Seventh International Solid State Chemistry Conference

ISSCC 2015

Organized by the

Société Chimique de Tunisie
Tunisian Chemical Society

December 21-24, 2015

Eden Star Hotel, Zarzis - Tunisia

Lectures and Communications Abstracts
List of Participants

Tunisian Chemical Society - Short Program of the ISSCC 2015

	Monday 21 December 2015
09.00 - 09.30	**Opening Ceremony**
09.30 - 10.10	**Lecture 1** **Pr. Latifa BERGAOUI** *National Institute of Applied Sciences and Technology, University of Carthage, Tunisia*
10.10 - 10.50	**Lecture 2** **Pr. Mohamed Naceur BELGACEM** *Grenoble Institute of Technology - Pagora, France*
10.50 - 11.15	***Coffee break***
11.15 - 12.30	**Oral Communications - First Session - OC 01 : OC 05**
13.00	***Lunch***
14.30 - 15.10	**Lecture 3** **Pr. Ezzeddine SRASRA** *Centre National des recherches en Science des Matériaux; Technopole Borj Cedria*
15.15 - 16.30	**Oral Communications - Second Session - OC 06 : OC 10**
16.30 - 17.30	**Coffee break + Poster Session 1 (P 1 - P 50)**
17.30 - 18.30	**Oral Communications - Third Session - OC 11 : OC 14**
19.30	***Dinner***
	Tuesday 22 December 2015
09.00 - 09.40	**Lecture 4** **Pr. Hassane OUDADESSE** *University of Rennes 1, Institut des Sciences Chimiques de Rennes, France*
09.40 - 10.20	**Lecture 5** **Pr. Lotfi BESSAIS** *University of PARIS 12, France*
10.25 - 11.10	**Oral Communications - Fourth Session - OC 15 : OC 17**
11.10 - 12.00	**Coffee break + Poster Session 2 (P 51 - P 100)**
12.00 - 13.00	**Oral Communications - Fifth Session - OC 18 : OC 21**
13.00	***Lunch***
	Afternoon off
19.30	***Dinner***
	Wednesday 23 December 2015
09.00 - 09.40	**Lecture 6** **Plenary Lecture 6** **Dr. Karim ZEHANI** *University of Paris Est, France*
09.45 - 10.45	**Oral Communications - Sixth Session - OC 22 : OC 25**
10.45 - 11.45	**Coffee break + Poster Session 3 (P 101 - P 145)**
11.45 - 12.30	**Oral Communications - Seventh Session - OC 26 : OC 28**
13.00	***Lunch***
15.00 - 16.15	**Oral Communications - Eighth Session - OC 29 : OC 33**
16.20 - 17.00	**Lecture 7** **Pr. Ahmed REBEY** *University of Monastir, Unité de Recherche sur l'Hétéro-épitaxie et Applications(URHEA), Faculty of Sciences of Monastir, Tunisia*
17.00	***Closing Remarks, Awards Distribution and ISSCC 2017 (8th edition) Announcing***
17.30	***Coffee break***
20.30	***Gala Dinner***

FOREWORD

The organizing committee of the Seventh International Solid State Chemistry Conference ISSCC 7 - 2015 is pleased to welcome you in Zarzis, Tunisia. The committee thanks you very much for answering our call and showing your commitment to this event and the subject matter.

I want to thank all my colleagues of the Organizing Committee and the Scientific Committee for their support, their effort and their availability.

Following the great success of previous events (JCS1- Monastir 2003, JCS2 - Mahdia 2005, JCS3 - Mahdia 2007, JCS4 - Zarzis 2009, JCS5 - Zarzis 2011 and JCS6 - Hammamet 2013, the Tunisian Chemical Society in collaboration with the Union of Arab Chemists organizes the Seventh International Solid State Chemistry Conference ISSCC 7-2015 from 21st to 24th December 2015 in Zarzis - Tunisia.

The scientific program promises to be exciting with seven plenary lectures, sixty-one oral presentations and one hundred fourty five posters.

This meeting between academics and industrials constitutes the promotion of research and the creation of a platform for discussion between researchers. This meeting is also open to young researchers who have a chance to present their work and interact closely with leading scientists.

We also wish to thank all the speakers and participants and hope that they can meet their expectations and aspirations.

On behalf of the Organizing Committee
Adel MEGRICHE
Chairman of ISSCC 7-2015
President of the
Tunisian Chemical Society

SCIENTIFIC COMMITTEE

Adel MEGRICHE (chairman)	Faculté des Sciences de Tunis
Mohamed KHITOUNI	Faculté des Sciences de Sfax
Nizar BELLAKHAL	Institut National des Sciences Appliquées et de Techologie - Tunis
Valérie PRALONG	CRISMAT Laboratory, ENSICAEN - Caen, France
Christian MASQUELIER	University of Picardie - France
Lotfi BESSAIS	Institut de Chimie et des Matériaux, University of Paris12 - France
Noureddine JOUINI	LSPM-CNRS, University of Paris13 - France
Naceur BELGACEM	EIPCIB, Grenoble INP-Pagora, France
Abderrahim MAAZOUZ	GMC Department - INSA - Lyon, France
Hassane OUDADESSE	Institut des Sciences Chimiques de Rennes, University of Rennes 1 - France
Ezzeddine SRASRA	Centre National de Recherches en Sciences des Matériaux - Borj Cédria
Samuel GEORGES	Université Joseph Fourier, Grenoble - France
M. T. WELLER	Royal Society of Chemistry - UK
Ahlem KABADOU	Faculté des Sciences de Sfax
Mohamed Kadri YOUNES	Faculté des Sciences de Tunis
Habib BATIS	Faculté des Sciences de Tunis
Houcine NAILI	Faculté des Sciences de Sfax
Najoua FRINI SRASRA	Faculté des Sciences de Tunis
Latifa BARGAOUI	Institut National des Sciences Appliquées et de Techologie - Tunis
Moheddine ABDELLAOUI	Institut National de Recherche et d'Analyse Physico-Chimique - Sidi Thabet
Mohamed BAGANNE	Ecole Nationale d'Ingénieurs de Gabès

ORGANIZING COMMITTEE

Mehrez ROMDHANE (chairman)	Ecole Nationale d'Ingénieurs de Gabès
Hatem BEN ROMDHANE	Faculté des Sciences de Tunis
Halim HAMMI	Centre National de Recherches en Sciences des Matériaux - Borj Cédria
Taïcir BEN AYED	Institut National des Sciences Appliquées et de Techologie - Tunis
Amine MNIF	Faculté des Sciences de Tunis
Imed LAAJIMI	Société Chimique de Tunisie
Mohamed Mouldi ABDELKAFI	Société Chimique de Tunisie
Hatem MAJDOUB	Faculté des Sciences de Monastir
Farhat REZGUI	Faculté des Sciences de Tunis
Ridha BEN SALEM	Faculté des Sciences de Sfax
Mohamed Lotfi EFRIT	Faculté des Sciences de Tunis
Rym ABIDI	Faculté des Sciences de Bizerte
Taoufik BOUBAKER	Faculté des Sciences de Monastir
Samir DJEMAL	Ecole Nationale d'Ingénieurs de Sfax
Béchir CHAOUACHI	Ecole Nationale d'Ingénieurs de Gabès
Abdelaziz TOUATI	Ecole Nationale d'Ingénieurs de Gabès

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

Sunday 20 December 2015	
15.00	***Welcoming participants, distribution of documents and check in***
19.30	***D i n n e r***

Monday 21 December 2015 (Morning)		
09.00	**O p e n i n g C e r e m o n y**	
09.30 - 10.10	**Plenary Lecture 1** **Pr. Latifa BERGAOUI** *National Institute of Applied Sciences and Technology, University of Carthage, Tunisia* **Surface modifications of aluminosilicate using manganese and synthesis of structured Mn-alumino-silicates**	*Chairpersons:* **Adel Megriche** *&* **Latifa Bergaoui**
10.10 - 10.50	**Plenary Lecture 2** **Pr. Mohamed Naceur BELGACEM** *Grenoble Institute of Technology - Pagora, France* **Inverse Gas Chromatography (IGC) as a tool to characterise the surface properties of powders and fibres**	
10.50 - 11.15	***C o f f e e b r e a k***	

Oral Communications - 1st Session				
	Room A - *Chairperson : H.Naili*		**Room B -** *Chairperson : M. Khitouni*	
	Com.	*communicating*	*Com.*	*communicating*
11.15 - 11.30	**OC-01A**	**Donia ZAMMEL** *FSM - Monastir*	**OC-01B**	**Marwa KHEMAKHEM** *ENIS - Sfax*
11.30 - 11.45	**OC-02A**	**Amal TOUNSI** *FSS - Sfax*	**OC-02B**	**Ahmed SOUEMTI** *FSB - Bizerte*
11.45 - 12.00	**OC-03A**	**Mouna SMIDA** *FSS - Sfax*	**OC-03B**	**Salam RHOUMA** *FST - Tunis*
12.00 - 12.15	**OC-04A**	**Najet SALAH** *FSS - Sfax*	**OC-04B**	**Marwène OUMEZZINE** *FSM - Monastir*
12.15 - 12.30	**OC-05A**	**Salem SAÏD** *FSS - Sfax*	**OC-05B**	**Kamel NOURI** *FSS - Sfax*
13.00	***L u n c h***			

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Monday 21 December 2015 (Afternoon)				
14.30 - 15.10	**Plenary Lecture 3** **Pr. Ezzeddine SRASRA** *Centre National des recherches en Science des Matériaux; Technopole Borj Cedria* **Tunisian clay and advanced Materials**			*Chairperson:* **Ismail Khattech**
Oral Communications - 2nd Session				
	Room A - *Chairperson : W. Smirani Sta*		**Room B -** *Chairperson : H. Tounsi*	
	Com.	*communicating*	*Com.*	*communicating*
15.15 - 15.30	**OC-06A**	**Lamia KHEDHIRI** *FSB - Bizerte*	**OC-06B**	**Arij MARZOUKI** *FST - Tunis*
15.30 - 15.45	**OC-07A**	**Ghaza RAHAL** *FSS - Sfax*	**OC-07B**	**Hana IBN GHARSALLAH** *FSS - Sfax*
15.45 - 16.00	**OC-08A**	**Narjess NGUILI** *FSS - Sfax*	**OC-08B**	**Imen HENTECH** *FSS - Sfax*
16.00 - 16.15	**OC-09A**	**Najla MAHBOULI RHOUMA** *FSS - Sfax*	**OC-09B**	**Mouldi BOUROUINA** *FSS - Sfax*
16.15 - 16.30	**OC-10A**	**Assila MAATAR BEN SALAH** *FSS - Sfax*	**OC-10B**	**Mohamed Amine BOUKADHABA** *FSM - Monastir*
16.30 - 17.30	**Coffee break**		**Poster Session 1 (P 01 - P 50) Alphabetical Order**	
Oral Communications - 3rd Session				
	Room A - *Chairperson : B. Ayed*		**Room B -** *Chairperson : N. Frini Srasra*	
	Com.	*communicating*	*Com.*	*communicating*
17.30 - 17.45	**OC-11A**	**Noura NOUIRI** *FSS - Sfax*	**OC-11B**	**Dhouha RTIBI** *FST - Tunis*
17.45 - 18.00	**OC-12A**	**Wahida LTAIEF** *ENIS - Sfax*	**OC-12B**	**Sana KEFI** *FST - Tunis*
18.00 - 18.15	**OC-13A**	**Fatma HMIDA** *FSM - Monastir*	**OC-13B**	**Amina GHARBI** *FSS - Sfax*
18.15 - 18.30	**OC-14A**	**Ramzi FEZAI** *FSB - Bizerte*	**OC-14B**	**Houda CHIHI** *FST - Tunis*
19.30	***D i n n e r***			

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Tuesday 22 December 2015 (Morning)				
09.00 - 09.40	**Plenary Lecture 4** **Pr. Hassane OUDADESSE** *University of Rennes 1, Institut des Sciences Chimiques de Rennes, France* **Presentation of the in vitro and in vivo behavior of three biomaterials: Aragonite, geopolymer/calcium phosphate and bioactive glasses**			*Chairpersons:* **Houcine Naili** *&* **Naceur Belgacem**
09.40 - 10.20	**Plenary Lecture 5** **Pr. Lotfi BESSAIS** *University of PARIS 12, France* **Nanomagnetic materials: Some emerging applications**			
Oral Communications - 4th Session				
	Room A - *Chairperson : A. Rebey*		**Room B -** *Chairperson : N. Bellakhal*	
	Com.	*communicating*	*Com.*	*communicating*
10.25 - 10.40	**OC-15A**	**Nabil HFIDHI** *FSS - Sfax*	**OC-15B**	**Aymen CHAABANI** *FSB - Bizerte*
10.40 - 10.55	**OC-16A**	**Nasreddine ENNACEUR** *FSG - Gafsa*	**OC-16B**	**Naceur ETTEYEB** *ISBAM - Médenine*
10.55 - 11.10	**OC-17A**	**Atef ELFERJANI** *FSS - Sfax*	**OC-17B**	**Maroua BOURKHIS** *FSB - Bizerte*
11.10 - 12.00	**Coffee break**		**Poster Session 2 (P 51 - P 100) Alphabetical Order**	
Oral Communications - 5th Session				
	Room A - *Chairperson : L. Bergaoui*		**Room B -** *Chairperson : I. Khattech*	
	Com.	*communicating*	*Com.*	*communicating*
12.00 - 12.15	**OC-18A**	**Jihene AISSAOUI** *FST - Tunis*	**OC-18B**	**Nabil BOUAZIZI** *ENIG - Gabès*
12.15 - 12.30	**OC-19A**	**Ines SAHLI** *FST - Tunis*	**OC-19B**	**Sonia BOUGHANMI** *FST - Tunis*
12.30 - 12.45	**OC-20A**	**Fatma WALHA** *ENIS - Sfax*	**OC-20B**	**Sahar BELGACEM** *FSB - Bizerte*
12.45 - 13.00	**OC-21A**	**Wided NOUIRA** *FSM - Monastir*	**OC-21B**	**Imen BOUCHMILA** *INRAP - Sidi Thabet*
13.00	***L u n c h***			
	Afternoon off			
19.30	***D i n n e r***			

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Wednesday 23 December 2015 (Morning)				
09.00 - 09.40	**Plenary Lecture 6** **Dr. Karim ZEHANI** *University of Paris Est, France* **Phase stability, EXAFS investigation and correlation between nanostructure and extrinsic magnetic properties of nanocrystalline $Pr_2(Co,Fe)_7$**			*Chairperson:* **Lotfi Bessais**
Oral Communications - 6th Session				
	Room A - *Chairperson : Z. Elaoud*		**Room B -** *Chairperson : M. Dammak*	
	Com.	*communicating*	*Com.*	*communicating*
09.45 - 10.00	**OC-22A**	**Raja JLASSI** *FSS - Sfax*	**OC-22B**	**Islem LABIDI** *FST - Tunis*
10.00 - 10.15	**OC-23A**	**Samia CHOUCHENE** *FSS - Sfax*	**OC-23B**	**Mahmoud CHEMINGUI** *FSS - Sfax*
10.15 - 10.30	**OC-24A**	**Nabaa BOUZIDIA** *FSS - Sfax*	**OC-24B**	**Safwen EL KOSSI** *FSM - Monastir*
10.30 - 10.45	**OC-25A**	**Khalil BOUZAZI** *FST - Tunis*	**OC-25B**	**Sabrine BOUKETTAYA** *FSS - Sfax*
10.45 - 11.45	***Coffee break***		**Poster Session 3 (P 101 - P 145) Alphabetical Order**	
Oral Communications - 7th Session				
	Room A - *Chairperson : M.Belhouchet*		**Room B -** *Chairperson : E. Srasra*	
	Com.	*communicating*	*Com.*	*communicating*
11.45 - 12.00	**OC-26A**	**Soumaya CHAOUACHI** *FSS - Sfax*	**OC-26B**	**Ines BEJAOUI** *FSB / INRAP*
12.00 - 12.15	**OC-27A**	**Dhouha BENHASSAN** *FSS - Sfax*	**OC-27B**	**Bakhta BOUZAYANI** *FSS - Sfax*
12.15 - 12.30	**OC-28A**	**Marwa BEN TAHER** *FSS - Sfax*	**OC-28B**	**Rabeb GRISSA** *Technopole Hélioparc - Pau, France*
13.00	***L u n c h***			

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Wednesday 23 December 2015 (Afternoon)			
Oral Communications - 8th Session			
	Room A - *Chairperson : F. Rezgui*		
	Com.	*communicating*	
15.00 - 15.15	**OC-29A**	**Abdessattar BEN CHRIFA** *FSS - Sfax*	
15.15 - 15.30	**OC-30A**	**Raoudha BEL HAJ SALAH** *FSB - Bizerte*	
15.30 - 15.45	**OC-31A**	**Houssem Eddine AHMED** *ENIS - Sfax*	
15.45 - 16.00	**OC-32A**	**Natija BARHOUMI** *FSG - Gabès*	
16.00 - 16.15	**OC-33A**	**Amira ANENE** *INRAP - Sidi Thabet*	
16.20 - 17.00	**Plenary Lecture 7** **Pr. Ahmed REBEY** *University of Monastir, Unité de Recherche sur l'Hétéro-épitaxie et Applications(URHEA), Faculty of Sciences of Monastir, Tunisia* **High Resolution X-Ray Diffraction of epitaxial thin films elaborated by MOCVD**		*Chairperson:* **Hatem Majdoub**
17.00	**Closing Remarks, Awards Distribution and ISSCC 2017 (8th edition) Announcing**		
17.30	***Coffee break***		
20.30	***Gala Dinner***		

Thursday 24 December 2015
Breakfast and Check out

Speakers' CVs & Abstracts

Latifa BERGAOUI

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University of Carthage, Tunisia
Centre Urbain Nord BP 676-1080 Tunis Cedex
Attached to the Laboratory of Materials Chemistry and Catalysis, FST, El Manar University, Tunisia
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Latifa Bergaoui is a professor of Chemistry at the National Institute of Applied Sciences and Technology (Tunisia). She took a Master's degree in chemistry in 1990 from the Faculty of Sciences of Monastir (Tunisia) then a Postgraduate Degree (DEA) of Inorganic Chemistry titled “From the molecule to Materials” in 1991 from the University Pierre and Marie Curie (France) and from the same University she got her PhD in 1994. Thereafter, she did a one-year postdoctoral at the National Institute for Agricultural Research of Versailles, (France). Between 1996 and 1998 she was an Assistant at the Preparatory School for Engineer Studies in Monastir (Tunisia). From 1999 to 2005 she was an assistant professor and from 2005 to 2010 she was Senior Lecturer then since 2011 she is a Professor at the National Institute of Applied Sciences and Technology belonging to University of Carthage (Tunisia). Her research activity deals mainly with the elaboration or modification of aluminosilicates to enhance their properties in adsorption and catalysis. She is interested in sol-gel and hydrothermal synthesis of manganese oxides and alumniosilicates containing manganese in their framework but also in modification of lamellar materials by surface functionalization, by modification of the interlamellar spaces and by grinding. Latifa Bergaoui authored 30 publications (H-index 11), several abstracts of communications presented at national and international conferences.

Surface modifications of aluminosilicate using manganese and synthesis of structured Mn-alumino-silicates

Latifa Bergaoui

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A review of the diversity of atomic architectures of Mn oxide and a presentation of aluminosilicates (clay minerals and zeolite) are first mentioned in this presentation, then the similarities between those two families is underlined and different strategies to modify aluminosilicates using manganese is shown.

In fact, Mn occurs in natural systems in three different oxidation states: II, III, and IV. Over 30 different natural and synthetic Mn oxide minerals are known for existing, largely due to the combination of varied valence states of Mn and the diverse arrangements of MnO_6 octahedra. Most Mn(III)/Mn(IV) oxides are categorized as having either a layer (like in clay minerals) or tunnel structure (as in zeolites). A wide variety of cations can be incorporated into the interlayers and tunnels. This cationic exchange capacity is one of the most important proprieties of zeolites and some clay minerals. Because of their properties, there is considerable interest in the use of these materials as catalysts and cation exchange agents. For this purpose, swelling can be used to allow access to the internal surface of the clay mineral, but the collapse of the swollen layers upon drying results in the loss of this accessibility. There are various methods to resolve this problem, which make clay minerals more attractive in catalysis. On the other hand, the zeolite-type materials have long attracted the attention of scientists and engineers with their properties combining large surface area, well-defined channel system and controlled density of active sites. Moreover, stable and interesting centers active in heterogeneous catalysis can be created by combining the open structure of zeolites and the catalytic properties of transition metal oxides.

In this talk, a particular attention is given to:

i) different used strategies to functionalize aluminosilicate by manganese

ii) our recent results about the incorporation of manganese in the aluminosilicate framework.

Mohamed Naceur BELGACEM

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Professor Belgacem received his Engineer diploma in wood science at the Saint Petersburg's Forest Academy (Russia: 1980-1986). He then got a PhD in Material Engineering and Science from the Polytechnic Institute of Grenoble, France in 1991. He then spent two years in Polytechnic School of Montreal (Canada) as a post-doc. On his return, he accepted a researcher position in a private company, before moving to University of Beira Interior in Portugal where he spent three years (from 1997 to 2000) as a lecturer and associate professor. Dr. Belgacem was nominated full professor in 2000 at the Polytechnic Institute of Grenoble, France, where he has been developing research activities on the rational use of vegetal biomass, valorization of industrial and agricultural resources and surface treatments and adhesion phenomena of lignocellulosic fibres. Professor Belgacem has supervised about 25 PhD theses, published about 180 works, including 2 books and ~25 book chapters. Since September 2007, Professor Belgacem is an Editor-in-chief of an Elsevier Journal: "Industrial Crops and Products" (ISSN: 0926-6690 http://ees.elsevier.com/indcro/). Finally, he is a Doctor Honoris Causa of the Saint Petersburg Forestry Academy, awarded in 2006.
From 2002 to 2010 he directed the CNRS research unity LGP2 and since 2014 he's directing Grenoble INP-Pagora.

Inverse Gas Chromatography (IGC) as a tool to characterise the surface properties of powders and fibres

Mohamed Naceur Belgacem

Univ. Grenoble Alpes, LGP2, F-38000 Grenoble, France
CNRS, LGP2, F-38000 Grenoble, France
Agefpi

After the end of World War II, science and technology related to polymers underwent a spectacular development that resulted in the production of a wide variety of materials covering a vast domain of applications. These thermoplastic and thermoset materials present many advantages compared with metallic, ceramic, and glass counterparts. During the last three decades, attention has been focused on the combination of these polymers with natural or synthetic fibres and fillers, in order to obtain high-added-value materials. Whereas fibre-based composites ensure a considerable improvement of the mechanical properties, which can reach those of metallic structures and provide applications, e.g., in transport and space technology, the addition of powders to polymers is particularly useful in the formulation of paints, inks, elastomers, coatings, blends, etc.
The performance of both types of composites depends on the properties of the individual components, as well as on their surface compatibility. The latter point is of great relevance because the physical and chemical interactions between the continuous phase (matrix) and the reinforcing fibres or fillers must be optimized in terms of maximum interfacial compatibility.
The bulk and surface properties of the individual components (fibres, powders, polymers, etc.) are often difficult to ascertain. Although the existence of several techniques, The present talk will give a wide survey about two techniques, namely Inverse Gas Chromatography (IGC) and Inverse Size Exclusion Chromatography (ISEC) and will show how they can be used to assess the surface and bulk properties of various solids with different morphologies (fibres and powders).
IGC and ISEC are, types of conventional gas and liquid chromatography, respectively, in which the material of interest constitutes the stationary phase rather than injected probes. Thus, the column containing the material under investigation is placed between the injector and the detector of any conventional chromatographic equipment and the measurements consist of injecting molecular or macromolecular probes possessing well-known properties through the column *via* an inert gas or liquid carrier (carrier vector). The retention time of each probe is related to the properties of the stationary phase.
The properties discussed in the present conference are the free energy of the surfaces under study, their acid-base character, the surface area of different solid morphologies, the transition temperatures of polymers, the fraction of a polymer crystalline phase, the interaction parameters of some polymer-solvent, polymer-polymer, polymer-filler and polymer-fibre systems (such as polymer blends, composites, and filled polymers like paints, tires, inks, etc.) and the pore-size and pore -size distribution of different solids.

These two techniques present many advantages, namely:

1. Simplicity
2. Lack of sensitivity to surface roughness compared to contact angle measurements
3. Applicability to non-film forming materials
4. Lack of surface contamination of samples under investigations from the surrounding atmosphere
5. Relative rapidity of data collection
6. Accuracy of results
7. Data collection over an extended temperature range
8. A large variety of probes, with different properties
9. Ease of temperature control
10. Low capital investment for the basic tools

However, IGC and ISEC have also some limitations, because they require a well-defined particle size range and cannot be applied to high-surface-energy materials because of chemi-sorption. Moreover, materials that cannot be easily ground to a fine powder cannot be studied.

Ezzeddine SRASRA

Date of Birth: September 6th 1955 in Tamezret – Tunisia
Nationality: Tunisian
Marital status: Married
Address: National Center Research of Materials Science Borj Cedria Technopark
Tel: 21679325470 - 216-98352973 - *Fax*: 21679325314
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FORMAL EDUCATION

1976 -1980: ***Graduation*** in Chemistry (june 1980): Science Faculty of Tunis
1980-1982: ***Master degree*** (june 1982) Science Faculty of Tunis
Subject: ***Liquid-liquid extraction of silver by pyridinethiol***.
1983-1987: ***Master thesis*** (June 1987): Science Faculty of Tunis
Subject: ***Physico-chemical characterisation of Tunisan Bentonites and their applications***
1994 - 2002: ***Higher doctoral thesis*** (Mars 2002)
Subject: c***lay and acidity - Mechanism of acid activation and the result properties***

ACTUAL POSITION

Professor in National Center Research of Material Science
Head of Physico-chemical mineral materials and application Laboratory

Responsibilities

2006-2009: Director of Research Unit of materials to CRTEn
since 2010: Director of the Laboratory of Physics Chemistry of Materials Minerals and their applications CNRSM

Research Projects Executed

1 Tunisian Chemical Group / Research Unit Materials: Setting heap of phosphogypsum and possibility of storage in clay: which aims to research a potential site for geological storage of emissions resulting from the manufacture of phosphoric acid (phosphogypsum). 2006-2009
2- Federator Research Program. Entitled .Conception and development of a fuel cell. 2008 -2010. Industrial partner ANME
3- SOWAEUMED FP7 project (2011-2012): Network in solid waste and water treatment entre Europe and Mediterranean countries
4- Cooperation Project with the National Office of Mining: Development of bentonite in the region of Zaghouan: hydrocycloning treatment (2013-2014)

international cooperation

1- Bilateral Tunisian-Portuguese (2008-2010) Project: The impact of harmful Elements derived from waste waters on the environment: Sorption and modeling studies of metals (Pb, Cd, Cr, Zn, Fe); Fluoride and phosphorous, into Tunisian and Portuguese clay minerals
2- Multilateral Cooperation FP7 SOWAEUMED project (2011-2012): Network in solid waste and water treatment entre Europe and mediterranean countries
3-Tunisian-Algerian bilateral cooperation (2012-2015) Project: Approach of green chemistry in the valuation of Tunisian materials by heterocycles

International Publications

100 published international paper (WWW.Scopus.com: h_{index}: 16)

INORPI Patents

1. Patent N1.
Bleaching properties of the oils by a Tunisian Clay:
Salem Hamdi, Insess Rekik Ellouze, Atef Malek and ezzedine Srasra
SN08472, 21 November 2008

2. Patent N2
Title: Method of obtaining a geopolymer cement from a Tunisian clay;
Noureddine Hamdi Ahmed Nmiri and Ezzeddine Srasra. TN2013 / 0451, October 30, 2013

3. Patent N3:
Title: Process for decontaminating based on adsorption by new activated carbon for the removal of hydrogen sulphide (H2S) in aqueous phase.
AZIZI Mohamed (CERTE) MAHMOUDI Khaled (CNRSM), Noureddine Hamdi (CNRSM) Boughanemi Hichem (CERTE), Mechi Lasaad (CERTE), Mohamed BEN AMOR (CERTE) SRASRA Ezzeddine (CNRSM). 2014

Students supervision

10 PhD defended since 2002
4 habilitations
25 Masters

Tunisian clay and advanced Materials

Ezzeddine SRASRA

***L**aboratoire de **P**hysico-**C**himie des **M**atériaux **M**inéraux et leurs **A**pplications (LPCMMA)*
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Recently, various nanohybrid materials have attracted considerable research interest due to their unusual and synergetic physicochemical properties, which cannot be achieved by any single component. Among them, the intercalation reaction – which is the reversible insertion of guest species into two-dimensional host materials – is expected to be one of the most effective ways of preparing noble materials with the desired functionality.

This regard, clay minerals, in particular, have attracted a great deal of attention, because they can keep various cationic species within their interlayer space, regardless of whether they are inorganic or organic ions or even polymeric ions. Therefore, numerous studies have been directed towards the intercalation chemistry of clay minerals.

In this study, a smectites Tunisian Clays are widely used in a range of applications because of their high cation exchange capacity, swelling capacity, high specific surface area. The combination of inorganic complex, surfactant and conducting polymers with tunisian clay materials having different characteristics opens a way to new hybrid materials showing novel properties.

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Hassane Oudadesse graduated from the University Blaise Pascal of Clermont-Ferrand, France. He obtained his PhD on nuclear physics in 1989. His works concern the use of nuclear methods for development in biomedical field. He worked as associate Professor and obtained his HDR in 1998. Since 2001, he works in the University of Rennes 1 as Full Professor. His works concern the conception, synthesis and physicochemical studies of new biomaterials for applications in orthopaedic surgery. Biocompatibility, kinetic of bio consolidation in the interface bone - implants, cells enhancement present his research filed. He is author of more than 130 papers published in international journals and about 65 international conferences. Prof. H. Oudadesse is a Head of the research unit on Biomaterials since 2001, Vice President of University of Rennes 1, human resources 2008 - 2012, Director of Master 2 Solid State Chemistry and Materials since 2006. He was the President of the Chemical Department from 2002 to 2004 and the President of the specialists commission CNU 33 (Materials Chemistry) from 2003 to 2008.

Presentation of the in vitro and in vivo behaviour of three biomaterials: Aragonite, geopolymer/calcium phosphate and bioactive glasses

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In orthopaedic surgery, grafts are used as a filling biomaterial for defective bone. The success of synthesised bony biomaterials depends on many determinant factors.
The synthesis, the physico chemical properties and the in-vivo behaviour of these materials were established by using multidisciplinary techniques such as DRX, SEM, PIXE, NAA, MAS-NMR and ICP-OES. Three bone biomaterials have been studied by using in vitro and in vivo investigations.

Vectabone: $CaCO_3$

biomaterial in pure aragonite form of 55% of porosity was synthesised and the active substance (gentamicin) was introduced at the first stage of the elaboration. The bony remodelling was studied by the in vivo experiments on the femoral diaphysis.
The kinetic resorption (Ca, P, Sr and Mg) of this biomatrial was insured with neutron activation analysis (NAA). Obtained results highlight a substantial mineral variation composition of $CaCO_3$. They show that the addition of the active substance causes an important activity of the bony metabolism and accelerates the bony neoformation.
Cartographies of every element of the biomaterial, of the interface (biomaterial-bone) and of the bony matrix were established by the Proton Induced X-Ray Emission method (PIXE).

Composite Geopolymer/calcium phosphate

The association between aluminosilicate synthesised with molecular ratio K_2O/SiO_2 = 0.54 and Si/Al = 21 and 13 % weight of calcium phosphate permit to have composites with about 64% of porosity and 6 MPa of compressive strength. The "*in vitro" and the "in vivo"* experiments were carried out to study the behaviour of these composites. The chemical element Al was particularly studied because of its toxic character in the organism. The ICP (Inductively Coupled Plasma) technique was employed in this goal. ICP analyses of compounds immersed in SBF solution show the total absence of the release of Al in the solution. Ca and P present the same behaviour and were not released to the SBF. This result traduces the good chemical stability of this composite. For the "in vivo" experiments, composites in the cylindrical form were implanted in the rabbit thighbone. Obtained results by PIXE show the important variations of atomic elements concentrations versus time after implantation. In the interface bone-composite, the intimate links traduce the high quality of the biointegration and the bioconsolidation of GPS composites.

Bioactive glasses

The reinforcement of scaffolds by the bioactive glass 46S6 induces several chemical modifications and a good bioactive character after immersion in the SBF. The textural properties are improved due to the increase of the content of chitosan. Indeed, the increase of specific surface area, average pore diameter and pore volume are observed. Histological study shows the osseointegration and the biodegradation of the glasses by the progression of the osteoblasts' deposition in the pores of biomaterial leading after nine months to it's totally degradation and its replacement by mineralized and osteoid bone.

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PhD, University of Paris VII (1990),
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Publications:
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Nanomagnetic materials: Some emerging applications

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Nanoscale magnetic systems have a great potential for technological applications and huge challenges in fundamental science. The main feature compared to bulk materials is the fact that the overall direction of the magnetization of a nanoscale system can switch between different orientations within the scale of the observation time. Indeed, the energy barriers between these orientations depend on the nanocluster size. For practical applications, this size is reduced for maximum information storage. However, this affects the stability of the magnetization since the energy barriers are reduced at room temperature. This phenomenon of macroscopic fluctuation of the magnetization of the monodomain under the thermal agitation effect is called superparamagnetism, by analogy with paramagnetism phenomenon taking place at the atomic level.

In this talk, we will present some approaches to solve this model and the different assumptions on the relevant physical parameters. Then we discuss some results of calculations (analytical and numerical) of the relaxation time.

In the second part we present an experimental approach to the correlation of structural, microstructural and magnetic properties of intermetallic nanomaterials. A nanostructured material has an enhanced behavior compared to the bulk material. This is due to its size in the order of magnitude of the correlation length of the characteristic properties studied. In fact, the size of a particle becomes less than the domain-wall thickness or less than the exchange length.

An important challenge is to control the composition and size of these materials in order to understand the mechanisms governing their behavior in a fundamental research approach, to coordinate more easily improve their resulting properties. Through some examples, we will show different areas of potential applications: the permanent magnets, high density magnetic recording, magnetic refrigeration and medical applications.

Key words: Relationship between structural nanostructral and magnetic properties, Mössbauer Spectroscopy, Superparamagnetism, Rare Earth-Transition metal alloys

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Productions scientifiques

- 10 conférences internationales.
- 15 publications dans des revues internationales avec comités de lectures

Publications les plus significatives

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- R. Guetari, R. Bez, A. Belhadj, K. Zehani, A. Bezergheanu, N. Mliki, L. Bessais, C.B. Cizmas,
  Influence of Al substitution on magnetocaloric effect of $Pr_2Fe_{17-x}Al_x$,
  Journal of Alloys and Compounds, Volume 588, 5 March 2014, Pages 64-69.

Phase stability, EXAFS investigation and correlation between nanostructure and extrinsic magnetic properties of nanocrystalline $Pr_2(Co,Fe)_7$

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Nanocrystalline $Pr_2Co_{7-x}Fe_x$ ($x = \leq 3.5$) powder was synthesized by high energy milling and was subsequently annealed at 973 K for 30 min. Their crystalline structures were investigated by means of X-ray diffraction (XRD), X-ray absorption spectroscopy (XAS) and transmission electron microscopy (TEM). Magnetic properties were also studied at room temperature and 10 K as a function of the iron composition. This compounds structure depends on the iron content; for $x \leq 1$ they crystallize in the Ce2Ni7-type hexagonal structure (space group: $P6_3/mmc$). For $1 < x \leq 3$, we observe a mixture of three phases, $Pr_2(Co,Fe)_7$, $Pr(Co,Fe)_3$ and $Pr_2(Co,Fe)_{17}$ while for larger values of x the $Pr_2(Co,Fe)_7$ phase disappears completely. Since the substitution of cobalt by iron in a pure $Pr_2Co_{7-x}Fe_x$ phase is limited to $x \leq 1$, a site preference can be suggested. Unfortunately standard XRD cannot be used unambiguously because Co and Fe have too close normal X-ray atomic scattering factors. Among the local and Fe selective available experimental methods, we have chosen to explore the P63/ mmc space group Wyckoff positions for iron by EXAFS at the Fe edge. This study shows that the local iron radial distribution function is much larger than expected for all the specific sites, at least two Fe-Pr distances, except for 12k which gives the best fit. A preferential substitution of cobalt by iron in 12k site is coherent with (i) the EXAFS result (ii) the substitution rate $x < 1$ (iii) the anisotropic increase of the crystal cell parameters. A mixture of 12k, 6h and 2a cannot be excluded. However, if we assume a unique preferential site, 12k is the only solution.
For $x \leq 1$, the magnetocryslalline anisotropy is observed in $Pr_2Co_{7-x}Fe_x$ alloys with fairly strong anisotropy fields at room temperature in range from 123 to 136 kOe for $x = 0$ and $x = 1$, respectively. For $x > 1$, the magnetocrystalline anisotropy field of the 2:17 phase is lower than that of the 2:7 phase, which causes a further reduction in anisotropy and coercivity.

Keywords: Phase stability, EXAFS study, Magnetic properties

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Ahmed REBEY was born in Gabès, Tunisia, in 1969. He received the M. A in Physics from the Faculty of Sciences of Monastir, Tunisia, in 1992. He then acquired the Master and Ph D. Degrees in Physics of condensed matter from the Faculty of Sciences of Tunis, Tunisia, in 1995 and 2001 respectively. In 1998, he joined the Department of Physics of Faculty of Sciences of Monastir, Tunisia, as an assistant professor. Where he is currently a Professor and Head of Department. His main areas of research interests are MOCVD of semiconductors, characterizations of thin films and nanostructures and ab-initio calculations of solid properties.

High Resolution X-Ray Diffraction of epitaxial thin films elaborated by MOCVD

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In this presentation, we describe the Metal-Organic Vapor Phase Epitaxy (MOCVD) as a technique of growth of thin crystalline films and nanostructures for electronic and optoelectronic applications. The growing conditions and their effects on the quality of the epitaxial layers will be discussed in details by using High Resolution X-Ray Diffraction (HRXRD) technique.

This technique is not used to determine the crystal structure, but to investigate deviations from an ideal crystal which can be induced i. e. by defects, mosaicity or strain.

We first give an overview on the basic concepts of how to describe an ideal crystal. Subsequently, we treat irregularities and defects in real crystals. We then present the experimental setup and the characterization of real crystals by HRXRD. Several configurations such as w/2θ(symmetric and asymmetric)curve, w-Rocking curve or mapping of reciprocal space are used to provide a wealth of information about crystal.

Keywords: HRXRD, thin crystalline films, strain, mosaicity.

Oral Communications' List

(alphabetically authors' name)

Name	**Ref**
H.E. Ahmed, S. Karoui, S. Kamoun, F. Michaud *ENIS - Sfax* Crystal structure, phase transition studies of $(C_6H_{18}N_2)_3SB4CL18$ compound	**OC 31A**
J. Aissaoui, M. Kacem, A.I Mgaidi, P. Dubujet *FST - Tunis* The impact of pyrene on the retention of lead in soil	**OC 18A**
A. Anene, R. Kalfat, Y. Chevalier, S. Hbaieb *INRAP - Sidi Thabet* Molecularly imprinted polymer based on silica supports for efficient adsorption of Patulin	**OC 33A**
N. Barhoumi , L. Labiadh, M. A. Oturan, N. Oturan, A. Gadri, S. Ammar, E. Brillas *FSG - Gabès* Mineralization of the antibiotic levofloxacin by electro-Fenton-pyrite process	**OC 32A**
I. Bejaoui, M. Baâzaoui, R. Kalfat, N. Amdouni, S. Hbaieb, Y. Chevalier *FSB / INRAP* Influence of the substitution of β-cyclodextrins by pyridinium groups on the complexation of adamantane derivatives	**OC 26B**
R. Bel Haj Salah, L. Khedhiri, M. Rzaigui *FSB - Bizerte* Synthesis, structure and physicochemical studies of hexakis (3-chloro 2-methylanilinium) cyclohexaphosphate dihydrate: $[C_7H_9ClN]_6 P_6O_{18}.2H_2O$	**OC 30A**
S. Belgacem, H.L Galai, K. Nahdi *FSB - Bizerte* Study of Magnesia –Spinel brick of a rotary cement Kiln	**OC 20B**
A. Ben Chrifa, A. Ben Salah, M. Loukil *FSS - Sfax* Structure spectroscopic and thermal behavior of two mixed trichlorocadmiate (II)	**OC 29A**
M. Ben Taher, N. Chaari, S. Chaabouni *FSS - Sfax* Synthesis, crystal structure and vibrational properties of (3-ammoniumpropyl) imidazolium pentachloroantimonate(III)	**OC 28A**

Name	**Ref**
D. Benhassan, W. Rekik, H. Naïli, T. Mhiri, T. Bataille *FSS - Sfax* Synthesis, crystal structure and thermal behaviour of two new hybrid nitrate compounds $(C_4H_{12}N_2)_2[M^{II}(H_2O)_6](NO_3)_6$ WITH M^{II}: Ni(II), Zn(II)	**OC 27A**
N. Bouazizi, R. Bargougui, A. Benghnia, R. Ben Slama, B. Chaouachi *ENIG - Gabès* Improvement properties of modified TIO_2 nanoparticles by grafted hexamethylenediamine and incorporated iron nanoparticles	**OC 18B**
I. Bouchmila, B. Bejaoui Kefi, R. Souissi, M. Abdellaoui *INRAP - Sidi Thabet* Desorption of rare earth elements from "Chert" of Gafsa Metlaoui basin	**OC 21B**
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M.A. Boukadhaba, A. Fouzri, M. Oumezzine *FSM - Monastir* Chracterization of ZnO thin film grown on vicinal-cut sapphire substrates α-Al_2O_3 (0001) (α<0.1°) by MOCVD	**OC 10B**
S. Boukettaya, M. Smida, M. Dammak, S. Garcia-Granda *FSS - Sfax* Structural, thermal decomposition and vibrational study of the new bidimentionel fluoroferrate Fe_3F_{10}(3,5-diamino-1,2,4-triazole)$_2$	**OC 25B**
M. Bourkhis, R. Abderrahim *FSB - Bizerte* Synthesis and physicochemical identification of amidine cyclodextrin derivative	**OC 17B**
M. Bourouina, A. Krichene, N. Chniba Boudjada, W. Boujelben *FSS - Sfax* Structural and magnetic properties of $Pr_{0.5-x}Gd_x Sr_{0.5}MnO_3$ manganites	**OC 09B**
B. Bouzayani, T. Mhiri, S. Chaâbane Elaoud *FSS - Sfax* Electrochemical behaviour of ferulic acid in aqueous solutions on glassy carbon by cyclic voltammetry	**OC 27B**

Name	**Ref**
K. Bouzazi, H. Boughzala, H. Zamali *FST - Tunis* Phase relationships in the KNO_3-$RbNO_3$ binary system at normal condition	**OC 25A**
N. Bouzidia, B. Hamdi, A. Ben Salah *FSS - Sfax* Synthesis, crystal structure and hirshfeld surface analysis of a new microporous phosphate organometallic	**OC 24A**
A. Chaabani, N. Souissi, M. Ben Messaouda, A. Moadhen, A. Madani, X.R. Nóvoa *FSB - Bizerte* The effects of *Laurus nobilis* leaves aqueous extracts on the corrosion behavior of chloride patina coated binary tin-bronze	**OC 15B**
S. Chaouachi, B. Hamdi, A. Ben Salah, R. Zouari *FSS - Sfax* X-ray single crystal, Hirshfeld analysis, vibrational spectra, optical studies and dielectric relaxation of new organic-inorganic compound $(C_2H_7N_4O)_2[CoCl_4(H_2O)_2]$.	**OC 26A**
M. Chemingui, V. Optasanu, M. Khitouni *FSS - Sfax* Characterization of amorphous Al-Co-Nb alloy prepared by high energy mechanical alloying	**OC 23B**
H. Chihi, I. Khattech, M. Jemal *FST - Tunis* Standard enthalpy, entropy and Gibbs free energy of formation of magnesium carbonate co-substituted fluorapatites	**OC 14B**
S. Chouchene, K. Jaouadi, T. Mhiri, N. Zouari *FSS - Sfax* Structural, thermal behaviour and vibrational study of a new cesium- rubidium dihydrogen arsenate: $Cs_{0.2}Rb_{0.8}H_2AsO_4$	**OC 23A**
S. El Kossi, J. Dhahri, K. Khirouni, E.K. Hlil *FSM - Monastir* Investigation of the magnetocaloric effect and dielectric properties in $La_{0.7}Sr_{0.25}K_{0.05}Mn_{(1-x)}Ti_xO_3$ perovskite manganite	**OC 24B**
A. Elferjani, M. Abdelhedi, A.W. Kolsi, M. Dammak *FSS - Sfax* Synthesis, Structural and vibrational studies of the new mixed solid solution thallium ammonium sulfate selenate tellurate	**OC 17A**

Name	**Ref**
N. Ennaceur, R. Henchiri, I. Ledoux-Rak *FSG - Gafsa* Combined theoretical and experimental studies on the molecular structure, spectral and Hirshfeld surface studies of NLO 1,4-Diazabicyclo[2.2.2]octane hydroxonium crystals	**OC 16A**
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R. Fezai, M. Rzaigui *FSB - Bizerte* Synthesis, physicochemical study and structure characterization of a new acidic cyclohexaphosphate $[O\text{-}(OCH_3)C_6H_4NH_3]_4.H_2P_6O_{18}.2H_2O$	**OC 14A**
A. Gharbi, H. Oudadesse, H. El Feki *FSS - Sfax* A new glance at physico-chemical comportments of silicate bioactive glasses: influence of partial substitution: Borosilicate versus Fluorosilicate glasses	**OC 13B**
R. Grissa, H. Martinez, S. Cotte, V. Pelé, B. Pecquenard, F. Le Cras *Technopole Hélioparc - Pau, France* Iron molybdates thin film as cathode material for lithium ion / sodium ion batteries	**OC 28B**
I. Hentech, A. Kabadou, K. Zehani, L. Bessais, A. Ben Salah, M. Loukil *FSS - Sfax* Synthesis, crystal structure, thermal and magnetic properties of CoMn $(HSeO_3)_2Cl_2.4H_2O$	**OC 08B**
N. Hfidhi, O. Kammoun, H. Naïli *FSS - Sfax* A 4-aminopyridinium based metal organic sulfate salts: Lamellar structure and supramolecular network	**OC 15A**
F. Hmida, B. Ayed, A. Haddad *FSM - Monastir* Synthesis, structure and characterization of an inorganic-organic hybrid dawson polyoxotungstate $Na_3(C_7N_2H_8)_3[P_2W_{18}O_{62}].16H_2O$	**OC 13A**

Name	**Ref**
H. Ibn Gharsallah, L. Escoda, J.J. Suñol, M. Khitouni *FSS - Sfax* Effect of the boron additions on the kinetic formation of Fe(Al) solid solution in nanostructured Fe_3Al alloys prepared by ball milling	**OC 07B**
R. Jlassi, W. Rekik, H. Naïli, A. P. C. Ribeiro, M. Fátima C. Guedes Da Silva, A. J. L. Pombeiro *FSS - Sfax* Template syntheses of copper(II) and cadmium(II) complexes from arylhydrazones of malononitrile and their catalytic activity towards alcohol oxidations	**OC 22A**
S. Kefi, D. Hellali, H. Zamali *FST - Tunis* Experimental investigation of the phase diagram of the binary system ($LiNO_3$+$TlNO_3$)	**OC 12B**
L. Khedhiri, E. Jeanneau, F. Lefebvre, M. Rzaigui, C. Ben Nasr *FSB - Bizerte* Synthesis and characterization of a new cyclohexaphosphate $(C_6H_7ClN)_6P_6O_{18.}$ $0.5(H_2O)$	**OC 06A**
M. Khemakhem, K. Lamnawar, A. Maazouz, M. Jaziri *ENIS - Sfax* Enhancement of physical and thermomecanical properties of polylactide with two types of core-shell rubbers	**OC 01B**
I. Labidi, S. Boughanmi, H. Tiss, A. Megriche *FST - Tunis* Hydration studies of different Tunisian HRS Portland cements	**OC 22B**
W. Ltaief, A. Mbarek, M. El-Ghozzi, D. Zambon, H. Naili *ENIS - Sfax* Synthesis, structural and luminescent proprieties of new lithium fluorophosphates $Li_xLn(PO_4)_{1-x}F_{4x}:Eu^{3+}$	**OC 12A**
A. Maatar, H. Naïli, T. Bataille, T. Mhiri *FSS - Sfax* Three noncentrosymmetric hybrid materials templated by racemic methylbenzylamine and it's enantiomorphic forms	**OC 10A**
N. Mahbouli Rhouma, A. Rayes, M. Loukil *FSS - Sfax* Synthesis, structural study and characterization of a new racemic non centrosymetric compound $[(C_2H_5)_4N]ZnI_3$	**OC 09A**

Name	**Ref**
A. Marzouki, H. Harzali, A. Megriche, K. Zehani, L. Bessais *FST - Tunis* Synthesis and characterization of ferroic nanomaterials $BiFe_{0.9}(Co_xMn_{(1-x)})_{0.1}O_3$	**OC 06B**
N. Nguili, J.J. Suñol, A. Bulou, M. Toumi *FSS - Sfax* Crystal structure and spectroscopic studies of $LiNH_4(H_2PO_4)_2$ A new solid acid in the LiH_2PO_4-$NH_4H_2PO_4$ system	**OC 08A**
W. Nouira, N. Jaffrezic-Renault, A. Maaref *FSM - Monastir* Conductometric enzyme biosensor based on proteinase K was developed using two types of nanoparticles	**OC 21A**
N. Nouiri, K. Jaouadi, N. Boudjada, T. Mhiri, N. Zouari *FSS - Sfax* Thermal study and superprotonic behavior of the crystal: $Rb_3(HSeO_4)_{2.5}(H_2PO_4)_{0.5}$	**OC 11A**
K. Nouri, M. Jemmali, S. Walha, K. Zehani, L. Bessais, A. Ben Salah *FSS - Sfax* Structural and magnetic properties of intermetallic compounds based on rare earth (Sm)	**OC 05B**
M. Oumezzine, A. C. Galca, Mo. Oumezzine, I. Pasuk, C. Chirila, A. Leca, A. Kuncser, C. Ghica, L. C. Tanase, C. M. Teodorescu, V. Kuncser *FSM - Monastir* Epitaxial growth of the electron doped manganites thin films through pulsed laser deposition	**OC 04B**
G. Rahal, B. Hamdi, R. Zouari *FSS - Sfax* Fer (III) Chloride Complex: Synthesis, structural study, vibrational and electrical properties of new compound $[C_7H_7N_2][FeCl_4]$	**OC 07A**
S. Rhouma, S. Said, A. Megriche, C. Autret-Lambert *FST - Tunis* Synthesis and characterization of $Ca_{1-x}Sr_xCu_{3-v}Ni_vTi_4O_{12}$ by solid-State	**OC 03B**
D. Rtibi, D. Hellali, H. Zamali *FST - Tunis* The phase diagram of (KNO_3+$TlNO_3$)	**OC 11B**

Name	**Ref**
I. Sahli, O. Ghodbane, M. Abdellaoui *FST - Tunis* Electrochemical Hydrogenation of $CeZr_2Cr_4Ni_5$ –based Alloys	**OC 19A**
S. Saïd, N. Mhadhbi, S. Elleuch, L. Tadeusz, T. Mhiri, H. Naïli *FSS - Sfax* Experimental and DFT characterizations of the monohydrated Co(II) complex with 2,6-diaminopyridine ligand	**OC 05A**
N. Salah, B. Hamdi, A. Ben Salah *FSS - Sfax* Hydrothermal synthesis and characterization of a new hybrid organic–inorganic compound $[Cu(en)]Cl_2$	**OC 04A**
M. Smida, M. Dammak, S. Garcia-Granda *FSS - Sfax* Hydrothermal synthesis, crystal structure and characterization of the new fluoroferrate bipyridine $Fe_2F_5(2,2'\text{-bipyridine})_2H_2O$	**OC 03A**
A. Souemti, L. Zayani, I. R. Martin, D. Ben Hassen Chehimi *FSB - Bizerte* Synthesis, characterization and optical study of a new langbeinite salts doped with Dy and Nd	**OC 02B**
A. Tounsi, B. Hamdi, R. Zouari, A. Ben Salah *FSS - Sfax* Crystal structure, Hirshfeld surface analysis, Vibrational spectroscopy and optical properties of a new Organic-Inorganic Hybrid Material: $[C_6H_{10}(NH_3)_2]CoCl_4 \cdot H_2O$	**OC 02A**
F. Walha, K. Lamnawar, A. Maazouz, M. Jaziri *ENIS - Sfax* The effect of a commercial melt strength enhancer additive on the physical and rheological properties of poly(lactic acid)	**OC 20A**
D. Zammel, I. Nagazi, A. Haddad *FSM - Monastir* Synthesis and characterization of a new organic–inorganic hybrid material based on isopolyoxomolybdate: $(C_2H_6N_4)_2[\beta\text{-}MO_8O_{26}] \cdot 4H_2O$	**OC 01A**

Oral Communications' Abstracts

Program of
Monday, December
21st 2015

SYNTHESIS AND CHARACTERIZATION OF A NEW ORGANIC–INORGANIC HYBRID MATERIAL BASED ON ISOPOLYOXOMOLYBDATE: $(C_2H_6N_4)_2[\beta\text{-}MO_8O_{26}]\cdot 4H_2O$.

