial at low temperature and which its blocking temperatures is 283 k.

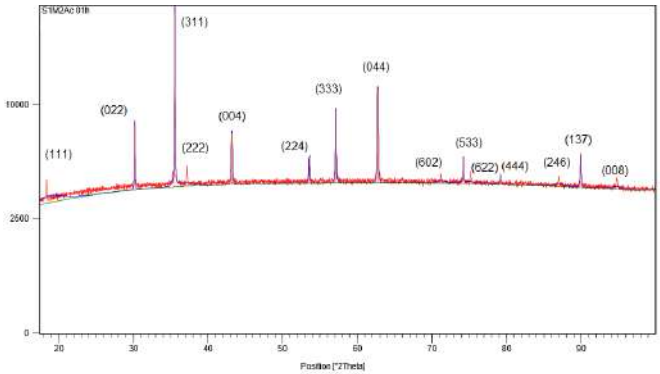

Fig. 1. The X-Ray Powder diffractogram of the Figure 2: The X-Ray Powder diffractogram nanomaterial $Ni_{0.2}Cu_{0.2}Co_{0.6}Fe_2O_4$

Keywords: Sol-Gel Auto Combustion method, Physico-chemical characterizations, Ferrimagnetic Nanoferrites of Co Ni and Cu, DRXP, IR-FT, Magnetic Measurements in ZFC-FC method.

Synthesis of mesouporous magnesium oxide: Its application to CO_2 capture

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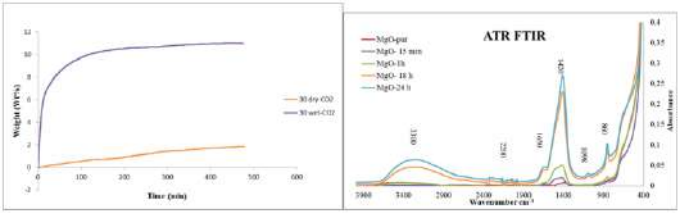
Since the industrial revolution, anthropogenic carbon dioxide come principally from combustion of fossil fuel, has produced a 40% increases in the atmospheric concentration of carbon dioxide[1]. This increase has moved researchers to find solution in order to avoid dangerous global warming. For this purpose, Seifritzet al [2]introduced the mineral carbonation, from that, previous studies focused on the integration of a CO_2 capture and storage process to treat gaseous emissions [3]–[6].

In this context, we synthesize mesoporous MgO via thermal decomposition method from hydromagnesite. After characterization, the obtained materiel was used for CO_2 capture since it presents good textural and morphological proprieties.

In contrast with previous works studying this reaction at high temperatures and high pressures, our results (Fig.1a) indicate that at low temperatures (T<50°C) and atmospheric pressure, steam is no more a catalyst but it participates to the reaction giving with CO_2 an hydrated carbonate product.To identify the resulting product, ATR-FTIR (Fig. 1b) and Raman spectroscopy were used as well as phase diagrams in the system $MgO\text{-}H_2O\text{-}CO_2$. All results confirm that 54% of MgO was converted to $MgCO_3 .3H_2O$.This transformation was accompanied with a noticeable change in the textural and morphological properties of the powder such as the decrease of both specific surface and pore volume and the change of the shape of powder crystals.



(a) (b)

Figure 1.(a) Comparison between carbonation (30°C, P_{tot} = 1 bar) in the absence (bottom) and presence of steam (15 mbar), (b) Infrared spectra of MgO before carbonation, after 15 minutes,1 hour, 18 hours and 24 hours of carbonation **(T= 30°C, P_{CO2}= 500 mbar et P_{H2O}= 15 mbar).**

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New coordination complexes with Fe (II) used as a metal center for lithium batteries

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Today, the development of new lithium battery materials is of great interest, especially since these devices have to be integrated into energy-intensive systems (electric vehicle batteries). The present materials are mainly inorganic and are chosen for their relative high operating potential and their ability to be able to reversibly undergo electron transfer (s) [1]. While it is difficult to combine these two properties, these materials must also have a structure for inserting/releasing the lithium ions. Thus, other alternatives to these materials have been envisaged, such as the use of organic materials whose capacity, durability, and stability deserve to be improved[2]. Surprisingly, the coordination copmlexes are studied very little as pasitive battery elctrode materials even though they are often cited in the context of redx flow[3], coordination complexes have many advantages such as their readily modulable operation by a modification of the ligands or of the metal ii) a relative high chemical stability iii) and a versatility with respect to the cation of the electrolyte.
In this context, we are interested in the use of polymer complexes based on tris-bipyridine iron. After the synthesis of monomers and polymers, an electrochemical study was carried out on model complexes to verify the stability *vs* time of the oxidized forms and to also determine the electron transfer kinetics. Then, the polymer materials were integrated as battery materials. Although the measured capacitances are relatively small (which takes into account the mass of the units undergoing electron transfer), these compounds exhibit a high durability and an operating potential compatible with the stability of the electrolyte (carbonates + lithium salt). These first results will be presented in this poster.

Acknowledgments: I would like to thank MESRS for accepting me in the scholarship program for residential education abroad for teachers and researchers for the academic year 2016-2017 (PNE)

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3 P.J. CabreraX. Yang, J. A. Suttil, R. E. M. Brooner, L.T. Thompson, M. S. Sanford*Inorg. Chem.*, (2015), 54 (21), pp 10214–10223.

Hybrid materials based on nanoparticles of Montmorillonite coating by Poly(pyrrole) used in supercapacitors

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Conductive polymers, such as polypyrrole, have been widely used as a pseudocapacitive electrode in materials for supercapacitors.[1; 2] This work demonstrates a simple method for improving the cyclic stability of conductive polymer electrodes by adding montmorillonite, carrying capacitive behavior. The conductive nanocomposite polymers (PNC), based on polypyrrole/Montmirillonite (PPy(Cl)/MMT) [3], are synthesized by oxidative polymerization using $FeCl_3$ as both dopant and oxidizing agent at the same time. The effect of mass ratio of monomer/reinforcement, influence the pH of medium on the adsorption of pyrrole on the surface of the MMT and temperature medium synthesis are studied. In addition, conductivities values measured by the four-point method of the PPy(Cl)/MMT are higher than that of pure PPy(Cl) or the percolation threshold is with a 0.4 mass ratio of MMT, synthesized at 0°C with an electrical conductivity of 5.04 $S.cm^{-1}$ which is significantly improved over pure polypyrrole. Supported by these overall benefic effect, high performance nanocomposite PPy(Cl)/MMT capacitor have been obtained with high specific capacity of 461 $F.gr^{-1}$ with respect to the nanocomposite synthesized at ambient temperature (220 $F.gr^{-1}$). [4] Analysis by infrared spectrometry shows a strong interaction between the MMT and the polypyrrole matrix confirmed by a shift of the vibration bands from PPy(Cl) towards lower values whereas the microstructures observed by scanning electron microscopy and powder X-ray diffraction analysis attests the good coating of MMT by PPy(Cl).

Acknowledgments: I would like to thank MESRS for accepting me in the scholarship program for residential education abroad for teachers and researchers for the academic year 2016-2017 (PNE)

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Yields, chemical composition, physicochemical properties and antioxidant activities of *Pinushalepensis* essential oils

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Laboratory of material Chemistry, Department of Chemistry,
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Since the middle ages, essential oils have been widely used for bactericidal, virucidal, fungicidal, antiparasitical, insecticidal, medicinal and cosmetic applications, especially nowadays in pharmaceutical, sanitary, cosmetic, agricultural and food industries. In this study, essential oil of PinusHalepensis, growing in Rimel forest was obtained by anhydrodistillationusing Clevenger Apparatus. Essential oils were extracted mainly from leaves and twigs of Pinus by various drying time and in different collection months. However, they were analysed by chromatography/mass spectrometric detection GC/MS. In vitro antioxidant activity was performed by the method of reduction of DPPH radical to evaluate the antioxidant characteristics. During our trials for yields optimization, we noticed a great variability especially according to:

- ✓ The state of vegetable matter
- ✓ The studied organ
- ✓ Vegetative cycle
- ✓ The aged vegetative material

Keywords: PinusHalpensis, essential oil, GC/MS, antioxidant activity, DPPH radical.

Synthesis, crystal structure, spectroscopic study, photoluminiscent properties and DFT calculations of 2-guanidinobenzimidazolium dichloride monohydrate salt

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Crystals of 2-guanidinobenzimidazolium dichloride monohydrate **(I)** were prepared and characterized through single-crystal X-ray diffraction, photoluminescence,IR and UV-Vis diffuse reflectance spectroscopy. The structural unit of **(I)** consists of a discrete guanidinobenzimidazolium, twoCl^- and one water molecule of crystallization**(Fig.)**. The crystal structure of the title salt is stabilized by N-H…Cl, O-H…Cl, N-H…O and C-H…Cl hydrogen bonds.Moreover, the cations show a π-π interaction adding extra stability to the three-dimensional architecture.The structure of elaborated compound was computed with the Gaussian 09 program using the DFT hybrid B3LYP functional and the 6-311+G(d,p) basis set.The electronic transitions were calculated using the time dependent TD-DFT method. The electronic absorption and infrared spectrums of the title compound were in good agreement with the experimental data.

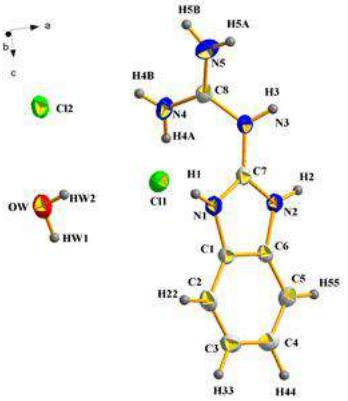

Fig. The asymmetric unit of the crystal structure of(I) showing the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level

Key words: crystal structure, photoluminescence, IR, UV-Vis diffuse reflectance spectroscopy, DFT calculations.

Removal of Eriochrome Black T by Adsorption onto Spent Coffee Grounds

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This work aims to reduce the environmental impact of an acid dye the black Eriocrome T (EBT) using coffee grounds, a domestic waste prepared and characterized previously. A set of experiments was then carried out in order to study the influence of certain parameters on the adsorption capacity, such as adsorbent dosage, initial dye concentration, contact time, pH, stirring rate and temperature. The whole of the results shows that the adsorption of the NET by coffee grounds is more favorable in an acidic. The adsorption process follows a pseudo-second order kinetic. The experimental results were analyzed using the Langmuir, Freundlich, Tempkin and Dubinin-Radushkevich models. It turns out that the adsorption of the NET by coffee grounds is better described by Tempkin equation with a high correlation coefficient (R^2= 0.971) and a weak normal standard deviation (Δq= 0.021). It has been demonstrated that the adsorption of the NET on coffee grounds is a physical type, endothermic and non spontaneous.

Keywords: Adsorption, dye, coffee grounds, isotherm, kinetic.

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Unusual bis(μ-aqua) bis(μ-sulfato) bridges in binuclear cobalt-based 4-aminopyridine: Synthesis, structural feature, electrical properties and magnetic behavior

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In order to improve the relationship between the structure of bridging dinuclear complexes and their magnetic behavior, we did synthesize metal-organic $[Co_2(\mu_2\text{-}H_2O)_2(\mu_2\text{-}SO_4)_2(C_5H_6N_2)_4].(C_2H_6OS)_4(C_4H_8O)_4$ (**1**). This dinuclear transition metal complex was characterized by infrared and UV-vis spectroscopy as well as single X-ray diffraction. The thermal and magnetic behavior of this complex was studied. Single crystal X-ray diffraction studies confirmed that **1** consists of discrete dinuclear cobalt entities and two free solvent molecules (DMSO, THF). The two cobalt ions are bridged by two water oxygen atoms and two bidentate sulfate groups to form a four bridged dinculear core. The optical band gap was determined by diffuse reflection spectroscopy to 3.06 eV for an indirect forbidden transition. The magnetic measurements suggest that the system reveals long–range magnetic ordering between Co (II) centers below 12 K.

Keywords: Crystal structure, binuclear cobalt, optical band gap, magnetic behavior

Rheological behavior of concentrate aqueous kaolin/magnesite suspensions

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[2)] Laboratoire Génie Physique des Hydrocarbures - Simulation et rhéologie des Fluides Complexes. Université M'Hamed Bougarab Boumerdes 35000,

The properties of porous cordierite ceramic depend firstly, on an amount of additives additional and secondly, with high concentration of ceramic particles (> 50%) which play a very important role for the phase transformation in sintering.
For this purpose, the focus of the present study is estimate the effect of additional magnesite amounts (3-15 % mass) into dispersed suspensions by optimal amount of both dispersant. The pH measurements were performed for all slurries in order to determine the most suitable amount of additives. The analysis of rheological tests led to the conclusion that an increase in the magnesite concentration above the limit 6% of saturation gave way to $Mg(SiO_3)$and $Mg_3(PO_3)_2$ soluble complexes.

The johillerite-like structure of the sodium cobalt arsenate $NaCo_4(AsO_4)_3$

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The chemical and structural investigations of the Na-Co-As-O system show that the arsenate $NaCo_4(AsO_4)_3$ exhibits two allotropic forms, synthesized by solid state reactions at different temperatures. The low-temperature form exhibit the Johillerite structure [1], as shown by Rietveld analysis of its X-ray powder data. The crystal cell is monoclinic, space group I2/a, with a=6.774(13), b=12.875(28), c=11.110(31) Å, β=100.207(3)° and Z=4. The structure was solved and refined to final agreement factors *R*p=0.011, *R*wp=0.015, *R*exp=0.014, $R(F^2)$=0.12188, χ^2=1.166. The two validation tools bond-valence-sum (BVS) and charge-distribution (CD) [2,3] are used to validate the obtained structural.

The unusual feature of the johillerite structure is the A(1)' site, a square-planar-coordinated site fully occupied by Co^{2+}.The A(2) site is similar to that in other alluaudite-group minerals, it is occupied by Na cations which are surrounded by eight O-atoms with a <A(2)–O> distance of 2.7232 Å. The octahedral sites M(1) and M(2) are reserved for CoO_6, whereas tetrahedral AsO_4 are distributed in the T(1) and T(2) sites.

