SCIENCE AND ENGINEERING OF POLYMERIC MATERIALS

SEPM 2014

Organized by the

Société Chimique de Tunisie

- Tunisian Chemical Society -

MARCH, 17th – 19th 2014

Le Royal Hotel - Yasmine Hammamet - Tunisia

Abstracts of Lectures and Communications
Participants' List

Short program of the SEPM 2014

Monday, March 17th 2014	
09.00 - 09.30	**Opening Ceremony**
09.30 - 10.10	**Plenary Lecture 1** Yusuf YAGCI *Istanbul Technical University - Faculty of Science and Letters - Turkey*
10.10 - 10.50	**Plenary Lecture 2** Alessandro GANDINI *University of Aveiro –Chemistry Département - Portugal*
10.50 - 11.20	**Coffee break**
11.20 - 12.35	**Oral Communications - First Session OC1 - OC5**
13.00	***Lunch***
14.30 - 15.00	**Key Note 1** Alain FRADET *Université Pierre et Marie Curie – Paris VI – France*
15.00 - 15.30	**Key Note 2** Mohamed M. CHEHIMI *Université Paris Diderot – CNRS – ITODYS – France*
15.30 - 16.45	**Oral Communications - Second Session OC6 - OC10**
16.45 - 17.15	**Coffee break**
17.15 - 17.45	**Key Note 3** Abderrahim MAAZOUZ *INSA, Département GMC, Lyon – France*
17.45 - 18.45	**Poster Session 1 (P 1 - P 37)**
19.30	***Dinner***
Tuesday, March 18th 2014	
08.30 - 09.10	**Plenary Lecture 3** Christophe DETREMBLEUR *Université de Liège – Fac. des Sciences – CERM – Belgique*
09.10 - 09.50	**Plenary Lecture 4** Philippe DUBOIS *University of Mons – SMPC - Belgique*
09.50 - 10.50	**Coffee break + Poster Session 2 (P 38 - P 74)**
10.50 - 11.20	**Key Note 4** Eric DROCKENMULLER *Université Claude Bernard – Lyon I – IMP – France*
11.20 - 12.50	**Oral Communications - Third Session OC11 - OC16**
13.00	***Lunch***
14.00	***Excursion to some touristic area***
20.30	***Gala Dinner***
Wednesday, March 19th 2014	
08.30 - 09.10	**Plenary Lecture 5** Daniel TATON *Université de Bordeaux – LCPO - France*
09.10 - 09.50	**Plenary Lecture 6** Philippe CASSAGNAU *Université Claude Bernard – Lyon I – IMP – France*
09.50 - 10.50	**Poster Session 3 (P 75 - P 111)**
10.50 - 11.20	**Coffee break**
11.20 - 12.20	**Oral Communications - Fourth Session OC17 - OC20**
12.20 - 12.50	**Key Note 5** Sami BOUFI *Université de Sfax - Faculté des Sciences de Sfax - Tunisie*
12.50	**Awards distribution for the 3 top poster communications and closing ceremony**
13.00	***Lunch and leaving the hotel***

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

FOREWORD

On behalf of the Chemical Society of Tunisia (Société Chimique de Tunisie - SCT), it is our great pleasure to welcome you to the international conference on Science and Engineering of Polymeric Materials (SEPM 2014).

This is the first time ever that the SCT organizes an International Conference fully devoted to polymeric materials. Through this event, we do hope to establish a tradition of gathering the polymer science and engineering communities: polymer experts, young researchers, students and SMEs and industries from Tunisia and the rest of the world.

SEPM covers many aspects of polymer science and engineering going from pure synthetic aspects to innovative (bio)polymer-based materials and devices.

We do hope that SEPM 2014 will be an exciting forum to discuss the latest advances in both polymer science and engineering research and an excellent opportunity for cooperation and networking.

For the delegates who visit Tunisia for the first time, we kept room for social events, and we do hope you will have a flavour of our over 20 century culture and heritage.

Many thanks for all participants, without them this event would not have been possible.

Many thanks too for all invited speakers who firmly accepted to contribute lectures at this event.

Last but not least, we warmly thank all colleagues and friends, members of the organizing committee who did a fabulous job organizing the Conference and make everything possible for its success.

Pr Hatem Ben Romdhane
Chairman
On behalf of the SCT

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Science and Engineering of Polymeric Materials

Yasmine Hammamet, March, 17th - 19th 2014

SEPM 2014's Program

	Sunday, March 16th 2014
16.00	**Welcoming the participants**
19.30	**Dinner**

<table>
<tr><th colspan="5">Monday, March 17th 2014 (Morning)</th></tr>
<tr><td>09.00 - 09.30</td><td colspan="4">Opening Ceremony of the SEPM 2014</td></tr>
<tr><td>09.30 - 10.10</td><td colspan="4">Plenary Lecture 1 Pr Yusuf YAGCI - Introduced by Pr Hatem Ben Romdhane
Istanbul Technical University - Faculty of Science and Letters - TURKEY
New photochemical methods for macromolecular synthesis</td></tr>
<tr><td>10.10 - 10.50</td><td colspan="4">Plenary Lecture 2 Pr Alessandro GANDINI - Introduced by Pr Sami Boufi
University of Aveiro –Chemistry Département - Portugal
Marriage of plant oils and furans to deliver new polymers through click chemistry</td></tr>
<tr><td>10.50 - 11.20</td><td colspan="4">Coffee break</td></tr>
<tr><td colspan="5">Oral Communications - First Session</td></tr>
<tr><td></td><td colspan="2">Room A - Chairperson : Mustapha Majdoub</td><td colspan="2">Room B - Chairperson : Philippe Dubois</td></tr>
<tr><td></td><td>Com.</td><td>communicating</td><td>Com.</td><td>communicating</td></tr>
<tr><td>11.20 - 11.35</td><td>OC-01 A</td><td>Safa Teka</td><td>OC-01 B</td><td>Ilhem Naghmouchi</td></tr>
<tr><td>11.35 - 11.50</td><td>OC-02 A</td><td>Paul Coupillaud</td><td>OC-02 B</td><td>Benjamin Carbonnier</td></tr>
<tr><td>11.50 - 12.05</td><td>OC-03 A</td><td>Imen Abdelhedi</td><td>OC-03 B</td><td>Imène Bekri</td></tr>
<tr><td>12.05 - 12.20</td><td>OC-04 A</td><td>Anthony Kermagoret</td><td>OC-04 B</td><td>Randa Ahmed</td></tr>
<tr><td>12.20 - 12.35</td><td>OC-05 A</td><td>Omer Suat Taskin</td><td>OC-05 B</td><td>Khouloud Jlassi</td></tr>
<tr><td>13.00</td><td colspan="4">Lunch</td></tr>
</table>

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<table>
<tr><th colspan="5">Monday, March 17th 2014 (Afternoon)</th></tr>
<tr><td>14.30 - 15.00</td><td colspan="4">Key Note 1 Pr Alain FRADET - Introduced by Pr Souhir Abid El Gharbi
Université Pierre et Marie Curie – Paris VI – France
Polycondensations in ionic liquids</td></tr>
<tr><td>15.00 - 15.30</td><td colspan="4">Key Note 2 Pr Mohamed M. CHEHIMI - Introduced by Pr Benjamin Carbonnier
Université Paris Diderot – CNRS – ITODYS – France
Surface chemistry of aryl diazonium salts. Historical development and applications in polymer science and engineering</td></tr>
<tr><td colspan="5">Oral Communications - Second Session</td></tr>
<tr><td></td><td colspan="2">Room A - Chairperson : Benjamin Carbonnier</td><td colspan="2">Room B - Chairperson : Philippe Cassagnau</td></tr>
<tr><td></td><td>Com.</td><td>communicating</td><td>Com.</td><td>communicating</td></tr>
<tr><td>15.30 - 15.45</td><td>OC-06 A</td><td>Saber Chatti</td><td>OC-06 B</td><td>Ramzi Belhassen</td></tr>
<tr><td>15.45 - 16.00</td><td>OC-07 A</td><td>Marwa Abbes</td><td>OC-07 B</td><td>Karama El Fehri</td></tr>
<tr><td>16.00 - 16.15</td><td>OC-08 A</td><td>Charlène Pouech</td><td>OC-08 B</td><td>Alàa Ladhar</td></tr>
<tr><td>16.15 - 16.30</td><td>OC-9 A</td><td>Raouf Medimagh</td><td>OC-9 B</td><td>Amira Bouaziz</td></tr>
<tr><td>16.30 - 16.45</td><td>OC-10 A</td><td>Sabrine Hbaïeb Kharrat</td><td>OC-10 B</td><td>Aziz Hassen</td></tr>
<tr><td>16.45 - 17.15</td><td colspan="4">Coffee break</td></tr>
<tr><td>17.15 - 17.45</td><td colspan="4">Key Note 3 Pr Abderrahim MAAZOUZ - Introduced by Pr Mohamed Jaziri
INSA, Département GMC, Lyon – France
Improvement of blowing film extrusion stability of poly (lactic acid) and its blends</td></tr>
<tr><td>17.45 - 18.45</td><td colspan="4">Poster Session 1 (P 1 - P 37)</td></tr>
<tr><td>19.30</td><td colspan="4">Dinner</td></tr>
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Tuesday, March 18th 2014				
08.30 - 09.10	**Plenary Lecture 3** Pr Christophe DETREMBLEUR - *Introduced by Pr Alessandro Gandini* Université de Liège – Fac. des Sciences – CERM – Belgique **Green and bio-inspired processes for the functionalization of surfaces**			
09.10 - 09.50	**Plenary Lecture 4** Pr Philippe DUBOIS - *Introduced by Pr Abderrahim Maazouz* University of Mons – SMPC - Belgique **Bio-sourced lactic acid-based polymeric nanocomposites: From reactive extrusion to high performance materials**			
09.50 - 10.50	**Coffee break + Poster Session 2 (P 38 - P 74)**			
10.50 - 11.20	**Key Note 4** Pr Eric DROCKENMULLER - *Introduced by Pr Mohamed M. Chehimi* Université Claude Bernard – Lyon I – IMP – France **Poly(1,2,3-triazolium ionic liquid)s: New generation of tailored ion conducting materials**			
Oral Communications - Third Session				
	Room A - *Chairperson : Claire Mangeney*		**Room B -** *Chairperson : Saber Chatti*	
	Com.	*communicating*	*Com.*	*communicating*
11.20 - 11.35	**OC-11 A**	Chirine Ben Osman	**OC-11 B**	Nedra Azizi
11.35 - 11.50	**OC-12 A**	Jean-Victor Richard	**OC-12 B**	Chaouki Belgacem
11.50 - 12.05	**OC-13 A**	Habiba Zrida	**OC-13 B**	Abdelkader Bougarech
12.05 - 12.20	**OC-14 A**	Souha Ben Mahmoud	**OC-14 B**	Sihem Bel Haaj
12.20 - 12.35	**OC-15 A**	Tayssir Ben Ghzaïel	**OC-15 B**	Besma Berrima
12.35 - 12.50	**OC-16 A**	Balkis Ben Salem	**OC-16 B**	Rosiyah Yahia
13.00	***Lunch***			
14.00	***Afternoon off*** ***Excursion to some touristic area***			
20.30	***Gala Dinner***			

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<table>
<tr><th colspan="5">Wednesday, March 19th 2014</th></tr>
<tr><td>08.30 - 9.10</td><td colspan="4">Plenary Lecture 5 Pr Daniel TATON - Introduced by Pr Eric Drockenmuller
Université de Bordeaux – LCPO - France
Selective organocatalyzed polymerization from N-heterocyclic carbenes (NHCs)</td></tr>
<tr><td>09.10 - 9.50</td><td colspan="4">Plenary Lecture 6 Pr Philippe CASSAGNAU - Introduced by Pr Ezzeddine Srasra
Université Claude Bernard – Lyon I – IMP – France
Linear viscoelasticity of suspensions of anisotropic nano-particles</td></tr>
<tr><td>9.50 – 10.50</td><td colspan="4">Poster Session 3 (P 75 - P 111)</td></tr>
<tr><td>10.50 - 11.20</td><td colspan="4">Coffee break</td></tr>
<tr><td colspan="5">Oral Communications - Fourth Session</td></tr>
<tr><td></td><td colspan="2">Room A - Chairperson : Alain Fradet</td><td colspan="2">Room B - Chairperson : Memia Benna Zayani</td></tr>
<tr><td></td><td>Com.</td><td>communicating</td><td>Com.</td><td>communicating</td></tr>
<tr><td>11.20 - 11.35</td><td>OC-17 A</td><td>Mohamed Braiek</td><td>OC-17 B</td><td>Mohamed Ammar</td></tr>
<tr><td>11.35 - 11.50</td><td>OC-18 A</td><td>Mohamed Amin Omri</td><td>OC-18 B</td><td>Carla Vilela</td></tr>
<tr><td>11.50 - 12.05</td><td>OC-19 A</td><td>Tasnim Kossentini Kallel</td><td>OC-19 B</td><td>Hajer Hrichi</td></tr>
<tr><td>12.05 - 12.20</td><td>OC-20 A</td><td>Nabawia Mechi</td><td>OC-20 B</td><td>Haythem Bennour</td></tr>
<tr><td>12.20 - 12.50</td><td colspan="4">Key Note 5 Pr Sami BOUFI - Introduced by Pr Hatem Majdoub
Université de Sfax - Faculté des Sciences de Sfax - Tunisie
Nanocellulose: A sustainable nanoparticles with outstanding reinforcing potential</td></tr>
<tr><td>12.50</td><td colspan="4">Awards distribution for the 3 top poster communications and closing ceremony</td></tr>
<tr><td>13.00</td><td colspan="4">Lunch and leaving the hotel</td></tr>
</table>

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Speakers' CVs & Abstracts

Yusuf YAGCI

Yusuf Yagci is a full professor of Chemistry at Istanbul Technical University, Turkey with expertise in photopolymerization. He received his Masters (1977) and Ph.D. (1979) degree from Liverpool University (UK). He joined Istanbul Technical University in 1980 and between 1996 and 2001 he was the Director of the Institute of Science and Technology of Istanbul Technical University. Professor Yagci's research interests involve photoinitiated cationic and radical polymerization, polymer modification, controlled polymerization methods and their transformations, block and grafted copolymers, nanocomposites, click chemistry, and benzoxazine-based thermosets. He has published about 500 original research papers in different peer-reviewed international journals and he is the inventor and co-inventor of five patents. He has also co-authored and co-edited five books and has been invited to write over 40 chapters in different books. Professor Yagci has supervised about 85 Ph.D. and M.Sc. students. He serves as member of the editorial board of many international journals. He is the recipient of several awards including the Turkish Scientific and Technological Research Council (Tubitak) Young Investigator Award (1989), Tubitak Science Award (1994), Turkish Chemical Society Honorary Member Award (2002), Elsevier Scopus Award (2007), The Society of Polymer Science, Japan (SPSJ) International Award (2008), and the Elginkan Foundation Technology Award (2008), COMSTEC Award (2011). He acts as the Associate Member of IUPAC Polymer Division. He was a member of the Turkish Academy of Sciences (1997-2011). He organized several international meetings in Turkey and abroad, and presented many plenary and invited lectures in international conferences.

NEW PHOTOCHEMICAL METHODS FOR MACROMOLECULAR SYNTHESIS

Yusuf Yagci

Istanbul Technical Universty, Department of Chemistry, Maslak, Istanbul 34469, Turkey
e-mail: yusuf@itu.edu.tr

Initiation of various modes of polymerization processes by photochemical methods has been widely used[1] in conventional applications in coatings, adhesives, inks, printing plates, optical waveguides, and microelectronics.
In this presentation, the development of new photoinitiating systems for radical[2, 3], cationic[4] and step-growth polymerization[5, 6]will be described. Besides film forming applications, these methods can also be used in polymer syntheses. What type of polymerization mode is applicable depends on the chemical structure of the monomers used. Whilst free radicals initiates the polymerization of unsaturated polyesters and (meth)acrylic monomers, epoxy and vinyl ether based formulations are activated by a cationic mechanism. Recent relevant studies[2] were devoted to make free radical and cationic photoinitiators responsive to light with wavelengths in the near UV and visible region of the spectrum With recent advances in the use of nanomaterials like metals, metal oxides and silicates in UV curing applications, it is now possible to prepare nanocompositesfilms with enhanced physical, chemical and biological properties. In the presentation, several synthetic methodologies for the preparation of epoxy and (meth)acrylate based formulations containing clay or metal nanoparticles[7,8]will also be described. The nanoparticles are homogenously distributed in the network without macroscopic agglomeration. Applicability to both free radical and cationic systems is demonstrated. Photoinduced "Click" reactions which may potentially be used in film formation[9, 10] will also be discussed.

References

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2) M.A. Tasdelen, M. Ciftci, Y. Yagci, *Macromol. Chem. Phys*., **2012**, 213, 1391
3) M.A. Tasdelen, M. Uygun, Y. Yagci, *Macromol. Chem. Phys*., **2011**, 212, 2036
4) G. Yilmaz, B. Iskin, F. Yilmaz, Y.Yagci, *ACS Macro Letters*, **2012**, 1, 1212
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8) Y.Yagci, M.Sangermano, G.Rizza, *Chem. Commun*. **2008**, 24, 2771
9) M.A. Tasdelen, G. Yilmaz, B. Iskin, Y. Yagci, *Macromolecules*,**2012**, 45, 56
10) M.A.Tasdelen, Yagci Y, *Angew. Chem. Int. Ed*., **2013**, 52, 5930

Alessandro GANDINI

1962-65: Ph.D. in Polymer Science, Keele University, UK. ***Studies on cationic polymerisation.***
1965-67: Postdoctoral fellow at NRCC, Ottawa, Canada. ***Primary processes in photophysics and photochemistry.***
1967-68: Research officer at Brookhaven Laboratory, Upton, USA. ***Gas-phase kinetics: H/D isotope effects in photochemically generated free radicals***.
1968-76: Professor of Physical Chemistry at the University of Havana, Cuba and head of the Polymer Laboratory at the Cuban National Research Council. *Courses in Polymer Chemistry, Polymer Physics and Photochemistry*. ***Research on the kinetics and mechanisms of the polymerisation of various monomers***.
1976-78: Research officer at NRCC, Ottawa, Canada. ***Multiphoton infrared photochemistry: H/D isotope effects in the decomposition of gaseous substrates.***
1978-2003: Professor of Chemistry at the National Polytechnic Institute of Grenoble, France. *Courses inPhysical Chemistry, Photochemistry, Polymer Chemistry and Physics, Materials Science at the Engineering Schools ofPapermaking and Printing*, *Electrochemistry, Physics* and *Mechanics*. Head of the Polymer Laboratory. ***Research on thesynthesis, characterization and properties of new polymers and composites, mostly derived from the biomass.***
2004-2005: ***Invited Professor*** at the São Carlos Chemistry Institute, University of São Paulo, Brazil. *Postgraduate courses on Polymer Materials and on Surfaces and Interfaces*. ***Research on thechemical modification of cellulose fibres and starch***.
2005-2011: Emeritus Professor, Chemistry Department, University of Aveiro, Portugal. *Postgraduate courses in Polymer Science, Polymers from Renewable Resources, Photochemistry and Photophysics and the Physical Chemistry of Surfaces and interfaces.* ***Coordinator of an industrial-academic research platform on polymersfrom renewable resources: cellulose fibres, starch, lignins, chitosan, suberin, furans and vegetable oils.***
2011-2013: Invited Professor at the São Carlos Institute of Chemistry, University of São Paulo, Brazil. *Postgraduate courses on Polymers from Renewable Resources.* ***Research on new polymers based on furans, vegetable oils, starch and cellulose.***
Present position: Research supervisor, Pagora, Grenoble Polytechnic, France
Visiting Professor at the Universities of Essex, UK, Sao Carlos, Brazil (*yearly* 1993-2003), Aveiro, Portugal, Pavia, Italy, Sfax, Tunisia (*yearly* 1988-2002) and Girona, Spain. ***Visiting scientist*** at NRCC, Canada. Courses on ***"Polymers from renewable resources"*** in a dozen Universities and Research Centres throughout the world.
>**80***PhD Theses*, >**440***publications*, including ***3*** *books*, >***60*** *monographs* (cited >**7000** times) and >**20** patents. >**470***communications* at scientific symposia, including >**100** *invited or plenary lectures*. >**140***invited seminars* in Universities and Research Centres in >**30** countries.
Fellow of the *International Academy of Wood Science* (elected in 1994) and of several scientific societies, including the *Royal Society of Chemistry* and the *ACS*.

Honorary Doctor of the *Saint Petersburg Forestry Academy (1995)*
Honorary Doctor of *Havana University (2000)*
Invested with the Ermine Decoration of Grenoble National Polytechnic Institute (1995)

MARRIAGE OF PLANT OILS AND FURANS TO DELIVER NEW POLYMERS THROUGH CLICK CHEMISTRY

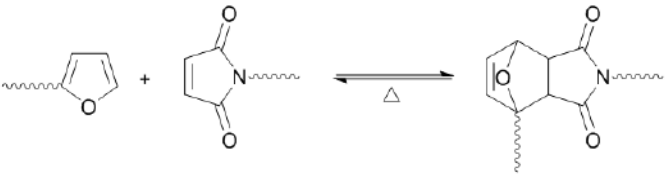
Alessandro Gandini

Grenoble INP, Ecole Internationale Du Papier,
de la Communication Imprimée et des Biomatériaux
E-mail:agandini@iqsc.usp.br

This lecture discusses recent work carried out both at Aveiro University and at São Paulo University in São Carlos with the valuable contribution of brilliant researchers during their postgraduate and postdoctoral experience, namely Carla Vilela and LetiziaCriciani in Aveiro and TalitaLacerda in São Carlos.

The working hypothesis of this investigation is to couple two types of precursors derived from renewable resources, viz. vegetable oils and furan derivatives, to synthesize monomers incorporating two classical complemenraty Diels-Alder (DA) moieties in the form of a diene structure (the furan heterocycle) and a dienophile counterpart (the maleimide ring) in different numbers and combinations. Some of these compounds were prepared using the thiol-ene click reaction, others called upon aminolysis, epoxy-amino condensations and other more conventional interactions.

The linear and branching polycondensations of these monomers was conducted at 60-65 °C thanks to the DA coupling of their complementary functions (another click chemistry mechanism) and were followed by different spectroscopic techniques. The ensuing materials received a thorough characterization before being submitted to a controlled depolymerization through the retro-DA reaction, carried out at 100-110 °C, which ultimately regenerated the starting monomers. The polymerization/depolymerization cycle could be repeated several times because of the absence of detectable side reactions.



Apart from the major interest of exploiting two different renewable resources for achieving these goals, the preparation of thermoplastic and crosslinked materials exhibiting such original features as self-healing and recyclability, represents the other notable achievement of this ongoing research venture.

A. Gandini, *Progr. Polym. Sci.* **2013**, *38*, 1.

Alain FRADET

Alain Fradet obtained his docteur-ingénieur degree in polymer chemistry in 1976 from the University of Rouen. He then pursued his research work at Université P. et M. Curie, Paris (UPMC) and defended his "doctorat ès sciences physiques" thesis in 1980, specializing in chemistry, catalysis and kinetics of condensation polymer synthesis. The kinetics and catalysis of bulk polyesterification by metal alkoxides was more specifically investigated. As a postdoctoral fellow, he then studied the synthesis and the liquid-crystalline properties of various thermotropic aromatic poly(polyester-block-polyether)s (University of Marburg, Germany).

He was appointed as CNRS researcher in 1978 (UPMC & CNRS) and was promoted Directeur de Recherche in 1994. In 1996 he became full professor at UPMC (Laboratoire de Chimie des Polymères), where he conducted research on the chemistry of condensation polymers, including: (i) the study of interchange reactions and ring-chain equilibrium in condensation polymers such as polyesters and polyamides, (ii) the synthesis of block-copolymers by bulk reactions between dicarboxy, diamino or dihydroxy oligomers and chain-coupling agents and (iii) the synthesis of various hyperbranched polymers by polycondensation of AB2-type monomers. Using a recursive probability approach, he also proposed and experimentally verified relationships between conversion, average molar masses, molar-mass dispersity and degree of branching for complex hyperbranched polymers.

His current work focuses on the synthesis and properties of biobased and biodegradable polymers and on the study of condensation polymerizations in ionic liquids. Alain Fradet has authored or co-authored more than 120 research papers and patents, and has given more than 100 national and international lectures in scientific meetings.

POLYCONDENSATIONS IN IONIC LIQUIDS

Alain FRADET, Shaodong ZHANG, Hervé LEFEBVRE, Martine TESSIER

Sorbonne-Universités UPMC-Paris-6, IPCM-UMR 8232, Chimie des Polymères, 4 Place Jussieu, 75252 Paris Cedex 05 (France)

The unique combination of properties of ionic liquids, i.e. negligible vapor pressure, high thermal and chemical stabilities, non-flammability and reusability, makes them very interesting as solvents for organic and polymer synthesis. Some polycondensation polymers, e.g. aromatic polyamides, polyesters and polybenzimidazoles, were prepared in ionic liquids, using simple direct reactions from non-activated monomers. Reaction rates and molar masses were generally higher than obtained by reacting the same monomers in conventional solvents. As Brønsted acid ionic liquids (BAILs) have been reported to act as solvents and efficient acid catalysts in esterification reactions, we undertook a study of their use for polyesterification reactions. We first investigated the polyesterification of 12-hydroxydodecanoic acid in various BAILs based on the 4-(3'-butyl-1'-imidazolio)-1-butanesulfonic acid cation. High molar mass polyesters were obtained at low to moderate temperature (90-130 °C) in short reaction time (30-60 min) and at atmospheric pressure,[1,2] i.e. in much milder reaction conditions than conventional ones (5-7 h, 180-220°C, vacuum). The method was then successfully applied to the synthesis of a series of linear and hyperbranched aliphatic polyesters.[3]

In some cases, a side reaction leading to hydroxy end-group etherification was observed. This side reaction was subsequently applied to develop a unique and very easy synthesis of long-chain aliphatic polyethers by direct polyetherification of diols with 7-12 methylene units.[4]

Ionic liquids may also be used as new reaction media for the synthesis of unusual polyamides: Poly(β-alanine) was synthesized by direct polyamidation of β-alanine in imidazolium-based ionic liquids, with triphenylphosphite as condensing agent. Similarly, copolymers of 12-aminododecanoic acid and various natural α-aminoacids were prepared in ionic liquids. The method was also applied to the direct synthesis of polypeptides, for instance poly(L-valine) and poly(L-leucine).[5] This approach is far simpler than the conventional anionic polymerization of N-carboxyanhydrides.

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Improvement of Blowing film Extrusion stability of poly (lactic acid) and its blends

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This work deals with the study of the blowing extrusion process of Poly (lactic acid) and its blends, which mainly present a poor shear and elongation viscosities. In order to enhance their processability, two main routes were selected: (i) the modification of its structure, rheological and thermo-mechanical properties by adding a multi-functional epoxide molecules (Joncryl ADR® - 4368) acting as a chain extension/branching agent and (ii) blending it with other ductile thermoplastic biopolymer such as the poly (butylene adipate-co-terephtalate) (PBAT). The effect of the improved rheological properties on the stability map of their blends has been also investigated. Based on the obtained results, a great enhancement of the blowing processing windows of modified PLA with Joncryl was demonstrated. The extension chain/ branching balance and the nucleating effects of the multifunctional epoxide have been analyzed. Higher Blow Up Ratio (BUR) and Take Up Ratio (TUR) values were obtained. Besides, the incorporation of PBAT into PLA matrix leads to an enlargement of the stability domain according to the improvement of their shear and elongation properties especially for the compatibilized blends. Indeed, the adding of Joncryl into PLA_PBAT matrix, the instability defects zone were reduced. Finally, the crystalline structure and the thermo-mechanical properties of blown films were characterized thanks respectively to the Differential Scanning Calorimeter (DSC) and the dynamic mechanical thermal analysis (DMTA).

Key-Words: Blown film extrusion ; Processing stability; Viscoelastic Properties ; Blends, Rheology, Structure-processing-properties relationships'.

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1995 HDR, Habilitation in Science, University Paris 7 (16/5/1995)
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• Over 200 articles • 2 edited books • 19 book chapters • 2 patents • 1 invited Gordon Conference • Over 100 oral and poster presentations • Supervision of PhD students: **17** • Over 4000 citations • H-index: 33.

• Honorary Medal from the Polymer Institute (Slovak Academy of Sciences, Slovakia) for long term international collaboration on surface and interface aspects of polypyrrole nanocomposites (2008).

PRESENT MAIN RESERACH PROJECTS

(i) Aryl diazonium as coupling agents of polymers and for surface initiated polymerization.
(ii) Electrochemical sensors based on molecularly imprinted polymer thin films.
(iii) Conductive polymer coatings for the development of flexible gas sensors.
(iv) Films, powders, latex particles and nanocomposites of conductive polymers.
(v) Clay/polymer and CNT/polymer nanocomposites.