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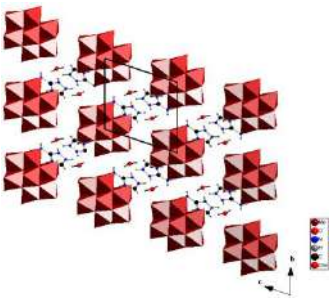
Polyoxometalates (POMs) are an immense class of metal–oxygen cluster compounds, which exhibit compositional diversity and structural versatility, as well as molecular identity in both the solid state and the solutions **[1]**. POMs, as a unique class of nanosized metal oxide clusters with abundant topologies and oxygen-rich surface, have exhibited remarkable properties in the field of catalysis, medicine and optics, etc. **[2–6]**. That's why a new polyoxometalate formulated as $(C_2H_6N_4)_2[\beta\text{-}Mo_8O_{26}]\cdot 4H_2O$ (1) has been isolated by conventional solution method and structurally characterized by single-crystal X-ray diffraction method, IR spectroscopy, UV-Vis absorption, thermogravimetric analysis and cyclic voltammetry measurements.

Compound (1) crystallizes in the Triclinic system, space group P1 with unit a = 8.348 (2) Å, b = 10.154 (2) Å, c = 10.823 (3) Å, α = 68.35° (2), β = 71.59° (2), ɤ = 78.55° (2), V = 805.5 (3) $Å^3$, Z = 2. The crystal structure of (1) is built up from an octamolybdate $[\beta\text{-}Mo_8O_{26}]^{4-}$ clusters connected through hydrogen-bonding interactions into a three-dimensional supramolecular network.



Packing view of compound (1) along the a axis, showing the arrangement of the $[Mo_8O_{26}]^{4-}$ clusters, $[C_2H_6N_4]^{2+}$ cations and the lattice water molecules in the crystal structure.

Keywords: Organic–inorganic hybrid materials, β-octamolybdate anion, 1, 2, 4-triazol-3-amine.

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Crystal structure, Hirshfeld surface analysis, Vibrational spectroscopy and optical properties of a new Organic-Inorganic Hybrid Material: $[C_6H_{10}(NH_3)_2]CoCl_4{\cdot}H_2O$

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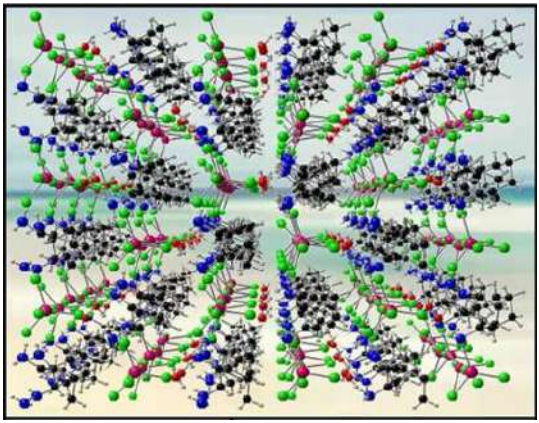
The present work accounts for a combined experimental and theoretical study on crystal structure and vibrational spectra of a new organic-inorganic hybrid compound. In fact, the single crystals $[C_6H_{10}(NH_3)_2]CoCl_4{\cdot}H_2O$, were obtained by slow evaporation of an aqueous solution at room temperature and crystallized in the non-centrosymmetric space group $Pna2_1$ of orthorhombic system. The infrared and Raman spectra of the title compound were recorded at room temperature and then analyzed. As for the vibrational spectra, they were also calculated theoretically by means of Gaussian 03 package of programs within the DFT framework using the B3LYP/LanL2DZ level of theory. The data thus obtained from vibrational wavenumber calculations were used to assign vibrational bands obtained in IR and Raman spectroscopy of the studied compound. Moreover, the optical study was also investigated by UV–vis absorption. The Hirshfeld surface and the breakdown of fingerprint plots were used for visualizing and exploring of title compound for quantifying intermolecular interactions in crystal lattice. The results also show that the title compound might have important NLO behavior and can be a potential new nonlinear optical (NLO) material of interest.



Perspective view of the structure

Hydrothermal synthesis, crystal structure and characterization of the new fluoroferrate bipyridine $Fe_2F_5(2,2'\text{-bipyridine})_2H_2O$

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A new metal-organic compound $Fe_2F_5(2,2'\text{-bipyridine})_2H_2O$ was hydrothermally synthesized yielded a new hybrid class II fluoroferrate. The structure was characterized by single crystal X-Ray diffraction data. The unit cell parameters with triclinic space group $P\bar{1}$, are: a=10.026(2)Å, b = 10.281(2)Å, c = 11.534(3)Å, α = 97.780(2)°, β = 102.755(2)°, γ = 107.952(2)°, V= 1076.28(4) $Å^3$ and Z = 2. The structure is built up by $Fe_4F_{10}N_8$ tetrahedral 'tetramers' connected by eight nitrogen atoms of four 2,2'-bipyridine molecules and separated by H_2O molecules. Thermal study by DSC and ATG shows that the total experimental mass loss value (68.5%) for $Fe_2F_5(2,2'\text{-bipyridine})_2H_2O$ is in good agreement for the theoretical mass loss (70.26%) assuming that the hematite is the final product of the decomposition. IR and magnetic characterization have been used and discussed for the title compound to confirm the presence of organic molecule 2,2'-bipyridine in the material and the paramagnetic compound, respectively.

Key words: Fluoroferrate, X-ray diffraction, Thermal analysis, Infrared.

Hydrothermal synthesis and characterization of a new hybrid organic–inorganic compound $[Cu(en)]Cl_2$

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A novel organic–inorganic hybrid material, $[Cu(en)]Cl_2$ (en:$C_2H_8N_2$) were grown by the hydrothermal technique. The X-ray crystal structure of (ethylenediamine)dichlorocopper(II) shows the compound crystallizes as a one dimensional chain with one chlorine non-bridging and the other chlorine triply bridging. It crystallizes at room temperature in the centrosymmetric space group $P2_1/m$. The copper surrounding is best described as distorted octahedron, the equatorial plane being built by three chlorines atoms (one triply bridging and the other μ^2 bridging). The rest of the copper coordination sphere is completed with the two nitrogen atoms of the chelating (en) and a non-bridging axial chlorine. The crystal structure was stabilized by an extensive network of N-H···Cl classical hydrogen bond interactions. The investigation of close intermolecular interactions between the molecules via Hirshfeld surface analyses is presented in order to reveal subtle differences and similarities in the crystal structures. The decomposition of the fingerprint plot area provides a percentage of each intermolecular interaction, allowing for a quantified analysis of close contacts within each crystal.

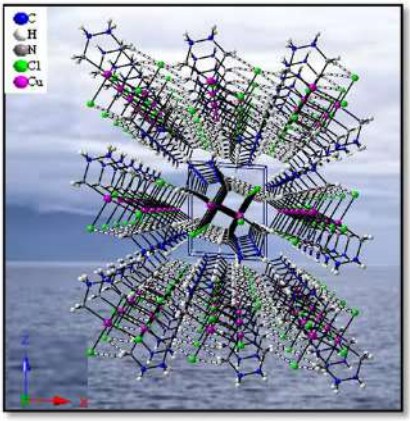

Fig. Sheet of compound $\{[Cu(en)]Cl_2\}_n$ parallel to the ab plane

Experimental and DFT characterizations of the monohydrated Co(II) complex with 2,6-diaminopyridine ligand.

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Single crystals of the new organic–inorganic compound $(C_5H_8N_3)_2[CoBr_4]\cdot H_2O$ were grown by hydrothermal technique and characterized by X-ray diffraction, infrared absorption, raman spectroscopy scattering, optical absorption and thermal analysis. The title compound belongs to the triclinic space group $P\bar{1}$ with the following unit cell parameters: a = 7.0482(2), b = 9.0578(3), c = 16.4723(4) Å, α = 82.912(2), β = 80.702(2) and γ = 69.075(3)°. In the crystal structure, the inorganic layers are built from tetrabromidocobaltate anions $[CoBr_4]^{2-}$ and free water molecules, linked together by O-H···Br hydrogen bonds and halogen···halogen interactions. The organic cations (2,6-HDAPY) are intercalated between the mineral layers via N-H···Br hydrogen bonds and form chains through π···π interactions. Therefore, the interlayer space is equal to the value of the b unit cell parameter. Theoretical calculations were performed using density functional theory (DFT) with the B3LYP/LanL2DZ level of theory for studying the molecular structure and vibrational spectra of the title compound. Good consistency is found between the calculated results and the experimental structure, IR and Raman spectra. The optical properties were investigated by optical absorption showing Ligand-to-Metal Charge-Transfer transitions (LMCT) in the visible region and organic related π–π* transitions in the UV region. The electronic spectrum of the cluster model was calculated by Time-Dependent Density Functional Theory (TD-DFT), which qualitatively reproduces the experimental transitions in the absorption spectrum of the title compound.

Key words: X-ray diffraction; Organic template; Metal halides; DFT calculations; Optical absorption and Thermal studies.

Synthesis and Characterization of a New Cyclohexaphosphate, $(C_6H_7ClN)_6P_6O_{18}.0.5(H_2O)$

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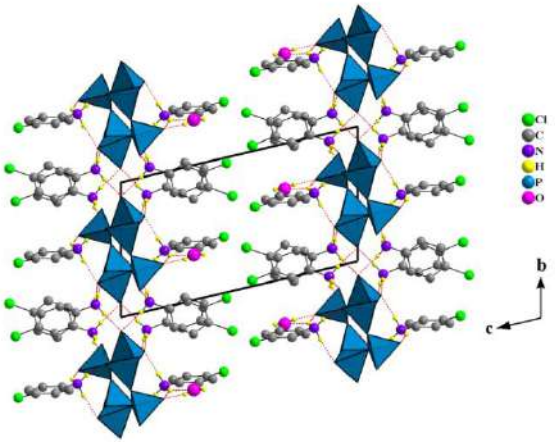
A new cyclohexaphosphate, of the composition $(C_6H_7ClN)_6P_6O_{18}0.5(H_2O)$, has been synthesized at room temperature in the presence of 4-chloroaniline as organic template and investigated by various physicochemical techniques. Its unit cell is triclinic P-1 with parameters a = 9.0054(8), b = 10.1053(9), c = 16.4454(14) Å, α = 100.476(7), β = 93.485(7), γ = 115.407(9) °, Z = 2 and V = 1313.0(2) Å^3. The structure involves a network of inorganic parallel layers built up from $P_6O_{18}^{6-}$ ring anions and water molecules. Charge balance is achieved by the protonated amine which is trapped in the interlayer space and interacts with the organic framework through strong hydrogen bonding. The ^{13}C, ^{15}N and ^{31}P CP-MAS NMR spectra are in 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 three-dimensional network of $(C_6H_7ClN)_6P_6O_{18}.0.5(H_2O)$, projected along the *a*-axis.

Fer (III) Chloride Complex: Synthesis, structural study, vibrational and electrical properties of new compound $[C_7H_7N_2][FeCl_4]$

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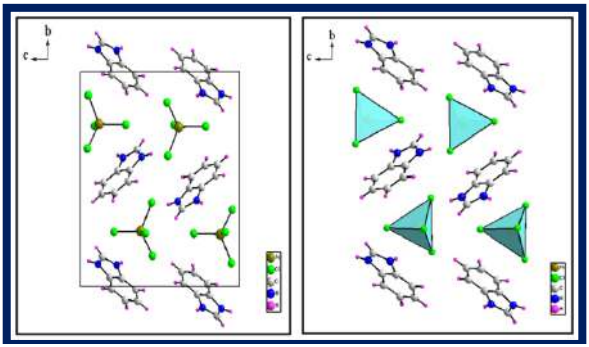
A new organic inorganic compound $[C_7H_7N_2][FeCl_4]$ has been synthesized and is characterized by a single -crystal X-ray diffraction, This compound crystallizes in the monoclinic system in $P2_1/c$ (N°: 14) space group With the following unit cell parameters a = 6.5311(7) (Å), b= 14.4270(13) (Å), c = 12.7011(11) (Å), β = 93.219(7)°,V = 1194.9(2) ($Å^3$), Z=4. Crystal structure was solved with a final reliability factor R = 0,0361for 2127 independent reflections. This vibrational bonds confirm the result find by X-ray diffraction.

This compound shows the presence of the organic cations as spacers between the inorganic isolated tetrahedral, the cohesion is assured by strong liaison assisted N–H···Cl interactions established between ammonium groups and discrete $[FeCl_4]^-$ tetrahedral and are combined by and π–π interactions which reinforce the association between cations.

Impedance spectroscopy study, reported in the sample, reveals that the conduction in the material is due to a hopping process. The temperature and frequency dependence of dielectric constants of the single crystal sample has been investigated to determine some related parameters to the dielectric relaxation.



Projection in the (b,c) plane of the atomic arrangement of the $[C_7H_7N_2][FeCl_4]$ compound.

Crystal structure and spectroscopic studies of $LiNH_4(H_2PO_4)_2$: A new solid acid in the LiH_2PO_4-$NH_4H_2PO_4$ system

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b *Department de Fisica, Universitat de Girona, Campus Montilivi, Girona 17071, Spain.*
c *LUNAM Université, Université du Maine, CNRS UMR 6283, Institut des Molécules et Matériaux du Mans (IMMM), Avenue Olivier Messiaen, 72085 Le Mans Cedex, France.*

A new solid acid of lithium ammonium bis-dihydrogeno phosphate LiNH4(H2PO4)2 (LADP) has been synthesized, from the NH4H2PO4 – LiH2PO4 system, by slow evaporation and its structure is determined by single crystal X-ray diffraction. The crystals are also characterized by the vibrational properties measured by Raman spectroscopy at room temperature in the wavenumbers 50 – 4000 cm^{-1}. It's found that the compound crystallizes in the monoclinic system with space group (P21/a, n°14) and with the following parameters a = 7.6811(19), b = 12.900(3) and c = 7.5078(18) Å, β = 100.092(4)° and Z = 4. The structure is characterized by LiO4 tetrahedra linked by corners with four H2PO4 groups to form infinite layer parallel to (001) plane. Each layer is connected by isolated NH_4 groups that form sheets parallel to the same plane giving a three dimensional network.
The Raman spectra show the vibration modes of H2PO4 and NH_4 groups.

Keywords: X-ray diffraction, Raman spectroscopy, dihydrogenophosphate.

SYNTHESIS, STRUCTURAL STUDY AND CHARACTERIZATION OF A NEW RACEMIC NON CENTROSYMETRIC COMPOUND $[(C_2H_5)_4N]ZnI_3$

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A new organic-inorganic non-centrosymmetric (NCS) polar zinc iodate, $[(C_2H_5)_4N]ZnI_3$, has been synthesized at room temperature and characterized by X-ray single crystal diffraction. The compound crystallizes in the tetragonal system with P-421m space group and the following lattice parameters a = 13.7431 Å, b = 13.7431 Å, c = 14.7852 Å, V = 2792.52 $Å^3$ and Z = 4. The asymmetric unit of the title compound contains only one type of ZnI_4 tetrahedral and three types of tetraethylammonim cation, denoted $[(C_2H_5)_4N(1)]_{0.25}$, $[(C_2H_5)_4N(2)]_{0.5}$ and $[(C_2H_5)_4N(3)]_{0.25}$ is shown in fig (1). The atomic arrangement shows an alternation of organic and inorganic entities. The cohesion between these entities is performed via both Van Der Waals and electrostatic interactions between tetaethylammonium cations and tetraiodozincate anions.

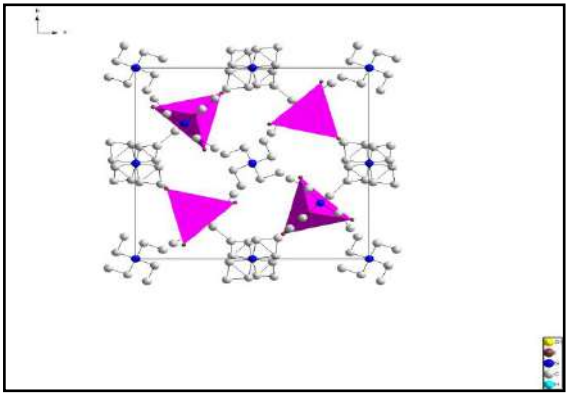

Fig1: Crystal packing of $[(C_2H_5)_4N]ZnI_3$ compound along the ac plane.

THREE NONCENTROSYMMETRIC HYBRID MATERIALS TEMPLATED BY RACEMIC METHYLBENZYLAMINE AND IT'S ENANTIOMORPHIC FORMS

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Single crystals of three novel layered inorganic–organic hybrids $[R\text{-}(C_8H_{12}N)_2][CuCl_4]$ (**1**), $[S\text{-}(C_8H_{12}N)_2][CuCl_4]$ (**2**) and $(C_8H_{12}N)_2[CuCl_4]$ (**3**) were grown by slow evaporation solution technique at room temperature through the use of copper chloride as the inorganic motif and enantiomorphically pure sources of either (R)-α-methylbenzylamine or (S)-α-methylbenzylamine as well as the racemic (RS) form as the organic constituent. The crystal structures were solved in the monoclinic symmetry, space group C2 for compounds **1** and **2,** and in the orthorhombic symmetry, space group C2cb for compound **3** (it was possible to solve the structure in the standard space group, Aba2, with cell transformation). The supramolecular crystal structure of these materials is built from the alternatively arranged inorganic and organic layers and stabilized by N—H···Cl hydrogen bonds between the inorganic and organic moieties and C-H···π interactions between the aromatic rings of the organic moieties themselves. These structures were determined using single-crystal X-ray diffraction, infrared spectroscopy, and thermal analysis.

Key words: Hybrid; layered; enantiomorphically pure; Crystal structure; X-ray diffraction; Thermal analysis.

Thermal study and superprotonic behavior of the crystal: $Rb_3(HSeO_4)_{2.5}(H_2PO_4)_{0.5}$

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Crystals of the new compound $Rb_3(HSeO_4)_{2.5}(H_2PO_4)_{0.5}$, have been synthesized for the first time by a slow evaporation method at room temperature. Thermal-differential analysis of the superprotonic transition in $Rb_3(HSeO_4)_{2.5}(H_2PO_4)_{0.5}$ showed that the transformation to high-temperature phase occurs at 374 K by one-step process. Thermal decomposition of the product takes place at much higher temperatures, with an onset of approximately 510 K.

The superprotonic transition was also studied by impedance and modulus spectroscopy techniques. The conductivity in the high temperature phase attained a value superior than 10^{-4} Ω^{-1} cm^{-1}, and the activation energy for the proton transport lower than 0.4eV. The conductivity relaxation parameters associated with the high disorder protonic conduction have been examined from analysis of the M''/M''max spectrum measured in a wide temperature range. Transport properties in this material appear to be due to proton hopping mechanism.

Keywords: $Rb_3(HSeO_4)_{2.5}(H_2PO_4)_{0.5}$; superprotonic phase transition; conductivity and Thermal behaviour.

SYNTHESIS, STRUCTURAL AND LUMINESCENT PROPRIETIES OF NEW LITHIUM FLUOROPHOSPHATES $Li_xLn(PO_4)_{1-x}F_{4x}:Eu^{3+}$

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[b)] Laboratoire des matériaux inorganiques, Université Blaise Pascal, Institut de Chimie de Clermont-Ferrand, BP 10448, F-63000 CLERMONT-FERRAND, France.
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Rare earth phosphors compounds have attracted much attention due their potential application as ionic conductors, optical display panel, mercury free lamp, field emission and high luminescence efficiency. In this work, we have developed a new family of fluorophosphates of general formula $Li_xLn(PO_4)_{1-x}F_{4x}$, resulting from the combination of fluoride $LiLnF_4$ stand orthophosphate $LnPO_4$ (Ln= Y, Gd and Eu). The obtained materials were characterized by X-ray diffraction XRD, NMR (^{31}P, ^{7}Li and ^{19}F) and Raman-IR spectroscopes. For optical measurements, Eu^{3+} doped samples $Li_xLn_{x'}Eu_{1-x'}(PO_4)_{1-x}F_{4x}$ were obtained by substituting the Eu^{3+} ions ones using appropriate amounts of europium fluoride $LiEuF_4$.

Key words: fluorophosphates, Rare earth, luminescence.

SYNTHESIS, STRUCTURE AND CHARACTERIZATION OF AN INORGANIC-ORGANIC HYBRID DAWSON POLYOXOTUNGSTATE $Na_3(C_7N_2H_8)_3[P_2W_{18}O_{62}].16H_2O$

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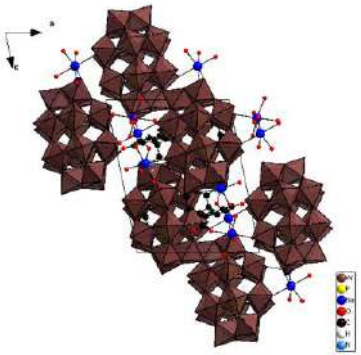
There exists growing interest in metal oxide clusters, or polyoxometalates, reflecting their diverse properties, which endow them with applications in the fields such as catalysis, medicine, analytical chemistry and photochemistry [1–4]. That's why a new inorganic-organic hybrid Wells-Dawson type polyoxotungstate, $Na_3(C_7N_2H_8)_3[P_2W_{18}O_{62}].16H_2O$ (1), has been synthesized under hydrothermal conditions and characterized by IR spectroscopy, UV spectroscopy, TG analysis, cyclic voltammetry and single crystal X-ray structural analysis.
The compound $Na_3(C_7N_2H_8)_3[P_2W_{18}O_{62}].16H_2O$ is a three-dimensional network composed of $[P_2W_{18}O_{62}]^{6-}$ polyanions, $(C_7N_2H_8)^+$ and Na^+ cations, and water molecules. 1 crystallizes as a triclinic system, in P^{-1} space group; with a = 14.063(1) Å, b = 17.009(1) Å, c = 17.868(1) Å, α = 79.882(1)°, β = 77.139(1) °, γ = 79.252(1)°, V = 4053.40Å^3 and Z =2.



A packing view of $Na_3(C_7N_2H_8)_3[P_2W_{18}O_{62}].16H_2O$ along the b axis

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

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SYNTHESIS, PHYSICOCHEMICAL STUDY AND STRUCTURE CHARACTERIZATION OF A NEW ACIDIC CYCLOHEXAPHOSPHATE $[O\text{-}(OCH_3)C_6H_4NH_3]_4.H_2P_6O_{18}.2H_2O$

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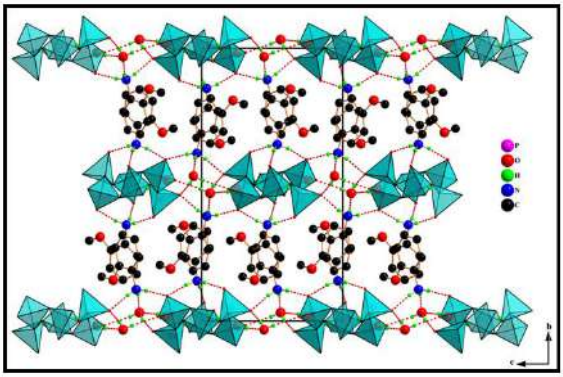
The literature shows that many research works have dealt with the characterization and properties of organic-inorganic materials with open structures which can optionally have applications. These compounds represent an advanced field in material science. Hybrid phosphates occupy an important position for their specific physical and chemical properties. As a contribution towards in search of these materials, we report in this communication the development and study of a new acidic, organic, cyclohexaphosphate $[O\text{-}(OCH_3)C_6H_4NH_3]_4H_2P_6O_{18}.2H_2O$. This compound crystallizes in the monoclinic $P2_{1/n}$ unit cell with a = 10.378(3) Å, b = 20.180(5) Å, c = 10.625(3) Å, β = 95.46 °(2), V = 2215.1(10) $Å^3$ and Z = 2. It exhibits a centrosymmetric atomic arrangement where $H_2P_6O_{18}^{4-}$ anions form layers parallel to the (a, c) plane at y = 0 and y = 1/2. The organic molecules are arranged in the interlayer space for ensuring the cohesion of the crystalline network by different types of interactions (electrostatic, Van der Waals, H-bonds) as shown in the projection along the $\vec{a}$ axis. Thermal analysis shows that this compound is thermally stable up to 420 K. Solid state NMR study of ^{31}P confirm X-ray diffraction results in terms of phosphoric sites and chemical environment.



Projection of the structure of $\mathbf{[o\text{-}(OCH_3)\ C_6H_4NH_3]_4\ H_2P_6O_{18}.2H_2O}$
Along the $\vec{a}$ axis.

Keywords: Cyclohexaphosphate, Hybrid materials, X-ray diffraction, Crystal structure.

Enhancement of physical and thermomecanical properties of polylactide with two types of core-shell rubbers

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The morphological, thermal and rheological behaviors of toughened polylactide (PLA) modified with two types of core-shell rubber particles (i.e., Paraloid EXL2330 and EXL2314) were studied. These rubber particles were based on (polybutylacrylate) core and (polymethylmethacrylate) shell.
Thermal analyses showed that both the EXL2330 and EXL2314 additives decreased the ability of PLA to crystallize. The rheological tests indicated that zero-shear viscosity and storage modulus of the PLA/EXL2330 and PLA/EXL2314 blends were significantly increased with the additive content.
These could be attributed to the entanglements between the PLA chains with those of the high molecular weight additive, creating a physical network which reduces the segmental mobility of PLA and improves the melt elasticity of the blend.

Key words: core-shell particles; polylactide; thermomecanical properties; rheological properties

Synthesis, characterization and optical study of a new langbeinite salts doped with Dy and Nd

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Langbeinite salts $K_2Mg_2(SO_4)_3$:Ln^{3+} (Ln^{3+} = Dy^{3+}, Yb^{3+}) in their cubic system were prepared by high temperature solid state reaction technique. The powder X-ray diffraction confirmed the high purity of the obtained phases. They crystallized in the cubic system with space group $P2_13$. Many spectroscopic measurements were done for the synthesized samples: The UV-VIS-NIR absorption of Dy^{3+} in this matrix revealed the transitions from the ground state $^6H_{15/2}$ to different excited levels. The emission spectra in the visible region shown four bands originated from the $^4F_{9/2}$ level to the $^6H_{15/2}$, $^6H_{13/2,}$ $^6H_{11/2}$ and $^6H_{9/2}$+$^6F_{11/2}$ levels of Dy^{3+} ions. Moreover, the lifetime dependence of this emitting level can be explained due to a thermalization effect. For the Yb^{3+}, emission luminescence verifies the $^2F_{5/2}\rightarrow{}^2F_{7/2}$transition. Cooperative emissions are observed at 476 nm due to blue up-conversion after powered the sample.

Key words: Langbeinite salts, Spectroscopy, Optical properties, Thermalization effect, Cooperative emission.

Synthesis and characterization of $Ca_{1-x}Sr_xCu_{3-y}Ni_yTi_4O_{12}$ by solid-State

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Recently, a new material has been discovered by Subramanian et al, named $CaCu_3Ti_4O_{12}$ (CCTO) [1]. This material presents a giant permittivity (10^4-10^5) and shows nearly temperature independent behavior in the temperature and frequency range 100–400K and 100–100 Hz respectively [2, 3].

In this work, the $Ca_{1-x}Sr_xCu_{3-y}Ni_yTi_4O_{12}$ (x=0.1-0.15 and y=0.1-0.15) ceramics were prepared by the conventional solid state reaction method. The structural study reveals that X-ray powder diffraction analysis confirmed the formation of cubic CCTO phase. Scanning electron microscopy (SEM) results show that doping enhanced the grain growth and appeared large grains surrounded by small grains. Dielectric proprieties of $Ca_{1-x}Sr_xCu_{3-y}Ni_yTi_4O_{12}$ reached a value as high as $4\ 10^5$.

As perspective, we will study the evolution of the permittivity as a function of temperature.

Key words: CCTO ceramics, Chemical synthesis, SEM, Dielectric proprieties

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Epitaxial growth of the electron doped manganites thin films through pulsed laser deposition

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Perovskite manganites, $R_{1-x}A_x$ $(Mn^{3+}{}_{1-x}Mn^{4+}{}_x)O^3$ (R–trivalent rare earth, A-divalent alkaline) have attracted considerable attention due to their magnetocaloric and/or magnetoelectric properties. With this respect, they are potential candidates to be used in various applications such as: electronics, spintronics, magnetic refrigeration, field sensors, magnetic information storage or faster reading devices. Downscaling perovskites is prerequisite for integrated them in nowadays miniaturized devices. Another important aspect regards the best/good functionality at room temperature. From the industrial application's perspective, these materials have to be prepared in the form of thin films. For this reason, much work has been done to obtain films with the best structure and physical properties. It has been suggested that the electrical and magnetic properties of CMR manganite films are closely associated with thin film preparation technology and the preparation condition.
In this work, the growth conditions of low doping non-magnetic ion Ti^{4+} replacing Mn^{4+} in $La_{0.67}Ba_{0.33}MnO_3$ thin films were optimized on $SrTiO_3$ substrate which has the lattice matching (+0.5%). The thin films grew epitaxially as proven by X-ray diffraction and transmission electron microscopy. The analysis of XPS spectra gives a stoichiometry close to the one of the ceramic target.

Keywords: Epitaxial growth; Manganite thin film, Lattice mismatch, giant magnetoresistance

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Structural and magnetic properties of intermetallic compounds based on rare earth (Sm)

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Structural, magnetic and magnetocaloric properties of $SmNi_2$ powder sample has been investigated by X-ray diffraction (XRD) and magnetic measurements. Our sample has been synthesized using an arc furnace. The Rietveld refinements and energy dispersive X-ray spectroscopy (EDS) detector show that the $SmNi_2$ intermetallic was single phase and crystallize in the $MgCu_2$-type cubic Laves phase structure (C15-type, Space group Fd-3m, number 227). The X-ray diffraction shows that the Sm_2Fe_{17} compound crystallizes in the rhombohedral system R-3m (Th_2Zn_{17} type structure) with the following cell parameters are: a = 8.5796 (3) Å and c = 12.4624 (2) Å. The hyperfine parameter was determined by the correlation between the volume of Wigner-Seitz V and isomeric shifts. Based on the sites volume of each iron atoms, which has the volume of the largest Wigner-Seitz has the largest of isomer shift [1-2]. So as V6>V18h>V18f>V9d, their corresponding isomeric will be as follows δ6c> δ18h> δ18f> δ9d. Their magnetic properties and magnetocaloric effect (MCE) have been studied by the magnetization and isothermal magnetization of different temperature measurements. It is found that the sample obeys to the second-order magnetic transition (SOMT) from ferromagnetic(FM) to paramagnetic(PM) state at the Curie temperatures T_c= 21 K. Using the thermodynamic Maxwell's relation, magnetic entropy change is estimated and discussed in terms of MCE. The maximal values of magnetic entropy change and the relative cooling power (RCP) for $SmNi_2$ are 1.22 J/Kg.K and 15.25 J/Kg.K, respectively, around their T_C with negligible thermal hysteresis for the field 0.05 T and weak magnetic hysteresis at low temperature. The results suggest that our alloy may be appropriate candidate for magnetic refrigerant working at low temperature region 10–100 K.

Key words: rare earth, Magnetic and magnetocaloric measurements, X-ray diffraction

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Synthesis and characterization of ferroic nanomaterials $BiFe_{0.9}(Co_xMn_{(1-x)})_{0.1}O_3$

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$BiFeO_3$ (BFO) is considered as the prototypical multiferroic oxide with the perovskite structure (ABO_3) and has intensively been studied because of its high ferroelectric–paraelectric transition temperature (T_C = 1083 K) and high antiferromagnetic–paramagnetic transition temperature (G-type, T_N = 634 K) [1-3]. The magnetoelectric effect (ME) of this multiferroic material involve significant potential in technological applications of the multifunctional devices such as the transistor gate at high temperature, the memory devices and the electronics spin.
In this work, we are particularly interested in the synthesis of $BiFe_{0.9}(Co_xMn_{(1-x)})_{0.1}O_3$ by hydrothermal method and the study of the effect of co-substitution on BFO properties. The perovskite structure belonging to the space group R3c of $BiFe_{0.9}Co_xMn_yO_3$ crystallites was confirmed by X-ray diffraction analysis and the variation of crystallite sizes as function of percentage substitution has been studied by the Scherrer method. Differential thermal analysis (DTA) of $BiFe_{0.9}(Co_xMn_{(1-x)})_{0.1}O_3$ nanopowders was used to the confirmation of the ferroelectric behavior of the samples. Infrared spectra allowed us to determine the different absorbance bands of our compounds. A magnetic study was focused on the variation of the magnetization as a function of applied magnetic field and the variation of samples compositions. Finally, as perspectives, we plan to investigate the variation of electrical polarization as function of applied electric field and to study the magnetoelectric effect of our samples.

Key words: Multiferroic, nanotechnology, magnetism, ferroelectricity.

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Effect of the boron additions on the kinetic formation of Fe(Al) solid solution in nanostructured Fe3Al alloys prepared by ball milling

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In the present work, structural and morphological properties of nanocrystalline Fe_3Al+X wt % B (X=0, 0.1, 0.2, 0.3, 0.5 and 0.7) powders produced by mechanical alloying (MA) were investigated by X-ray diffraction and scanning electron microscopy. The patterns so obtained were analyzed using the Rietveld program. The results show the presence of two crystalline phases: the majority one corresponds to bcc-FeAl phase and another in low proportion corresponds to Fe_2B phase type. This later appeared due to the presence of boron. The crystallite size reduction to the nanometer scale is accompanied by an increase in lattice strains. Increasing the boron content addition causes a progressive decrease in the lattice parameter. This behavior can be explained by the fracture phenomenon that intensifies with the age hardening of the alloy by the boron addition. In the case of the tetragonal Fe_2B phase, there is no apparent dependency of its microstructural parameters with the added boron content or the milling time. In addition, on can see a delay in the formation of Fe(Al) solid solution during the mechanical alloying.

Keywords: mechanical alloying, X-ray diffraction, Nanostructure, Fe-Al alloy.

Synthesis, crystal structure, thermal and magnetic properties of CoMn $(HSeO_3)_2Cl_2.4H_2O$

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The new compound $CoMn(HSeO_3)_2Cl_2.4H_2O$ (CoMnSe) has been synthesized by slow evaporation method. Their crystal structure has been determined by using single crystal X-ray diffraction. The CoMnSe compound crystallizes in the orthorhombic Pnma space group with the following lattice parameters: a = 9.1074(3) Å, b = 17.9950(5) Å, c = 7.1610(2) Å and Z = 4. The new compound are isostructural to Cu $(HSeO_3)_2Cl_2.4H_2O$ **[1]**. This structure is three-dimensional made of layers, parallel to the (010) plane, which contain Mn /Co atoms and $HSeO_3^-$ anions. The substitution of the Cu^{2+} by Mn^{2+} and Co^{2+} in acid-base conditions well determined (PH value), this substitution didn't changed the type structure but has been an effect on the unit cell volume and also influences in the magnetic properties of our compound. The grown crystal was characterized by FT-IR spectra, which were recorded in the 4000-400 cm^{-1} ranges. Also this compound was analysis by differential scanning calorimetry DSC and TG-DTA measurement. The measurement of the magnetization as a function of the temperature (M (T)) of CoMnSe compound indicates a ferromagnetic transition at the Curie temperature Tc = 19.5K. In the paramagnetic region of 50-300 K, the magnetic susceptibility follows a Curie-Weiss modified. The negative Weiss constant implies the existence of an antiferromagnetic interaction at low temperature **[2, 3]**.

Key words: single crystal X-ray diffraction, Magnetic measurements, Thermal measurements and Infrared spectrum

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Structural and magnetic properties of $Pr_{0.5-x}Gd_xSr_{0.5}MnO_3$ manganites

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We have investigated the effect of gadolinium doping on the structural and magnetic properties in $Pr_{0.5-x}Gd_xSr_{0.5}MnO_3$ manganites prepared by the solid state reaction method. Rietveld refinement of the X- ray diffraction patterns shows that all our samples are single phase and crystallize in the orthorhombic structure with Pbnm space group**.** Magnetic measurements versus temperature in an applied magnetic field of 0.05T show that the all samples exhibit a paramagnetic to ferromagnetic transition when temperature decreases. The x=0.1 sample shows a clear transition, from the ferromagnetic state to the antiferromagnetic one at T_N =125 K. The absolute values of the maximum of magnetic entropy change $|\Delta S_M|$ for an applied magnetic field of 2 T are 1.26, 1.23 and 1.041J kg^{-1} K^{-1} for x = 0, 0.05 and 0.1 respectively at the Curie temperature.

Chracterization of ZnO thin film grown on vicinal-cut sapphire substrates α-Al_2O_3 (0001) (α<0.1°) by MOCVD

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In recent years, ZnO thin films have attracted much attention because of their potential applications such as transparent electrodes, solar cells, surface acoustic wave devices, optical wave guides, varistors, LDs, LEDs, etc… Many different deposition methods have been used to grow ZnO thin films on various substrates such as sapphire, silicon, GaAs, and glass. Among them, the sapphire substrate plays an important role in applying ZnO thin films to optoelectronic devices. Despite the large differences in the thermal expansion coefficient and lattice constant between the ZnO thin films and sapphire substrates, it's widely used as a substrate because of its physical robustness and high-temperature stability. It is now well recognized that the surface treatment of the substrate prior to growth has crucial effects on both the growth mechanism and the material properties. Furthermore, there are few reports on the surface treatment effects of sapphire substrates on the epitaxial ZnO thin films. Among the stable surface orientations, the c-plane Al_2O_3 (0001) substrate is so far the most widely studied and most commonly used surface. These templates have recently attracted additional interest as they offer the possibility of reconstructing their surfaces into a hill and valley structure [1]. It was demonstrated that annealing of the (0001) surface can produce periodic step-and-terrace arrangements with a step height h= 1.2 nm [2]. The step rearrangements of c-plane -Al2O3 have been previously investigated as a function of parameters such as annealing temperature [2, 3], time [1, 3], pressure [4], off-cut angle [3, 5] and orientation [3].
In this work, we report epitaxial growth by metal organic chemical vapor deposition (MO-CVD) of ZnO films on c-plane (0001) sapphire substrate with a nominally vicinal-cut angle α <0.1°, which are prior annealed at 1000, 1050 and 1100 °C for 3 h under oxygen. We are interested on the influence of substrates annealing temperature on the microstructural, morphological and optical properties of ZnO thin films.

Keywords: ZnO Films, MOCVD, HRXRD, AFM, MEB et Photoluminescence.

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THE PHASE DIAGRAM OF (KNO_3+$TlNO_3$)

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Nowadays, the use of renewable energy has been the subject of much research. Alkali nitrates have a great interest in this area, especially in the storage and transfer of solar energy. These salts are characterized by low melting points, relatively high transition enthalpies and good thermal conductivities. Thallium nitrate, often encountered in industrial processes, has great similarities with alkali nitrates and it is interesting to associate it with the study of the latter.

The study of phase diagrams of the binary and/or of higher order systems is of great importance. Indeed, it allows to know compositions having low melting temperatures (such as: eutectic composition).

So, we are interested in this work to investigate experimentally, the phase diagram of the isobaric binary (KNO_3 + $TlNO_3$) system in the range 300 to 700 K. We used the techniques of thermal analysis STA (Simple Thermal Analysis), DTA (Differential Thermal Analysis), DSC (Differential Scanning Calorimetry) and XRD (X-Ray Diffraction).

The obtained phase diagram is characterized by four invariants: two eutectoids, a peritectoid and an eutectic at 382 K, 409 K, 352 K and 457 K respectively.

Key words: Potassium nitrate, Thallium nitrate, Thermal analysis, XRD, Phase diagram

EXPERIMENTAL INVESTIGATION OF THE PHASE DIAGRAM OF THE BINARY SYSTEM ($LiNO_3$+$TlNO_3$)

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Alkali and pseudo-alkali nitrates and their mixtures are of interest in both fundamental and industrial research. They have properties that allow them to be used in various fields such as chemical fertilizer industry, military engineering and in the storage and transfer of solar energy. Because of their relatively low melting temperatures, they are often used as molten salt baths in the metallurgy, calorimetry and electrochemistry

All these applications require knowledge of their thermodynamic properties such as phase diagrams of their binary systems and/or higher order. Indeed, they summarize in a condensed way the thermodynamic data of the considered system. But there is dispersion in the experimental results reported by different authors. The phase diagram of the binary system ($LiNO_3$+$TlNO_3$) has been the subject of some works leading to different results. The purpose of this study is to investigate experimentally, at atmospheric pressure, solid/solid and solid/liquid equilibria in the ($LiNO_3$ + $TlNO_3$) phase diagram. The techniques of simultaneous simple and differential thermal analysis (ATS/ATD), differential scanning calorimetry (DSC) and X-ray diffraction (DRX) have been used for this purpose.

This diagram is characterized by a single point eutectic at 404 K.

Key words: Alkali nitrates, phase diagrams.

A new glance at physico-chemical comportments of silicate bioactive glasses - influence of partial substitution: Borosilicate versus Fluorosilicate glasses

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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. 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. X-ray analysis showed that the introducing of these elements in the chemical composition does not affect the amorphous character of glasses. The FTIR spectra revealed that the main structural units in the glass network were predominantly silicate species. The presence of phosphate and borate units in the structure of the glasses was also evident in these spectra. The presence of fluorine is assigned to the displacement in infrared spectra of the Si-O vibration mode. These results were confirmed by EDS spectra. SEM images show the similar morphological aspects of fluoride glasses versus borate glasses surface. A significant difference in thermal behavior between the two types of glasses was approved. This study allows the tailoring of novel fluoride and borate-containing bioactive glasses to address orthopedic needs.

Key words: Bioactive Glasses, Boron, Fluorine, Physical and Chemical Characteristics.