The asymmetric unit is composed of a square-planar-coordinated $Co(1)O_4$, two Co(2) and Co(3) octahedron and two As(1) and As(2) tetrahedra. All cobalt polyhedra are connected by sharing edges and share vertices with As tetrahedra.

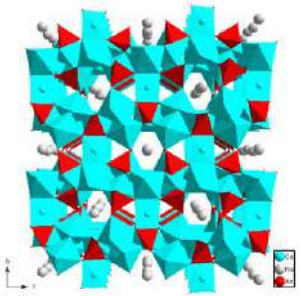

Figure 1: Projection of structure along the [100] direction.

Keywords: $NaCo_4(AsO_4)_3$, johillerite, BVS, CHARDI.

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SYNTHESIS AND STRUCTURAL STYUDY OF THE TETRAGONAL FORM OF $NaCo_4(AsO_4)_3$

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A new allotropic form of the arsenate $NaCo_4(AsO_4)_3$ has been synthesized by solid state reaction at T=1100°C and its structure determined by single-crystal X-ray diffraction. It is isostructural to $NaMg_4(VO_4)_3$ [1] with tetragonal symmetry, space group I-42d, a=6.854(2) Å, c=18.949(2) Å and Z=4. The structure was solved and refined to final agreement factors $R(F^2)$=2.20%, $Rw(F^2)$=5.60%, $S(F^2)$=1.13. The obtained structural model was supported by both bond-valence-sum (BVS) and charge-distribution (CD) validation tools [2,3].

The asymmetric unit consists of a Co_2O_{10} dimer formed by two edge-sharing CoO_6 octahedra, two independent AsO_4 tetrahedra and one Na atom. Each O atom connects two Co octahedra and one As tetrahedron (Fig. 1). The three-dimensional network encloses cavities in which Na^+ cations are located (Fig. 2). The possible motion of monovalent cations in the anionic framework is simulated by means of a generalization of the BVS model.

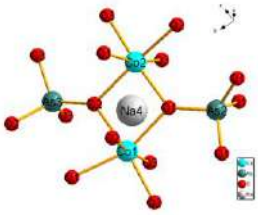

Fig. 1: The asymmetric unit of $NaCo_4(AsO_4)_3$.

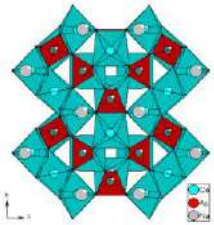

Fig. 2: Projection of structure along the [100] direction.

Key words: $NaCo_4(AsO_4)_3$, BVS, CHARDI, Ionic Conduction.

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Synthesis, Crystal structure, Thermal stability and Vibrational analysis of two hybrid compounds based on 5,5'-dimethyl-2,2'-bipyridine

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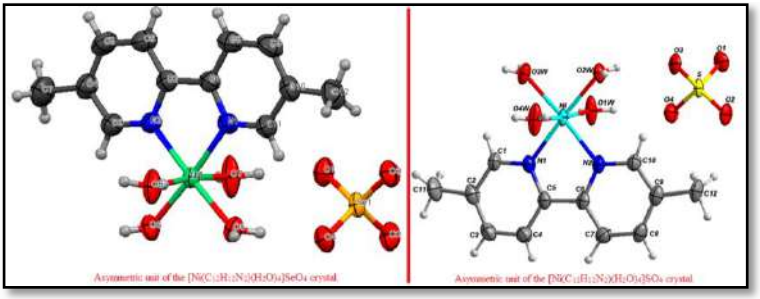
Two new Ni-based hybrid materials, $[Ni(C_{12}H_{12}N_2)(H_2O)_4]SeO_4$ (**1**) and $[Ni(C_{12}H_{12}N_2)(H_2O)_4]SO_4$ (**2**), have been synthesized by hydrothermal method and crystallographically characterized. They crystallizes isotypically in the monoclinic system, space group $\mathbf{P2_1/c}$, with the following unit-cell parameters: a = 9.6916 (2) Å, b = 9.7534 (2) Å, c = 17.8240 (4) Å, β = 93.20°, V = 1682.20 (6) $Å^3$ and Z = 4 for (**1**) and a = 9.492 (7) Å, b = 9.539 (7) Å and c = 18.411 (1), β =102.616 (1) Å, V = 1626.8 (2) $Å^3$ and Z = 4 for (**2**). Their crystal structure consist of metallic cations octahedrally coordinated by four water molecules and bidentate chelating 5,5'-dimethyle-2,2'- bipyridine ligand $[NiN_2(H_2O)_4]^{2+}$, and sulfate/selenate anions $(S/SeO_4)^{2-}$ linked together via hydrogen bonds, Ow–H···O. The IR spectroscopic study confirms the results obtained by X-Ray Diffraction. The thermogravimetric measurement indicates that the dehydration occurs in one step for (1) and in two steps for (2) by the departure of four water molecules giving rise to an anhydrous phase.



Keywords: Hydrothermal synthesis, Crystal structure; selenate/sulfate.

Nonlinear optical properties of a new centrosymmetric compound (4-Dimethylaminopyridinium nitrate gallic acid monohydrate)

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A new centrosymmetric material has been successfully synthesized using the slow evaporation method. Despite the fact that of having a centrosymmetric structure, the nonlinear optical properties of the single crystal, which was tested by Kurtz-Perry technique, proved that its second harmonic generation efficiency was 1.22 times more than that of KDP (potassium dihydrogen phosphate) single crystal. This nonlinear optical behavior of the studied compound was also determined throughthe calculations of polarizability and first hyperpolarizability values. So, we can say thatthe experimental and theorical results have shown that this material provides a high value of quadratic susceptibility and certainly having high quality single crystal with equitable thermal stability, valuable transparency and a high nonlinear optical efficiency is the best choice for the technologists. Therefore, the title compound is a suitable choice for doubling frequency.

Synthesis, rietveld refinement, vibrational and UV–visible studies of $Tl_2Mn_2(MoO_4)_3$

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The $Tl_2Mn_2(MoO_4)_3$compound has been prepared by the solid state reaction method and characterized by X-ray powder diffraction at room temperature. This compound crystallizes in the Cubic system with $P2_13$space group. Vibrational study by means of Raman and FTIR spectroscopies confirmthe existence of MoO_4^{2-} functional group. Optical properties were studiedat room temperature using UV–visible spectroscopy in the spectral range of 200–800 nm. The Kubelka–Munk function was used to determine the optical gap energy of the synthesized $Tl_2Mn_2(MoO_4)_3$compound. The dispersion parameters (E_0 and E_d) of this compound were estimated using the Wemple Di-Domenico model.

Key words: Rietveld, UV–visible spectroscopy, Urbach energy, Refractive index.

CHEMICAL SYNTHESIS, CRYSTAL STRUCTURE AND VIBRATIONAL SPECTROSCOPY OF NEW ORGANIC-INORGANIC SULFATE

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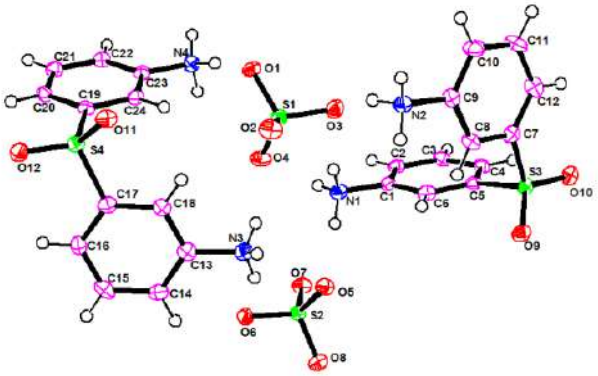
Single crystals of a new organic-inorganic sulfate were grown by slow evaporation at room temperature of a mixture of 4,4-diaminobiphenylsulfone and sulfuric acid dissolved respectively in ethanol and water. The synthesized compound crystallize in the monoclinic system $P2_1/c$ space group with the following parameters a = 30.1122 (18) Å; b = 11.7047 (5) Å; c = 8.0292 (3) Å; β = 96.307 (5) ° and Z = 4.This compound was characterized by infrared absorption, UV-Vis, NMR spectroscopy and crystal X-ray diffraction. The structure was solved using 5010 independent reflections and refined to the reliability factor R = 0.0572. The atomic arrangement can be described as two infinite parallel planes built by $[SO_4]^{2-}$ anions and $[C_{12}H_{14}N_2O_2S]^{2+}$ cations connected by N-H…O hydrogen bonds to form a three-dimensional network. The infrared spectrum of this compound reported from 4000 to 400 cm^{-1} confirm the presence of the principals bands assigned to the internal modes of the organic cation and sulfate anion.



Asymmetric unit of $(C_{12}H_{14}N_2O_2S)(SO_4)$

Keywords: Crystal structure, IR spectroscopy, UV visible, X-ray diffraction.

Catalytic activity of Cu-Al_2O_3catalysts prepared from aluminumscraps in the selective catalytic reduction of NO by NH_3 in presence of water vapor

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In this study, we reported the preparation, the characterization and the application in the selective catalytic reduction of NO by NH_3 (NH_3-SCR) of Cu loaded η-alumina catalysts prepared from aluminum scraps. Accordingly, five catalysts with different copper amounts (1%, 2%, 3%, 5 % and 7.5 %) have been prepared by impregnation/evaporation method and calcined at 500°C for 10h. This method was used to obtain highly dispersed copper oxide species on the alumina surface which were shown to be very active in the NH_3-SCR of NO.

XRD technique showed the presence of CuO particles for higher Cu loadings (3%, 5% and 7.5 %); whereas this species were absent for lower Cu amount 1% and 2%. The nature of copper species for lower copper amounts has been determined by H_2-TPR technique. The reduction profiles showed the reduction of small CuO clusters at 125°C and a broad reduction peak in the range 300-600°C, attributed to copper aluminate. With the increase of copper loading, there is an agglomeration of the CuO particles and reduction profiles showed broad peaks around 260°C associated to big sized CuO particles.

The catalytic tests were carried out in a temperature programmed surface reaction (TPSR) from 180 to 550 °C at 10°C/ min in the presence of water vapor. The following gas composition (1000 ppm NO, 1000 ppm NH_3, 8% O_2 and 2.5% H_2O balanced with Helium) was used for NH_3-SCR of NO. The outlet gases from the reactor were analyzed continuously by sampling on line to quadruple mass spectrometer (Pfeiffer Omnistar).The introduction of copper to η-Al_2O_3, which is inactive for NO reduction, produces marked changes in the catalytic behavior. For Cu(x)-η-Al_2O_3 (xis the Cu amount)catalysts, the NO conversion increases with the temperature, reaches a maximum and then decreases more slowly. This behavior reveals a competition between two reactions. The first correlated to NO reduction to N_2 and the second to NH_3 oxidation into NO or/and N_2O by the oxygen present in the reactionmixture as shown in reaction (1), (2) and (3).

$$4NH_3 + 3O_2 \rightarrow 2\,N_2 + 6H_2O \quad (1)$$
$$2NH_3 + 2O_2 \rightarrow N_2O + 3H_2O \quad (2)$$
$$4NO + 4NH_3 + O_2 \rightarrow 4\,N_2O + 6H_2O \quad (3)$$

It was shown that Cu(1%)-η-Al_2O_3 and Cu(2%) η-Al_2O_3catalysts were the most active in the NH_3-SCR of NO in the presence of water vapor. For the Cu(2%) η-Al_2O_3catalyst, the NO conversion is about 12% at 275 °C, reached 86 % at 475 °C and then decreased progressively to 65% at 550 °C. On the other hand, Cu(2%)-η-Al_2O_3catalyst showed about100% N_2 selectivity; while Cu(1%)- η-Al_2O_3 catalyst was N_2O selective at temperature higher than 500°C.

Development of New modified Cyclodextrins for pharmaceutical nanoformulation

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Jose Ignacio Miranda[3], Jesús M. Aizpurua[3]

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Cyclodextrin derivatives are known to be efficient delivery systems with low toxicity. In this study, a nanoformulation of bioactive molecules (baclofene®, synthesis aziridine and active propolis) with amphiphilic cyclodextrin (β-CD-amph) was achieved from native β-cyclodextrin (β-CD) via the simple co-precipitation method. It was of interest to find out a model of molecule derivatives that would be sufficiently water-stable and form a stable complex with (β-CD-amph) in aqueous medium, so that it could be used as a reference in future formulations or vectorization work[1]. Among the nanoformulation, NMR studies of the inclusion complex of this derivative with β -cyclodextrin provided useful parameters related to the stoichiometry of the complex and the association constant Ka[2]. The geometry of the complex was assessed by 2D-ROESY experiments, suggesting a deep insertion of the guest into the cavity of (β-CD-amph)[3].

Keywords: Nanoformulation, β-cyclodextrin, inclusion complex, NMR.

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Electrochemical and optical properties of Fe_2O_3/TiO_2, Fe_2O_3/ZnO and Fe_2O_3/SnO_2 nano-heterojunctions

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Fe_2O_3/TiO_2, Fe_2O_3/ZnO and Fe_2O_3/SnO_2 hetero-nanostructures were fabricated via a simple hydrothermal method at 150 °C during 5 hours. The phase structures, morphologies, chemical compositions of the composites have been characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) photoluminescence spectroscopy (PL) and ultraviolet-visible (UV-vis) diffuse reflectance spectroscopy. The results confirm the nanostructure characters of the compounds. The microstructure the as-prepared composites reveal that the Fe_2O_3 nano particles are uniformly dispersed on the surfaces of TiO_2, ZnO and SnO_2 oxides. It also indicates that after addition of Fe_2O_3 on the oxide's surface, the absorption is induced towards the visible light range. The optical gap bands of the nano-composites Fe_2O_3/TiO_2, Fe_2O_3/ZnO and Fe_2O_3/SnO_2 were found to decrease respectively compared to the TiO_2, ZnO and SnO_2. This study offers a simple method for preparing the nano composites, which will be a choice to greatly expand potential applications in the treatment of water pollution, pollutant degradation and other related fields.
The study by cyclic voltammetry shows that the compounds exhibit excellent electrochemical performances for the Na-system and presents an application project in the development of high-power anode materials for the Na-ion battery.