SELECTED PUBLICATIONS RELEVANT TO THE SURFACE CHEMISTRY OF DIAZONIUM SALTS

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4. *Core/shell, protuberance-free MWCNT/polyaniline nanocomposites via interfacial chemistry of aryl diazonium salts*. A. Mekki, S. Samanta, A. Singh, Z. Salmi, R. Mahmoud, M. M. Chehimi, D. K. Aswal. Journal of Colloid and Interface Science, 418 (2014) 185–192.
5. *Design of molecularly imprinted polymer grafts with embedded gold nanoparticles through the interfacial chemistry of aryl diazonium salts. S. Gam-Derouich, S. Mahouche-Chergui, S. Truong, D. Ben Hassen-Chehimi, M. M. Chehimi*. Polymer, 52 (2011) 4463-4470.
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1. *Aryl diazonium salts : new coupling agents in polymer and surface science*. M. M. Chehimi (Ed.), Wiley-VCH, Weinheim (Germany), 2012, ISBN978-3-527-32998-4.
2. *Applied surface chemistry of nanomaterials* . M. M. Chehimi and J. Pinson (Eds.), Nova Science Publishers, Hauppauge (NY, USA), 2013, ISBN: 978-1-62808-351-4.

SURFACE CHEMISTRY OF ARYL DIAZONIUM SALTS. HISTORICAL DEVELOPMENT AND APPLICATIONS IN POLYMER SCIENCE AND ENGINEERING

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The discovery of diazotation of compounds by the German chemist Peter Griess dates back to 1858. Henceforth, aryl diazonium salts are commonly used for the synthesis of a large series of organic compounds such as azo dyes which are industrially produced. However, the study of the surface chemistry of these salts remained sparse despite its interest in modifying materials such as liquid mercury electrodes [1] or to label enzymes [2].

In 1992, Jean Pinson and co-workers described the mechanisms of the reaction between aryl diazonium salts and glassy carbon electrodes and demonstrated by XPS the existence of surface-tethered aryl groups [3]. Provided that the functional group, in para position of the diazonium, is reactive, it becomes possible to graft polymers, enzymes, catalysts, etc. Since then, the literature witnessed a quantum jump in the number of publications pertaining to the surface chemistry of aryl diazonium salts and uses thereof. The interest in using these compounds obviously lies in their ease of preparation, rapid reduction by a large range of methods and strong aryl-surface covalent bonding. The applications concern polymer coatings, molecular electronics, electrocatalysis, (bio)sensors, to name but a few, as testified by over 4000 articles [4] and one edited book [5]. Several surface processes using diazonium salts were also patented and industrial products, though not too many, are on the market (modified carbon black [6] and drug eluting stents [7]).

These extraordinary academic and industrial achievements will be summarized first, then the emphasis will be on polymer grafting to aryl diazonium-modified materials surfaces. Grafting can be achieved either *via* click chemistry or *via* a range of surface-initiated polymerization methods (radical, iniferter, anionic) on sp^2 and sp^3 carbons, metals, ceramics, clay and semi-conductors. The applications concern catalytic hybrid nanomaterials, molecularly imprinted polymer-based sensors and bioactive surfaces.

From the above, the toolbox of the polymer chemist is enriched with aryl diazonium salts which undoubtedly represent alternative coupling agents to the traditional silanes and thiols for tethering polymers to surfaces.

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[7] http://www.youtube.com/watch?v=cRPPhFWsyqI

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Christophe Detrembleur is a member of the Center for Education and Research on Macromolecules (CERM) of the University of Liege (ULg), Belgium. He did his undergraduate and graduate work at ULg. He received his PhD degree in 2001 under the supervision of Prof. Robert Jérôme on the development of new regulators for the controlled radical polymerization of (meth)acrylic monomers. He was an Invited Researcher at IBM, Almaden Research Center, San José, California, USA, under the supervision of Dr. J. Hedrick for a few months in 1998. After his PhD thesis, he joined the Research Center of Bayer AG, Leverkusen in Germanyin May 2001 as a laboratory leader. He was involved on materials synthesis and polymer processing. In January 2003, he moved to the polyurethane research division at Bayer AG, where he was in charge of the development of new high performance UV coatings. In October 2003, he became a permanent researcher at CERM under the auspices of the National Funds for Scientific Research (F.R.S.-FNRS). In October 2008, he was promoted Senior Research Associate and in October 2012, he became Research Director by the F.R.S.-FNRS.
C. Detrembleur heads a research team that is active in three main fields:
1- The development, understanding, optimization and applications of new controlled radical polymerization techniques such as *in-situ*nitroxide mediated polymerization (*in-situ* NMP) and more recently, cobalt-mediated radical polymerization (CMRP). The implementation of these processes to the synthesis of advanced functional materials in green media such as in water and in supercritical carbon dioxide is one of the recent research topics of his team.
2- Thepreparation of novel well-defined (multi)functional (co)polymers for coating applications. Besides others, C. Detrembleur is involved in the synthesis and characterization of long-lasting antibacterial coatings using bio-inspired green processes. Some of his recent works have been awarded by an ACS press release, and several interviews in specialized journals and web sites (www.laboratoryequipment.com).
3- Thepreparation of polymer/carbon nanofillers (carbon nanotubes and graphene) nanocomposites foams by the supercritical carbon dioxide technology. These materials are specifically designed to act as electromagnetic interference (EMI) absorbers, that are able to suppress the electromagnetic pollution. C. Detrembleur is also setting up routes to the chemical modification of these carbon-based nanofillers by “grafting onto” approaches.

C. Detrembleur published more than 170 peer-reviewed papers, 7 book chapters and filed 40 patents.

A full list of publications, patents, oral presentations and posters is available at http://orbi.ulg.ac.be

Green and bio-inspired processes for the functionalization of surfaces.

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Due to its exceptional properties, stainless steel (SS) is widely used in the daily life (food industry, household appliances, surgery …).[1] However, it is unable to prevent bacteria from adhering, proliferating and forming a resistant biofilm over time. Therefore, surface modification is needed for providing durable antibacterial (AB) properties. Various techniques are used for imparting these AB properties to the substrate but are most often difficult to implement at the industrial scale and/or are not environmentally friendly procedures.

In this talk, we will discuss on new routes towards the preparation of long-lasting antibacterial coatings for stainless steel using aqueous-based and bio-inspired processes. We will first report on the design of a bio-inspired glue that is required for the strong adhesion of the functional coating on the substrate, and therefore for the long-term activity of the coating.[2] Then, various routes towards the functionalization of stainless steel with antibacterial coatings will be discussed. In this section, we will emphasize the role of catechol groups present on specifically designed novel polymers[3] (1) for the grafting of biocides to the surface and (2) for the film crosslinking in mild and aqueous conditions. Beside the formation and immobilization of silver based nanoparticles as biocides in the coating,[4] we will describe how the same catechol-bearing polymers can be exploited for the grafting of antibacterial peptides[5] and antibiofilm enzymes.[6] The long-term antibacterial/antibiofilm activity of the surface will be discussed. Finally, we will focus on how the deposition procedure of the active species can be simplified to render it industrially attractive by speeding up the deposition technique on stainless steel surfaces while maintaining a high and long-term surface activity.[7]

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Philippe DUBOIS

Philippe DUBOIS is the Vice-Rector/executive Vice-President for Scientific Research at University of Mons – UMONS, Belgium.
Born in Charleroi (BE), he is full professor, Director of the Center of Innovation and Research in Materials & Polymers (CIRMAP; ca. 165 pers. from 24 different nationalities) and Vice-Chair of the UMONS Research Institute for Materials Sciences and Engineering. In in Laboratory of Polymeric and Composite Materials), he supervises ca. 50 people including 14 PhD students and 16 post-doctoral researchers (http://morris.umons.ac.be/smpc).
His main fields of research are: the synthesis and characterization of polymeric materials for applications in the biomedical domain and electric and electronic industries. His fields of interest also include the catalysis in macromolecular chemistry and polymeric nanocomposites. Dr Dubois is specialized in the production and the applications of polymeric and composite materials issuing from renewable bio-resources.
In November 2013, he had authored more than 530 peer-reviewed scientific publications (h index = 63, total number of citations >18500) and is inventor of 60 patents. He is personally ranked in the "Top 100 materials scientists of the 2000-2010 decade" by Thomson Reuters (World ranking: 18th, European ranking: 5th, Belgian ranking: 1st). He is member of the editorial board of 15 international scientific journals in the field of polymer chemistry and materials science. Among them, he is associate editor of Materials Science and Engineering: R: Reports edited by Elsevier (average Impact Factor of 18.9 for the last 5 years). He also edited 8 books and special issues of scientific journals specialized in macromolecular chemistry and polymer materials.
Philippe Dubois is also scientific director of the private Research Center Materia Nova asbl, where he supervises the R&D activities in the field of polymer nanocomposites and bioplastics. In 2011, he founded the spin-off NANO4 S.A., which upon specific industrial requests, produces masterbatches of polymer blends and nanocomposites, incl. (nano)filled bioplastics (from few kg-to-ton scale production) by (reactive) extrusion (http://www.nano4-materials.com).
Prof. Dubois is Past-President of the Belgian Royal Chemical Society and in 2010 he was elected as Academician - permanent titular member of the Royal Academy of Sciences of Belgium. In 2012, he becomes inaugural member of Europe Science Committee.

BIO-SOURCED LACTIC ACID-BASED POLYMERIC NANOCOMPOSITES: FROM REACTIVE EXTRUSION TO HIGH PERFORMANCE MATERIALS

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Polylactide (PLA) represents the frontrunner of biobased and biodegradable, finding broad applications not only for short-time applications, including food-packaging, but more and more for long-term and high-performance applications, e.g., in automotive and electronic industries. Nevertheless, achieving such high added value applications requires, at least, improving the low crystallization kinetics, the resistance-to-hydrolysis and the low Heat Deflection Temperature (HDT) of PLA-based materials.

In this contribution, we propose the straightforward and continuous synthesis of melt-processable PLA-based stereocomplexes according to a solvent-free process [1,2]. Starting namely from the corresponding monomers (D- and L-lactide), three successive reactions (ROP, chain extension and stereocomplexation) have been performed through a multi-step reactive extrusion (REx) process. REx affords a continuous and solvent-free process, which can be considered sustainable and easily adapted if the reaction rate is high enough. In the multi-step process, ROP was used to produce well-defined low molecular weight α,ω-dihydroxyl polylactide in bulk, while chain extension was subsequently performed in the presence of coupling agent and chain extender, respectively, 4,4'-diisophenylmethane diisocyanate (MDI) and 4,4'-diaminodiphenylmethane (MDA). Depending on the molecular weight of the PLA macrodiol initially produced, the content in chain-extender/coupling agent was limited to max. 5 wt% forming the hard segments spread along the PLA-based chains and increasing their overall thermal stability. Regarding the third and last step, i.e., stereocomplexation, two twin-screw extruders connected in T-shape configuration was employed, being particularly relevant in this case to obtain PLA-based stereocomplexes since the simultaneous polymerization of L- and D-lactide would have led to amorphous PLA-based materials. Interestingly, for the entire process, a single and unique catalytic system was used to promote both ROP and chain extension reactions while stereocomplexation took place. Actually, FDA-approved tin(II) octoate specifically complexed with one equivalent of triphenylphosphine added as Lewis base, i.e. $Sn(Oct)_2/PPh_3$, was studied for catalysing both chain-growth polymerization (i.e. L- and D-Lactide ROP) and the consecutive step-growth polymerization (coupling/extension reactions of macrodiols) by REx process.

Furthermore, the profile of PLA properties have been be tuned by combining PLA with well-selected nanofillers paving the way to novel biosourced materials of high added values, i.e., renewable biosourced PLA-based nanocomposites [3-7]. Different types of nanofillers (with 1, 2 or 3 nanodimension(s)) have been investigated for improving the mechanical (tensile and impact resistance), thermal (crystallinity extent and kinetics), rheological (processing ability) properties of PLA matrices and as well as other more specific properties of the related nanocomposites materials such as their gas barrier resistance, electro-conductive ability, flame resistance, UV absorbance, and antibacterial performances.

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Eric DROCKENMULLER

Eric Drockenmuller was born in 1973 in Thionville (France), and received his PhD degree in 2002 from the University of Strasbourg (France) after working on nitroxide-mediated radical polymerization. He undertook a two years postdoctoral position with Prof. C. J. Hawker (IBM Almaden Research Center, California, USA) and Prof. T. P. Russell (University of Massachusetts, Amherst, USA) working on the synthesis and functionalization of nanostructured materials. In October 2004 he was appointed assistant Professor at the University of Lyon 1 (France) and full professor in September 2011 at the same University. He is a junior member of the "Institut Universitaire de France" (IUF) since October 2010. His main interests include controlled radical polymerization techniques, step growth polymerizations, polymer functionalization, polymers issued from renewable resources as well as polymer materials for the field of energy.

POLY(1,2,3-TRIAZOLIUM IONIC LIQUID)S: NEW GENERATION OF TAILORED ION CONDUCTING MATERIALS

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Polymerized ionic liquids (PILs) are unique polyelectrolytes with cationic and anionic groups included in the repeating unit. They are extremely attractive in the field of materials science as they combine the properties of ionic liquids (high ionic conductivity, thermal and chemical stabilities) with those of polymers (mechanical stability, processing and tunable macromolecular design).[1] Many examples have demonstrated their potential in applications such as dye sensitized solar cells, fuel cells, batteries, permselective membranes for CO_2 recovery or catalysis.[2] In all these applications, imidazolium-based PILs are by far the most widespread and investigated materials. Since the striking development of the copper-catalyzed azide-alkyne cycloaddition (CuAAC) modular ligation, a large variety of materials containing 1,2,3-triazole groups have been reported. However, applications of 1,2,3-triazolium derivatives as anion recognition structures, organocatalysts, metal ligands and precursors of *N*-heterocyclic carbenes has only been explored lately. We have recently pioneered the synthesis of 1,2,3-triazolium-based PILs (**TPILs**).[3] The tuning of structural parameters has a tremendous effect on ionic conductivity, thermal stability and crucial properties in most applications of PILs. The first generation TPILs not only demonstrate ionic conduction similar to that of imidazolium-based PILs with analogous structure, pendant substituent and anion, but their synthesis also benefits from the versatile, robust and orthogonal nature of CuAAC. This oral communication demonstrates the potential of 1,2,3-triazolium chemistry to tackle new synthetic challenges using unexplored approaches competent to broaden the current structural variety and functionality of PILs (**Figure 1**).

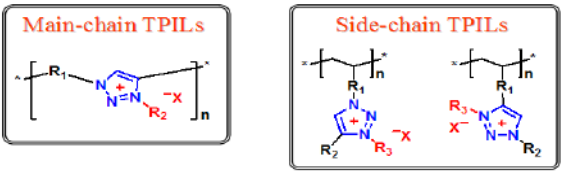

Figure 1. Structural variety of 1,2,3-triazolium-based polymerized ionic liquids.

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Daniel TATON

Daniel Taton received his PhD in polymer chemistry in 1994 at the UniversityPierre et Marie Curie (Paris VI). He has been appointed as an assistant Professor in 1995, then as a full Professor in 2008, at the University of Bordeaux. He develops his research activities at the "Laboratoire de Chimie des PolymèresOrganiques" (LCPO). He is coordinator of one of the four main research topics developed at LCPO, and which is entitled "Polymerization Catalyses and Engineering".

He is co-author of more than 100 articles in peer review journals and 12 patents. His main contributions are in the field of *macromolecular engineering*, through the development of original methodologies based on controlled radical or ionic polymerizations for the synthesis of "complex" macromolecular architectures, including star-like polymers, dendrimer-like polymers, nanogels, and for the design of block copolymers featuring specific blocks (oligosaccharides, polypeptidic, or polymeric ionic liquids) that can self-assemble into sub-micron size structures, in bulk or in solution.

In recent years, he has been focusing on the use of *N*-heterocyclic carbenes(NHCs) as organocatalystsfor various precise and selective polymerization reactions, including group transfer and ring-opening polymerizations as well as step-growth polymerization for the synthesis of polybenzoins or polyurethanes. In view of better handling and recycling NHCs, he also develops masked NHCs and/or polymer-supported versions that can *in-situ* deliver the catalytic active form. In this context, his approach relies on specifically designed poly(ionic liquids) (PILs) derived from imidazolium-based precursors to supported NHCs or poly(NHCs).

Selective Organocatalyzed Polymerization From *N*-Heterocyclic Carbenes (NHCs)

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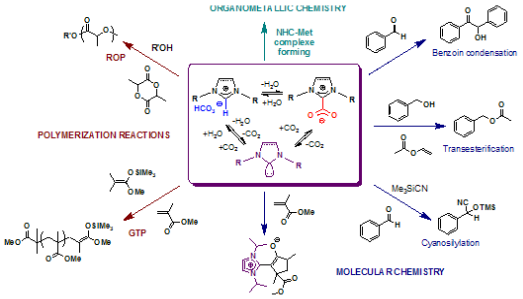
In the last decade, *N*-heterocyclic carbenes(NHCs) have been intensely investigated as organic catalysts in metal-free polymerization reactions.[1]The success of NHCs is due to their steric and/or electronic properties that can be finely tuned through a proper selectionof their substituents (e.g. R groups in the Figure below). Despite their enormous catalytic potential, some issues still limit theirwidespread adoption. For instance, their poor stability makes NHCs difficult to handle.[1]

In this context, we have shown that imidazol(in)ium hydrogen carbonates and imidazolium carboxylates (see Figure)behave as air-stable NHCprecursors, allowing for a facile use for various reactions of molecular chemistry (e.g. benzoin condensation, transesterification, …) and polymer synthesisas well, e.g. for group transfer polymerization (GTP) of methyl methacrylate (MMA) or ring-opening polymerization (ROP) of D,L lactide.[2] Recyclable polymer-supported NHCs generated from imidazolium-based poly(ionic liquid)s (PILs),have also been investigated.[3]

More recently, We have evidenced that reactionof1,3-bis-(diisopropyl)imidazol-2-ylidene (NHCiPr) with MMA affords an unprecedented imidazoliumenolatecyclodimer (NHCiPr:MMA = 1:2; bottom of the Figure). In contrast, the NHC homologue with *tert*-butyl groups(= NHCtBu) allows directly polymerizing MMA in DMF, highlighting the remarkable selectivity of NHCs.[4]



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Ingénierie des Matériaux Polymères (UMR 5223),
Université Claude Bernard, Lyon1,
Position/Title: Professor

EDUCATION
Highest degree: Ph. Doctorat
University : PAU (France) Date : October 28, 1988

Field : Physics

EMPLOYMENT HISTORY
• 1988-1999: Research senior at the National Council for the Scientific Research (CNRS)
• 1999: Full Professor at the University of Lyon. Department of Polymer Engineering
• 2005: Promote to the first rank of full professor
• 2011: Head of Laboratoire Ingénierie des Matériaux Polymères (UMR5223)
• 2013: Promote to the exceptional rank of full professor

MAJOR FIELD OF RESEARCH
- Rheology and processing
- Suspensions, Polymer blends and Structuration under flow
- Morphology developments
- Functional materials
- Composites and nanocomposites

SCIENTIFIC PRODUCTION AND ANOTHER ACTIVITIES
- **Author** or co-author of **160** papers, **10** patents, **6** book chapters and more **200** oral or poster presentations.
- **Supervisor** or co-supervisor of **50** PhD students and **6** Post docs
- **Head** of the Master "Matériaux" Polytech-Lyon, University of Lyon1
- Fellow, the Polymer Processing Society, Award 2006-Fellow, the French Society of Rheology (elected to the board)-Fellow, the American Institute of Physics -Elected fellow, Scientific council of University Lyon1
- **Award of the Polymer Processing Society, Yamagata 2006**

Linear viscoelasticity of suspensions of anisotropic nano-particles

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The objective of the present communication is to discuss the linear viscoelasticity of suspensions filled with nano-size particles of different aspect ratios and structuration. The viscoelastic behaviour of liquid suspensions of well-dispersed and stabilised particles proves that the Brownian motion is the dominant mechanism of relaxation. Accordingly, dilute and semi-dilute suspensions of stabilised carbon nanotubes, cellulose whisker and PS nanofibres obey a universal diffusion process according to the Doi-Edwards theory. As expected from this theory, these straight rigid rods obey a master curve in the variation of the reduced viscosity (or rotary diffusivity) versus the particle concentration. The following power laws on viscosity ($\eta_0 \propto \phi^3$) and diffusivity ($D_r \propto \phi^{-2}$) were clearly observed in semidilute regime in agreement with the Doi-Edwards theory.

However, regarding exfoliated but not well stabilized nano-particles such as carbon nanotubes, graphite oxide and graphene, the particular rheological behaviour of these suspensions arises from the presence of a network structure (inter-particle interaction), which leads to a drastic decrease in the percolation threshold. Fractal exponents are then derived from scaling concepts and related to the structure of the aggregate clusters. For example, we studied the aggregation of untreated CNTs in a low-viscosity PDMS matrix from simultaneous time resolved measurements of electrical conductivity and dynamic shear modulus under different strains. It was shown that the kinetics of CNT aggregation strongly depend on the amplitude of the dynamic shear deformation. Actually, the kinetics of aggregation increases with increasing the deformation. However, above a critical deformation γ_c the equilibrium value of the storage modulus and electrical conductivity decreases with increasing the deformation. It was finally concluded from these measurements that this network structure is broken up under non linear flow and rebuilt upon the cessation of the flow under static or linear conditions (annealing or rest time experiments).

Acknowledgments: The works presented in this paper have been made in the framework of several collaborations. I would particularly like to thank Prof Ph Dubois (Univ Mons), Prof B. Charleux (Univ Lyon1) and my colleagues Drs Seytre, Fuchiron and Beyou. Ph D students (W. Zhang, L. Moreira and S. Marceau) are also thanked for their valuable contributions.

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Academic qualifications

1989- Engineering diploma : Speciality : Material Science Ecole d'Ingénieurs de Sfax-Tunisie.
1990- Master degree in Macromolecular material and composite: INSA de Lyon- France
1993 - PhD Specialized in Sciences of Polymeric Materials – Institut Polytechnique National de Grenoble (France)
2000- "Diplôme d'Habilitation à Diriger les Recherche" - Specialized in Physical Chemistry – Faculté de Sciences de Sfax-Tunisia
PhD in Macromolecualr chemistry and physics- Institut Polytechnique National de Grenoble (France) (1993), Habilitation in Chemistry-University of Sfax-Tunisia in 2001

Employment

University of Sfax, Chemistry Department: Professor, Since 2007
University of Sfax, Chemistry Department: Associate Professor, 2002-2007
University of Gabes: Chemistry Department: Assistant Professor, 1994 – 2002

Current Main Research Activities

- Nanocellulose extraction from wide range of annual plants and crops residue
- Nanocomposite based on biobased nanofillers and a polymer matrices.
- Hybride materials based on metallic NPs and biobased polymer
- Nanoparticles from starch and natural biopolymer

Scientific achievements in numbers (1994 - 2013)

Publications	78
Book chapters	3
Patents	2
International conferences:	15

Selected publications

-BelHaaj Sihem; Ben Mabrouk Ayman; Thielemans Wim; Sami Boufi, A one-step miniemulsion polymerization route towards the synthesis of nanocrystal reinforced acrylic nanocomposites, *Soft Matter 9 (2013) 1975-1984.*
-S Kalia, S Boufi, A Celli, S Kango, Nanofibrillated cellulose: surface modification and potential applications (a review), *Colloid and Polymer Science 2014, 292 (1), 5-31*
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- S BelHaaj, A Magnin, C Pétrier, S Boufi, Starch nanoparticles formation via high power ultrasonication
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Nanocellulose: A sustainable Nanoparticles with outstanding reinforcing potential

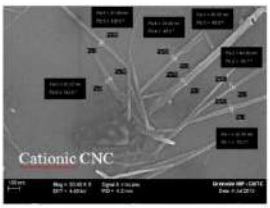
Sami Boufi

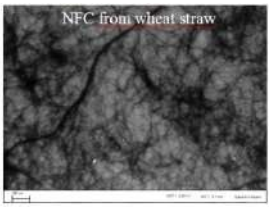
LMSE-University of Sfax-Tunisia

The extraction of nanosized fibrils from cellulose fibres represents, undoubtedly, the most breakthroughs in cellulose-based materials during the past two decades. This is not only evident in academic articles, but it is also manifested by the increasing number of nanocellulose patents that are published every year. Unlike the synthetic nanoparticles, nanocellulose is completely renewable, lightweight material with density around 1.5 g/cm^3, inexpensive to produce and safer to handle compared to synthetic nanoparticles.

Currently, cellulose nanocrystals (CNC) and nanofibrillated cellulose (NFC) constituted the two main families of nanosized cellulose. The former is extracted from fibres after a complete dissolution of the non-crystalline fractions, while the latter results from the application of high shearing forces of disintegration leading to a high degree of fibrillation, which yields highly interconnected fibrils. Applications of nanocellulosics include reinforcement of composite materials, tissue engineering scaffolds, moistening masks for cosmetic applications, filtration media, thickening agents, rheology modifiers, adsorbents, paper reinforcement, etc.

In this presentation, an overview on the preparation, characterization and applications of nancellulose will be reviewed. The emphasis will be put on the activity we have carried out on this topic during the last decade and the approach adopted to reduce the energy demand during the preparation of NFC. Some aspects regarding the use of NFC as nanofiller, adsorbent or strength additive for paper will be also presented.