STANDARD ENTHALPY, ENTROPY AND GIBBS FREE ENERGY OF FORMATION OF MAGNESIUM CARBONATE CO-SUBSTITUTED FLUORAPATITES

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Magnesium-carbonate co-substituted fluorapatites (Mg-CO_3-Faps), with similar Mg/Ca+Mg molar ratio of 0.1 and different amount of carbonate, have been prepared in this study by a double decomposition method. The as-prepared powders were characterized by X-ray diffraction, infrared spectroscopy, and chemical analysis.

Using an isoperibol calorimeter, the heat of solution of these products was measured at 298 K in 3 wt% chlorhydric acid solution. Thermochemical cycle was proposed and complementary experiences were performed in order to get the standard enthalpy of formation of the studied apatites.

Estimation of the values of entropy of formation allowed the determination of standard Gibbs free energies of formation of these compounds. The results showed that simultaneous incorporation of magnesium and carbonate ions results in a decrease of the stability of the apatite structure.

Key words: Magnesium-carbonate co-substituted fluorapatites, Enthalpy of solution, Enthalpy of formation, Entropy of formation, Gibbs free energy of formation

Program of Tuesday, December 22nd 2015

A 4-AMINOPYRIDINIUM BASED METAL ORGANIC SULFATE SALTS: LAMELLAR STRUCTURE AND SUPRAMOLECULAR NETWORK

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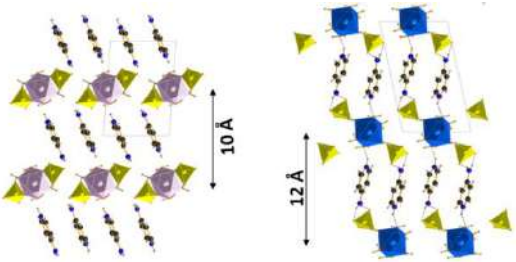
In the field of hybrid materials, compounds that posses layered crystal structure consisting in stacked inorganic sheets with intercalated inorganic and/or organic guests, have attracted a great attention due to their potential applications, such as nonlinear optical materials, conductors, photoactive materials, nanomagnets, polymer additives, ion-exchangers, electrodes and catalysis [1]. Nevertheless, materials having supramolecular crystal structures governed by weak bonding energy in three lattice directions are one of the most studied and used, namely organic-inorganic sulfate salts builting from transition metals and amines that have been mainly described in the literature as belonging to well-known families as alums and Tutton's salts [2].

In this context, we designed a novel structure topology that gathers between the supramolecular aspect and the lamellar aspect, which is not very much encountered in the literature. Indeed, we present a new class of layered transition metal sulfates, for which interlayer spacing confines chains of aromatic amine connected together through extended π-stacking networks that have a relevant role in stabilizing the lamellar structures. Interlayer distances are modified according to the metal, which leads to accessible vacancies allowing possible catalytic reactions.



Key words: hybrid material, lamellar structure, supramolecular, sulfate salt

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Combined theoretical and experimental studies on the molecular structure, spectral and Hirshfeld surface studies of NLO 1,4-Diazabicyclo[2.2.2]octane hydroxonium crystals

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Transparent single crystals of 1,4-Diazabicyclo[2.2.2]octane hydroxonium were grown by slow evaporation technique at room temperature from an aqueous. The experimental and theoretical studies on the molecular structure and vibrational spectraof ZTS were investigated by single crystal X-ray diffraction, FT-IR and density functional theory (DFT).have been synthesized and characterized by X-ray structural studies and the electronic structure was calculated at the DFT level with a detailed analysis of Hirshfeld surface and fingerprint plot facilitating a comparison of intermolecular interactions. The crystal packing of (3) exhibits intermolecular O–H---O and C–H---O hydrogen bonds forming linear chains propagating parallel to [1 0 0] and [0 1 0] directions. Investigation of intermolecular interactions and crystal packing via Hirshfeld surface analysis reveals that more than two-thirds of the close contacts are associated with weak interactions.

Hirshfeld surface analysis for visually analyzing intermolecular interactions in crystal structures employing molecular surface contours and 2D fingerprint plots have been used to examine molecular shapes.

Keywords: NLO material, FT-IR, TD-DFT, Hyperpolarizability, Hirshfeld analysis, XRD

Synthesis, Structural and vibrational studies of the new mixed solid solution thallium ammonium sulfate selenate tellurate

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The new mixed solid solution $Tl_{0.92}(NH_4)_{0.08}(SO_4)_{0.65}(SeO_4)_{0.35}Te(OH)_6$ crystallizes in the monoclinic system with space group $P2_1/c$. It was analyzed at room temperature using X-ray diffractometer data. The unit cell parameters are: a= 12.336(2) Å, b= 7.203(14) Å, c= 12.017(2) Å, β= 110.631(10)°, V= 999.4(3) $Å^3$, Dx= 5.152gcm^{-3} and Z= 4.

The structure can be regarded as being built of isolated TeO_6 octahedra, S/SeO_4 tetrahedra and Tl^+/ NH_4^+ cations. The main feature of this structure is the coexistence of two types of hydrogen bonds O–H…O and N–H…O ensuring the cohesion of the crystal. The Raman and IR spectra recorded at room temperature, in the frequency ranges (50–1200cm^{-1}) and (400–4000cm^{-1}), respectively, confirm the presence of SO_4^{2-}, SeO_4^{2-} and TeO_6^{6-}, groups in the crystal independently.

THE IMPACT OF PYRENE ON THE RETENTION OF LEAD IN SOIL

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The fate of heavy metals in soils with co-contaminants of polycyclic aromatic hydrocarbons is worthy to be elucidated. This study examined sorption of lead as a representative of heavy metals by a soil contaminated with pyrene using batch experiments and saturated column tests. Lead and pyrene were chosen as model pollutants because of their toxicity and abundance in contaminated soils. The soil used in the laboratory experiments is constituted from mixture of Hostun sand (96.7%), clay (3%) and compost used as a source of organic matter (0.3%). In a first step**,** sorption isotherms of the system lead–soil were obtained by carrying out batch experiments in two ways: sorption of lead alone and sorption of lead with pyrene at three different concentrations (46.5 µg/L; 150 µg/L and 250 µg/L). In all cases, results fit well with Langmuir isotherm [1]. Langmuir parameters were identified. Results clearly indicate that sorption was lowered by pyrene addition. The distribution coefficient (k_d) for lead sorption by the model soil spiked with pyrene was 23% lower than that by unspiked one.

In a second step, soil column experiments in saturated regime were carried out. The experimental device consists of a Plexiglas column with a height of 6 cm and a diameter of 2 cm. The column was filled with dry soil, then saturated by water-methanol mixture from bottom to top at 1.4 mL/min. After equilibrium, lead solution were percolated through of columnsoil at the same flow. Lead concentrations were measured in the eluted fractions. Two solutions were prepared: lead solution at a concentration of 200 mg/L and the second containing the same lead content added with pyrene at a concentration of 150 µg/L. Results show no effect of pyrene on lead sorption at this concentration. The mass of sorbed lead is the same in the presence and absence of pyrene.

Key words: heavy metals/polycyclic aromatic hydrocarbons/ co-contaminants/batch experiments/saturated column tests.

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Electrochemical Hydrogenation of $CeZr_2Cr_4Ni_5$ –based Alloys

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In the last decade, a number of studies demonstrated that AB_3-type alloys are promising alternatives for reversible hydrogen storage [1, 2]. They gained a considerable development owing to their high hydrogen storage capacity and good electrochemical properties compared to AB_5-type alloys [3]. In this context, a novel AB_3-type compound $CeZr_2Cr_4Ni_5$ was successfully prepared by mechanical alloying (MA) process starting from a mixture of AB_5-type compound $CeNi_5$ and AB_2-type compound $ZrCr_2$. The corresponding synthesis reaction is the following:

$$CeNi_5+2ZrCr_2 \rightarrow CeZr_2Cr_4Ni_5$$

$CeNi_5$ and $ZrCr_2$ precursors were prepared by Ultra High Frequency (UHF) induction melting of pure elements Ce, Ni and Zr, Cr respectively. For an alloying duration varied from 2 to 25 hours, the as-synthesized $CeZr_2Cr_4Ni_5$ alloys were constituted by a main hexagonal phase of $PuNi_3$-type structure mixed with other residual $ZrCr_2$, $CeNi_5$ and Cr phases. The crystal structure was determined and refined from X-ray diffractograms and the electrochemical hydrogenation properties of the novel AB_3-type $CeZr_2Cr_4Ni_5$ –based alloys were investigated by electrochemical measurements such as chronopotentiommetry, cyclic voltammetry and chronoamperometry techniques. The cycle-life investigation of the as-prepared $CeZr_2Cr_4Ni_5$ -based materials shows a promising electrochemical stability following 100 charge/discharge cycles in alkaline medium. The specific discharge capacity of $CeZr_2Cr_4Ni_5$ was equal to 270 mAh/g. Such properties are beneficial for the application of $CeZr_2Cr_4Ni_5$ –based materials as negative electrodes in Ni-MH batteries. The values of the hydrogen diffusion coefficient determined by cyclic voltammetry indicate a good absorption-desorption kinetic and a high hydrogen diffusivity through the $CeZr_2Cr_4Ni_5$ rich alloy.

Key words: Hydrogen storage, Ni-MH batteries, Mechanical alloying.

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The effect of a commercial melt strength enhancer additive on the physical and rheological properties of poly(lactic acid)

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In order to find out the effect of a commercial melt strength enhancer additive on the thermal, rheological and morphological properties of polylactide (PLA), different blends of PLA/Paraloid BPMS-260 were melt-blended using a screw extruder. Thermal analyses results showed only one Tg suggesting the miscibility of the melt strength enhancer additive with PLA resin. The cold crystallization onset of the PLA blends shifts to higher temperatures and their crystallinity decreased with an increase of the BPMS additive compared to neat PLA. This behavior indicates that BPMS additive decreases the ability of PLA to crystallize and/or recrystallize during processing. The rheological analyses showed that zero-shear viscosity and storage modulus of the PLA/BPMS-260 were significantly increased with the additive content. These findings suggest the entanglements of PLA chains with those of the high molecular weight additive, creating a physical network which reduces the segmental mobility of PLA and leads to a high melt elasticity of the blend. The analysis of SEM micrographs seems to confirm the miscibility of the acrylic additives with the PLA.

Key words: melt strength modifier, physical properties

Conductometric enzyme biosensor based on proteinase K was developed using two types of nanoparticles.

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Biosensors can be considered as competitive tools for environmental monitoring because of their specificity, fast response and low cost. The use of nanoparticles (NPs) in biosensors is a relatively new area of research, nevertheless, the literature already shows numerous examples of the incorporation of NPs into biodevice. Nanoparticles, specifically gold nanoparticles (AuNPs), are materials of interest in a rapidly developing area of biosensors. AuNPs exhibit strong surface plasmon resonance (SPR) that depends on the size of AuNPs and the relative distance between AuNPs. We can find nanobiosensors for the specific detection of biologically-relevant molecules like enzymes nucleic acids and proteins. Magnetic nanoparticles (MNPs) have found a lot of applications in immunoanalysis, immobilization, and purification of enzymes, DNA and proteins, and anti-tumor drug transportation and in the conception of biosensors.
In this work, proteinases are involved in a multitude of important physiological processes, such as protein metabolism. For this reason, a conductometric enzyme biosensor based on proteinase K was developed using two types of nanoparticles (gold and magnetic). The enzyme was directly adsorbed on negatively charged nanoparticles and then deposited and cross-linked on a planar interdigitated electrode (IDE). The biosensor was characterized with bovine serum albumin (BSA) as a standard protein. Higher sensitivity was obtained using gold nanoparticles.

The effects of *Laurus nobilis* leaves aqueous extracts on the corrosion behavior of chloride patina coated binary tin-bronze

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Several methods are available to prevent or retard corrosion of metallic materials, numerous researchers used organic molecules as corrosion inhibitors. However, most of these inhibitors are toxic. In this work, the effects of *Laurus nobilis* leaves aqueous extracts on the corrosion behavior of chloride patina coated binary tin-bronze was studied using stationary electrochemical technique, Raman spectroscopy and quantum chemical calculations. The electrochemical results obtained revealed that the time of immersion in the extract of Laurus nobilis is the most significant factor for experimental responses Ecorr, βa, βc, Icorr, Q_{Cu} and Q_{Sn}.

Moreover, the interaction between the weight of leaves and the time of immersion in the extract is the most influent factor in the B, R and% EP.

Keywords: corrosion, bronze, chloride patina, laurus nobilis extracts, inhibitor.

Eucalyptus Globulus a green inhibitor of corrosion of steel reinforcement in chlorinated simulated pore solution: A preliminary study

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This work present the corrosion inhibition of carbon steel in alkaline medium contaminated with 3% NaCl that simulates the interstitial solution of concrete contaminated with chlorides by aqueous extract of *Eucalyptus Globulus* leaves.
The electrochemical behaviour was evaluated by d.c. potentiodynamic polarisation and by electrochemical impedance spectroscopy (EIS). These electrochemical results were compared with those obtained from gravimetric method.
We found that the aqueous extract of *Eucalyptus Globulus* is an excellent green-inhibitor for steel in alkaline chloride environment and its effectiveness reached 95% at optimum conditions of extraction: maceration of 20g of plant material during 6 hours in 100 mL of H_2O. The Adsorption of "*EG*" aqueous extract on the carbon steel surface follows the Langmuir isotherm.

Key words: corrosion, carbon steel, green-inhibitor, alkaline chloride medium.

Synthesis and physicochemical identification of amidine cyclodextrin derivative

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Isolated from starch in 1891, cyclodextrin was then called by A. Villiers "cellulosine", since then their obtaining; cyclodextrins are widely used in pharmaceutical formulation to increase the solubility and stability of poor soluble drug. As a consequence of their composition of glucopyranose in chair conformation, they are shaped as a truncated cone with a wider side formed by the secondary hydroxyl groups and a narrow side formed by primary hydroxyl groups (figure 1). Those hydroxyl groups influence the aqueous solubility of cyclodextrin because of the strong intermolecular hydrogen bonding in the crystal state. Therefore many modified cyclodextrins has been synthesis by the substitution of one or more hydroxyl groups, to improve solubility and physical feature of cyclodextrin; their applications have developed in the latest decade.

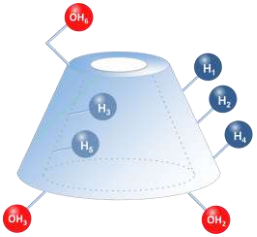

Figure 1. Geometric structure of β-cyclodextrine.

In this present paper we describe the efficient synthesis of a modified β-cyclodextrin within a co-evaporated dispersion reaction between β-cyclodextrin and N'-(1H-benzimidazo)-N-ethylacetamidine with no addition of bases or catalyses.
The structure of the amidine derivative of β-cyclodextrin was studied by FT-IR spectra, 1H NMR spectra, 13C NMR spectra, NOESY NMR spectra, UV absorption, thermogravimetric analysis, differential scanning calorimetry, X-ray diffractometry and scanning electron microscopy.

Key words: β-cyclodextrin, amidine, 2-aminobenzimidazole.

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IMPROVEMENT PROPERTIES OF MODIFIED TIO_2 NANOPARTICLES BY GRAFTED HEXAMETHYLENEDIAMINE AND INCORPORATED IRON NANOPARTICLES

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Modification of synthetic TiO_2 nanoparticles by grafting of Hexamethylemediamine (HMD) and an in-situ dispersion of iron nanoparticles (Fe^o-NPs) show changes in structural, optical and electrical properties of the novel resulting material TiO_2-HMD-Fe. The covalent surface-grafting of HMD within mesoporous tine dioxide was confirmed by the results from Fourier-transformed infrared (FTIR) spectroscopy. Electron microscopy and X-ray diffraction showed that dispersion of fine iron Nps occurs mainly inside the mesopores of TiO_2, producing a slight structure compaction. This was accompanied by a significant improvement of both the affinity towards electrical conductivity. Incorporing iron nanoparticles and grafting of HMD are resulted in a slight decrease in the optical band gap energy. Slightly red shifts to the longer wavelength are attributed to the change in the acceptor level induced by the iron nanoparticles. The capacitance, conductance and relaxation phenomenon of the compounds were studied by means of impedance spectroscopy measurements at several temperature. The electrical properties of the material are strongly improved by the grafting of HMD and the incorporation of Fe^o-NPs. Both the incorporation of Fe^o-NPs and HMD grafting appear to be, at least partly, responsible for conductance variations. The effects of the surface groups of ZnO-HMD-Fe and charge transfer are almost proportional to the capacitance. These properties make these materials to be regarded as very promising electrode materials for high-efficiency energy storage.

Keywords: TiO_2; surface properties; band-gap; impedance spectrum; HMD; iron Nps.

Quantitative study of Portland cement hydration by X-ray Diffraction: advantages and limits

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X-ray diffraction (XRD)/Reitveld has proved its efficiency as a powerful technique for the study of anhydrous Portland cement. It is considered as the most powerful method for the determination of quantitative phase amounts in multiple phase mixtures. Now many researchers are interested to apply this method on hydrated cement phases [1] [2]. However the presence of amorphous phase represents a major obstacle which can remains a source of error.

The present work describes a quantitative study of the hydration of Portland cement, in which we discuss the advantages and the limits of this method on the quantification of hydrated Portland cement phases. The studied cement belongs to Bizerte Cement Factory, the chemical composition was determined by X-ray fluorescence (XRF), and their mineralogical composition was calculated by X-ray diffraction.

Key words: XRD, Reitveld, anhydrous Portland cement, hydrated Portland, cement, XRD

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STUDY OF MAGNESIA –SPINEL BRICK OF A ROTARY CEMENT KILN

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Magnesia bricks-spinel $MgAl_2O_4$ are widely used to protect the burning zone of a rotary cement kiln. This amounts to their high thermal and chemical resistance. Corrosion of their refractory matrix is usually caused by the clinker phases enriched with iron, calcium and aluminum, which diffuse into the brick structure and react with the spinel and the magnesia [1-2].

In this work, the chemical and mineralogical composition of a sample of a magnesia brick -spinel MgO -$MgAl_2O_4$, taken from the burning zone, was investigated by ICP -AES, infrared, X-ray diffraction and heating microscopy to highlight the relationship between the thermal and chemical stresses and their effect on the structure of the brick. In fact, the results indicate excessive temperature that have accelerated the infiltration of the liquid phase of the clinker in the brick structure (the presence of belite C_2S). Therefore, the phenomenon of corrosion occurs giving rise to the formation of new phases such as Magnesium iron aluminum oxide $Mg_{0,7}Fe_{0,23}Al_{1,97}O_4$, Calcium magnesium aluminum oxide silicate, Potassium magnesium silicate K_2MgSiO_4 and $Na_2Ca_4Mg_2SiO_{15}$.

Key words: Magnesia – spinel bricks $MgAl_2O_4$, clinker, burning zone, corrosion.

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DESORPTION OF RARE EARTH ELEMENTS FROM "CHERT" OF GAFSA METLAOUI BASIN

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Currently silica arouses the interest of researchers given its multiple physical and structural properties, dimensional characteristics and it's abundance in the natural state.

The 'Chert' studied in this work is a highly silified sedimentary rock collected in Gafsa Metlaoui basin in Tunisia. It underwent purification treatments and was characterized by various analytical methods (XRD, FX-ED, MEB, FTIR**,** ICP-AES, laser granulometry).

The adsorption- desorption of rare earth elements was studied in this work. The present study focused on desorbing these ions from 'Chert' by using eluting agents capable of removing all the metals previously adsorbed. The goal of this work was to verify the reversibility of the sorption reaction thus the possibility of the desorption process for metal recovery.

Several eluting agents at different concentrations were tested to desorb the lanthanides including nitric and hydrochloric acids, calcium chloride salts.

Lanthanum desorption was 96% with $CaCl_2$, 66 % with HCl and 24% with HNO_3. Cerium desorption was 93% with $CaCl_2$, 40 %with HCl and 23% with HNO_3.

Key words: Chert, Purification, Adsorption, Desorption, Adsorbent, Rare earth, Eluting agents.

Program of Wednesday, December 23rd 2015

Template syntheses of copper(II) and cadmium(II) complexes from arylhydrazones of malononitrile and their catalytic activity towards alcohol oxidations

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The reaction of cadmium acetate hydrate with sodium 2-[2-(4,4-dimethyl-2,6-dioxocyclohexylidene)hydrazinyl]terephthalate (Na_2HL^2) leads to the new complex $[Cd(im)_3(\mu,\eta^2\text{-}HL^2)]_n$ **(1)**, while $[Cu(im)(\mu\text{-}HL^1\text{-}1\kappa O{:}2\kappa^3 NOO')]_2$ **(2)** is obtained from $Cu(NO_3)_2{\cdot}2.5H_2O$ and sodium 2-[2-(2,4-dioxopentan-3-ylidene)hydrazinyl]terephthalate (Na_2HL^1) in the presence of imidazole (im). Both compounds were characterized by IR spectroscopy, elemental analysis, UV–vis absorption spectroscopy and X-ray diffraction analysis. Di- or tri-anionic deprotonated derivatives of ligands display different coordination modes and lead to topologies and nuclearities of the complexes depending on metal ions and conditions used for the syntheses. Extensive intermolecular H-bonds form an infinite three dimensional architectures. Complexes **(1)** and **(2)** were successfully tested as catalysts. As a matter of fact, they can be applied as homogeneous catalysts for the oxidation of alcohols to ketones in acetonitrile, with mild conditions. The use of TEMPO as additive leads to an increase in the yield.

Keywords: Arylhydrazones of β-diketones / Multinuclear Cu^{II}, Cd^{II} complexes / Oxidation of cyclohexanol / Oxidation of phenylethanol / Catalysis

Structural, Thermal behaviour and Vibrational study of a new Cesium- Rubidium Dihydrogen Arsenate: $Cs_{0.2}Rb_{0.8}H_2AsO_4$

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The structure of a new inorganic solid acid caesium rubidium dihydrogen arsenate $Cs_{0.2}Rb_{0.8}H_2AsO_4$ (CRDA) has been examined by X-ray single crystal performed at room temperature. This compound crystallizes in tetragonal space group $I\bar{4}2d$ with lattice parameters a = 7.8090(1) Å and c = 7.5010 Å. It has a unit cell volume of 457.415 (1) $Å^3$ and four formula units per cell. The title compound is isostructural with the tetragonal phases of CsH_2AsO_4 (CDA) and RbH_2AsO_4 (RDA). The main feature of this structure is the coexistence of two different cautions (Cs^+ and Rb^+) in the same crystal.

In this structure, the arsenic atom as well as the rubidium and cesium ions lie on points with site symmetry S_4, the oxygen atoms lie in general positions about the arsenic atoms, in a tetrahedral arrangement. The AsO_4 tetrahedra are connected by O-H…O hydrogen bonds laying essentially in the *a*,*b* plane. The infrared spectrum performed at room temperature in the frequency range 4000–400 cm^{-1} confirm the presence of structural disorder in this material. The differential scanning calorimetric (DSC) show the presence of a structural phase transition of the title compounds at 474 K.

Keywords: Inorganic materials; $Cs_{0.2}Rb_{0.8}H_2AsO_4$; X-ray single crystal; Thermal behavior; Infrared spectrum

Synthesis, crystal structure and hirshfeld surface analysis of a new microporous phosphate organometallic

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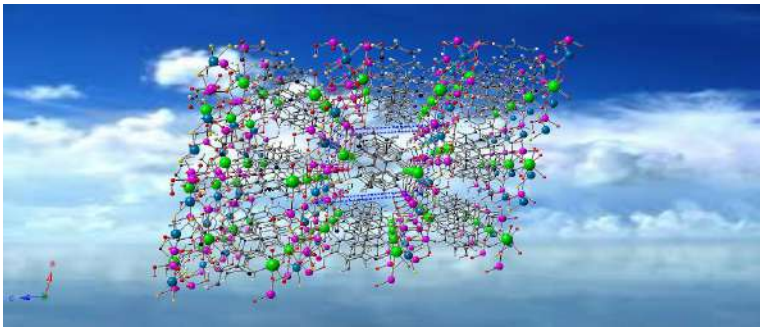
The study of microporous phosphate organometallic has been a proactive area of research owing to its optical and catalytical importance. In this regard, the compound $FeAlF_2(C_{10}H_8N_2)(HPO_4)_2(H_2O)$ has been prepared and characterized. An open framework structure of the material was a desirable feature. We performed X-ray structural studies. The compound is quantitatively analyzed with the aid of Hirshfeld surface analysis and 2D fingerprint plot for decoding of the intermolecular interactions. The molecules are connected by hydrogen bonds of the type O–H...O and O–H...F. In addition, The 2, 2'-bipyridine ligand involved in the stabilization of the structure and the creation of the three-dimensionality via π...π stacking.

Graphical abstract



View of the crystal packing of the title compound along the b-axis.

Keywords: Crystal Structure; Intermolecular Interactions; Hirshfeld Surfaces

Phase relationships in the KNO_3-$RbNO_3$ binary system at normal condition.

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Phase relationships in the KNO_3-$RbNO_3$ binary system at normal condition were investigated by powder X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray analysis (EDX) techniques. The limit of the solid solubility was determined. In the high potassium concentration, the solid solution $K_{(1-x)}Rb_xNO_3$ (denoted κ) adopts Pmcn orthorhombic symmetry were Rb^+ may substitute K^+ up to 52 mol %. In the Rb-rich phase $Rb_{(1-y)}K_yNO_3$ (denoted ρ) the $P3_1$ trigonal symmetry is preferred. We found that K^+ may substitute Rb^+ up to 75 mol %. Furthermore, the quantitative phase analysis (QPA) was performed for every sample leading to the amorphous and the crystalline proportions. Refining the occupation rates of every alkaline cationic site in the solid solutions allows, not only knowing the global cationic substitution rate in κ and in ρ phases but also the preferred crystallographic sites in every crystalline phase. These results were confirmed by microstructure investigations.

Key words: Phase relationships in the KNO_3-$RbNO_3$, XRD, SEM, QPA.

X-Ray single crystal, Hirshfeld analysis, Vibrational spectra, Optical studies and Dielectric relaxation of new organic-inorganic compound $(C_2H_7N_4O)_2[CoCl_4(H_2O)_2]$.

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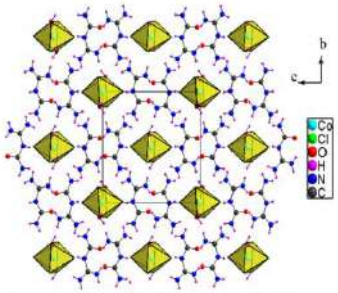
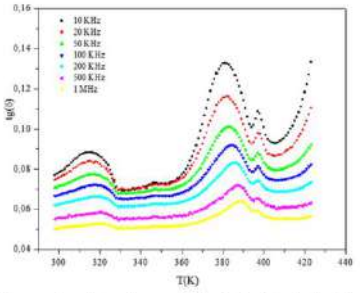
In this paper we study the new organic-inorganic hybride compound $(C_2H_7N_4O)_2[CoCl_4(H_2O)_2]$ has been grown by the solvent evaporation method. The compound was characterized by XRD, FT-IR, Raman, the UV-visible technique and Dielectric properties. The crystal structure was determined at room temperature by means of single-crystal X-ray diffraction methods in the monoclinic space groupe $P2_1/n$. The structure can be described by the alternation of organic-inorganic layers along the *a*-axis. The cohesion of compound entities is ensured by hydrogen bonding (N-H…Cl, N-H…O, O-H…Cl). Furthermore, the room temperature FT-IR and Raman spectra of the title compound were recorded and analyzed. The UV-visible absorption spectrum is characterized by two strong absorptions are assigned to the n–π*, π–π* transitions of the crystal and d-d transitions of Co anions. Dielectric data were analyzed using complex permittivity ε*(dielectric constante (ε') and dielectric loss (ε")) and dissipation factor (tan δ) were undergo two phase transition at 310 and 390K respectively.



Projection of the $(C_2H_7N_4O)_2[CoCl_4(H_2O)_2]$ structure along the b-axis.



Temperature dependence of the dissipation factor (tan δ) at various frequencies.

SYNTHESIS, CRYSTAL STRUCTURE AND THERMAL BEHAVIOUR OF TWO NEW HYBRID NITRATE COMPOUNDS $(C_4H_{12}N_2)_2[M^{II}(H_2O)_6](NO_3)_6$ WITH M^{II}: Ni(II), Zn(II)

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Two new hybrid nitrate materials, $(C_4H_{12}N_2)_2[M^{II}(H_2O)_6](NO_3)_6$, ($M^{II}$: Ni (**1**), Zn (**2**)), have been synthesised by slow evaporation method and crystallographically characterized. The X-ray diffraction analysis shows that the two compounds crystallise isotypically in the triclinic system, space group $P\bar{1}$, with closed unit-cell parameters. Their supramolecular crystal structure consists of metallic cation surrounded by six water molecules in an octahedral fashion, isolated nitrate anions and piperazine cations. These entities are linked together by OW-H…O and N-H…O hydrogen bonds. The FTIR spectroscopy analysis proves the existence of the different entities. The thermal decomposition of these two materials, studied by thermogravimetric analysis (TG), takes place in several step and allowed to the formation of metal oxides NiO and ZnO.

Key words: hybrid materials, nitrate, supramolecular, crystal structure, decomposition.

SYNTHESIS, CRYSTAL STRUCTURE AND VIBRATIONAL PROPERTIES OF (3-AMMONIUMPROPYL) IMIDAZOLIUM PENTACHLOROANTIMONATE(III)

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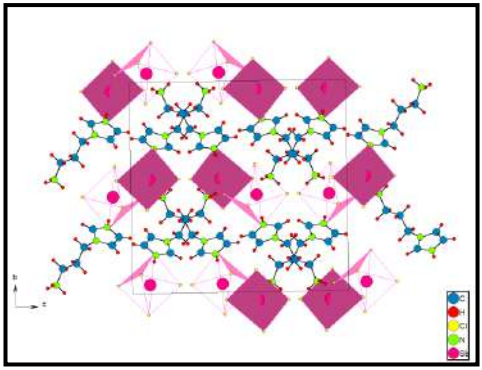
A new organic – inorganic crystal (3-ammoniumpropyl) imidazolium pentachloroantimonate(III) has been synthesized and characterized by FT-IR, Raman, PXRD and fluorescence studies. Single-crystal X-ray measurement result revealed that this compound crystallizes in the orthorhombic system with space group Pbca and has lattice parameters a = 9.6884 (4), b = 16.8479 (7), c = 17.3825 (8) Å and Z = 2. The atomic arrangement can be described as an alternation of organic and inorganic layers along the C-axis. The FT-IR and the Raman spectra of the polycrystalline $[C_6H_{13}N_3]$ $[SbCl_5]$ were performed at room temperature in the frequency region between 4000 and 400 cm^{-1} and 500-50 cm^{-1} respectively. This Spectra shows the presence of some bands characterized of cationic group $[C_6H_{13}N_3]^{2+}$ and anionic group $[SbCl_5]^{2-}$. This vibrational study confirms the results already found by Single-crystal X-ray measurement. Moreover, the fluorescent properties of the compound have been investigated in the solid state at room temperature, the PL spectrum of the sample is excited at 330 nm. This material shows two strong blue emission bands centered at 420 nm and 470 nm.



Projection of crystal structure of $[C_6H_{13}N_3]$ $[SbCl_5]$, on (b, c) plane

Key words: $[C_6H_{13}N_3]$ $[SbCl_5]$, Crystal structure, Luminescence, vibrational studies

STRUCTURE SPECTROSCOPIC AND THERMAL BEHAVIOR OF TWO MIXED TRICHLOROCADMIATE (II)

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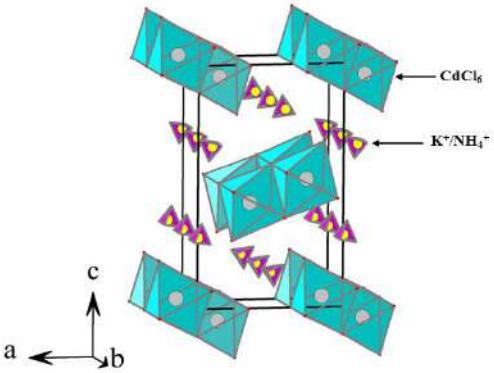
$K_{0.57}(NH_4)_{0.43}CdCl_3$ and $K_{0.25}(NH_4)_{0.75}CdCl_3$ are orthorhombic, space group Pnma, Z = 4, with a = 8.8760(4) Å, b = 3.9941(2) Å, c =14.7004(7) Å, and Z = 4, a = 8.9567(9) Å, b = 3.9957(4) Å, c = 14.855 (2) Å, respectively. Final R values are 0.01 and 0.02 for 608 and 834 reflections, respectively. In both materials, the crystal structure has been determined by X-ray single crystal analysis at room temperature (293 K). The compounds structure consists of K^+ (or NH_4^+) cations and double chains of $CdCl_6$ octahedra sharing one edge extending along ''b" axis. The mixture of K^+/ NH_4^+ cations are located between the double chains ensuring the stability of the structure by ionic and hydrogen bonding contacts N/K–H ...Cl. Spectroscopic and DSC measurements were performed to discuss the mechanism of the phase transition. These studies show that these materials $K_{0.57}(NH_4)_{0.43}CdCl_3$ and $K_{0.25}(NH_4)_{0.75}CdCl_3$ undergo a phase transition at 438 K and 454 K, respectively.



Perspective view of the structure of $K_{0.57}(NH_4)_{0.43}CdCl_3$.

Keywords: X-ray diffraction; Phase transition; DSC; IR absorption; Raman scattering.

Synthesis, Structure and Physicochemical Studies of Hexakis (3-Chloro 2-methylanilinium) Cyclohexaphosphate dihydrate: $[C_7H_9ClN]_6P_6O_{18}.2H_2O$

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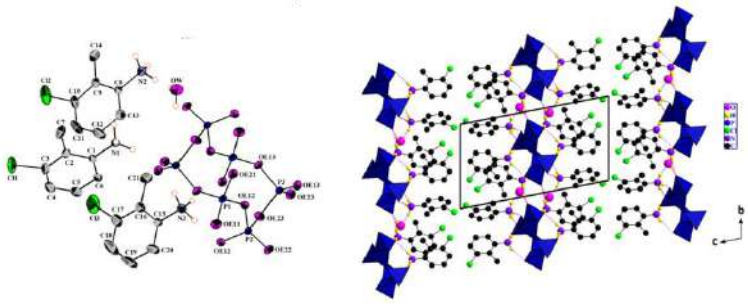
The interest of hybrid organic-inorganic materials has been continuously growing because of their potential applications in several fields (catalysis, biomoleculaire sciences and nonlinear optics) (Shi et *al*., 2000, Yokantani et *al.*,1989). Interest of these compounds stems from the intriguing possibility of combining the features of both inorganic and organic systems within a single material (Koo et *al*., 2004, Xiao et *al*., 2005)

This paper reports synthesis, structural characterization and physicochemical studies of a new organic cyclohexaphosphate. The X-ray diffraction study of the title compound leads to the determination of its chemical formula. Configurations of the different organic and inorganic entities are depicted in the **Figure 1**. The examination of the atomic arrangement shows that the structure consists of inorganic layers made up from P_6O_{18} rings and water molecules connected via hydrogen bonds and extended in the *ab* plane. The organic 3-chloro-2-methyl-anilinium cations are displayed in the interlayer spaces compensating their negative charges and establishing H-bonds with the oxygen atoms of the anionic framework as shown in **Figure 2.**

This compound is also characterized by infrared spectroscopy, solid state MAS-NMR and differential thermal analysis



CRYSTAL STRUCTURE, PHASE TRANSITION STUDIES OF (C6H18N2)3SB4CL18 COMPOUND

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Recently, a great attention has been focused on organic-inorganic hybrid materials. The materials are frequently characterized by their various physical and chemical properties that could lead to technological innovations like magnetic or ferroelectric transitions, conductivity (super conductivity).The tri (N, N, N', N'-tétraméthyléthylènediamonium) octadecachlotetraantimonate (III) has been characterized by X-ray powder and single crystal analysis, DSC, AC conductivity and dielectric measurements. The chemical preparation, the main structural, vibrational are given for a new zero dimensional alkyl di-ammonium antimonate halogenide. ($C_6H_{18}N_2$) Sb_4Cl_{18} crystallize in triclinic system, space group P-1. The phase transition is detected at 356K,

Key words: alkyl polyammonium antimonate (III) chloride, chemical preparation, structural, DSC, X-ray powder, dielectric measured.

Mineralization of the antibiotic levofloxacin by electro-Fenton-pyrite process

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Solutions of Levofloxacin (Lev) in 0.05 M Na_2SO_4 was studied by a novel heterogeneous electro-Fenton (EF) process, so called EF-pyrite, in which pyrite powder was the source of Fe^{2+} catalyst. Experiments were performed with a cell equipped with born-doped-diamond (BDD) anode or a Pt and a carbon-felt cathode, where Lev and its products were destroyed by hydroxyl radicals formed at the anode surface from water oxidation and in the bulk from Fenton's reaction between Fe^{2+} and H_2O_2 generated at the cathode.
Addition of 2.0 gL^{-1} pyrite provided an easily adjusted the pH to 3.0 and Lev solution with 0.46 mM content were then completely mineralized applying a constant current of 300 mA.

Key words: Heterogeneous catalysis, Hydroxyl radical, Levofloxacin, Mineralization, Electro-Fenton-pyrite.

References

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MOLECULARLY IMPRINTED POLYMER BASED ON SILICA SUPPORTS FOR EFFICIENT ADSORPTION OF PATULIN

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In this work, surface modified Stöber silica particles were used as support for the development of molecular imprinted polymer matrix for separation of Patulin. The polymer matrix was developed from methacrylic groups anchored at the surface of silica support using maleic acid (MA) or methacrylic acid (MAA) as functional monomer. The resulting imprinted materials SiO_2polymer@MIPs were characterized by means of FT-IR, ^{13}C NMR and elemental analysis. The uptake capacity of Patulin by the synthesized adsorbent materials was assessed under the same conditions. SiO_2MA@MIP has exhibited the best adsorption capacity of PAT 1.55 µmol.g^{-1} which was 3.85 times higher than that obtained with SiO_2MA@NIP. It was noticed that the adsorption of Patulin on SiO_2MA@MIP has attained a steady state in only 20 min whereas SiO_2MAA@MIP have taken 120 min under the same conditions. Pseudo-first order, pseudo-second order and intraparticle diffusion, were used as kinetic models for the fitting of the results of adsorption kinetics. It has been shown that the adsorption kinetic process is best described by the pseudo-second order kinetic model. Adsorption isotherm recorded with SiO_2MA@MIP as adsorbent was examined using Langmuir, Freundlich, Temkin and Harkins-Jura models. Freundlich was identified as the most appropriate model in analyzing the binding behavior of the adsorbent. The obtained results indicate that SiO_2MA@MIP could be employed as an efficient adsorbent much more than the SiO_2MA@NIP material for Patulin removal from complex systems.

Keywords: Molecularly imprinted polymers (MIPs), silica support, Patulin toxin, adsorption.

HYDRATION STUDIES OF DIFFERENT TUNISIAN HRS PORTLAND CEMENTS

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According to the Tunisian standard, HRS Portland cements hydraulic binders for work in environments with high content of sulfate ions such as sea water, soils rich in gypsum. These cements have a very special mineralogical composition (C_3A<3% and 2 C_3A +C_4AF < 20%) that allows the concrete to confer increased resistance to the aggression of sulfates ions in the setting and thereafter [1].
Today, in most Tunisian cement industries, the production of this cement type was made by increasing the iron in raw materials or by addition of limestone at the moment of clinker milling to obtain a cement with a low C_3A, but this behavior can be a source of heat of hydration rising, which became a cause of cracking of concrete and penetration of aggressive agents such us SO_4^{2-}. In this context, we collected some HRS cements and we made a study performed this cements presented as a powder and paste to highlight the hydration occurring in the cement in various time intervals (1, 2, 7and 14 days) making use of the X-ray diffraction (XRD) method and an infrared spectroscopy technique (IR).
As regards the cement hydration study, we performed several tests of HRS cement samples by means of a precision microcalorimeter.

Key words: HRS cement, mineralogical composition, hydration, XRD, IR, cement hydration.

References

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CHARACTERIZATION OF AMORPHOUS Al-Co-Nb ALLOY PREPARED BY HIGH ENERGY MECHANICAL ALLOYING

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In recent decades, research to produce, understanding and using nanostructures continue to intensify. Nanosciences are based on the fact that the material properties vary depending on their dimensions. To synthesize these nanostructures, several technologies have been explored. Mechanical high energy milling is one of the synthesis methods of nanomaterials. It allows one hand carrying alloy with a mixture of chemical elements at the atomic scale (mechanical alloying) and secondly obtaining nanostructured and/or amorphous powders by milling of crystalline systems (mechanical milling).

The purpose of this work is the synthesis of a metallic glass type Al-Co-Nb by high energy ball mill (type mill Fritsch P7) from pure elemental powders of Al, Co and Nb and investigate the mechanisms of the amorphization of the alloy. Morphological, microstructural and thermal characterizations of the powders milled several times were investigated by scanning electron microscopy, X-ray diffraction and differential scanning calorimetry. The patterns obtained were analyzed using the Rietveld refinement. At early stage of milling, the products of the mechanically alloying process were a duplex of Al(Co,Nb) nanocrystals and little amount of amorphous phase. The increase of mechanical milling induced a decrease of the crystallite size and an increase of the microstrains. A continuous increase of the amorphous phase content was also observed. The thermal properties of powders were determined from DSC measurements under an argon atmosphere. These measures have enabled us to determine the glass transition, recrystallization and the stability temperatures.

Keywords: mechanical alloying, nanostructured compounds and metallic glasses.

Investigation of the magnetocaloric effect and dielectric properties in $La_{0.7}Sr_{0.25}K_{0.05}Mn_{(1-x)}Ti_xO_3$ perovskite manganite

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In this paper, the magnetic phase transition and the magnetic entropy change (ΔS_M) in the electron-doped $La_{0.7}Sr_{0.25}K_{0.05}Mn_{(1-x)}Ti_xO_3$ ($LSKMTi_x$) (x = 0.0-0.1) have been investigated in details by measuring the magnetization as a function of temperature. The polycrystalline sample ($LSKMTi_x$) was prepared using a conventional solid state reaction method**.** X-ray power diffraction indicates that these compounds have a rhombohedrally distorted structure with a space group $R\bar{3}C$. The maximum magnetic entropy change (ΔS_M) and the relative cooling power (RCP) are found to be, respectively, 4.85 J/kg.K and 280 J/kg for a 5 T magnetic field change along with a negligible hysteresis loss, making of this material a promising candidate for the magnetic refrigeration. The magnetic entropy change versus the temperature curves for the different applied fields collapses into a single curve, revealing a universal behavior of MCE. The dielectric behavior of $LSKMTi_x$ sample was particularly investigated via AC impedance spectroscopy method. The complex impedance plot show a single semi-circle in the complex plane is derived from the ac response with frequency which exhibits some depression indicative of non-Debye type of relaxation. The studies of frequency-dependent conductivity show the effect of Ti^{4+} in our sample and the bulk and the grain boundary relaxation can be distinguished in the M'-M'' plots. Dielectric loss was analyzed by the Maxwell Wagner interfacial polarization given by Koop's theory.

Keywords: X-ray methods; magnetic; magnetocaloric properties; impedance spectroscopy.

Structural, thermal decomposition and vibrational study of the new bidimentionel fluoroferrate Fe_3F_{10}(3,5-diamino-1,2,4-triazole)$_2$

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A new crystal structure Fe_3F_{10}(3,5-diamino-1,2,4-triazole)$_2$ was prepared under hydrothermal conditions, at 393 K for 72 hours, by a mixture of FeF_2/FeF_3, (3,5-diamino-1,2,4-triazole) molecule and hydrofluoric acid solution (HF 4%) in ethanol solvent. Crystalline structure determination is performed from single crystal diffraction data. It crystallizes in the triclinic system space group P1 with unit cell parameters a= 7,100(1)Å, b= 7,658(2)Å, c= 8,321(1)Å, α=107,330(2)°, β= 111,842(1)°, γ =93,049(1)°, Z=1 and V=394,00(1)Å^3. The new iron fluoride Fe_3F_{10}(3,5-diamino-1,2,4-triazole)$_2$ exhibits both oxidation states of iron (Fe(II) and Fe(III)) in the unit cell. Each iron cation is associate by fluorine anions of FeF_4N_2 and FeF_6 octahedral and only iron (II) connected with two nitrogen atoms of monoprotonate organic molecules (3,5-diamino-1,2,4-triazole) to build 2D material. The thermal analysis TG, DTG and DTA of the title compound show that the decomposition introduce a three step between 310 K and 873 K. IR spectra recorded, at room temperature, in the frequency range 400-4000 cm^{-1} confirm the presence of (3,5-diamino-1,2,4-triazole) molecules.

Key words: Hydrothermal synthesis, X-ray diffraction, Thermal analysis, Infrared.

INFLUENCE OF THE SUBSTITUTION OF β-CYCLODEXTRINS BY PYRIDINIUM GROUPS ON THE COMPLEXATION OF ADAMANTANE DERIVATIVES

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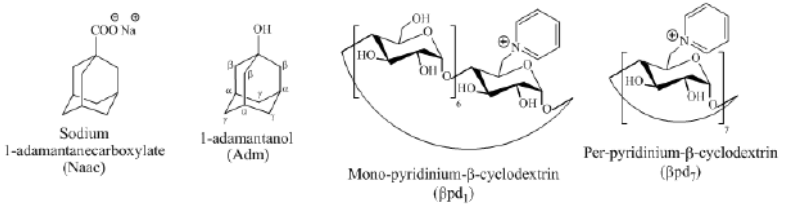
Cyclodextrins are cyclic oligosaccharides built of α-1,4-linked D-(+)-glucopyranose units that possess the overall shape of a hollow truncated cone, the narrow rim bearing primary hydroxyl groups and the wide rim secondary hydroxyl groups. An important property of cyclodextrins is their ability to include in their hydrophobic cavity a large variety of guest molecules in aqueous solutions, without formation of any covalent bond. The potential utility of inclusion complexes includes solubilization, encapsulation, and transport of small hydrophobic molecules such as toxins and drugs. However cyclodextrins are poorly soluble in water because of strong intramolecular hydrogen-bonding between secondary hydroxyl groups. Cyclodextrins modified with hydrophilic substituents have enhanced solubilities in water compared to their native form.
A series of cationic β-cyclodextrins with various positive charges has been synthesized by selective functionalization of the primary face of β-cyclodextrin with pyridinium groups. All modified cyclodextrins were characterized by elementary analysis, FTIR, ^{1}H and ^{13}C NMR spectroscopy. The inclusion complexes of adamantane derivatives were of the 1:1 stoichiometry; the stability constants ($K_{1:1}$) have been evaluated by spectroscopy ^{1}H NMR. The presence of pyridinium groups increased the complexation ability. β-cyclodextrin fully substituted at the primary face with pyridinium groups showed the strongest inclusion binding ability towards the 1-adamantanecarboxylate guest. The enhanced complexation for anion guest was ascribed to the cationic pyridinium groups. A simple thermodynamic model of the electrostatic contribution to the complexation is also presented.



Key words: Cationic β-cyclodextrins, adamantane derivatives, inclusion complexes, Stability constant.

ELECTROCHEMICAL BEHAVIOUR OF FERULIC ACID IN AQUEOUS SOLUTIONS ON GLASSY CARBON BY CYCLIC VOLTAMMETRY

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A glassy carbon electrode (GC) was used for the investigation of the electrochemical oxidation of ferulic acid (4-hydroxy-3-methoxycinnamic acid), molecule which is selected as a model that can characterize the wastewater of olive oil extraction, using cyclic voltammetry. The electrochemical behavior of ferulic acid was studied as a function of its concentration, the potential scan rate and the temperature. At low concentration, ferulic acid shows one irreversible anodic peak. The peak current shows adsorption characteristics. For ferulic acid concentration higher than 10^{-4} mol dm^{-3}, the voltammogram shows two anodic peaks. In fact ferulic acid is oxidized in two forms: free and adsorbed ones. The free form corresponds to the first peak. The adsorbed or the stabilized form, called also "post-peak" corresponds to the second peak and oxidizes at more anodic potential. Furthermore, we have shown that the competitiveness of these two forms is controlled by the ferulic acid initial concentration value. The activation energy from the slope of the curve representing the neperian logarithm of the anodic current relative to the first peak depending on the temperature inverse (Ln (i) = f (1 / T)) is equal to 23.45 kJ / mol. value consistent with a purely diffusionnel regime.