Keywords: Nano composites, optical, electrochemical

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Quantum study of zinc phthalocyanine by DFT method with deferent functional

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In this work, we realized a theoretical study at the structural, vibratory and electronic level of zinc phthalocyanine. Based on the DFT calculation method ("density functional theory") with the combination of the two bases, one base used for metal and the other base used for ligand and functional B97D3, B3LYP, M062X, PBE1PBE and BMK, we determined the geometrical parameters, the vibratory frequencies and the UV-visible of zinc phthalocyanine .

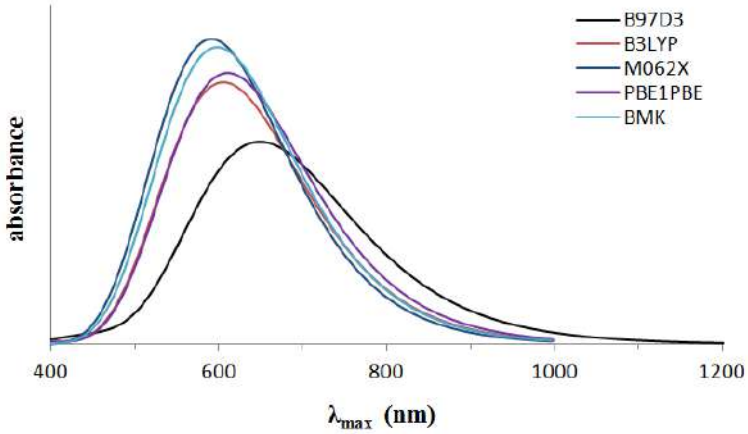

Figure1: UV-visible absorption of ZnPc calculated by various functional

Keywords: DFT, theoretical study, ZnPc.

Synthesis, crystal structure and magnetic properties of 1-(2,5-dimethylphenyl)piperazine-1,4-dium terachloridocuprate(II)

L. Khediri, M. Rzaigui, C. Ben Nasr

Laboratoire de Chimie des Matériaux, Faculté des Sciences de Bizerte, 7021 Zarzouna, Université de Carthage, Tunisie.

The synthesis and the physico-chemical characterization of a novel organic-inorganic hybrid material, 1-(2,5-dimethylphenyl)piperazine-1,4-dium tetrachloridocuprate have been reported. This compound crystallizes in the monoclinic system with the space group $P2_1/n$ and cell parameters a = 9.833(3), b = 16.337(2), c = 10.452(3) Å, β= 102.13(4), Z = 4 and V =1641.5(7) $Å^3$. In the title salt, $(C_{12}H_{20}N_2)[CuCl_4]$, the geometry of the $CuCl_4^{2-}$ ions is intermediate between tetrahedral and square planar. In the atomic arrangement, tetrachlorocuprate anions and the piperazine-1,4-dium cations are held together via N-H…Cl and C-H…Cl **(Fig. 1)** to give a 1-D hybrid chain running along the *a*-axis **(Fig. 2)**. The six-membered piperazinedium ring adopts a chair conformation. The vibrational absorption bands were identified by infrared spectroscopy. Magnetic measurements reveal that moderate antiferromagnetic (AF) Cu–Cu interactions predominate in the title compound.

Keywords: Cu(II) complex; X-ray diffraction; FT-IR; Magnetic properties.

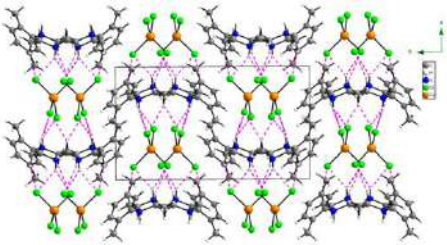

Fig. 2: Projection along the *c*-axis of the crystal packing of the title compound. The dotted lines indicate hydrogen bonds.

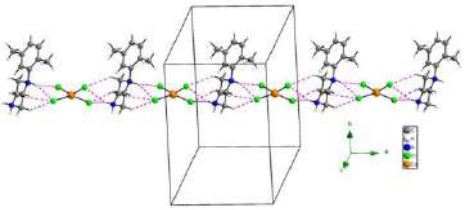

Fig. 1: A view of 1-D hybrid chain running in $(C_{12}H_{20}N_2)[CuCl_4]$.

The structural properties of the nanostructured Fe-based alloy prepared by mechanical alloying

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Nanocrystalline $Fe_{50}Co_{25}Ni_{15}B_{10}$ powder mixture was prepared by mechanical alloying (MA) in a high-energy planetary ball-mill under argon atmosphere. Structural and morphological properties and thermal stability changes as a function of milling times were followed by X-ray diffraction (DRX), scanning electron microscopy (SEM) and differential scanning calorimetry (DSC), respectively. The final products of the MA process achieved after 100 h milling were the nanocrystalline Fe(Co,Ni) and FeB_3 within a partially amorphous matrix. The nanostructure formation was commonly attributed to the generation and movement of dislocations defects. A decrease in Ms values can be attributed to the modification of magnetization of the elements and their changes in their configurations during milling by the presence of crystalline defects. Recovery, recrystallization and ferro-paramagnetic transition at Curie temperature were revealed in the thermal analysis in the temperature range 35–700 °C. The microstructural evaluation was used to understand physical properties of nanocrystalline $Fe_{50}Co_{25}Ni_{15}B_{10}$.

Keywords: FeCoNi alloy; High energy ball milling; X-ray diffraction; Nanostructure; Thermal stability.

Synthesis and characterization of new organometallic complexes for Nonlinear Optical Applications

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The ruthenium(II) acetylides containing an azobenzene moiety in the main π-conjugated system can constitute important components of molecular materials for electrooptic applications and for formation of surface relief gratings (SRGs) for optical information storage and photonic devices. Azobenzene dyes form a class of molecules currently, widely investigated because of their optical properties.

Novel photoresponsive organometallic complexes based on ruthenium(II) σ-acetylides coupled to an azobenzene moiety in the main π -conjugated chain have been synthesized and have been characterized using elemental analyses, infrared, ^{1}H, ^{13}C NMR, ultraviolet-visible spectrometer and mass spectrometry .

The photoinduced trans→cis isomerization and the thermal cis→trans back reisomerization of these compounds were studied in solution. These materiels show satisfactory processability and give rise to spin-coated uniform thin films. Formation of surface relief gratings on their amorphous thin films using a picosecond pulsed laser at 532 nm results in instantaneous inscription: saturation of the first order diffraction efficiency and of the modulation amplitude of gratings were obtained in less than 1 s, while the orientation of these azodyes remains unchanged for up to 6 months.

Key words: Organometallics, ruthenium, azobenzene, nonlinear optics.

Synthesis and characterization of new inorganic solid catalysts for decomposition of hydrogen peroxide

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In this study, we report the synthesis of oximato-nickel complexes by adopting the hydrothermal method. This process is rarely used for the preparation of coordination compounds.
New binuclear complexes of nickel were isolated and were thoroughly characterized by elemental analyses and the commonly used spectral techniques: EPR investigation, Infrared spectroscopy (IR) and nuclear magnetic resonances (^{1}H and ^{13}C).
Decomposition of hydrogen peroxide catalyzed by nickel complexes as heterogeneous catalysis has been investigated by measuring the evolution of oxygen at different time intervals 35-60^{0}C . The effect of variation in the concentration of catalysts on the extent of disproportionation has also been studied. The reaction rate is found to increase with increase in temperature and quality of the catalyst.

Keywords: Complexes of nickel, decomposition, hydrogen peroxide.

A Hirshfeld Surface Analysis, Crystal Structure and Physicochemical studies of a New Cd(II) complex with 2-amino-4-methylpyrimidine ligand

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Coordination polymers architectures formed by metal-organic moieties have attracted considerable attention during the past decade for a variety of topologies as well as potential applications [1,2]. A new Cd(II) complex with the monodentate ligand 2-amino-4-methylpyrimidine, $[Cd(NO_3)_2(C_5N_3H_7)(H_2O)_2]$, has been prepared and characterized by single crystal X-ray diffraction, elemental analysis, CP–MAS NMR and IR spectroscopy. The title compound crystallizes in the monoclinic space group $P2_1/c$ with a = 10.3495 (7) Å; b =11.9110 (8) Å; c = 7.5919 (5) Å; β = 104.981° (3) and Z = 2. The basic coordination patterns of the 2-amino-4-methylpyrimidine coordinated metal cations are slightly distorted octahedra in this compound. The crystal structure is characterized by CdN_2O_4 octahedra interconnected via O–H…O and O–H…N hydrogen bonds to form layers parallel to the (**b**, **c**) plane. The crystal structure is stabilized by sets of hydrogen bonds, one of which is trifurcated. Intermolecular interactions were investigated by Hirshfeld surfaces. The ^{13}C and ^{15}N CP–MAS NMR spectra are discussed and the vibrational absorption bands were identified by infrared spectroscopy. Electronic properties such as HOMO and LUMO energies were derived.

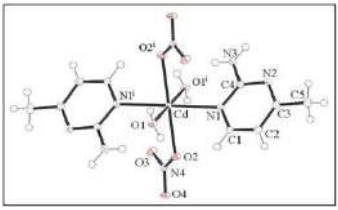

Fig. ORTEP of the title compound with displacement ellipsoids drawn at the 40% probability level. (i: −*x*+1, −*y*, −*z*.).

Keywords: Cadmium (II) complex; X-ray structure; Hirshfeld surface; contact enrichment ratio; DFT calculations.

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Valorization of ceramics contained in aluminum slag

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During the melting of Aluminum, important quantities of slag are generated. These slags contain significant quantities of aluminum oxide (Al_2O_3), together with other substances, some of which are harmful to the environment and to human life. The valorization of these slags has as aim, the recovery of the useful substances (example: Al_2O_3 ...) and the environmental protection which can be achieved by transformation of the harmful substances.
In this work, we extracted the alumina (Al_2O_3) contained in the aluminum slags by a chemical method. We started by washing slag with water. Then, we treated the washed slag with sulfuric acid (H_2SO_4). Thereafter, we heat treated the obtained powders at 1000 °C.
The obtained results show the effectiveness of the washing with water in order to eliminate the salts contained in the slag and the consequent increase in the alumina content. The treatment of slag with sulfuric acid increases the alumina and spinel ($MgAl_2O_4$) contents by dissolving the calcite ($CaCO_3$) contained therein. By the treatments applied, a relatively high level of alumina was obtained in the collected residues (~ 79.3%).

Keywords: Aluminum slags, alumina, slag treatment, acid dissolution.

Application des nanoparticules fonctionnalisées dans le domaine des biocapteurs

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Nowadays nanotechnology is creating a wealth of new materials and manufacturing possibilities, which in turn will profoundly impact our economy, our environment, and our society. The use of nanotechnology, more specifically, nanoparticles, in the field of medicine offers some exciting possibilities such us diagnosing and treating some pathologies (cancers, Alzheimer, infections…)[1][2]. In this work we report a protocol for the synthesis and the functionalization of ZnO and ZnS-$AgInS_2$ (ZAIS) nanoparticles with single stranded oligonucleotide. The binding of DNA to these particles was achieved through a covalent binding via a carbodiimide activation. The combination of the optical proprieties of those quantum dots (QDs) and DNA melting characteristics contributes to the development of a new DNA detection technologies with remarkably high selectivity and efficiency.

Keywords: nanoparticles, ZnO, ZAIS, oligonucleotide.

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Electrical and electrochemical properties of $CePO_4$ and $Ce_{0.9}Cd_{0.15}PO_4$ nanomaterials

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The inorganic phosphates are usually used in the fabrication of electrochemical, electrical and optical nanodevices [1]. For example, the cerium phosphate $CePO_4$ nanomaterials are being studied for technological applications [2]. Generally, the doping could modify or improves the compounds properties and even leads to new properties [3,4].

In this context, Synthesis of metal ions Cd^{2+}doped $CePO_4$ nanocrystals were achieved through a simple hydrothermal route at 150 °C using poly-ethylene-glycol (PEG-6000) as surfactant. The morphology (figure 1) and structure of the obtained products were characterized using X-ray diffraction and scanning electron microscopy. The Results obtained reveal that the low percentage of Cd^{2+}dopant affect the structure showing a weak decrease in the lattice parameters compared to those of $CePO_4$. UV-vis spectroscopic study showed an apparent change in the energy band gap of $CePO_4$ with the metal ions (Cd^{2+}) doping. The total electrical conductivity plots obtained from impedance spectra, between 300 and 450°C, indicated that the activation energies for $CePO_4$ and $Ce_{0.9}Cd_{0.15}PO_4$, are 1.05, and 0.98 eV, respectively, showing an increase of the total conductivity as Cd-content increases (figure 1). The partial substitution leads to the improvement capacity from 58 $F.g^{-1}$ for $CePO_4$ to 65 Fg^{-1} for $Ce_{0.9}Cd_{0.15}PO_4$ and a stable cycling performance of the nanomaterials (figure 2).

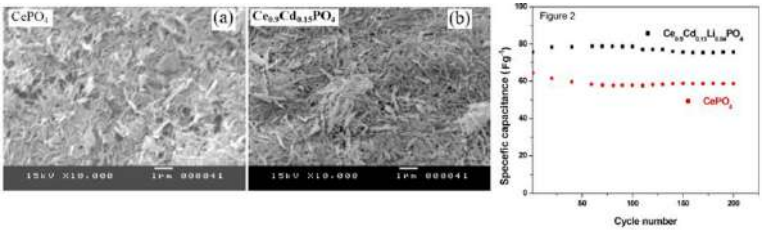

Fig.1a,b: SEM images of $CePO_4$ and $Ce_{0.9}Cd_{0.15}PO_4$ nanorods
Fig.2: $CePO_4$ and $Ce_{0.9}Cd_{0.15}PO_4$ cycling performance.