Oral Communications' List

(alphabetically authors' name)

Name	Ref
M. Abbes, S. Salhi, S. Brogly, C. Delaite, S. Abid, R. El Gharbi *FSS - Sfax* Synthesis and structure–property relationship of poly(ester-amide)s derived from amino-acids	**OC7A**
I. Abdelhedi, É. Drockenmuller, H. Ben Romdhane *FST - Tunis* Photochemical isomerization of norbornadiene-containing polytriazoles obtained by click chemistry polyaddition	**OC3A**
R. Ahmad, C. Mangeney, A. Lamouri, N. Felidj, J. Grand, L. Boubekeur, S. Lau, P. Decorse, J. Aubard *ITODYS - Paris 7 University, France* Functionalization of gold nanorods by molecularly imprinted polymers for extraction, detection or drug release	**OC4B**
M. Ammar, R. Khiari, M.N. Belgacem, E. Elaloui *FSM - Monastir* Thermal characterization and comparisons of lignin-formaldehyde and lignin-glyoxal adhesives	**OC17B**
N. Azizi, Y. Chevalier, M. Majdoub *ENIM - Monastir* Core-shell bio-based polyurethane microcapsules obtained by interfacial polycondensation for textile application	**OC11B**
I. Bekri, E. Srasra *CNRSM - Borj Cédria* Preparation of polyaniline clay nanocomposite using mechanically activated clay as polymerization initiator	**OC3B**
S. Bel Haaj, A. Magnin, C. Petrier, S. Boufi *FSS - Sfax* A one-step emulsion polymerization route towards the synthesis of starch nanoparticles reinforced nanocomposites	**OC14B**
C. Belgacem, R. Medimagh, A. Fildier, A. Bulete, H. Kricheldorf, H. Ben Romdhane, S. Chatti *INRAP - Sidi Thabet* Synthesis and characterization of new isosorbide-based α,ω-dihydroxyethersulfones oligomers as precursors of biomaterials	**OC12B**

Name	Ref
R. Belhassen, P. Mutjéb, S. Boufi *ISSAT - Gabès* Thermoplasticized starch modified by reactive blending with epoxidized soybean oil	**OC6B**
T. Ben Ghzaiel, W. Dhaoui-Ammar, F. Mazaleyrat *FST - Tunis* Electromagnetic properties of polyaniline synthesized by different pathways	**OC15A**
S. Ben Mahmoud, W. Essafi, F. Boue *INRAP - Sidi Thabet* Pearl-Necklace-Like Chain Conformation of Hydrophobic Polyelectrolyte	**OC14A**
C. Ben Osman, F. Stoffelbach *Pierre et Marie Curie University, Paris - France* Synthesis of hybrid particles containing a supported catalyst and use in the atom transfer radical polymerization of methyl methacrylate	**OC11A**
H. Bennour, R. Medimagh, A. Fildier, M. Hangouet, S. Chatti, H.R. Kricheldorf *INRAP - Sidi Thabet* Hyperbranched cyclic and multicyclic poly(etherketone)s by polycondensation of isosorbide and isomannide with 2, 6, 4'-trifluorobenzophenone (TFB) and 1, 3, 5-tris(4-fluorobenzoyl) benzene (TFBB)	**OC20B**
B. Ben Salem, K. Hriz, N. Jaballah, M. Majdoub *FSM - Monastir* New poly(phenylene vinylene-ALT-anthrylene vinylene) semi-conducting polymers: Synthesis and optical properties	**OC16A**
B. Berrima, G. Mortha, N. Belgacem, S. Boufi, L. Aloui *FSS - Sfax* Optimization study of lignin oxypropylation in view of the preparation of polyurethane rigid foams	**OC15B**
A. Bouaziz, M. Jaziri, V. Massardier *ENIS - Sfax* Properties of polypropylene/ethylene–propylene-rubber copolymer/silica: Effect of the high shear process	**OC9B**

Name	Ref
A. Bougarech, M. Abid, F. Gouanvé, E. Espuche, S. Abid, R. EL Gharbi, E. Fleury *FSS - Sfax* Furanic sulfonated biobased copolyesters water sorption and hydrolytic degradation	**OC13B**
M. Braiek, I. Rassas, F. Lagarde, J.F. Chateaux, A. Maaref, N. Jaffrezic-Renault *FSM - Monastir* Electrospun of polymer nanofibrous membranes for biosensor application	**OC17A**
B. Carbonnier, M. Guerrouache, S. Mahouche-Chergui, M.M. Chehimi *Paris-Est University - CNRS - ICMPE - France* Click chemistry as a smart strategy for the site specific chemical functionalization and nanostructuration of porous monolith surface	**OC2B**
S. Chatti, A. Fildier, H. Ben Romdhane, C. Cren-Olive *Lyon University - France* Synthesis and characterization of diahnydrohexitols based biomaterials	**OC6A**
P. Coupillaud, D. Mecerreyes, D. Taton *Bordeaux University - France* Reactive poly(ionic liquid)s (PILs) and nanostructures from PIL-based block copolymers	**OC2A**
K. Elfehri, M. Jaziri, C. Carrot *ENIS - Sfax* The effect of fiber content and alkali treatment on the properties of biocomposites based on MATER-BI® and Alfa fibers	**OC7B**
A. Hassan, R.A. Lafia-Araga, R. Yahya, N. Abd Rahman *Malaya University - Malaysia* Effect of wood heat treatment on the dynamic mechanical and impact properties of injection moulded wood/LDPE composites	**OC10B**
S. Hbaieb-Kharrat, W. Kamoun, M. Abid, S. Abid, R. El Gharbi *FSS - Sfax* New copolyesters derived from furan dicarboxylic acids: A step forward in the development of biobased polyesters	**OC10A**
H. Hrichi, L. Monser, N. Adhoum *INSAT - Tunis* Determination of etoposide based on electropolymerized-molecularly imprinted overoxidized polypyrrole	**OC19B**

Name	Ref
K. Jlassi, M.M. Chehimi, M. Benna-Zayani *FSB - Bizerte* Novel ternary, clay/polypyrrole-silver nanocomposite. UV-induced preparation and catalytic application	**OC5B**
T. Kallel, M. Haddar, M. Kammoun, B. Elleuch *ENIS - Sfax* Biological properties and biodegradation studies of chitosan biofilms plasticized with PEG and glycerol	**OC19A**
A. Kermagoret, Y. Nakamura, C. Detrembleur, C. Jérome, S. Yamago, A. Debuigne *Liège University - CERM - Belgique* Combination of organometallic- and tellurium-mediated radical polymerizations for the synthesis of new block copolymers	**OC4A**
A. Ladhar, M. Arous, H. Kaddami, M. Raihane, A. Kallel, M.P.F. Graça, L.C. Costa *FSS - Sfax* AC and DC electrical conductivity in natural rubber / nanofibrillated cellulose nanocomposites	**OC8B**
N. Mechi Ammar, R. Khiari, E. Elaloui, M.N. Belgacem *FSM - Monastir* Synthesis of a composite material made from phosphogypsum reinforced by two cellulosic fibres (*Prunus amygdalus*, *tamaris*)	**OC20A**
R. Medimagh, S. Mghirbi , A. Saadaoui, A. Fildier, H. R. Kricheldorf, S. Chatti *INRAP - Sidi Thabet* Dianhydrohexitol based diamines for the synthesis and characterization of biosourced polymers	**OC9A**
I. Naghmouchi, M. Pereb, S. Boufi *FSS - Sfax* Olive stones flour as reinforcement in PVC composite: A step forward in the valorization of the solid waste from the olive industry	**OC1B**
M. Omri, A. Triki, M. Guicha, Med. Ben Hassen, M. Arous, H. Ahmed El Hamzaoui, A. Bulou *FSS - Sfax* Influence of Hybridization treatment on the interface region of non-woven alfa fibres reinforced unsaturated polyester composites	**OC18A**

Name	Ref
C. Pouech, L. Prouteau, S. Chatti, C. Cren-Olive *Lyon University - France* Elaboration and characterization of new polydimethyl siloxane based biomaterials used for the extraction of additives released by plastic packaging into water	**OC8A**
J. Richard, C. Delaite, G. Riess, A.S. Schuller *ENSC, Mulhouse - France* Determination of reactivity ratios by proton NMR spectroscopy in free radical copolymerization: Co-monomer carbon chain length influence	**OC12A**
Ö.S Taskin, B.A. Temel, M.A. Tasdelen, Y. Yagci *Istanbul Technical University - Turkey* Synthesis of block copolymers by photoinduced coupling of ketyl macroradicals generated from benzophenone/benzhydrol system	**OC5A**
S. Teka, K. Hriz, N. Jaballah, D. Kreher, F. Mathevet, M. Majdoub *FSM - Monastir* New semi-conducting rotaxane based on β-cyclodextrin and anthracene moieties	**OC1A**
C. Vilela, F. Figueiredo, A.J.D. Silvestre, C.S.R. Freire *Aveiro University - Portugal* Novel functional bacterial cellulose-poly(methacroylcholine chloride) composite membranes	**OC18B**
R. Yahya, N. Mohd Salleh, A. Hassan *Malaya University - Malaysia* Thermal, optical and electrochemical study of side chain liquid crystalline polymers bearing schiff base ester chromophore	**OC16B**
H. Zrida, K. Hriz, N. Jaballah, M. Majdoub *FSM - Monastir* PPV derivatives containing isosorbide side-groups: Synthesis and optical properties	**OC13A**

Oral Communications' Abstracts

Program of
Monday, March
17th 2014

NEW SEMI-CONDUCTING ROTAXANE BASED ON β-CYCLODEXTRIN AND ANTHRACENE MOIETIES

Safa Teka[a], Khaled Hriz[a], Nejmeddine Jaballah[a], David Kreher[b, c], Fabrice Mathevet[b], Mustapha Majdoub[a]

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A rotaxane (**BPAAn/β-CD**), consisting of an anthracene-based material (**BPAAn**) encapsulated into β-cyclodextrin (**β-CD**), has been synthesized via the Williamson reaction in solvent-free conditions. The supramolecular structure of the compound was confirmed by NMR and FT-IR spectroscopies. The optical properties of this semi-conducting organic material were investigated by UV-visible absorption and photoluminescence (PL) spectroscopies. An optical gap of 2.80 eV was estimated from the absorption-onset of the rotaxane thin film. The **BPAAn/β-CD** exhibits a blue photoluminescence in dilute solution; whereas, a green emission was obtained in the solid state, due to the π-π interaction in the anthracene moieties. The rotaxane material shows a significantly enhanced PL quantum yield in comparison with the free **BPAAn**. The HOMO and LUMO levels were estimated using cyclic voltammetry analysis, and show an improved **BPAAn** electronic affinity in complexed form. A single-layer device with the configuration [ITO/rotaxane/Aluminium] has been elaborated and showed low turn-on voltage of 5V.

Keywords: Rotaxane; β-Cyclodextin; Semi-conducting materials; Photoluminescence; Thin solid film.

Reactive Poly(ionic liquid)s (PILs) and Nanostructures from PIL-based Block Copolymers

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[b)] CNRS, LCPO, UMR 5629, F-33600 Pessac (France)
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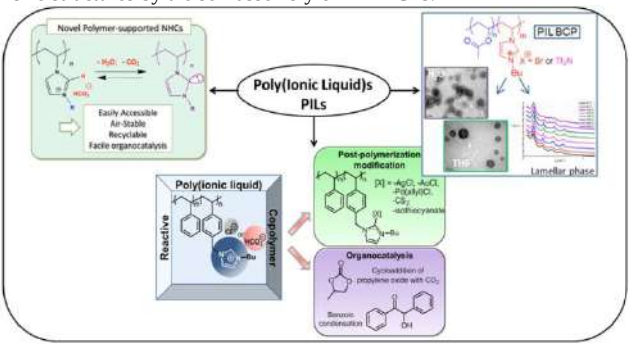
Poly(ionic liquid)s (PILs) represent a special class of polyelectrolytes that have emerged in the past decade. PILs combine the physicochemical qualities of molecular ILs (chemical and thermal stability, high ionic conductivity, etc.) with the specific properties of polymers (film formation and processability). This work aims at taking benefits of PILs in different applications, i) as reactive polymers for the purpose of organic catalysis, ii) as novel precursors for post-polymerization modifications and iii) as building blocks for the design of sub-micron size structures by the self-assembly of PIL BCPs.



Firstly, poly(1-vinyl-3-alkylimidazolium hydrogen carbonate)s were designed and proved useful as air stable to generate polymer-supported *N*-heterocyclic carbenes, poly(NHC)s, formally by a loss of "H_2CO_3" (H_2O +CO_2) in solution.[1] Organocatalyzed reactions such as benzoin condensation, transesterification and cyanosilylation, could be performed with excellent yields until five runs and the *in situ* generated poly(NHC)s is recycled by re-carboxylation.

Secondly, a statistical poly(ionic liquid)-based copolymer (coPIL), -of poly(*N*-vinylbenzyl imidazolium)-type instead of a poly(*N*-vinyl imidazolium) backbone- could serve as a reactive polymer precursor for various quantitative post-chemical modifications such as metallation, stoichiometric addition of organic substrates or transition metals, and for organocatalysis as well, depending on counter-anion (bromide *versus* hydrogen carbonate).[2]

Thirdly, novel block copolymers consisting of a PIL block were synthesized by sequential "cobalt mediated radical polymerization" (CMRP) of vinyl acetate and *N*-vinyl-3-butylimidazolium bromide, utilizing $Co(acac)_2$ as controlling agent.[3] Their ability to generate ordered self-assembled mesostructures, both in solution depending on the solvent and at the solid state was next evidenced. Well-defined nanodomains could be achieved depending on the counter-anion (bromide *versus* bis-triflimide).

REFERENCES AND NOTES

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2. P. Coupillaud, J. Vignolle, D. Mecerreyes, D. Taton, submitted in Polymer (2014).
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PHOTOCHEMICAL ISOMERIZATION OF NORBORNADIENE-CONTAINING POLYTRIAZOLES OBTAINED BY CLICK CHEMISTRY POLYADDITION

Imen ABDELHEDI MILADI[a,b], Éric DROCKENMULLER[b], Hatem BEN ROMDHANE[a]

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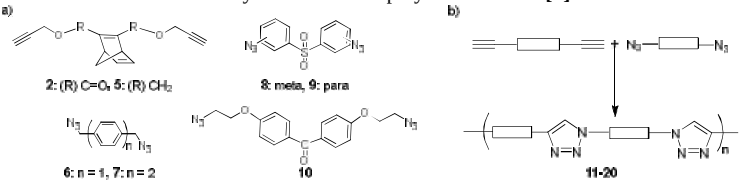
The increasing energy consumption and the rapid depletion of fossil resources are the driving forces to develop new technologies to harvest the solar energy. A promising method for the conversion, storage and release of solar energy is to rely on the photochemical valence isomerization (PVI) of norbornadiene (NBD) into quadricyclane (QC). However the introduction of chromophores into the NBD molecules is necessary since NBD does not absorb in the visible light region. The use of this photoinitiated reaction in polymer materials has been widely discussed **[1]**. Recently Ben Romdhane and coworkers have detailed the synthesis and photochemical properties of polyimides **[2]** based on pentamethylated-NBD units as well as polyamides **[3]**, obtained by polycondensation of dianhydride and carboxylic acid NBD derivatives with different aromatic diamines.
Since the advent of the click chemistry philosophy in 2001 by Sharpless and coworkers **[4]**, it has been an immense source of inspiration for the design of advanced multifunctionnal materials **[5]**. The most studied and reliable click reaction to date is the copper-catalysed azide-alkyne cycloaddition (CuAAC). The step growth polymerization of different diazides and dialkynes (AA+BB) or α-azide-ω-alkynes (AB+AB) to obtain linear polytriazoles and polytriazoles networks has been widely studied **[6,7]**. Herein we present the CuAAC polyaddition of dialkynes **2** and **5** with different aromatic diazides **6-10** to build a series of NBD-containing polytriazoles suitable for PVI (Scheme 1). The resulting polytriazoles have been characterized by ^{1}H NMR, SEC, TGA and DSC. The PVI of the NBD residues into QC units was investigated by UV/vis spectrophotometry from polymer films and was demonstrated for the first time by ^{1}H NMR from polymer solutions.**[8]**



Scheme 1. a) Diazide and dialkyne monomers; b) polytriazoles obtained by CuAAC polyaddition.

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COMBINATION OF ORGANOMETALLIC- AND TELLURIUM-MEDIATED RADICAL POLYMERIZATIONS FOR THE SYNTHESIS OF NEW BLOCK COPOLYMERS

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Shiregu YAMAGO[b], Antoine DEBUIGNE[a]

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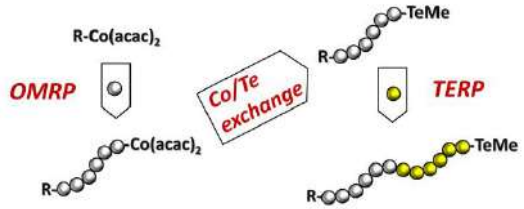
Organometallic-Mediated Radical Polymerization (OMRP) based on $Co(acac)_2$ is one of the most efficient controlled radical polymerization technique for non-conjugated monomers such as vinyl esters or vinyl amides.[1] However, this system shows a low efficiency for most of conjugated monomers including acrylates or acrylamides due to the weakness of the polymer-cobalt bond and is very sensitive to coordinating functionalities of monomers or solvents.[1] On the other side, Tellurium-Mediated Radical Polymerization (TERP) is a versatile process to control the polymerization of numerous monomers containing polar group.[2] Nevertheless, in contrast to OMRP,[3] TERP suffers from severe limitations regarding vinyl esters due to accumulation of too stable P-CH_2-TeR species resulting from Head-to-Head monomer additions.[4] Therefore, we took the best of both techniques and combined them to synthesize new block copolymers following the present strategy: (1) synthesis of a poly(vinyl acetate) by OMRP using $Co(acac)_2$ as controlling agent, (2) Co/Te exchange reaction and (3) chain extension by TERP of N-isopropylacrylamide, 2-(dimethylamino)ethyl acrylate, butyl acrylate, isoprene or vinylimidazole.[5]



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SYNTHESIS OF BLOCK COPOLYMERS BY PHOTOINDUCED COUPLING OF KETYL MACRORADICALS GENERATED FROM BENZOPHENONE/BENZHYDROL SYSTEM

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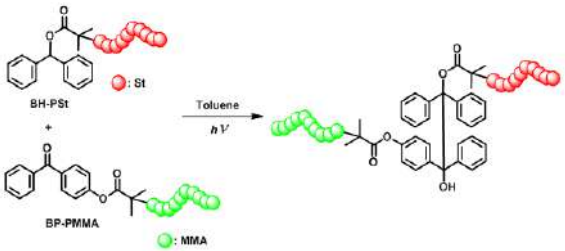
Block copolymers are generally defined as macromolecules, which consist of covalently connected different blocks (or segments) to combine their macroscopic properties and to design hybrid materials. In the area of polymer chemistry, block copolymers can be synthesized either via coupling reactions of different homopolymer segments, or sequential polymerization of diverse monomers. In coupling reactions, two or more polymer chains bearing a reactive group are reacted together to obtain the desired block copolymers.

In this work, a new photochemical strategy has been reported for the successful synthesis of block copolymers by coupling reaction of equivalent ketyl macroradicals. It is demonstrated that benzophenone and benzhydrol end-functional polymers can easily be prepared by ATRP using functional initiators. The benzophenone end-functional polymer can easily abstract hydrogen from benzhydrol end-functional polymers to obtain two ketyl macroradicals. These resonance stabilized polymeric ketyl radicals undergo facile coupling reactions which are responsible for block copolymer formations.



Scheme 1. Synthesis of block copolymer by photoinduced radical coupling process.

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SYNTHESIS AND CHARACTERIZATION OF DIAHNYDROHEXITOLS BASED BIOMATERIALS

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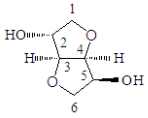
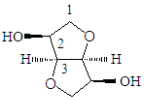
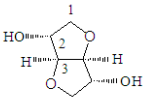
The development of bio-based chemicals has the potential to reduce the amount of petroleum consumed in the chemical industry and also to open new high-value-added markets to agriculture. 1,4;3,6-dianhydrohexitols (isosorbide, isomannide and isoidide) are examples of such chemicals.



isosorbide



isomannide



isoidide

We have synthesis and characterized several biomaterials from these sugar diols such as polyethers [1], polycarbonates [2], polyesters [3], polyether-sulfones [4] and polyetherketones [5]. We have studied the influence of reaction conditions (reaction time, temperature, excess and stereochemistry of monomers) on the fraction of cyclic and molecular weights. The resulting polymers have been characterized by NMR spectroscopy, MALDI-ToF mass spectrometry, SEC and DSC measurements.
Sufficiently high molecular weights and glass transition temperature mean that such polymers can be useful as transparent engineering plastics, films and membranes (less sensitive to an attack of oil, gasoline and other organic solvent or liquid) and as bio-sensors (steroids, metals).

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SYNTHESIS AND STRUCTURE–PROPERTY RELATIONSHIP OF POLY(ESTER-AMIDE)S DERIVED FROM AMINO-ACIDS

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Poly(ester-amide)s (PEAs) have been extensively studied for several years; because they generally demonstrate desired properties of both polyesters and polyamides [1]. Polyamides generally reveal preferred mechanical and processing properties, such as thermal stability, tensile strength and biocompatibility, but are generally not biodegradable [2]. Polyesters exhibit varying degrees of biodegradability; however, lack of the preferred physical properties demonstrated for example by polyamides limits their applications [3].

Therefore when these two different types of linkages are combined in the main chain of one single polymer, the biodegradability caused by the ester linkage and the enhanced thermal and mechanical properties caused by amide-induced intermolecular hydrogen bonds can be combined in one polymer, giving rise to a superior material which could be applied in a wide variety of areas.

In this context, we are interested in the synthesis and characterization of novel aliphatic PEAs in a single step, by the method of melt polycondensation in the presence of a titanium catalyst ($Ti(OBu)_4$), between different amino acids (L-alanine, β-alanine), diacids (succinic acid and adipic acid), and diols (ethylene glycol, propanediol, butanediol and hexanediol).

We also studied the influence of molar fraction amide / ester on the thermal properties of the resulting polymers; indeed increasing the percentage amide generates a significant improvement of decomposition temperatures and glass transition of our polymers. The Thermal behavior of our polymers is susceptible to be compared to that of a commercial polymer named BAK 1095 based on adipic acid, butanediol and 1,6-aminohexanoic acid, synthesized by melt polycondensation and produced by Bayer [4].

The structure of various PEAs was elucidated by FTIR, ^{1}H NMR, ^{13}C NMR, SEC, DSC and TGA. These analyzes clearly confirm the expected structures of polymers, synonymous with the good progress of the copolymerization reaction.

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ELABORATION AND CHARACTERIZATION OF NEW POLYDIMETHYL SILOXANE BASED BIOMATERIALS USED FOR THE EXTRACTION OF ADDITIVES RELEASED BY PLASTIC PACKAGING INTO WATER

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An increasing interest for plastic materials has been observed over the last decades. Besides an excellent quality/price ratio, plastic materials are seen as malleable, strong, lightweight, recyclable and transformable materials. These properties are partly explained by the presence of various additives in the polymer matrix. These additives modify or improve the properties and the performance of the polymer. In this way, the polymer industry is able to provide a specific polymer for each type of application and to respect the requirements imposed in each field. One of the major fields of applications of plastic materials is the packaging in food and pharmaceutical industries.

However, as a packaging product should be inert, it is crucial to study the different interactions that may occur between the packaging and the content. Indeed, the release of substances contained initially into the packaging material represents a significant health concern since it may affect the quality of the product and/or make it toxic. In order to protect and ensure the quality of their products, the food and pharmaceutical industries are more and more concerned by this migration phenomenon. Thus, in order to control migration, several factors such as: the number, the nature, and the maximum quantities of additives allowed were imposed.

In this context, the main objective of this work is to elaborate and characterize new polydimethylsiloxane (PDMS) based biomaterials in order to extract additives authorized by the European Pharmacopeia which can be released by plastic materials into water. Firstly the manufacturing work to prepare the new phase extraction in the form of stir bar (including synthesis, shaping and conditioning) will be presented. Secondly, the optimization of the extraction conditions of this *home-made* stir bar will be exposed. Thirdly, the effectiveness of these new biomaterials in the form of "*home-made* stir bar" will be discussed and compared with that obtained with conventional stir bars.

Dianhydrohexitol based diamines for the synthesis and characterization of biosourced polymers

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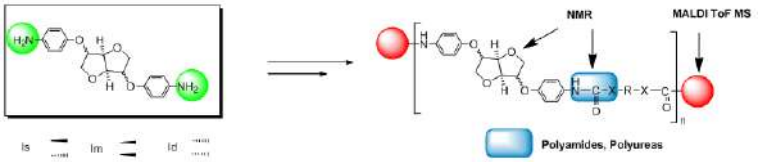
Polymers obtained from renewable feed stock building blocks represent a currently and rapidly expanding field of research. The 1,4:3,6 dianhydrohexitols (**DAH**) are considered as promising biomonomers in this field.[1] However some limitations due to a lack of stability and reactivity of hydroxyle functions gave rise to several attempts in order to significantly broaden their applications field. Modified **DAH**, displaying diamine or extended diol functions found application in polyamides, polyurethanes…[2, 3] We propose herein an original plateform of diamines and derivatives based on three DAH for the building of several families of polymers.[4] Combination of NMR and MALDI ToF mass spectrometry permit to ascertain all the characteristic signals proper to dianhydrohexitol core and to identify the polymers functions and their end chain nature. In another side MALDI Tof MS was used as a powerful and precise analytical tool to investigate the influence of DAH stereochemistry on the structures of the obtained polymers leading to interesting findings. These observations were confirmed when studying thermal properties of the obtained polymers. High glass temperatures as well as melting temperatures were obtained showing that cristallinity was dependent on the DAH and the co-monomer nature.



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NEW COPOLYESTERS DERIVED FROM FURAN DICARBOXYLIC ACIDS: A STEP FORWARD IN THE DEVELOPMENT OF BIOBASED POLYESTERS

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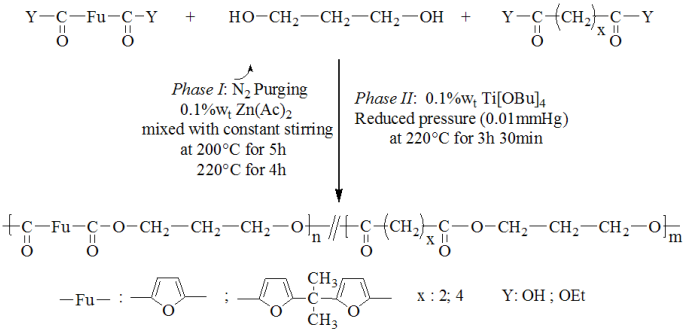
Within the last decade, growing attention has been focused on the synthesis of polymers derived from renewable resources (cellulose, lignin, starch, chitin, etc)[1-2]. The interest in this strategy is double: (i) a large variety of products are available either directly or after chemical modification, (ii) these sources are continuously renewed, cheap and ubiquitous, as well as to counteract the environmental problems related to fossil CO_2 emissions.

The present work aims at studying and characterizing the synthesis of novel copolyesters based on: (i) 2,5-furandicrboxylic acid (**FDCA**)/1,3-propandiol (**PD**)/succinic acid (**ASu**) or adipic acid (**AAd**), and (ii) 5,5'-isopropylidene-bis(ethyl 2-furoate) (**DEF**)/1,3-propandiol (**PD**)/diethyl succinate (**ESu**) or diethyl adipate (**EAd**):



Different stoichiometric amounts of these monomers were used and the ensuing copolyesters were characterised in detail by several physical chemistry and thermal techniques. All copolyesters have the expected chemical structure incorporating both aliphatic and furanic units in different amounts accordingly to the stoichiometric feed-ratio. Only two polyesters were partially crystalline, whereas all the others were amorphous. Thermogravimetric analysis revealed that thermal decomposition began above 320°C in all instances.

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Olive stones flour as reinforcement in PVC composite: A step forward in the valorization of the solid waste from the olive industry

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The production of olive oil leads to considerable amounts of solid wastes mainly composed of hard woody endocarp called here olive stones. The aim of the present work is to explore the possible use of ground olive stones as filler for PVC to elaborate composite material with a solid loading up to 50wt%. After grinding, the olive stone flour (OSF) was incorporated into a PVC matrix via a melt compounding and injection molding to elaborate a PVC-OSF-based composite with filler content up to 50wt%. The evolution of the mechanical performances, impact properties and water absorbance behavior according to the OSF content, were investigated. The addition of OSF was shown to enhance the stiffness of the matrix but at the expense of a reduction in the mechanical strength. The thermal properties of the ensuing composite were also studied by thermal thermogravimetric analysis (TGA). A decrease in the temperature of the onset of PVC thermal degradation was observed as OSF is added into the PVC.

CLICK CHEMISTRY AS A SMART STRATEGY FOR THE SITE SPECIFIC CHEMICAL FUNCTIONALIZATION AND NANOSTRUCTURATION OF POROUS MONOLITH SURFACE

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Combining metallic structures with length scales in the nanometer range with organic polymers provides fascinating opportunities for the design of smart heterostructures with applications in various scientific and technological fields.[1]
Considering porous polymer containing metal nanoparticles (MNPs), the mostly applied preparation pathway invloves the immobilization of MNPs on the pores surface of pre-formed prolymers. This appraoch proved particularly efficient for macroporous polymers with an interconnected network of pores, namely monoliths, allowing for a dynamic loading with colloidal solution of MNPs. Moreover, adsorption behaviour of MNPs can be controlled by adjusting the surface functionality of porous monolith *via* either a judicious choice of monomers, or post-polymerization functionalization.[2] Indeed, using classical organic reactions, such as the epoxide ring opening, surface grafting of amino or thiol groups has been successfully achieved allowing the further immobilization of gold nanoparticles (GNPs). With this approach, good and homogeneous composites can be produced, but control over the spatial distribution of the GNPs was not considered.
In this contribution, we will demonstrate that this gap can be filled spatially controlled surface functionalisation of pore surface.[3] To establish the method, monolith surface with alkyne functionality was reacted with cysteamine through thiol-yne addition reaction providing hydrophilic and chelating interface. Photochemical initiation affords spatial control over the reaction site and further site-specific immobilisation of gold nanoparticles.
Implementation of Huisgen,[4] thiol-ene,[5] and diels-alder click-type reactions will also discussed for the surface grafting of molecular and oligomeric units.
This work undoubtedly highlights click-surface chemistry as a powerful surface modification strategy for spatial control, at the molecular level, the chemical nature of pores surface.

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Preparation of polyaniline clay nanocomposite using mechanically activated clay as polymerization initiator

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Technopole de Borj Cedria; Tunisia

A simple and facile method was used to synthesize polyaniline (PANI) nanocomposites with sodium montmorillonite clay (Na+-MMT) using solid solid reaction as a promoting method and mechanically activated clay as an initiator of polymerization.

Smectite clay was activated mechanicaly by varying period of grinding for a from 5 to 90 mn. Then the activated clay has been mixed with aniline chloride and grounded until changing of the colour of the mixture to dark blue, characteristic coulour of the emeraldine form of polyaniline. Polymerization has been initiated by oxidative properties of activated clay. The intercalation and polymerization of PANI into the clay layers was confirmed by X-ray diffraction FTIR spectra, particularly by the narrowing of the Si–O stretching vibration band confirmed the interaction between PANI and the clay. The AC conductivity data of different synthesized nanocomposites were analyzed as a function of frequency. It was shown that ac conduction shows a regime of constant dc conductivity at low frequencies and a crossover to a frequency-dependent regime of the type A ω at high frequencies.