Keywords: anodic oxidation; cyclic voltammetry; Ferulic acid; glassy carbon; Wastewater treatment.

Iron molybdates thin film as cathode material for lithium ion / sodium ion batteries

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The recent technological development of miniaturized systems has induced a strong demand for developing compact power sources with high efficiency and small dimensions that are suitable for portable devices. Among these systems, the lithium microbattery may be relevant for a wide range of applications linked to the field of smart cards, implantable medical devices, MEMS,..

Many researchers are, as well, focusing on sodium-ion batteries as a new alternative to lithium-ion batteries in large scale applications thanks to the higher abundance and lower cost of sodium.

For microelectronic applications, the voltage delivered by the micro-battery should remain relatively low around 3V which requires the research of new positive electrode materials fulfilling this condition (figure 1). Iron molybdate $Fe_2(MoO_4)_3$ (FM) is a potential candidate to be applied in this kind of systems[1]. The purpose of this study is to investigate, for the first time, redox processes in iron molybdate thin films (elaborated by radiofrequency Magnetron Sputtering) cycled versus lithium and sodium using X-Ray Photoelectron Spectroscopy (XPS).

XPS gives essential information not only on the electrode materials but also on the phenomena occurring during electrochemical cycling. An an example, the figure 2 presents Fe 2p XPS spectra at the1st (end of discharge and end of charge) and 20th cycles versus lithium.

Our results lead to a better knowledge of the redox process and the reversibility of FM electrodes cycled vs Li and Na.

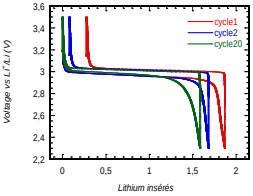

Figure 1: *Discharge curves of the 1st, the 2nd and the 20th cycles of FM electrode vs. Li*

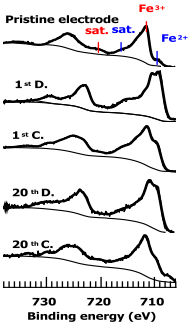

Figure 2: *The XPS Fe 2p core peaks spectra of iron molybdate thin films cycled versus Li at the first and the 20th cycle.*

Key words: XPS, iron molybdate, lithium ion batteries, sodium ion batteries.

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Posters Communications' Abstracts

THERMODYNAMIC ASSESSMENT OF THE ($CsNO_3$+$NaNO_3$+$TlNO_3$) TERNARY SYSTEM

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The thermodynamic evaluation of binary systems and/or higher order, by CALPHAD method, occupies increasingly considerable interest both in research than in industry. In the case of ternary systems or higher order, the advantage of this method which based on thermodynamic calculations is to reduce the number of experimental measures and avoid the use of different techniques such as direct and differential thermal analysis, microscopic, X-ray diffraction at different temperature. Indeed, the compilation of all the raw experimental data for pure component and binary mixtures allow to study the coherence between thermodynamic data and phase diagrams of limiting binary systems of a ternary one. The results for these limiting binary systems are then used to estimate their ternary system.

In this context we are interested in the assessment of the ternary system ($CsNO_3$+$NaNO_3$+$TlNO_3$).

The three limiting binary systems, ($CsNO_3$ + $TlNO_3$), ($NaNO_3$ + $TlNO_3$) and ($CsNO_3$ + $NaNO_3$) systems have been well optimized [1, 2, 3].

By means of an optimization procedure, the ($CsNO_3$+$NaNO_3$+$TlNO_3$) ternary system has been assessed in the present work. The parameters describing the Gibbs energies of all binary phases and the ternary liquid are used to predict the phase diagram of this ternary system, using THERMOCALC software.

This work represents the first step of the thermodynamic description of the ternary ($CsNO_3$+$NaNO_3$+$TlNO_3$) system.

Key words: CALPHAD, Thermodynamic evaluation, limiting binary systems, ($CsNO_3$+$NaNO_3$+$TlNO_3$) ternary system.

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Cellulose-Ag -TiO_2 thin films : Characterization and photocatalytic activity under visible light irradiation

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In recent years, many attentions have been focused on organic/inorganic composite materials composed of polymer matrix and inorganic nanoparticles, which can be widely applied in many fields including photocatalysis, adsorption and bulk-heterojunction solar cells.

Among the great variety of natural materials, it is well known that cellulose substances are of special importance both in industries and in daily lives as essential materials. Natural cellulose, chemically a polysaccharide, is a linear natural polymer of β-(1 4)-D-glucopyranose possessing abundant surface hydroxyl groups that forming plentiful inter- and intra-molecular hydrogen bonds. The abundant surface hydroxyl groups possessed by the natural cellulose provide a suitable substrate for metal oxide thin film deposition onto the cellulose surface.

Titania-based coatings with high specific surface area and narrow pore size distribution proved to have great prospects for practical application in solar energy transformation, photocatalysis, water and air cleaning. However, because of its large forbidden band, only radiation with energy larger than3.2 eV (in ultraviolet range) can stimulate its photocatalytic action. So the first step of this study has been to coat the TiO_2 with silver because of the strong absorbance of silver in the visible range.

In our present work, cellulose-Ag-TiO_2 hybrid thin films were successfully prepared at low temperature. They were characterized using raman spectroscopy, time resolved luminescence, the ground state diffuse reflectance spectra and scanning electron microscopy (SEM).

The photocatalytic activity of the prepared cellulose-Ag-TiO_2 thin films, under visible light irradiation, was evaluated using dyes (Methyl blue, Methyl red and Acid orange) as pollutant models. A great enhancement of the photocatalytic efficiency was evidenced.

LUMINESCENT FLUORENYL DENDRIMERS

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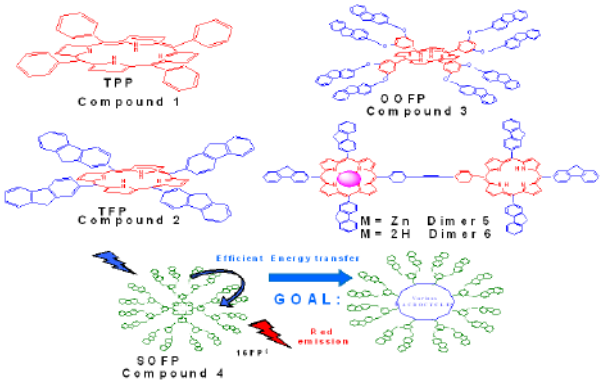
In 2004, we have reported the synthesis of porphyrin possessing four fluorenyl arms (**TFP,** compound **2**).[1-5] Surprisingly, **TFP** exhibited a remarkably high quantum yield (24%), compared to the reference tetraphenylporphyrin (**TPP**, compound **1**) demonstrating the capacity of the fluorenyl units to enhance quantum yields. Then, to exploite this efficiency, a series of porphyrin dendrimers bearing fluorenyl dendrons was prepared: new porphyrin **OOFP** possessing eighth fluorenyl pendant arms, [6] and the **SOFP** possessing sixteen fluorenyl arms; namely **3** and **4**.[6, 7]

As an applications, we next tested corresponding platinum(II) complexes in the fabrication of red Organic Light Emitting Diodes (OLEDs).[8-10] Also, supramolecular assemblies possessing **12 or 24 fluorenyl arms**, using these efficient building blocks have been obtained by our group.[11] Very recently we synthesised smaller units possessing 6 fluorenyl arms like dimers **5** and **6**, [10] as well as trimers. [11] Encouraged by these results, we will design Various Macrocycles substituted by fluorenyls units.



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Crystal structure and vibrational of N-(3-ammoniumpropyl)-1,3 diammoniumpropane hexachloroantimonate monohydrate: [C6H20N3]SbCl5.Cl H2O

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This work is a part of our study of the crystal structures of alkyl polyammonium antimonate (III) chloride. This investigation were extended to three aliphatic polyamines NH2[(CH2)2NH2]n(CH2)2NH2 (with n=0, 1 and 2) in order to examine the effect of the flexible cation on the antimony (III) coordination geometry. The chemical preparation, the main structural, vibrational are given for a new zero dimensional alkyl tri-ammonium antimonate halogenide. (C6H20N3) $SbCl_5$.Cl H_2O crystallize in triclinic system, space group P-1. The crystal structure consists of discrete ionic pairs of N-(3-ammoniumpropyl)-1,3 diammoniumpropane cations, (pentachloromoantimonate and free chloride) anions and neutral water molecules linked via simple and bifurcated N–H–Cl(OW) and O(W)–H–Cl hydrogen bonds. Infrared and Raman spectra of (C6H20N3) $SbCl_5$.Cl H_2O were recorded at 298 K and discussed. The assignment of the observed IR and Raman lines was performed by comparison with the homologous compounds and DFT calculated.

Key words: alkyl polyammonium antimonate (III) chloride, chemical preparation, structural, vibrational, DFT calculated

STUDY OF PYRENE SORPTIVE BEHAVIOR BY AN ARTIFICIAL SOIL USING BATCH EXPERIMENTAL PROCEDURE

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Sorption of polycyclic aromatic hydrocarbons (PAHs) has been extensively studied under various experimental conditions. Researchers agree that PAHs fate and distribution in the subsurface environment are greatly influenced by sorptive interactions with sorbents. For example, the amount of organic matter on the soil influences a compound's sorption behavior.

PAHs are classified as priority environmental pollutants and their accumulation in soil may lead to contamination of vegetables and food chains [1]. Pyrene is a typical PAH found in contaminated soils.

Very little information on sorption behavior of pyrene in the soil matrix is available. Most studies have been conducted on non-carcinogenic PAHs which have less than 4rings.

The aim of this study is to evaluate the sorption behavior of pyrene in a reconstituted soil and to investigate the effect of organic content on the pyrene sorption behavior. At the beginning, isotherm studies were performed by carrying out batch experiments. The pyrene solutions were prepared at concentration ranging from 50 to 250 µg/L. The choice of the composition of the soil used in the laboratory experiments was made by referring to a natural soil collected at a depth of 40 cm from an industrial site sixty kilometers northwest of Tunis. It's constituted from mixture of Hostun sand (96.7%), clay (3%) and compost used as a source of organic matter (0.3%).

Our results show that the data of pyrene sorption fitted well with the Freundlich model. The model constants were calculated and we noted that at higher organic matter content (varied from 0.3% to1.2% w/w), sorbed concentrations of pyrene are relatively low.

Key words: pyrene/organic content /batch experiments.

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Synthesis, spectroscopic characterization and X-ray structure analysis of $[C_5H_8N_3]_2ClSbCl_4.H_2O$

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The synthesis, crystal structure, and characterization of a new organic–inorganic hybrid material with $[C_5H_8N_3]_2ClSbCl_4.H_2O$ formula are described. The crystal structure refinement shows that this compound crystallizes in the monoclinic space group $P2_1/m$ with the following unit cell dimensions: a = 5.671(1) Å, b = 18.628(2) Å, c = 9.018(1)Å, b = 90.448(1) °, V = 952.61(2) $Å^3$, and Z = 2. The atomic arrangement of the title compound can be described by the three-dimensional network (Fig. 1). The cohesion of compound entities is ensured by hydrogen bonding (N–H…Cl, Ow–H…Cl, N–H…N, and C–H…Cl) and π–π interactions. The ^{13}C CP-MAS-NMR spectrum shows five isotropic resonances, confirming the existence of five non-equivalent carbon atoms. This result is in good agreement with the X-ray single-crystal study. The infrared spectrum of this compound reported from 4000 to 400 cm^{-1} confirmed the presence of the principal bands assigned to the internal modes of the organic cation and water molecule.

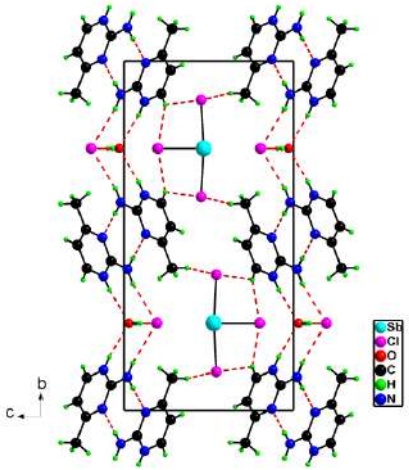

Fig. 1 A Projection along the [100] direction of the $[C_5H_8N_3]_2ClSbCl_4.H_2O$ structure

THERMAL CHARACTERIZATION AND COMPARISONS OF LIGNIN-FORMALDEHYDE AND LIGNIN-GLYOXAL ADHESIVES

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In the present study, the main focus was the use of lignin isolated through an industrial waste (black liquor) to prepare phenolic-type resins. Thermal properties of control lignin-formaldehyde adhesive (RLF) and lignin–glyoxal adhesive (RLG) have been investigated in detail. The effect of methylolation of lignin by formaldehyde and glyoxal have been studied using thermogravimetric analysis (TG) and differential scanning calorimetry (DSC). Methylolation has significant effect on the structure and the thermal stability of lignin. Rate of curing is enhanced by the type of curing agent and temperature.

Key words: Lignin; glyoxal; formaldehyde; resin; thermal stability.

Kinetics and thermodynamics of the attack of fluorapatite by sulfuric acid at 25° C

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In the wet manufacture process of phosphoric acid, the ore is attacked either by sulfuric acid solution or by a mixture of the latter with phosphoric acid.

Modeling the attack by phosphoric acid [1] and the mixture [2] has been carried out by following attack with synthetic fluorapatite (Fap), using a SETARAM C-80 microcalorimeter at different temperature.

In this work a calorimetric study of kinetics and thermodynamics of the attack of a synthetic fluorapatite (Fap) by sulfuric acid is undertaken at 25°C using C-80 SETARAM microcalorimeter with the reversal cells.

The global enthalpy of the attack equals −371.3 kJ mol^{-1}. The recorded thermograms show one peak leading to anhydrous calcium sulfate (AH). The deconvoluted curves agree with a model based on simultaneous reactions, both of order 1, supposing for this phenomenon a dissolution/precipitation mechanism. Iteration procedure led to values of kinetic and thermodynamic parameters corresponding to the best coincidence between the calculated and deconvoluted curves. The precipitation enthalpy of (AH), determined experimentally, is close to the value deduced from iteration. Furthermore the sum of the enthalpies ($\Delta_1 H + \Delta_2 H$), deduced from iteration, not differ from the enthalpy determined by integrating the rough signal by more than 3%.

Key words: Fluorapatite, Microcalorimetry, Kinetic, Dissolution, Sulfuric acid.

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Synthesis and structural characterization of 3-chloro-2-methylanilinium chloride monohydrate

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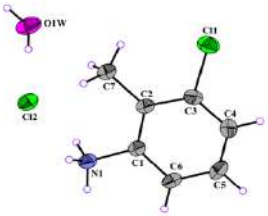
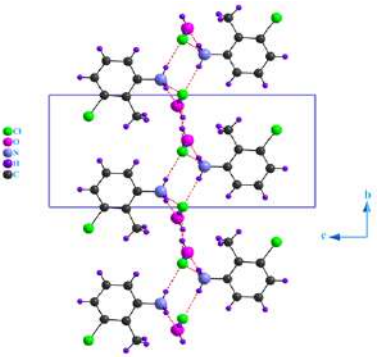
Hydrogen bonding is of interest because of their prevalent occurrence in biological systems (Steiner 2002; Jayaraman et al., 2002). Therefore, it is extremely useful to search simple molecules allowing understanding the configuration and the function of some complex macromolecules. The title compound, was prepared as part of our ongoing studies of hydrogen-bonding interactions in the crystal structure of protonated amines (Hamdi et al., 2014). Structures containing the 3-chloro-2-methylanilinium cation have been already reported with dihydrogenphosphate (Khemiri et al., 2008) and cyclohexaphosphate (Bel Haj Salah et al., 2014).

The asymmetric unit of the title compound (I), illustrated in Fig. 1, consists of one organic cation, one Cl^- anion and one water molecule. All bond distances and angles are within the ranges of accepted values. Moreover, they are close to respect the geometrical features observed in the crystal structure of 5-chloro-2-methylanilinium chloride (Oueslati et al., 2005). Components of the asymmetric unit develop different H-bonds, N-H...Cl, N-H...OW and OW-H...Cl (Fig. 2) that keep them in a state where the aromatic rings of organic buildings are oriented in the direction of the bc planes to form centro-symmetric pairs interconnected by hydrogen bonds in the direction of the b axis (Fig. 2). The resulting sequences are alternated to form the crystal packing of the title compound.





Study on the structural and morphology properties of Chemical Vapor Deposition of Tin sulfide (SnS) thin films

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In recent years, Tin chalcogenides thin films have received much attention because of their world-wide applications in various fields of science and technology [1]. Tin monosulfide (SnS) belongs to IV-VI compound semiconductor materials [2]. It is a promising material for the absorber layer in solar cells, primarily due to the earth-abundant elements comprising the material [3], secondly it has an optical band gap of 1.3 eV [4], closed to the ideal band gap (1.5 eV).
In order to try a new approach to form tin sulfide thin films using an organophosphorus compounds as a precursor, we report in this work, the synthesis of SnS thin film by simple chemical vapor deposition (CVD) technique using the triphenylphosphine sulfide ligand ($Ph_3P=S$).
Tin sulfide, which is grey/black adherent film, has an orthorhombic structure with the distorted NaCl type [5].
The thin film produced by the CVD technique was characterized by XRD, EDAX, SEM, Raman, and AFM, which revealed that the layer is indeed a SnS film with a good uniformity and adhesion.

Key words: Thin films, triphenylphosphine sulfide, SnS, CVD

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Morphology Controlled Solvothermal Synthesis of Bismuth Oxyhalides and Characterization of their Polyaniline/BiOX (X= Cl, Br, I) Composites

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Developments of organic-inorganic hybrid materials on nanometer scale have been receiving significant attention due to a wide range of potential applications, high absorption in the UV-Visible part of the spectrum and high mobility of the charge carriers. A series of Polyaniline/BiOX (X= Cl, Br, I) composites were successfully synthesized by solid state route with different content of hierarchical bismuth oxyhalides which were elaborated via solvothermal process. The composition, morphology, structure and optical properties of these hybrids were characterized by Fourier Transform-Infrared Spectroscopy (FT-IR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and UV–Vis Diffuse Reflectance Spectroscopy (DRS). The decomposition was visualized by ThermoGravimetric Analysis (TGA). The PANI modified bismuth oxyhalides are of great importance for environmental applications due to their optical properties which are in progress in our laboratory.

Key words: Polyaniline, Bismuth oxyhalides, solvothermal synthesis.

Synthesis, X-ray single crystal, and vibrational properties of new organic–inorganic compound $[C_7H_7N_2]_2\ Cu_2Cl_6$

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In the present work, the synthesis, structural characterization and and vibrational spectroscopy of $[C_7H_7N_2]\ Cu_2Cl_6$ are reported.
The title compound crystallizes in the Monoclinic system P 21/n at room temperature with the following parameters: a = 16.7794(18)Å, b = 6.4216(6) Å, c = 18.535(2)Å, β = 92.931(8)°, and Z = 8.The crystal structure was solved and refined to R[F>4σ(F)] = 0,04 and Rw=0,12.
The structure of this compound might be described as layered with two parallel anionic and cationic layers.
In the crystal structure the organic cations are linked to $CuCl_5$ by C-H....Cl and N H....Cl hydrogen, forming infinite chains. The weak hydrogen bonds between the anions and the cations give further rise to a 3D network structure.
In addition to these hydrogen interactions, we also note that the cohesion in the crystal is ensured by π-π interactions.
In order to give more information on the crystal structure, we have studied the vibrational properties of our compound using Raman scattering and infrared absorption. The assignment of the vibrational bands was based on comparisons with vibrational mode frequencies of homologous compounds.

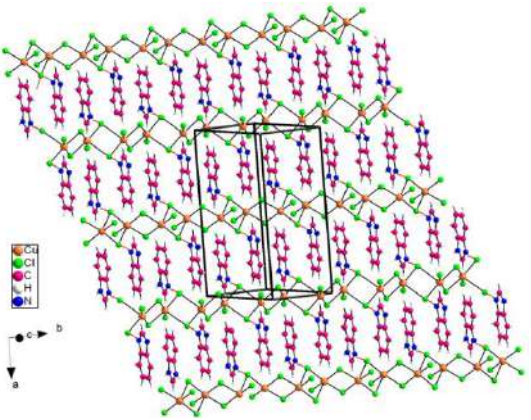

Fig: Packing diagram showing the alternation between different entities in $[C_7H_7N_2]\ Cu_2Cl_6$

EVALUATION OF SUFACE PROPRIETIES OF NEW TENTIOACTIVE AGENTS: AMPHIPHILIC CYCLODEXTRINS

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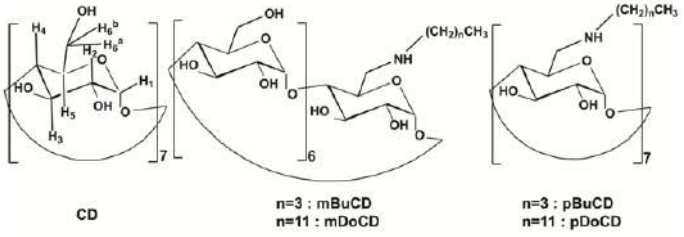
The preparation of aqueous suspensions of nanoparticles of amphiphilic β-cyclodextrins mono and per-modified on the primary face by aliphatic alkyl amino chains of varying length (C4 and C12) was studied. In the aim to evaluate the potentiality of the new amphiphilic β-CDs as excipients of the preparation and optimization of nanoparticles without using surface-active agent, stable aqueous suspensions of nanoparticles were prepared using sonication and nanoprecipitation methods. Amphiphilic β-CDs derivatives were characterized by ^{1}H and ^{13}C NMR, FTIR, mass spectroscopy, thermogravimetric analysis and elemental analysis. The solubility (s) of amphiphilic β-cyclodextrins has been determined by measurement of their conductivity and surface tension in aqueous solution at 25°C. Mono(6-deoxy-6-N-butylamino)- β-cyclodextrin (mBuCD) derivative exhibits a larger solubility compared to the other derivatives. The solubility decreases with increase of aminoalkyls chains number and length. The nanoparticles prepared from these new amphiphilic β-cyclodextrin derivatives according to optimized conditions have a diameter size ranging to 450 at 500 nm using sonication method and to 120 at 250 nm using nanoprecipitation method. Suspensions were stable over at least 45 days storage. The result indicate that mono(6-deoxy-6-N-dodecylamino)- β-cyclodextrin (mDoCD) proved to be the most efficient among the amphiphilic β-cyclodextrins due to its smaller diameter size and higher nanoparticles stability in aqueous solution.



Key words: amphiphilic β-cyclodextrins, amino alkyl-β-cyclodextrins, water solubility, nanoparticles.

THE DECOMPOSITION OF THE LAYERED DOUBLE HYDROXIDES OF Co AND Al: PHASE SEGREGATION OF A NEW SINGLE PHASE SPINEL OXIDE.

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Monophasic Co-Al-CO_3-like layered double hydroxides has been prepared by the coprecipitation method. It has been characterized by Rietveld refinement of the X-ray powder diffraction pattern, DTA-TGA, infrared and Raman spectroscopies. Its structure is trigonal, $R\bar{3}m$ with cell parameters a =0.3061(4) nm and c = 2.252 (3) nm. The decomposition of this hydrotalcite-like structure on heating up to 800°C yields to a single phase spinel oxide. Besides, infrared and Raman spectroscopies showed the presence of spinel-like domains. The results of Rietveld refinement have revealed that this compound has the $Fd\bar{3}m$ space group (a= 0.8088(4) nm), with crystallographic formula $[Co^{II}_{0.75}\ Al_{0.25}]^{8a}$ $[Co^{II}_{0.252}\ Co^{III}_{0.77}\ Al_{0.98}]^{16d}\ O_4$, which is of the general formula $Co_{1.77}Al_{1.23}O_4$. This structure is also validated by the charge distribution (CD) analysis.

KEYWORDS: Inorganic compounds; Rietveld; X-ray diffraction; Infrared spectroscopy; Raman spectroscopy.

SYNTHESIS, STRUCTURAL AND SPECTROSCOPIC CHARACTERIZATIONS OF MAGHEMITE γ-Fe2O3 PREPARED BY ONE-STEP COPRECIPITATION ROUTE

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Maghemite (γ-Fe_2O_3) is synthesized by one step coprecipitation method from mixed salt solutions containing Fe(II) and Fe(III) salts. This material was characterized using Rietveld refinement of the X-ray powder diffraction, heating stage Raman microscopy and Mössbauer spectroscopies. The Rietveld refinement of XRD pattern has indicated that the γ-Fe_2O_3 shows a cubic cell structure. The superstructure reflection related to the long-range ordering of cation lattice vacancies was not detected in the diffraction pattern. The extra peaks corresponding to the Lepidocrocite phase γ FeOOH in a small amount were observed in the XRD pattern. The crystallite size of maghemite compound is 18 nm calculated using Williamson-Hall method. Using Mössbauer spectroscopy, the result of Rietveld refinement was confirmed by the presence of magnetic sextet arising from the metallic iron of γ-Fe_2O_3, and a paramagnetic doublet inferring the presence of paramagnetic iron of γ FeOOH. Heating stage Raman microscopy reveals that the temperature of transition to maghemite phase up to hematite is 550 °C. The heating of our maghemite (γ-Fe_2O_3) powder up to 550°C yields a single phase of Hematite hexagonal-corundum structure with $R\bar{3}c$ space group.

KEYWORDS: Maghemite (γ-Fe_2O_3); coprecipitation, nanoparticles; Crystallographic studies; ^{57}Fe Mössbauer; phase transition.

STRUCTURE OF PALLADIUM COMPLEX WITH HETEROCYCLIC LIGAND

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The free radicals of nitronyl nitroxide are among the best group characterizing the free radicals. They were mainly used as probes in the biological systems, most of the previous studies on the interactions metal-nitroxide were bound to this domain. It is in the last decades when they became more used in magnetic materials. They allow the synthesis of molecular buildings with varied magnetic properties.

Several transition metal complexes with stable nitronyl nitroxide radical ligands have been prepared and extensively investigated.

We are interested in the study of the coordination of palladium with heterocyclic reactive ligands. So here, we report synthesis and structure characterization of palladium complex with stable nitronyl nitroxide radical ligand.

In the complex with nitronyl nitroxide radical, the palladium atom is bound to one chelating imino nitroxide radical through two N atoms, one from the pyridyl ring and the other from the imidazoline ring, the coordination of the metal is completed by two chlorine atoms in a cis configuration.

Key words: Palladium, Complex, Nitronyl Nitroxide, Radical, Ligand.

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Crystal structure and spectroscopic studies of a new hybrid compound $[C_7H_{13}N_3S]SO_4.H_2O$

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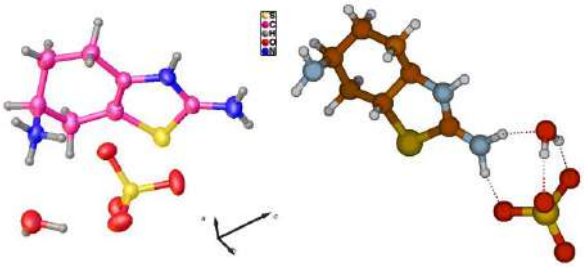
In this document we report the synthesis, structural characterization, IR and Raman spectroscopy of new hybrid compound namely 2,6-diammonium-4,5,6,7-tetrahydrobenzothiazole hydrated sulfate $[C_7H_{13}N_3S]SO_4.H_2O$. This compound crystallizes in the orthorhombic, space group $P2_12_12_1$ with: a = 7.0014(12) Å, b = 8.7631(15) Å, c = 19.773(3) Å, V = 1213.1(4) $Å^3$ and Z = 4. The crystal structure is determined and refined to R_1 = 0.046 and wR_2 = 0.103 using 3018 independent reflections. The structure of the title compound consists of an arrangement of inorganic layers of $[SO_4]^{2-}$ anions parallel to (001) plane. In this layer, we note the presence of three types of rings. Organic cations $(C_7H_{13}N_3S)^{2+}$ are situated between anionic chains. Multiple N-H…O and O-H…O hydrogen bonds connect cations and anions together.



Asymmetric unit and the optimized geometry of the title compound

Pyrite electro-Fenton degradation of the antibiotic sulfamethazine with Pt and BDD. Kinetics, mechanism and toxicity evolution

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The degradation of 0.20 mM sulfamethazine (SMT) was studied by heterogeneous electro-Fenton process pyrite-EF, in which pyrite powder was the source of Fe^{2+} catalyst. Using an undivided electrochemical cell equipped with Pt or BDD anode and a carbon-felt cathode. The higher oxidation power of pyrite-EF process using BDD anode was demonstrated. In both treatments, SMT and its products were destroyed by hydroxyl radicals formed at the anode surface from water oxidation and in the bulk from Fenton's reaction between Fe^{2+} and H_2O_2 generated at the cathode. Effect of pyrite concentration and applied current on the kinetics of oxidative degradation and mineralization efficiency have been investigated. SMT decay in this process followed pseudo-first order kinetics. The rate constant of the oxidation of SMT by $^{\bullet}OH$ has been determined to be 1.872×10^9 mol^{-1} L s^{-1} as determined by the competition kinetics method. A number of stable intermediate products have been identified using HPLC and GC-MS analyses. Ion chromatography confirmed the main released of SO_4^{2-}, NH_4^+ and NO_3^- ion in relatively smaller extent. Based on the identified reaction intermediates, a plausible reaction pathway was proposed for the mineralization by pyrite-EF process. Toxicity assessment by the Microtox method revealed that some cyclic intermediates are more toxic than the parent molecule.

Key words: Sulfamethazine, Pyrite Electro-Fenton, Electro Fenton, Wastewater treatment, Born-doped diamond anode, Platinum anode.

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Removal of phosphorous from wastewater by struvite precipitation for the production of a slow release crystal fertilizer

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Phosphorous (P) is an essential element and plays a key role for life on Earth. It stiffens the bone structure, is a major component of DNA and is involved in the metabolism of living cells. One of the most widely known metabolic processes is photosynthesis, which, amongst many other things, fuels the growth of plants. Modern agriculture uses fertilizers to supply plants with additional phosphorous in order to support photosynthesis and maximize yields. Still today, the primary source for fertilizers is phosphate rock, the supply of which is finite and estimated to be depleted within in the next century. In recent years, several techniques were developed to recycle phosphorous and prevent future scarcity of this vital element. This study examines a method to recover P from nutrient-rich wastewater by crystallizing struvite ($MgNH_4PO_4.6H_2O$) **[1]**.The valorization of the precipitate as a fertilizer constitutes an asset of the process. In this general context, the objective consists in developing a methodology and experimental approach. A synthetic effluent containing phosphorus NaH_2PO_4, magnesium $MgSO_4$ and ammonium NH_4Cl was used as a model solution to study the effect of pH on struvite precipitation efficiency in a stirred tank reactor**[2]**. The residual concentration of magnesium, ammonium and phosphorus allows to determine the conversion rate of these compounds and to study a likely formation of a co-product. for an effluent initially containing 600 mg (PO_4^{3-})/l and an $Mg:NH_4:PO_4$molar ratio of 1:1:1, 91% of the $PO_4^{3-}$was removed in only 120s.

Key words: Struvite- Precipitation- p-recovery- Stirred reactor

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Synthesis, structural study and physico-chemical characterization of a new Perchlorate $(C_6H_{10}N_2)$ $[ClO_4]_2$

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Studies of perchlorate salts containing organic cations have experienced considerable growth in recent years, because of their ferroelectric and dielectric behavior [1, 2]. It was shown that dynamics of pyridinium cations contribute mainly to the mechanism of solid-solid phase transition and lead ferroelectricity in these molecular-ionic crystals [3]. In this work, we report the synthesis and the characterization of a new organic perchlorate, $(C_6H_{10}N_2)(ClO_4)_2$.

The physicochemical properties of the title compound are discussed on the basis of a single-crystal X-ray diffraction investigation. The crystal structure determination reveals that the structure consists of one 3-ammoniomethylpyrinidinium dication and two perchlorate anions (Fig. 1). In the crystal, the three H atoms of the ammonium group are involved into N—H…Cl and N—H…O hydrogen bonds. These hydrogen bonds link the ionic units (NH_3^+ and $[ClO_4]^-$) to form columns running along the *a*-axis direction and situated at $y = z = 1/2$ (Fig.2). The organic groups are located between these columns *via* two kinds of hydrogen bonds. This compound is also characterized by infrared spectroscopy, solid state NMR and thermal analysis.

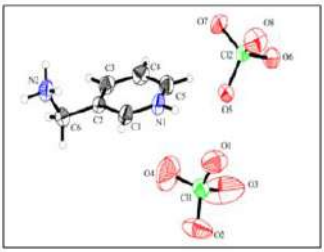

Fig. 1. Asymmetric unit of the title compound.

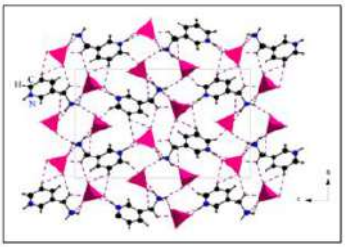

Fig. 2. The crystal packing of the title compound viewed along the *a* axis.

Keywords: Hybrid material; perchlorate; single-crystal; X-ray study

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Elaboration 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 confirme 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.

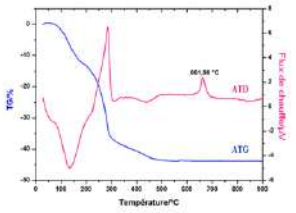

Figure 1: The thermogram DTA-TGA coupled of the Nanomaterial $Li_{2,8}Hf_{1,755}Si_{0,9}P_{2,1}O_{12}$

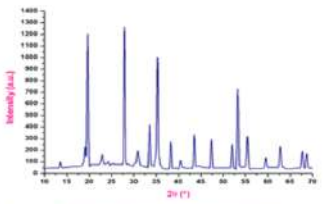

Figure 2: The X-Ray Powder diffratogram of the nanomaterial $Li_{2,8}Hf_{1,755}Si_{0,9}P_{2,1}O_{12}$

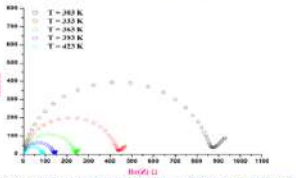

Figure 3 : The impedance diagram of the nanomaterial $Li_{2,8}Hf_{1,755}Si_{0,9}P_{2,1}O_{12}$

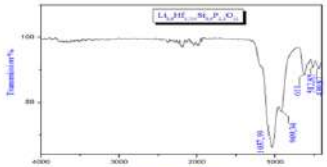

Figure 4 : The infrared absorption spectrum of the nanomaterial $Li_{2,8}Hf_{1,755}Si_{0,9}P_{2,1}O_{12}$

CRYSTAL STRUCTURE, VIBRATIONAL AND OPTICAL PROPERTIES OF A NOVEL ORGANIC-INORGANIC HYBRID MATERIALS $(C_9H_8NO)_2HgCl_4$

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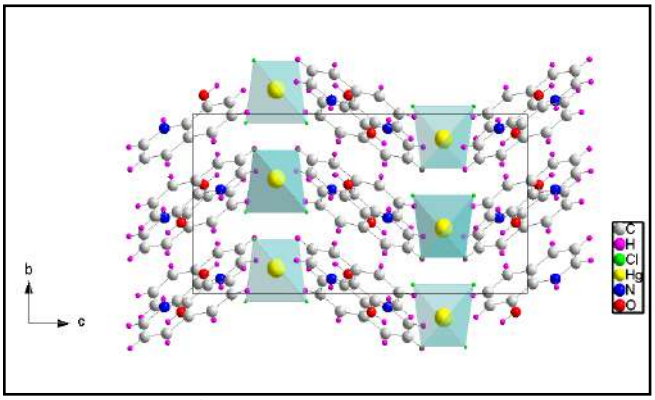
A novel organic inorganic hybrid material $(C_9H_8NO)_2HgCl_4$ was synthesis and studied by means of a single-crystal X-ray diffraction, UV–Visible absorption, IR, and Raman spectroscopy and PXRD techniques.

The crystal structure of bis(8-hydroxyquinolinium) tetrachloromercurate (II) crystallizes in the monoclinic space group C2/c with a = 15,303(3), b = 8.372(2), c = 16.549 (4) Å, (14), β = 91.068(6) and Z = 4. The crystal lattice is composed of discrete $[HgCl_4]^{2-}$ tetrahedra surrounded by $[C_9H_8NO]^+$ cations which are interconnected by means of hydrogen bonding contacts N-H…Cl and O-H…Cl, in which they may be effective in the stabilization of the crystal structure. The powder x-ray diffraction was used to confirme the homogeneity and purity of the crystals. Furthermore, the room temperature IR and Raman spectra of the title compound were recorded and analyzed. The UV-visible absorption spectrum is characterized by a strong absorption at 242 characteristic of n ➔ π* transition of organic cation.



Projection of the structure $(C_9H_8NO)_2HgCl_4$ in the (b, c)

Elaboration of a cermet from clay and powder of aluminum waste

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Composite materials have recently attracted much attention due to singular combination of their mechanical and microstructural properties. They are amply used in different applications. In the present study, the cermet was fabricated from clay and powder of aluminum waste. The aluminum has a double role: Firstly, to improve the mechanical strength of the ceramic part and secondly, to increases the porosity of cermets composites. Its mechanical properties were analysed by the Brazilian test. The evolution of the microstructure, the density and the mechanical strength of the ceramic was studied as a function of sintering temperature and percentage of aluminum added.

Key words: Ceramic, kaolin, Aluminum, Cermret, Sintering, Mechanical properties.

Degradation of azo dyes by rapidly solidified metallic particles

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Azo compounds are one of the most common families of dyes used in textile and leather treatments. An important step during the treatment of water polluted by these compounds, is the degradation of the compounds by decomposition of the -N = N- bonds, producing the de-colorization of the water. This de-colorization reaction can be activated by the presence of zero valent metallic particles.

The metastable structures generated during rapid solidification tend to increase the chemical activity of the alloys. Recently, it has been discovered that the use of metallic particles in a metastable phase (amorphous or nanocrystalline) multiplies significantly the efficiency of the de-colorization water-treatment step. Here we present the results obtained in the de-colorization of water using alloys based on different metals (Fe, Mn, Ni and Al) produced by rapid solidification and posterior ball milling. For some Al-containing alloys the results show a fast reaction, even in neutral pH conditions. In this work, the efficiency of the different metastable alloys in the de-colorization process and the effects of the metastable structure are presented and assessed.

Keywords: *Palabras clave,* powder, metastable alloys, de-colorization.

Synthesis and Characterization of New Quinolinium Dihydrogenphosphate, $(C_9H_8N)H_2PO_4$

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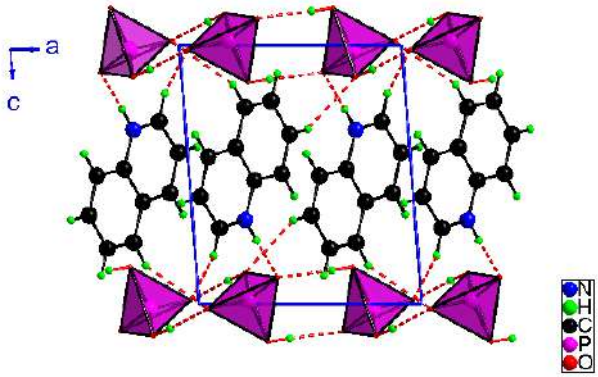
The crystal structure of a new organic-inorganic compound of stoichiometry $[C_9H_8N](H_2PO_4)$, was determined. The new quinolinium dihydrogenphosphate (QDHP) has been synthesized and single crystals were grown from aqueous solution by slow evaporation technique. Single crystal X-ray diffraction study on grown crystal reveals that they belong to triclinic crystal system with space group $P\bar{1}$. The unit cell dimensions are: a = 7.5866(9) Å, b = 8.0105(9)Å, c = 8.7048(9)Å, α = 77.379(4)°, β = 82.520(4)°, γ = 74.867(5)° with V = 496.82(10)Å^3 and Z = 2. The structure has been solved using a direct method and refined to a reliability R factor of 0.032. The structure is built up from anionic chains parallel to the *a*-axis. These chains are interconnected by the organic cations via hydrogen bonds so as to build a three dimensional network. FT-IR spectrum confirms the presence of the functional groups in the synthesized material. UV–Vis spectrum indicates that the crystal is transparent in visible region with a lower cut off wavelength of 285 nm and the band gap of QDHP was found to be 3.7 eV. The DSC analysis and $_{31}$P MAS– NMR spectroscopy are also reported for the QDHP.



Projection of $[C_9H_8N]H_2PO_4$ along the *b* axis.

Key words: Single crystal X-ray; IR Spectroscopy; UV-visible spectroscopy; DSC Analysis.

Synthesis and physico-chemical studies of a novel organo-metallic compound $CdCl_2(C_6H_4FNH_2)_2$

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Polynuclear d^{10}-metal complexes have been found to exhibit important structural and photoluminiscent properties [1]. Among these metals, cadmium gives rise to structural flexibility with coordination number varying between 4 and 8 with often severely distorted coordination geometries [2]. Herein, we report the crystal structure and the spectroscopic characterization of the new Cd(II) complex with the monodentate ligand 3-fluoroaniline, $CdCl_2(C_6H_4FNH_2)_2$. In the title complex, the Cd(II) ion is surrounded by four chlorine atoms and two nitrogen atoms, forming a chain of edge-charing octahedra spreading along the a-axis direction. These chains are interconnected via N-H…Cl hydrogen bonds to form inorganic layers parallel to the (a, b) plane (Fig. 1). The organic 3-fluorophenyl groups are inserted between these layers and are interconnected via C-H…F hydrogen bonds to perform an infinite three-dimensional network (Fig. 2). Intermolecular p–p stacking interactions between neighboring aromatic rings are also observed with a face-to-face distance of 3.6816 (4) Å. The ^{13}C and ^{19}F CP-MAS NMR spectra are in agreement with the X-ray structure. DFT calculations allow the attribution of the carbon peaks to the different atoms. The absorption bands of the $Cd_2Cl_4N_2$ units were identified by Raman spectroscopy. The solid-state UV–vis spectrum of the complex has been assigned to ligand and charge transfer transitions.

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

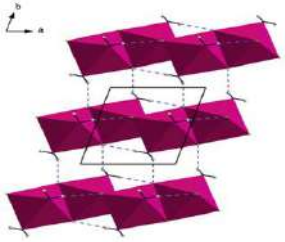

Fig. 1. Inorganic layer in $CdCl_2(C_6H_4FNH_2)_2$ at z =1/2.

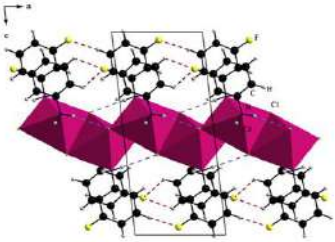

Fig. 2. Projection along the b-axis of the $CdCl_2(C_6H_4FNH_2)_2$ structure.

References

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Synthesis, structural characterization and spectroscopic studies of a novel coordination compound $ZnCl_2(C_6H_4FNH_2)_2$

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The synthesis and characterization of metal organic frameworks has been an active area of research in coordination chemistry [1]. This arises not only from the fundamental properties of these materials such as their intriguing topological frameworks, but also from their unexpected potential applications in a wide range of various fields such as engineering, device manufacturing, materials science,etc. [2].We report here the synthesis, the crystal structure and the spectroscopic characterization of a new Zn(II) complex with the monodentate ligand 3-fluoroaniline, $ZnCl_2(C_6H_4FNH_2)_2$. In the title material, the Zn(II) ion, located on a special position, is tetracoordinated by two nitrogen atoms of two 3-fluoroaniline monodentate ligands and two chlorine ligands. In the atomic arrangement, the $ZnCl_2(NH_2)_2$ entities are interconnected via N-H…Cl hydrogen bonds to form inorganic layers parallel to the (b, c) plane (Fig. 1). The organic 3-fluorophenyl groups are located between these layers and connect each other via C-H…F hydrogen bonds to perform an infinite three-dimensional network. An F…F interaction between neighboring fluorine atoms contributes also to the structure cohesion (Fig. 2). The ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray structure. DFT calculations allow the attribution of the carbon peaks to the different independent atoms. Solid-state UV-Vis spectrum of the complex has been assigned to ligand and charge transfer transitions.

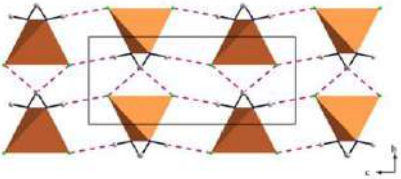

Fig. 1. Inorganic layer in $ZnCl_2(C_6H_4FNH_2)_2$.

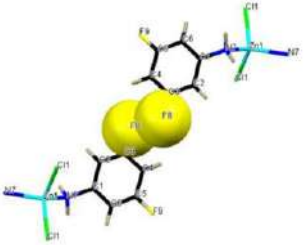

Fig. 2. F…F interaction in $ZnCl_2(C_6H_4FNH_2)_2$.

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

References

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Mo-M (M=Ni, Co) materials dispersed on silica and alumina supports for catalytic evaluation in ethane oxydehydrogenation

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Unsupported and supported M-Mo catalysts (M = Ni or Co) over γ-alumina or silica were prepared, characterized and tested in oxidative dehydrogenation of ethane (ODHE). Coprecipitation route is used for catalysts synthesis and TG-DTA, HT-XRD, XRD, LRS, FTIR, Uv-Vis-NIR, B.E.T, SEM-EDX, XPS and SAA are used for their characterization. A novel soft route was developed by combining the coprecipitation and impregnation methods for supported catalysts synthesis. For all solids, the $MMoO_4$ phase was identified by XRD as a main phase. Physicochemical and catalytic properties of oxides depended strongly on the transition metal (M) nature. They were, also, markedly affected by interactions-type between support and active metal. The catalytic activity in ODH of ethane, measured without any pretreatment previously, was higher with alumina supported catalyst than with unsupported $MMoO_4$ or silica supported $MMoO_4$ in spite the low specific surface area of $NiMoO_4/Al_2O_3$ catalyst (5 m^2/g). The presence of $NiAl_2O_4$ phase, observed for $NiMoO_4/Al_2O_3$ system, seem to play an important role in oxidative dehydrogenation of ethane. For $MMoO_4/Al_2O_3$ system and in our catalytic conditions, the measurements showed that C_2H_6 conversion and C_2H_4 selectivity can reach 43% and 85%, respectively. The relationships between textural and structural properties of catalysts with catalytic performance were proposed.