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Synthesis and structural study of $(C_8H_{12}N)_6V_{10}O_{28}.16H_2O$

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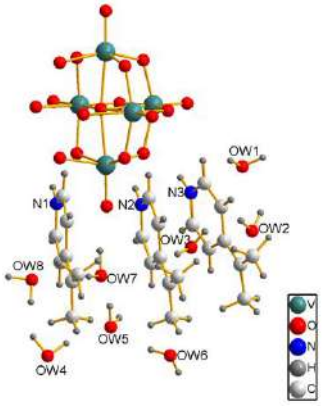
Study of polyoxometalates has been a very active research area over the past decade, mainly due to their potential applications in catalysis and material science [1].

In the present work, the synthesis and crystal structure of vanadium(V) complex, 4-isopropylpyridin-1-ium decavanadate hydrate $(C_8H_{12}N)_6V_{10}O_{28}.16H_2O$, is reported.

The compound has been prepared by slow solvent evaporation at room temperature and its structure was elucidated using single-crystal X-ray diffraction. It crystallizes in the triclinic system, $P\bar{1}$ space group with the cell parameters: a=11.258(4)Å, b=11.783(3)Å, c=15.060(2)Å, α=93.43(2)°, β=102.52(3)°, γ=100.69(5)° and volume V=1905.9(4)Å^3.

The asymmetric unit consists of one half-decavanadate anion of Ci symmetry, three organic cations and eight water molecules.



The asymmetric unit of $(C_8H_{12}N)_6V_{10}O_{28}.16H_2O$

Key words: Decavanadate, vanadium complexes, crystal structure.

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Characterization and catalytic reactivity of catalysts based on acidified mesoporous zirconia with telluric acid: Acetic acid esterification with ethanol

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Mesoporous xerogel catalyst based on acidified zirconia with telluric acid prepared by sol-gel method has been tested in esterification reaction of acetic acid with ethanol. The influence of some preparation parameters on the textural, structural and catalytic properties of the prepared catalysts was studied such us the effect of the addition of nitric acid in the gelling step, acidifying agent content and the calcination temperature. Characterization was made by N_2 physisorption at 77K, X-ray Diffraction, FTIR spectroscopy and adsorption of Hammett's indicators.

The addition of nitric acid in the gelling step causes the development of texture and the generation of acidic sites on the surface of catalyst, thus enhances acetic acid conversion. The doping of the zirconia with telluric acid causes a slight lowering of the specific surface, a reduction of the pore size and the pore volume. Furthermore, the calcination catalyst gives rise to the falling of the specific surface, the development of the tetragonal ZrO_2 phase and makes lose of the dopant, therefore it reduces the acidity and reactivity of the surface of the catalyst.

Key words: Xerogel; zirconia; telluric acid; acetic acid esterification.

Synthesis, Structural characterization, Hirshfeld surface and Antioxidant activity analysis of a novel organic cation antimonate complex

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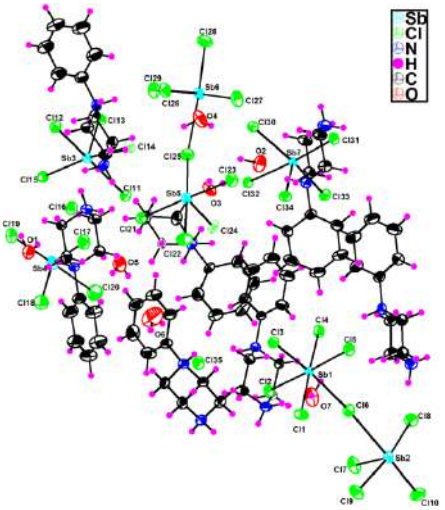
A new organic-inorganic hybrid material of formula $(C_{10}H_{15}N_2)_7$ Sb_2Cl_{10} Sb_2Cl_9 $(SbCl_5)_2$ $SbCl_4$ $2Cl.7H_2O$ was synthesized and characterized by X-Ray diffraction. It crystallizes in the triclinic system with the $P\bar{1}$ space group and the following unit cell parameters a=11.8127(3) Å, b=15.7557(4) Å, c=35.4511(8) Å, α=89.409(1)°, β=84.04(1)°, γ=71.116(1)°, Z=2 and V=6207.3(3) $Å^3$. The examination of the structure shows that the two dimensional frameworks are produced by O–H…Cl, N–H…Cl and N-H…O hydrogen bonding. In addition, the most important features of crystal packing and intermolecular interactions in the title complex were quantified via Hirshfeld surface analysis. Differential scanning calorimetry (DSC) has revealed a dehydration phenomenon at around 348 K. The investigation of the antioxidant activity of the title compound was carried out using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) and Ferrous iron chelating (FIC) methods.



Keywords: Antimonates, Crystal structure, Hirshfeld surface, Photoluminescence, Antioxidant activity.

Synthesis and magnetic characterization of Spinel ferrites MFe_2O_4 (M= Ni, Co, Zn and Cu) via chemical co-precipitation method

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Nanostructured ferrite spinels MFe_2O_4 (M= Ni, Co, Zn and Cu) were successfully synthesized via a chemical co-precipitation method. The structure, composition, optical, morphology and magnetic properties of the samples were characterised. The thermal decomposition of the MFe_2O_4 was investigated by TGA\DTA. X-ray diffraction (XRD) confirmed the formation of single phase MFe_2O_4 (M= Ni, Co, Zn and Cu) with space group Fd-3m and spinel structure and the crystallite size was found to be between 25 and 41 nm. SEM and TEM images showed cubic-like nanostructure. The Fourier Transform Infra-Red (FT-IR) spectroscopy showed absorption bands, which were assigned to the vibration of tetrahedral and octahedral complexes. Raman spectroscopy is used to verify that we have synthesized ferrite spinels and determines their phonon modes. The UV-visible study is used to calculate the band gap energies. The vibrating sample magnetometer (VSM) was used to obtain the hysteresis parameters.

Synthesis, crystal structure and characterization of a new Organic–inorganic hybrid material 4-(ammonium methyl) pipyridinium hexachloro stanate (II) trihydrate

Mohamed Saber Lassoued, [a, b, c*] Mohammed. S. M. Abdelbaky, [c] Salah Ammar,[a] Abdellatif Gadri, [a] Abdelhamid Ben Salah, [b] and Santiago García-Granda [c]

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The present paper undertakes the study of($C_6H_{16}N_2$) $SnCl_6$, $3H_2O$ which is a new hybrid compound. It was prepared and characterized by single crystal X-ray diffraction, X-ray powder, Hirshfeld surface, Spectroscopy measurement, thermal study and photoluminescence. The single crystal X-ray diffraction study revealed that the compound crystallizes in monoclinic *Cc* space group with cell parameters *a*= 8.3309(9) Å, *b*= 22.956(2) Å, *c*=9.8381(9) Å, β= 101.334(9) ° and Z=4. The atomic arrangement shows an alternation of organic and inorganic entities. The cohesion between these entities is performed via N–H…Cl, N-H…O and O–H…Cl hydrogen bonding to form a three-dimensional network. Hirshfeld surface analysis was used to investigate intermolecular interactions, as well 2D finger plots were conducted to reveal the contribution of these interactions in the crystal structure quantitatively. The X-ray powder diffraction analysis is in agreement with the single crystal X-ray structure results. Scanning electronic microscopy (SEM) was carried out. Furthermore, the room temperature infrared (IR) spectrum of the title compound was recorded and analyzed on the basis of data found in the literature. Solid state ^{13}C NMR spectrum shows four signals, confirming the solid state structure determined by X-ray diffraction. Besides, the thermal analysis studies were performed, but no phase transition was found in the temperature range between 30 and 450 °C. The optical and PL properties of the compound were investigated in the solid state at room temperature and exhibited three bands at 348 and 401cm^{-1} and a strong fluorescence at 440 nm.

Synthesis, characterization and electrical properties of a novel biological V-substituted polyoxotungstates

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A new V-substituted polyoxotungstates based on V-substituted Lindqvist-type $[V_2W_4O_{19}]^{4-}$ and piperazinium (H2pip) cations $(H2pip)_2[V_2W_4O_{19}].2H_2O$ was synthesized and characterized by powder and single-crystal XRD methods, ICP elemental analysis, IR, UV-Vis and photoluminescence spectroscopy measurements. X-Ray diffraction studies reveal that the titled compound crystallizes in the orthorhombic space group Pnmn with a = 8.931 (2), b = 14.287 (2) Å, c = 10.737 (3) Å. In its crystal packing Two crystallographically independent water molecules (O1W and O2W) generate with related symmetry a based H-bond tetramer (O1W…(O2W)2…O1W).These water tetramers and $[V_2W_4O_{19}]^{4-}$ cluster anions are extensively involved hydrogen bonding interactions to form a three-dimensional porous inorganic framework, wherein the guest cations reside in the channel voids. Moreover, the biological properties of **1** were also carried out, showing a promising enhancement of biological activity of reported material in comparison with its parent reactants. Furthermore, the conductivity and dielectric behavior of both compounds was studied as a function of temperature and frequency showing that the activation energy responsible for relaxation was evaluated and found to be close to that of the conductivity.

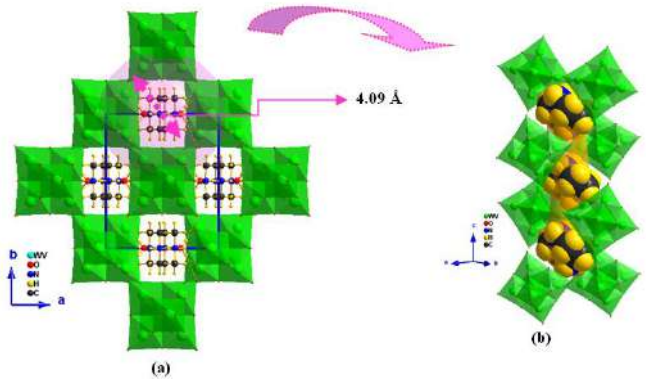

Fig. (a) Crystal packing of structure **1** down *c* -axis evidencing the porous architechture filled by organic moities. **(b)** Perspective view of channel voids extending along b -direction in structure **1**.

Structural and spectroscopic properties of terbium complex with thiosemicarbazone

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The significance of semicarbazone and thiosemicarbazone and their metal complexes, apart from their diverse chemical and structural characteristics, stems from their potential application as biologically active molecules. In this study, we report and discuss the comprehensive results of vibrational and optical research performed for Tb^{3+} complex with thiosemicarbazone novel ligand (N'-[2,4-dihydroxyphenyl)methylidene]-4-(4-fluorophenyl)piperazine-1-carbothiohydrazide). A brief description of synthesis and spectroscopy of this complex is given. Infrared and Raman spectroscopy were used as a sensitive technique for determination of local structure. The problem to be solved is, which mechanism is responsible for the ligand-to-metal energy transfer after femtosecond laser excitation.

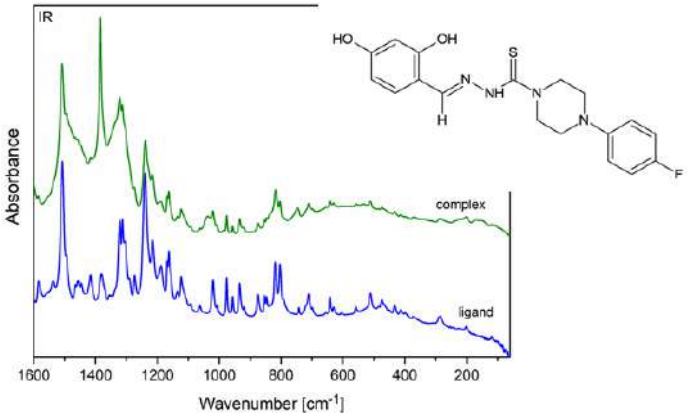

Fig. 1. IR spectra of the ligand and Tb/complex.

Acknowledgement: The present work was supported by Polish National Centre of Science under grant No. 2014/15/B/ST5/04730

Synthesis Polyol and structural characterisation of Nano crystals of Ni-Zn-Cu ferrite with spinel structure

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The physical and chemical properties of spinel nanoparticles are greatly affected by synthesis route. For this reason, various methods have been reported in the literature for the preparation of these Nano scale spinel particles such as hydrothermal technique, Sol Gel,…[1]

The present work deals with the elaboration of $Ni_{0.4}Zn_{0.4}Cu_{0.2}$ ferrites by chemical method named "polyol", the polyol process was described as a novel route for preparing finely divided metal powders of easily reducible metals such as copper, noble metals and their alloys by reduction of inorganic precursors in liquid polyols.

The X-ray diffraction patterns of the synthesized ferrite nanocrystals confirm the formation of spinel cubic structure with the Fd3m space group. The obtained powders were also investigated by Fourier transform infrared spectroscopy. Two main absorption bands (υ1 and υ2) characteristic for ferrites were observed: υ1= 587.57 cm^{-1} and υ2 = 426.94 cm-1. According to Hashim et al. [2] the band (υ_1) could be attributed to the stretching vibration of Fe^{3+}- O^{2-} in the tetrahedral complexes and the band (υ_2) could be associated to the bending vibrations in octahedral complexes. The interpretation of the experimental curves DSC has been advanced in the temperature range 40-140 ° C. The polyol method is efficient for obtaining ferrite Ni0.4Zn0.4Cu0.2 Fe_2O_4 with the ethylene glycol solvent.