FUNCTIONALIZATION OF GOLD NANORODS BY MOLECULARLY IMPRINTED POLYMERS FOR EXTRACTION, DETECTION OR DRUG RELEASE

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Anisotropic gold nanorods (GNRs) have received a great deal of attention recently due to their large absorption cross-section in near-infrared frequencies which opens new opportunities for localized hyperthermia in cancer therapeutics[1]. Their use generally requires their functionalization in order to protect colloidal particles from aggregation, to manipulate the optical, electronic and catalytic properties of the gold core, as well as to control interfacial properties. At the same time, **molecular imprinting** has become a widely used method for creating ligand-specific polymeric materials with recognition properties analogous to biomolecular systems[2]. Combining the exceptional characteristics of molecularly imprinted polymers and anisotropic gold nanorods in a single system should afford a hierarchical structure enabling the hybrid particles to report quickly, easily, sensitively and directly a molecular recognition event **without any transducers and treatments** for analytes (label-free). In this talk, I will present an original method for the functionalization of gold nanorods by molecularly imprinted polymer layers. The recognition properties of these new hybrid systems will also be investigated.

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NOVEL TERNARY, CLAY/POLYPYRROLE-SILVER NANOCOMPOSITE UV-INDUCED PREPARATION AND CATALYTIC APPLICATION

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Clay–polypyrrole–silver (MMT–Sil–PPyAg) hybrid material was synthesized via one pot photopolymerization of pyrrole using silver nitrate as a photosensitizer in the presence of montmorillonite (MMT) clay (Fig. 1). Clay was silanized using a pyrrol–functionalized silane to provide anchor sites for the polypyrrole/silver nanocomposite coating (PPyAg). The silanized clay was effective in the making of a ternary hybrid material which exhibits a PPyAg-rich surface. PPy and Ag mass loadings were 19 and 27 wt.%, respectively. Interestingly, silanization by pyrrole-functionalized silane permits to fully exfoliate the clay upon the photopolymerization process. In contrast, without any silanization, the surface of the ternary hybrid material MMT–PPyAg is rather aluminosilicate-rich despite a black colour of the nanocomposite. The anchored Ag particles were found to be in the metallic state. The MMT–Sil–PPyAg nanocomposites were found to act excellent catalysts of the reduction of methylene blue by $NaBH_4$ (Fig. 2) with 94% yield within a few minutes. This work conclusively shows that UV-induced polymerization of pyrrole in the presence of a layered silicate is simple and fast; in addition, it permits to design novel hybrid materials with high catalytic performances.

Figure 1. Protocol of synthesis of ternary MMT–Sil–PPyAg nanocomposite

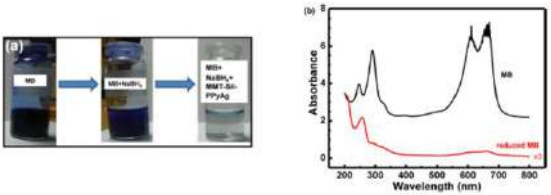

Figure 2. (a) Digital photographs of a solution of pure methylene blue (MB), MB + $NaBH_4$ mixture and MB + $NaBH_4$ mixture after addition of MMT–Sil–PPyAg. (b) UV spectra for the MB + $NaBH_4$ mixture before and after addition of the nanocomposite for the catalyzed reduction of methylene blue.

THERMOPLASTICIZED STARCH MODIFIED BY REACTIVE BLENDING WITH EPOXIDIZED SOYBEAN OIL

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Biopolymer based on thermoplastic starch (TPS) was prepared by melt extrusion of a mixture of starch, glycerol and water. Aiming to improve the stiffness and the mechanical strength of the ensuing TPS, in-situ modification with epoxidized soybean oil (ESO) was carried out using melt reactive blending in an internal mixer. Evidence of the condensation reaction between the epoxide ring of ESO and the hydroxyl groups was supported by FTIR. The evolution of the properties of the modified TPS, in term of mechanical properties, microstructure and water absorption, were investigated using tensile mechanical, dynamic mechanical analysis (DMA), X-ray diffraction (XRD) and water uptake.The addition of ESO results in a huge enhancement in both Young's modulus and tensile strength. The hydrophobicity of the TPS was also increased upon the addition of ESO.

THE EFFECT OF FIBER CONTENT AND ALKALI TREATMENT ON THE PROPERTIES OF BIOCOMPOSITES BASED ON MATER-BI® AND ALFA FIBERS

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In a first stage of this study, Alfa fibers were subjected to an alkali treatment by two different concentrations. The structural modifications of the alkali treatment on the fibers were examined by using Fourier transform infrared (FTIR) spectroscopy, thermogravimetric analysis (TGA), optical microscope, X-ray diffraction and solid-state ^{13}C NMR spectroscopy. Experimental results revealed that the thermal stability, crystallinity index and the amount of cellulose in the fibers were found to improve on alkali treatment. The composites of Materbi® and (treated and untreated) Alfa fibers were elaborated in twin-screw extruder at different fiber contents. The thermal, mechanical and the morphological properties of the composites were investigated. The thermal analysis reveals a nucleant effect of treated or untreated alfa fibers in the Materbi® which accelerated the beginning of the crystallization and increased the crystallinity of the biopolymer. The study of the mechanical properties showed that the addition of treated or untreated alfa fibers to Materbi®, result a decrease of the ductility as well as a reduction in the impact strength of the material. On the other hand, we noted a very important gain of rigidity. The improvement in Young's modulus and tensile strength increased with the highest concentration of alkali treatment and also with the highest fiber content in the composite. The morphological study carried out with the MEB on fracture topographies, showed a good dispersion of the fibers in the matrix and also a good adhesion at the interface matrix-fiber particularly with fibers treated with highest concentration.

AC and DC electrical conductivity in natural rubber / nanofibrillated cellulose nanocomposites.

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We report the results of studies on natural rubber filled, at various doping rates, with naofibrillated cellulose extracted from the rachis of date palm tree. The complex impedance, the direct current (dc) and alternating current (ac) electrical conductivities of cellulose nanocomposites have been investigated in the temperature interval from 100 °C to 200 °C and frequency range 0.1 Hz - 1 MHz. The dc conductivity of all samples is suitably fitted to Arrhenius model. The large activation energy values, extracted from the slopes of the curves, confirm the ionic conduction phenomenon. The compounds have a similar behaviour of organic semiconductors as their dc electrical conductivity increases with increasing temperature. For ac measurements, Argand's plots of the complex impedance show that interfacial polarization contributes to conduction phenomenon. Moreover, at high frequencies, the ac conductivity follows the universal power law $\sigma_{ac}(\omega) = A(T)\omega^{s(T)}$, which is characteristic for charge transport in disordered materials by hopping or tunnelling processes. The observed variation in the frequency exponent *s* with temperature suggests that the non-overlapping small polarons tunnelling (NSPT) and the correlated barrier hopping (CBH) describe the dominant ac conduction mechanism. A direct correlation between conductivity, structure and morphology was obtained in our systems.

Keywords: Nanocomposite, Natural rubber, Nanofibrillated cellulose, Electrical properties.

PROPERTIES OF POLYPROPYLENE/ETHYLENE–PROPYLENE-RUBBER COPOLYMER/SILICA: EFFECT OF THE HIGH SHEAR PROCESS

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The structure and properties of polypropylene and ethylene propylene copolymer blends (PP/EPR) filled with hydrophilic Silica nanoparticles (SiO_2) have been investigated. These nanocomposites were prepared via melt mixing method using high shear processing technology in an intermeshing twin co-rotating screw extruder, in which silica nanoparticles were directly melt blended with (PP/EPR). This preparation method presents a new attractive technique to improve the silica nanoparticles dispersion throughout polymer matrix and to enlarge miscible regions for an originally partial immiscible blend (polypropylene and elastomeric phase). Comparative study with and without compatibilizing copolymer agent Fusabond (maleic anhydride grafted polyethylene: (PE-g-MA) was conducted. Melting blending process of PP/EPR formed a two-phase morphology in which EPR appeared as dispersed phase. The obtained results were interpreted in terms of polymer/polymer; polymer/filler interactions.

The microstructure of the nanocomposite was assessed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). High shear processing has proved to be an efficient process to decrease the nodule size of the dispersed phase (EPR). The (PP/EPR) nanocomposites containing maleic anhydride groups show a core-shell structure (silica: shell, EPR: core) and an important enhancement of mechanical properties while nanocomposites without compatibilizer show a structure containing dispersed silica in the PP phase and a lower improvement of properties.

The results obtained show that speed screw rotation is a crucial factor to reach better properties of the material and show that PE-g-MA worked as an effective compatibilizer. Mechanical properties of the PP/EPR-based nanocomposites were improved significantly with the increase of the speed screw extrusion from 300rpm to 800rpm. A better adhesion between the phases and the elasticity of the interface provided by the terpolymer PE-m-MA could be the explanation of the so good results obtained for the elongation at break measurements.

EFFECT OF WOOD HEAT TREATMENT ON THE DYNAMIC MECHANICAL AND IMPACT PROPERTIES OF INJECTION MOULDED WOOD/LDPE COMPOSITES

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Red Balau saw dust was heat-treated at 180°C and 200°C for one hour and compounded with low density polyethylene, LDPE, at 9, 20 and 37 wt% using a co-rotating twin screw extruder (Brabender KETSE 20/40, Germany). The compounded materials were pelletised and injection moulded into test specimens using injection moulding machine (BOY® 55M, Germany). Both compounding and moulding procedures were carried out at a temperature range of 150–155°C. Charpy impact assessment of the notched samples of the composites revealed that the peak load, P and the critical stress intensity factor, K_c, increased with wood content and treatment temperature. While the energy to failure, W and the critical strain energy release rate, G_c, decreased with wood content, the values were highest in composites made from wood flour treated at 180°C and reduced in 200°C treated wood composites. This behaviour revealed that heat treatment of wood flour at the 200°C resulted in poorer impact properties of the composites. Dynamic mechanical analysis showed an increase in storage and loss modulus of composites made from untreated wood flour relative to the heat-treated ones. Tan delta values were also found to reduce in the heat-treated wood composites as a result of decreased damping. Heat treatment of wood flour at appropriate temperature enhanced both the mechanical and dynamic mechanical properties of the composites. Mohanty *et al.* [1] and Joseph *et al.* [2] also reported a similar trend for PP reinforced with jute and sisal fibre composites respectively. In the same way, Kalaprasad *et al.* [3] observed that incorporation of short sisal fibre into LDPE resulted in an increase in loss modulus.

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Program of Tuesday, March 18th 2014

SYNTHESIS OF HYBRID PARTICLES CONTAINING A SUPPORTED CATALYST AND USE IN THE ATOM TRANSFER RADICAL POLYMERIZATION OF METHYL METHACRYLATE

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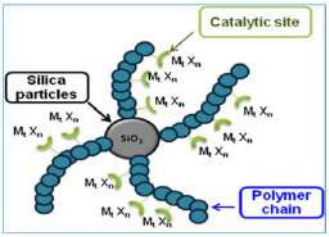
Atom Transfer Radical Polymerization (ATRP) represents one of the most versatile synthetic tools for preparing new polymer materials with well-controlled molecular weights and well-defined structures. A notorious drawback, however, is the tedious separation of the catalyst from the reaction products. For this reason, there is a need to develop innovative methods and strategies in order to overcome this limitation by designing new recoverable and reusable catalyst system. In order to avoid purification steps, catalytic systems that combine the properties of homogeneous and heterogeneous catalysis have been developed by immobilization of the catalyst on organic or inorganic particles [1] (silica, magnetic particles [2]). However, the control level of the polymerization by these covalently solid-supported systems is generally lower than with unsupported catalyst mainly due to the deactivation rate constant of the Cu(II) linked to the immobilized catalysts [3].
In this context, the aim of our work is to develop nanoparticles bearing "catalytic" well defined polymer arms (Figure 1). This new generation of "semi heterogeneous" catalysts are prepared by immobilizing the polymer arms on silica particles by covalent and low-energy bonds [4] (hydrogen bonds). In this presentation, we will show the synthesis by nitroxide mediated polymerization of well defined styrene or azlactone functionalized copolymers and their use (after immobilization) for ATRP of methyl methacrylate.



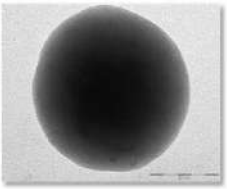


Figure 1: (Left) Silica particle supported by functionalized copolymer for the ATRP of MMA, (Right) hybrid particle observed by transmission electron microscopy (TEM)

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DETERMINATION OF REACTIVITY RATIOS BY PROTON NMR SPECTROSCOPY IN FREE RADICAL COPOLYMERIZATION: CO-MONOMER CARBON CHAIN LENGTH INFLUENCE

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Free-radical copolymerizations of n-alkyl maleates with styrene were investigated to evidence carbon chain length influence on reactivity. Alkyl-maleates carbon chain length varies from four to eight carbons. ^{1}H-NMR spectroscopy has been successfully applied to investigate kinetic parameters of radical copolymerization, therefore this technique has been used in this study[1]. Indeed, by monitoring the variation of the vinylic proton signal of each monomer, corresponding conversions are determined. Thanks to these results, polymer composition at low conversion is known and reactivity ratios are calculated by different linear least-squares methods, such as Mayo–Lewis[2], Finemann–Ross[3] and Kelen–Tudos[4].
The reactivity ratios for styrene-butyl maleate copolymerization are respectively r_1=0.8 and r_2=0.01. Reactivity ratios have also been calculated for copolymerizations of hexyl maleate and octyl maleate with styrene. The reactivity ratio of styrene tends to increase as the carbon chain length of maleate increases. In order to confirm this result, reactivity ratios of n-alkyl itaconates and n-alkyl acrylates with styrene have also been determined (Figure 1).

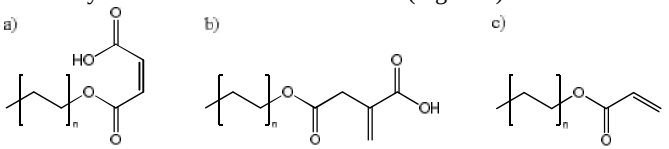

Figure 1: Structures of monomers copolymerized with styrene: a) n-alkyl maleate b) n-alkyl itaconate c) n-alkyl acrylate

Determination of reactivity ratios for n-alkyl itaconates and n-alkyl acrylates gives the same trend as n-alkyl maleate reactivity ratios when carbon chain length increases.

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PPV derivatives containing isosorbide side-groups: Synthesis and optical properties

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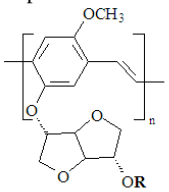
The discovery of electroluminescence in poly(*p*-phenylenevinylene) (PPV) 20 years ago[1] created a new area in organic polymer applications. Since then, enormous progress has been made in the molecular engineering of π-conjugated polymers and in their uses as active materials in polymeric light-emitting diodes (PLEDs).[2] In our contribution, we report the first PPV derivatives containing polar isosorbide-based side-groups.



	R
P1	ethyl
P2	hexyl
P3	dodecyl

P1-3

Figure 1: Polymer structures **P1-3**.

New conjugated PPV derivatives containing isosorbide moieties (**P1-3**) have been synthesized via the Gilch reaction. The polymers are soluble in common organic solvents and show good film-forming abilities. High number-average molecular weights were determined by steric exclusion chromatography (SEC) (46.10^3-56.10^3 g. mol^{-1}). The molecular structures of the polymers were established by NMR and FT-IR spectroscopies. Thermogravimetric analysis of the polymers showed good thermal stability up to about 320 °C.

The optical properties of these materials were investigated by UV-vis absorption and photoluminescence (PL) spectroscopies. The polymers show a yellow fluorescence in dilute solution, and an orange emission is observed in thin films. The introduction of the isosorbide groups improves the PL intensity, and quantum yields between 50 and 73% were obtained. The HOMO-LUMO energy levels were estimated by cyclic voltammetry, and the electrochemical gaps were 1.81, 1.83 and 2.48 eV for **P1**, **P2** and **P3**, respectively. Single-layer diode devices of the [indium tin oxide/**polymer**/aluminum] configuration were fabricated and show relatively low turn-on voltages between 3.1 and 3.4 V.

Keywords: π-conjugated polymer; isosorbide; poly(*p*-phenylenevinylene); thin film; photoluminescence; side-chain effect.

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Pearl-Necklace-Like Chain Conformation of Hydrophobic Polyelectrolyte

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A series of polyelectrolytes with narrow molecular weight distributions and well-characterized fractions of charged monomers on the hydrophobic backbone has been prepared. These polymers are derived from the controlled sulfonation of a series of hydrogenated and deuteried polystyrene (PSSNa-h and PSSNa-d). The sulfonation level of each sample has been determined by NMR and elemental analysis.

The main objective is measuring the form factor of single chain using the Zero Average Contrast method chain of partially sulfonated polystyrene PSSNa at polymer concentrations 0.34 M. The total scattering function is also measured, allowing us to extract the distinct interchain function and an apparent structure factor.

The main result is the behavior of the form factor, which shows contributions of spherical entities as well as extended chain parts.

We demonstrate also the influence of solvent quality on the structure of one polymer chain in the semi dilute solution of partially sulfonated polystyrene sulfonate through the added amount of organic solvent THF.

This dependence is more pronounced when the sulfonation rate is low (more hydrophobic polyelectrolyte). It is proposed that when THF is added, the chain conformation evolves from the pearl-necklace shape already reported in pure water toward the conformation in pure water for fully sulfonated PSS.

ELECTROMAGNETIC PROPERTIES OF POLYANILINE SYNTHESIZED BY DIFFERENT PATHWAYS

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Recently intrinsically polymers, particularly Polyaniline (Pani), have been the focus of many researchers as advanced engineering materials due to their improved physical and mechanical properties. A part from their mechanical flexibility, these properties include improved optical activity, environmental stability and electromagnetic properties.

Among conductive polymers, Pani can be synthesized by either of chemical or electrochemical oxidative polymerization of aniline. However, these methods of Polyaniline synthesis have their advantages and disadvantages. Therefore, it is worth to compare and understand the differences between them.

In the present work, we report results about the elaboration and characterization of Polyaniline synthesized by different pathways.

The structure, morphology, magnetic and dielectric properties of Pani samples were characterized by powder X-ray diffraction (XRD), Fourier Transform Infrared (FTIR), UV-visible Spectroscopy (UV-vis), Scanning Electron Microscopy (SEM), Vibrating Sample Magnetometer technique (VSM) and network analyser.

Keywords: Polyaniline; polymerization; Magnetic properties, conductivity.

NEW POLY(PHENYLENE VINYLENE-*ALT*-ANTHRYLENE VINYLENE) SEMI-CONDUCTING POLYMERS: SYNTHESIS AND OPTICAL PROPERTIES

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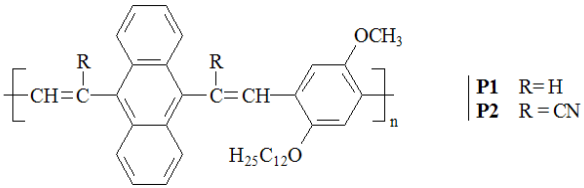
Two polymers containing alternated phenylene-vinylene and anthrylene-vinylene moieties have been synthesized via the Wittig and Knoevenagel polycondensations. Their molecular structures were confirmed by ^{1}HNMR and FT-IR spectroscopies. The polymers are soluble in common organic solvents and have number-average molecular weights of 13750 and 6430 g.mol^{-1} for **P1** and **P2**, respectively. The DSC analysis shows a good thermal stability and an amorphous morphology for these organic materials.



The molecular structures of the polymers

The optical properties of the polymers were investigated by UV-visible absorption spectroscopy. The optical gaps were estimated from the absorption-onsets of the polymer films; the values were 2.53eV and 2.60eV for **P1** and **P2**, respectively. In dilute solution, the **P1** exhibits a yellow photoluminescence; whereas,blue emission was observed in the case of **P2**, due to the introduction of the CN groups on the π-conjugated system.The HOMO and LUMO levels were estimated using cyclic voltammetry analysis; the values show a significantly effect of the cyano groups on the electronic structure of thesesemi-conducting polymers.

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

CORE-SHELL BIO-BASED POLYURETHANE MICROCAPSULES OBTAINED BY INTERFACIAL POLYCONDENSATION FOR TEXTILE APPLICATION

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Encapsulation is a good way to control fragrance release and to make more durable perfumed textiles.[1,2] New core shell microparticles made of a bio-based polyurethane wall and neroline fragrance as a core material, were produced by interfacial polymerization technique.[3] Neroline-loaded microcapsules have been investigated for their properties and their suitability for cosmetotextile applications. The chemical structure of the microcapsules was confirmed by IR and ^{1}H NMR spectroscopies. Microcapsules of spherical shape consisted of a liquid core and a polymer wall, as revealed by means of optical microscopy, scanning electron microscopy (SEM) observations and small-angle light scattering measurements. The encapsulation efficiency of perfume was determined using ^{1}H NMR analysis technique. The microcapsules were deposited onto cotton fabrics by an impregnation process.[3,4] The durability and the perfume release behaviour of the impregnated textile were evaluated through SEM observations, UV-visible spectroscopy and Gas Chromatography.

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Synthesis and characterization of new isosorbide-based α,ω-dihydroxyethersulfones oligomers as precursors of biomaterials.

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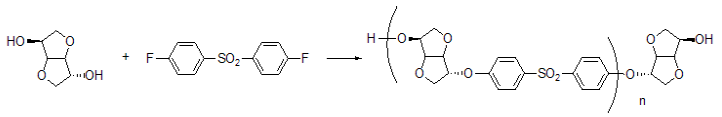
The scope of this study is the synthesis of new bio-based dihydroxy functional oligomers of polyethersulfones based on isosorbide as precursor of biomaterials [1]. The hydroxy end-groups were controlled by the variation of the stoichiometry ratio between isosorbide monomer issued from corn starch [2-4] and 4,4'-difluorodiphenylsulfone. A comparative study was conducted to investigate the effect of isosorbide excess and its concentration in the reaction mixture on structural composition and molecular weights of prepared oligomers.
Several oligo(ether-sulfones) with different end-groups were synthesized by finely tuning the experimental parameters. Optimized polycondensation conditions, yielded over 94% of α,ω-dihydroxy-linear oligomers (Scheme) with low molecular weights (6kDa) and narrow polydispersity index (Đ=1.11). The chemical characterization of the synthesized PES was carried out by MALDI-ToF, ^{1}H and ^{13}C NMR, differential scanning calorimetry and size-exclusion chromatography.



Scheme

This work demonstrates that novel oligomers with tuned physicochemical properties can be designed in a simple and efficient step growth polymerization using stoichiometric adjustments and leading to promising precursors of biomaterials.

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FURANIC SULFONATED BIOBASED COPOLYESTERS WATER SORPTION AND HYDROLYTIC DEGRADATION

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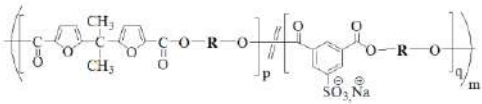
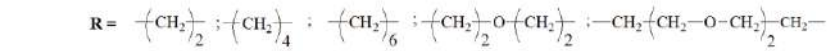
Working with bio-sourced monomers is of interest in the case of polyesters since such polymers are widely used in industry or home applications. Thus hydrophilic copolyesters which can be ionic [1] or non ionic [2] polymer are found as soil release molecules or surfactants in home care application or as sizing agent and dye enhancers in the textile domains. The aim of present work was to synthesis a novel furanic based sulfonated polyesters by melt polycondensation [3].
Furanic based sulfonated copolyesters were prepared by melt polycondensation from diols: (Ethandiol, Butanediol, Hexanediol, Diethylene glycol, Triethylene glycol) and mixture of (5,5'-Isopropylidene-bis(ethyl 2-furoate)) and (dimethyl 5-sodiosulfoisophthalate). Structural characterization of copolyesters was investigated by (^{1}H, ^{13}C NMR and 2D NMR). The NMR investigation for all copolyesters indicates that copolymerization takes place and the microstructure was found to be blockiness. The thermal properties show that the level of Tg was correlated to the diols nature and increases with content of sulfonated moieties ranging from 5 to 40%. Liquid water sorption was also performed on these copolyesters. Sorption isotherms revealed a high dependence of liquid water sorption in the diols nature. The hydrolytic degradation in acidic aqueous conditions show that the mechanism of the hydrolysis of copolyesters was elucidated in relation with the microstructure.





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A one-step emulsion polymerization route towards the synthesis of starch nanoparticles reinforced nanocomposites

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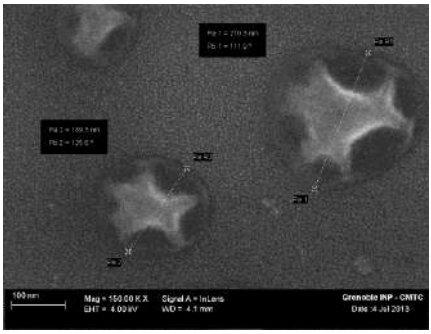
Recently, there has been a growing interest in Pickering emulsions in different fields including cosmetics formulation, food engineering, pharmaceuticals, and nanocomposite materials. Using solid particles, conventional emulsifying agents can be omitted and hazardous surfactants may be replaced by less harmful materials and more environmentally-friendly components. Furthermore, the use of surfactants is known to adversely affect the mechanical and the water resistance properties of the resulting films. All of these attributes make the Pickering polymerization approach of great interest.Furthermore, Preparation of the emulsion with the nanoparticles present (in-situ approach) has the further advantage of: (i) creating the formulation in one step that than through the so-called ex-situ approach requiring the nanoparticles to be blended with the polymer latex after polymerisation has taken place, and (ii) reducing the necessary processing steps.

In the present work, a one-pot approach for the synthesis of stable nanocomposite dispersions was reported via emulsion polymerisation, using starch nanoparticles (SNP) as a sole stabilizer. The particle size of the dispersion ranged from 120 nm to about 300 nm, depending on the SNPs' content; The higher the amount of SNPs, the lower the particle size is. The ensuing dispersion gives rise to a transparent nanocomposite film at room temperature after water evaporation and particles coalescence.



FE-SEM micrographs of freeze-dried poly(butyl methacrylate) emulsions stabilised with 8 wt% SNP

Optimization study of lignin oxypropylation in view of the preparation of polyurethane rigid foams

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Lignin is a renewable material obtained in huge quantities as a by-product of the pulp industry. Until now, it is mainly used as fuel and only a small amount is isolated from spent pulping and commercialised. This work deals the utilisation of lignin as a macromonomer in rigid polyurethane foam synthesis after chemical modification. For such purpose the lignin extracted from black liquor were converted into liquid polyols by chain extension reaction with propylene oxide (oxypropylation). The polyol formulation was optimised by modifying the lignin/propylene oxide ratio, and the catalyst content (KOH). The influence of the studied variables was inspected following the evolution of the hydroxyl index, viscosity and FTIR spectroscopy.

Key words: Lignin, Polyol, oxypropylation, viscosity, hydroxyl index.

THERMAL, OPTICAL AND ELECTROCHEMICAL STUDY OF SIDE CHAIN LIQUID CRYSTALLINE POLYMERS BEARING SCHIFF BASE ESTER CHROMOPHORE

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A series of new side chain liquid crystalline polymers (**P1-P4**) containing Schiff base ester mesogen in the side chain were successfully synthesized by conventional free radical polymerization. The chemical structure, thermal stability and mesophase behaviors of the synthesized polymers were characterized. All the polymers showed good thermal stability and revealed liquid crystalline property. From TGA analysis, all the resulting polymers were thermally stable below 389°C which is really excellent for the practical processing or for the possible use in devices [1]. The synthesized polymers exhibited blue photoluminescence and **P1** exhibited highest intensity among **P1-P4**. The absorption peaks from UV-Vis spectroscopy of these polymers varied from 300 to 400 nm, while the fluorescence peaks varied from 380 to 560 nm. The absorption maxima of UV-vis and photoluminescence spectra were influenced by the electron donating substituent located at the side chain of polymers [2]. All polymers have the characteristic of emissive polymers. The obtained HOMO and LUMO values of polymers are around -5.59 and -2.59 respectively which are close to that of the materials currently use in hole-transporting layers [3,4]. The band gap values of polymers (3.03-3.18 eV) revealed that the newly synthesized SCLCPs could be potential candidate in photovoltaic applications.

Keywords Schiff base ester, Side-chain liquid crystal polymers, Optical properties, Electrochemical properties

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Program of Wednesday, March 19th 2014

Electrospun of polymer nanofibrous membranes for biosensor application

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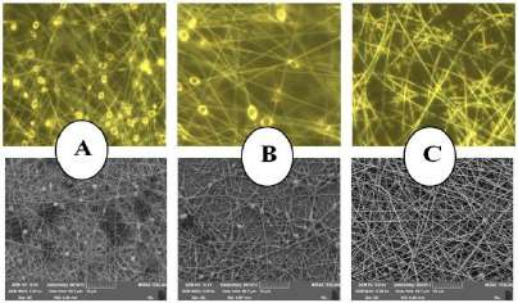
Electrospinning is a physical process enables the production of non-woven mats of fibers with diameters ranging from the sub-micrometer size down to the nanometer range from a polymer solution [1,2]. The electrospun have been employed successfully to immobilize several organic compounds such as bacteria, vitamins, virus, enzymes, etc. [3]. In this work, biosensors elaborated by direct electrospinning of two different polymers, poly(ethylene co-vinyl alcohol) (EVOH) and poly-vinyl-pyrrolidone (PVP), containing enzyme onto gold electrodes was proposed for urea detection using electrochemical impedance spectroscopy (EIS). The effects of the polymer concentration, the solvent nature and parameters of experiment on the fiber diameter and morphology were determined.