Key words: characterization, $NiMoO_4$, $CoMoO_4$, ODH of ethane, ethylene

Rheological behavior of cellulose nanofiber suspensions from marine biomass

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[a] *University of Monastir-Faculty of Science-URCAE 13 ES 63 -Tunisia.*
[b] *Univ. Grenoble Alpes, LGP2, F-38000 Grenoble, France.*
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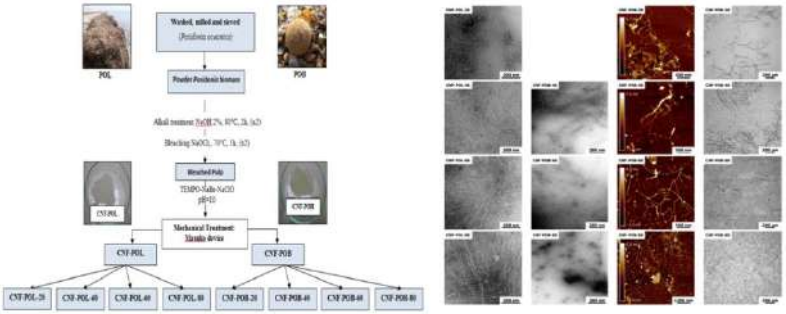
In this work, different grades of cellulose nanofibrils (CNF) were prepared from *Posidonia oceanica* balls and leaves (POB and POL). Pretreatment using 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO)-mediated oxidation was performed to facilitate the fibrillation during ultrafine friction grinding process. The ensuing CNF batches were compared in terms of morphology and degree of fibrillation. The rheological properties of the produced CNF suspensions were also analyzed for varying doses of sodium hypochlorite used during the TEMPO-mediated oxidation procedure. The stronger fibrous network structures were formed when increasing the oxidant concentration, which was confirmed by the increase of the storage moduli value. *Posidonia oceanica* balls were found to undergo stronger fibrillation and, consequently, to form stronger networks, compared to Posidonia oceanica leaves, when using equivalent concentration of the oxidizing agent.



Keywords: *Posidonia oceanica*; cellulose nanofibrils (CNF); Nanofibrillated cellulose (NFC); TEMPO-oxidation; TEM; rheological behavior.

Acknowledgements
The authors express their sincere gratitude to Dr. Jean-Luc Putaux (CERMAV, Grenoble) and Dr. Frédéric Pignon (Laboratory of Rheology and Processes, Grenoble), for their support and availability as well as to the "PHC-UTIQUE CMCU" (project 13G1114) and "CMIRA" for financial support.

Analysis of thermomechanical reprocessing effects on Silica reinforced rubber-toughened materials.

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The impact of recycling by grinding and re-extrusion on the physical and mechanical properties of silica reinforced polypropylene (PP)/ethylene propylene rubber (EPR) nanocomposites was investigated.
The considered EPR content was 20 wt%, and the investigated number of recycling passes (extrusions) was set to 0, 1, 2 and 3cycles using a high shear twin screw extruder by varying the screw rotation speed (300; 800 and 1200 rpm). The concentration of silica nanoparticles was set to 3wt% and the nanocomposites were compatibilized with polyethylene modified with maleic anhydride MAPE.
Morphological, and thermomechanical analyses were carried out on as-produced and reproduced samples in order to explore different parameters such as number of extrusions, shear rate level, SEM (specific mechanical energy) and residence time, on properties of the recycled systems.
The analysis of the different findings illustrate that the properties were affected by the multiple extrusion. In particular, this process provokes a decrease of the molecular weight and the melt flow. The first recycling procedure induced an increase of the Young's modulus and tensile yield stress, while for higher recycling numbers, these two parameters changed. The MAPE copolymer stabilized the tensile elongation at break at 3 recycling procedures due to a decrease of the size of EPR nodules and to a homogenization of their shape, while that of neat PP continuously decreased with recycling numbers.

Key words: thermomechanical, high shear, rubber-toughned

SYNTHESIS OF BIOHYBRID LAYERED DOUBLE HYDROXIDE-BOVINE SERUM ALBUMIN: STRUCTURAL AND MORPHOLOGICAL STUDIES

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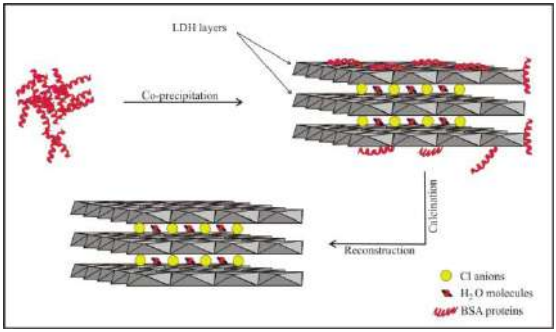
b) Laboratoire des Sciences Analytiques UMR CNRS 5280, Université Claude Bernard-Lyon 1, 5 Rue de la Doua, 691000 Villeurbanne, France. (b)

The present work introduces a new biohybrid material involving layered double hydroxide (LDH) and biomolecules such as bovine serum albumin (BSA). BSA has been chosen as a model protein, being immobilized [1] onto MgAl LDH material via co-precipitation route in the first time. Then the resulting biohybrid has been calcined/regenerated [2] to remove the BSA biomolecules and to get macroporous materials. To better study the interaction and association between BSA biomolecules and LDH particles, structural and morphological investigations were carried out, using X-ray diffraction (XRD) study, Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM), respectively. However, the modified LDH materials showed a decrease in crystallinity as compared to the unmodified LDH. A bipyramid aggregates were obtained up to 1.



Schema representation of different reactions steps

Key words: Layered Double Hydroxide (LDH), Bovine Serum Albumin (BSA), immobilization

References

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POLY [PENTAAQUASTRONTIUM (II) BIS [HYDROGENSQUARATE] HEMIHYDRATE]

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[a] *Unité de Recherche Chimie de l'Environnement et Moléculaire Structurale (URCHEMS), Université des frères Mentouri Constantine, route Ain El-Bey, Constantine, Algérie*
[b] *Sciences Chimiques de Rennes UMR 6226 CNRS – Université de Rennes 1, F- 35042 Rennes Cedex, France*
[c] *Université Européenne de Bretagne UEB, F- 35000 Rennes, France*

Many organic species have been used as templating agents or as bases of hybrid organic–inorganic compounds. Squaric acid is known as an oxocarbon possessing an aromaticity, strong acidity, hydrogen bonding, and metal chelating properties. Squaric acid and its anion (Hsq^- and sq^{2-}) is also a useful tool for constructing crystalline architectures and they proton donating and accepting capabilities for hydrogen bonding. This molecule presents high degree of electron delocalization, which is a very interesting characteristics discussed in many structural and is very important in crystal packing. In the crystal structure of synthetically prepared title compound, the strontium atoms form infinite chains which are bridged by the hydrogensquarate group acting as a bidentate ligand in a *trans* position (fig.1). The Sr atom is coordinated by the O atoms from three hydrogensquarate anions and six water molecules. The nine-fold coordination polyhedral of strontium can be described as a monocapped square antiprism (Fig. 2).

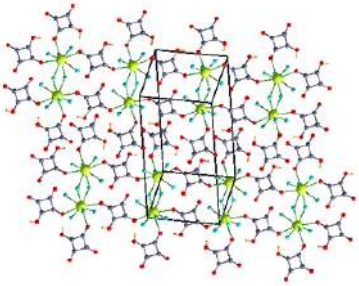

Fig.1

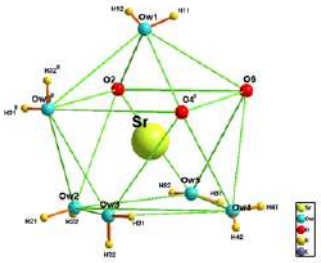

Fig.2

Key-words: Squaric acid, X-ray diffraction, polyhedral, hydrogensquarate group

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SYNTHESIS, CRYSTAL STRUCTURE, VIBRATIONAL SPECTROSCOPY AND THEORETICAL STUDIES OF A NEW Bi(III) HALIDE COMPLEX: [p-$C_7H_{10}N]_3[BiBr_6].H_2O$

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Bismuth complex chemistry has attracted significant interest not only because of the low cost and high abundance of bismuth resources on Earth, but also due to their practical applications in pharmaceutics and medicine, catalysis, superconductors and semiconductors, etc. [1]. We report in the present work, the synthesis and the characterization of a new hexabromobismuthate compound: [p-$C_7H_{10}N]_3[BiBr_6].H_2O$. Single-crystal X-ray diffraction analysis revealed that the complex crystallizes in the monoclinic system with $P2_1/c$ space group. The crystal lattice is composed of discrete $[BiBr_6]^{3-}$ anions surrounded by p-toluidinium cations. Theoretical calculations were performed to study the molecular structure (Figure 1), the vibrational spectrum and the NBO natural charges of the investigated molecule in the ground state. All calculations were carried out at the DFT/B3LYP/LanL2DZ level of theory using Gaussian 09 program [2]. Good consistency is found between the calculated and the experimental vibrational wavenumbers. The NBO analysis reveals that the N–H …Br intermolecular interactions significantly influence crystal packing in this molecule.

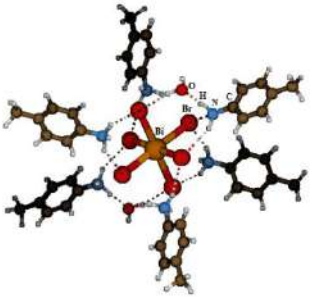

Figure 1. Optimized structure of [p-$C_7H_{10}N]_3[BiBr_6].H_2O$ compound at DFT/B3LYP/LanL2Dz level.

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

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Structural, electrical and magnetic properties of $Pr_{0.4}$ $Gd_{0.1}Sr_{0.5}MnO_3$ manganite

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The Structural, electrical and magnetic properties have been studied in $Pr_{0.4}$ $Gd_{0.1}Sr_{0.5}MnO_3$ manganite prepared by the solid state reaction method. Rietveld refinement of the X- ray diffraction patterns shows that our sample is single phase and crystallizes in the orthorhombic structure with Pbnm space group. Scanning electron microscopy (SEM) shows that the grains are irregularly spherical like shaped with sizes of 460 nm. Electrical resistivity measurements versus temperature showed that our sample exhibit different behavior as a function of temperature**.** Magnetic measurements versus temperature in an applied magnetic field of 0.05T show that our sample exhibit two distinct transitions, one from PM to FM at T_C = 250K, this transition is followed by another one to AFM at T_N=125K. The absolute value of the maximum of magnetic entropy change $|\Delta S_M|$ for an applied magnetic field of 2 T is 1.041J kg^{-1} K^{-1}.

Phase relationships in the KNO_3-$NaNO_3$

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Phase relationships in the KNO_3-$NaNO_3$ binary system at normal condition were investigated by Rietveld method from powder X-ray diffraction (XRD). The solid limit solubility was determined and showed that for samples with a high potassium content, the solid solution (denoted as κ)$K_{(1-x)}Rb_xNO_3$ adopts orthorhombic symmetry (Pmcn) and Na^+ may substitute K^+ up to 15 mol %. In the Na-rich phase (denoted as ν) $Na_{(1-y)}K_yNO_3$ with R-3c space group miscibility was determined up to 15 mol %. Furthermore, the quantitative relative concentration of the components of a two-phase samples and amorphous content were calculated.

Key words: Phase relationships in the KNO_3-$NaNO_3$, XRD, Rietveld refinement, QPA.

Hydrothermal synthesis, Structural studies and decoding Intermolecular Interactions with Hirshfeld Surface Analysis of a new Non-Centrosymmetric Microporous compound: $[Fe_4(H_2O)_2F_4(H_2PO_4)(PO_4)(HPO_4)_2](H_2O)_{2.92}$

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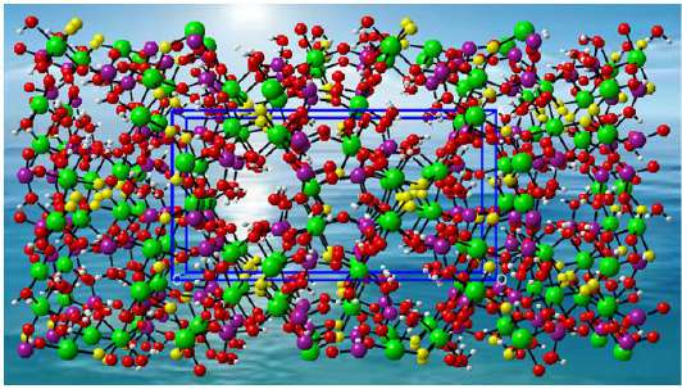
The synthesis of $[Fe_4(H_2O)_2F_4(H_2PO_4)(PO_4)(HPO_4)_2](H_2O)_{2.92}$ was done and its single crystals were grown by hydrotermal method. Crystal structure is determined by single crystal X-ray diffraction analysis and reveals that it belongs to the orthorhombic system with four molecules in the unit cell (space group $P2_12_12_1$). The various vibration patterns of the specimen have been investigated by Fourier transform infrared and Fourier transform Raman spectroscopy. The inorganic sheets, stacked along [010] highlight the presence of FeF_2O_4 and $FeF_2O_3H_2O$ octahedra and H_2PO_4, HPO_4 and PO_4 tetrahedra. The new d_{norm} Hirshfeld surface and the breakdown of fingerprint plots were used for visualizing and exploring of title compound for quantifying intermolecular interactions in crystal lattice.

Graphical abstract



View of the structure along the b-axis showing the connection of the layer via the PO_4 tetrahedra and octahedra $Fe(O,F)_6$

Keywords: Non-centrosymmetric structure; iron fluorinated phosphate; Hirshfeld Surface Analysis.

Synthesis, Thermal stability, AC electrical conductivity and dielectric properties of the Bis [8-Hydroxyquiolinium] tetrachlorocobaltate, $(C_9H_8NO)_2CoCl_4$.

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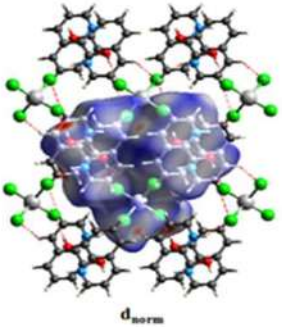
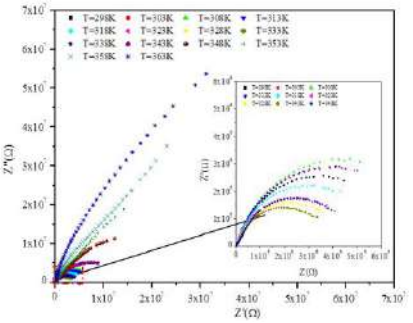
A new organic-in organic compound, Bis [8-Hydroxyquiolinium] tetrachlorocobaltate, $(C_9H_8NO)_2CoCl_4$ has been obtained by slow evaporation at room temperature and characterized by single cristal X-ray diffraction, Hirshfeld surface analysis, electrical and dielectric properties. Atomic Hirshfeld surface and two- dimensional fingerprint plots estimate the intermolecular interactions accountable for the generation of crystal packing. The material was characterized by impedance spectroscopy technique measured in the 209–5 Hz frequency range from 298 to 443K. Besides, the Cole–Cole (Z" versus Z') plots were well fitted to an equivalent circuit built up by a parallel combination of a resistance (R), capacitance (C) and fractal capacitance (CPE). The frequency dependence of the conductivity is interpreted in tern of Jonschers law: $\sigma(\omega) = \sigma_{dc} + A\omega^s$. It was found that the data of the ac–measurement follow the correlated barrier–hopping (CBH) model and the model's parameters were determined. The dielectric characteristics show diffuse phase transition phenomena for title compound.



Hirshfeld surface of the asymmetric units.



Cole-Cole plots of the $[C_9H_8NO]_2CoCl_4$ compound at several temperatures.

INFLUENCE OF MICROSTRUCTURAL PARAMETERS OF THE DAMAGE OF AN INDUSTRIAL Al-Zn-Mg ALLOY (7020)

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Mesmacque GERARD[b] and Mohamed DAMMAK[a]

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Metal structures are naturally submitted to varying stresses (cyclic) in which the time, although lower than the yield limit of the material may lead to breakage when their application is repeated a number of times: this is the process of fatigue damage. Despite periodic checks on service parts, highlighting the fatigue phenomenon is sometimes difficult because it is very slow and allows a crack to propagate until a critical value. Structural fatigue is particularly insidious because of its progressive nature hidden. This behavior is even more serious than fatigue cracking often leads to a sudden break which can cause an accident.

The objective of this work was to understand the influence of microstructural parameters on the fatigue strength of an industrial aluminum alloy noted 7020. We focused our attention on the relationships between changes in the microstructure and ageing Study the alloy. We proved close liaison between the yield strength and resistance to aging. Indeed, the metallurgical state having the highest yield has the largest life duration and cracking is the slowest. These results open up many opportunities for future work, both from the industrial point of view than scientific. Despite it occupies a very large fraction of time compared to the total life of an alloy; the time of priming hardly interests a core mechanic. Improved performance over this period, with adequate surface treatment, can significantly increase the lifespan of a material. It should also be noted that, to a certain damage threshold, it is possible, by appropriate mechanical and / or thermal treatments, to remove damage and thus return to an undamaged initial state.

Keywords: Aluminum alloys, Precipitation hardening and Damage.

Structure and thermochemistry of calcium sodium phosphate glasses

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The poor chemical durability of phosphate glasses limits their use in common applications. However, this property makes them candidates in alternative fields such as fertilization [1] and treatment of trace element deficiency in cattle [2]. Phosphate glasses are also developed as bioactive materials in bone repair and tissue engineering [3]. Thus, the fundamental study of those materials seems necessary to adapt the glass composition to the applications. Therefore, this work focuses on the synthesis, physical, structural and thermochemical properties of the phosphate glasses with the formula $(100\text{-}x)NaPO_3\ x(CaO)$.

Glasses were prepared with the melt quenching technique using the appropriate amount of $CaHPO_4$ and $NaH_2PO_4.H_2O$. The mixture was heated at 400°C in order to evacuate water and the melt was obtained between 750°C and 1100°C from $NaPO_3$ to $75NaPO_3$-25CaO respectively. Up to this content of CaO, the glasses are obtained readily after the quenching.

The glass density, glass transition (T_g) and crystallization temperatures increase (T_c), revealing the shrinking and the reticulation of the phosphate network when the calcium oxide is introduced. Furthermore the addition of CaO enhances the chemical durability of the glasses.

The structure of the glasses was investigated by FTIR, Raman and ^{31}P solid state NMR. The results show that the metaphosphate chains are disrupt. The CaO added create polyphosphate chains and cyclophosphates.

The heat of dissolution of the glasses in 4,5% weight H_3PO_4 was measured using a SETARAM C-80 microcalorimeter. This heat decreases when the calcium oxide amount increases. This variation could be explained by the diminution in the average chain length of polyphosphate groups.

Key words: calcium phosphate glass, chemical durability, structural investigation, microcalorimetry.

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STRUCTURAL, THERMAL, SPECTROSCOPIC AND MAGNETIC PROPERTIES OF $(C_5H_6ClN_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 1.5H_2O$

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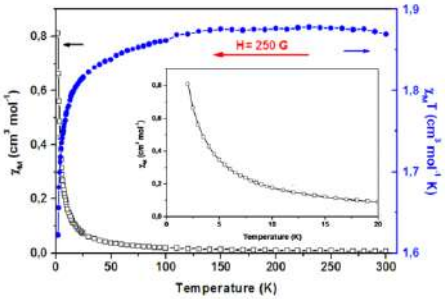
It is well known that the use of hydrogen-bonding and π-π stacking interactions is a successful way to obtain a large variety of hybrid (organic/inorganic) compounds with extended supramolecular networks through self-assembly [1-2]. Following this strategy, the compound $(C_5H_6ClN_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 1.5H_2O$ has been synthesized by the reaction of chromium(III) salt in ethanol with 2-amino-5-chloropyridine in the presence of two equivalents of oxalic acid. The pink-violet crystals were structurally characterized by X-ray diffraction. Thermal stability and spectroscopic properties were discussed on the base of thermogravimetric analysis and UV-Visible absorption study respectively. The temperature dependence of the magnetic susceptibility measured down to 2 K reveals the occurrence of weak antiferromagnetic interaction between the chromium centers in accordance with the crystal structure.



Thermal variation of χ_M (□) and χ_MT (●) for $(C_5H_6ClN_2)[Cr(C_2O_4)_2(H_2O)_2]\cdot 1.5H_2O$. The inset shows the susceptibility curve in the low temperature region 0 – 20 K.

Keywords: Crystal structure; Magnetic measurements; Thermal analysis; X-ray diffraction.

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SYNTHESIS AND STRUCTURAL STUDY OF $Ag_{0.32}Na_{0.68}Nb_4AsO_{13}$

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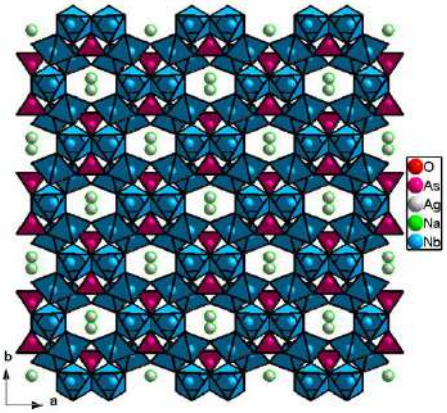
For research of layer or tunnel structures with fast alkalin or pseudo-alkalin–ion mobility, A-Nb-As-O system has been investigated. Several phases have been identified [1-4] among which the compound $Ag_{0.32}Na_{0.68}Nb_4AsO_{13}$. This stoichiometry is confirmed by the result of the structural determination carried out by X-ray diffraction on single crystal. This study shows that this compound has a three-dimensional framework delimiting tunnels, with hexagonal section, where Na^+ and Ag^+ cations are located.



Projection of the structure of $Ag_{0.32}Na_{0.68}Nb_4AsO_{13}$ along c axis showing tunnels where Na^+ and Ag^+ cations are located.

Keywords: three-dimensional framework, X-ray diffraction, crystal structure.

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Zn(II) Chloride complex with 1,2 diaminocyclohexane: Synthesis, molecular structure and vibrational investigation

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The synthesis and characterization of a novel compound [1,2-$C_6H_{10}(NH_3)_2$]$ZnCl_4$ was presented in this study. After an X-ray investigation, it has been shown that the title compound crystallizes at 296 K in a monoclinic system, in the space group C2/c with the following lattice parameters a = 32.394(9) Å, b = 12.217(4) Å, c = 10.175(3) Å and β = 97.852(13) with Z = 12 and the refinement converged to R = 0.034 and wR = 0.065. The spectroscopic properties of the compound were investigated by FT-IR, Raman and MAS-NMR and UV spectroscopy. The structure consists of isolated $[ZnCl_4]^{2-}$ tetrahedral anions and 1,2 diammonium cyclohexane $[1,2\text{-}C_6H_{10}(NH_3)_2]^{2+}$ cations, which are connected together via N–H…Cl, hydrogen bonds. Figure 1 shows also the different alternation of the two organic molecules along *a*-axis.

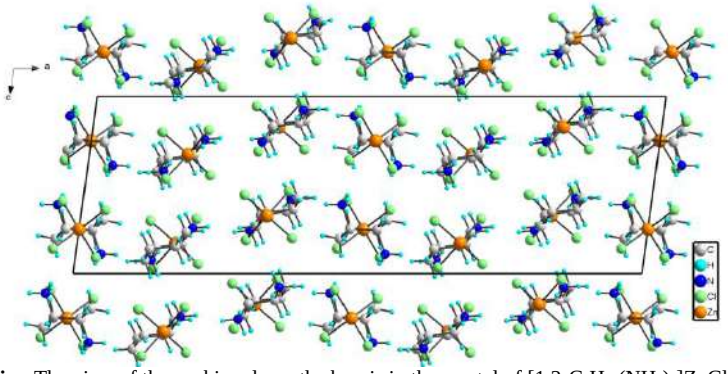

Fig : The view of the packing down the b-axis in the crystal of [1,2-$C_6H_{10}(NH_3)_2$]$ZnCl_4$.

PREPARATION, CHARACTERIZATION OF MAGNESIUM CARBONATE CO-SUBSTITUTED FLUORAPATITE NANOPOWDERS FOR BIOMEDICAL APPLICATIONS

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Properties of hydroxyapatite (Hap) or fluorapatite (Fap), such as bioactivity, biocompatibility, solubility, and adsorption properties can be tailored over a wide range by modifying the composition via ionic substitutions. The aim of this work concerns the preparation and the characterization of magnesium-carbonate co-substituted fluorapatite nanopowders (Mg-CO_3-Faps).

Mg-CO_3-Faps with similar Mg/Ca+Mg molar ratio of 0.1 and different amount of carbonate were prepared by the precipitation method. Several techniques such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were utilized to characterize the obtained powders. Results showed that the prepared compounds are pure B-type carbonate apatite. The simultaneous incorporation of CO_3 and Mg ions into the fluorapatite caused the following effects: a decrease in a-dimension with a slight increase in c-dimension and a decrease in crystallinity associated with reduction in crystallites size. Moreover, the morphology changed progressively with increasing CO_3 content from rod-like particles poorly crystallized to small spherical particles ranging from 30–50 nm. Nanometer size characteristic for obtained powders renders them similar to natural bone [1]. Furthermore, Granules with a smooth spherical geometry were useful in growth and attachment of bone tissue for improving osseointegration [2]. Therefore, the powders thus synthesized (Mg-CO_3-Fap) may be have applications in bone tissue engineering.

Key words: Magnesium-carbonate co-substituted fluorapatites. Precipitation. Characterization methods. Nanostructured materials.

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Nanostructured VO_2(B) photocatalysts for degradation of methylene blue under visible light

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The highly colored effluent generated by many industries, using dyes to color their products, poses not only severe public health concern, but also many serious environmental problems. In this context, heterogeneous semiconductors photocatalysis can be applied to resolve many environmental problems in addition to water purification. The TiO_2 is the most commonly used photocatalysts but its activation is limited to the ultraviolet light which is about 3-5 % of the solar spectrum explaining the current low solar photoconversion efficiency of this photocatalyst. The need of photocatalysts more efficient under visible light for further solar applications catalyzed the research in this field [1-4].
This work deals with the VO_2(B) nanobelts prepared by solvo/hydrothermal treatment. The as-synthesized samples were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Fourier Transform Infrared spectroscopy (FTIR) and UV-Visible spectroscopy. The photocatalytic activity of VO_2(B) nanobelts has been evaluated by the degradation of methylene blue under visible light irradiation. The photocatalytic experiments revealed that the VO_2(B) nanobelts showed promising results for degradation of methylene blue. Indeed, after the lapse of 120 min the methylene blue was completely photodegraded.

Key words: VO_2(B), visible-light photocatalysis, Methylene blue.

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Fast preheating of olive paste by microwave: Impact on olive oil quality and Yield

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Olive paste conditioning using microwave technology was integrated into an olive oil extraction plant using laboratory-scale microwave-assisted apparatus. The aim of this work was to evaluate the impact of the microwave treatment as well as the effect of sodium citrate concentration on legal parameters, oil yield, chlorophyll and carotenoids content. The short process time of the rapid microwave treatment resulted in a low oxidation of the olive oil and consequently a reduction in the peroxide value compared with the conventional method. Therefore, a fast preheating of olive paste not longer than 72 s followed by 20 min malaxation is the most promising extraction technique to improve the oil recovery, quality indices and pigment content of the extracted olive oil without compromising other quality parameters. Microwave processing was therefore confirmed as an attractive alternative to the conventional malaxation, with the main advantages being the rapid processing time and the high olive oil quality.

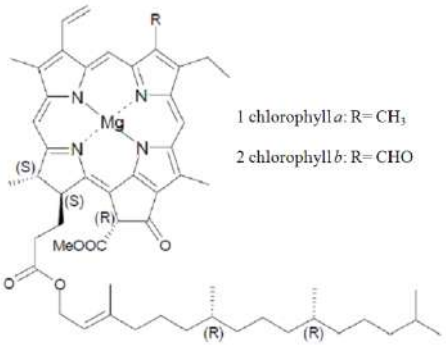

Figure1: Chemical structure of chlorophyll *a* et *b*.

Key words: Microware treatment, olive oil, Fast preheating, sodium citrate.

Conductivity behavior of a new mixed cesium-ammonium dihydrogen phosphate–arsenate: $Cs_{0.55}(NH_4)_{0.45}H_2(PO_4)_{0.39}(AsO_4)_{0.61}$

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The mixed cesium ammonium dihydrogen phosphate arsenate, $Cs_{0.55}(NH_4)_{0.45}H_2(PO_4)_{0.39}(AsO_4)_{0.61}$ (CADAP) crystallizes in the tetragonal system $I\bar{4}2d$ at room temperature with the following parameters: a = 7.6473(1) Å; c = 7.6803(1) Å and Z = 4. Two distinct endothermic peaks at T_1 = 204 and T_2 = 407 K, were detected and studied by differential scanning calorimetric, dielectric measurements, impedance and modulus spectroscopy. The evolution of the dielectric constant as a function of frequency and temperature revealed that the first transition (204 K) to be first order and antiferroelectric paraelectric. The high temperature phase transition (407 K) leads to a superionic protonic phase, characterised by an unusual high conductivity. The conductivity relaxation parameters associated with the high-disorder protonic conduction have been determined from analysis of the M''/M''_{max} spectrum measured in a wide temperature range. Transport properties in this material appear to be due to proton hopping mechanism.

Keywords: Inorganic materials; $Cs_{0.55}(NH_4)_{0.45}H_2(PO_4)_{0.39}(AsO_4)_{0.61}$; Thermal behavior; Conductivity investigation

Physicochemical characterization and in-vitro drug dissolution study of niflumic acid/ß-cyclodextrin complexe inclusions.

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The Niflumic acid is a non-steroidal anti-inflammatory drug (NSAID) and a poorly water-soluble derivative of nicotinic acid with relatively low bioavailability. So, the present paper is devoted to the preparation of new formulations based on the combination of niflumic acid and the beta-cyclodextrin in order to optimize its pharmaceutical and physic-chemical properties. The effect of beta-cyclodextrin on the aqueous solubility and dissolution rate of niflumic acid was studied and elucidated. The binary systems of the compounds (1:1) molar ratios were prepared by different methods and the inclusion complexes were characterized by FTIR, XRD, and UV. The interaction between niflumic acid and ß-cyclodextrin in solution was studied by phase solubility analysis. The results of this study revealed that the complexation of niflumic acid with ß-cyclodextrin can improve the therapeutic efficacy of the drug through the greater efficiency of the drug dissolution.

Key words: ß-cyclodextrin, complexation, niflumic acid, phase solubility.

The structural and magnetic studies of new phases formed after oxidation electrochemical and chemical reduction of $Na_4VO(PO_4)_2$

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Polyanionic frameworks have been the object of numerous studies as electrode materials for Li or Na-ion batteries **[1, 2]**. This is exemplified by the vanadium based compounds such as $LiVOPO_4$ **[3],** $Li_3V_2(PO_4)_3$ [4], $Li_4VO(PO_4)_2$ **[5, 6]**, with a great interest as cathode materials for rechargeable lithium batteries. These phases are showing reversible capacity of about 130 mAh/g at the redox potential ~ 4.0 V. the iso-structural phase based on sodium vanadium phosphate have been also studied: $NaVOPO_4$ **[7]** leading to a capacity of 90mA h g^{-1} at 1/15 C potentiel redox ~3,6V Versus Na^+/Na, $Na_3V_2(PO_4)_3$ **[8, 9]** given a capacities of 147, 136, 118, and 103 mA h g^{-1} obtained at rates of 0.1, 0.2, 0.5, and 1 C.

In the continuing search for new intercalation host for sodium ion batteries, we have investigated the phase $Na_4VO(PO_4)_2$. This phase show a 3D type structure built of Zigzag-like infinite chains of the corner-sharing VO_6 octahedra run along the c-direction of the unit cell. Within each chain the octahedral are additionally linked to each other by phosphate groups.

The $Na_4VO(PO_4)_2$ phase synthesized by the solid state route in sealed tubes at 700°C starting from $Na_4P_2O_7$ and VO_2. We found that 0.6 sodium could be extract reversibly leading to a capacity of 49 mAh/g at 3.5V vs Na^+/Na. From chemical oxidation and reduction, we could also prepare the fully oxidized and reduced phases. These results will be discuss in details in the communication.

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Synthesis, crystal structure, physico-chemical characterization and dielectric properties of a new hybrid material 1-Ethylpiperazine-1,4-diium tetrachlorocadmate

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A new organic-inorganic hybrid metal complex, 1-Ethylpiperazine-1,4-diium tetrachlorocadmate was synthesized and the structure was determined by single crystal X-Ray diffraction analysis. The title compound crystallizes in the orthorhombic system with space group Pbca. The unit cell parameters are a = 11.5129 (2) Å, b = 9.7801 (2) Å, c = 23.8599 (4) Å with Z = 8 and V = 2686.56 (8) $Å^3$. The examination of the structure shows that Cd(II) is coordinated by 4 chlorine atoms and adopts a tetrahedral geometry.

Three-dimensional frameworks of the title compound are produced by N-H···Cl and C-H···Cl hydrogen bonding. IR, Raman and UV-Visible spectroscopies were also used to characterize this complex (Fig. 1). Moreover, the fluorescent properties of the compound have been investigated in the solid state at room temperature. Solid state ^{13}C MAS NMR spectroscopy results are in agreement with the X-ray structure.

Differential scanning calorimetry (DSC) has revealed a structural phase transition of the order-disorder type around 373 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.

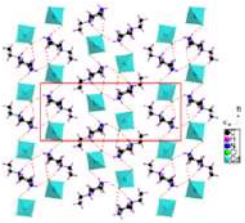

Fig. 1 View of the ribbons showing the R^4_2 (10) and R^4_2 (12) motifs. Hydrogen bonds are shown as dotted lines.

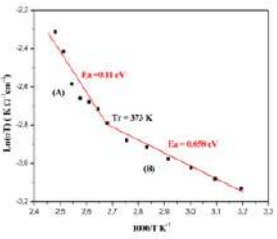

Fig. 2 Temperature dependence of the conductivity of $(C_6H_{16}N_2)CdCl_4$.

Key words: X-Ray diffraction, IR-Raman spectroscopies, NMR spectroscopy Photoluminescence, Phase transition, Dielectric properties

IMPEDANCE SPECTROSCOPY AND DISORDERED STRUCTURE OF ORAGANIC-INORGANIC HYBRID

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***$[CH_3CH_2CH_2]_4N(H_2AsO_4)(H_3AsO_4)_2$:** The salt Tetrapropylammonium dihydrogenmonoarsenate bis arsenic acid compound crystallizes in non-centrosymmetric space group *Ia* system with the following unit cell dimensions: a = 8.116(2)Å, b = 33.673(4)Å, c = 8.689(2)Å, β = 95.34(2)°. The structure was solved by the direct method and refined to final R value of 0.0655 and R_w = 0.1284. The structure consists of infinite parallel two-dimensional planes built of mutually $[H_2AsO_4^-]$, $[H_3AsO_4]$ tetrahedra and $[CH_3CH_2CH_2]_4N^+$ cations connected by strong O–H···O and C–H···O hydrogen bonding. Impedance spectroscopy study, reported for powder sample, reveals that the conduction in the material is due to the proton jumping between the oxygen atoms (figure 1).

***$[(CH_3CH_2CH_2)_4N]_2MnCl_4$:** An hybrid compound $[(CH_2CH_2CH_3)_4N]_2$ $MnCl_4$ was characterized by single-crystal X-ray diffraction (DRX). This compound crystallizes in the space groups C2/c with unit cell parameters: a = 32.6563(5), b=13.8790(2), c=15.0126(2)Å, β= 109.182(1), R= 0.0537 and R_w=0.2278. Manganese environment is bound by four chlorine atoms giving rise to isolated MnCl4 tetrahedra and chlorine atoms are disordered with an occupancy rate ½, ½, ¼. The cationic groups have an occupation rate ½ therefore these carbons have a disordered structure (figure 2).

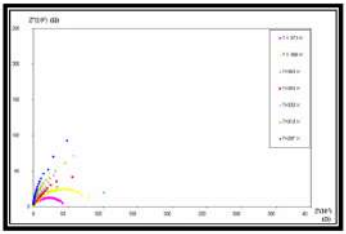

Figure 1

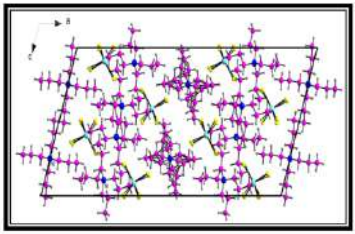

Figure 2

SYNTHESIS AND CRYSTAL STRUCTURE OF A NEW CHROMIUM COMPLEX $(C_7H_7N_2)[Cr(C_2O_4)_2(H_2O)_2].2H_2O$

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The search of bis(oxalato) chromium (III) compounds obtained through an impressive synthetic strategy based on supramolecular chemistry has attracted intense interest not only because of their appealing structural novelty, but also due to their fascinating properties and applications, such as catalysts, luminescence, NLO and magnetic [1].
Following these lines of investigation, we have focused our work on the synthesis and the characterization of a new compound of formula $(C_7H_7N_2)[Cr(C_2O_4)_2(H_2O)_2]$ $2(H_2O)$.
This compound crystallizes in the triclinic P-1 space group with a = 7.245(1)Å, b = 7.725(2)Å, c = 15.162(3)Å, α = 97.37(1)°, β = 98.17(2)°, γ = 96.04(1)° and Z= 2.
The resolution of the structure leads after several cycles of refinement followed by some Fourier-Differences to reliability factors R(F) = 2.89 % and $wR(F^2)$ = 8.27 %.
The structure is made up of one $[Cr(C_2O_4)_2(H_2O)_2]^-$ anion, $(C_7H_7N_2)^+$ cation and two crystallization water molecules, which are held together by hydrogen bonds and π-π stacking interactions.
The structure of the title compound consists of layers formed by $[Cr(C_2O_4)_2(H_2O)_2]^-$ and free water molecules intercalated by organic cations $(C_7H_7N_2)^+$ (Fig. 1).

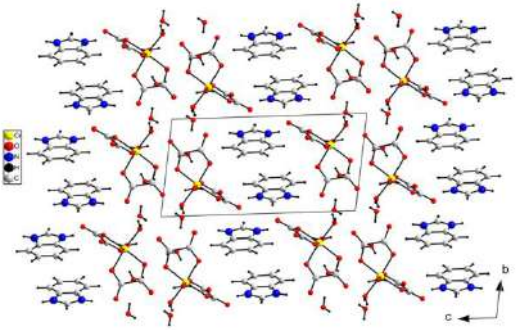

Figure 1: Projection of the $(C_7H_7N_2)[Cr(C_2O_4)_2(H_2O)_2].2H_2O$ structure along the *a* axis

The crystal packing is stabilized by N—H...O and O—H...O hydrogen bonds [2,3] between $(C_7H_7N_2)^+$ and $[Cr(C_2O_4)_2(H_2O)_2]^-$ involving the uncoordinated water molecule. These interactions link the layers together forming a three-dimensional network and reinforcing the cohesion of the ionic structure.

Keywords: Chromium(III) complexes, Crystal structure, X-Ray Powder diffraction.

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Synthesis and Crystal structure of new double molybdate $Na_9Cr(MoO_4)_6$

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Nonasodium chromium (III) hexakis [molybdate(VI)] was prepared by solid-state reactions. Single crystals of $Na_9Cr(MoO_4)_6$ were obtained and its structure was solved (the space group R-3c, a= 14.707(5) Å and c= 19.175(7) Å, V= 3592(2) $Å^3$, Z=6, R= 0.028). Morphology and elemental analysis, were made using a scanning electron microscope type FEI QUANTA 200. This analysis confirms the presence of the expected elements: Na, Cr, Mo and oxygen. The basic structure units are isolated polyhedral clusters composed of a central CrO_6 octahedron sharing vertices with six MoO_4 tetrahedra to form an open framework in which the Na^+ cations are bound to the free vertices of the MoO_4 tetrahedra (Fig. 1). The Cr^{3+} cation has site symmetry of 32 (6a), one Na atom sits on a twofold axis (18e), and all other atoms are at general positions. The bond-valance-sum model confirms the expected values of ion charges. The title compound is isotypic with $Na_9Sc(MoO_4)_6$ [1] and $Na_9Fe(MoO_4)_6$ [2]. It is compared and discussed with similar structures. As a perspective of this work, we will prepare its microcrystalline powders, in sufficient quantity in order to study their physical properties.

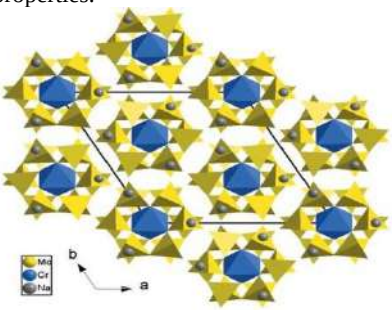

Fig. 1 : projection of the structure according to c

Key words: crystal structure, molybdate (VI), CrO_6 octahedron, quaternary systems, open framework.

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Thermal beavior, dielectric and vibrational studies of the new thallium rubidium sulfate selenate tellurate

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At room tempurature, the new compnoud $Tl_{1.90}Rb_{0.10}(SO_4)_{0.92}(SeO_4)_{0.08}Te(OH)_6$ crystallizes in the monoclinic system with space group $P2_1/c$ The structure can be regarded as being built of isolated TeO_6 octahedra, S/SeO_4 tetrahedra and Tl^+/Rb^+ cations. The main feature of this structure is the coexistence of three different anions (SO_4^{2-}, SeO_4^{2-} and TeO_6^{6-} groups) in the unit cell, connected by O–H…O hydrogen bonds.

Crystals of $Tl_{1.90}Rb_{0.10}(SO_4)_{0.92}(SeO_4)_{0.08}Te(OH)_6$ undergo three endothermic peaks at 393 K, 446 K and 474 K. These transitions detected by DSC and analyzed by dielectric measurements using the impedance and modulus spectroscopy techniques. Raman spectra of the material recorded at various temperatures (300–600 K) in the frequency range (100–1100 cm^{-1}) and IR spectra achieved at room temperature between 400 and 4000 cm^{-1}, confirm the presence of all the phase transitions detected in the material and show that anionic groups coexist independently in the same crystal.

The structure, Raman spectroscopy and Evidence of Ferromagnetic transition in $CuCr_{1-x}M_xO_2$ (M = Mn and Rh) compounds

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The $CuCr_{1-x}M_xO_2$ (M = Mn, Rh) polycrystalline powders were synthesized by the direct solid state reaction of Cu_2O and M_2O_3 (M = trivalent cation or mixer of trivalent cation for transition metal). The magnetic susceptibility was measured in the temperature range of 0 –300 K. It was found that the magnetic susceptibility (χ) increases rapidly with the doping of Cr^{3+} by Mn^{3+} and Rh^{3+} ions with existence of paramagnetic substance at low temperature. With the substitution of magnetic Mn^{3+} and Rh^{3+} for Cr^{3+} (S=3/2), the antiferromagnetic (AF) transition becomes broader and the transition temperature increases. However, at low temperature, the magnetic data for $CuCr_{1-x}M_xO_2$ (M = Mn, Rh) show evidence for weak ferromagnetic (FM) transition between 100K and 130K. Clear hysteresis loops indicate that FM order exists in both of them Mn or Rh-doped samples at 4K. All samples behave like semiconductors. The ferromagnetism properties can be attributed to the double exchange interaction between the Mn^{3+} or Rh^{3+} and Cr^{3+} semiconductors compounds.

Keywords: Delafossite, Powder diffraction, Magnetic susceptibility, Ferromagnetic transition, Raman spectroscopy

Evidence of Development a New Spin orders Benefiting to Enhanced Magnetic Properties in Co^{2+}-Doped delafossite-type oxide $CuCrO_2$

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This paper reports the effect of high-spin Co^{2+}-doped $CuCrO_2$ delafossite-type oxide on the structure and physical properties. X-ray diffraction and Raman spectroscopy show that the structure is maintained for all Co-doped simples for chromium. The incorporation of this element generates anisotropic microstrains in the structure. The temperature dependence of zerofield-cooling magnetization was measured. All samples exhibit an AFM transition around 24K. The high-spin state and the shift due to the exchange splitting of the conduction band suggest strong hybridization between carriers in the Cr 3d t_{2g} band and the t_{2g} states of the high-spin Co^{2+} to developed other spin orders benefiting to enhanced magnetic susceptibility and grow to put the evidence of new FM transition. The coupling between the magnetic order and ferroelectric order is also characterized.

Keywords: Delafossite oxides, DRX, magnetic properties, Raman spectroscopy

ELECTROCHEMICAL BEHAVIOR OF ACID ORANGE 7 BY CYCLIC VOLTAMMETRY IN TWO DIFFERENT SOLVENTS

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In the present work the electrochemical oxidation of acid orange 7 (AO7) on a glassy carbon (GC) electrode has been investigated by cyclic voltammetry in two different solvents: aqueous solvent (H_2O) and dimethyl sulfoxide (DMSO) using sulfuric acid as a supporting electrolyte in function of the experimental parameters controllable namely the initial concentration of AO7, the scanning speed, the temperature and the solvent nature. The results show that the oxidation peak current of the AO7 increased linearly with the concentration in the two solvents. In addition, the anodic peak current logarithm (log ipa) varies linearly with the logarithm of the potential scan rate with a leading coefficient equals to 0.5, based on these results, we can conclude that the oxidation process reaction kinetics of AO7 at vitreous carbon electrode was controlled by diffusion except. Finally, the analysis of AO7 oxidation voltammograms in two different solvents showed that the more donor number is important, the oxidation of AO7 becomes easy.

Keywords: Azo dye; Eectrochemical oxidation; Cyclic voltammetry; Glassy carbon electrode; Solvent effect; Donor number.

Cr-substitution effect on structural, optical and electrical properties of $Cr_xCe_{1-x}PO_4$ (x = 0.00, 0.08, 0.10 and 0.20) nanorods.

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$Cr_xCe_{1-x}PO_4$ (x = 0.00–0.20) nanorods were synthesized using the hydrothermal method. The as-prepared samples were characterized by X-ray diffraction (XRD), infrared absorption spectroscopy (IR) and transmission electron microscopy (TEM). The XRD results revealed the formation of a pure $CePO_4$ hexagonal phase. TEM images confirmed the nano-size character of the as-prepared samples. Impedance spectroscopy analysis was used to analyze the electrical behavior of samples as a function of frequency at different temperatures. The increase of Cr-amount led to an increase in the total conductivities and decreased the activation energies (E_a (x = 0.00) = 1.08 eV to E_a (x = 0.20) = 0.80 eV). The optical properties of $Cr_xCe_{1-x}PO_4$ nanomaterials were investigated using UV–vis spectroscopy. The band-gap energy values decreased with increasing Cr-content showing a red-shift trend. The improvement of the electrical conductivity and optical properties makes the $Cr_xCe_{1-x}PO_4$ nanomaterials possible candidates to be used as electrolytes in solid oxide fuel cells, in photocatalytic and photovoltaic applications.

Key words: Nanostructures, Chemical synthesis, Impedance spectroscopy, X-ray diffraction, Transmission electron microscopy (TEM).

DIELECTRIC AND ELECTRIC PROPERTIES OF ACIDIC CYCLHEXAPHOSPHATE $[o\text{-}(OCH_3)C_6H_4NH_3]_4H_2P_6O_{18}.2H_2O$

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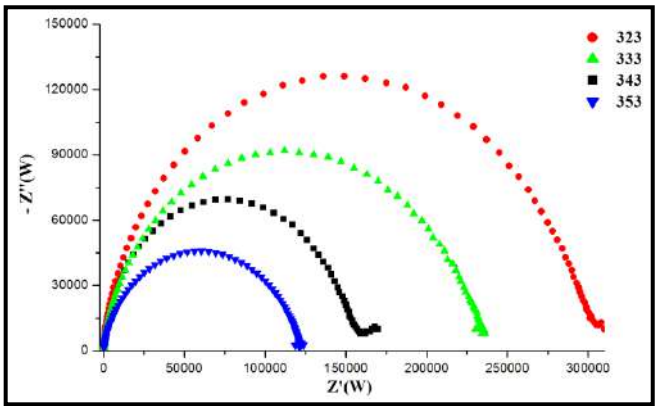
Several studies are devoted to research of organic-inorganic materials with open structures with interesting physicochemical properties and therefore be used in various applications. Such as structures are suitable in ionic conduction. In the present communication, we report crystal growth and investigation of an hybrid cyclohexaphosphate **$[o\text{-}(OCH_3)C_6H_4NH_3]_4H_2P_6O_{18}.2H_2O$.** This later exhibit an open structure containing inorganic layers between which the organic entities are located. The complex impedance of this compound has been studied**.** Analysis of impedance spectra (Z'' versus Z') can be fitted based on a, parallel, equivalent electric circuit consisting of a resistor (R) and capacitance (C) elements. The AC conductivity of this compound is performed, modulus results are given showing that conductivity is thermally activated and the spectra follow the Arrhenius law. Dielectric constant drastically decreases at low frequencies, which is governed by different components of polarizability, while at high frequencies, it reaches a constant value.