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FROM T (*Bmab*) → Pseudo-S (*A*2/*m*) → T' (*I*4/*mmm*) TO T-$La_{1.8}Nd_{0.2}CuO_{4-\delta}$ USING CaH_2 AS REDUCTANT VIA LOW TEMPERATURE TOPOTACTIC REACTION

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c) Institut de Chimie Moléculaire et des Matériaux-UMR 5253, C2M: Chimie et Cristallochimie des Matériaux, Université de Montpellier 2, 5 Place Eugène Bataillon, Bat 15, CC1504, F-34095 Montpellier, France

La_2CuO_4 is known to crystallize in the so-called "T-phase" with K_2NiF_4-type structure, which is usually obtained by classic high temperature synthesis. As reported recently, La_2CuO_4 exists in a second polymorphic phase, adopting the so-called T′-structure (Nd_2CuO_4-type)[1,2]. T′-La_2CuO_4 shows a 6 % higher volume compared to T-La_2CuO_4 and its synthesis via the molten salt technique thus seems somehow unusual. The present paper lies in this context to deal with the synthesis of T-$La_{1.8}Nd_{0.2}CuO_4$. This phase is crystallized in the space group *Bmab*. Structure analyses were carried out by X-ray diffraction on a Bruker AXS D8 Advance diffractometer using Cu Kα radiation at the ILL Grenoble. T-$La_{1.8}Nd_{0.2}CuO_4$ can be transformed into T′-$La_{1.8}Nd_{0.2}CuO_4$ via a two-step topotactic reaction mechanism. First T-$La_{1.8}Nd_{0.2}CuO_4$ is reduced by CaH_2 at 200 °C to $La_{1.8}Nd_{0.2}CuO_{3.5}$ which can be subsequently reoxidized to T′-$La_{1.8}Nd_{0.2}CuO_4$ at 300 °C in air[3]. $La_{1.8}Nd_{0.2}CuO_{3.5}$ does not adopt the Sr_2CuO_3 type "S-phase" structure, as taken for granted for almost 20 years, but constitutes an oxygen deficient T′-framework, with copper in 4-fold planar and 2-fold linear dumbbell coordination. Upon heating the T- $La_{1.8}Nd_{0.2}CuO_4$ phase is reobtained from T′-$La_{1.8}Nd_{0.2}CuO_4$ above 650 °C, confirming the "T-phase" to be the thermodynamically stable modification at least at higher temperature.

Keywords: Lanthanum copper oxide, Calcium hydride reduction, Nonstoichiometric oxides, Topotactic reaction mechanism, X-ray diffraction.

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CRYSTAL STRUCTURE, VIBRATIONAL AND MAGNETIC PROPERTIES OF THE DIHYDRATED COBALT (II) COMPLEX WITH THE BIS (2,6-DI-METHYL-ANILINIUM) CATION, $(C_8H_{12}N)_2[COCl_4].2H_2O$

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The new complex of Co(II) with 2,6-dimethylaniline, $(C_8H_{12}N)_2[CoCl_4].2H_2O$ (I), has been prepared and characterized by various physicochemical studies. This compound crystallizes in the monoclinic system, with the space group P21/c and the following cell parameters: a = 8.2853 Å, b = 38.3127 Å, c = 7.3827 Å, β = 113.023 ° and Z = 4. The crystal structure was solved and refined to R = 0.0456 using 4564 reflections. The atomic arrangement can be described by inorganic layers built by $CoCl_4^{2-}$anions and H_2O water molecules where the organic molecules perform different interactions to form a stable 3D network. The compound is characterized with a single-crystal X-ray diffraction, Hirshfeld surface analysis, magnetic susceptibility, IR and UV–vis spectroscopies, fluorescence and differential scanning calorimetric study. Magnetization was used to investigate the magnetic properties. This compound exhibits an antiferromagnetic to paramagnetic phase transition at a temperature (TN) lower than 2 K. The values of paramagnetic Curie–Weiss temperature (θcw) emphasize the existence of an antiferromagnetic interaction between the neighboring cobalt ions. The biological activity of this material has been evaluated in vitro for antibacterial activity.

Keywords: Crystal structure, Spectroscopies, Magnetic properties, antibactirial activity, Hirshfeld surface analysis.

Synthesis and structural studies of (morpholine) [5,10,15,20-tetrachlorophenyl)porphyrinato]cadmium(II) complex in solution and solid states.

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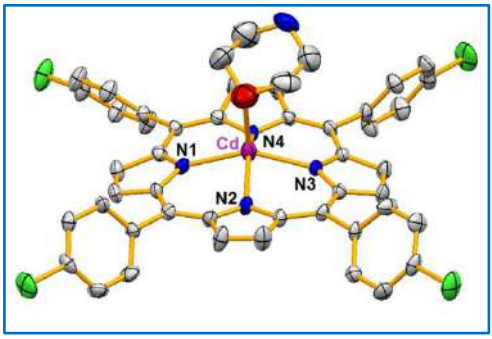
Porphyrins occupy a prominent role both in chemistry and biology. Indeed they are present as ligands in many molecules active in different fundamental biological processes. Furthermore, due to their rigid and highly stable macrocyclic structure, porphyrins constitute a unique class of ligands, able to provide distinctive chemical characteristics to a coordinated metal.

The structural diversity of cadmium porphyrins opens up high opportunities for application in various fields. In fact, the physical and chemical properties of cadmium porphyrins have gained our interest because cadmium ion is widely used in a variety of industrial processes including batteries, alloy and coloring matters as well as electroplating products.

The aim of the present work concerns the synthesis of (morpholine) [5,10,15,20-tetrachlorophenyl)porphyrinato]cadmium (II) complex, named [Cd^{II}(TClPP)(4,4'-mda)] and their structural and photophysical studies in solution and solid states by using X-ray diffraction,UV-visible, fluorescence, ^{1}H NMR, and IR techniques.



ORTEP drawing of the structure of the [Cd^{II}(TClPP)(morpholine)] complex.

Capacitive Screen Printed Touchpad

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A novel transparent touch sensor was fabricated by a printed capacitive matrix with silver conductive ink.
The printing of the matrix was printed in both sides of a C2MIMNTF2 (10%)/PVDF composite [1] by screen printing and curing the silver ink, in order to develop the conductive patterns. Thus, a high efficiency and low cost production process was implemented for the fabrication of capacitive touchscreen.
The screen printing method and opaque silver ink showed to be a viable procedure to fabricate touch screens based on capacitive touch surfaces with the corresponding reading electronics.
This platform can be applied in relevant industries due to its simple design, low cost, robustness and good performance.

Key words: Capacitive touch screen, sensor, active layer…

References

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Constant Property Newtonian Fluid Flow and Heat Transfer over Cascaded Fins

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Turbulent flow and heat transfer characteristics were studied and analyzed numerically for a constant property Newtonian fluid flowing through a two-dimensional horizontal rectangular cross section channel with staggered, transverse cascaded rectangular-triangular fins (CRTFs) and a constant temperature along both walls. The governing equations based on k-ε model used to describe the turbulence phenomenon, are solved by the finite volume method using the SIMPLEC-algorithm. Computations were carried out in the fully-developed regime for different Reynolds numbers, and geometric locations. In particular, velocity and temperature fields, skin friction coefficient, and local and average Nusselt numbers were obtained. This study can be a real application in the field of heat exchangers and air plane solar collectors.

Key words: Cascaded fin - Heat transfer - Numerical simulation - Rectangular channel.

MODELING OF THE PLANETARY BALL MILLING PROCESS FOR THE CASE OF TiC POWDER

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Stirred media milling is the main technology for producing colloidal nano-crystalline suspensions for laboratory and industrial scale. In this work we discussed the milling parameters of high-energy ball mill (Fritsch Pulverisette 7) like vial geometry, ball number and size, and speed of the mill. Simulations provide correlation between the milling parameters and the resulting microstructure of the TiC powder. A mathematical model is used to improve and develop this process.

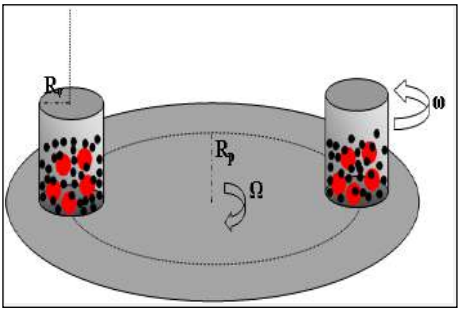

Fig. Schematic representation of a high- energy planetary ball mill.

Key words: Mechanical alloying; Planetary ball-mill; Milling energy; Modeling.

Crystal Structure and physicochemical features of a new hybrid arsenate templated by mono and di-protonated 4-aminopyridine cations: $(C_5H_7N_2)(C_5H_8N_2)[AsO_4]\cdot H_2O$

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Single crystals of a new organic–inorganic hybrid compound, with the formula (4-APH)(4-APH$_2$)[AsO$_4$]·H$_2$O, was synthesized at room temperature by slow evaporation and characterized by X-Ray diffraction at 150 K, DSC–TG measurement, FT-IR and Raman spectroscopies. The title compound, $(C_5H_7N_2)(C_5H_8N_2)[AsO_4]\cdot H_2O$, contains two 4-aminopyridinium cations, an arsenate trianion and water molecules. One of the diprotonated 4-ammoniumpyridinium dication $[C_5H_8N_2]^{2+}$ is disordered over two positions with refined site occupancies of 0.73 and 0.27 and monoprotonated 4-aminopyridinium cation $[C_5H_7N_2]^{+}$. The 4-aminopyridinium rings are essentially planar and occur in stacks along *b* axis. In the crystal, the As^{III} atom is coordinated by four O atoms in a slightly distorted tetrahedral geometry. The arsenate O atoms link the 4-aminopyridinium cations and water molecules into a three-dimensional network via intermolecular O–H···O and N–H···O hydrogen bonds. Furthermore, the room temperature IR and Raman spectra of the title compound were recorded and analyzed. On the basis of detailed vibrational studies, the detailed assignment confirms the presence of the organic groups and the anionic entities. Besides, the thermal analysis studies have been performed, but no phase transition was found in the temperature range between 298 and 625 K. Results from X-ray crystallography, Raman, IR spectroscopy and thermal analysis are combined to provide a description of the new organic arsenate monohydrate, $(C_5H_7N_2)(C_5H_8N_2)[AsO_4]\cdot H_2O$.

Key words: Organic-inorganic hybrid; X-ray diffraction; Infrared and Raman spectroscopy; Thermal studies.

Physico-chimical study of the bacterial membership (support) on surfaces modified by photopolymérisation

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Introduction

The food sector is constantly confronted with the presence of biofilms on the walls of the equipment despite the procedures for cleaning and disinfection routine. Given the complexity of the adhesion mechanism bacteria, many factors affect cell adhesion.

Methodology

- In the first phase, we proposed to prepare, bioactive surfaces madeof ultra-thinlayers of polymer PHEMA prepared on ITO –glass surfaces by olymer by photopolymerization.
- In the second part we studied the effect of hydrophobicity of *S*almonella *Typhimirium* and Bacillus cereus on modified surface substrate, using the contact angle measurement. The free energy interaction between the bacteria and the substratum treated by photopolymerization were quantified based on the thermodynamic approach.
- The impedimetriques measurements were powerful tools to evaluate the properties of the surface modified electrode, and to study the effect of this bioactive surface on the bacterial adhesion. The optic microscopy was used to evaluate the resistance of the polymer biofilm formation.

Main Results

The overall results show that photopolymerization initiated at the surface of non-toxic polymer represents a simple, effective and inexpensive method to modify the solid-liquid interface and create a physical barrier against bacterial adhesion. Our results confirm that besides the hydrophobic properties of bacteria and surfaces substrates, the biological aspects of the bacteria should also be considered by studying adhesion phenomena.

Synthesis, crystal structure and physico-chemical characterization of a new organic-inorganic material, bis(1-methylpiperazine-1,4-diium) hexachloroplumbate(II) trihydrate $[C_5H_{14}N_2]_2PbCl_6{\cdot}3H_2O$

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A new organic-inorganic hybrid metal compound, $[C_5H_{14}N_2]_2PbCl_6{\cdot}3H_2O$ was synthesized and the structure is determined by single crystal X-ray diffraction analyses. The title compound crystallizes in the monoclinic *P*2/n space group (Z = 4) and the following unit cell dimensions: *a*= 16.6066(5) Å, *b*= 8.3501(2) Å, *c*= 18.4889(7) Å, β = 115.129(4)° and *V* = 2321.14(13) Å^3. The powder X-ray diffraction and scanning electron microscopy (SEM) confirm the phase purity of the crystal sample. The examination of the structure shows a layered organization of the $(PbCl_6)^{4-}$ anions, $(NH_2)^+$ groups and water molecules developed in the *bc*-plane at x= (2n+1)/4. The crystal structure is stabilized by N−H···Cl, N−H···O, O−H···Cl, O−H···O and C−H···Cl hydrogen bonds. The intermolecular contacts are quantified using Hirshfield surfaces computational method. The major inter contatcs contributing to the Hirshfield surfaces are H···Cl, H···H and O···H. The vibrational modes were identified and attributed by IR and Raman spectroscopy. The optical proprieties were investigated by UV-visible and photoluminescence spectroscopy studies. The compound was characterized by thermal analysis to determine its thermal behavior with respect to the temperature. Finally, X-ray photoelectron spectroscopy analysis (XPS) is reported for analyzing the surface chemistry of $[C_5H_{14}N_2]_2PbCl_6{\cdot}3H_2O$.

Structural Investigation of Co-Fe-B-Ta Compound Obtained by Mechanical Alloying

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[b)] University of Burgundy , Dijon · Laboratoire Interdisciplinaire Carnot de Bourgogne (ICB).

In this study, Co-Fe-B-Ta alloy was obtained by mechanical alloying (MA) of the elemental powder mixture under argon atmosphere. The structural, microstructural and thermal properties of the obtained samples were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD) and differential scanning calorimetry (DSC).
The powders were milled for 200 h and the MAUD refinement program was used to carry out the microstructural study.
The results showed that at 15 h of the milling, microstructure consisted of nanocrystalline bcc-(Fe, Co) phase whereas the formation of Co_2Ta occurred only after 45 hours of milling. From 45 h to 105 h of milling time, we noticed a decrease of the crystal size of Co_2Ta that was eliminated after 135 h of milling, contrary to bcc-(Fe, Co) phases. However, it is noted that the main peak of the bcc- (Fe, Co) phase still persists at 200 h of the milling..
The DSC analysis of the initial powder and the milled samples for 30 h, 75 h, 135 h and 200 h revealed only exothermic peaks which can be related to the recovery of defects, recrystallization and formation of new phases.

Synthesis, crystal structure and characterizations of a new triple molybdate $Ag_{0.90}Al_{1.06}Co_{2.94}(MoO_4)_5$

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The title compound $Ag_{0.90}Al_{1.06}Co_{2.94}(MoO_4)_5$ was synthesized by solid state reaction. It crystallizes in the triclinic system, space group P-1 with the following unit cell dimensions: a=6.854(8) Å, b=6.941 (8) Å, c= 17.597 (2) (7) Å (les angles???) and V= 820.20 (2) $Å^3$. The structure can be described as a three-dimensional framework formed by M_2O_{10} dimers and M_3O_{14} units (M = Co/Al) linked to MoO_4 tetrahedra by sharing corners, with the cavities occupied by Ag^+ cations (Fig. 1).
This compound is isotypic with $NaMg_3Al(MoO_4)_5$ [1].
The structural model was confirmed by charge distribution (CD) and bond valence sum (BVS) methods. The material was characterized by Scanning electron microscope (SEM) and infrared spectroscopy.