OM and SEM images of poly(ethylene co-vinyl alcohol) (EVOH) electrospun nanofibers prepared with different voltages: (A) 10 kV, (B) 15 kV, (C) 20 kV

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Influence of Hybridization treatment on the interface region of non-woven alfa fibres reinforced unsaturated polyester composites

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Dielectric measurements of the alfa/wool/thermo-binder fibres reinforced polyester composite were carried out in the frequency and temperature ranges 10^{-1} - 10^{6} Hz and 40 - 150°C, respectively. Two dielectric relaxations processes were identified, namely the α mode relaxation associated with the glass transition of the polyester resin matrix and the relaxation process associated with conductivity occurring as a result of the carrier charges diffusion noted for high temperatures above glass transition temperature and low frequencies. Analysis of the dielectric relaxation for high temperatures according the Havriliak-Negami model revealed an additional dielectric relaxation process attributed to the interfacial polarization effect. Such effect is due to the accumulation of charges at the fibres/polyester interfaces, which depends on the adhesion between constituents of the composite. Vibrational study performed by using the FT-IR technique revealed that adhesion mechanism is based on inter-diffusion and chemical bonds formed by secondary bonding thanks to wool and thermo-binder fibres.

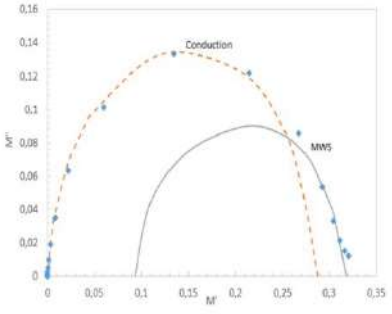

Figure 1: Argand's plots of the electric modulus, M* of the composite at 150 °C

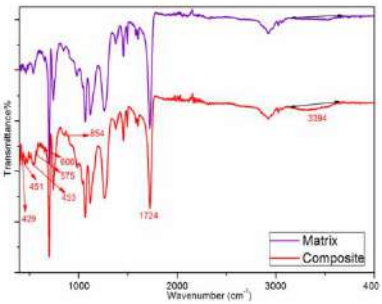

Figure 2: FTIR spectra of Matrix and composite in the wavenumber range 400-4000 cm^{-1}

BIOLOGICAL PROPERTIES AND BIODEGRADATION STUDIES OF CHITOSAN BIOFILMS PLASTICIZED WITH PEG AND GLYCEROL

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Chitosan biofilms, prepared by casting method at various percentage of plasticizer (PEG and glycerol), were evaluated for their biological, structural and thermal properties. The addition of PEG at 30 % (w/w) and Glycerol at 10 % (w/w) to chitosan (Figure 1) has increased the antioxidant activity of biofilm with the percentages of 22 and 26 %, respectively[(1)]. The increase of ferric reducing power was noted for the mixtures of chitosan-PEG (70-30) and chitosan- GLY (75-25). Additionally, the antibacterial properties of several biofilms were tested against E. coli and S. aureus. Biofilms with 70-30 and 90-10 blends ratios of chitosan-PEG and chitosan-GLY showed the best inhibitory effect against E[(2)]. coli and S. aureus with 12 and 27 %, respectively. All biofilms were degraded in compost in liquid and the addition of plasticizer PEG to chitosan increased his biodegradability with a value of BOD5 about 2.33 O2/mg CO.
FT-IR spectra showed that the addition of plasticizer promoted the interactions through hydrogen bonding as reflected on the shifting of main peaks but there is no effect on biodegradation.

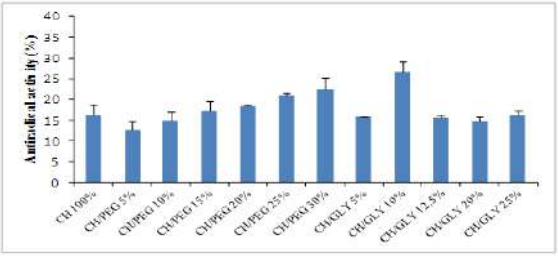

Figure 1: Antiradical activities of chitosan biofilm and chitosan biofilms plasticizer with PEG and GLY measured on DPPH. BHT was used as a reference. IC_{50} of BHT measured after 30 minutes of incubation is 2.27 µg/ml.

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Synthesis of a composite material made from phosphogypsum reinforced by two cellulosic fibres (*prunus amygdalus, tamaris)*

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The production of phosphoric acid from natural phosphate rock by the wet process gives rise to an industrial by-product called phosphogypsum (PG). About 5 tons of PG are generated per ton of phosphoric acid production, and worldwide PG generation is estimated to be around 100–280 Mt per year. The aim of this work is to add value to this waste; After characterization, the PG was calcined and treated with calcium carbonate to reduce curing time. The cellulose fibers are characterized at the end to choose the most effective way to reinforced the phosphogypsum (PG) by two types of cellulosic fibers are witch introduced (*prunus amygdalus, tamaris)* into PG with different mass percentages of 0%, 5%, 15%, 25%, 30% and 40%.The study involved physical, chemical, mechanical and environmental tests on the obtained plaster. The obtained results showed that the incorporation of cellulosic fibers increases the mechanical property and decrease the water solubility. The density of the final product depends on nature of fiber and is low compared to the commercial product.

KEYWORDS: composite material, *cellulosic fibers*, phosphogypsum, *plaster*, *curing time.*

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 word: Lignin; glyoxal; formaldehyde; resin; thermal stability.

NOVEL FUNCTIONAL BACTERIAL CELLULOSE-POLY(METHACROYLCHOLINE CHLORIDE) COMPOSITE MEMBRANES

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Cellulose, an ubiquitous natural polymer, is considered an inexhaustible source of new functional and sustainable materials for a wide range of applications.[1] Bacterial cellulose (BC), also known as microbial cellulose, is produced by several non-pathogenic bacterial strains and shows unique properties due to its tridimensional nano- and microfibrilar network.[2] Particular attention has been devoted in recent years to the development of BC-based (nano)composite materials, either by simply blending of BC with different polymeric matrices or by *in situ* polymerization of monomers within the BC network, for a panoply of end-uses[1] including ion-exchange membranes for fuel cell applications.

In the present study, a series of BC-based nanocomposite membranes was prepared by the *in situ* free radical polymerization of methacroylcholine chloride (MACC), an ammonium functionalized monomer for the synthesis of cationic polymers,[3] within the BC network (Figure 1). The obtained composites were characterized in terms of chemical structure, crystallinity, morphology, thermal and mechanical properties, and water absorption behaviour. The membranes exhibited higher thermal stability and mechanical performance than the pristine homopolymer poly(MACC). Finally, preliminary conductivity tests envisage their use as a new generation of low-cost and environmentally sustainable membranes for anion exchange fuel cells.

Figure 1. *In situ* free radical polymerization of MACC within BC membranes.

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DETERMINATION OF ETOPOSIDE BASED ON ELECTROPOLYMERIZED-MOLECULARLY IMPRINTED OVEROXIDIZED POLYPYRROLE

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In this work, preparation of a molecularly imprinted polymer (MIP) film and its recognition properties for a chemotherapeutic agent Etoposide were investigated. The sensor was prepared by electropolymerization via cyclic voltammetry of pyrrole on a Glassy Carbon Electrode in the presence of Etoposide (template). The template molecules were removed from the modified surface by overoxidation in sodium hydroxide solution. A non-imprinted polymer modified electrode (NIP) was used as a control electrode and prepared under the same condition without adding Etoposide to check the reliability of the measurements.

The performance of imprinted (MIP) and non-imprinted (NIP) film was evaluated with differential pulse voltammetry (DPV). Several important parameters controlling the performance of the molecularly imprinted polymer (MIP) modified sensor were optimized.

In the optimal condition, the response of the imprinted sensor to Etoposide was linearly proportional to its concentration over the range 510^{-7}M to 10^{-5}M with a detection limit of 0.28 10^{-8}M (S/N=3). Meanwhile, good stability and reproducibility were observed. The MIP sensor was successfully applied for the determination of Etoposide in injectable dosage form.

Hyperbranched Cyclic and Multicyclic Poly(etherketone)s by Polycondensation of Isosorbide and Isomannide with 2, 6, 4'-trifluorobenzophenone (TFB) and 1, 3, 5-Tris(4-fluorobenzoyl) benzene (TFBB)

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The step growth polymerizations based on difunctional and trifunctional monomers (''a_2+b_3'') were called by P. Flory "three-dimensional polycondensations".[1] Most of the authors did not take care of the influence of cyclization reaction on the normal course of a polycondensation reaction and it was demonstrated that cyclization may be particularly effective in a_2+b_3 polycondensations leading to cyclic and multi-cyclic polymers. The latter are interestingly soluble.[2] These types of materials could be useful as adhesives and thermo sets. Preliminary study on the polycondensation of isosorbide and (2,6,4'-trifluorobenzophenone) TFB wasn't successful and led only to linear polymers. The cyclization tendency was improved when the TFB was replaced by (1,3,5-tris(4-fluorobenzoyl) benzene) TFBB. All products obtained from all the feed ratios showed good solubility. The feed ratio was varied from equimolarity to equifunctionality (1:1 to 1:1.5) and the structures were investigated using MALDI-ToF MS. Therefore the ideal feed ratio to be chosen for the obtaining of multicyclic structures was found between 1.31/1.0 and 1.41/1.0. At 40 % and 50% excess of diol, in addition to cyclic and multicyclic species new transition structures appears representing a multifunctional polyhydroxylated material. To improve the cyclization tendency isomannide was reacted with TFBB leading mainly to cyclic, bicyclic and multi cyclic species.[3] This can be explained by the higher cyclization tendency of isomannide compared to isosorbide. DSC measurements demonstrated that cyclic and linear species behaves thermally differently. The conducted SEC measurements demonstrated how the increasing of feed ratio correlates with the molecular weights and molecular weight distributions giving rise to high molecular value for the optimal ratio.

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Poster Communications' List

(alphabetically authors' names)

F. Abdelkafi, H. Ammar, M. Tessier, A. Fradet, M.Delmas, R. El Gharbi *FSS - Sfax* Characterization and modification of lignin biopolymer	**PC1**
M. Abidi, A. Kriaa, A. Hedhli *ESSTT - Tunis* Synthesis and characterization of fluorinated quaternary ammonium surfactant intended for mesoporous silica nanoparticles	**PC2**
H. Abiras, L. Belarbi, L. Bennabi *Djilalli Liabes University - Algeria* Synthesis and caracterization of polyesteramide derived from bis (2-oxazoline), anhydrid and diol study of degradation	**PC3**
S. Alila, A. Chaker, Sami Boufi *FSS - Sfax* Nanofibrillated cellulose: Effect of the hemicelluloses content and the fibers' morphology on the nanofibrillation efficiency of cellulose fibers	**PC4**
I. Alkskas *Misurata University - Libya* Synthesis and properties of thermotropic liquid crystalline poly(azomethine-ether)s containing 1-phenethyl-4-piperidone moiety	**PC5**
F. Aloulou, S. Boufi *FSS - Sfax* Cellulose based aerogel as an adsorbent for metallic cations	**PC6**
H. Ayadi, T. Mekhalif, Z. Salmi, B. Carbonnier, M. M. Chehimi *20 Août 1955 University, Skikda - Algeria* Lysozyme-imprinted polyacrylamide thin layers on PVC beads	**PC7**
M. Ayadi *ENIT - Tunis* Extraction des nanocharges du liège, élaboration et caractérisation d'un matériau composite à matrice biodégradable de PHBV	**PC8**
N. Azizi, Y. Chevalier, M. Majdoub *ENIM - Monastir* Morphology and structure of bio-based microcapsules for perfume release	**PC9**

S. Azzouz, F. Meganem *FSB - Bizerte* Adsorption of metals by EDTA-Chitosan	**PC10**
S. Badi, S. Seridi, M. Kadri *Guelma University - Algeria* Investigation of the inclusion processes of adrenaline with B - cyclodextrin: A computational study	**PC11**
G. Bayramoğlu, S. Eşiyok *Yalova University - Turkey* Preparatıon, thermal and mechanıcal properties of uv curable poss epoxy hybrıd coatıngs	**PC12**
S. Belaidi, A. Kerassa *Biskra University - Algeria* Electrical and structural properties of chlorothiophenes as monomers for new conducting	**PC13**
H. Ben Abderrazak, H. Ben Romdhane, S. Chatti, R. Medimagh, D. Prim *FST - Tunis* New bio-based vinyl monomers from dianhydrohexitol isomers and/or furan	**PC14**
H. Ben Ali, S. Besbes-Hentati, M. Rzaigui *FSB - Bizerte* The adsorption of the acidic textile dyes onto Chitosan	**PC15**
N. Ben Ammar, T. Saied, A. M'nif, A.H. Hamzaoui *CNRSM - Borj Cédria* Synthesis of PVP/ PEG hydrogel and effect of agar on the super absorbing properties of the copolymer	**PC16**
A. Ben Chaabane, M.A. Sanhoury ,Z. Ben Hamed, F. Kouki, A. Zeinert, H. Bouchriha *FST - Tunis* Optical characterization of PVK/ZnSe hybrid nanocomposites for photovoltaic application	**PC17**
S. Ben Cheikh *ENIT - Tunis* Development and characterization of a biocomposite based on biodegradable matrix, nanocharges extracted from marine plants and active principles; and modeling the kinetigs of delivery of the active substance; such as: Ibuprofen, β cetamol, cafeine, salicycate	**PC18**

S. Ben Cheikh *ENIT - Tunis* Extraction of cellulose microfibers from the alfa and the elaboration and characterization of a PHBV / MFC biocomposite	**PC19**
M. Ben Gara, W. Kamoun, S. Salhi, S. Brogly, C. Delaite, S. Abid, R. El Gharbi *FSS - Sfax* Synthesis and structural study of copolyesters based on bis(2-hydroxyethyl terephthalate) and various lactones	**PC20**
M. Ben Haj Yahia, R. Ben Cheikh *ENIT - Tunis* Formulation and characterization of a drug delivery system based on PHBV copolymer matrix	**PC21**
A. Ben Mabrouk, A.M. Ferraria, A.M. Botelho Do Rego, S. Boufi *FSS - Sfax* Highly transparent nancomposite films based on polybutylmethacrylate and functionalized cellulose nanocrystals	**PC22**
N. Besbes, K. Diaf, F. Aboumessaad, M.L. Efrit, E. Srasra, A. Mesli *CNRSM - Borj Cédria* Synthesis of dimers and copolymers from aldehydes under homogeneous and heterogeneous medium	**PC23**
A. Bessadok, J. Bras; A. Dufresne, N. Belgacem *IPEIG - Gafsa* Effects of OH-bearing matrices for copolymerization routes	**PC24**
A.U. Birnin-Yauri, A.P. Abbott, W.R. Wise *Kebbi State University, Aliero - Nigeria* Development of borax modified starch-based plastics	**PC25**
M. Boukari, A. Kriaa, A. Hedhli *ESSTT - Tunis* Synthesis, characterization and evaluation of physico chemical properties of polymeric components of nonionic hydrocarbonated and perfluorinated surfactants	**PC26**
G. Boulila, E. Darque-Ceretti, M. Vincent *Ecole des Mines de Paris - France* Analysis of different treatments of aluminum mold for easy release of epoxy pieces	**PC27**

A. Bourguiba, W. Dhaoui, E. Ghorbel *FST - Tunis* Study of the rheological behavior of epoxy - amine system cross-linked at 30°C with and without thinner	**PC28**
F. Brahmi, F. Meganem *FSB - Bizerte* Chemical modification of β-amino alcohols and their use in deppolution	**PC29**
A. Chaker, S. Alila, M. Rei Vilar, S. Boufi *FSS - Sfax* Naofibrillated cellulose from Alfa and sunflower plant: Effect of the delignification on the Nanofibrillation behavior	**PC30**
M. Chehimi, A. Mekki, S. Samanta, A. Singh, Z. Salmi, R. Mahmoud, D. K. Aswal *Paris Diderot University, CNRS - ITODYS - France* Core/Shell, multiwalled carbon nanotube/polyaniline nanocomposites by a facile approach using in-situ generated diphenylamine diazonium salt	**PC31**
M. Chemli, A. Haj Said, K. Hriz, N. Jaballah, M. Majdoub *FSM - Monastir* Synthesis, structure and properties of a new poly(p-phenylene) derivative	**PC32**
N. Chihaoui, B. Hamdi, R. Zouari *FSS - Sfax* No centrosymetric new organic-inorganic hybrid crystal structure of 1, 2 ammonium cyclohexane tetrabromozincate (II) monohydrate	**PC33**
S. Çubuk, M.V. Kahraman, E.K. Yetimoğlu *Marmara University - Turkey* Photocured polymeric optical fluorescence sensor for the determination of Hg (II)	**PC34**
I. Dabbabi, M. Ammara, E. Elaloui *FSG - Gafsa* Etudes des lignines de l'esparto (stipa tenacissima)	**PC35**
L. Dammak, I. Jarraya, M. Kammoun, M. Abid, S. Abid *FSS - Sfax* Synthesis of new polyoxadiazoles bearing furanic-pyridinic moieties	**PC36**

S. Djebabra, Djamel Barkat *Biskra University - Algeria* Effet du milieux aqueux sulfate et nitrate sur l'extraction du cuivre(II) par l'acide di-(2-éthyl hexyl) phosphorique	**PC37**
A. Douibi, D. Benachour, A. Hellati *Ferhat Abbas University, Sétif - Algeria* Synergic effect of a combined use of two chemical blowing agents on the foaming efficiency of an extruded rigid PVC compound	**PC38**
M. El Aassar, M.S. Mohy Eldin, A.A. Elzatahry, M.M.B. Al-Sabah *ATNMRI - Egypt* Poly (acrylonitrile-co-methyl methacrylate) nanoparticles for enzyme immobilization: Preparation and characterization	**PC39**
F. Elaieb, A. Hedhli *ESSTT - Tunis* Synthesis, Characterization and Reactivity toward diethanolamine of α, ω–ditosylates	**PC40**
F. El-Ashhab, L. Sheha, F. Eltaboni, K. Elsherif, S. Gaballah *Benghazi University - Libya* Influence of microwave radiation on structure-property relationship of chitosan in solution	**PC 41**
M. Elrebii, S. Boufi *FSS - Sfax* Surfactant-free Waterborne Hybrid Alkyd–acrylic Dispersion: Synthesis, Properties and Long term stability	**PC42**
S. Eşiyok, G. Bayramoğlu, S. Şen *Yalova University - Turkey* Polystyrene-clay nanocomposıtes: Effect of phosphıne oxide ıntercalant for clay modıfıcatıon	**PC43**
G. Farhani, I. Bekri, E. Srasra *CNRSM - Borj Cédria* Synthesize of benzidine-clay hybrid material by solid-solid reaction and reaction of polymerization of benzidine in situ	**PC44**
Z. Ferchichi, A. Hedhli *ESSTT - Tunis* Synthesis, Characterization and Reactivity toward of hydrazine of α, ω ditosylates	**PC45**

M. Fersi, I. Chaabane, M. Gargouri *FSS - Sfax* Dielectric proprieties of the new organic-inorganic hybrid compound $[C_8H_{10}NO][ZnCl_3]$	**PC46**
H. Fodil, O. Mahmoud *Biskra University - Algeria* Physics properties and defect structure of Al-doped $SrFeO_{3-\delta}$	**PC47**
A. Gharbi, R. Bel Hassen, S. Boufi *FSS - Sfax* Olive stones flour from solid-waste of the olive oil industry as a filler in thermoset unsaturated polyester resin	**PC48**
R. Guermazi, G. Clavier, A. Hedhli, P. Audebert *ESSTT - Tunis* Synthesis, characterization and fluorescence properties of s-tetrazines	**PC49**
M. Guettari, A. Bitar, L. Ajroudi, A. Elaissari, T. Tajouri *IPEIT - Tunis* Characterization of poly(n-vinylcaprolactam) aqueous solution by a capillary viscometer	**PC50**
N. Haddadine, K. Benfattoum, N. Bouslah, A. Benaboura *USTHB - Algeria* Preparation and characterization of alginate-carbopol beads for oral insulin delevery	**PC51**
H. Hadj Ammar, S. Ajili, A. Bouraoui, H. Majdoub *FSM - Monastir* Physico-chemical characterization of extracted and degraded polysaccharide from Tunisian Algae: Antioxidant anti-inflammatory and anti-proliferative activity	**PC52**
Y. Hadj Kacem, A. Bougarech, M. Abid, S. Abid, R. El Gharbi, E. Fleury *FSS - Sfax* Synthesis and characterization of sulfonated aliphatic polyesters	**PC53**
J. Hafsa, B. Charfeddine, M.A. Smach, A. Letaif, L. Ben Othman, S. Neffeti, H. Dridi, O. Bouallegue, H. Majdoub, K. Limem, R. Sonia *Faculty of medicine - Sousse* Extraction of chitin and chitosan from shrimp shells (*Parapenaeus longirostris*): Physicochemical and biological characterization	**PC54**

S. Halladja, H. Ayadi, T. Mekhalif, Z. Boucerib *20 Août 1955 University, Skikda - Algeria* Adsorption of the BSA on the poly (acrylamide co acrylic acid) complexed with copper ion	**PC55**
S. Hrichi, A. Ladhar, H. Kaddami, M. Raihane, M. Arous *FSS - Sfax* Effect of coupling agent on the dielectric properties of low density polyethylene / short palm lignocellulosic fibers composites	**PC56**
K. Hriz, N. Jaballah, M. Majdoub *FSM - Monastir* New biphenylvinylanthracene-based semiconducting polymer synthesis and optical proprieties	**PC57**
F. Ibn El Haj Amor, F. Bouchnag, C. Ahmed *FSM - Monastir* Determination of affinity coefficient of cations exchange membrane at different ionic strengths	**PC58**
A. Imragaa *Benghazi University - Libya* Preparation and Physical Properties of Polymer for Photovoltaic Applications	**PC59**
H. Irinislimane, N. Belhaneche *National Polytechnic School - Algeria* Adsorption kinetics of two cationic dyes on starch from aqueous solutions	**PC60**
J. Isaad *ENSAIT, Roubaix - France* Colorimetric and fluorescent chemosensors for the toxic ions detection in envoronmental mediums	**PC61**
J. Isaad, A. El Achari *ENSAIT, Roubaix - France* Novels geotextiles for the selective capture of the heavy metals from wastewater based on bio-polymers grafted functionalized crown ethers	**PC62**
N. Jaballah, K. Hriz, M. Chemli, M. Majdoub *FSM - Monastir* New semi-conducting poly(arylene sulfide)s for opto-electronic applications	**PC63**

I. Jarraya, M. Kammoun, M. Abid, S. Abid, R. El Gharbi *FSS - Sfax* Characterization of partially sulfonated polyoxadiazoles bearing furan moieties	**PC64**
M. Kahraman, M. Eminoğlu, F. Beypınar *Marmara University - Turkey* Development of photo-cross linked polyethyleneimine nanofibers as CO_2 sensor	**PC65**
A. Kallel, H. Ben Romdhane *FST - Tunis* New poly(triazolo-imide)s obtained by click polyaddition	**PC66**
T. Kallel, N. Mnif, B. Elleuch *ENIS - Sfax* Analysis of the double effect of the coupling agent upon the structure and properties of short glass fibre polypropylene composite	**PC67**
M. Khemakhem, K. Lamnawar, A. Maazouz, M. Jaziri *ENIS - Sfax* Biocomposite based on polylactide acid and olive solid waste: Rheological, processing and mechanical studies	**PC68**
D. Khorramdel, A. Aghili *Islamic Azad University, Shiraz - Iran* Modeling of condensed mode fluidized bed reactors for polyethylene production at steady state conditions	**PC69**
F. Laatar, M.R. Ben Romdhane, E. Srasra *CNRSM - Borj Cédria* The effect of the incorporation of the clay on acrylic polymer	**PC70**
A. Labidi, M. Abderrabba *FST - Tunis* Synthesis and characterization of graft copolymers of potato starch and acrylonitrile	**PC71**
F. Lhumeau, C. Delaite, M. Kutnik *ENSC, Mulhouse - France* Cellulose acetylation by functional anhydride - A potential tool for the development of non-biocidal wood treatments	**PC72**
J. Longlade, A.S. Schuller, C. Delaite *ENSC, Mulhouse - France* New polymeric dispersant for titanium dioxide pigments: Synthesis and characterization	**PC73**

K. Mabrouk, N. Souissi *ISET - Zaguouan* Study of the effects of the temperature and composition on dynamic viscosity and density of vinyl acrylic emulsions	**PC74**
S. Madakbaş, F. Şen, M.V. Kahraman *Marmara University - Turkey* Structural, thermal and morphological properties of polyaniline / huntite composites	**PC75**
J. Mahfoufh, S. Salhi, S. Brogly, C. Delaite, S. Abid, R. El Gharbi *FSS - Sfax* Poly (ester-amide)s and polydepsipeptides based on aminoacids	**PC76**
C. Mangeney, M. Nguyen, S. Lau Truong, N. Felidj, J. Grand, L. Boubekeur, P. Decorse, G. Levi, J. Aubard *Paris Diderot University - France* Manipulation of bio-adhesion by thermoplasmonic activated phase-changes	**PC77**
N. Maouche, B. Nessark, M.M. Chehimi *Setif University - Algeria* Electrosynthesis, Characterization and Electrocatalytic Behaviour of Organic–Metal thin-Film based on 2,2'-Bithiophene-5-carboxylic Acid and Metallic Ions	**PC78**
D. Maraii, M. Dammak, J. Farjas, P. Roura *FSS - Sfax* Decomposition of Yttrium acetate in the form of films	**PC79**
F. Masmoudi, A. Bessadok, S. Belgaied, M. Jaziri, E. Ammar *ENIS - Sfax* Elaboration and characterization of plasticized starch film	**PC80**
F. Mbarki, F. Meganem *FSB - Bizerte* Chemical modification of polyvinyl chloride with aromatic and aliphatic amines. Synthesis, characterization and extractanting properties for some metals	**PC81**
H. Merine, H. Merine, K. Mehida *Djilalli Liabes University - Algeria* Delay effect evaluation of liberation of antipyrine dispersed in matrix copolymere based on N-2vinylpyrrolidone	**PC82**

R. Mesbeh, B. Hamdi, R. Zouari *FSS - Sfax* New organic-inorganic hybrid crystal structure of of 1,3,5-triazinidium-2,4,6-triamine hexachlorodicuprate (II)	**PC83**
A. Messaoudi, N. Hamdi, E. Srasra *CNRSM - Borj Cedria* Characterization of geopolymer synthesised from Tunisian illitic clay	**PC84**
A. Mezni, I. Balti, N. Jouini, L. Smiri, A. Mlayah *FSB - Bizerte* Hybrid Nanoparticles: plasmonic, SERS and phase transition properties	**PC85**
A. Mezni, A. Fkiri, L.S. Smiri, Adnen Mlayah *FSB - Bizerte* Facile synthesis of monodisperse triangular gold nanoplates based on a polyol process	**PC86**
S. Mhiri, A. Bougarech, M. Abid, S. Abid, R. El Gharbi *FSS - Sfax* Synthesis, characterization and hydrolytic degradation of new biobased (furanic-sulfonated copoly(ester-amide)s	**PC87**
R. Milad, M. Abderrabba *IPEST - La Marsa* Theoretical study of new conjugated polymers containing thiophene and 1,3,4-oxadiazole or 1,3,4-thiadiazole in the main chain	**PC88**
M. Mouffok, I. Abdelmalek, A. Mesli *Djilalli Liabes University - Algeria* Preparation of the poly (N-2-vinylpyrrolidone-co-vinyl acetate) for application matrix-type loaded microspheres of 2-aminobenzothiazole	**PC89**
M. M'Sahel, R. Medimagh, E. Drockenmuller, M.S. Zina *INRAP - Sidi Thabet* Synthesis and characterization of novel bio-based platform from dianhydrohexitols for application in polymers synthesis	**PC90**
A. Saadaoui, R. Medimagh, S. Marque, S. Chatti, D. Prim *INRAP - Sidi Thabet* New biosourced AA and AB monomers from 1,4:3,6 dianhydrohexitols	**PC91**