Complex impedance diagrams on $[o\text{-}(OCH_3)C_6H_4NH_3]_4H_2P_6O_{18}.2H_2O$

Keywords: Cyclohexaphosphate; X-ray diffraction; Complex Impedance; AC Conductivity; Dielectric Modulus.

Structural, thermal analysis of rubidium Potassium phosphate tellurate compound $Rb_{1,6}K_{0,4}HPO_4Te(OH)_6$

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The new crystal structure of $Rb_{1,6}K_{0,4}HPO_4Te(OH)_6$ have been synthesized. Structural and thermal analysis has been investigated. This compound crystallizes in the monoclinic system with space group P 2. Its unit cell dimensions are: a = 7.950 (7) Å, b = 6.308(6) Å, c = 9.5008(9) Å, β = 109.783 (9), Z = 4, V = 448.37 (7) $Å^3$ and its crystal structure was determined and refined down to R_1= 0.057 and W R_2 = 0.058.

The main feature of this atomic arrangement is the coexistence of two independent and different anions (PO_4^{3-} and TeO_6^{6-}) in the unit cell, connected by O−H…O hydrogen bonds which make the building of the crystal. The cohesion of the structure is assured on the one hand by the interaction between Rb^+/ K^+ and the oxygen atoms and on the other hand, by the presence of hydrogen bonds.

The IR spectra achieved, at room temperature, between 400 and 4000 cm^{-1}, confirm the presence of PO_4^{3-} and TeO_6^{6-} groups and elucidate the hydrogen bonds in their crystal lattice.

The thermal analyses (DTA) and (TG) of the title compound prove that the decomposition of this material starts at T= 485 K and the differential scanning calorimetry (DSC) analysis show three phase transitions detected respectively at T_1 = 451 K, T_2 = 463 K and T_3 = 481 K.

SOLID STATE CARACTERISATION OF STATIN: CONTRIBUTION OF FLORINE NUCLEAR MAGNETIC RESONANCE

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The pharmaceutical industry in Tunisia has experienced a considerable growth since 1989, giving the industry full of promise rise even if it is restricted to import raw materials. Strengthening the credibility of local products requires complementary analytical tools for their control and development.
In this work we are interested in the comparative study of two rosuvastatin raw materials and two atorvastatin tablets using thermal and spectrometric analysis.
Regarding rosuvastatin, DSC and 19 F ss NMR showed for each product, an appropriate profile which explained by two diastereoisomeric mixtures of different compositions.
For atorvastatin, only 19F ss NMR, was able to detect a structural deformation within one of the two products, which is probably due to a partial amorphization.

Synthesis and characterization of $[C_8H_{14}N_2](NO_3)_2$

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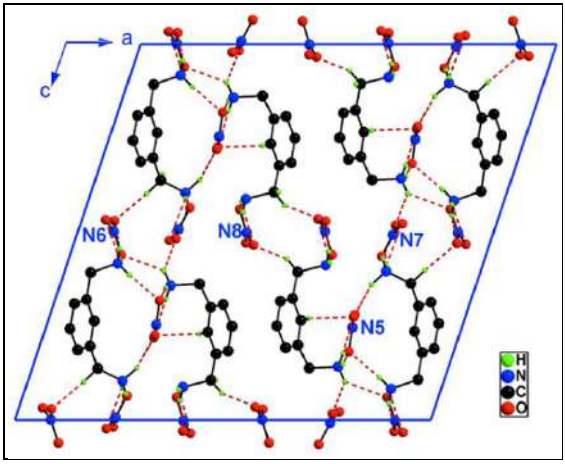
As a part of our study of crystal packing containing the nitrate anion, we report here the preparation and the structural investigation of a new organic nitrate, $[C_8H_{14}N_2](NO_3)_2$. This compound is a monoclinic $P2_1/c$ unit cell with the following parameters: a = 21.430(7), b = 5.725(2), c = 20.447(5) Å, β = 108.502(1)°, V = 2379.28(13) Å^3, and Z = 8. The asymmetric unit of the title salt, $[C_8H_{14}N_2](NO_3)_2$, contains two independent dications and four independent nitrate anions. The crystal structure consists of discrete nitrate ions, three of which stack in layers parallel to (001) at z = 0 and 1/2. These layers are connected via m-xylylendiaminium dications. The fourth anion is sandwiched by the two independent organic cation in the asymmetric unit. In the crystal, the ions are connected by a large number of bifurcated and non-bifurcated N-H…O(O) hydrogen bonds, forming sheets parallel to (100). These sheets are connected by C-H…O hydrogen bonds, forming a three-dimensional network. This compound is also characterized by infrared spectroscopy and thermal analysis.



Projection of $[C_8H_{14}N_2](NO_3)_2$ along the b axis.
The H-atoms not involved in H-bonding are omitted.

Novel alkali Borosilicate glasses: Thermal behavior for use as bone biomaterial.

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Bioactive glasses are indicated for use as bone substitutes in orthopaedic or dental surgery, because they have a high reactivity when in contact with the biological medium. Boron is known as a glass network former and an activator of the bioactivity of the glasses. In this work bioactive glass was doped by, 5; 10 and 20 wt % of B_2O_3 according to the melting process. The aim of our study was to investigate the effect of boron on the thermal characteristics of our bioactive glass for the use as bone biomaterial. Obtained results show that increasing the boron content in the glass network decreases the melting temperature and increases the thermal stability.

Key words: Glasses, Melting, Thermal proprieties, Biomedical applications.

Preparation and characterization of activated carbon from peach stones by physical activation

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

Key words: Peach stones, Activated carbon, Physical activation, Adsorption

SYNTHESIS, STRUCTURE AND CHARACTERIZATION OF A NEW ORGANIC-INORGANIC COMPOUND: $(C_5H_6N_5)_2[Cr_2O_7]$

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When dealing with organic-inorganic hybrid materials, two main underlying concepts are "combination" and "synergy", since the basic idea behind the development of this rapidly expanding class of materials is to combine organic and inorganic building blocks to afford a material endowed with the properties of both components and eventually to overcome the structural limits of conventional materials. That's why a new inorganic-organic hybrid $(C_5H_6N_5)_2[Cr_2O_7]$, has been synthesized under slow evaporation from aqueous solution and characterized by IR spectroscopy, UV spectroscopy and single crystal X-ray structural analysis.

The title compound crystallizes as a triclinic system, in P-1 space group; with

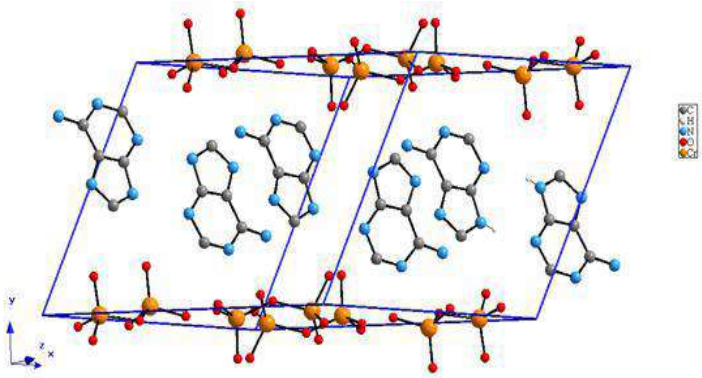
a = 11,685(1) Å α = 83,96(1)°

b = 11,753(1) Å β = 70,48(1)°

c = 14,560(1) Å γ = 61,86(1)° 1658,7 $Å^3$ and Z= 4

Single-crystal X-ray analysis shows that compound (1) possesses a 3D framework, which is built up by isolated $Cr_2O_7^{2-}$ anions and $(C_5H_6N_5)^+$ cations, the extensive net hydrogen bonds between cations and anions contribute to the crystal packing.

Keywords: Organic–inorganic hybrid, Infrared spectroscopy, High-dimensional structure



SYNTHESIS, CRYSTAL STRUCTURE, PHYSICO-CHEMICAL PROPERTIES AND THEORETICAL CALCULATIONS OF A CADMIUM(II) COORDINATION POLYMER ASSEMBLED BY TRIETHANOLAMMONIUM

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Chemical preparation, structural, thermal behavior, IR, Raman and UV studies are reported for a monoclinic new organic-inorganic chlorocadmate, $[NH(C_2H_4OH)_3]CdCl_3$, having the following parameters: a=9.5124(6), b=16.5639(8), c=7.7181(4) Å, β=101.737(7)°, Z=4, P21/c. The atomic arrangement is organized as layers in the (100) plane. Connection in these layers is assured by three types of hydrogen bonds, O–H···O, O–H···Cl and N–H···O. The stability between successive layers is performed by van der Walls interactions C–H···O originating from the organic cations. Thermal behavior exhibits one phase transitions. IR and Raman bands are attributed by comparison with spectra of similar other compounds. The molecular HOMO-LUMO compositions and their respective energy gaps were also drawn to explain the activity of our compound. The role of the intermolecular interaction in this crystal is analyzed. The optical study was also investigated by UV–vis absorption spectrum. Cyclic voltammetry was studied to evaluate the spectral and structural changes accompanying electron transfer.

Keywords: Chlorocadmate (II). X-ray diffraction. Thermal analysis (TG/DTA). Infrared, Raman and UV-Vis spectroscopy. DFT calculations. Cyclic voltammetry.

NOVEL TWO-DIMENSIONAL CHLOROCADMATE TEMPLATED BY ETHANOLAMMONIUM: SYNTHESIS, CRYSTALLOGRAPHICAL, VIBRATIONAL, THERMAL AND DFT STUDIES

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Chemical preparation, X-ray single crystal diffraction, thermal analysis, electrochemical measurements, IR, Raman and UV spectroscopic investigations of a novel organic-inorganic hybrid material $(C_2H_8NO)_2Cd_{2.5}Cl_7.H_2O$ are described. The compound crystallizes in the triclinic system with P-1 space group. Its unit cell dimensions are a = 9.5112(5)Å, b = 10.1603(7)Å, c = 11.0191(8) Å, α = 108.623(9)°, β = 110.278(9)°and γ = 100.615(8) with V = 892.9(1) $Å^3$ and Z = 2. The structure provides a new interesting example of infinite inorganic layers of $[Cd_{2.5}Cl_7O]_n^{2n-}$ anions interconnected by N–H···Cl and O–H···Cl hydrogen bonds. Acidic protons of the chloride group were transferred to the organic molecule, giving the singly-protonated cations. The ability of ions to form a spontaneous three-dimensional structure through O–H···Cl and N–H···Cl hydrogen bond is fully utilized. These hydrogen bonds induce notable vibrational effects. IR and Raman spectra are reported and discussed on the basis of group theoretical analysis The molecular HOMO-LUMO compositions and their respective energy gaps were also drawn to explain the activity of our compound. The role of the intermolecular interaction in this crystal is analyzed. The optical study was also investigated by UV–vis absorption spectrum. Thermal analysis reveals the hydrous character of the compound. Cyclic voltammetry was studied to evaluate the spectral and structural changes accompanying electron transfer.

Keywords: Chlorocadmate (II). X-ray diffraction. Thermal analysis (TG/DTA). Infrared, Raman and UV-Vis spectroscopy. DFT calculations. Cyclic voltammetry.

Synthesis and spectroscopic studies of the Li $Gd_{(1-x)}Eu_x(PO_3)_4$ compounds (x=0, 0.01, 0.02, 0.03 and 0.04)

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The polycrystalline powder of the title compounds were synthesized via traditional solid-state reaction. The crystal structures of the powders were obtained from the powder diffraction data and were refined using the Rietveld method. It was shown that the main diffraction picks of the studied solids are present. Moreover, Infrared and Raman spectra were recorded at room temperature. The observed vibrational results show the presence of the P-O-P bridges and the O-P-O groups in the structure of the obtained materials.

The excitation and the emission spectra of the lanthanide phosphate composites doped with different concentration of Eu^{3+} have been studied.

Besides, the condensed phosphates of rare earths with general formula $MGd_{(1-x)}Eu_x(PO_3)_4$ were the object of several researches and they have a considerable interest mainly for their optical properties, especially in laser technology. So, we have systenized some other compounds, Eu^{3+} doped $MGd(PO_3)_4$, where M is a monovalent cation: Li, Na, K. The corresponding spectroscopic characterizations are under study.

CRYSTAL STRUCTURE AND PHYSICOCHEMICAL PROPERTIES OF A NEW β-OCTAMOLYBDATE: $[Ba_2(H_2O)_8][Ba_{0.5}(OH)][MO_8O_{26}]\cdot 3H_2O$

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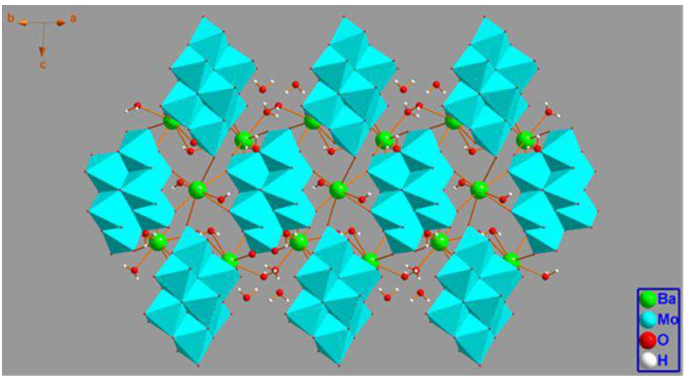
A novel β-octamolybdate compound $[Ba_2(H_2O)_8][Ba_{0.5}(OH)][Mo_8O_{26}]\cdot 3H_2O$ (1) has been synthesized and characterized by X-ray single crystal analysis, scanning electron microscopy (SEM), IR, TG, Cyclic Voltammetry analyzes and UV–Vis. The title compound crystallizes in monoclinic system, space group C2/m, a = 16.8575(10) Å, b = 12.8418(9) Å, c = 15.2298(10) Å, β = 97.225(5)°, V = 3270.79(7) $Å^3$, Z = 4, λ(MoKα) = 0.71073 Å. The refinement of the structure leads to a residual factor R = 0.0448 for 2638 reflections [I>3σ(I)].The structure of (1) consists of β-$[Mo_8O_{26}]^{4-}$,OH^- anions, Ba^{2+}cations and H_2O molecules. The β-$[Mo_8O_{26}]^{4-}$ subunits are connected together through Ba−O−Mo bridges.



View along the [110] direction of compound (1)

Key words: Synthesis, octamolybdate, barium, crystal structure, characterization.

Thermal analysis, dielectric properties and conduction mechanism of $Cu_{1.09}Ni_{0.91}(HSeO_3)_2Cl_2.4H_2O$ compound

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The $Cu_{1.09}Ni_{0.91}(HSeO_3)_2Cl_2.4H_2O$ (noted CuNiSe) compound was prepared from a aqueous solutions reaction. This compound crystallizes in the orthorhombic Pnma space group. It was analyzed by single-crystal x-ray diffraction and we have used the X-ray powder diffraction to confirm the purity of the synthesized compound. The structure of this compound consists of a three–dimensional fram work structure made of layer parallel to the (010) plan formed by copper nickel octahedral $HSeO_3^-$ trigonal pyramidal [1]. The thermal analysis of the CuNiSe compound shows two distinct endothermic peaks at 433 k and 463 k (phase transition). The Raman spectra of CuNiSe have been recorded from 299 to 523 K. The evolution of the dielectric constant as a function of frequency and temperature revealed one anomaly at about 433K attributed to a ferro-paraelectric phase transition. However, the evolution of the conductivity versus temperature showed the presence of an ionic-protonic phase transition at 463K [2].

Keywords: Thermal analysis, Dielectric properties

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SYNTHESIS AND CHARACTERIZATION OF TITANIUM OXIDE NANOTUBES

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Titanium oxide nanotubes are especially interesting because of their high dimension ratio and high specific surface area. In last few years, properties of TiO2 nanostructures were extensively studied due to their potential applications in solar cells, medicine, electrochemistry, gas sensors and photocatalysis.

A number of methods have been developed for preparing TiO_2 derived nanotubes, for instance, the chemical "template" method [1], the electrochemical "anodic oxidation" method [2] and the alkaline hydrothermal treatment [3]

For large scale production of nanostructured TiO_2 materials, the hydrothermal method, proposed by Kasuga et al. [4] is the most suitable. It involves treating the precursor of TiO_2 (anatase and/or rutile) in an autoclave by a concentrated alkaline solution NaOH at high temperature and times going from one to several days. Synthesis is followed by a washing step with water and acid solutions. This method is simple and not expensive with respect to other described methods. Several factors can influence the formation of titanates nanotubes such as: the structure of the starting material (anatase, rutile or amorphous), the nature and concentration of the alkaline solution, the hydrothermal temperature and duration and the post-treatment processes (acid washing).

In this paper, we present a hydrothermal synthesis of titanates nanotubes. The products were characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD) and Raman spectroscopy.

Keywords: Titanium oxide nanotubes, Hydrothermal treatment, Raman spectroscopy

Synthesis and characterization of the phosphates $NaSrCr_2(PO_4)_3$ and $NaPbCr_2(PO_4)_3$

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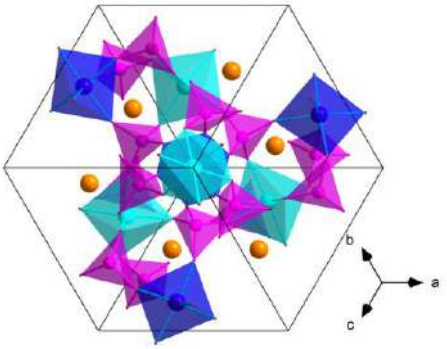
This work describes the synthesis and characterization of two new phosphates $NaSrCr_2(PO_4)_3$ (I) and $NaPbCr_2(PO_4)_3$ (II). These compounds were synthesized by the solid state reaction technique. Their structural analysis using powder X-ray diffraction showed that they crystallize in the cubic system with space group $P2_13$ and the unit cell parameter a = 9.648(1) Å for (I) and 9.654(1) Å for (II) and four formula units per cell. Corresponding structural data showed their belonging to the large group of $A_xM_2(XO_4)_3$ compounds isostructural to the mineral langbeinite $K_2Mg_2(SO_4)_3$. The framework consists of a 3D assemblage of CrO_6 octahedra and PO_4 tetrahedra linked to each other via common corners, in such a way that each octahedron connects six neighboring tetrahedra and reciprocally each tetrahedron links four adjacent octahedra. An alternative description of this framework can be made on the basis of $[Cr_2P_3O_{18}]$ units (labeled U units), composed by two octahedra, bridging three tetrahedra. Eight of these units share the corners of their polyhedra leading to the formation of two kinds of large cavities M(1) and M(2), statistically occupied by the Na^+ and M^{2+} (M = Sr or Pb) cations. The cavities and the units alternate along the ternary axis generating finite chains with a stacking sequence of -U-U-M(1)-M(2)-.

The IR spectra were interpreted on the basis of internal vibrations of PO_4 tertrahedra. EPR and specific heat results showed that there is no structural transition at low temperature.



A projection of the structure along the [111] direction

SYNTHESIS, STRUCTURE AND CHARACTERIZATION OF AN INORGANIC-ORGANIC HYBRID DAWSON POLYOXOMOLYBDATE $(C_4H_{10}N)_6(P_2Mo_{18}O_{62}).4H_2O$

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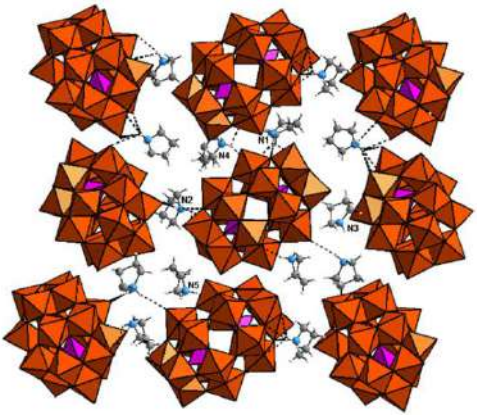
A new inorganic-organic hybrid materials based on heteropolyoxometalates, $(C_4H_{10}N)_6[P_2Mo_{18}O_{62}].4H_2O$ (1) where $C_4H_{10}N$ is protonated pyrrolidine has been synthesized and structurally characterized by physic-chemical methods. Single-crystal X-ray diffraction method, infrared, ultraviolet spectroscopy, Thermogravimetric analysis and cyclic voltammetry measurements
of the title hybrid materials indicate that there are hydrogen bond interaction between O atoms of the heteropolyoxometalates and water molecules as well as the N and O atoms of the organic compound. The molecular structures of synthesized hybrid materials contain discrete entities of pyrrolidinumion and water molecules surround every $[X_2Mo_{18}O_{62}]^{6-}$ anion over the extended crystalline network that the $[X_2Mo_{18}O_{62}]^{6-}$ anion retains its "Dawson structure". Crystal data: monoclinic, space group P21/a, a = 13,453(1) Å, b = 24,046 (1)Å, c = 24,119(1) β = 97, 99(1)◦, V = 7726,30(5)$Å^3$ and Z = 4.



View of three-dimensional network in the compound

Keywords: Polyoxometalate; Hybrid materials; Crystal structure; Infrared spectroscopy; Thermogravimetric analysis.

Ultrasound assisted synthesis of nanocrystalline zinc oxide

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ZnO nanoparticles have been produced via polyol process by forced hydrolysis of zinc acetate in diethylene glycol (DEG). This reaction was assisted with ultrasonic (US) irradiation in the absence of mechanical stirring. Under optimal conditions of 600 watt total ultrasound power and 20 kHz frequency, spherical shape nanoparticles of 5 to 10 nm in diameter was found after only 10 min of ultasonic irradiation in a alterned cycle on/off of 50 seconds duration each. As compared to thermally activated conventional polyol process [1], (US) permits to considerably reduce reaction time as well as size particles. Indded, in the presence of ultrasound, the implosive collapse of to bubbles generates hotspots with transient temperature of ~5000 K and pressures of ~1800 atm [2]. For these conditions, a new growth mechanism is proposed in this paper that is based on equilibrium calculations of the polyol system in temperature runge from ambient to 5000K with 51 molecular and atomic species as a combination of H, C, O and Zn atoms (figure 1).

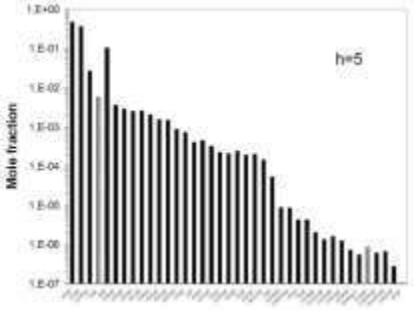

Figure 1: Equilibrium calculation of bubble gas composition at T=5000 K : P=1800 atm ; h=5 ; z=0.5

Key words: DEG, polyol process, ultrasound, nanoparticles.

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Elaboration and characterization thin films of $AgSbS_2$ by CBD: Application of the experimental design methodology

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$AgXS_2$ sulphosalts (in which X is As, Bi or Sb) are a group of semi-conducting sulphides that occur naturally as minor components of polymetallic sulphide ores [1]. The ternary Ag–Sb–S semiconductor system has two phases: Ag_3SbS_3 (Eg = 1.8 eV) and $AgSbS_2$ (Eg = 1.7 eV) [2]. This last one managed to produce a good film with a high absorption coefficient of $\alpha \sim 10^5$ cm^{-1} that matches that of $CuInSe_2$, making it a potential solar absorber [3]. This ternary can be one of the most promising candidates for the next generation of solar cells.

In this work we have successfully synthesized $AgSbS_2$ thin films using the chemical bath deposition (CBD). The optimal operating parameters depositions of this layer have been investigated using Doehlert matrix.

X-ray diffraction pattern indicates that film exist in polycrystalline with cubic phase. Optical study revealed that this ternary has an optical band gap of 1.71eV.

Key words: Thin films, semiconductor, Doehlert Matrix, $AgSbS_2$.

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Magnetic and structural characterization of the mechanically alloyed Fe-Al-B powders

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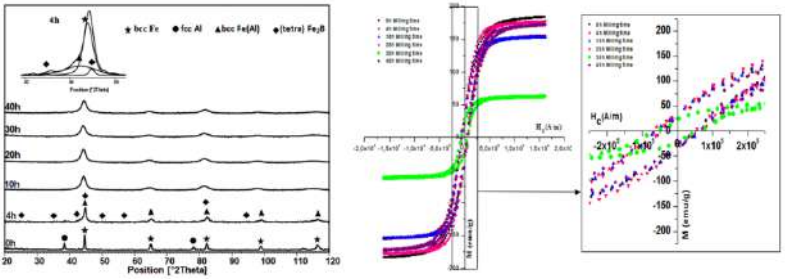
Nanostructured iron-aluminium alloy of Fe-25at%Al composition doped with 0.2 at% B was prepared by mechanical alloying. Morphological, microstructural, structural and magnetic changes during the milling process were followed by scanning electron microscopy, X-ray diffraction and magnetic measurements. The transformation of the phase depends upon the milling time. At the early stage of the MA process, inhomogeneous Al distribution in the Fe(Al) solid solution is found and three phases, namely, Fe, Fe(Al) and Fe_2B are needed in the XRD profile analysis. With increasing milling time to 6 h all Al atoms become dissolved in the Fe(Al) lattice. The two phase of BCC Fe(Al) and Fe_2B was formed after milling time exceeds 6 h and the resulting powder has a nanometer scale structure. From the magnetic point of view there is a correspondence between microstructural evolution and magnetic behavior in the form of that decreasing the crystallite size increases the magnetism by increasing coercivity and squareness ratio. Consequently as low crystallites size is, stronger hard ferromagnetic material results. Due to thermal vibrations higher temperature produces a decrease in magnetic properties.



X-ray diffraction patterns and Hysteresis loops for Fe-Al-B powders a function of the milling time.

Key words: Nanocrystalline materials, Mechanical alloying, X-ray diffraction, Magnetization, Microstructure.

PREPARATION, CHARACTERIZATION AND BIOLOGICAL EVALUATION OF DECAVANADATE POLYANION DECORATED WITH ORGANIC CATION

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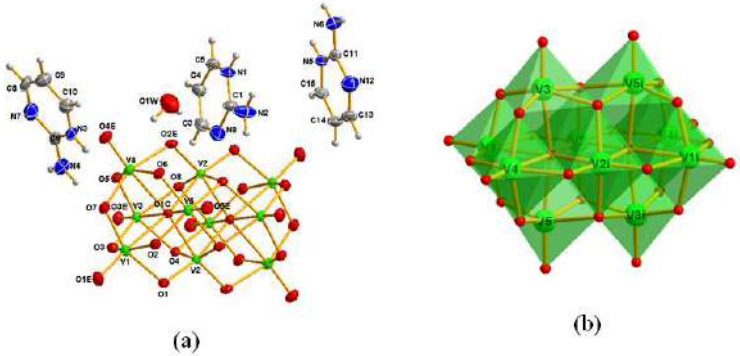
A novel decavanadate cluster with organic cation $(C_4H_6N_3)_6V_{10}O_{28}.2H_2O$ (I) has been synthesized and characterized by X-Ray diffraction, elemental analysis, IR, UV-Vis and fluorescence. The structure determination reveals that I crystallizes in the monoclinic space group $P2_1/c$ with a = 9.8177(10) Å, b = 18.1521(17) Å, c = 14.2793(14) Å and β = 97.217(6)°. Its crystal structure consists of polymeric inorganic layers $[V_{10}O_{28}(H_2O)_4]^{6n-}$ developed perpendicular to a-axis interconnected by means organic cations through H-bonds, Van der Waals and electrostatic interactions giving rise to 3D supramolecular assembly. In addition, optical and biological properties of I are evaluated.



(a) View of the title compound with displacement ellipsoids drawn at the 30 % probability level. H atoms are represented as small spheres of arbitrary radii. Symmetry code: (i) 1 – x, 1 – y, 1 – z, (b) Polyhedral view of the decavanadate cluster showing the VO_6 octahedra.

OCTAHEDRON-LIKE Cu_2O: HYDROTHERMAL SYNTHESIS, OPTICAL AND ELECTROCHEMICAL PROPERTIES

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The morphology of inorganic microcrystal has significant influence on their physical and chemical proprieties. Indeed, the control the crystal shape is becoming one of the most important challenges in the field of advanced materials. Some works have been devoted to synthesize materials with distinct structures such as urchins, hollow spheres, and wire, all of which have electrical, optical, and electrochemical singular properties. Among these, the Cu_2O material is inexpensive and non toxic with a narrow band gap of 2.17eV at room temperature. It has aroused great interest owing to its distinguished and promising applications for chemical sensors [1], oxide solar cells [2], lithium-ion batteries [3], and catalytic converter [4].

In this communication, we will present the synthesis of Cu_2O octahedron via the hydrothermal route from the copper nitrate trihydrate as a copper source and hydroquinone which, acts as a reducing and structure-directing agent. The effect of the amount of hydroquinone on the particle size, morphology and the crystallinity was investigated. The electrochemical properties of the Cu_2O octahedron material have been investigated using cyclic voltammetry (CV) in the presence of li^+ in propylene carbonate electrolytic solution. Although many methods have been used to elaborate microstructured copper, to our knowledge, this is the first study that of Cu_2O octahedron is prepared using $Cu(NO_3)_2.3H_2O$ and hydroquinone which, acts as a reducing and structure-directing agent.

Key words: Hydrothermal synthesis, Cu_2O microcrystal, electrochemical properties, Optical properties.

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Chitosan-TiO_2 –ZnS photocatalysts: Characterization and photocatalytic activity

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Photocatalytic degradation of water pollutants using visible light on semiconductor photocatalysts has attracted intense research interests in recent years. Many researchers have proven that degradation efficiency of pollutants is principally determined by the properties of the photocatalysts, underscoring the importance of selecting suitable photocatalysts. Visible light response of photocatalysts must satisfy the functional requirements of having the capacity of absorbing visible light and the ability to separate photoexcited electrons from the holes. Among several semiconductor photocatalysts, titanium dioxide has attracted worldwide interest due to its strong oxidation activity and stability. Because of the band gap (3.2 eV) corresponding to UV light irradiation, modifying TiO_2 photocatalysts to enhance light absorption and photocatalytic activity under visible light irradiation has already been reported in the literature. Chitosan (CS) is one of the most extensively used biopolymers thanks to its nontoxic and excellent film forming ability with good mechanical strength, higher permeability and cost effectiveness. Chitosan is a polysaccharide derived from chitin, a linear chain of acetyl-glucosamine groups, generally extracted from crab shell or squid pen. It is obtained by removing most of the acetyl groups from chitin. This process gives rise to amine groups, and chitosan can be considered as a natural solid base. Moreover, Chitosan is an excellent adsorbent in wastewater treatment for the adsorption of various pollutants, including organic com-pounds and dyes. This is due to its high content of amino and hydroxyl groups, which can improve the photocatalytic efficiency of the hybrid samples as the adsorption is the first step of the photodegradation process. The as-prepared hybrid photocatalyst (CS-TiO_2-ZnS) was characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) and UV–vis diffuse reflectance spectroscopy (DRS). The results of the photodegradation of acid carboxylic, used as a model pollutant, revealed that the hybrid photocatalyst (CS-TiO_2-ZnS) exhibited an interesting photocatalytic performance under visible-light irradiation which can be attributed to the cooperative role of the components of the photocatalyst; chitosan as a template for the immobilization crystalline TiO_2 nanoparticles, that promote the light absorption in the visible range and TiO_2 acting as the origin of electrons-hole generated by the photons absorption to produce superoxide radicals.

Key words: Sol–gel processes, Raman and IR spectroscopy; Chitosan-TiO_2

ALUMINUM SCRAP AS LOW COST MATERIAL FOR THE SYNTHESIS OF ALUMINUM HYDROXIDE AND OXYHYDROXIDE

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Boehmite, aluminum oxyhydroxide (γ-AlOOH), is an important precursor powder for the production of γ-Al_2O_3, a low-temperature metastable polymorph of α-Al_2O_3. The utility of γ-Al_2O_3 can be outlined to a favorable combination of textural properties (i.e., surface area, pore volume, and pore size) and acid-base characteristics. These properties make γ-Al_2O_3 a potential material in many industrial applications as catalyst or catalyst support, membranes and adsorbents. The γ-Al_2O_3 phase is formed upon the dehydration of the γ-AlOOH at temperatures ranging from 400 to 700 °C. This preparation route is favored to the thermal decomposition of gibbsite (γ-$Al(OH)_3$) since γ-AlOOH has low dehydration enthalpy (72 kJ/mol Al_2O_3) compared to the corresponding one of gibbsite γ-$Al(OH)_3$ (187 kJ/mol Al_2O_3). Many methods for the synthesis of γ-AlOOH are reported in the literature, mainly involving expensive raw materials and harsh synthetic conditions that include high temperatures and pressures. Thus, the aim of this study was to develop a facile and environmentally friendly method to prepare γ-AlOOH, precursor for the production γ-Al_2O_3. For this purpose, we used aluminum scrap (99.8% purity) collected from aluminum workshop as aluminum source. The alkaline attack of the scrap with 2 M NaOH solution provides the sodium aluminate ($NaAlO_2$). This solution was neutralize with different acids (HNO_3, H_2SO_4 and CH_3COOH) at room temperature (RT) to different pH values (6, 7, 8, 9 and 10), resulting in the precipitation of a white gel. After a ripening time of 48 h in the mother liquor at RT, the gels were washed, dried over night at 80°C and then ground in porcelain mortar. The samples were then calcined for 3h in a muffle furnace at 500°C.

This study showed that the acid nature and the neutralization pH are important parameters that control the nature of the precipitated phase. Using HNO_3 acid and for pH=6, the XRD pattern of the white gel showed the characteristic peaks of well crystallized bayerite ($Al(OH)_3$) and pseudo-boehmite, poorly crystalline boehmite which has a very small particle size. The increase of the neutralization pH to 7, 8 and 9 and whatever the acid nature, we precipitated only pseudo-boehmite. At pH=10, we obtained a mixture of bayerite ($Al(OH)_3$) and pseudo-boehmite when HNO_3 and H_2SO_4 were used. Whatever the neutralization pH, and the nature of acids, the calcination of the white gel at 500 °C for 3h transformed it to γ-Al_2O_3. The ^{27}Al NMR spectra of non-calcined gel, prepared at pH=6 and using HNO_3, exhibit a single peak at 6 ppm attributed to Al nuclei in octahedral coordination. The calcined-gel reveals a second signal around 68 ppm corresponding to Al nuclei in tetrahedral coordination, characteristic of γ-Al_2O_3.

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

SYNTHESIS, CRYSTAL STRUCTURE AND CHARACTERIZATION OF A NEW COORDINATION COMPOUND: TETRA(2-AMINO-6-METHYLPYRIMIDIN-4(*1H*)-ONE) CUPRATE(II) SULFATE TETRAHYDRATE

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The crystal structure of the new complex tetra (2-amino-6-methylpyrimidin-4(1H)-one) cuprate(II) sulfate tetrahydrate has been determined by single crystal X-ray diffraction and refined by full-matrix least-squares methods to R = 0.050. The compound crystallizes in the orthorhombic space group Pccn with lattice parameters: a = 12.4064 (3), b = 14.4307 (4), c = 17.5011 (4) Å. Each Cu(II) ion is tetra-coordinated by four N atoms of four chelating 2-amino-6-methylpyrimidin-4-one molecules, forming a square-planar geometry. Intermolecular O-H...O, N-H...O and C-H...O hydrogen bonds link the molecules into a three-dimensional network. FT–IR and TGA were also used to characterize the complex.

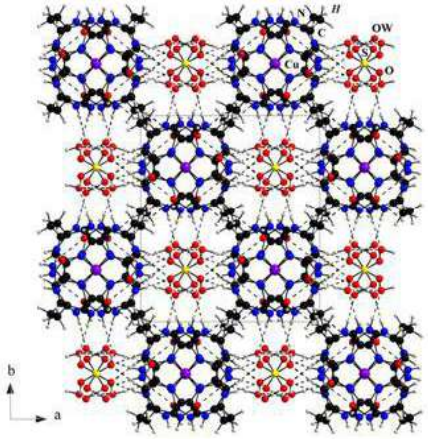

Figure: The packing diagram of the compound viewed down the $\vec{c}$ axis. Hydrogen bonds are shown as dashed lines.

Key words: X-ray diffraction; Crystal structure; Hydrogen bonds; IR spectroscopy.

SYNTHESIS, CHARACTERIZATION AND IONIC CONDUCTIVITY OF NEW NASICON-TYPE PHOSPHATES

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This work is part of systematic studies of the physicochemical properties of NASICON type phosphates, in an effort to highlight their applications in Li-batteries having high energy density.

The NASICON compounds with general formula $Ba_{x/2}Li_{1-x}Ti_2(PO_4)_3$ ($0.4 \leq x \leq 1$) have been prepared by a conventional solid state reaction process and characterized by X-ray Diffraction (XRD), Fourier Transform infrared (FTIR), Raman spectroscopy, Nuclear Magnetic Resonance (NMR) and Complex Impedance Spectroscopy (CIS).

The XRD analysis of the samples indicated the formation of a single-phase with rhombohedral structure (space group $R\bar{3}C$). The refinement of cell parameters has been performed by the Rietveld method [1], showing that these compounds form a continuous solid solution. FTIR, Raman and ^{31}P, ^{7}Li MAS-NMR spectra of these compounds showed the formation of isolated PO_4 groups in these phosphates.

From the electric measurements the frequency dependence of bulk conductivity is found to obey the Jonscher's universal power law. The maximum conductivity of 7.86×10^{-4} $S.cm^{-1}$ and minimum activation energy of 0.39 eV, were obtained for $Ba_{0.3}Li_{0.4}Ti_2(PO_4)_3$ (x=0.6) at 623 K.

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Nanocellulose from agricultural residues: Mechanical Properties and morphological features

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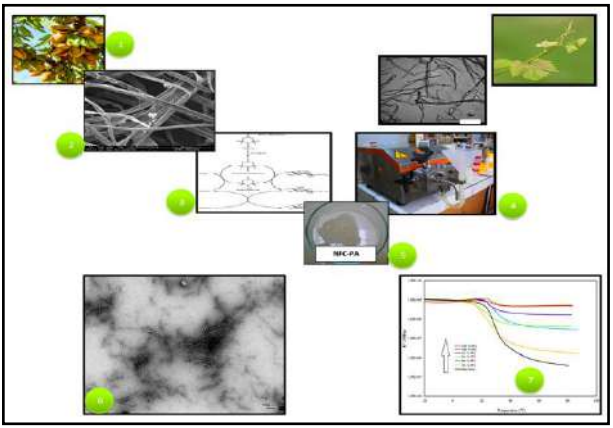
Important quantities of agricultural residues such as *Prunus amygdalus* and vine stem accumulate every year in Tunisia. The rational valorization of this available renewable resource fits very well with the recent sustainable approach, established nowadays. In this context *Prunus amygdalus* and vine stem was used as the starting material to produce nanofibrillar cellulose. Thus, NFC gel was prepared by disintegration in a high pressure homogenizer of cellulose pulp extracted from *Prunus amygdalus* and vine stem. The ensuing NFC suspension was characterized by several methods such as the fibrillation yield, transparency and morphological features. The reinforcing potential of the ensuing NFC was also established using dynamic mechanical analysis (DMA) from measurements carried out on nanocomposite films prepared by casting a mixture of NFC suspension and commercial acrylic latex. The prepared materials have showed promising mechanical properties.



Keywords: *Prunus amygdalus*, Vine stem, NFC gel, TEM, DMA.

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The authors express their sincere gratitude to Dr. Jean-Luc Putaux (CERMAV, Grenoble) for his support and availability as well as to the "PHC-UTIQUE CMCU" (project 13G1114) for financial support.

Microstructural properties and structure defects of the mechanically alloyed $Fe_{50}Co_{25}Ni_{15}B_{10}$ powder mixture

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Nanocrystalline $Fe_{50}Co_{25}Ni_{15}B_{10}$ powder mixture was prepared by mechanical alloying in a high energy planetary ball-mill under argon atmosphere. Structural and morphological properties and thermal stability changes during the milling process were followed by X-ray diffraction and scanning electron microscopy and differential scanning calorimetry. A multiple nanostructure of Fe(B), Co (CFC), (FeNi et FeCo), $Fe_{23}B_6$ et (Fe_3B, Fe_2B et Fe(Co, Ni)) nanocrystals within a partially amorphous matrix were achieved for 1, 2, 4, 10 et 15 h milling. For extended milling time, a single nanocrystalline Fe(Co, Ni, B) phase was identified. The phase transformations are related to the increase of dislocation and accumulation of stacking faults. The nanostructure formation caused by mechanical alloying is commonly attributed to the generation and movement of dislocations. Depending on the structural state of the milled powders, two or several overlapping exothermic peaks over the temperature range 100–700°C were revealed in the DSC scans.

Keywords: Mechanical alloying; Nanostrucure; X-ray diffraction; Thermal stability.

SYNTHESIS AND CRYSTAL STRUCTURE DETERMINATION OF THE NEW $[Cu^{II}_{0.5}\ Cu^{I}_{1}(SCN)_2DMSO]$ COMPOUND

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In recent years, thiocyanate transition metal complexes have received considerable interest due to their diverse structures [1]. This work presents a new mixed valence copper complex $[Cu^{II}_{0.5}\ Cu^{I}_{1}(SCN)_2DMSO]$ (SCN = thiocyanato and DMSO = dimethyl sulfoxide). Compound has been synthesized and characterized by crystallographic method analysis. The crystal structure was determined by single crystal X-ray diffraction studies. It crystallizes in the monoclinic system with space group $P2_1/n$, $Z = 4$, with unit cell parameters a = 5.9156(4) Å, b = 7.2304(5) Å; c = 21.9025(16) Å and β = 93.991(4)°. The three-dimensional structure of the binuclear thiocyanato bridged compound afforded two different environments for both copper centers in the unit cell: a tetrahedron and a square-planar. The zig-zag tetrahedron chains are inter-linked through a sulfur atom between the neighboring copper atoms and then bridged through SCN^- groups giving rise to sheets intercalated by square planar copper center. The DMSO ligands involved in the square planar coordination occupy the cavities formed by the metallic centers.

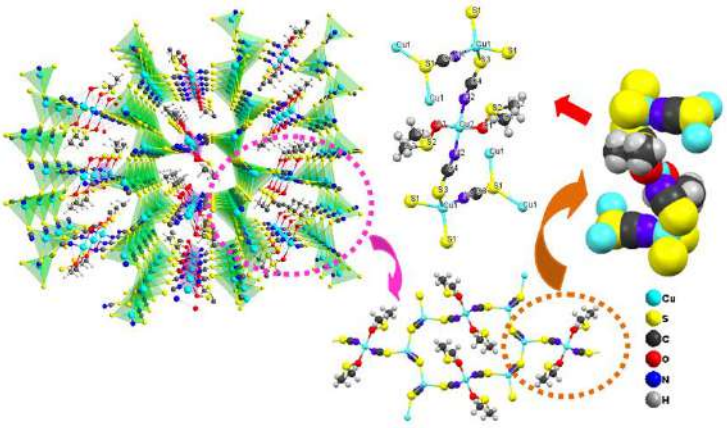

Fig : The molecular structure of $[Cu^{II}_{0.5}\ Cu^{I}_{1}(SCN)_2DMSO]$.

Key words: Crystal structure, X-ray diffraction, Cu(II/I) complexes, Thiocyanate

Reference:

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Effect of copper addition on structure defects in nanostructured Fe-Al-Cu alloys

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The evolution of the microstructure of nanostructured disordered iron–aluminium-copper alloys of $Fe_{50}Al_{50-x}Cu_x$ (x = 10, 20 and 30) compositions during the high energy mechanical alloying process was investigated by X-ray line profile analysis. Scanning electron microscopy (SEM) was employed to examine the morphology of the samples as a function of milling times. Analysis of line breadths was carried out to get an insight into the interrelated effects of grain size, lattice strain and dislocations. The transformation of the phase depends upon the milling time. With the increase of milling time all Al and Cu atoms became dissolved in the bcc Fe and the final product of the mechanical alloying process was the nanocrystalline Fe(Al,Cu) solid solutions with a mean crystallite size of about 10 nm. On the basic on the modified Williamson–Hall plots, the root-mean squared strains were explained by the presence of the high density of the dislocations. The identified steady-state saturation values of the microstructural parameters can be related to accumulate strain hardening of the powder material during longer milling times.

Keywords: Mechanical alloying; Nanostructured materials; Phase transitions; X-ray diffraction; Dislocations.

Synthesis and structural study of $(C_5H_7N_3)_3.[VO_2(C_2O_4)_2].3H_2O$

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Much attention is devoted to the oxalate ion, $C_2O_4^{2-}$, well known to be an attractive ligand, aiming at the synthesis of novel oxalates of transition metals.
In the present work, we sought to synthesize a novel vanadium oxalate of composition $(C_5H_7N_3)_3.[VO_2(C_2O_4)_2].3H_2O$ (I) (Fig 1).
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, C2/c space group with the cell parameters: a=38.698(2)Å, b=7.583(5)Å, c=21.206(4)Å, β= 116.34(6)° and volume V=5576,5(5)Å3.
The phase was characterized by infrared spectroscopy and by UV-visible spectroscopy, and both IR and UV-visible measurements support the structural results.

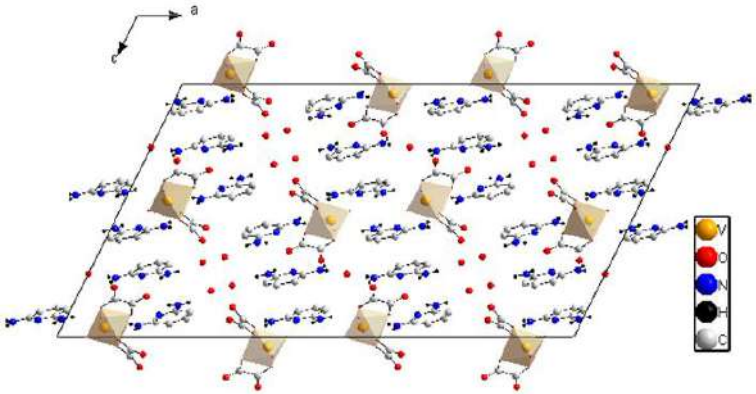

Fig 1: Projection along the b axis of the atomic arrangement of (I)

Key words: Oxalate, vanadium complexes, crystal structure.

Orientation Imaging- ASTAR Investigation of the Grain and Precipitate morphology in Al-Cu-Mg Alloy processed by Equal Channel Angular Pressing.

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Multiphase alloys are usually serious candidates for structural applications. Their mechanical properties depend on the precipitate distribution. We have studied the effects of Equal Channel Angular Pressing (ECAP) on the morphology of grains and precipitates in an Al-Cu-Mg alloy. Precipitates are mainly incoherent θ-Al_2Cu and S-Al_2CuMg. ECAP was performed using a die with an angle 90° up to three passes. TEM shows that high density of dislocations is created for the two first passages then for N=3, new grains 200nm in size are clearly observed (fig.1). Orientation imaging (ASTAR) shows that many precipitates are crushed by ECAP and that the new grains are randomly oriented. The new high angle grain boundaries are pinned by precipitates and the grain size seems to be limited by the distance between precipitates.[1,2]

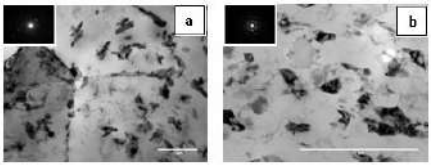

Fig. 1. Bright field images of the Al-Cu-Mg alloy before and after ECAP. (a) N = 0 and (b) N = 3 with corresponding SA diffraction patterns.