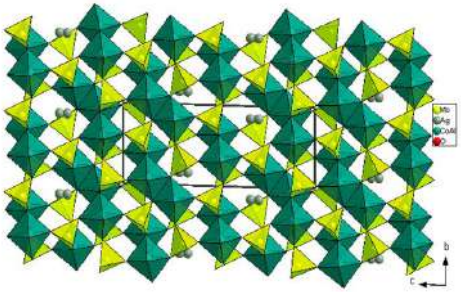

Fig. 1: Projection of the structure along direction near [100].

Key words: Crystal structure; X-ray diffraction; BVS; IR spectroscopy.

References

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ELECTROCHEMICAL PERFORMANCE OF OLYVANADATES $[NH_3(CH_2)_4NH_3][V_6O_{14}]$ PLATE-LIKE CRYSTALS

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During the past few years, a great interest in electrochemical energy storage systems attracts many researchers [1]. Some of these systems consist of transition-metal oxides that enable the access of lithium ions into their network. Among them, hybrid organic-inorganic vanadium pentoxide (V_2O_5) with a layered structure provides a lot of interlayer space, which facilitates faster mobility of the Li^+ ions [2].
The present communication deals with the electrochemical performances of polyvanadates $[NH_3(CH_2)_4NH_3][V_6O_{14}]$ plate-like crystals. Electrochemical measurements were carried out on thin films deposited on ITO modified glass electrodes and revealed that the cathodic and anodic peaks can be attributed to intercalation and deintercalation of Li^+ ion throw the material and corresponds to the reduction of V^{5+} to V^{4+} and the oxidation of V^{4+} to V^{5+}. To estimate the diffusion of Li^+, EIS measurements were carried out and the Nyquist plots, measured at 10 k mV ac voltage in the frequency range from 0.001 Hz to 100 kHz, are composed of one high-to-medium frequency semicircle, one medium-to-low frequency semicircle, and a low-frequency sloping line. The diffusion coefficient (D_{Li}) of lithium ion, deduced from the EIS plots in the low-frequency region, were found to be decreasing with increasing in Li^+ content intercalated in the material.

Key words: Layered compounds; Electrochemical performances.

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Physico-Chemical Studies of a new non-centrosymmetric organic-inorganic hybrid material: Tris (4-Chloroanilinium) Hexachlorobismuthate (III) Monohydrate

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Halogenometallate compounds are interesting systems owing to their opportunity to combine the different properties of both organic-inorganic components into one material [1]. Especially, chlorobismuthates possessing excellent tunable anionic size and electron donating ability, which can exhibit flexible photo induced electron transfer as well as photochromism with viologen acceptors [2]. As a part of our continued involvement in the investigation of hybrid compounds, a novel non-centrosymmetric organic-inorganic hybrid compound was synthesized, with the formula $(C_6H_7NCl)_3[BiCl_6].H_2O$, by slow evaporation method at room temperature. It was characterized by single crystal X-ray diffraction (SCXRD), spectroscopy measurement, thermal study and Hirshfeld surface analysis of intermolecular interactions. A preliminary SCXRD structural analysis revealed that the compound is crystallized in the orthorhombic system (space group $P2_12_12_1$) with the following unit cell parameters a = 7.3432 (1) Å, b = 13.8257 (2) Å, c = 28.2140 (5) Å with Z = 4 and V = 2864.42 (8) $Å^3$. The examination of the structure shows that its atomic arrangement can be described as an inorganic $[BiCl_6]^{3-}$ units isolated from each other by the organic cations and the co-crystallized water molecules. The cohesion between 4-dichloroanilinium cations, the $[BiCl_6]^{3-}$ anions and water molecules is performed via the N-H…Cl, O-H…Cl and C-H…Cl hydrogen bonding interaction forming a 3D network (Fig.1). The Hirshfeld surface calculations are computed around the asymmetric unit of the title compound and reveals that the H…Cl and H…H are the most frequent interactions (Fig.2).

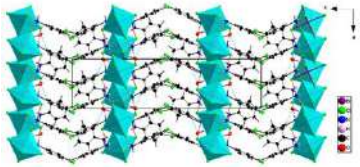

Fig. 1. Molecular arrangement of the title compound along b axis.

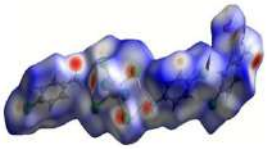

Fig. 2. Hirshfeld surface mapped with d_{norm}.

Keywords: Crystal, Hirshfeld surface, enrichment ratio, spectroscopy

References

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Synthesis and physico-chemical studies of a new hybrid compound based on polymeric chlorocadmate anions $(C_4H_{10}N)[CdCl_3]$

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Hybrid organic-inorganic materials have attracted intensive research interests due to their magnificent physical properties that take advantage of both organic and inorganic components and their promising applications in functional devices such as light emitting diodes, solar cells, and flexible thermoelectrics [1, 2]. The hybrid organic-inorganic crystal $(C_4H_{10}N)[CdCl_3]$ based on pyrrolidinium cations and chlorocadmate anions was prepared by slow evaporation at room temperature. The title compound crystallizes in the orthorhombic system with *Cmc2₁* space group and the following unit cell parameters a = 7.6410 Å, b = 17.1595 Å, c = 6.7015 Å with Z = 4 and V = 878.672 Å^3. The title compound adopts a layered structure consisting of polymeric chains of $[CdCl_6]_n$ octahedra running along the c axis on y= n/2, each of them sharing opposite faces with its two neighbors (Fig.1). The pyrrolidinium molecules are connected to these chains via N–H···Cl and C–H···Cl hydrogen bonds to form layers parallel to (b c) plane (Fig.2). In addition to the single crystal X-ray diffraction analysis, this compound has been investigated by thermogravimetric study, FT-IR spectral analysis and solid-state UV-visible spectroscopy.

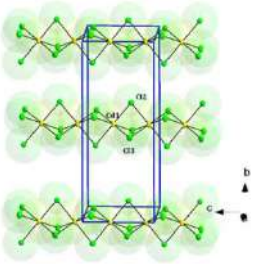

Fig.1. $[CdCl_3]_n$ polymeric chains in $(C_4H_{10}N)[CdCl_3]$.

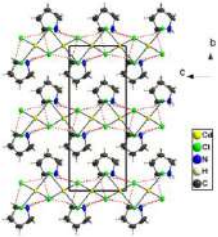

Fig.2. A view of the structure along a axis.

Keywords: Hybrid materials, crystal structure, chlorocadmate, spectroscopy

References

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High pressure phase transitions in perovskite-type metal-organic frameworks

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We present our recent high-pressure Raman, X-ray diffraction and luminescence studies of formate metal-organic frameworks (MOFs) with general formula $[A]Na_{0.5}M^{III}_{0.5}(HCOO)_3$, where A = Imi^+ (imidazolium cation) or Am^+ (ammonium cation) and M^{III} = Al, Cr. The aim of this work is understanding of the structure-properties relationship and answering the question what is the dependence between the templated protonated amine and the stability of synthesized MOF. We employed the X-ray diffraction method and high pressure Raman spectroscopy, therefore they are very sensitive probes for monitoring dynamics of ions, molecules and local structure as well. We discuss the main factors that are responsible for occurring high pressure phase transitions, including the size of the templated amine, the strength and symmetry of created hydrogen bonds and the flexibility of metal-formate frameworks. We also compare our results with the temperature-dependent Raman and IR studies showing the absence of structural phase transitions in this family of compounds.

Key words: Raman, X-ray diffraction, high pressure, phase transition

Electrochemical study of N-benzimidazole-O-acetyl-chitosan

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b) Equipes de Physique et Chimie des Polymères, Université de Pau et des Pays de l'Adour, Pau, France

Cyclic voltammetry study of N-benzimidazole-O-acetyl-chitosan in acetonitrile solution reveals its anodic oxidation at a platinum disk and a progressive growing of a thin film, through the cycling of potential.

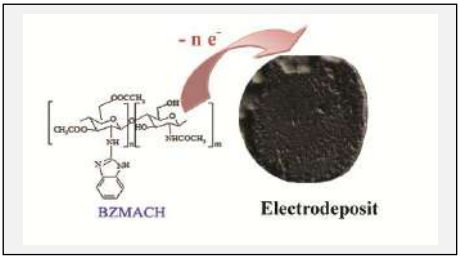

Fig. Electrodeposit of N-benzimidazole-O-acetyl-chitosan

Key words: N-Benzimidazole-O-acetyl-chitosan; Cyclic voltammetry; Coating electrode.

Electrosynthesis and characterization of oxidation products of chitin

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By means of macroscale electrolysis of chitin at platinum grid, in separated cell (El1) and non-separated cell (El2), we highlight the formation of electroactive deposits. The oxidation products present new electrochemical and physico-chemical properties, from which the solubility is more important than this of the parent compound.

Key words: Electrolysis; oxidation product of chitin; characterization

DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBS) AND ORGANOCHLORINE PESTICIDES (OCPS) IN SURFACE SEDIMENTS FROM THE GHAR EL MELH LAGOON, TUNISIA

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The aim of this study is to measure the concentrations of polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs) in 25 surface sediment samples collected from Ghar El Melh lagoon, North of Tunisia. The analytical procedure involved the application of microwave extraction, followed by purification by adsorption chromatography on a silica column. Finally, the gas chromatographic analyzes were carried out on a capillary column with an electron capture detector (GC-ECD-ECD). The total concentrations of eight PCBs (∑PCBs) and of eight OCPs (∑OCPs) in the sediments from this area ranged from 0.7 to 2.9 ng g^{-1} and from 0.7 to 2.37 ng g^{-1}), respectively. Compositional analyses of the POPs indicated that PCB 153, 138 and 180 were the predominant congeners accounting for 60 % of the total PCBs. In addition, p,p′-DDT was found to be the dominant DDTs, demonstrating recent inputs in the environment. Compared with some other regions of the world, the Bizerte lagoon exhibited low levels of PCBs and moderate levels of HCB and DDTs.

Keywords: PCB, OCP, sediments, microwave extraction, GC-ECD-ECD.

Asymmetric Reduction of Ketones by Ruthenium Catalysis under mild and eco-friendly conditions

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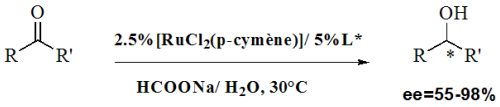
Nowadays a major concern for chemists is the design of new low-cost and highly sustainable methodologies following green chemistry principles [1]. The use of water as solvent allows one to fulfill most of these requirements and catalysis in water represents a major area [2]. Asymmetric transfer hydrogenation in water is an alternative method to perform reactions under mild conditions with high enantioselectivities and have been widely employed for the preparation of chiral alcohols. In this area we have studied catalysts for ATH reduction of ketones in water and reported on ruthenium-based catalysts which are reusable both with the same substrate or changing the ketone for each reduction without a loss of enantioselectivity [3]. Various ligands for the ruthenium-catalyzed enantioselective reduction of ketones in water have been investigated. Variety of heterocyclic aromatic ketones was reduced with ruthenium catalysis as in aqueous media. Prochiral ketones containing furan, thiophene, chroman, and thiochroman moieties are reduced with up to 98% ee. High enantioselectivities have been observed especially for the reduction of tetralone and thiochromanone with respectively 94% and 98% ee. These chiral benzylic alcohols are used as synthon-key in various syntheses of the many drugs. In conclusion, reduction catalyzed by ruthenium complex provides an attractive approach to access enantio enriched alcohols under mild and eco-friendly conditions.



Keywords: heteroaryl alcohols,ruthenium, enantioselctive reduction

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Tetrakis(μ-2-phenylacetato-κ2O:O′) bis{(DMSO κO′)]copper(II)}(Cu-Cu)]

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Carboxylate groups may interact as bridging ligands with divalent transition metals present in biological environments, thereby altering the bioavailability of drugs. Moreover, it is well known that many complexes of divalent transition metals are capable of catalyzing the hydrolysis of RNA (Stem et al., 1990; Kimura, 1994). The coordination chemistry of Cu2+ complexes bridged by phenylacetate has been reported. We have found only two reports of a dinuclear Co2+ complexes, namely tetrakis (phenylacetato)bis[(quinoline-N)-cobalt(II)](Cui et al.,1999),μ-aqua-κ2O:O-di-μ-phenylacetato-κ4O:O′-bis[(1,10-phenanthroline-κ2N,N′) (phenyl acetato-κO)cobalt(II)](Kong et al., 2005) and dinuclear Cu2+ complex, namely tetrakis (phenylacetato)bis-[(N,N-dimethylformamide)copper(II)], in which all phenylacetate groups are in bidendate bridging modes. In this presentation, the crystal structure of a new dimeric complex obtained by reaction of phenylacetic acid with copper(II) acetate is described. Each Cu(II) atom is six-coordinated by five O atoms from carboxylate groups of the phenylacetate and DMSO ligands and is completed by a Cu-Cu bond in a strongly distorted octahedral coordination, in which an inversion center is located at the mid-point of the Cu-Cu bond with a Cu…Cu distance of 2.6321(4) Å. This is longer than the 2.251(2)Å distance found in the polymeric complex [Cu2(C8H7O2)4]n. However, it is similar to the 2.6414(8) Å and 2.6261(8)Å distances found in the complex [Cu2(C8H7O2)4(C3H7NO)2] (Kong et al., 2005). The Cu-O phenylacetate bond length lies in the range 1.9644(14) to 1.9734 (14) Å and the Cu-ODMSO bond length is 2.1319(13) Å.