H. Salhi, S. Ayadi, M. Abderrabba *IPEST - La Marsa* Theoretical study of the copolymer based on: carbazole and 4,7-di(thiophen-2-yl)-2,1,3-benzothiadiazole	**PC92**
S. Salhi, A. Ali Mohamed, S. Abid, R. El Gharbi, A. Fradet *FSS - Sfax* Random and alternated polyesteramides derived from β-alanine and ε-caprolactone	**PC93**
K. Seffah, A. Zafour-Hadj-Ziane, E. Flahaut *Saad Dahlab University, Blida - Algeria* Double-walled carbon nanotube dispersion via biopolymers	**PC94**
L. Seid, D. Chouder, N. Maouche *Setif University - Algeria* Doping polypyrrole particles by removing of Cd^{2+}, Co^{2+} from aqueous solutions	**PC95**
F. Şen, S. Madakbaş, M.V. Kahraman *Marmara University - Turkey* Polypyrrole / hexagonal boron nitride nanocomposites	**PC96**
S. Seridi, S. Badi, A. Seridi, M. Berredjem, M. Kadri *Guelma University - Algeria* Host-guest inclusion complex between β-cyclodextrin and sulfonamide: A theoretical approach	**PC97**
M. Stouri, I. Bekri, E. Srasra *CNRSM - Borj Cédria* Valorization of polystyrene waste foam as polymer/clay nanocomposite: Application as methanol fuel cell membrane	**PC98**
S. Teka, K. Hriz, N. Jaballah, M. Majdoub *FSM - Monastir* Synthesis and characterization of semiconducting polyrotaxane based on anthracene and β-cyclodextrin	**PC 99**
L. Toukal, N. Maouche, B. Nessark, M.M. Chehimi *Setif University - Algeria* Synthesis and characterization of copper/ poly (5- aldehyde-2,2',5',2"-terthiophjene) composite material and its application for electrocatalysis	**PC100**
W. Trigui, A. Oueslati, H. Faouzi *FSS - Sfax* Crystal structure, thermal analyze and vibrational spectroscopy of the new compound $[(C_4H_9)_4N]_3Bi_2Cl_9$	**PC101**

M. Trimeche, R. Ben Cheikh, H. Oudadesse, H. Smaoui, H. Keskes, T. Rbaï *ENIT - Tunis* Characterization of a new composite bone implant : Polyhydroxybutyrate -co -3- hydroxyvalerate - phosphate ceramic (CPC- PHBV)	**PC102**
F. Walha, K. Lamnawar, A. Maazouz, M. Jaziri *ENIS - Sfax* Morphological, rheological and mechanical studies of bio-sourced polymer blends based on polylactide-acide and polyamide 11	**PC103**
N. Weslati, I. Chaabane, F. Hlel *FSS - Sfax* Synthesis, crystal structure, thermal analyze and Raman spectroscopy of the new compound $[(C_3H_7)_4N]SbCl_4$	**PC104**
S. Yedbri, S. Louhibi *Tlemcen University - Algérie* Nouveaux complexes de cobalt(III) avec des ligands amides. Synthèse et caractérisation	**PC105**
R. Zidi, I. Bekri-Abbes, E. Srasra *CNRSM - Borj Cédria* Anticorrosive and dielectric properties of polypyrrole/ Na^+ – montmorillonite (PPy/Na^+ –MMT) clay nanocomposites	**PC106**
S. Zouaoui, A. Ben Hadj Amor, F. Meganem *FSB - Bizerte* Adsorption of heavy metals onto oxidised and cross-linked corn starch	**PC 107**
H. Zrida, K. Hriz, N. Jaballah, M. Majdoub *FSM - Monastir* New anthracene-based π-conjugated polymer: Synthesis and optical proprieties	**PC 108**

Posters Communications' Abstracts

CHARACTERIZATION AND MODIFICATION OF LIGNIN BIOPOLYMER

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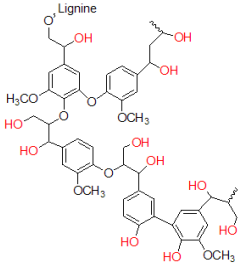
The negative impact of petrochemical-based platform chemicals and industrial commodities has led to use of green materials to reduce toxic emission and reduce energy demand. As a consequence there has been focus on the use of natural polymers and biopolymers. Lignin is a renewable resource produced in large quantities during pulping and, until now, mainly used to generate energy. Only a small percentage is used in the field of material, mainly because of its structural heterogeneity and complexity, which, among others, depend on the plant origin and on the separation and purification method.

This communication concerns our recent study on the extraction, characterization of alfa and wheat straw lignin. An original organosolve process developed by CIMV (*Compagnie Indusriel de la Matière Végatale*), based on the use of a mixture formic acid/ acetic acid/water as cooking reagent allowed the destructuration of alfa and wheat straw, to give selectively cellulose, lignin and hemicelluloses [1]. Lignin was extracted and purified separately from hydrolysates and residual solids after treatment. The chemical structure of lignin was characterized through FT-IR, SEC, ^{1}H, ^{13}C-^{1}H 2D HSQC. The quantification of aliphatic, phenolic and carboxylic OH groups was determined by ^{31}P NMR [2]. Lignin extracted from the CIMV process was a low molecular weight polymer with a relatively low polydispersity index and high free hydroxyl group content.

The second part of this work was carried out on the chemical modification of CIMV wheat straw lignin, structurally close to alfa lignin, in order to improve its reactive and its solubility in organic solvents. This modification was achieved by esterification with acetyl and hexanoyl chlorides in pyridine solution at low temperature. The FT- IR and ^{1}H NMR spectra proved that lignin modification took place.These changes were reflected by the results of thermal analyses such as thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) and by Size Exclusion Chromatography (SEC). The potential applications of lignin as precursor for the elaboration of new polymeric materials will also be discussed.



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Synthesis and characterization of fluorinated quaternary ammonium surfactant intended for mesoporous silica nanoparticles

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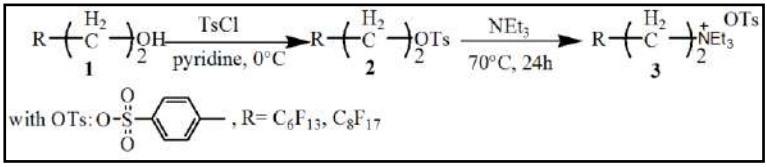
Fluorinated surfactants are more efficient than hydrogenated surfactant since their surface tensions are lower. Hydrogenated surfactants are used in various domains as emulsifiers, wetting agents, detergents, or solubilizers. Both families adopt similar behavior in solution, and those surfactants can be employed for the preparation of mesoporous materials.

In a previous study, silica nanoparticles have been prepared by sol-gel method using quaternary ammonium surfactant (cetyltrimethylammonium bromide C_{16}TAB and C_{18}TAB) as porogen in a basic medium.

In this study and in order to compare between the specific properties of the materials synthesized with the hydro-carbonated surfactant cited above and those with the fluorinated surfactant with always the aim to prepare new silica nonoparticles, we have interested to the synthesis of new fluorinated quaternary ammonium, in this case the N, N, N-triethyl-F-alkyl-1-ammonium tosyl ($R(CH_2)_2N(Et)_3$,OTs) was chosen. It was synthesized with an addition of the 4-Toluenesulfonyl chloride (TsCl) to the alcohol **1** in presence of pyridine as a base leading to a tosylate **2**. Then, the ammonium salt **3** was obtained with an addition of the triethylamine to this tosylate (Sheme1).



Sheme1. Synthesis of the N, N, N-triethyl-F-alkyl-1-ammonium tosyl

The fluorinated quaternary ammonium having the structure $R(CH_2)_2N(Et)_3$,OTs (with R: C_6F_{13} and C_8F_{17}) are obtained with acceptable yields. These products were characterized by ^{1}H NMR, ^{13}C NMR and ^{19}F NMR.

SYNTHESIS AND CARACTERIZATION OF POLYESTERAMIDE DERIVED FROM BIS (2-OXAZOLINE), ANHYDRID AND DIOL STUDY OF DEGRADATION

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Environmental issues raise concern on restrict the use of non degradable polymers and encourage the development of degradable once. Poly (ester-amide)s are an emerging group of biodegradable polymers that may cover both commodity and specialty applications. These polymers have ester and amide groups on their chemical structure which are of a degradable character.
This cadre our study consists to prepared the poly (ester-amide) by reacting bis (2-oxazoline), carboxylic anhydride and diol. The polymerization proceeds through the formation of a dicarboxy In ester by reaction of two anhydride molecules with one diol molecule and subsequent 2-oxazoline ring opening by attack of the dicarboxy ester[1]. The polymers resulting from polycondensation were characterized by means of FTIR, ^{1}H-NMR, ^{13}C-NMR, and determination the viscosimetric mass. Hydrolytic degradation of the polyesteramide was examined in acidic or basic aqueous solutions of varying pH [2] and biodegradation of polymer in a liquid medium activated sludge, followed by kinetic the biochemical oxygen demand (BOD) [3].
The poyesteramide studied reveal excellent hydrolytic degradability, depending on the acidic and basic conditions of the Dioxane solution and the biodegradation of polymer was practical by evolution of BOD.

Key words: □ degradation, poly (ester-amide), polycondensation, bis(2-oxazoline).

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Nanofibrillated cellulose: Effect of the hemicelluloses content and the fibers' morphology on the nanofibrillation efficiency of cellulose fibers

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LMSE-Faculté des Sciences de Sfax

The extraction of nanosized fibres from cellulose fibres undoubtedly represents most of the breakthroughs in cellulose-based materials during the last decade. The nanofibrillated cellulose (NFC) extraction process depends on two main factors: (i) the biosynthesis of the crystalline cellulose microfibrils, which is dependent on cellulose source material, and (ii) the extraction process of the cellulose fibrils from cell walls calling on an intensive mechanical disintegration process. Today, one of the most challenges to face resides in the reduction of the energy demand during the production of NFC.

In the present work, the impact of hemicellulose content and fibre morphology on the nanofibrillation behaviour of delignified cellulose pulps was studied. For this purpose, pulps from two non-woody plants, Alfa (Stipa tenacissima) and Sunflower (Helianthus annuus), were delignified using $NaClO_2$/acetic acid and the NaOH pulping procedures to obtain fibres with different hemicellulose contents. The ensuing pulps were characterized by chemical analysis, SEM, FTIRS and XRD and then fibrillated using high pressure homogenizer and a domestic blender to obtain cellulose nanofibrils (NFC).

Synthesis and Properties of Thermotropic Liquid Crystalline Poly(azomethine-ether)s Containing 1-Phenethyl-4-Piperidone Moiety

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A novel series of poly (azomethine-ether)s containing 1-phenethyl-4-piperidone moiety was synthesized by solution polycondensation of various diformyl-α,ω-diphenoxyalkanes, I-VIII with 3,5-bis(m-aminobenzylidene)-1-phenethyl-4-piperidone X, and characterized by ^{1}H-NMR, IR and elemental analyses. The inherent viscosities of the polymers were in the range 0.45-0.65 dI/g. All the poly (azomethine-ether)s were insoluble in common organic solvents but dissolved completely in aprotic solvents. The mesomorphic properties were studied as a function of the diphenoxyalkane space length. Analysis by DSC and optical polarized microscopy demonstrated that the poly (azomethine-ether)s form nematic mesophases over wide temperature ranges.

CELLULOSE BASED AEROGEL AS AN ADSORBENT FOR METALLIC CATIONS

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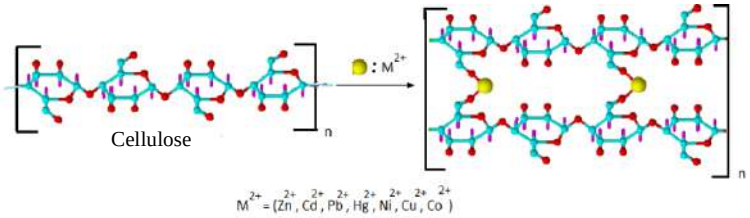
Cellulose aerogel prepared from corsslinked nanofibrillated cellulose was prepared, characterized and used as a sustainable adsorbent for the removal of heavy metallic cations. The elaboration of the adsorbent involved three steps; (i) the first one is the preparation of the nanofibrillated cellulose by high pressure homogenization, (ii) then the aerogel was prepared by cryo-solvent exchange process, and finally (iii) a chemical reticulation with tri-isocyanate was implemented in order the preserve the physical integrity of the adsorbent in water.

The adsorption properties of the cellulose based adsorbent toward a large range of metallic cationic were investigated under batch condition. The adsorption isotherms were analysed using both Langmuir and Freundlich isotherm models from which the thermodynamic adsorption parameters were evaluated. FTIR and zeta-potential measurement have shown that the surface complexation and ion exchange were the major mechanism involved in the adsorption process. The reusability of the new cellulose adsorbent was also studied. It was shown that once exhausted the adsorbent might be reused by treatment with solution of EDTA at pH 5-6.



Keywords: Adsorption, Heavy metal ions, Cellulose.

LYSOZYME-IMPRINTED POLYACRYLAMIDE THIN LAYERS ON PVC BEADS

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The objective of this study is to prepare thin films of MIPs on PVC beads for purification of lysozyme. The acrylamide (AM) was selected as the functional monomer, and N, N, methylenebisacrylamide (MBAA) as crosslinking agent; they interact with lysozyme to form a complex in the prepolymerization step. In order to confine the polymerization to the surface of the grains, we grafted iniferter initiator (sodium diethyldithiocarbamate, DEDTC) to the surface of the beads of PVC. We obtained core/shell structure (PVC/ MIPs) with the immobilized protein in the shell which was extracted after the cleaning step. MIPs are then used as artificial receptors for the rebinding of lysozyme. We prepared the non-imprinted polymer (NIP) in the same way but in the absence of the template molecule (PVC / NIP). We have demonstrated by XPS and infrared spectroscopy that PVC is covered with a thin layer of MIP (or NIP). The binding properties of biomimetic beads were determined using UV spectroscopy from adsorption isotherms of lysozyme and myoglobin. The imprinted beads were found to be highly specific and selective towards lysozyme. This work shows the interest of MIPs for the uptake of proteins, but more importantly emphasizes the major interest to produce biomimetic polymeric materials as thin layers to develop rapid detection system.

Keywords: molecular imprinting, protein, PVC beads, thin coatings, iniferter

EXTRACTION DES NANOCHARGES DU LIEGE, ELABORATION ET CARACTERISATION D'UN MATERIAU COMPOSITE A MATRICE BIODEGRADABLE DE PHBV.

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National engineering school of Tunis (ENIT)

Polymer materials reinforced with synthetic fibres such as carbon fibres or glass fibres are widely used in various application fields such as the automotive, construction and aerospace industries.

Currently, oil polymers price increases sharply [1]. In addition, composite materials formed from such products generate dying tons of non degradable wastes which are difficult to recycle.

Composites elaborated with biopolymers reinforced by natural fibres can solve these problems of waste and provide some interesting properties such as biodegradability, lightness, rigidity... [2].

This work is devoted to the study of a vegetable fiber which is cork for the development of a lightweight thermoplastic biocomposite, having a lower cost and respecting the environment. The biocomposite is made with a matrix of PHBV and cork nanofibres extracted from the tunisian suberais.

Key words: PHBV, Cork, Biocomposite, Nanofibres, Biodegradability

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MORPHOLOGY AND STRUCTURE OF BIO-BASED MICROCAPSULES FOR PERFUME RELEASE.

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Microencapsulation is an effective method for save the properties and control release of the encapsulated active product.[1, 2] New polyurethane microcapsules based on renewable materials and containing perfume were designed for cosmeto-textile application. The polymer wall was synthesized by interfacial polycondensation technique.[3] The chemical structure of the microcapsules was characterized in details by IR spectroscopy. The core-shell morphology of the microparticles was confirmed using optical microscopy and scanning electron microscopy (SEM). The diameter distribution was revealed by means of small-angle light scattering measurement. The microparticles were deposited onto textile fabrics by an impregnation process using a binding agent.[4, 5] The durability and the perfume release behavior of the impregnated textile were evaluated through SEM, UV–visible spectroscopy and gas chromatography. The cosmeto-textiles released slowly its microcapsule content and the perfume remained until twentieth washing cycles.

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ADSORPTION OF METALS BY EDTA-CHITOSAN

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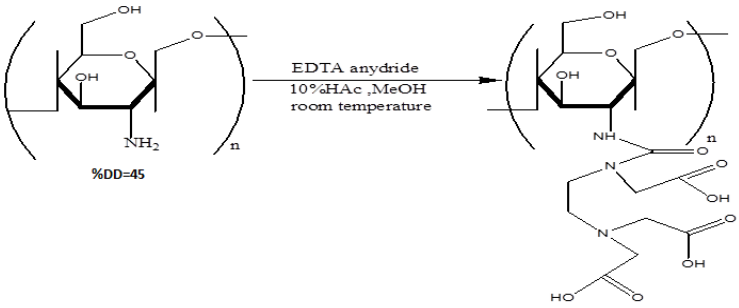
The aim of the present study was to investigate the adsorption properties of surface modified chitosan in aqueous metals solutions. For this purpose, ethylene diamine tetra acetic acid (EDTA) was immobilized onto polymer matrices of chitosan.
Adsorption of metals by prepared adsorbents was investigated in batch techniques. The effects of pH, contact time, and the concentration of metals were studied.
These tests demonstrated that very clear-cut mutual separations between these pairs of metal ions can be successfully achieved by these complexing agent types of chemically modified chitosan.



Synthesis of EDTA-chitosan

Key words: Modified chitosan , Adsorption ,EDTA ,metals.

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INVESTIGATION OF THE INCLUSION PROCESSES OF ADRENALINE WITH β -CYCLODEXTRIN: A COMPUTATIONAL STUDY

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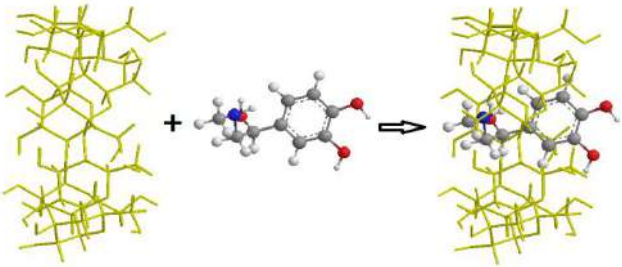
Adrenaline is one of the most frequently used in the treatment of cardiovascular diseases [1]. Cyclodextrins are known to form inclusion complex with many kinds of compounds [2].In this study geometry optimizations of 2-benzothiazolethiol /β-Cyclodextrin complex were carried out using PM6, HF, and ONIOM methods. The most stable structure was obtained at the optimum position and angle. The geometry of the most stable complex shows that the aromatic ring is deeply self-included inside the hydrophobic cavity of β -CD also an intermolecular hydrogen bond is established between host and guest molecules. Furthermore, the quantification of the donor–acceptor interaction between host and guest molecules is performed via Natural Bond Orbital (NBO) analysis .This suggests that hydrophobic effect and hydrogen bond play an important role in the complexation process.

Key words: adrenaline, β-cyclodextrin, inclusion complexes, quantum mechanics.



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PREPARATION, THERMAL AND MECHANICAL PROPERTIES OF UV CURABLE POSS EPOXY HYBRID COATINGS

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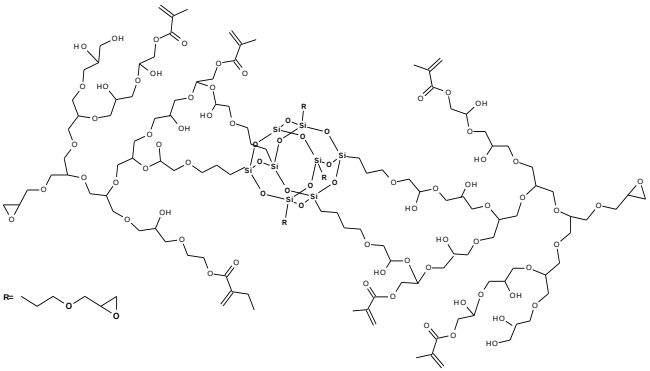
Photopolymerization is a high-speed process where UV light induces the polymeric film formation leading to a fast transformation of the wet film into the solid film. It offers many advantages such as low energy consumption and less environmental pollution that are of great interest in industrial applications. Owing to the increasing necessity of physically and mechanically improved materials due to developing technology, researchers head towards new materials. Therefore many different attempts have been made to obtain materials, which combine mechanical toughness and flexibility of the organic solids[1]. The development of hybrid organic polymer-inorganic nanocomposites with a variety of new and improved properties have attracted considerable attention due to their lightweight character and excellent thermal and mechanical properties[2]. By incorporating cross-linkable side chains, the hybrid materials can be made photosensitive and UV curable, which offers many advantages including low processing temperature, low equipment cost and compatibility. Polyhedraloligomeric silsesquioxanes (POSS) reagents offer a unique opportunity for preparing hybrid organic-inorganic materials. Functionalized POSS can be incorporated into any existing polymer system through grafting, copolymerization or blending[3]. By this was the inorganic structural units truly molecularly dispersed within the nanocomposites.

In this study UV curable methacrylated polyglycidol (MAPG) oligomer was synthesized and hybrid coatings were prepared using glycidyl POSS and MAPG. The structures of oligomers were characterized by FT-IR. UV curable clear hybrid coatings were prepared by mixing MAPG, POSS modified MAPG and photoinitiator (Irg-184) then applied on aluminum substrates. The physical and mechanical properties of UV curable clear hybrid coatings such as gel content, solvent resistance, hardness, gloss, flexibility and tensile tests were examined. In addition, the thermal behavior of coatings was also evaluated.



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ELECTRICAL AND STRUCTURAL PROPERTIES OF CHLOROTHIOPHENES AS MONOMERS FOR NEW CONDUCTING

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Electronically conducting polymers possess different properties related to their electrochemical behavior. The importance and the potentiality of this class of materials were recently recognized by the world scientific community when H. Shirakawa, A.J. Heeger and A.G. MacDiarmid were laureate in 2000 with the Nobel Prize in Chemistry by their research in this field [1–4].

Conducting polymers are frequently called ''synthetic metals'' because they present electric, electronic, magnetic and optical properties inherent to metals or semiconductors, while retain the mechanical properties of conventional polymers. In intrinsic conducting polymers, the conductivity is assigned to the delocalization of π-bonded electrons over the polymeric backbone, exhibiting unusual electronic characteristics, such as low energy optical transitions, low ionization potentials and high electron affinities [5]. Electron delocalization is a consequence of the presence of conjugated double bonds in the polymer backbone.

Electrical and structural properties of mono-, di-, tri- and tetrachlorothiophenes and their radical cations have been studied using the ab initio/MP2 method with 6-311+G** basis set. The effects of the number and position of the substituent of chlorine atoms on the properties of the thiophene ring for all chlorothiophenes and their radical cations have been studied. Vibrational frequencies, spin-density distribution, size and direction of dipole moment vector, ionization potential, electric polarizabilities and orbital energy spacings including HUMO-LUMO gap (HLG) and electronic spatial extent (ESE) of these compounds have been calculated as well. The analysis of these data showed that double bonds in 3-chlorothiophene are more delocalized and it is the best possible candidate monomer among all chlorothiophenes for the synthesis of corresponding conducting polymers with modified characteristics

Analysis of the results of this study shows that 3-chlorothiophene molecule has the smallest value of the ***Fn*** coefficient while thiophene radical cation has the smallest value of the *Fn* coefficient. Therefore, it can be suggested that double bonds in 3-chlorothiophene are more delocalized among all chlorothiophenes molecule and thiophene are more delocalized among all radical cations.

Electrochemical stability of 3-chlorothiophene and 3,4-dichlorothiophene is greater than other chlorothiophenes. Thermal stability and Zero-Point Energy of thiophene are greater than all chlorothiophenes. Accordingly, compound B (3-chlorothiophene) is the best candidate monomer among all chlorothiophenes for the synthesis of corresponding conducting polymers with modified characteristics.

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NEW BIO-BASED VINYL MONOMERS FROM DIANHYDROHEXITOL ISOMERS AND/OR FURAN

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Hatem BEN ROMDHANE [a] and Saber CHATTI [d]

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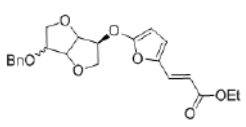
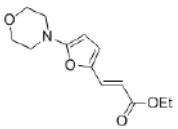
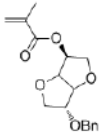
One of the most important items on the agenda of the global chemical industry is the substitution of petroleum-based feed-stocks by those from renewable sources. Hence, the use of biomass-derived building blocks has been investigated in the chemical and commodity industries. Among the important byproducts of biomass are the dianhydrohexitols (DAH) obtained in the sugar industry by the double dehydration of starch.
In this work we report the preparation of new vinyl monomers derived from DAH isomers (isosorbide, isomannide) and furan (**1a-1d**).



(1-a, 1-b)

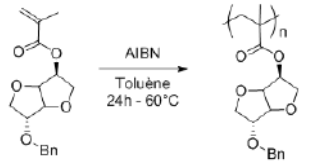


(1-c)



(1-d)

The benzylisosorbide polymethacrylate **BIPMA** [1] prepared by the radical polymerisation of monomer (**1-d)** was characterized by ^{1}H and ^{13}C NMR, FT-IR and SEC. Thermal properties of the biopolymer were investigated by differential scanning calorimetry (DSC).



1-d **BIPMA**

BIPMA has an Mn = 14 kDa with $Đ$= 1,35 and DSC results demonstrated its amorphous nature with a glass transition temperature Tg = 147°C. These features make this material as a promising new class of biopolymers.

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THE ADSORPTION OF THE ACIDIC TEXTILE DYES ONTO CHITOSAN

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Chitosan is a linear polysaccharide, obtained by the deacetylation of the shells chitin **(Fig. 1)**. In acidic medium, the positively charged amino groups of chitosan readily interact with the anionic groups of textile dyes through an adsorption phenomenon. For this reason, we have explored the possibility of removing of two acidic red colorant, AR 183 and AR 114, from wastewater by using such process.

In this study, the interaction between the chitosan cationic groups and the AR anionic groups has been investigated by the conventional study of the adsorption process and the cyclic voltammetry approach.

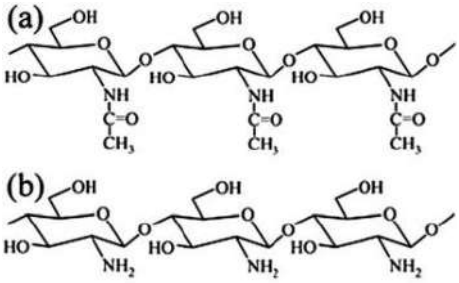

Fig. 1 Structures of (a) chitin and (b) chitosan

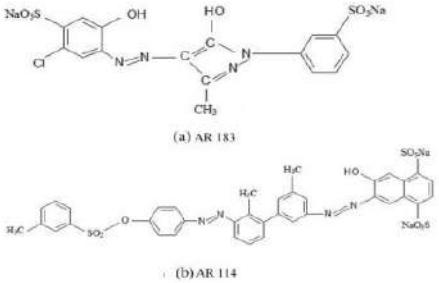

Fig.2 Structures of AR 183 and AR 114

SYNTHESIS OF PVP/ PEG HYDROGEL AND EFFECT OF AGAR ON THE SUPER ABSORBING PROPERTIES OF THE COPOLYMER.

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Polymerization process with Gamma irradiation technique is an important method for obtaining superabsorbent polymer whose composition is based on polyvinyl pyrrolidone (PVP), poly (ethylene glycol) (PEG) and agar used as cross linking agent.

In this study, the effect of agar proportion on the swelling behavior of the hydrogel was firstly investigated and showed that increasing the proportion of agar leads to an increase in the cross linking density. The pH and time dependent swelling was studied and showed that the swelling decrease is proportional to the increase of the initial amount of agar in the hydrogel [1]. The graft copolymer was characterized by Fourier transform infrared spectra analysis. The removal of Cu(II) from aqueous solution by the prepared PVP/PEG/agar chelating hydrogel was studied and the influence of pH and contact time were investigated in the absorption experiments.

Our results showed that pH = 3 and 4,5 goes with the adsorption maximum of 1% and 2% of agar hydrogels, respectively. It must be mentioned that since we act in acidic solutions, the absorption capacities of hydrogels were low due to the presence of hydronium groups which induced an electrostatic repulsion of Cu (II) ions. As for Rate of metal ion absorption are concerned, the observed differences in the retention duration of copper are explained by a more developed three dimensional network of the 2% agar hydrogel; which allows an easier integration of copper ions into this network [2].