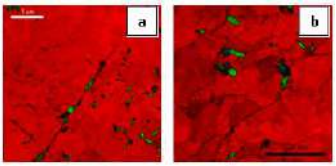

Fig.2: Precipitate morphology in the Al-Cu-Mg alloy before and after ECAP as revealed by ASTAR/Megastar program. (a) N=0, (b) N=3Bc.

Keywords: Aluminum alloys, ECAP, TEM, ASTAR

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Synthesis and characterization of un-doped ZnS particle: Study of their Antibacterial and Antioxidant Activity

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Zinc sulfide (ZnS) particles were synthesized and characterized by X-ray diffraction (XRD)**,** transmission electron microscopy (TEM) and Fourier transform infra-red (FT-IR) spectroscopies. The sizes of the crystallites were estimated using the Debye-Scherrer formula based on the XRD data. Zinc sulfide particles were screened for their antibacterial activity and for their antioxidant activity with 1,1-diphenyl-2-picrylhydrazyl (DPPH), hydroxyl radical (OH•) and hydrogen peroxide (H_2O_2) scavenging activity, ferric reducing power (FRP) assay and ferrous ion chelating (FIC) methods. It was found that ZnS particles showed appreciate both antioxidant and antibacterial activity.

In the last two decades, synthesis of nanoparticles has a rapid advancement. Among various classes of nanoparticles, semiconductor nanomaterials such as ZnS, have emerged as important materials with promising applications in nanotechnology [1, 2].

Keywords: ZnS particles, antioxidant activity, antibacterial activity.

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DEGRADATION OF SYNTHETIC DYES BY A NEW ELECTRO-FENTON PROCESS USING CHALCOPYRITE AS HETEROGENOUS FERROUS IRON AS CATALYST

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The mineralization of synthetic azo dye Reactive Orange 16 (RO16) has been studied by an electrochemical advanced oxidation process (EAOP) consisting in electro-Fenton (EF) oxidation catalyzed by chalcopyrite (CP) as heterogeneous catalyst. In this process the ferrous ions as catalyst needed for the Fenton's reaction has been supplied by the continuous dissolution of solid CP surface instead of a soluble iron salt. The experiments were performed at constant current in an undivided cell equipped with boron-doped diamond (BDD) anode and commercial carbon felt cathode to electrogenerate in situ H_2O_2 and regenerate ferrous ions as catalyst. The effect of operating conditions such as applied current, CP concentration, and initial dye content were investigated. RO16 mineralization efficiency was monitored during the electrolysis by TOC measurements. The experimental results showed that RO16 was quickly oxidized by the reaction with hydroxyl radicals ($^{\bullet}$OH) produced simultaneously both on BDD surface by water discharge and in solution bulk from electrochemically assisted Fenton's reaction. RO16 solutions with 100 mg L^{-1} were completely mineralized in 3 h applying a constant current of 300 mA in the presence of 1 g L^{-1} of CP as solid catalyst.

Keywords: CP, electro-Fenton, RO16, Heterogeneous catalyst, BDD anode, Mineralization, Hydroxyl radical

INFLUENCE OF ANODE MATERAL ON THE ELECTROCHEMICAL DEAGRADATION OF SYNTHETIC DYES

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The anodic oxidation of methyl Orange has been studied by cyclic voltammetry and bulk electrolysis, using a range of electrode materials such as Ti-Ru-Sn ternary oxide, lead dioxide and boron-doped diamond (BDD) anodes. The results show that polymeric films, which cause electrode fouling, are formed during oxidation in the potential region of supporting electrolyte stability.

While the Ti-Ru-Sn ternary oxide surface cannot be reactivated, $PbO_2$and BDD can be restored to their initial activity by simple anodic treatment in the potential region of electrolyte decomposition. In fact, during the polarization in this region, complex oxidation reactions leading to the complete incineration of polymeric materials can take place on these electrodes due to electrogenerated hydroxyl radicals. Moreover, it was found that BDD deactivation was less pronounced and its reactivation was faster than that of the other electrodes.

The bulk electrolysis showed that the complete COD and colour removal were only achieved using lead dioxide and boron-doped diamond while Ti–Ru–Sn ternary oxide only permitted a partial oxidation of MO.

Keywords: Methyl Orange; voltammetry; anode materials; electrode fouling; Bulk electrolysis; oxidation.

Phase transformations and microstructural properties of mechanically alloyed Fe-Si-Nb-B solid solutions

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Nanostructured Fe-Si- Nb-B powders were prepared by mechanical alloying of Fe, Si, B and Nb elements in a high-energy planetary ball mill. The structural properties and morphology changes were investigated as a function of milling time (in the 0–100 h range) by means of X-ray diffraction and scanning thermal analysis. At early stage, the dissolution of B and Si atoms into the Fe lattice leads to the formation of bcc Fe(Si,B) solid solution. After milling until 100 h, a mixture of Fe(Si,B) (~20 nm) and Nb(B) (~10 nm) is obtained. On the other hand, an interesting phenomenon called mechanical recrystallization, related to the formation of a crystalline phase of a partial amorphous powder (obtained at ~56 h), was investigated. Depending on the structural state of the milled powders, several transition temperatures were revealed in the DSC scans of the ball-milled powders in the temperature range 25-700 °C.

Keywords: Mechanical alloying, Fe-Si-Nb-B alloy, X-ray diffraction, Thermal analysis.

Synthesis, spectroscopic characterization and X-ray structure of N-3-pyrazolyl amidine

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Recently, new synthetic routes to substituted amidines are investigated [1]. Amidines may take the Z or E configuration [2]. Their derivatives have several applications in various fields, such as anti-inflammatory [3], antibacterial [4]. The synthesis of N-3-pyrazolyl-amidine crystal was prepared by a recristalisation of the obtained powder and characterized by IR, ^{1}H NMR, ^{13}C NMR, 2D NMR, SEM, X-ray diffraction and DFT calculation. The crystallographic study showed that the amidine is stabilized by the existence of intermolecular hydrogen bonds and probably of intramolecular hydrogen bonds and has Z configuration.

Keywords: 3-Amino-5-methylpyrazole, FT IR, X-ray diffractometry, 2D NMR, SEM and DFT.

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Spectroscopic studies of inclusion complex of β -cyclodextrin and 3-Amino-5-methylpyrazole Picrate

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In this paper, we have synthesized a new picrate derived from 3-amino-5-methylpyrazole by the reaction between heterocyclic amines and picric acid in ethanol at room temperature. Then we synthesized a new complex between the pyrazolyl picrate and β-cyclodextrin.

Picric acid is having tendency to form stable picrate with various organic molecules through ionic and hydrogen bonding and π-π interactions. [1-2]. The complexation with cyclodextrins can increase the solubility of compounds [3-5]. The optical properties of the picrate and the complex were determined by the UV-Vis Spectroscopy. The formation of inclusion complex was confirmed using the powder X-ray diffractometry, SEM, ^{13}C NMR, ^{1}H NMR, dept 135, NMR ^{1}H–^{1}H Cosy, NMR ^{1}H–^{1}H Noesy, which were used to provide information about the position of picrate inside the cavity of β-cyclodextrin.

Keywords: β-cyclodextrin, 3-Amino-5-methylpyrazole Picrate, UV-Vis Spectroscopy, FT IR, X-ray diffractometry, SEM,, ^{13}C NMR, ^{1}H NMR, dept 135, NMR ^{1}H–^{1}H Cosy and NMR ^{1}H–^{1}H Noesy.

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AMORPHIZATION OF CELECOXIB IN THE PRESENCE OF POLYVINYLPYRROLIDONE

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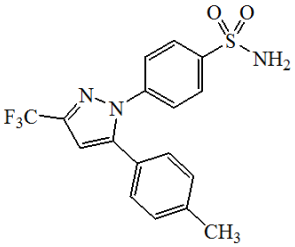
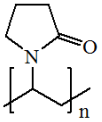
Bioavailability, stability, and processability are key considerations in drug formulations [1]. Crystallinity, which can critically influence these considerations, has been an essential subject in pharmaceutical research and development [2].Unfortunately, the amorphous state prepared by melt quenching is only stable for days to months due to its molecular mobility [3]. As part of this work, we studied the possibility of developing a celecoxib (CLX) dispersion in a polymer matrix polyvinylpyrrolidone (PVP) by adopting the technique of grinding, for to achieve a stable amorphous mixture.



Celecoxib (CLX)



Polyvinylpyrrolidone (PVP)

A characterization of CLX/ PVP binary systems by DRX, DSC and FTIR techniques provided greater insight into the molecular interaction between the two species. The stability of the resulting mixture (partially amorphous) is mainly due to the establishment of strong intermolecular interaction between the active pharmaceutical ingredient and the additive. Particularly, a hydrogen bond is established between the NH_2 group of CLX and the C = O of PVP.

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COMPLEXING AND EXTRACTANT PROPERTIES OF CALIX[4]ARENES

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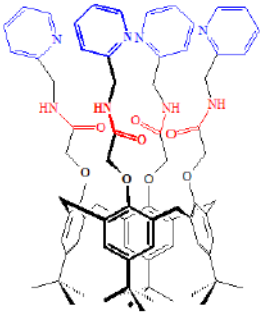
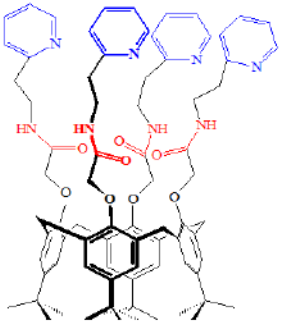
The present study deals with the analysis of the complexing and extractant properties of two derivatives.





The metal cations considered are alkali metals and some transition metals. Experimentation was conducted mainly in acetonitrile. To assess the stoichiometries of the formed complexes and location of the coordination sites as well as the selectivity, we used several study methods including UV-visible spectrophotometry and proton NMR.
The solid liquid extraction of metallic picrates followed by proton NMR showed an intra- or intermolecular exchange of cations within the calixarene units of the studied compounds.

Key words: Calix[4]arene, coordination, extraction

Synthesis and characterization of a new Organic- Inorganic sulfate $(C_5H_6N_2O)_2[Co(H_2O)_6]_3(SO_4)_4.2H_2O$

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A new organic-inorganic hybrid compound,$(C_5H_6N_2O)_2[Co(H_2O)_6]_3(SO_4)_4.2H_2O$, were synthesized in aqueous solution and characterized. The structure was determined by single crystal X-Ray diffraction analysis. This compound crystallizes in the triclinic system with the space group P-1, the unit cell parameters are a=6.632(3) Å, b=11.769(5) Å, c=14.210(6) Å, α=67.86(4)°, β=81.32(4)°, γ=85.18(4)° and V=1015.14(8) $Å^3$. The asymmetric unit of the title complex contain one and a half hexaaquacobalt(II) cations, one 2-amino-6(1H)-pyridone cation, two isolated sulfates anions and one water molecule of crystallization. Its crystal structure can be described as a packing of alternated inorganic and organic layers. The different components are connected by a three-dimensional network of O-H…O and N-H…O hydrogen bonds (Fig. 1). IR and UV-Visible spectroscopies were also used to characterize this complex. Moreover, differential scanning calorimetry (DSC) has revealed a structural phase transition of the order-disorder type around 373 K (Fig. 2).

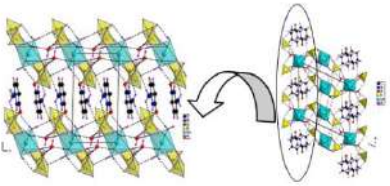

Fig.1 crystal packing of the complex $(C_5H_6N_2O)_2[Co(H_2O)_6]_3(SO_4)_4.2H_2O$

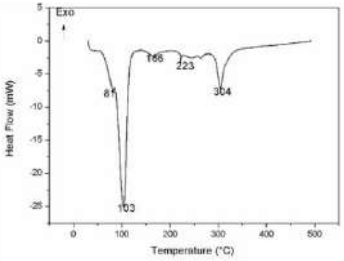

Fig. 2 DSC thermogramme

Key words: X-Ray diffraction, IR spectroscopy, Band-gap, Phase transition.

A new member in the chlorocobaltate hybrid family: Synthesis, Crystal structure and Characterization

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Organic–inorganic hybrid materials have received extensive attention in recent years owing to their great specific properties, such as electronic, optical, thermal and catalytic. Halometallates (II) have been of special interest of their structural flexibility. As a contribution to the investigation of the above materials, we report here the crystal structure of bis(imidazo[1,2-a]pyridin-1-ium)tetrachlorocobaltate (II) dihydrate$[C_7H_7N_2]_2[CoCl_4].2H_2O$, synthesized by slow evaporation at room temperature of an acidic solution of $CoCl_2.6H_2O$ and imidazo[1,2-a]pyridine in a 1:1 molar ratio. The synthesize product crystallizes in the monoclinic C2/c space group, with the following unit cell dimensions: a=11.573 (3)Å, b=9.777 (4)Å, c= 17.512 (5)Å. The crystal structure was determined and refined down to R=0.028. The structural unit consists of two $[C_7H_7N_2]^+$ cations, one isolated $[CoCl_4]^{2-}$ tetrahedron and two water molecules, The different entities are held together by hydrogen bonds ensuring the cohesion and the structure stability and building a three dimensional network exhibiting empty channels running along c axis as shown in Figure1.

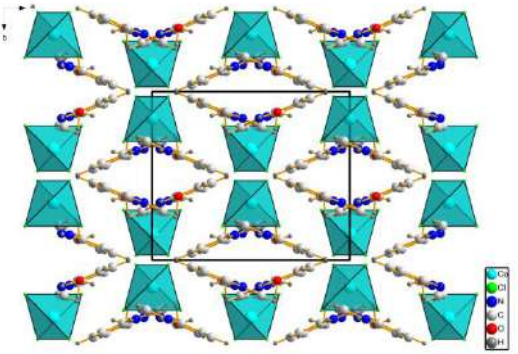

Figure 1: Projection of the structure of the title compound $[C_7H_7N_2]_2[CoCl_4].2H_2O$ exhibiting empty channels running along c axis.

Raman scattering study of phase transitions in LAMOX compounds

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LAMOX are new materials deriving from $La_2Mo_2O_9$ extensively studied due to their recently discovered high ionic conductivity (better than best stabilised zirconia above 600°C), so that applications as fuel cell electrolytes can be predicted. Other applications as electrode materials and oxidation catalysts are also possible with regard to controls of the molybdenum oxidation states. In the reference compound $La_2Mo_2O_9$, the transition from a weakly conducting phase (α-$La_2Mo_2O_9$, monoclinic, ordered) to the highly conducting phase (β-$La_2Mo_2O_9$, cubic, disordered) occurs at 580°C.

The purpose of the present work is to explain the mechanisms of conduction and of the transition by using theoretical approaches based upon group theory and *ab initio* calculations of the phonon spectra and experimental results provided by Raman scattering.

The study by Raman spectroscopy of LAMOX was undertaken in order to: follow the dynamic properties of the system according to the temperature, contribute to the understanding of the mechanism of transitions and find the origin of the conduction regime change depending on the temperature.

Key words: LAMOX; $La_2Mo_2O_9$; mechanisms of conduction; *ab initio* calculations; mechanism of transitions; Raman spectroscopy.

Calcium Hydride as a Powerful Reducing Agent for Topotactic Oxide Deintercalation: Synthesis, Characterization and Crystallographic Evolution, T→ pseudo S → T' of the Topotactically Reduced $La_{2-x}Ca_xCuO_{4-\delta}$ ($0 \leq x \leq 0.2$).

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Low-temperature reactions are a powerful approach to generate new transition metal oxides that are inaccessible by conventional high-temperature reactions. In this review, we describe the recent progress of the topochemical reduction method using metal hydrides (CaH_2, NaH, LiH) for transition metal oxides, in particular, focusing on structural modifications, chemical and physical properties, and the factors that direct selective and rational preparations. The capability of calcium hydride as a reducing agent in oxide deintercalation reactions is explored. Reaction of the n = 1 Ruddlesden-Popper phase $La_{2-x}Ca_xCuO_{4-\delta}$ ($0 \leq x < 0.2$) with CaH_2 in a sealed evacuated tube at $200 \leq T/°C \leq 300$ yields the topotactically reduced phase $La_{2-x}Ca_xCuO_{3.5}$ (A/2m, a = 8.6224(6), b = 3.8446(2) Å, c = 13.0179(10), β = 109.690(5) °). The variation in the anion vacancy distribution of these reduced phases as a function of stoichiometry is discussed in relation to the coordination polyhedra of the metal cations. Structural characterization indicates the coexistence of a new oxygen-vacancy-ordered arrangement of cooperatively distorted Cu_2O_3 planes containing 2- and 4-coordinate Cu^+/Cu^{3+} sites is found.

Key words: Nonstoichiometric oxides, Topotactic reaction mechanism, Oxygen mobility, Ruddlesden-Popper phase.

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Synthesis and characterization of mesostructured materials composition SiO2 Application in the adsorption of heavy metals

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A large number of rejections, particularly liquids, carry heavy metals in varying amounts. This is a major concern for public of the consequences that may occur on living species and their environment

The main objective of this work is the use of mesoporous silica-based materials for the adsorption of cobalt ions from synthetic aqueous solutions simulating the origin of industrial effluents.

Two types of mesoporous materials CMI-1 and SBA -15 were synthesized by the self-assembly mechanism cooperative (CTM), and characterized by X-ray diffraction and adsorption-desorption nitrogen (BET) and tested for the adsorption of cobalt (II) ions. The equilibrium and adsorption kinetics were modeled by Langmuir, Freundlich, Temkin and Dubinin-Radushkevich (D-R).

Keywords: mesoporous silica, adsorption, kinetic, isotherm, heavy metals, cobalt (II)

Synthesis, Crystal structure and Characterization of a new hybrid compound based on iodide lead

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Organic–inorganic hybrid materials have received extensive attention in recent years owing to their great unique structural, magnetic and optoelectronic functionality.

In particular, the lead iodide-based hybrid materials extensively studied for their excitonic and magneto-optical properties as they form natural quantum-well structures. We report in this work the crystal structure of $[C_4H_{12}N_2]_2[PbI_5].I.H_2O$, synthesized by slow evaporation at room temperature of a solution containing lead iodide and piperazine in a 1:2 molar ratio. The prepared compound crystallizes in the orthorhombic, P nma space group, with the following unit cell dimensions: a=8.7477(10) Å, b=13.488(2) Å, c= 20.336(3) Å, V=2399.44 (3) $Å^3$. The crystal structure was determined and refined down to R = 0.037. The structural unit consists of two $[C_4H_{12}N_2]^{2+}$ cations, one $[PbI_5]^{3-}$, one I^- and one isolated water molecule. The different entities are held together by hydrogen bonds building a three dimensional network and ensure the cohesion and the structure stability as shown in figure 1.

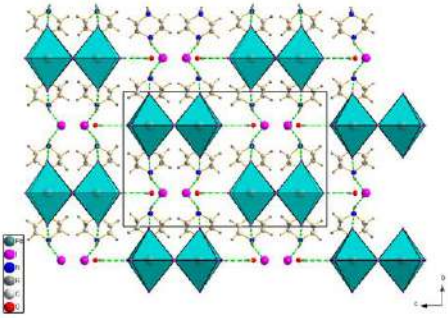

Figure 1: Projection of the structure of the title compound $[C_4H_{12}N_2]_2[PbI_5]I^- H_2O$ along a axis.

FUNCTIONALIZATION OF SILICA NANOPARTICLES

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Silica particles were synthesized by co-condensation of tetraethoxysilane (TEOS) and vinyltrimethoxysilane (VTMS) in basic medium by sol-gel synthesis according to Stöber-modified method. The porosity of the material was obtained by mesophase surfactant surrounded by polymerized silica. In this study, we investigated the effect of the functionalization of the silica particles by metallic nanoclusters (Au, Pt and Al) on the morphology.

The investigation of the morphology of the obtained mesoporous silica by MET and SEM (Figure1) showed that i) The silica particles have a surface area of about 1000 $m^2.g^{-1}$. ii) The particle morphology functionalized by Au depends on the synthesis temperature. Particles synthesized at room temperature (25°C) have cylindrical shape however particles are spherical at 80°C. iii) Adding platinum nanoclusters in the porous structure has no effect on the morphology of silica particles. iv) The morphology of the functionalized particles by aluminum depends on the molar ratios of Si/Al. Cylindrical particles are obtained by Si/Al = 80/2 and spherical for Si/Al = 60/4.

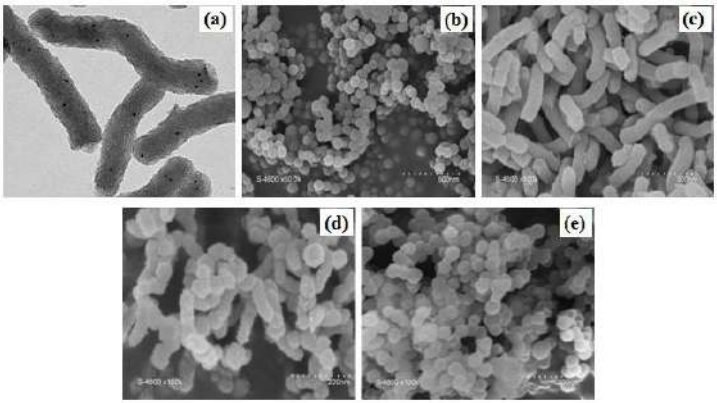

Figure1: Morphology of silica particles: (a) Au@SiO_2, r.T. ; (b) Au@SiO_2, 80°C ; (c) Pt@SiO_2, r.T. ; (d) Al@SiO_2, Si/Al = 80/2 ; (d) Al@SiO_2, Si/Al = 60/4.

Synthesis and characterization of a new di-adamantan-1-aminium monohydrogenophosphate acide fumarique $[C_{10}H_{18}N]_2[HPO_4].1.5[C_4H_4O_4]$

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Organic phosphate complexes have been widely studied due to their numerous practical and potential uses in various fields such as biomolecular sciences, catalysis, fuel cell research, liquid crystal-material development and quadratic nonlinear optics studies. Here, we report the synthesis and the crystal structure of a new hydrogen phosphate with an organic cation, $[C_{10}H_{18}N]_2[HPO_4].1.5[C_4H_4O_4]$, formed by the reaction of adamantan-1-aminium fumarate with orthophosphoric acid. This compound crystallizes in the monoclinic system, with the space group $P2_1/n$ and the following cell parameters: a = 12,755(2), b = 11,185(1), c = 20,251(2) Å, β = 105,795 (2)°, V = 2780,1(6) $Å^3$ and Z = 4. The crystal structure was solved and refined to R = 0.042 using 9001 reflections. The asymmetric unit of the title compound contains two adamantan-1-aminium cations, one hydrogenophosphate anion, and one and a half molecules of fumaric acid. Each $[HPO_4]^{2-}$ anion is hydrogen bonded, via all of its oxygen atoms, to four $[NH_3]^+$ groups of the adamantan-1-aminium cations, forming chains spreading along [100]. These chains are, in turn, interconnected via a set of O-H…O hydrogen bonds involving the fumaric acid forming layers parallel to (a, b) plane (Figure). Weak C-H…O interactions lead to a consolidation of the three-dimensional network.

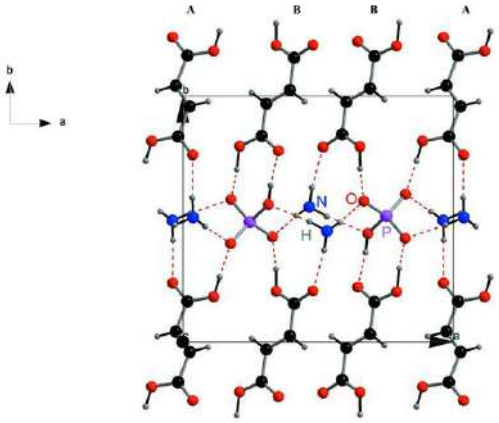

Figure. Projection along [001] of a layer in the structure of $[C_{10}H_{18}N]_2[HPO_4].1.5[C_4H_4O_4]$

Synthesis and physicochemical characterization of a new nitrate organic $[C_5H_9N_4].NO_3$

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The blending of the organic and inorganic components in the hybrid materials allows to the development of compounds having novel properties. Among these materials, salts of amines attracted more attention due to their potential importance. As a contribution to the study of this compound family, we report in this work the synthesis and the crystal structure of a new organic cation nitrate $[C_5H_9N_4].NO_3$. This compound crystallizes in the orthorhombic system, with the space group Pnma and the following cell parameters: = 19,7213 (2), b = 6,4245 (2),c = 6,7197 (6) (Å), V = 851,38(8) $Å^3$ and Z = 4. The crystal structure was solved and refined to R = 0.0429 using 2321 reflections. In the title compound, the organic cations and the nitrate anions are linked via N-H…O hydrogen bonds, forming infinite chains running along the *c*-axis direction. The values of the N-O bond lengths [1.2256 (19)-1.2642 (18) Å] and O-N-O angles [118.39 (16)-121.64 (15) °] indicate that the nitrate anion exhibits a slightly distorted C_{3h} geometry. The N atom of the NH_2 group has sp^2 character. This compound has also been characterized by infrared spectroscopy, solid state MAS-NMR and thermal analysis.

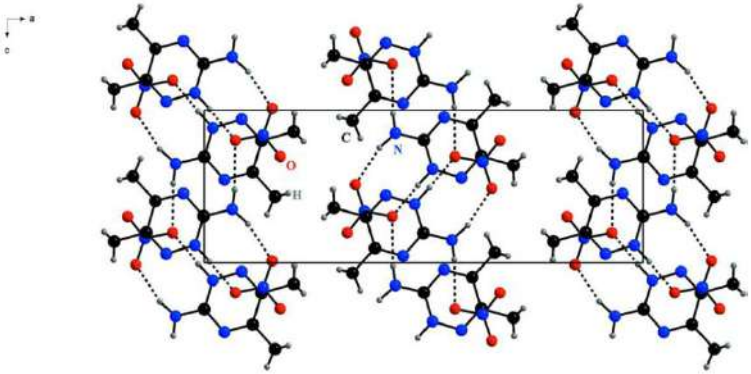

Figure. Crystal packing of the title compound, viewed down the *b*-axis, showing the chains formed between the nitrate anions and the organic cations.

MORPHOLOGICAL AND STRUCTURAL CHARACTERIZATION OF CELLULOSE FROM DEGLET NOUR SEEDS (*Phoenix dactylifera L.)*

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[b)] Laboratory of Pulp, Paper and Graphic arts sciences, UMR CNRS 5518, Grenoble INP-Pagora-461, rue de la papeterie, 38402 Saint-Martin-d'Hères, France

Date palm seeds (DS) were subjected to mechanical grinding then sodium hydroxide delignification and sodium chlorite single step bleaching. The optimization of the extraction was performed using response surface methodology. The obtained holocellulose was treated with potassium hydroxide to extract cellulose. The structural features of the isolated cellulosic sample were examined by Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). The morphological characteristics of the cellulosic preparation were investigated with fiber morphology analyser (MorFi). Grain size distribution was studied with laser particle size analyser CILAS 1190.
The degree of polymerization (DP) was determined by standard test method for intrinsic viscosity. It was found that cellulose extracted from date seeds (CDS) composed of fines (elements with the length under 100 μm and width under 5 μm) with a degree of polymerization of 950. The extracted cellulose (CDS) was composed of 90% of amorphous phase and has a monoclinic crystal structure Iβ. FTIR spectra show that extracted cellulose was free from lignin and hemicellulose.

Keywords: Date seeds, cellulose Iβ, X-ray diffraction (XRD), morphology analyser (MorFi),

EXPERIMENTAL DETERMINATION AND MODELING OF SORPTION ISOTHERMS OF LEAVES OF *CAPPARIS SPINOSA.L* (SOUTH OF TUNISIA)

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[b)] *Applied thermodynamics Unit Research, ENIGabes,*

The knowledge of the relationship between the equilibrium moisture content and the water activity is very important in order to describe the drying process.
The aim of this work was to determine the sorption isotherms of leaves of *Capparis Spinosa.L* (Matmata, south of Tunisia) and their modeling. Moisture adsorption/desorption isotherms were studied within the range of 10%-90% relative humidity at different temperatures (40, 50 and 60°C), using the gravimetric method. The results (Figure 1) showed that the sorption isotherms of leaves of *Capparis Spinosa.L* are of type II and that the equilibrium moisture content is temperature – dependent.
Experimental values were fitted using different models. The GAB model was found to be the more suitable for describing the sorption isotherms of studied leaves (Figure 2). We have also determined the parameters of this model. Using experimental results we have calculated the isosteric heat (Q_{St}) of sorption using Clausis-Clapeyron equation. The results (Figure 3) showed that the Q_{St} dropped when the equilibrium moisture content (EMC) increase.

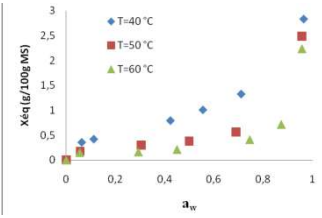

Figure 1: Desorption isotherms of water (leaves of Capparis Spinosa.L)

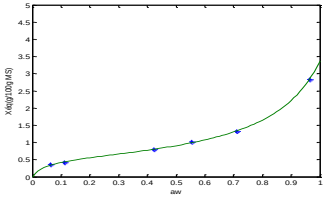

Figure 2 : Application of GAB's model for water desorption of Capparis spinosa.L at T=40°C

Key words: Sorption isotherms, modeling, Capparis Spinosa.L, isosteric heat.

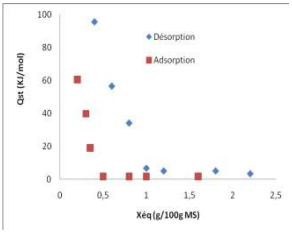

Figure 3 : Isosteric heat (Capparis spinosa.L).

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SYNTHESIS, STRUCTURE AND CHARACTERIZATION OF A NEW NONCENTROSYMMETRIC Zn(II) COMPLEX WITH THE 2-AMINO-5-CHLOROPYRIDINE LIGAND (ACLPY)

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A new noncentrosymmetric Zn(II) complex with the monodentate ligand 2-amino-5-chloropyridine (AClPy), $ZnCl_2(C_5H_5ClN_2)_2$, has been prepared at room temperature and characterized by single crystal X-ray diffraction and ^{13}C CP-MAS-NMR and IR spectroscopies. The basic coordination pattern of the AClPy coordinated metal cations is slighly distorted tetrahedral. The crystal structure is characterized by $ZnCl_2N_2$ tetrahedra interconnected via N-H···Cl hydrogen bonds generated by the NH_2 amino group to form chains extending along the (*a*-*c*) direction. The exocyclic N atom is an electron receiving center, which is consistent with features of imino resonance as evidenced by bond lengths and angles. The crystal structure is stabilized by sets of intra and intermolecular hydrogen bonds**.** The ^{13}C CP-MAS NMR spectrum is discussed and the vibrational absorption bands are identified by infrared spectroscopy and theoretical calculations.

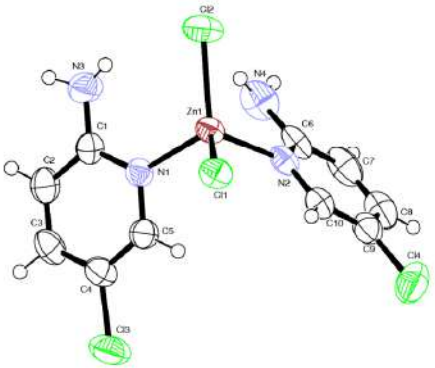

Figure. 1 Asymmetric unit of $[ZnCl_2(C_5H_5ClN_2)_2]$ with the atom numbering scheme and thermal ellipsoids at 50 % probability.

Key words: X-ray diffraction; Crystal structure; ^{13}C CP-MAS NMR; IR spectroscopy; DFT calculations.

Synthesis, X-ray structure, Thermal behaviour and Vibrational study of a new solid acid $K_2(HSO_4)_{1.5}(H_2AsO_4)_{0.5}$

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Crystals of a new compound with a superprotonic phase transition $K_2(HSO_4)_{1.5}(H_2AsO_4)_{0.5}$, have been synthesized for the first time by a slow evaporation method at room temperature. The structure of this new solid acid has been determined by single-crystal X-ray diffraction methods. The title compound crystallizes in the Triclinic system P1 with lattice parameters a = 7,160(6) Å b = 7,517 (1) Å, c = 7,584 (4) Å, α = 72,05(2)°,β = 88,11 (3)°,γ = 86,36 (4)° and a unit cell volume of V = 387,5 (4) $Å^3$, implying a density of 2.522 Mg. m^{-3}.

The structure is characterized by HSO_4 and disordered $(H_xS/As)O_4^-$ tetrahedral connected to dimers via hydrogen bridges. These dimers are linked and stabilized by an additional hydrogen bonds (O–H–O) and hydrogen bonds (O–H...O) to built an anionic chains of dimers, which are parallel to the [0 1 0] direction at x=0.5.

The infrared spectra of $K_2(HSO_4)_{1.5}(H_2AsO_4)_{0.5}$ recorded at room temperature in the frequency range 4000 - 400 cm^{-1}confirm the presence of two different anions (AsO_4^{3-} and SO_4^{2-}) in the same crystal.

Thermal-differential analysis of the superprotonic transition in $K_2(HSO_4)_{1.5}(H_2AsO_4)_{0.5}$ showed that the transformation to high-temperature phase occurs at 463 K by one-step process. Thermal decomposition of the product takes place at much higher temperatures, with an onset of approximately 674 K.

Keywords: Inorganic compounds; Mixed dipotassium hydrogensulfate dihydrogenarseniate; Crystal structure; IR spectroscopy.

Structural, magnetic and magnetocaloric properties of $SmNi_5$ compound

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The $SmNi_5$ intermetallic compound obtained under arc-melting conditions, have been investigated. Powder X-ray diffraction analysis and Rietveld refinement revealed that the sample is a pure compound stabilized with a hexagonal $CaCu_5$-type structure (space group P6/mmm) with the following lattice parameters: a= 4.9200 (3) Å, c= 3.9662 (4) Å [1]. These lattice parameters for our this compound are in good agreement with previous theoretical result and experimental data. The performed EDX analysis has been to confirms the composition of this compound. The chemical bonding in $SmNi_5$ was analyzed using atomic Hirshfeld surfaces, and the analysis supports the presence of the structural elements and coordination of Sm, Ni (2c) and Ni (3g). However, indications of interatomic interactions between the $SmNi_5$ atoms and the weak interactions between Sm atoms were also observed along the c axis. In this work, the magnetic properties and magnetocaloric effect (MCE) have been established by the magnetization and isothermal magnetization of different temperature measurements. The magnetization curve as a function of temperature shows a magnetic transition from ferromagnetic to paramagnetic state at the Curie temperatures T_C = 29 K. We have studied the MCE phenomena in the vicinity of magnetic phase transitions in terms of magnetic entropy change. The magnetocaloric effect of $SmNi_5$ is calculated in terms of isothermal magnetic entropy change for a field change of 50 kOe near T_C. This value of the magnetic entropy (ΔS_M) is about 2.38 J/mol.K and the relative cooling power (RCP) is in the range of 14.28J/mol for a field of 50 kOe [2].

Key words: rare earth, Magnetic and mangetocaloric measurements, X-ray diffraction

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Substitution of magnesium and zinc of the hydroxyfluorapatite

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Institut Préparatoire aux Etudes d'Ingénieur de Monastir, (Tunisia)

The biological apatite contains minor substituents such as F^-, Cl^-, CO_3^{2-}, SiO_4^{4-}, Na^+, K^+, Sr^{2+}, Zn^{2+} and Mg^{2+}. Therefore, the incorporation of such ions into the synthetic hydroxyapatite would enhance its biocompatibility and bioactivity. Magnesium and zinc co-substituted hydroxyfluorapatites with the general formula $Ca_{9.8}Mg_{0.1}Zn_{0.1}(PO_4)_6F(OH)$. The obtained powders have been characterized using different analyses. The results showed that the co-substitution of two cation for Ca^{2+} was incorporated. Indeed, no secondary phases were formed. Furthermore, the incorporation of Mg and Zn into the hydroxyfluorapatites lattice hasn't influenced its thermal behavior. The samples exhibit a thermal behavior stable. The Fullprof program was used to determine the lattice parameter for the powders. The obtained results showed that the 'a' parameter decreased with the insertion of the cation Mg and Zn.

A NEW ACENTRIC POLYMORPH MONETITE, $CaHPO_4$

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The chemistry and crystal structure of calcium phosphates is an interesting field of study for a long time. These compounds have been largely studied for their important properties and applications in different domains. They are studied for biomedical applications [1], for bone regeneration [2], for bio-implant [3], for dental and orthopedic applications [4]. The synthesis and structural characterization of anhydrous calcium hydrogen phosphates has been well known for a long time [5]. In this work we interest in crystal structure of a new acentric polymorph monetite. It was synthesized by hydrothermal reaction and characterized by single crystal X-ray diffraction. It crystallized in orthorhombic system, space group $Ccm2_1$ with z = 2. The crystallographic parameters are a=6.242(1)Å, b=6.994(2)Å and c=7.003(3)Å. A three-dimensional crystal structure can be described by $\{HPO_4\}_n$ infinite zigzag chains linked by Ca—O bonds. The comparison to the crystal structures with other polymorphs is given. The structural model is validated by both bond-valence-sum and charge-distribution methods, the distortion of the coordination polyhedra is analyzed by means of the effective coordination number and compared with others allotropic forms.

Key words: Calcium hydrogen phosphate, Polymorphs, Synthesis, Single crystal X-ray diffraction, Monetite, Crystal structure.

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SYNTHESIS, CRYSTAL STRUCTURE, CHARGE-DISTRIBUTION AND BOND-VALENCE SUM INVESTIGATIONS OF A NEW IRON PHOSPHATE: $TlFe(HP_3O_{10})$

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The novel thallium iron hydrogen tri-polyphosphate, $TlFe(HP_3O_{10})$, was synthesized under hydrothermally conditions at 453K and studied by single-crystal X-ray diffraction at room temperature. It crystallized in monoclinic system, space group C2/c with z = 4. The crystallographic parameters are a = 11.9938(1)Å, b = 8.4766(1)Å, c = 9.2766(1)Å and β = 111.99(1)°. The title compound framework is isostructural to $(NH_4)Fe(HP_3O_{10})$ [1], $RbMn(HP_3O_{10})$ [2], $KMn(HP_3O_{10})$ [3]. The crystal structure is built up from FeO_6 octahedral and (HP_3O_{10}) groups linked by corners. This arrangement leads to a three-dimensional anionic framework with tunnels parallel to the c-axis, where Tl^+ cations are located. The structural model is validated by both bond-valence-sum (BVS) and charge-distribution methods (CDM). The distortion of the coordination polyhedra is analyzed by means of the effective coordination number and compared with other similar compounds. The BVS model is extended to simulate the ionic migration pathways of Tl^+ cations in the anionic framework.

Key words: Thallium hydrogen phosphate, Synthesis, Single crystal X-ray diffraction, Crystal structure, Charge distribution.

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ELECTRO-FENTON-PYRITE TREATMENT OF VANILLIC ACID USING PT ANODE AND CARBON-FELT CATHODE

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Due to its advantages over classic electro-Fenton process such as less iron sludge formation, cheaper cost, wider working pH and possible recycling of iron promoter, a heterogeneous electro-Fenton process using a natural pyrite as catalyst was used to degrade vanillic acid (VA). Experiments were performed with a cell equipped with a Pt anode and a carbon-felt cathode. The effect of operating conditions such as pollutant and pyrite concentrations, applied current and initial pH on VA degradation has been studied. The VA decay kinetics and the mineralization of the aqueous solutions were monitored during the electrolysis, respectively, by HPLC analysis and TOC measurements. The experimental results showed that VA was completely removed by the reaction with $HO^{\bullet}$ radicals generated from electrochemically assisted Fenton's reaction and that the decay kinetic always follows a pseudo-first order reaction. At pH 3, applying a current of 300 mA and with pyrite concentration of 0.2 g/L, 93.7% of the initial TOC was removed in 360 min of electrolysis, meaning the almost complete mineralization of the organic content of the treated solution.

Key words: Pyrite, Heterogeneous catalysis, phenolic compounds, electro-Fenton, advanced oxidation processes.

ELECTRICAL PROPERTIES AND INTERNAL DYNAMICS OF BIS(PIPERAZINE-1,4-DIIUM) PENTACHLOROANTIMONATE (III) MONOHYDRATE

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Halogenoantimonates(III) and halogenobismuthates(III) with organic cations are interesting group of compounds due to their ferroelectric properties. In these compounds the metal shows a tendency towards distorted octahedral coordination with some rather long Sb–Cl bonds, which is attributed to aspherical distribution of Sb(III) lone electron pair. The polarity of these crystals is associated with phase transitions, which are mainly caused by the changes in rotational motions of the organic cations. Bis(piperazine-1,4-diium) pentachloroantimonate(III) monohydrate crystallizes in the monoclinic system with space group P21/n (a = 9.5409(2)Å, b = 14.1455(1)Å, c = 10.0525(7)Å, β = 99.113(8)°, Z = 4).The structure consists of piperazinediium cations, $[SbCl5]^{2-}$anions and a water molecules in which distorted SbCl5 square pyramids sharing two commons Cl atom are held together a bioctahedra of form $[Sb_2Cl_{10}]^{4-}$ and was stabilized by intermolecular N—H….Cl, O—H….Cl and N—H….O hydrogen bonds. The dielectric properties were measured using a Hewlett-Packard 4192A impedance analyzer at 50 kHz (E_a = 1 V/cm) in the range from 30 K to 325 K. At Tc=70 K the electrical permittivity exhibit their maximum value. The Curie –Weiss dependence was analyzed. The conductivity measurements were carried out in temperature range from 373 K to 448 K by means of impedance spectroscopy using a Novocontrol Alpha A Frequency Analyzer (0.1Hz, 10MHz). The impedance representations (Argand plots) have been used for the precise evaluation of their σ_{dc} conductivity. The temperature dependence of σ_{dc} obeys the Arrhenius formula which is characteristic for the thermally activated hopping process. Above 390 K the diffusion process of crystalline water influence on their electrical properties. In order to propose the model of internal dynamics of studied compounds, the 1H NMR investigation were performed in temperatures 50 K-500K. The activation parameters of piperazine-1, 4-diuum and crystalline water reorientations were obtained.

Keywords: molecular-ionic crystal, electrical permittivity, conductivity, internal dynamics.

Preparation and characterization of bis (4-methoxyanilinium) tetrachlorozincate

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A new zinc complex of formula $[C_7H_{10}NO]_2ZnCl_4$ has been prepared and characterized by X-ray diffraction, IR, UV-Visible and impedance spectroscopies.

The complex crystallizes in the monoclinic space group $P2_1/n$ with a minimal tetrahedral distortion of the $ZnCl_4^{2-}$ anion and with the lattice parameters: a = 12.054 (2) Å, b = 7.129 (3) Å, c = 23,480 (2) Å, β = 100.67 (2)°, V = 1983.03 (1) $Å^3$ and Z = 4.

The crystal structure can be described by organic layers of p-anisidinium cations held together by C-H…O hydrogen bonds parallel to (010) plane, linked to the inorganic groups of $ZnCl_4^{2-}$ through N-H…Cl hydrogen bonds, electrostatic and Van Der Waals interactions to form a three- dimensional network.

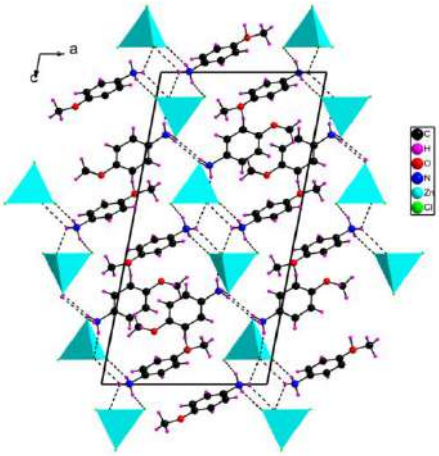

Figure 1. Projection of the structure of $[C_7H_{10}NO]_2ZnCl_4$ along the b axis

Keywords: complex, X-ray diffraction, IR spectroscopy, impedance spectroscopy, hydrogen bonds

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

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Microstructure evolution of 99.1% aluminum after equal-channel angular extrusion (ECAE) and subsequent heat-treatment was investigated. The deformed and annealed states were characterized by Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), and microhardness tests. It was shown that the observed microstructure changes during subsequent annealing have to be associated with recovery and cells formation. The initial stages of recovery were investigated using weak-beam technique. The microstructure obtained after annealing for 1 h at 100°C consists of some arrangements of the dislocations into sub-grain boundaries within the wide preexisting grains. Annealing at 300°C led to the appearance of a duplex microstructure consisting of bands of slightly coarsened grains associated with refined grains. No growth of dislocation cells was observed up to 400°C. In XRD measurements, the lattice parameter increase with subsequent heating. This indicates a continuous grain growth during annealing. This is due to the important increase of coherency length, D observed parallel to a substantial decrease of rms-strain, ε.

Key words: aluminum, equal-channel angular extrusion (ECAE), Transmission Electron Microscopy (TEM) and X-ray diffraction (XRD).

CATALYTIC ACTIVTY OF MOLYBDENUM SUPPORTED ON TITANIUM PILLARED CLAY: EFFECT OF MOLYBDENUM AMOUNTS

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The reduction of nitrogen oxide (NO), emitted from industrial plants, is a challenging task. Various processes have been proposed for removal of nitrogen oxides and amongst them, the selective catalytic reduction of NO (SCR-NO) is considered as the most efficient technology. The commercial catalyst for SCR-NO is based on TiO_2 as a support and V_2O_5/MoO_3 as promoters [1]. The properties of TiO_2 make of titanium-pillared clay (Ti-PILC) an interesting subject to study. Thus, considerable efforts have been made to investigate the substitution of conventional TiO_2 by Ti-PILC support in order to design a new efficient catalyst. Titanium pillared clays have been recognized as the promising catalysts for SCR-NO [2, 3]. In this work, a series of molybdenum supported on titanium pillared clay, prepared with different molybdenum amounts is prepared and characterized by different techniques. The catalytic activity of all samples has been evaluated for the SCR-NO (Fig. 1) and the results are discussed in terms of the physicochemical properties. The structural, textural, acidic, redox and catalytic properties are largely influenced by the amounts of molybdenum added to titanium pillared clay.

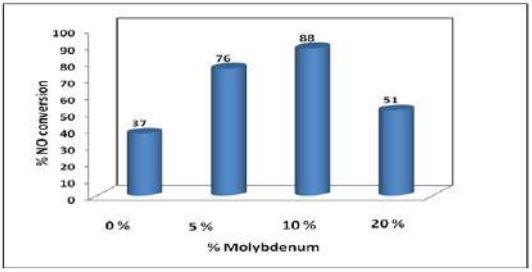

Fig. 1: NO Conversion over Mo supported on Ti-PILC.

Key words: Molybdenum, Titanium, Pillared clay, SCR-NO activity.