Keywords: Carboxylate ligands, Transition metal, Single-crystal X-ray-study

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Phase transition from disordered high-temperature to twinned low-temperature structure of tris(ethylenediamine)zinc(II) selenate

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The tris(ethylendiamine)Zinc(II) selenate has been successfully synthesized and crystallographically characterized. The title compound crystallizes at room temperature in the non centrosymmetric space groupe P31c with the following unit cell parameters: a = b = 9.1137(1) Å; c = 9.8369(2) Å; $\alpha = \beta = 90°$; $\gamma = 120°$; V = 707.58(2) $Å^3$ and Z = 2. Its crystal structure consist zinc(II) atom coordinated by three ethylenediamine molecules in octahedral geometry and disordered selenate anion linked together by hydrogen bonds only. The title complex undergoes a reversible phase transition at about 230 K detected by DSC. Below this temperature, it becomes fully ordered and crystallizes in the non centrosymmetric space group P3 with the following unit cell parameters: a = b = 9.0260(2) Å; c = 9.9010(2) Å; $\alpha = \beta = 90°$; $\gamma = 120°$; V = 698.55(3) Å and Z = 2. The twinned low-temperature structure is built from $[Zn(en)_3]^{2+}$ cations and ordered $(SeO_4)^{2-}$ anions linked by N-H…O hydrogen bonds.

Key words: Hybrid material, non centrosymmetric complex, order-disorder phase transition.

Effect of $SrTiO_3$ doping on the microstructure and the dielectric properties of $Ca_{0.9}Sr_{0.1}Cu_{0.9}Mg_{0.1}Ti_4O_{12}$ ceramics

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In this work, $Ca_{0.9}Sr_{0.1}Cu_{0.9}Mg_{0.1}Ti4O_{12}$ ─ x $SrTiO_3$ (STO) (x = 0, 1, 2, and 3 vol. %) nanocomposites were prepared by a conventional solid state method. The influence of STO on the microstructure and dielectric properties of $Ca_{0.9}Sr_{0.1}Cu_{0.9}Mg_{0.1}Ti4O_{12}$ ceramics has been investigated using SEM and dielectric spectrum measurements. The SEM results show that the grain morphology varies significantly with increasing of STO content. The impedance spectroscopy revealed that addition of 1 vol. % STO can improve the dielectric constant and minimize the dielectric losses.

Synthesis, structural characterization, spectroscopic, thermal, dielectric and Hirshfeld surface analysis of 1-(2-methoxyphenyl) piperazinuim chloranilate

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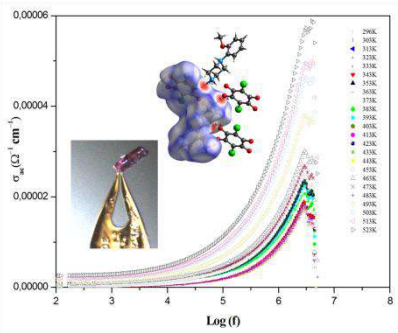
The new compound 1-(2-methoxyphenyl)piperazinium chloranilate (MPP.CA) was synthesized and studied by the single crystal X-ray diffraction method. Its structure was confirmed by infrared spectroscopy. The crystal structure consists of ribbons of chloranilate anions and 1-(2-methoxyphenyl)piperazinium cations linked together by NH…O hydrogen bonds. Two protons are transferred from a chloranilic acid molecule to the nitrogen of the piperazine in this structure. Measurements of AC conductivity as a function of frequency at different temperatures indicated the hopping conduction mechanism. In addition, the variation of dielectric constant as a function of T confirmed the transition phase indicated by the differential scanning calorimetry (DSC). The physicochemical properties, UV-Vis, DSC and dielectric properties are described. Hirshfeld surface analyzes all the intermolecular interactions involved within the structure, which are important to stabilize the structure.



Key words: Chloranilate; charge-transfer; crystal structure; hydrogen bonds; conductivity

CRYSTAL STRUCTURE, VIBRATIONAL AND ANTIMICROBIAL ACTIVITY OF TERT-BUTYLAMMONIUM CHLORANILATE

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The salt of tert-btylamine (TerB) and chloranilic acid (2, 5 dichloro- 3, 6 dihydroxy 1, 4 benzoquinone CA) was grown from the ethanol solution. The title compound crystallizes in the $P2_1/n$ space group with the lattice parameters a= 9.1590(14) Å b=6.7998(11) Å c= 14.308(2) Å and β=91.599(7)° with Z=2. The structure was solved from 8195 independent reflections with R_1=0.0389 and wR_2= 0.0929. The structure consists of a protonated tert-Butylammonium cation paired with the dianion of chloranilic acid, which are interconnected via strong hydrogen bonds N-H ... O, electrostatic (cation-anion) and Cl…Cl (d_{Cl-Cl} = 3.480 Å) interactions. The new compound has been characterized by X-ray diffraction, differential scanning calorimetry (DSC), Infrared spectroscopy, UV-vis spectra and antimicrobial activity

Key words: crystal structure, chloranilic acid, hydrogen bonding, antimicrobial activity.

Influences of Ni doping on the structural and dielectric properties of $CaCu_3Ti_4O_{12}$ ceramics

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$CaCu_{3-x}Ni_xTi_4O_{12}$ (x = 0, 0.1, 0.15, 0.2, 0.3, 0.4, 0.5, 0.6) polycrystalline samples were prepared by a conventional solid state reaction. The influence of Ni substitutions on the structure and dielectric properties of CCNTO ceramics were investigated by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), dielectric Spectrometry and Raman Spectroscopy. Results confirmed the presence of a single phase of Ni-doped ceramics. Scanning electron microscopy micrographs of Ni-doped samples showed a striking change from regular polyhedral particle type in pure $CaCu_3Ti_4O_{12}$ (CCTO) to inhoùogéneous and voluminous grains.

Such as dielectric properties of these ceramic have a close relation with microstructure [1]. According to the internal barrier layer capacitance (IBLC) model [2], the effective dielectric constant ε_{eff} of the CCTO ceramics can be expressed by $\varepsilon_{eff} = \varepsilon_{gb}t/d$, where ε_{gb} is the dielectric constant of the grain boundary, t and d are the average grain size and the thickness of the grain boundaries, respectively. So, according to this model, the dielectric constant is function of the average grain size. In the context, the effect of Ni concentration on the electric behaviour of CCTO is investigated.

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Key words: $CaCu_3Ti_4O_{12}$; electrical properties; microstructure; colossal dielectric constant.

Facile Synthesis of Ag-ZnO Photocatalysts for Waste-water Treatment

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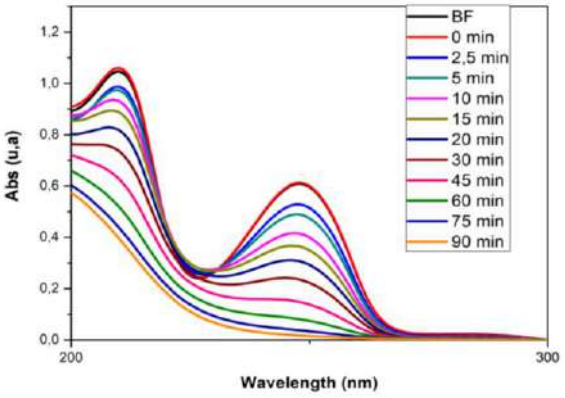
In the present communication, mono-disperse Ag-ZnO nano-photocatalyst were prepared via a simple, fast and effective chemical route at low-temperature, based on a modified polyol process using triethylene-glycol (TREG) as solvent, reducing and stabilizing agent. The synthesis procedure was conducted without any post-synthesis thermal treatment. The as prepared samples were characterized by different techniques such as X-ray diffraction, N2 adsorption study, UV-Vis diffuse reflectance spectroscopy, and transmission electron microscopy. Based on TEM images, the Nanocomposite consist mainly of quasi-spherical silver cores surrounded by closely packed zinc oxide nanoparticles. The photocatalytic performances of the hybrid Ag-ZnO nanomaterials was tested using Diuron pesticide as a model contaminant under simulated solar light irradiation. The sample with 0.68% Ag exhibits the best photoactivity performance compared to pure ZnO. The improvement of the photocatalytic activity may be attributed to the charge separation of photogenerated electrons and holes by Ag-ZnO hetero-nanostructure.



The absorbance spectra of diuron aqueous solution in the presence of 0.68% Ag-ZnO nanocomposites after different irradiation time

Keywords: Photocatalysis, Ag-ZnO Nanocomposites, Polyol process, Diuron, waste water treatment

The phase evolution of Lithium Iron Phosphate at elevated temperatures

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An amorphous compound Lithium Iron Phosphate was synthesized, The phase composition of the chemosynthetic powders was determined with X-ray diffraction (XRD) and the differential thermal analysis. The reaction of synthesis was carried out with mixtures of LiH2PO4 (2 g), and H3PO4 (1,3 ml g) with a molar ratio of 1 heated at 250°C for 48h, the mixture was filled into a 10 ml of water, a quantity of Fe(OH)2 was addict in this solution until saturation.
The XRD traces of lithium Iron Phosphate material unheated and heated at varied temperatures clearly shows that unheated compound appeared amorphous. The onset temperature of crystallization was 550 °C, three crystalline phases identified as $Fe_3(P_2O_7)_2$,$Fe(PO_4)$ and $LiFe(P_2O_7)$. During the thermal treatment up to 750 °C, the phase of $Fe_3(P_2O_7)_2$ was transformed to $Fe_4(P_2O_7)_3$.
TG/DTA analyses of the title compound revealed the presence of small exothermic peak was observed between 500 °C and 510 °C, it was correlated with the crystalline phase transformation.

Keywords: Amorphous compound, Phase transformation, Thermal analysis.

Elaboration, structural study and validation (CHARDI and BVS) of a new NASICON type $Na_{0.72}(Cr_{0.48}Al_{1.52})(Mo_{2.77}Al_{0.23})O_{12}$.

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The title compound $Na_{0.72}(Cr_{0.48}Al_{1.52})(Mo_{2.77}Al_{0.23})O_{12}$ was prepared by solid-state reaction. Its crystal structure is related to NASICON-type compounds. It crystallizes in the rhombohedric space group R-3c with a= 9.2169 (9) Å, c= 22.646 (2) Å, V = 1666.1 (4) $Å^3$, Z= 6. Crystal structure was refined to R (F^2) = 0.014and Rw (F^2) = 0.027. The framework is built up from an $M1O_6$ (M1= Cr1/Al2) octahedron and an $M2O_4$ (M2= Mo1/ Al1) tetrahedron interconnected by corners. The three-dimensional framework contains cavities in which the sodium cations are located.

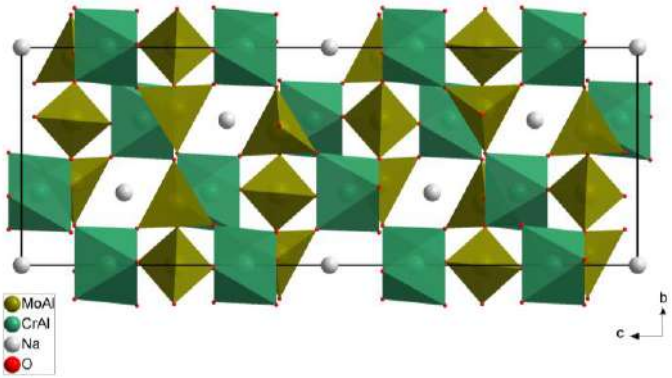

Fig. Projection of $Na_{0.72}(Cr_{0.48}Al_{1.52})(Mo_{2.77}Al_{0.23})O_{12}$ along the a axis.

Key words: NaSICON structure, framework, crystal structure, BVS, CHARDI.

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Synthesis, X-ray single crystal structure, likelihood of occurrence of intermolecular contacts, spectroscopic investigation and DFT quantum chemical calculations of zwitterionic complex: 1-Ethylpiperaziniumtrichlorozincate (II)

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Polymeric and extended metal (II) halide complexes have recently received a great attention for their particular properties related to their ability to optimize both the inorganic and organic components. The polymeric halometallates with Zn(II) metal ion, which have been reported to exhibit a wide range of attractive properties, such as optical, photoluminescent, catalytic, and electrical properties [1–3], belong to this class of compounds. In fact, we are now interested in the synthesis, the Hirshfeld surface analysis and the physicochemical characterization of the 1-ethylpiperaziniumtrichlorozincate (II) zwitterionic complex in the course of an ongoing program of research regarding the metal complexes of 1-ethylpiperazine. The synthesis and the X-ray structure of the Zn(II) zwitterionic complex:1-ethylpiperaziniumtrichlorozincate (II) are described. In the atomic arrangement, the $ZnCl_3N$ entities, grouped in pairs, are deployed along the *b*-axis to form layers. The organic entities are inserted between these layers through N-H…Cl and C-H…Cl hydrogen bonds to form infinite three-dimensional network (Fig.1). The 3D Hirshfeld surfaces were investigated for intermolecular interactions. The optimized geometry, Mulliken charge distribution, molecular electrostatic potential (MEP) maps and thermodynamic properties have been calculated using the Lee-Yang-Parr correlation functional B3LYP with the LanL2DZ basis set. The HOMO and LUMO energy gap and chemical reactivity parameters were made (Fig.2). The ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray crystal structure. The vibrational absorption bands were identified by infrared spectroscopy. DFT calculations allowed the attribution of the NMR peaks and of the IR bands.

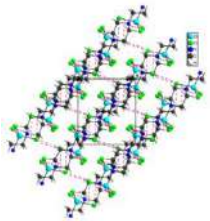

Fig. 1. Projection along the *b*-axis of the crystal packing of the title compound.

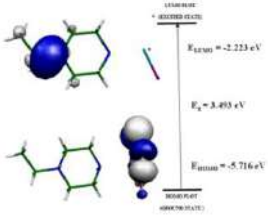

Fig. 2. Frontier molecular orbitals (HOMO and LUMO) of $C_6H_{15}N_2ZnCl_3$.