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Optical characterization of PVK/ZnSe hybrid nanocomposites for photovoltaic application

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Polymer/inorganic nanocomposites have attracted much attention due to their low cost, substrate flexibility [1], ease of processing [2] and size tunable absorption-emission properties. For the polymer component, poly(N-vinylcarbazole) (PVK) is a good hole transporting material in photovoltaics [3]. It has been mixed with inorganic compounds to achieve good conductivity [4] for new promising optoelectronic devices. In this work, optical properties of hybrid organic/inorganic PVK/ZnSe layers are investigated as a function of the nanoparticle volume fraction using the effective medium model (EMM). The layers are deposited by spin coating technique on glass substrate and their transmission and reflection spectra were performed in the range 200-800nm. Optical constants such as refractive index n and extinction coefficient k are extracted and injected in the EMM formula to determine dielectric permittivity ε and absorption coefficient α. The optical band gap of the hybrid structure was also obtained as a function of nanoparticle volume fraction. The results show an increase of band gap energy after ZnSe doping (from 3.52 eV to 3.42 eV), depending on ZnSe concentration. Structural characterisation of the PVK/ZnSe nano-composite films was carried out using scanning electron microscopy.

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DEVELOPMENT AND CHARACTERIZATION OF A BIOCOMPOSITE BASED ON BIODEGRADABLE MATRIX, NANOCHARGES EXTRACTED FROM MARINE PLANTS AND ACTIVE PRINCIPLES; AND MODELING THE KINETIGS OF DELIVERY OF THE ACTIVE SUBSTANCE; SUCH AS: IBUPROFEN, β CETAMOL, CAFEINE, SALICYCATE…

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The pharmaceutical industry is looking for new drug forms, gradually releasing the active ingredient in order to overcome the defects of conventional galenic forms, in particular, avoiding the need for repeated administration. Moreover, more recently there has been great interest in the development of vectors for medication using nanoparticles, microparticles composed of biodegradable polymers. Polymer matrices modify the release, the pharmacokinetics and distribution of active principles in the body

For our work we have considered, in a first step, the extraction of biodegradable nanocharges from natural plant Posidonia Oceanica marine origin, and through dissolution under reflux in a solution of NaOH followed by acid hydrolysis.

Then we proceed to develop the composite formed substantially of nanofillers PO, biodegradable polyesters (such as PLA and PHA), and the active principle (such as ibuprofen, salicylates, β Cétamol, Caffeine, ...), then its characterization using spectral analysis to study the morphology of the different mixtures and to better understand the various existing interactions between the active ingredient and the matrix in question.

Finally, the last objective is modeling the kinetics of delivery of the active substance for each formulation.

EXTRACTION OF CELLULOSE MICROFIBERS FROM THE ALFA AND THE ELABORATION AND CHARACTERIZATION OF A PHBV / MFC BIOCOMPOSITE.

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The Alfa is the arab name of the esparto grass or stippa tenacissima plant, widely cultivated in the dry region of north Africa and especially in the center of Tunisia, the Alfa stem is built of strong, stiff and light cellulosic fibers which are mostly used in the production of high quality papers, dielectric applications for condensers and composites[1].

This work is devoted to the study of a vegetable fiber for the development of a lightweight thermoplastic biocomposite, having a lower cost and respecting the environment [2]. The biocomposite is made with a matrix of PHBV and Alfa microfibers.

We approach in this work the study and the characterization of Alfa fibres before and after its basic and acid treatment. The techniques used for this characterization are the electronic scanning microscopy (SEM), the energy dispersive X-ray spectrometry, and Fourier transform infrared spectroscopy(FTIR). The Thermal parameters of PHBV/Alfa biocomposite were determined by differential scanning calorimetry (DSC). This technique showed their decrease after reinforcement adding.

Key words : biocomposite, PHBV, Alfa, microfiber, cellulose.

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SYNTHESIS AND STRUCTURAL STUDY OF COPOLYESTERS BASED ON BIS(2-HYDROXYETHYL TEREPHTHALATE) AND VARIOUS LACTONES

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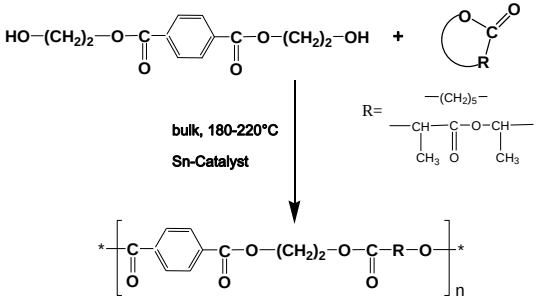
Every year, several hundred thousand tons of non-degradable plastic products are introduced into the environment. The continuously increasing extent of pollution of the environment has recently given rise to demands for novel biodegradable polymers mainly for applications related to food packaging [1]. Poly(ethylene terephthalate) (PET) is been known as industrially important polyester which is widely used as commercial fibers and engineering plastics. Post-consumer PET can be recycled into new items or into monomers or oligomers which are generally hydroxy-terminated and can be used for the synthesis of derived polyesters of PET such as PET – poly(lactide), PET –poly(glycolide)[2]

In this paper, we describe the synthesis of novel biodegradable aliphatic-aromatic copolyesters PET–lactones prepared by ring-opening polymerization (ROP) from bis (2-hydroxyethyl terephthalate) (BHET) and ε-caprolactone or lactide respectively. The BHET was previously synthesized by glycolysis of PET waste (plastic bottles) in the presence of ethylene glycol and cobalt acetate as catalyst.



Various copolyesters were synthesized by using different mass ratio ranging from 90/10 to 10/90 (PET-PCL or PET-PLA) and were characterized by FTIR, ^{1}H NMR, SEC, DSC and TGA. The microstructure of these copolyesters was determinate by ^{1}H NMR, showing the insertion of aliphatic units into PET chains. Copolyesters thermal properties were studied by DSC and TGA. The resulting materials are amorphous and semi cristallin polymers with good stability; their decomposition temperature can reach 370°C.

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FORMULATION AND CHARACTERIZATION OF A DRUG DELIVERY SYSTEM BASED ON PHBV COPOLYMER MATRIX

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Drug delivery systems (DDS) based on biodegradable polymer matrixes have become increasingly importance, mainly due to the non-toxicity, biocompatibility and efficiency of this type of matrix. The Polyhydroxyalkanoates (PHAs) matrix, particularly the poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), a biodegradable thermoplastic copolymer produced by renewable resources, is increasing continuously for the elaboration of microspheres systems by the technique of microencapsulation using solvent evaporation.

The obtained polymer microspheres, with drugs trapped inside, can degrade, and release the encapsulated drugs slowly, with a specific release profile. This controlled drug release, has outstanding clinical benefits: reduction of dosing frequency, more convenience and acceptance for patients, and drug targeting to specific locations resulting in a higher efficiency.

In this work, we propose to elaborate microspheres systems based on PHBV matrix, charged with cork powder issue from Quercus suber L trees from the north west region of Tunisia, in which diclofenac sodium (DS), a non steroidal anti-inflammatory, is dispersed and trapped.

The microspheres are analyzed by scanning electronic microscopy (SEM) in order to determine the morphology and size of the particles. Infrared spectroscopy and differential scanning calorimetry are performed to verify that there are no chemical interactions between drug and matrix, and that the drug is stable in the formulated microspheres product.

In-vitro drug release studies are performed, in order to understand the kinetics and mechanisms of the drug release and to determine the best kinetics models.

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Highly transparent nancomposite films based on polybutylmethacrylate and functionalized cellulose nanocrystals

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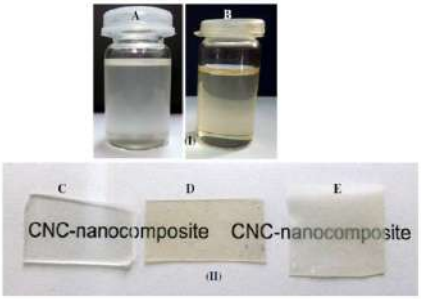
Efficient surface functionalization of cellulose nanocrystals (CNC) with hydroxyl butyl acrylate monomer (HBA) was carried on under mild condition using N,N'-carbonyldiimidazole as an activator. The grafting of the acrylic monomer was shown to bring about high yield grafting of polymer chains on the functionalized CNC during the in situ polymerization process. Surface functionalization of CNC with HBA and the polymer grafting on the modified CNC were confirmed by Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy. The grafting of polymer chains was found to bring on the effective dispersion in organic solvent over a long time without any coagulation, nor any sedimentation. Nanocomposite film prepared from in situ polybutylmethacrylate polymerization process using HBA functionalized nanocrystals exhibited high transparency degree here assigned to improved dispersion. DMAanalysis proved that the composite storage tensile modulus is highly increased in the rubbery region for HBA–CNC contents of 4 %, this composition yielding the best mechanical/rheological performance.



(I) Visual aspect of PBMA-CNC solution in toluene at a content of 0.5 %(based on CNC) using ***A*** *unmodified CNC*; and ***B*** *HBA-modified CNC*
(II) Visual aspect of ***C*** *PBMA matrix;* ***D*** *PBMA nanocomposite films containing 4 % HBA modified CNC*; and ***E*** *PBMA nanocomposite films containing 4 % of unmodified CNC,* prepared via in situ polymerization

SYNTHESIS OF DIMERS AND COPOLYMERS FROM ALDEHYDES UNDER HOMOGENEOUS AND HETEROGENEOUS MEDIUM

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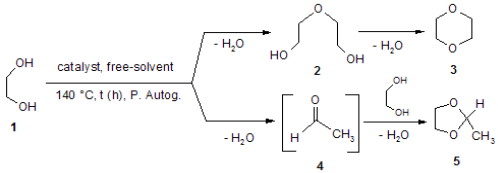
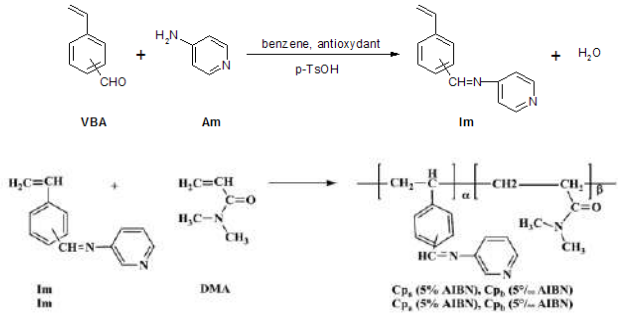
We have shown in the first part of this study that ethylene glycol **1** is unstable in the presence of sulfuric acid, silica gel, synthetic silica, alumina, zeolite and acid activated clays under solvent free. The yield of diethylene glycol **2**, 1,4-dioxane **3,** acetaldehyde **4,** and 2-methyl-1,3-dioxolane **5** depended closely on the experimental condition and the nature and acidity of various catalysts used. The products **2-5** were resulted from a competitive between linear dimerisation and cyclization dimerisation.



On the other hand, m,p-vinylbenzaldehyde **VBA** is reacted with 3-aminopyridine in the presence of para-toluene sulfonic acid to get N-(m,p)-vinylbenzyliden-3-aminopyridine **Im**. The monomer **Im** is then copolymerized with N,N-dimethylacrylamide **DMA** to afford a mixture of copolymers $\mathbf{Cp_a}$ and $\mathbf{Cp_b}$.



KEY WORDS: acetaldehyde, (m,p)-vinylbenzaldehyde, dimerisation, copolymerisation, dioxolane.

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Effects of OH-bearing matrices for copolymerization routes

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Nowadays there is a rapid evolution of materials in the context of sustainable development [1]. These materials are said composite materials consisting of a polymer matrix and a fibrous reinforcement [2]. The current trend is the use of vegetable fibers [3]. The used matrix is constituted by a biopolymer based on cellulose derivatives [4].

The aim of this work is the elaboration of biocomposite materiel obtained by biopolymer matrices Methyl cellulose and hydroxypropyl cellulose by compatilibisation between vegetal fibre (hydrophilic) and matrix (hydrophobic) like copolymerisation process (chemical grafting) [5,6,7].

The thermal and viscoelastic properties of blend were investigated via differential scanning calorimetry (DSC) and dynamic mechanical analysis (DMA) [8].

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DEVELOPMENT OF BORAX MODIFIED STARCH-BASED PLASTICS

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The foremost interest in this research is assaying the potential of sodium tetraborate decahydrate (borax) in the plasticisation of agro-based biopolymers such as starch and cellulose to develop starch-based plastics via compression molding. These compression moldable materials have benign nature and biodegradability and could be employed as alternative to MDF which uses environmentally non-benign adhesive *i.e.* urea-fomaldehyde. Cornstarch together with wood flour, straw flour, blue roll, orange peels and banana peels were exploited. The starch was the thermoplastic component and the celluloses were the filler reinforcements. As starch cannot form true thermoplastics without plasticisers, different borax systems *viz.* saturated aqueous borax solution, 1:4 borax-glycerol and 1:6 borax-glycerol solutions were employed as possible plasticisers. It was observed that pure wood plastics were weak, soft and brittle, while starch-wood composites were strong and the strength increase upon increase in starch content. The most excellent borax system that gave starch-wood composites of optimum strength was 1:4 borax-glycerol, while the saturated aqueous borax solution gave materials with considerable mechanical properties. Pure water and glycerol were respectively employed as plasticisers as they are well-known for their starch plasticising power in order to substantiate the role of borax in the saturated aqueous borax and borax-glycerol solutions in the plasticisation process. Even though pure water and glycerol gave materials with impressive mechanical properties, they suffered the problems of evaporation and recrystallization upon ageing. Almost all the materials were denser than conventional MDF.

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Synthesis, characterization and evaluation of physico chemical properties of polymeric components of nonionic hydrocarbonated and perfluorinated surfactants

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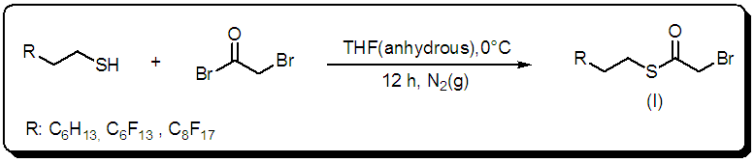
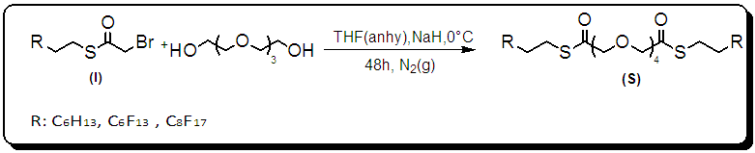
The synthesis of nonionic surfactants is widely used in many areas such as cosmetic, detergent and pharmaceutical industries as disinfectants, antimicrobial agents and also in electronics industry. It should be noted that perfluorinated nonionic surfactants are nowadays increasingly used in such synthesis mainly due to the reduction of surface tension.

In this paper, we describe the synthesis of alkyl bromoéthanthiolate or thioester by the action of hydrocarbon and perfluorinated thiols with bromoacetyl bromide. This reaction leads to three intermediates I_1, I_2 and I_3, respectively (scheme 1).



Scheme 1

The conversion of intermediates I_1, I_2 and I_3 to nonionic surfactants S_1, S_2 and S_3 respectively was performed with triethylene glycol according to a convenient operating procedure (scheme 2).



Scheme 2

The polyethoxylated nonionic surfactants having the structure R-$(CH_2)_2$-S-CO-CH_2-O-$(C_2H_4O)_4$-CH_2-O-CH_2-CO-S-$(CH_2)_2$-R with R: C_6H_{13}, C_6F_{13} and C_8F_{17} are obtained with acceptable yields. The structural characterization of all compounds, is confirmed by using conventional spectroscopic techniques namely ^{1}H NMR, ^{13}C and ^{19}F. The surface properties of the nonionic surfactants obtained is carried out by measuring the surface tension (γ_s) and determination of the critical micelle concentration (CMC).

Analysis of different treatments of aluminum mold for easy release of epoxy pieces

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Silicon-based coatings are used to prevent adhesion of pre-impregnated epoxy (prepreg) to Aluminum molds during polymer processing.
As silicon coatings are semi-permanent, the coated mold may be used for many mold releases before the coats need to be reapplied. Coatings durability depends on many factors like the number of applied layers, the curing temperature of the coating and the interaction between the coating and both the mold and the epoxy.
Our work presents a comparative study of adhesion between epoxy prepreg and various coatings. It contains two main parts.
In a first step, we characterized the non-coated mold and the coated molds. First, we studied the surface wettability of the mold by the liquid treatments using a goniometer. Then, we measured the roughness evolution of the mold surface according to the treatment layers in order to determine the final thickness of each coating. Finally, we analyzed the chemical composition of the surface by two different methods, namely EDS and XPS.
In a second step, we evaluated the durability of different coats according to the number of mold releases qualified as easy. Any transfer to the resin is considered as contamination of the surface of the piece. We therefore analyzed both fracture surfaces with SEM and XPS in order to measure coat transfer.

EDS: Energy-Dispersive X-ray Spectroscopy
XPS : X-ray Photoelectron Spectroscopy
SEM : Scanning Electron Microscope

Work performed under the chair DAHER-Mines Paristech

Study of the rheological behavior of epoxy - amine system cross-linked at 30°C with and without thinner

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Mortars based on epoxy resin are mainly used in the implementation of prefabricated parts used in the field of civil engineering. The workability of these mortars represents a major obstacle to the development of this field. In order to improve this property, we introduce a thinner in its composition. This addition is optimized to ensure the final mechanical properties of the mortar.

In this work, the rheological behavior during curing reaction of epoxy – amine system was studied. Isothermal rheological tests at 30°C were performed in order to determine the gel point of the resin and to study its viscosity during the crosslinking reaction. These tests were also conducted with adding a thinner at different rates (2%, 3%, 5% and 7%) to the epoxy – amine system for the purpose to study its influence on the rheological properties. This study showed that the addition of the thinner decreases the viscosity of the system, which leads to improving the workability of the mortar.

Key words: Mortar, workability, epoxy – amine, rheological behavior

CHEMICAL MODIFICATION OF β-AMINO ALCOHOLS AND THEIR USE IN DEPPOLUTION

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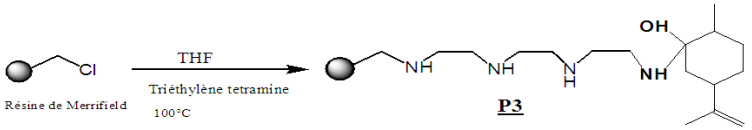
In the present work, we are interested in the synthesis of β-amino alcohols from limonene oxide. The three following amines: ethylenediamine, diethylenetriamine and triethylenetetramine using water as a catalyst were reacted with the limonene oxide. The aim of the second part, is the reaction of β-amino alcohols with Merrifield Resin and characterization of obtained products by physico-chemical analyses (IR, UV, DSC). The obtained polymers were used in the extraction of heavy metals.

The functionalized polymer by triethylenetétramine has a high selectivity for the metals Fe (III) and Bi (III) with percentages of extraction of the order of 97, 1% and 96, 4% respectively.



Keywords: limonene oxide, β-amino alcohols, Merrifield Resin (MR), extraction, heavy metals.

Naofibrillated cellulose from Alfa and sunflower plant: Effect of the delignification on the Nanofibrillation behavior

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Nanofibrillated cellulose arouses much interest both in the academic and the industrial level. These nanosized nanoparticles maigh be used in board range of application including nanocomposite, nanopare, reinforcing additive for paper etc… In the present work nanofibrillated cellulose from two annual plants, namely Alfa (Stipa tenacissima) and Sunflower (Helianthus annuus), was produced and their properties and morphological features fully characterized. The effect of the hemicellulose content and the fibre morphology on the nanofibrillation behaviour of delignified cellulose pulps was studied. The degree of fibrillation and the morphology of the nanofibrillated fractions were evaluated by centrifugation and FE-SEM. Pulps with the highest hemicellulose content showed higher yields of the nanofibrillated fraction and a better aptitude for the individualization of the microfibrils. Furthermore, it was shown that fibres from Sunflowers, recognized by having thinner cell walls, were easier to fibrillate and could be disintegrated into NFC using a simple domestic-blender when the deliginification was carried out using the $NaClO_2$/acetic acid method.

CORE/SHELL, MULTIWALLED CARBON NANOTUBE/POLYANILINE NANOCOMPOSITES BY A FACILE APPROACH USING IN-SITU GENERATED DIPHENYLAMINE DIAZONIUM SALT

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Recently carbon nanotube and conductive polymers have been combined in view of designing novel nano-composite materials [1,2] of importance for applications in capacitors, organic light emitting diodes, gas sensors, etc. Highly uniform core–shell like multi-walled carbon nanotubes-polyaniline (MWCNT-PANI) nanocomposites (Figure 1) were prepared in two steps: *(i)* surface modification of MWCNTs with a 4-aminodiphenylamine group via in-situ diazonium generation process; and *(ii)* polymerization of PANI onto surface modified MWCNTs. This functionalization helped to easily disperse the MWCNTs in acidic solutions; hence it is suitable for the chemical oxidative polymerization of aniline. It was found that MWCNT-PANI nano-composites with higher MWCNTs loading yield PANI chains with more quinoid units than the pure PANI, which results in significant improvement of the conductivity of the composites [3]. The latter were further drop casted on gold electrodes to evaluate their gas sensitivity, by measuring the change in their electrical resistance in presence of different concentration of hydrogen sulfide H_2S and ammonia NH_3 gas. The selectivity of the nano-composites decreases in the order $NH_3 > H_2S$.

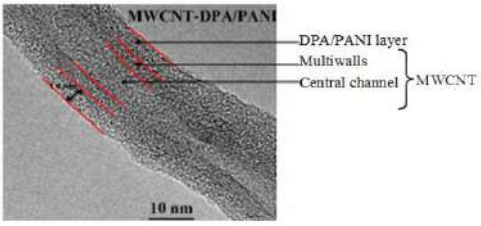

Figure 1. High resolution TEM image of MWCNT-DPA/PANI.

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Synthesis, Structure and Properties of a New Poly(*p*-phenylene) Derivative

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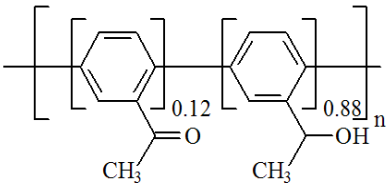
Poly(*p*-phenylene) (PPP) and its derivatives are a promising class of organic materials since they combine the excellent thermal and mechanical properties of the rigid aromatic macromolecules to the attractive opto-electronic properties of the semi-conducting polymers [1]. These organic materials are currently used as active components in several electronic devices, solar cells and light-emitting diodes [2,3]. Full color display applications require red, green, and blue emission and while all three colors have been demonstrated there are relatively few blue-emitting polymers. Here we report the synthesis and the characterization of a blue luminescent PPP derivative for opto-electronic application.



Polymer structure

The polymer has been synthesized via chemical reduction of a precursor acyl-functionalized polymer, previously prepared upon the Ni-catalyzed Yamamoto cross-coupling reaction. The structure was characterized by NMR and FT-IR spectroscopies. The polymer showed a reduction rate of 88% and exhibits a good solubility in THF, DMSO and DMF. Its dilute solution and its thin solid film emit in the blue region. The optical gap, estimated from the absorption onset of the polymer film, was about 3.21 eV. The HOMO and LUMO energy levels were determined by cyclic voltammetry and showed a p-type semi-conducting behavior.

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No centrosymetric new organic-inorganic hybrid crystal structure of 1, 2 ammonium cyclohexane tetrabromozincate (II) monohydrate.

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An X-ray diffraction single crystal study of the orthorhombic phase of $C_6H_{10}(NH_3)_2$ $ZnBr_4H_2O$, which occurs at room temperature, has been performed with the aim of determining the atomic structure. This compound crystallizes in the space group $Pna2_1$ with unit cell dimension a= 13.0317(5) Å, b= 9.3398(4) Å, c= 12.0334(5) Å and Z=4 : The refinement converged to R = 0.025 and wR = 0.065 using 2695 independent reflections (i□ 2i). The packing of viewed along b axis shows an alternation of organic cation $[C_6H_{10}(NH_3)_2]^{2+}$ and a half isolate $[ZnBr4]^{2-}$ combined into a framework by hydrogen bonds. This arrangement governed by an extensive three-dimensional network of N—H….Br, N—H …O and O—H…… Br hydrogen bonds. The zinc (II) metal centre has slightly distorted tetrahedral coordination geometry.

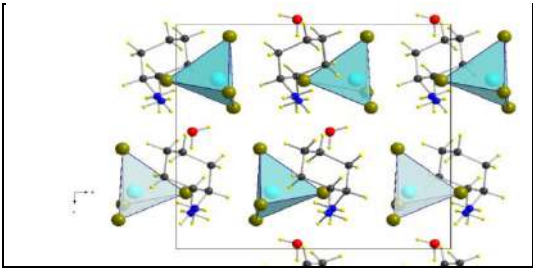

Figure: Projection of $C_6H_{10}(NH_3)_2$ $ZnBr_4H_2O$ compound structure in the ac plane.

Table: Crystallographic parameters and summary of data collection and refinement

Crystal data	
$C_6H_{10}(NH_3)_2ZnBr_4$, H_2O	*F*000 = 2256
Mr = 519	*D*x = 1.592 Mg m−3
Orthorhombic, $Pna2_1$	Mo *K*α radiation λ = 0.71073 Å
	Cell parameters from 25 reflections
a = 13,0317(5) Å	θ = 14–16º
b = 9,3398(4)Å	μ = 2.87 mm−1
c = 12,0334(5)Å	*T* = 289 (2) K
V = 1464.63(10) Å3	Prism, pink
Z = 4	0.11 × 0.09 × 0.04 mm
Data collection	
Enraf–Nonius CAD-4 Diffractometer	Rint = 0.036
Radiation source: fine-focus sealed tube	θmax = 25.38º
Monochromator: graphite	θmin = 2.68º
Non–profiled ω/2θ scans	h = -15→15
Absorption correction: ψ scan	
(North et al., 1968)	k = -11→11
Tmin = 0.34 Tmax = 0.65	l = −14→14
45757 measured reflections	2 standard reflections
independent reflections	every 120 min
reflections with $I > 2\sigma(I)$	intensity decay: 11%
Refinement	
Refinement on *F*2	
$R[F2 > 2\sigma(F2)]$ = 0.037	(Δ/σ)max = 0.001
wR(*F*2) = 0.053	Δρmax = 0.28 e Å−3
S = 1.070	Δρmin = −0.30 e Å−3

Photocured Polymeric Optical Fluorescence Sensor for the Determination of Hg (II)

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Among the various transition metal ions, mercury is known as one of the most toxic and hazardous. Due to the high toxicity of mercury, considerable attention has been intended to design and synthesis of optical fluorescent sensor for the determination of low level Hg (II)[1-5].

In this work, a novel photocured polymeric membrane containing vinylphosphonic acid was designed and synthesized as optical fluorescence sensor for Hg (II) ion. Characterization of the membrane was carried out by Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR) and Scanning Electron Microscope (SEM) techniques. The photophysical and photochemical characteristics of membrane were investigated in detail. The effects of conditions such as pH, response time and interference of foreign ions were systematically investigated. Samples were also analyzed with cold vapor-atomic absorption spectrophotometry (CV-AAS). Hg (II) ion of real samples was determined by our proposed method and produces satisfactory results.

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ETUDES DES LIGNINES DE L'ESPARTO (STIPA TENACISSIMA)

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L'Esparto (Stipa tenacissima), également appelé Alfa, est une plante herbacée vivace, largement répandue dans le sud de l'Espagne et en Afrique du Nord (Tunisie). Les propriétés de ses fibres (souplesse, finesse, contenu en cellulose) font de cette plante une matière première inestimable aussi bien en qualité qu'en quantité pour l'industrie papetière, qui l'utilise bien souvent comme seule et unique source d'approvisionnement. Nous présentons ici les résultats de l'étude physico-chimique de trois variétés de lignines (alcaline, CIMV et Acétosolv) isolées selon trois méthodes à partir d'esparto poussant dans les hautes plaines de Tunisie. Les structures et la stabilité thermique sont étudiées par analyses spectrales (RMN et infrarouge IR), thermogravimétriques (TGA).

Les résultats en spectroscopie IR indiquent que ces lignines se classent dans le groupe de lignine HGS (pics à 1497 et 1503 cm^{-1})

Cette étude confirme que les lignines ne devraient plus être considérées comme des déchets mais comme des polymères naturels de valeur disponibles en quantité et récupérables facilement depuis les liqueurs de pâte à papier. Ces conclusions feront l'objet de propositions d'applications concrètes et appropriées par la suite.