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Effect of the Al addition on nanocrystallisation ability Fe-Al-Ni-Zr-B alloy produced by mechanical alloying

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Mixtures of Fe, Al, NiZr and B powders with the nominal composition of $Fe_{50-x}Al_x(Ni_{70}Zr_{30})_{10}B_{10}$ (x=0; 20 et 30 at% Al) were mechanically alloyed in a planetary ball mill. The microstructure analysis was carried out via X-ray diffraction and scanning electronic microscopy according to milling time. It was found that a nanocrystalline structure could be achieved in the mixture after extended milling times. The patterns obtained were analyzed using the Rietveld refinement. The final products of the mechanically alloying process were a duplex nanostructure of Fe(Ni,Zr,B) and Fe_2B type boride nanocrystals. The predominant bcc structural phase has a lattice parameter larger than that of α-Fe. The increase of mechanical milling induced a continuous decrease in the crystallite sizes and an increase in the lattice strains. The thermal stability of the as-milled powders was analyzed by differential scanning calorimetry. At early stages of milling, the exothermic peaks observed at higher temperature corresponded to the reaction between the mixed powder elements, while at intermediate stages they were related to the recrystallization and crystallite growth. From the Mössbauer spectrometry results, it is observed that the magnetic hyperfine field decreases with the progressive mixing of the elemental powders, at the atomic level. This decrease mainly results from the presence of Al, Zr and B atoms as first near neighbor of Fe probe since Ni also does occur.

Keywords: Mechanical alloying; Fe-based alloy; Nanocrystalline; Thermal stability. Mössbauer spectrometry.

Purification of [6]-shogaol from *Zingiber officinale Rosco*, a phenolic compound having a high effectiveness against bacterial strains

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Rhizome of *Zingiber officinale Rosco*, one of the well-known medicinal plants, was analyzed for their chemical composition, and then antioxidant activity and antibacterial effect of extracts against some pathogenic bacteria were evaluated. For the first time, one step purification of [6]-shogaol from methanolic (20 %) extract was carried out. In the present study a solid phase extraction procedure was optimized for the purification of [6]-shogaol (N) which was eluted from a silica cartridge with 150 ml of *n*-hexane-diethyl ether (70/30, v/v). The structure elucidation of compound N was based on the spectral evidence UV, MS, ^{1}H-NMR and ^{13}C-NMR. The antioxidant properties of the pure compound as well as root extracts were investigated by different methods. In addition, antimicrobial activity of [6]-shogaol was estimated using a micro-dilution technique against some human pathogenic bacteria such as *Staphylococcus aureus, Escherichia coli, Pseudomonas savastanoa, Sallmonella enterica, Listeria monocytogenes and Agrobacterium tumefaciens*. The results of the present study can be proved useful in the purification of [6]-shogaol to produce a potentially bioactive compound.

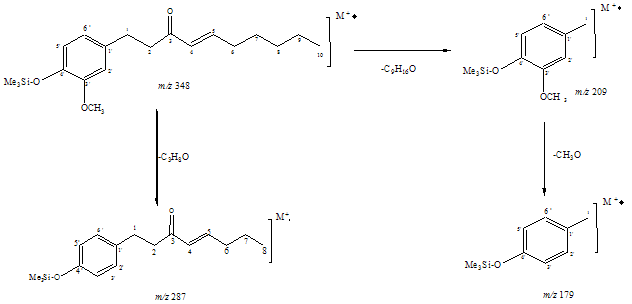

Figure 1: A proposed mass fragmentation of a sylilated [6]-shogaol.

Key words: *Zingiber officinale Rosco*, shogaol, DPPH, FRAP.

SYNTHESIS AND CHARACTERIZATION OF A NEW COMPLEX $[Co(C_4H_{11}N_2)_2(H_2O)_4](SO_4)_2.H_2O$

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In view of the importance of piperazines, which are found in biologically active materials across a number of areas of therapeutic importance [1], the structural chemistry of metal salts that include piperazine and its derivatives does not cease to develop and continues to be a subject of research in many laboratories. In this report, we present the crystal structure of Tetraaquabis(piperazin-1-ium)cobalt(II) bis(sulfate) dehydrate, $[Co(C_4H_{11}N_2)_2(H_2O)_4](SO_4)_2.2H_2O$, in which the metal ion is coordinated to the piperazinium moiety. Such coordination mode is not reported so far (Fig.1). Thermal analysis, IR and UV-V absorption spectroscopies, electric conductivity and magnetic proprieties of Tetraaquabis(piperazin-1-ium)cobalt(II) bis(sulfate) dehydrate are also reported.

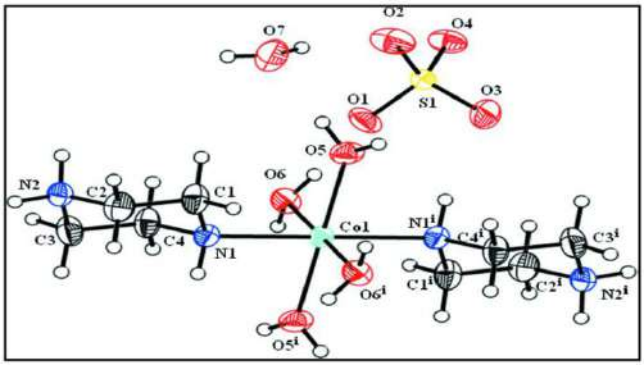

Fig.1 View of $[Co(C_4H_{11}N_2)_2(H_2O)_4](SO_4)_2.2H_2O$ with atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

Key words: Sulfates, piperazine, X-Ray diffraction, thermal analysis, Dielectric properties and magnetic proprieties.

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Ferroelectric and relaxor behaviour in the Perovskite-type $Bi_{0.5}(Na_{1-x}Li_x)_{0.5}TiO_3$ ($0 \leq x \leq 0.2$) solid solution

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New ferroelectric ceramics $Bi_{0.5}(Na_{1-x}Li_x)_{0.5}TiO_3$ ($0 \leq x \leq 0.2$) were synthesized by solid state reaction method. Room temperature X-ray diffraction and dielectric properties of ceramics sintered at different temperatures were measured in wide temperature (25-800°C) and frequency (100 Hz - 1 MHz) ranges. The studied materials adopt a very particular behaviour since they are classical ferroelectric in certain conditions and show a relaxor character in other conditions. The classical ferroelectric with rhombohedric symmetry was observed when they were sintered at 1050°C, 1100°C and 1150°C. The material became relaxor without changing symmetry if the sintering temperature is ≥ 1190°C. The influence of sintering temperature and initial lithium contents in the solid solution $Bi_{0.5}(Na_{1-x}Li_x)_{0.5}TiO_3$ on the physical properties were investigated. The Curie temperature Tc and also the maximum temperature T_m of ε'_r increase when the lithium content increases. The evolution of $\varepsilon = f(T)$ revealed two anomalies. The first occurs around 200°C, it is well known for all the rich NBT solid solution. It was attributed to the ferro-antiferroelectric transition. The second was diffuse and occurs around 320°C. It was associated to the phase transition in these compounds. At this temperature a weak dispersion appears as the frequency increases. However the dispersion is so small that the Curie Weiss law for relaxor compounds was not verified. Only when sintering temperature exceeds 1190°C, the relaxor behaviour became more and more pronounced and persisted even after several measuring cycles. The relaxor state could be attributed to increased number of vacancies likely due to the lack of Li^+, Bi^{3+} and O^{2-}.

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Synthesis of monodisperse silver nanoparticles

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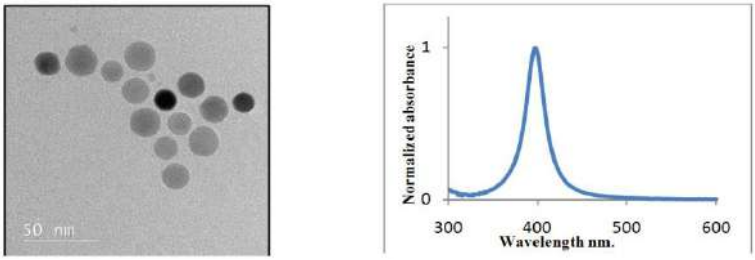
This communication describes a protocol to synthesize silver nanoparticles (AgNPs) of few tens nanometers diameter. By reducing silver nitrate by ethylene glycol in the presence of poly(vinylpyrrolidone) under inert atmosphere in the presence of a trace amount of NaSH, monodisperse AgNPs were obtained in high amount. By introducing an aged solution of sulphide ions in the reaction medium, the formation of anisotropic shape could be avoided and the process yielded in few minutes to the elaboration of spherical silver particles. Similarly to previous findings in silver photography technology, the presence of traces of sulfides and polysulfides, in the range of ppm, affected the particle growth by accelerating the rate of reducting of silver precursor. Due to the low solubility of silver sulfides species, sulfides nanocrystallites formed immediately and served as catalyst for the growth process. By merely adjusting the reaction time, this approach allowed to produce monodisperse particles over a tunable range of 20-40 nm. Moreover its are used to form a new core-shell nanostructures Silver@silica.



TEM micrograph and the corresponding absorption spectrum of silver nanoparticles obtained after 3 min.

Key words: nanoparticles, polyol approach, silver.

Magnetite based nanoparticles: Preparation, characterization and removal essays of Cu^{2+} from aqueous solution

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Copper is a metal of choice for technologies having industrial wide applications. Further, the worldwide increasing demand of the metal generates serious environmental problems because it's poisonous impact to mankind as well as to flora and fauna. The major contributors of Cu^{2+} pollution are the industrial effluents. Therefore, importance should be attached to how to effectively remove copper entities from wastewater effluents. Recovery of copper and then its reusing, and recycling can help community and the environment by saving money, energy, and natural resources. Today numerous techniques and methods are described for the removal of cupper entities from wastewaters [1][2]. Only a little number of them is considered to be environmentally friendly and economical. For instance, precipitation of copper by chemical precipitation (which is the most widely used technique) is achieved by the addition of coagulants such as alum, lime, iron salts and other organic polymers. The large amount of sludge containing toxic compounds produced during the process is the main disadvantage of the technique [2]. Nowadays, nanotechnology is involved in various application areas such as biology, medicine, environmental protection, etc. Several publications have studied the effects of increasing the specific surface area by switching to small magnetic nanoparticles or by modifying the surface with active surfactants against Cu^{2+} [3][4].

In the present communication we focused on recovering Cu^{2+} from aqueous solutions as models for wastewater of electroplating industry, using magnetite based nanoparticles (NPs). For this purpose, Fe_3O_4(Magn) and ZnO/Fe_3O_4(ZnO/Magn) NPs were prepared by co-precipitation and hydrothermal method, respectively. The structure, microstructure, the magnetic properties and the kinetic adsorption of Cu^{2+} by the NPs were investigated. XRD revealed a spinel-type structure and the presence of both spinel-type and wurtzite-type structure for Magn and ZnO/Magn powders, respectively. The results are supported by IR analysis. In addition, for the ZnO/Magn NPs, a quantitative phase analysis using Riveted refinement was performed. TEM images of the produced powders showed almost non agglomerated nanoparticles with an average size of about 100 nm and 10 for Magn and ZnO/Magn, respectively and with a relatively large dispersion in the shape and size for the last ones. Hysteresis loops revealed a superparamagnetic behaviour of the NPs at 300K. In addition, as expected, the saturation magnetization of ZnO/Magn NPs was found to be much lower than that of Magn NPs due to the diamagnetic character of ZnO [5]. The kinetic tests of Cu^{2+} removal by the as-produced NPs were conducted at 25 °C and at pH = 6.9, by mixing 10 mL of 100 $mg.L^{-1}$ Cu^{2+} solution with 10 mg of NPs. The stirring time was varied between 5 min and 24 hrs, while maintaining constant the other thermodynamic parameters. The kinetic's process of adsorption of Cu^{2+} can be described by two types of equations; the pseudo-first-order and the pseudo-second-order [6]. Kinetic constants obtained from different samples strongly depend on the microstructure of the NPs. The adsorption isotherms followed the Langmuir model. The maximum sorption capacity of Cu^{2+} of about $95mg.g^{-1}$ was found with the sample designed ZnO/Magn2. Further, the adsorption capacity varied from one sample to another depending on the composition of the NPs. In addition, it was observed that the increase of ZnO to Magn ratio increased the adsorption capacity of Cu^{2+}.

Keywords : co-precipitation, hydrothermal, nanoparticle, superparamagnetic, adsorption, kinetic.

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Structure and magnetic properties of the extented binary in to the ternary system GdCu2-xFex (x = 0.18)

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The study of the ternary Gd-Fe-Cu diagram enabled us to demonstrate the existence of a ternary composition $GdFe_{0.18}Cu_{1.82}$ in the domain of homogeneity $GdCu_{2-x}Fe_x$. This compound was synthesized by melt-solidification reaction using a vacuum arc furnace by a pump incorporated in the system. Alloy chip Ti / Zr is then melted to ensure the absence of any trace of oxygen.

The structural study by X-ray diffraction powder was performed on a polycrystalline obtained after annealing at 800 ° C for one week in a silica tube under the primary dynamic vacuum sealed after several purging with argon.

The intermetallic compound $GdFe_{0.18}Cu_{1.82}$ crystallizes in the hexagonal system. The structure was solved in the space group Fm-3m a = 7.309 (3) Å. The refinement structure converges to the reliability factors RP = 7.16 , RWP= 8.78

The structural arrangement is characterized by a substitution Fe / Cu on all crystallographic positions of the iron atom.

Magnetic moment measurements performed in the temperature range 475–700 K show conventional Curie-Weiss paramagnetic behaviour for both compounds above 690 K. At High temperature, magnetic susceptibility data reveal magnetic phase transitions. In the paramagnetic state, magnetic susceptibility for this compound follows the modified Curie–Weiss law and can be then written:

$$\chi = C/(T-\theta p) + \chi_0$$

The fitting of the inverse susceptibility for $GdFe_{0,18}Cu_{1,82}$ compound gives a paramagnetic Weiss temperature θp = 655 K, a Curie constant C = 9,81uem K/mol and an effective paramagnetic moment μ_{eff} = 8,86 (2) μ_B; et $\chi_0 = 610^{-4}$ emu/mol.

Contact angle, structural and optical properties and compatibility of Sol-Gel prepared B_2O_3 doped TiO_2 thin films deposited on glass substrates

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Titanium dioxide (TiO_2) is a versatile band-gap (3.2 eV for the anatase crystalline phase) semiconductor with a unique combination of chemical and physical properties, which includes its high refractive index, high dielectric constant, high UV photo-catalytic activity and good chemical stability. These properties make it suitable for the development of various optical thin film applications. When exposed to UV radiation with photon energy greater than the band gap energy of the material, the TiO_2 surfaces become super-hydrophilic and self-cleaning [1]. It is one of the most prominent oxide materials for performing various kinds of industrial applications such as photovoltaic [2], photocatalytic [3], water purification devices [4], optical coatings [5] and photochromic systems.

In this investigation, the boron-doped and undoped high transparent TiO_2 films on glass substrates were prepared by sol–gel spin coating method using titanium (IV) isopropoxide as a Ti source and boric acid (H_3BO_3) as B precursors.

The structure was investigated by X-ray diffraction (XRD) and the morphology by Atomic Force Microscopy (AFM) techniques. Results show an amorphous structure for one layer 50 nm thick. Crystalline structures were obtained after two or more layers corresponding to at least 100 nm TiO_2-B_2O_3 thin films.

The optical transmittance was investigated and the values of the optical band gap have been obtained for the studied films. We also determined the effect of B_2O_3 content on the contact angle after and before film exposition to UV radiations.

Keywords: Spin coating, thin films, TiO_2, hidrophilicity

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The new copper(II) complex with 4-Aminoquinaldine ligand: Structure and Hirshfeld surface

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A new coordination compound, $(CuCl_2)_4[H(C_{10}H_{10}N_2)]_4$ (1) where $C_{10}H_{10}N_2$ = 4-Aminoquinaldine (4-AQ) has been prepared under mild hydrothermal conditions and characterized by elemental analysis, Raman, IR and UV–Vis spectroscopic techniques. X-ray crystal analysis of the complex confirmed that the copper(I) ion has a distorted Tetrahedral environment. The inter-chain face to face π-π interactions among the aromatic rings of 4-AQ and the hydrogen bond interactions between 4-AQ molecules and Cl atoms result in 0-D network in the complex. The investigation of close intermolecular interactions between the molecules via Hirshfeld surface analyses is presented in order to reveal subtle differences and similarities in the crystal structures. The decomposition of the fingerprint plot area provides a percentage of each intermolecular interaction, allowing for a quantified analysis of close contacts within each crystal.

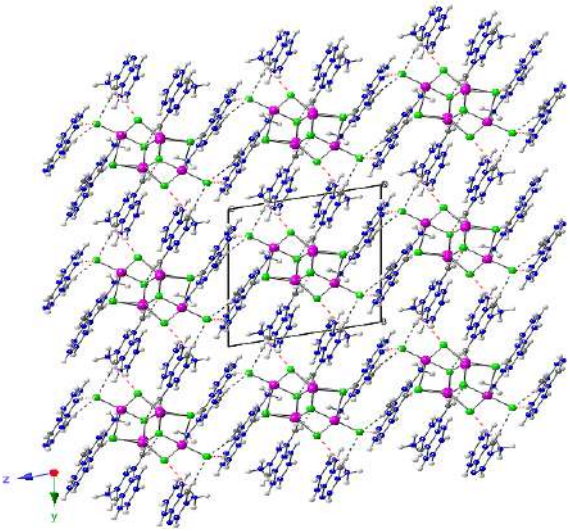

Fig.: Projection in the plane bc of the atomic arrangement of $(CuCl_2)_4[H(C_{10}H_{10}N_2)]_4$

SHORT TIME SYNTHESIS OF TITANATE NANOTUBES BY HYDROTHERMAL TREATMENT

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Titanate nanotubes (TNTs) have attracted extensive and engrossing interest since their first synthesis due to their special physicochemical properties. They have been used in various applications such as photocatalysis [1], energy production (ion-exchangeable and adsorption abilities in lithium batteries and dye sensitized solar cells respectively) [2-3], medicine (carriers of medicament) [4] and other applications.
In this work, titanate nanotubes were successfully synthesized by hydrothermal treatment for a short time reaction using a vigorous mechanical stirring. The effect of stirring time variation on the morphology of TNTs has been investigated. Results showed that agitation reduced significantly the reaction time. However, a continuous mechanical stirring leads to nanoribbons formation instead of nanotubes.
These nanostructures were characterized by a large range of methods including powder X-ray diffraction (XRD), transmission electron microscopy (TEM), X-ray Photoelectron Spectroscopy (XPS), thermal analysis (TGA) and Surface area measurements.

Key words: titanate nanotubes, nanoribbons, hydrothermal treatment, stirring.

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SYNTHESIS OF HIGH VALUE-ADDED ZEOLITIC MATERIALS USING WASTE GLASS AND ALUMINIUM SCRAP

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Zeolites are hydrated crystalline aluminosilicate materials with 3-dimensional framework of $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ tetrahedra linked at their corners to form pores with molecular dimensions (3-15 Å) and a regular arrangement of cavities and canals. Because of their molecular sieve properties and high thermal, mechanical and chemical stability zeolites have accounted of the majority of industrial applications such as adsorption, ion-exchange, separation and catalysis. The preparation of synthetic zeolites is expensive due to the use of high-purity reagents, such as the sources of silica, alumina and the structure directing agents. Hence, the recent studies are focused on the use of cheap natural raw materials or low-cost waste materials, as well as on the implementation of modern highly effective methods for their preparation. Inexpensive materials are mainly clays (kaolin, halloysite, illite,…), volcanic glasses (perlite and pumice) or waste products as ashes (coal fly, paper sludge and rice husk), and ceramic or glass wastes. Therefore, the aim of this study was to investigate the synthesis of high value-added zeolitic materials using waste glass and aluminum scrap as starting materials. The aluminum source was a scrap (99.77% purity) collected from aluminum workshop and the silica source was obtained by crushing the glass of fluorescent tubes collected from municipality waste. The crushed waste glass was sieved through a 125 µm steel mesh. The chemical analysis of the amorphous waste glass, assessed by X-ray dispersive fluorescence spectrometer is: SiO_2 (64.60%), alkalis (Na_2O_{eq}=Na_2O+0.658 K_2O) 22.04%, CaO (6.91%), MgO (3.00%), Al_2O_3 (2.59%) and Fe_2O_3 (0.21%). The alkaline fusion followed by a hydrothermal treatment was adopted for the preparation of the zeolitic materials. Eight grams of the waste glass (ø <125 µm) was mixed with the appropriate amounts of NaOH pellets in the weight ratios NaOH/waste= 0.8; 1.0 and 1.2. It was ground to obtain a homogeneous mixture. This mixture was then heated in a nickel crucible in air at 550°C for 1 h. The fused product was then mixed thoroughly with 80 mL of aluminate solution prepared from aluminum scrap. The slurry was first subjected to crystallize at 60°C for 6 days. The solid product was recovered by filtration and washed thoroughly with deionized water until the filtrate had a pH of 10–11.The XRD patterns of the recovered solids after crystallization using NaOH/waste ratio of 0.8 showed the reflexions of Na-X type zeolite and a small quantity of Na-P1. With the increasing of alkalinity (NaOH/waste= 1.0 and 1.2), the intensity of Na-X zeolite reflections decreased and Na-P1 zeolite phase appeared. This finding showed that higher alkalinity was required for the formation of the more stable Na-P1 zeolite.

Key words: Waste glass, fluorescent tubes, alkaline fusion, zeolite.

Synthesis and structural study of $(C_7H_{11}N_2)_2[VO(C_2O_4)(H_2O)].2H_2O$

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Much attention is devoted to the oxalate ion, $C_2O_4^{2-}$, well known to be an attractive ligand, aiming at the synthesis of novel oxalates of transition metals.
In the present work, we sought to synthesize a novel vanadium oxalate of composition $(C_7H_{11}N_2)_2[VO(C_2O_4)(H_2O)].2H_2O$ (I) (Fig 1).
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 triclinic system, P-1 space group with the cell parameters: a=7,639(4)Å, b=8,274(3)Å, c=21,283(2)Å, α=96,12(2)°, β=90,59(3)°, γ=117,42(5)° and volume V=1184,5(4)Å3.
The phase was characterized by infrared spectroscopy and by UV-visible spectroscopy. Both IR and UV-visible measurements support the structural results.

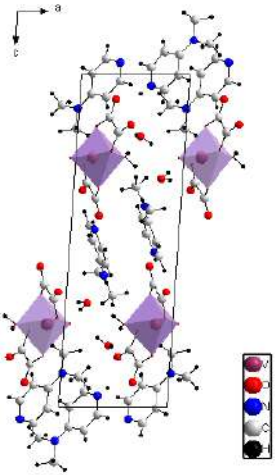

Fig 1: Projection along the b axis of the atomic arrangement of (I)

Key words: Oxalate, vanadium complexes, crystal structure.

Magnetic properties and DFT calculation of an oxo-bridged dinuclear iron(III) complex $(H\text{-phen})_2[Fe_2(H_2O)_6(phen)_2(\mu\text{-}O)](NO_3)_6$

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The crystal structure 1 of formula $(H\text{-phen})_2[Fe_2(H_2O)_6(phen)_2(\mu\text{-}O)](NO_3)_6$ has been determined by single-crystal X-ray crystallography. In the binuclear complex, two iron(III) centres are bridged by one oxygen atom to form al-oxo-diiron(III) complex with an Fe-Fe distance of 3.568(2) Å and a non linear Fe–O–Fe angle of 162.62(5) °. This binuclear complex features strong antiferromagnetic coupling between the two iron(III) centers. The complex has off set face-to-face π-π interactions between the dimers, and possesses a supramolecular network structure. The magnetic susceptibility of 1 can be fit with g= 2, S=5/2, and J= -220 cm^{-1}. These indicate that very strong antiferromagnetic interactions occur via the oxo bridge within the Fe(III) dimer[1] and weak antiferromagnetic interactions between the dimers.
The IR and UV spectra of oxo-diiron complex was investigated by using density functional theory (DFT) and time-dependent DFT (TDDFT) methods[2]. The theoretical study are devoted to the analysis of the interesting supramolecular assemblies in the solid state of the structures, paying special attention to lp−π and π−π interactions involving the 1,10-phenanthroline moiety.

Keywords: Dinuclear iron (III) complexes; Magnetic properties; DFT; TD-DFT.

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Synthesis, crystal Structure and Spectroscopic Studies of Noncentrosymmetric (S)nicotiniumtrichloridozincate monohydrate Complex

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Transition metal complexes play a very important role in many areas of chemistry and biology, such as catalysis [1], medicinal chemistry, photochemistry and electrochemistry [2]. Herein, we report the crystal structure and the spectroscopic characterization of the novel noncentrosymmetric (S)nicotiniumtrichloridozincate monohydrate complex. The atomic arrangement can be described as built up by chains of $ZnNCl_3$ tetrahedra and water molecules interconnected via O-H…Cl hydrogen bonds and spreading along the c-axis at y = n/2. (Fig. 1). The organic entities are inserted between these chains through C-H…Cl and N-H…Cl hydrogen bonds (Fig. 2). The ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray structure. The vibrational absorption bands were identified by infrared spectroscopy. DFT calculations allowed the attribution of the NMR peaks.

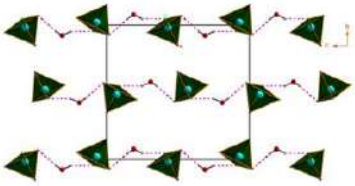

Fig.1 Projection along the *a*-axis showing the chains of $ZnCl_3$ entities and water molecules in the crystal of the title compound.

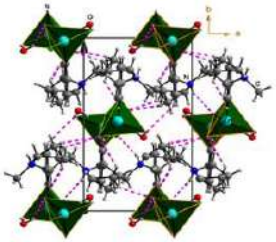

Fig. 2 Projection along the *c*-axis of the crystal packing of the title compound.

Keywords: Trichloridozincate; X-ray diffraction; NMR; DFT calculations.

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Synthesis, structural characterization and spectroscopic studies of cadmium (II) chloride complex with 4-hydroxy-1-methylpiperidine

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Heterocyclic organic compounds with nitrogen atom, which have basic properties, play important roles as cyclic components in the industrial field. They are also used in manufacturing pharmaceuticals [1]. Moreover, the 4-substituted-1-methylpiperidine derivatives, especially with the OH group, reveal interesting antimicrobial properties [2]. We report here the synthesis and the characterization of a new halide complex with 4-hydroxy-1-methylpiperidine**.** The atomic arrangement can be described as built up by an anionic framework, formed by edge sharing $CdCl_6$ and $CdCl_5O$ octahedral linear chains spreading along the *a*-axis (Fig.1). These chains are interconnected by water molecules via O-H…Cl and O-H…O hydrogen bonds to form layers parallel to the (a, b-c) plane (Fig.2). The organic cations are inserted between layers through C-H…Cl hydrogen bonds (Fig. 1). The ^{13}C and ^{15}N CP-MAS NMR spectra are in agreement with the X-ray structure. The vibrational absorption bands were identified by infrared spectroscopy. DFT calculations allowed the attribution of the NMR peaks and the IR bands.

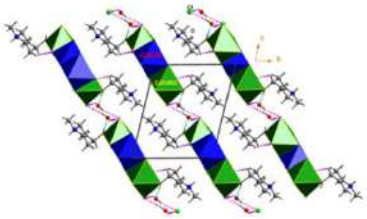

Figure.1. Projection along the *a*-axis of the crystal packing of the title compound.

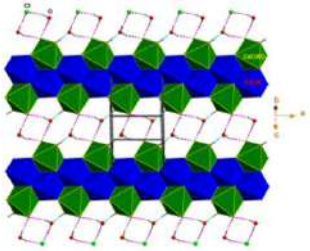

Figure.2. View of an inorganic layer parallel to (a, b-c) plane in the title compound.

Keywords: 4-Hydroxy-1-methylpiperidine; hexachloridocadmate; X-ray diffraction; NMR; DFT calculations.

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Synthesis and characterization of a new chromium phosphate $NaCuCr_2(PO_4)_3$

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The present work reports the synthesis, X-ray structure, spectroscopic investigation by IR and EPR, magnetic, specific heat and dielectirc measurements of a new chromium phosphate $NaCuCr_2(PO_4)_3$. This compound crystallizes with the unit cell parameters: a = 10.410(1) Å; b = 13.102(1) Å; c = 6.302(1) Å of the orthorhombic space group Imma. Its structure, belonging to the α-$CrPO_4$ type, features a three-dimensional $[CuCr_2(PO_4)_3]^-_\infty$ anionic framework with interesting tunnels where the Na^+ ions are located (figure 1). This framework consists of two building blocks: (1) sheets parallel to the (b,c) plane and constructed by M_2O_{10} (M = 0.5 Cu + 0.5 Cr) binuclear units, corner-linked by PO_4 tetrahedra, and (2) chains running parallel to the b axis, formed by CrO_6 octahedra, alternating with PO_4 tetrahedra.

Spectroscopic investigation by IR and EPR gives clear evidence of the occurrence of two crystallographically distinct phosphorus sites and confirms the octahedral environment of the Cr^{3+} ions. The magnetic and specific heat show two magnetic transition at low temperature. the dielectric properties are discussed.

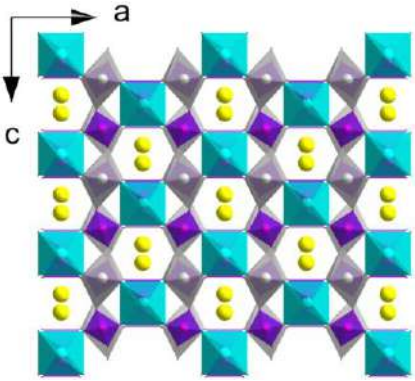

Figure 1: A projection along the [010] direction of the $NaCoCr_2(PO_4)_3$ structure.

DESİGN AND SYNTHESİS OF A SET OF HYDROPHOBİC PHTHALOCYANİNES FOR SAR İNVESTİGATİONS OF THE PHOTODYNAMİCTHERAPY EFFİCİENCY

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Vefa Ahsen[b], Fabienne Dumoulin[b]

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The treatment of cancer with photodynamic therapy (PDT) based on the combination of photosensitizing molecules (Ps) able to concentrate in tumor cells, and a focused light suitable wavelength (dependent on the Ps), exciting molecular oxygen into its singlet form. This will be the combination of these three factors which will specifically target tumor tissue and to destroy them. Phthalocyanines are among the most promising photosensitizers to be developed.
Even if most of the chemists attempt to prepare water-soluble biocompatible phthalocyanines[1], several approved photosensitizers are hydrophobic, such as hypericin[2] and Foscan.[3] A few hydrophobic phthalocyanines have been recently reported as well.[4]
In these works, a set of hydrophobic phthalocyanines for SAR investigations of their photodynamic therapy efficiency has been designed. Their design rational as well as their synthesis and characterizations will be presented.

Keywords: Phthalocyanine, hydrophobic, SAR structure-activity relationships, photodynamic therapy of cancer.

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A Novel Organic–Inorganic Hybrid Compound Based On Anderson-Type Polyoxometalates And Anthranilamide

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A new organic-inorganic hybrid material, $(C_7H_9N_2O)_2[Al(OH)_7Mo_6O_{17}].12H_2O$**(1)**, was prepared and characterized by elemental analysis, IR and UV–visible spectroscopies, TG–DTA analysis and single-crystal X-ray diffraction**.** The title compound crystallizes in the triclinic system, space group P-1 with a=10,237(2)Å, b=10,415(1)Å, c=10,545(1)Å, α=87,27(1)° β=102,17(1)°, γ=95,24(1)°, Z =1.

Single-crystal X-ray analysis shows that the compound**(1)** possesses a 3D framework, which is built up of Anderson polyoxoanions, organic cations and water molecules; the extensive net hydrogen bonds between cations and anions contribute to the crystal packing. Furthermore, the luminescent property of **(1)** in the solid state was investigated.

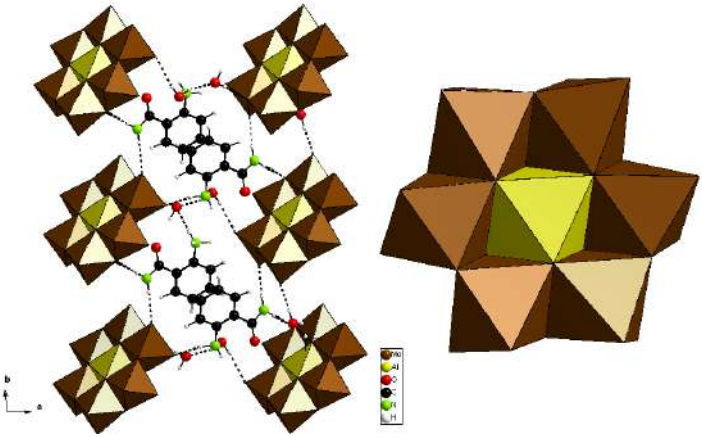

Fig. View of the three-dimensional network of the compound along the c-axis.

Synthesis and characterization of $CaCu_{3-x}Ni_xTi_4O_{12}$ by solid-State reaction

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Solid solution of the perovskite type compounds with $CaCu_{3-x}Ni_xTiO_3$ formula (x=0, 0.025, 0.05, 0.075, 0.1, 0.2, 0.3, 0.4 and 0.5) were prepared by a conventional solid state reaction method. The samples were then calcined in air at 850, 900, 950 and 1000°C for 7, 10 and 12 h. Undoped and Ni-doped samples were sintered at 1100°C for 12h. The influence of Ni additive on microstructure, dielectric and Raman properties were studied.

X –ray diffraction results showed that cubic structure does not change on doping with nickel in all compositions. The dielectric properties of sintered ceramics were investigated in wide range frequency 1 kHz-1 MHz at room temperature. The measurement of the dielectric properties at room temperature revealed higher dielectric constants at all frequency ranges [1]. But its origin is not fully understood up to now. Several theories to explain this behavior have been proposed. In addition the dielectric constant in CCNTO strongly depends on the grain size [2].

The microstructure indicates that the average grain size is different and depends on the concentration of Ni. There is great difference between the samples owing to the microstructure. Somes compositions exhibit exaggerated grain sizes.

Keywords: $CaCu_3Ti_4O_{12}$, Dielectric properties, Giant dielectric constant, Microstructures, CCNTO

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Synthesis and Bio-Physico-Chemical Characterization of Decavanadate Cage-like Cluster Templated by Organic Counter Cation

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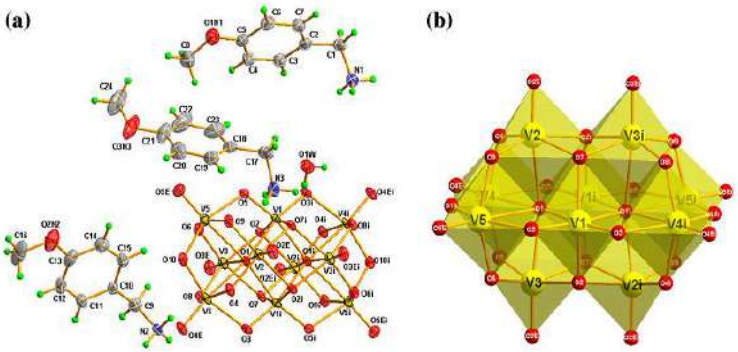
A novel Decavanadate cluster decorated with organic cation [4-$(CH_3O)C_6H_4CH_2NH_3]_6V_{10}O_{28}.2H_2O$ (*Abbr*. $4MBAV_{10}$) has been synthesized and characterized by powder and single-crystal XRD methods, elemental analysis, IR, UV-Vis, fluorescence and 1H, 13C-NMR spectroscopies. X-Ray diffraction analyses reveal that 4MBAV10 crystallizes in the monoclinic space group $P2_1/c$ with a = 10.032 (9) Å, b = 19.066 (2) Å, c = 17.9648 (15) Å and β = 93.909 (7)°. Its crystal structure consists of inorganic layers $[V_{10}O_{28}(H_2O)_4]^{6n-}$ stacked parallel to the (a, b) plane interconnected by means organic cations through H-bonds, Van der Waals and electrostatic interactions giving rise to 3D supramolecular assembly. Moreover, electronic, optical and biological properties of $4MBAV_{10}$ are also reported.



(a) View of the title compound with displacement ellipsoids drawn at the 30 % probability level. H atoms are represented as small spheres of arbitrary radii. Symmetry code: (i) 1 – x, 1 – y, 1 – z,
(b) Polyhedral view of the decavanadate {V10} cluster showing the {VO6} units.

Key words: Decavanadate, Synthesis, Single-crystal XRD, Fluorescence, Antibacterial activity.

Structural characterization, vibrational spectra, DFT investigation and Hirshfeld surface analysis of a new non-centrosymmetric hybrid: $[C_6H_{10}(NH_3)_2]_3CoCl_4.4Cl.3H_2O$

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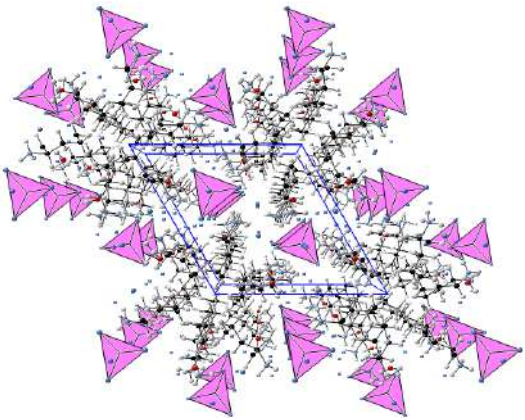
Cobalt-chloride type zero-dimensional (0-D) organic/inorganic hybrid compound $[C_6H_{10}(NH_3)_2]_3CoCl_4.4Cl.3H_2O$ was synthesized and characterized by single-crystal X-ray diffraction. It crystallizes in the hexagonal space group $P6_3$. The inorganic part of this compound is formed by isolated chlorides and isolated tetrahedral, between them, the organic part is inserted. The cohesion is performed via hydrogen bonding network. Experimentally observed spectral data (FT-TR and FT-Raman) were compared with the spectral data obtained by DFT/B3LYP method using 6-31+g(d,p) basis set. Good agreement has been found between the calculated results and the experimental data. Hirshfeld surface analysis for visually analyzing intermolecular interactions in crystal structures employing molecular surface contours and (2D) fingerprint plots have been used to examine molecular shapes. Crystal structure analysis supported with the Hirshfeld surface and fingerprint plots enabled the identification of the significant intermolecular interactions.



Perspective view of $[C_6H_{10}(NH_3)_2]_3CoCl_4.4Cl.3H_2O$ structure

Bis(4-aminopyridinium) dichromate (VI): Synthesis, crystal structure and spectroscopy studies

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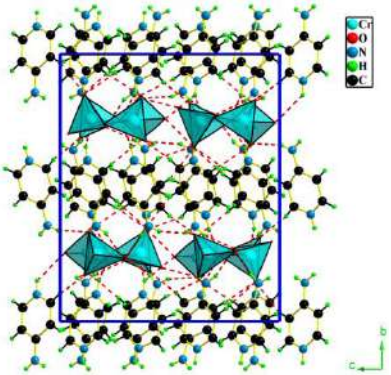
The structure of bis(4-aminopyridinium) dichromate(VI) $[C_5H_7N_2]_2Cr_2O_7$ [1] was determined from X-ray diffraction data. The compound crystallizes in the monoclinic system (space group $P2_1/c$) with the lattice parameters: a = 13.8505(4)Å, b = 16.2486(4)Å, c = 15.2586(4)Å, β = 118.923 (2)°, V = 3005.65 (14) $Å^3$ and Z = 8. The structure was solved with 6868 independent reflections with R = 0.035 and Rw = 0.097. The crystal structure consists of discrete dichromate anions with an eclipsed conformation stacked in layers parallel to (010) at y = 1/4 and y = 3/4. These layers are linked via 4-aminopyridinium cations by N-H…O and weak C-H…O hydrogen bonds, forming a three-dimensional supramolecular network. In addition, π-π interactions are present in this structure; the shortest distance separating mean planes through 4-aminopyridinium cations is 3.679 (6) Å. Crystal structure, thermal Analysis and spectroscopic studies are reported for the bis(4-aminopyridinium) dichromate(VI).

Key words:
Dichromate (VI); Single crystal X-ray; IR Spectroscopy; UV-visible spectroscopy; thermal Analysis.



Projection of $[C_5H_7N_2]_2Cr_2O_7$ along the *a* axis.

Reference:

[1] S. Trabelsi, T. Roisnel, H. Dhaouadi & H. Marouani. (2014) *Acta Cryst.* **E70**, m263-m264.

Properties of polyethylene high density/polystyrene nanocomposites

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The incorporation of nonoclay into polymers can improve the thermal stability and improved mechanical properties. This work is focuses on the study of thermoplastic nanocomposites based on Polyethylene high density/Polystyrene blends compatibilized with SBS-g-MA and reinforced with nanoclay. All PEhd/PS/SBS-g-MA/nanoclay formulations were prepared by using internal mixer and single screw extruder followed by injection molding. Maleic anhydride styrene-butadiene-styrene (SBS-g-MA) was used as the compatibilizer and the nanoclay content was varied between 0 - 7 wt percent. The mechanical and thermal properties of PEhd/PS nanocomposites were examined.

Keywords: Compatibilization, PEhd / PS, nanocomposite nanoclay / polymer, polymer blend.

References

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$(C_5H_{14}N_2)_3Fe_4Cl_{18}$: Crystal structure and magnetic properties

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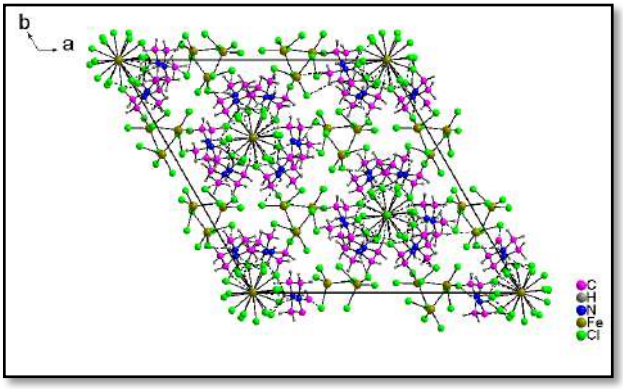
During the past few decades, self-assembly of organic and inorganic molecules has attracted much attention in chemistry, physics and material science owing to their abilities to combine the desired properties of organic and inorganic compounds, so as to exhibit some interesting crystal structures and special properties, such as second order nonlinear optical (NLO) response, magnetism, and even multifunctional properties and their technologic applications. In the present investigation, we report the synthesis, crystal structure and magnetic properties of the title compound, tris 1-methylpiperazinediium octadecachlorotetraferrate (III)



Synthesis, crystal structure, physico-chemical characterization and dielectric properties of a new organic material 1-benzhydrylpiperazinium maleate

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A new single crystal of organic material, 1-benzhydrylpiperazinium maleate $(C_{17}H_{22}N_2)(C_4H_3O_4)_2$, was synthesized and characterized by single-crystal X-ray diffraction. It crystallizes in the monoclinic space group $P2_1/c$. The asymmetric unit is composed by two maleate anions and one 1-benzhydrylpiperazinium dication. In the crystal structure, the molecular aggregations are stabilized through a two-dimensional hydrogen bonding pattern. The grown crystal was characterized by FT-IR which was recorded in the 4000-400 cm^{-1}. The placement of carbons was determined using ^{13}C NMR analysis. The optical band gap was calculated and found to be 4.14 eV. Different molecular motions are separated via dielectric relaxation spectroscopy. The (AC) electrical conduction and the impedance spectroscopy studies, reported in the sample, reveal that the conduction in the material is due to a hopping process.

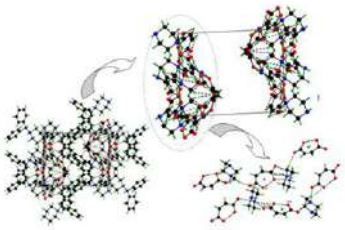

Fig. 1 View of the crystal packing of the title compound showing the hydrogen – bonding interactions between the two tartrate anions A and B

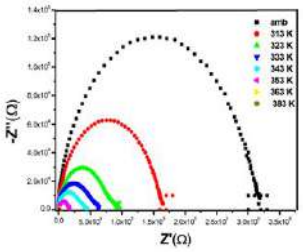

Fig. 2 Complex impedance plot as a function of temperature of the title compound

Key words: X-Ray diffraction, IR spectroscopy, NMR spectroscopy, electrical conduction, Impedance spectroscopy, Dielectric properties.

SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF β –TYPE OCTAMOLYBDATE: $(C_4H_7N_2)_2(H_3O)_2[\beta\text{-}Mo_8O_{26}].2H_2O$.

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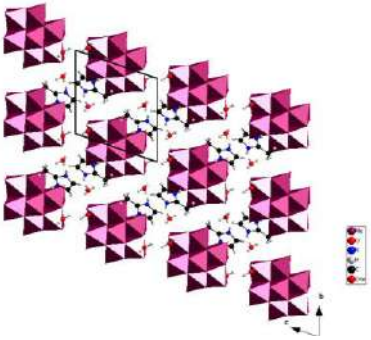
Polyoxometalates (POMs) as early-transition-metal oxide clusters, have received much attention in fields such as catalysis, ion exchange, electrochemistry, magnetism and medicine for their enormous variety of structures and unique properties **[1-4]**.
Recently polyoxomolybdates containing organic molecules have received considerable attention because of their use as models of the interactions between organic substrates and catalytic metal oxide surfaces in heterogeneous catalysis employing solid molybdenum oxides. That's why a new organic–inorganic hybrid material based on isopolyoxomolybdate formulated as $(C_4H_7N_2)_2(H_3O)_2[\beta\text{-}Mo_8O_{26}].2H_2O$ (1) has been isolated by conventional solution method and structurally characterized by single-crystal X ray diffraction methods, IR spectroscopy, UV-Vis absorption, thermogravimetric analysis and cyclic voltammetry measurements. Compound (1) crystallizes in the Triclinic system, space group P1 with unit a = 8.379 (3) Å, b = 10.200 (5) Å, c =10.861 (5) Å, α = 68.27° (3), β = 71.61° (2), ɤ = 78.40° (3), V = 814.5 (6) Å3, Z = 2. The crystal structure of (1) is built up from an octamolybdate $[\beta\text{-}Mo_8O_{26}]^{4-}$ clusters connected through hydrogen-bonding interactions into a three-dimensional supramolecular network.



Packing view of compound (2) along the a axis, showing the arrangement of the $[Mo_8O_{26}]^{4-}$ clusters, $[C_4H_7N_2]^+$, H_3O^+cations and the lattice water molecules in the crystal structure.

Keywords: Organic–inorganic hybrid materials, β-octamolybdate anion, 2-méthylimidazole.

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ACTION OF HEAT TREATMENTS ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AN ALUMINUM FOUNDRY ALLOY

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The aim of this work is to study the microstructural and mechanical properties of an aluminum alloy AS9U3, and follow its evolution during thermal treatments. Then, equal channel angular extrusion (ECAE) was used as a tool of sever plastic deformation. For this purpose, optical microscopy, X-ray diffraction (XRD), differential scanning calorimetry (DSC), Vickers hardness and compression tests were used to characterize the alloy. Results were appreciated and can be listed as follows:

Metallographic study of the alloy shows that after aging up to 400 °C, precipitation sequence took place as follows: $\alpha \rightarrow$ GP $\rightarrow \theta'' \rightarrow$ Phase $\theta' \rightarrow \theta$ phase (Al_2Cu).

The X-ray diffraction shows the appearance of fundamental peaks of aluminum and silicon, and small and fine peaks related to the second phase (Al_2 Cu).

DSC analysis reveals the appearance of different peaks (exothermic and endothermic) which are related to the formation and dissolution of precipitates.

Mechanical tests show that hardness increases at temperatures about 150°C (coherent precipitate) and it gradually decreases beyond 150°C (incoherent precipitate). These results are validated by compression tests which were consistent with the results of DSC and Vickers hardness.

The results obtained after deformation by ECAE show a cracking of the material at the first pass in homogenized and aged at 400°C states.

Keywords: Aluminum alloy, microstructure, mechanical properties, ECAE.

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