Keywords: Trichlorozincate (II); Crystal structure; Hirshfeld surface; enrichment ratio; Quantum; DFT; DSC; Infrared

References:

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THE SYNTHESIS, CRYSTAL STRUCTURE AND IR VIBRATIONAL OF TWO NEW ORGANIC-INORGANIC HYBRIDS $(C_5H_9N_3)Br_2$ AND $(C_5H_9N_3)Cl_2$

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Two new crystals of 2, 5-diaminopyridinediium dichloride and 2,3-diamino-pyridinediim dibromide were successfully synthesized and crystallized by slow evaporation method at room temperature. The two compounds (I) and (II) are characterized by single-crystal X-ray diffraction and FT–IR spectroscopic analyses. Both amine N atoms are protonated. In the crystal, moderately strong and weak intermolecular N-H…Br, C-H…Br and N-H…Cl hydrogen bonds link the anion and cation units into an infinite two-dimensional network.

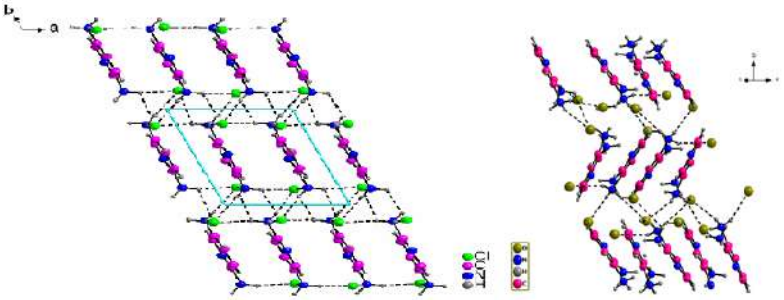

Fig: Projection of the crystal structure of the title compounds, with hydrogen bonds indicated as dashed lines.

Keywords: Organic template, molecular structure and IR spectroscopy.

Synthesis, Structure and Characterization of a Bioactive Te- Disubstituted Decavanadate Complex

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A new Te-substituted decavanadate cluster; $(C_9H_8N)_4[Te_2V_8O_{28}].8H_2O$ Tetraquinolinium ditelluro(VI) octavanadate(V) octahydrate (*Abbrev.* QTe_2V_8) has been grown by slow evaporation method and characterized by means of X-ray diffraction, IR, UV-Vis and fluorescence spectroscopy measurements. The powder and single crystal XRD analysis revealed that the isolated pure crystals belong to the triclinic system with a = 10.907 (3)Å, b = 11.302 (3) Å, c = 13.169 (2) Å, α = 106.45 (4)°, β = 107.71 (4)° and γ = 105.34 (4)°. Its crystal packing displays a 3-D supramolecular assembly built by $[Te_2V_8O_{28}(H_2O)_8]_n^{4n-}$ anionic sheets associated with $[C_9H_8N]^+$ cations through multiple H-bonds. These inter-contacts are also elucidated and quantified by Hirshfeld surface analysis. Moreover, the luminescent emission indicated a blue photoluminescence and the antimicrobial trials revealed a promising enhancement of biological activity compared to its basic reacting agents.

Key words: Te-disubstituted decavanadate, X-Ray Diffraction, Hirshfeld surface analysis, Photoluminescence, Antimicrobial activity.

E 235 CARBON STEEL CORROSION BEHAVIOR IN NEUTRAL CHLORIDE MEDIA

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Carbon steel is not stable in contact with air. In fact, oxygen, humidiy as well as pollutants could lead to materials corrosion.
Hence, we used electrochemical and surface analytical techniques to investigate the corrosion behaviour of two E235 carbon steel samples used on shock absorber manufacture in Tunisia (raw sample and phosphating sample). Furthermore, we tested Benzotriazole (BTA) as materials corrosion inhibitor in neutral aqueous chloride medium.
The experimental results revealed for both carbon steel samples that:

- the material corrosion rate (CR) increased when the medium chloride concentration increased reaching a maximum at 0.5 M;
- the CR decreased when the pH medium increased from 3 to 11;
- the CR decreased when the medium BTA content increased from 0.1mM to 0.5mM.

Keywords: steel E235, shock absorber, corrosion rate, polarization, Chloride ions, pH, BTA.

Thermal and Structural Analysis of Ni-based Heusler Alloy Ribbons

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Heusler alloy $Ni_{50-x}Mn_{36}Sn_{14}Pd_x$ (x=0, 1, 2) and $Ni_{50}Mn_{36}Sn_{13}Pd$ was produced as specially textured ribbon flakes by melt spinning, discovering the existence of martensitic-austenic transformation which was checked by calorimetry technique. The austenite state has an L21 structure, whereas the structures of the martensite can be 10M, 14M, or L10 depending on the Pd composition. Furthermore, the microstructure followed by scanning electron microscopy confirms the coexistence of equiaxial and columnar grains with an inhomogeneous distribution.
The temperature shift directly reports the valence electrons concentration per atom *e*/*a* of alloys.

Keywords: Shape memory alloys, Melt-spun process, Martensitic transformation, differential scanning calorimetry

Fabrication of CuO-NiO nanocomposites thin films and experimental study of the effect of the NiO proportions on its physical properties

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Copper oxide (CuO) and nickel oxide (NiO) and CuO/NiO nanocomposites thin films were deposited on glass substrates by the sol–gel method and spin-coating technique. Their structural and optical properties were investigated. XRD diffraction spectra allowed phase identifications of CuO and NiO with no appearance of secondary phases and the crystallinity of elaborated nanocomposite films improved with doping concentration increase. The optical properties of the films were characterized by UV–Visible spectrophotometry, which shows that the films are transparent in the visible region and their transparency is slightly influenced by the NiO ratio, with an average value above 70 %. The optical band gaps were in the range (3.6–1.75 eV).

Structure and microstructure properties of mechanically synthesized and thermally treated Fe-Ni-B-Si alloy

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Fe-Ni-B-Si alloy was prepared by mechanical alloying (MA) of elemental powders in a high-energy planetary ball mill. Phase evolutions, microstructure, thermal properties and behaviors after annealing were investigated. It was found that a body-centred cubic structured solid solution started to form after 25 h milling and a faced-centred cubic structure solid solution started to form after 50 h of milling and its amount increases gradually with increasing milling time. The crystallite size of the BCC and the FCC phases coexisted after 150h of milling with refined microstructure was 13 nm and 10 nm, respectively. After annealing at 450 ° C, the BCC phases and FCC coexist again and the amount of the last phase in the milled powder has increased. At 650 ° C, the most of the as-milled powder is converted to a single homogeneous solid solution BCC and exhibited good phase stability.

Titanocène/Nafion® graphite felt electrode for electrocatalytic reduction of dimetridazole

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The feasibility of an electrochemical pretreatment using a titanocène/Nafion® modified graphite felt for the removal of dimetridazole was investigated [1]. The stability of modified graphite felt was examined by cyclic voltammetry in acidic medium showing a good stability of the catalyst. Heterogeneous catalytic reduction of dimetridazole was then carried out at -0.45 V/SCE in a home-made flow cell. UPLC analysis showed an important elimination yield (>80%). After electrolysis, the modified graphite felt was examined by scanning electron microscopy coupled with energy dispersive spectroscopy analysis, revealing the lost of titanocène.

Key words: Dimetridazole- modified electrode- Electrocatalytic reduction- graphite felt

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Addition to booklet
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Electrochemical study of N-benzimidazole-O-acetyl-chitosan

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Cyclic voltammetry study of N-benzimidazole-O-acetyl-chitosan in acetonitrile solution reveals its anodic oxidation at a platinum disk and a progressive growing of a thin film, through the cycling of potential.

Fig. Electrodeposit of N-benzimidazole-O-acetyl-chitosan

Key words: N-Benzimidazole-O-acetyl-chitosan; Cyclic voltammetry; Coating electrode.

LITHIUM MOBILITY IN NASICON $Li_{1+x}Ti_{2-x}Sc_x(PO_4)_3$ ($0 \le x \le 0.5$) SERIES: A COMBINED NMR AND IMPEDANCE STUDY

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Rhombohedral NASICON compounds with general formula $Li_{1+x}Ti_{2-x}Sc_x(PO_4)_3$ ($0 \le x \le 0.5$) have been prepared using a conventional solid-state reaction and characterized by X-ray diffraction (XRD), nuclear magnetic resonance (NMR) and impedance spectroscopy (IS). The partial substitution of Ti^{4+} by Sc^{3+} and Li^{+} in the pristine $LiTi_2(PO_4)_3$ increases unit cell dimensions and the number of charge carriers. In Sc-rich samples, the analysis of XRD data and $^{6}Li/^{7}Li$, ^{31}P and ^{45}Sc MAS-NMR spectra confirms the presence of secondary $LiScO_2$ and $LiScP_2O_7$ phases that reduce the amount of lithium incorporated in the NASICON phase. For $x \ge 0.3$, ionic conductivity decreases because of secondary non-conducting phases formed at grain boundaries of the NASICON particles (core-shell structures). For $x=0.2$, high bulk conductivity (2.5×10^{-3} S.cm^{-1}) and low activation energy ($E_a=0.25$ eV) measured at room temperature makes $Li_{1.2}Ti_{1.8}Sc_{0.2}(PO_4)_3$ one of the best lithium ionic conductors reported in the literature. In this material, the vacancy arrangement enhances Li conductivity.

Magnetic and electrochemical properties effects of iron–aluminum substitution in new sodium nickel polyphosphate cathode materials $Na_2Ni_2(Fe,Al)(PO_4)_3$

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Herein we report the studies of different physical properties (structural, magnetic, thermal, morphologic, electrical and electrochemical) of two new allotropic β-$Na_2Ni_2M(PO_4)_3$ (NNMP) phosphates, with M = Fe and Al. Pure orthorhombic single-phase powders were prepared under air, using autocombustion synthesis method, Crystal structures of both compounds were determined using X-ray powder diffraction and Rietveld method refinements, which indicate the occurrence of Ni^{2+} in the 8g site, and of M^{3+} in the 4a site of the structure. The structure consists of three-dimensional anionic framework obtained by the association on MO_6, NiO_6 and PO_4 polyhedrons, sharing edges and corners. The resulting three-dimensional structure creates mono-dimensional channels along the [100] and [010] directions, formed by face-shared oxygen polyhedra and occupied by Na^+ cations. This non-disordered cationic distribution in confirmed by a significant change of magnetic properties. Thus, both NNAP and NNFP samples show paramagnetic to ferromagnetic transition at 14 and 19 K, respectively. For the two compounds, thermal stability, electrical conductivity and electrochemical properties have been also studied. The interaction/desintercalation properties of NNMP compounds as positive electrode were tested in sodium-ion batteries. The cycling curves exhibit a significant polarization even for both prepared samples.

Keywords: Na-ion battery; Crystal structure; Surface morphology; Curie temperature, Discharge capacity.

DESIGNING OF A NOVEL SYNTHETIC MITOCHONDRION-TARGETING TRI PHOSPHONIUM-FUNCTIONALIZED PHOTOSENSITIZING PHTHALOCYANINE

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Photodynamic therapy (PDT) is based on the association of photosensitizing molecules (PS) able to concentrate in tumor cells and of a focused light of appropriate wavelength (dependent on PS), to generate singlet oxygen which is highly reactive and toxic, thus induces cell death locally, without damaging healthy tissues. Among the different ways to optimize the efficiency of a photosensitizer, the targeting of mitochondria remains scarcely explored, especially for phthalocyanine-based photosensitizer, despite their advantages due to the maximum absorption at NIR wavelengths allowing deeper penetration of the light and excitation into the phototherapeutic window. Triphenylphosphonium moieties are known to have an excellent mitochondrion-targeting effect, hence triphosphonium-functionalized Zn(II) phthalocyanine was designed. The synthetic strategy will be presented and discussed together with its so-far completed synthetic and photo characterization.

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Experimental and theoretical investigations of a novel organic photovoltaic compound

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The technology of photovoltaic cells based on small organic molecules is a promising technology that successful reach a great success in the field of renewable energy. The study of structural, electronic and optical properties for these compounds could help to design more efficient functional photovoltaic organic materials. Therefore, designing and synthesizing conjugated molecules with interesting properties play a crucial role in technology at the same time it is important to understand the nature of the relationship between the molecular structure and the electronic properties to provide guidelines for the development of new materials in this field.

In this context, a structural study of a new organic photovoltaic compound was performed using X-ray powder diffraction data technique (figure 1) and quantum chemical calculation based on density functional theory (DFT) to predict the band gap of the conjugated system. All calculations were done with Gaussian 09 program package. Finally, more results will be presented at the seminar.

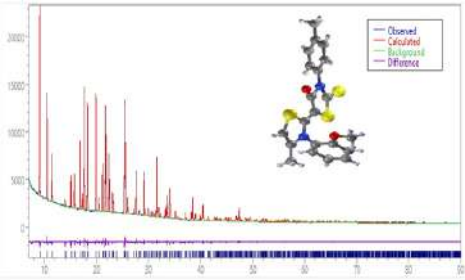

Figure 1. X-ray diffraction diagram of the title compound

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Facile Synthesis of Gold Nanoparticles with Irregular Shapes in Polyol and Aquatic Ecotoxicity Effects

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A simple chemical route, based on a modified polyol process, is used to synthesize mono-disperse triangular gold nanoprisms (Tr-AuNPs) in triethylene glycol (TREG) with polyvinyl-pyrrolidone (PVP) as capping agent. Both the solvent and the surfactant in the solution play important roles in the formation of the Tr-Au NPs. Shape, size and optical properties of the particles were tuned by changing the molar ratio of PVP to metal salts. The formation of such large, single-crystal Tr-Au NPs is explained by the preferential adsorption of some species of molecules from the solution onto the {111} planes of Au nuclei. The anisotropy in the nanoparticles' shape shows strong localized surface plasmon resonance (SPR) in the near infrared region of the electromagnetic spectrum. These nanostructures may be used in areas that include photonics, optoelectronics, optical sensing and also for inducing hyperthermia in tumors. For this reason, the ecotoxicological effects of the Tr-Au NPs and their interactions with marine organisms were also studied in this work. It was found that Tr-Au NPs don't seem to have a large effect on the non-target marine organism *R. decussatus*. Indeed, no significant modification ($p > 0.05$) was observed after 2 days on the glutathione-S-transferase activity of the clams exposed to Tr-Au NPs when compared to the control, both in the gill and the digestive glands.