SYNTHESIS OF NEW POLYOXADIAZOLES BEARING FURANIC-PYRIDINIC MOIETIES

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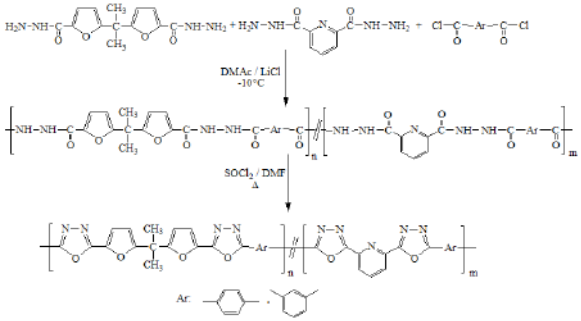
Aromatic poly(1,3,4-oxadiazole)s are known by their high chemical and thermal resistance. For this reason, polyoxadiazoles are used in preparation of membranes for gas separation and fuel cells [1, 2]. Most polyoxadiazoles showed poor solubility in common organic solvents due to the chain rigidity from a heterocyclic group. It was demonstrated the interest to prepare the polymers which contain pyridine rings in the backbone and to employ the polymers as reverse osmosis membranes, since such polymers may improve reverse osmosis performance in view of their rigid backbone and polarity of pyridine rings [3]. As a part of our research interest in the development of materials based on monomers from renewable resources, we have showed that the substitution of aromatic groups by furanic units containing two furanic heterocycles linked by an aliphatic bridge in the final macromolecules of some polymers did not affect their thermal and mechanical properties and their chemical resistance. We report in this communication our recent work devoted to the synthesis and characterization of new class of polyhydrazides containing pyridine rings and 2,5-substituted furan moieties in their backbone which were converted to the corresponding polyoxadiazoles by chemical cyclodehydration.



All the copolymers were readily soluble in polar solvents such as 1-methyl-2-pyrrolidone (NMP) and dimethyl sulfoxide (DMSO) and showed inherent viscosities equal to 0.52 - 0,95 dL/g. Work is in progress to assess their physico-chemical and mechanical properties and to examine their biodegradability.

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EFFET DU MILIEUX AQUEUX SULFATE ET NITRATE SUR L'EXTRACTION DU CUIVRE(II) PAR L'ACIDE DI-(2-ETHYL HEXYL) PHOSPHORIQUE

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Ce travail porte sur l'étude de l'extraction du cuivre (II) en différents milieux aqueux sulfate et nitrate par l'acide di-(2-ethylhexyl) phosphorique (D2EHPA) dans le chloroforme à 25°C.

La stœchiométrie des complexes extraits a été déterminée par la méthode des pentes. Les complexes organométalliques extraits dans la phase organique sont du type $CuL_2 2HL$ dans le chloroforme.

Suivant le milieu aqueux utilisé, l'extraction du cuivre (II) par D2EHPA a diminué suivant l'ordre:

Milieu nitrate > milieu sulfate

L'évaluation de la nature de l'interaction avec les ions de chaque milieu pour les systèmes d'extraction utilisés se fait par utilisation de la loi de Debye Hükel et le calcul de la constante d'interaction.

Mots clés : Extraction liquide - liquide, Acide di-(2-ethylhexyl) phosphorique, Cuivre (II), Effet du milieu aqueux.

Synergic effect of a combined use of two chemical blowing agents on the foaming efficiency of an extruded rigid PVC compound

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This work deals with the effect of the addition of a combination of two chemical Blowing Agents (BA); AzoBisFormAmide (ABFA) and/or Sodium BiCarbonate (SBC) , on the foaming efficiency and cell structure of a rigid Poly(Vinyl Chloride) (PVC), using a laboratory scale extruder and optical microscope. It was found that the foaming efficiency increased with BA loading to an optimum value, which is characterized by a Critical Concentration (CC) of BA . This CC was noticed to be higher in the case of SBC. On the other hand, the foaming efficiency (expressed in term of diameter of foamed extrudates) showed a synergistic effect according to some mixing ratio of the two BA, which were based on their CC.

Conclusions
- Foaming efficiency would rise with increasing ABFA level to an optimum value corresponding to the CC, and then dropped with additional BA.
-The CC would shift to higher value when SBC was used
-In all cases surface quality was improved below the CC
-A combined use of two BA, with a high ABFA content (70%), would improve more the foaming efficiency of extrudates

keywords : Poly(vinyl chloride), azobisformamide, sodium bicarbonate, cell structure, expansion, extrusion, decomposition, foaming efficiency, coalescence and synergism.

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Poly (Acrylonitrile-co-Methyl Methacrylate) Nanoparticles for Enzyme Immobilization: I. Preparation and Characterization

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This work concerns preparation and characterization of poly (acrylonitrile-co-methyl methacrylate) Copolymer nano-particles using precipitation polymerization technique. Potassium per-sulfate redox initiation system was used to perform polymerization process in alcoholic aqueous system. The impact of different polymerization conditions such as co-monomer concentration and ratio, polymerization time, polymerization temperatures, initiator concentration and co-solvent composition on the polymerization yield and particle size were studied. Maximum polymerization yield, 70%, was obtained with MMA: AN (90%:10%) comonomer composition. Particles size ranged from 16 nm to 1483 nm was obtained and controlled by variation polymerization conditions. The co-polymerization process was proved through FT-IR and TGA analysis. The copolymer composition was investigated by nitrogen content analysis. Copolymers with progressive percentage of PAN show thermal stabilities close to PAN Homopolymer. SEM photographs prove spherical structure of the produced copolymers. The investigated system shows promising future in preparation of nanoparticles from comonomers without using emulsifiers or dispersive agents.

Keywords: Nano-poly (acrylonitrile–co-methylmethacrylate); Precipitation polymerization; Particles size; Copolymer composition; TGA; FT-IR; XRD.

Synthesis, Characterization and Reactivity toward diethanolamine of α, ω–ditosylates

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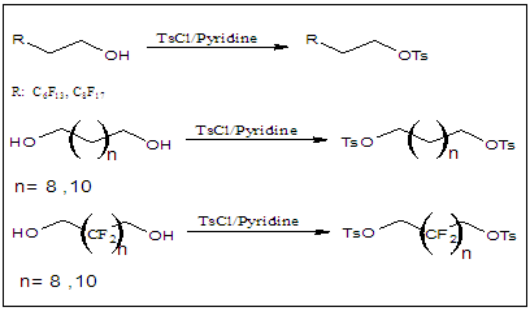
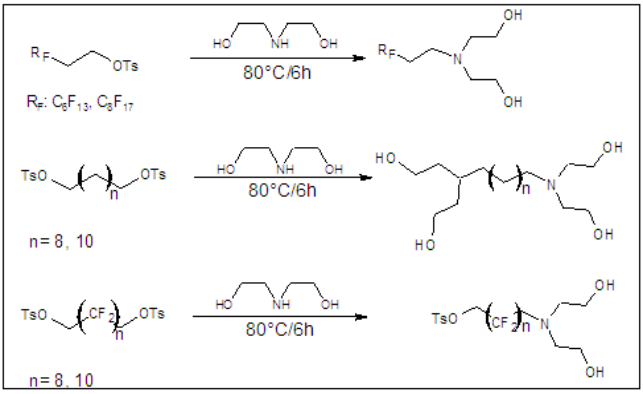
Mono- and ditosylates are prepared from the corresponding alcohols or diols and tosyl chloride in pyridine. Both fluoro- and hydrocarbon tosylates are obtained according this procedure (scheme1).



Scheme 1: Preparation of tosylates

Introduction of hydrophilic head into these tosylates was accomplished by treatment with diethanolamine (scheme 2).



Scheme 2: Conversion of tosylates into amphiphiles

Surface properties of the obtained amphiphiles are under investigation.

INFLUENCE OF MICROWAVE RADIATION ON STRUCTURE-PROPERTY RELATIONSHIP OF CHITOSAN IN SOLUTION

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Khaled ELSHERIF, Salma GABALLAH

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The aim of this work is using a simple way to investigate the structure-property relationship of extremely diluted chitosan in salt solution before and after microwave (MW) – irradiation. Accordingly, equal weights of solid chitosan were exposed to MW- radiation for different periods of time. Then, 0.5 mg cm^{-3} of native and irradiated chitosan in 0.1M HAc / 0.2M NaCl solutions were prepared. UV- spectra of accumulated carbonyl groups due to the cleavage of glycosidic linkages of chitosan chains by MW- irradiation were observed in the range of 200-270 nm. Accordingly, the progress of MW- degradation for chitosan was monitored spectrophotometrically. An increase in UV-absorbance of irradiated chitosan was detected at 247 nm and interpreted in terms of coils concentration via Beer-Lambert law. Also, a simple viscometric technique by one point method of measurement was used to detect the relative viscosity of native and irradiated chitosan. A decrease in relative viscosity of irradiated chitosan were observed and interpreted in terms of the scaling laws. Consequently, structure-viscosity parameters such as perturbed and unperturbed intrinsic viscosities and coil dimensions, molar mass and steric hindrance parameter were decreased. In contrast, the values of its coil density, critical concentration and second virial coefficient were increased. MW-irradiation reduced the molecular weight and size of chitosan, but increased its rigidity and solvation.

Key Words: chitosan; microwave; UV-absorbance; viscosity; scaling laws.

Surfactant-free Waterborne Hybrid Alkyd–acrylic Dispersion: Synthesis, Properties and Long term stability

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Surfactant-free waterborne hybrid alkyd-acrylic dispersions with a solid content of 40% were synthesized, characterized and their long term stability investigated. The hybrid resin was synthesized by melt co-condensation reaction between acrylic prepolymer bearing carboxylic groups, and low molecular weight alkyd resin. Spontaneous emulsification of the ensuing hybrid resin was achieved, following the addition of base aqueous solution such as NH_3, triethylamine (TEA) or NaOH that neutralize the carboxyl groups. The key role of the carboxylic groups in the stabilization process was discussed and the effect of their content on the properties of the dispersion was analysed. The insertion of anhydride moiety within the acrylic prepolymer was shown to ensure the efficient coupling between the acrylic and the alkyd resin, thus impeding destabilization by phase separation process. According to the neutralizing base, the particle size of the dispersion, its viscosity and the hydrolytic resistance of the hybrid resin were greatly altered. The present dispersion is easy to implement and the colloidal stability might be easily tailored.

POLYSTYRENE-CLAY NANOCOMPOSITES: EFFECT OF PHOSPHINE OXİDE INTERCALANT FOR CLAY MODIFICATION

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Thermoplastic polystyrene (PS) is a well known and characterizad hydrophobic polymer. The main aim of this study is to preparation and investigation the thermal and mechanical properties of PS-modified clay nanocomposites. Montmorillonite clay (MMT) was pre-modified using different concentrations of quaternized derivative of Bis(3-aminophenyl) phenyl phosphine oxide (BAPPO) via ion exchange between Na^+ ions in the clay and BAPPO cations in aqueous medium. Polystyrene–clay nanocomposites were prepared by free radical polymerization of styrene containing dispersed clay. X-ray diffraction (XRD) and transmission electron microscopy (TEM) indicate that exfoliation of MMT was achieved. The effect of increased nanofiller loading in thermal and mechanical properties of the nanocomposites was investigated by thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA).

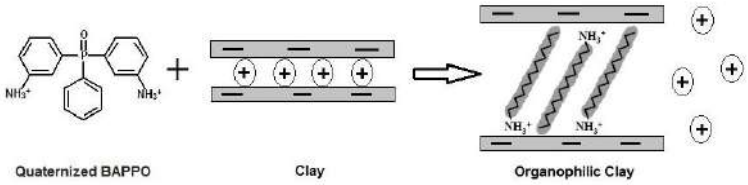

Figure: Modification of montmorillonite clay with BAPPO

Synthesize of benzidine-clay hybrid material by solid-solid reaction and reaction of polymerization of benzidine in situ

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In recent years, the intense uses of solid-solid reaction have much attention owing to their ease of operation when compared to the classical method using solutions. This work aims to synthesize benzidine/clay hybrid materials by grinding mixtures of various amounts of benzidine (BZ) and two clays with different nature which are montmorillonite (natural) and laponite (synthetic). Different techniques have been used in order to characterize the obtained hybrid material such as: DRX, UV visible and FTIR spectroscopy.
From DRX analysis, it was shown the successful intercalation of benzidine molecules in the clay interlayer. The arrangement adopted by these molecules depends on the nature of the clay and loading amount of benzidine. In fact; it was showed that BZ molecule intercalate with a monolayer arrangement parallel to the plan of montmorillonite layers and with a twisted configuration with laponite clay. A second step of this work was the study the reaction of solid state polymerization of benzidine between the clay interlayer using ammonium peroxodisulfate as polymerization initiator. Impedance spectroscopy has been used to study electrical properties of the obtained conductive polybenzidine-clay nanocomposite.

Keyword: Montmorillonite; Laponite; polybenzidine; nanocomposite

Synthesis, Characterization and Reactivity toward of hydrazine of α, ω ditosylates

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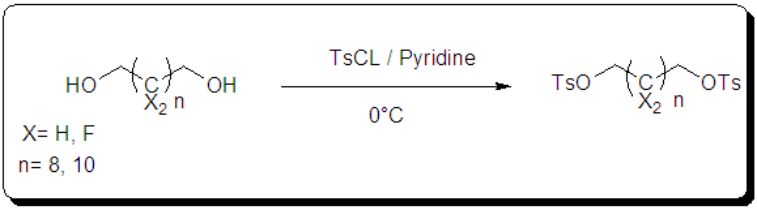
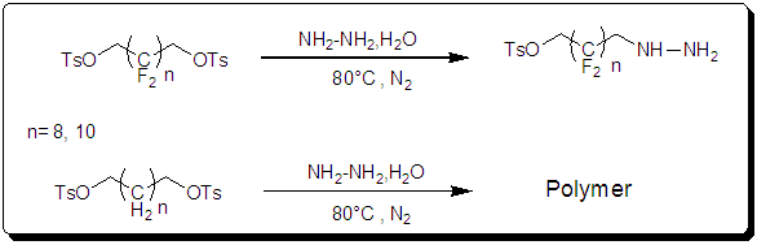
Hydro- and fluoroditosylates are obtained from the corresponding diols and tosyl chloride in pyridine (Schema 1).



Schema 1: Ditosylates preparation

While fluorinated ditosylates react with hydrazine to furnish the corresponding monosubstituted hydrazine, the hydrocarbon analogues lead to polymer (Schema 2).



Schema 2: Reaction of ditosylates with hydrazine

Dielectric proprieties of the new organic-inorganic hybrid compound $[C_8H_{10}NO][ZnCl_3]$

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Bis(4-aminoacetophenone) tetrachloridozincate has been synthesized and characterized by X-ray at 298K and impedance spectroscopy. At room temperature, the title compound crystallize in the orthorhombic system (Cmca space group) with Z = 8 and the following unit cell dimensions: a = 19.4331 Å, b = 15.5209 Å, c = 13.7318 Å. The dielectric proprieties of this compound have been measured in the temperature range 388-513K and the frequency range 209Hz-5MHz. The analysis of their proprieties shows the existence of a relaxation process at 448K which can be explain by the reorientation motion of the 4-aminoacetophenone groups where the high dynamical disorder of NH^+_3 in the N-H…Cl and N-H…O hydrogen bond.

Keywords: Organic-inorganic hybrid, X-Ray powder, Impedance Spectroscopy

PHYSICS PROPERTIES AND DEFECT STRUCTURE OF AL-DOPED $SrFeO_{3-\delta}$

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A defect chemical model for the behavior of acceptor and donor-doped SrFeO3 as a function of oxygen pressure is proposed. The non-stoichiometric deviation was calculated as a function of oxygen partial pressure, pO_2, at different temperatures. The mathematical approach allows us to calculate the oxygen partial pressure dependent properties of SrFe1-x AlxO3-δ in the range $0.10 \leq x \leq 0.30$. The results show that the conductivity was dependent of pO_2 and proportional to the dopant concentration.

Stability regimes and compensation mechanisms at various oxygen partial pressures and at various temperatures are proposed. This model examines also the charge compensation mechanisms that dominate under the different regimes and their implications for transport properties. From equilibrium constants, Thermodynamic quantities such as standard enthalpy and entropy charge for the defect formation reactions were calculated.

Key words: Defect modeling; SFA; Non stoichiometry; Conductivity

Olive stones flour from solid-waste of the olive Oil industry as a filler in thermoset unsaturated polyester resin

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Tunisia is one of the leading olive oil producers in the world. Nevertheless, the extraction system generates large volume of liquid/solid waste which represents a disposal and potentially environmental pollution problem. About 15% based on the whole olive fruit is composed of hard indigestible stone which is a solid lignocellulosic material composed of hemicellulose, cellulose and lignin as main components. Although the solid waste residue might constitute a renewable source of energy, their use as sustainable filler for thermoplastic and thermosetting polymer to elaborate composite materials with enhanced stiffness might also constitute an interesting alternative of valorization within the field of materials.

In the present study, olive stone flour (OSF) was used as filler in a thermoset based unsaturated polyester (UP) resin. The composite was prepared by mixing the OSF with the liquid resin UP followed by compression moulding at 120°C and 4 MPa. Composites with filler content ranging from 10 up to 60 wt% were prepared and their thermal, mechanical and water absorption properties investigated. The addition of OSF was shown to enhance the stiffness of the matrix but at the expense of a reduction in the mechanical strength and impact strength. The quality of the filer-matrix interfacial adhesion was also analyzed using scanning electronic (SEM) observation.

SYNTHESIS, CHARACTERIZATION AND FLUORESCENCE PROPERTIES OF *s*-TETRAZINES

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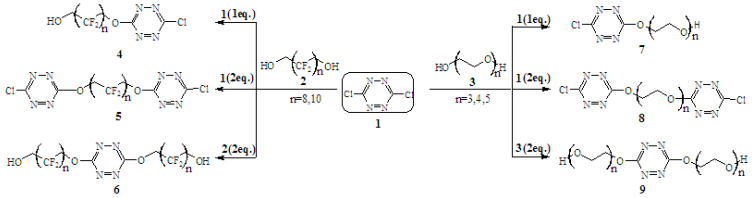
Treatment of dichloro-*s*-tetrazine **1** with fluorinated diols **2** or polyethylene glycols **3** leads to the corresponding fluorinated tetrazines in moderate to good yields (Scheme).



Scheme: S_NAr^1 of dichloro-*s*-tetrazine

Absorption and fluorescence properties of the synthesized compounds are investigated (e.g. Figure).

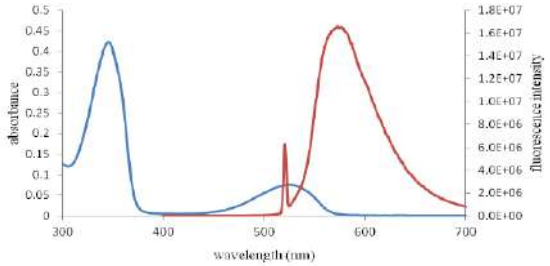


Figure: Absorption (blue) and fluorescence (red) spectra of *s*-tetrazine **8a**

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CHARACTERIZATION OF POLY(N-VINYLCAPROLACTAM) AQUEOUS SOLUTION BY A CAPILLARY VISCOMETER

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Poly(N-vinylcaprolactam) is prepared via batch radical precipitation polymerization using N-vinylcaprolactam and cationic radical initiator. The viscosity of diluted polymer was examined as a function of temperature ranging from 25 to 40°C. The intrinsic viscosity and the polymer-solvent interactions were determined and examined on the basis of the Huggins's equation. The Huggins constant was found to be positive below 32°C which the Lower Critical Solution Temperature (LCST) for this homopolymer. However, in the LCST range, the sign of the Huggins constant changes due to the turbidity of the medium induced by polymer precipitation. The intrinsic viscosity and Huggins constant variation as a function of temperature prove the associative nature of the prepared end-charged poly(N-vinylcaprolactam).

PREPARATION AND CHARACTERIZATION OF ALGINATE-CARBOPOL BEADS FOR ORAL INSULIN DELEVERY

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A variety of approaches have been investigated to address the problems associated with oral insulin delivery, but the bioavailability of oral insulin is still low. Insulin is rapidly degraded by the enzymes in the GI tract and is not transported across the epithelial barrier easily. Present study indicated that alginate-carbopol beads are resistant and can be utilized as a carrier system for controlled delivery of insulin or other proteins for bioadhesive various therapeutic applications. Alginate-carbopol beads were prepared by ionotropic gelation technique utilizing calcium chloride ($CaCl_2$) as a cross linking agent, to take the advantage of swelling and degradation property of alginate beads for improving the oral delivery of insulin. Depending upon the variability in the concentration of polymers, percentage of cross linking agent, time of curing, the factors like particle size and incorporation efficiency of different beads varies. Insulin release, at pH 1.2, was almost prevented by the incorporation of polymers. When uncoated beads were transferred to pH 6.8, a fast dissolution occurred, independently of the concentration of polymers, and insulin was completely released. Thus, carbopol was able to avoid insulin release at pH 1.2, protecting the protein from the acidic environment and reducing the total insulin released at pH 6.8.

Fig: Alginate-carbopol beads

Keywords: Alginate; carbopol; beads; Insulin; oral administration.

Physico-chemical characterization of extracted and degraded polysaccharide from Tunisian Algae: Antioxidant anti-inflammatory and anti-proliferative activity

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Seaweed polysaccharides are highly active natural substances having valuable applications. Brown algae represent a rich and renewable source of key polysaccharides with structural and biological interest. Alginates and fucoidans are the parietal polysaccharides from brown algae. Alginates are phycocolloids that can be used in different fields. Their uses are based on their gelling properties. However, fucoidans have recently known a great interest as a sulfated polysaccharide offering multiple biological activities. In this work, we are interested in the extraction, degradation, physico-chemical characterization and study of biological activities of polysaccharides from Tunisian brown seaweeds.

Polysaccharides were extracted with an acceptable yield. The different rates of total carbohydrate, uronic acids and sulfate content, were determined by colorimetric techniques.

In order to obtain oligosaccharides we choose the autohydrolysis process. Polysaccharides were changed to H-form using amberlite IR -120. After 24h, 48h and 72h at 37°C a volume was sampled and neutralized with 5% NH_4OH.

The physico-chemical characterizations of extracted and degraded polysaccharides were performed by size exclusion chromatography equipped with a triple detection: refractometric detection, viscosity and multiangle light scattering (SEC/MALLS/RI). The average weight of the extracted polysaccharides was about 10^5 Da. After degradation, the decrease in mass was greater for fucoidans (971000 Da to 137700 Da) than for alginates (167100 Da to 44000 Da). Furthermore, we have shown that these products have potential applications as anti-free radical, anti-inflammatory and anti-proliferative.

Key words: brown algae, polysaccharides, degradation, SEC/MALLS

SYNTHESIS AND CHARACTERIZATION OF SULFONATED ALIPHATIC POLYESTERS

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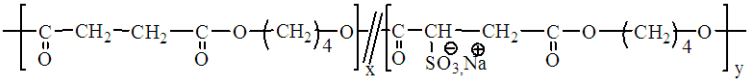
Recently, there has been considerable interest in the synthesis of biodegradable aliphatic polyesters because of the growing importance of environmentally degradable polymers [1]. However, sulfonated aliphatic polyesters PEAS are little mentioned in the literature if one refers to their aromatic counterparts. This ionomer, may be of interest particularly interesting in specific areas where biodegradability is a key parameter. In fact, the introduction of ionic units along the polyester chain reduces the crystallinity [2]. On the other hand, aliphatic polyester ionomers showed interesting properties, including shape memory characteristics due to the presence of ionic groups [3]. In this context aliphatic based sulfonated polyesters containing 10 to 40 mol% of sulfonated units were prepared by two different method the first one based on the melt polycondensation from 1.4-butanediol dimethyl sodiosulfosuccinate and diethyl succinate and the second one using a post-sulfonation of unsaturated polyesters Structural characterization of copolyesters was investigated by (^{1}H,^{13}C NMR and 2D NMR). The molecular weight of copolyesters was performed with (SEC). The thermal properties (DSC, TGA) show that the level of Tg was correlated to the sulfonate concentration units. The hydrolytic degradation in acidic aqueous conditions (pH= 4,35) at 37°C over the period of four weeks show that the mechanism of the hydrolysis of copolyesters was elucidated in relation with the microstructure. This study allows tuning their properties as a function of the final applications.



Poly (butylene succinate-co-butylene sodiosulfosuccinate)

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Extraction of chitin and chitosan from shrimp shells (Parapenaeus longirostris): physicochemical and biological characterization

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Chitosan is a natural copolymer of Dglucosamine (GlcN) and N-acetyl-Dglucosamine (GlcNAc) and is produced by alkaline deacetylation of chitin, extracted from an abundant source of shellfish exoskeletons or the cell walls of some microorganisms and fungi (Hirano, Ohe, & One, 1976). It is insoluble at neutral and alkaline pH but it is soluble in acidic media. It has attracted marked interest as a biomedical material, because it is biodegradable, biocompatible, and non-toxic (Kumar et al., 2000; Jayakumara et al., 2010).

In this study, chitin was extracted from shrimp shells (*Parapenaeus longirostris*) of the source of Tunisian origin and chitin obtained was converted into chitosan more useful. The preparation of chitin and chitosan from shrimp waste was produced by the conventional method (demineralization, deprotenization and deacetylation). The degree of deacetylation of chitin and chitosan was determined by Fourier Transform Infrared Spectroscopy (FTIR).

Then, we evaluated the antimicrobial activity of different concentrations of chitin and chitosan against 4 species of bacteria and 2 fungi by micro-well dilution assay. The minimal inhibitory concentrations (MIC) of chitin and chitosan depended on the bacterium and fungi being studied.

Also their antioxidant activity was investigated, with 1, 1-diphenyl 1-2-picryl hydroxyl (DPPH) radical scavenging activity and the inhibition of linoleic acid peroxidation.

As the concentration of chitin and chitosan increased, the antioxidant effects were strengthened.

Chitosan from *parapenaeus longirostris* was good in antimicrobial and antioxidant activity, and may be used as a source of antioxidants, as a possible food supplement or ingredient in the pharmaceutical industry.

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Adsorption of the BSA on the poly (acrylamide co acrylic acid) complexed with copper ion

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For its great importance in purification of proteins, used in medicine and biology, we have described in this study how resins synthetic macromolecules chelated with metal ions of copper (Cu^{2+}) witch bind protein (BSA) as biological macromolecules.

The Cu-resin poly (acrylamide co acrylic acid) was synthesized in two steps: preparation of a polymer resin Poly (MAA-co-AA), followed by fixing the copper ions on this one. Chelation of copper was characterized by FTIR analysis and UV-Visible spectroscopy.

Before carrying out the adsorption, we have firstly optimized the pH of the analysis; pH of 7 seems to be adequate to achieve our tests.

The adsorption is carried out in aqueous solution of bovine serum albumin (BSA) ; we observed an increase of the adsorption until the formation of saturation plate. This corresponds at the maximum adsorption capacity (Q_m) and it was estimated at 129.69 $mg.g^{-1}$ and 240.36 $mg.g^{-1}$ for resins without copper and with copper respectively.

Specific surface values have been calculated and its equal to $0.65x10^6$ $cm^2.g^{-1}$ in the case of Poly (MAA-co-AA) and $1.2x10^6$ $cm^2.g^{-1}$ for Poly (MAA-co-AA)-Cu. This indicates that chelating polymers by metals increases the protein power retention.

Keywords: adsorption, protein, chelate, polymer

Effect of coupling agent on the dielectric properties of low density polyethylene / short palm lignocellulosic fibers composites.

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In this work, the use of short palm tree lignocellulosic fibers as a reinforcing phase in the low density polyethylene (LDPE) matrix has been reported. Compatibilization of these fibers was carried out with the use of maleic anhydride copolymers. In order to investigate the coupling agent effect, the broadband dielectric spectroscopy (BDS) is used for composites at equal filler content, but with different ratio of compatibilizer. For the neat matrix, only the ionic conduction phenomenon has been detected. After the incorporation of cellulose filler, the dielectric losses increases and two relaxation processes appeared: a dipolar relaxation and the interfacial polarization. It was shown that the use of the compatibilizer improves the interfacial adhesion and the dielectric properties to a critical content of compatibilizer beyond which the dielectric properties decrease was observed.

Keywords: Composites; Low density polyethylene (LDPE); Lignocellulosic fibers; Coupling agent; Dielectric properties.

NEW BIPHENYLVINYLANTHRACENE-BASED SEMICONDUCTING POLYMER SYNTHESIS AND OPTICAL PROPRIETIES

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Anthracene was one of the first aromatic materials used in organic light emitting diodes (OLEDs). The first experiments were carried out by Pope in the early 1960s.[1] Soon after, several reports on single-crystal anthracene-based OLEDs were published, and good quantum yields were obtained (up to 5%). Nevertheless, such devices are thick and hence require very high operating voltage (over 100 V). The improvement in the operating voltage was achieved by vacuum evaporating thin layers of anthracene; in this case the operating voltage was lowered up to 30 V.[2] However, the anthracene tends to recrystallize with diode operating time, which led to a degradation of device performance.
In this communication, we report the synthesis and characterization of new soluble **biphenylvinylanthracene-based** semi-conducting polymer (**Figure1**) for organic thin-layer electronic applications.

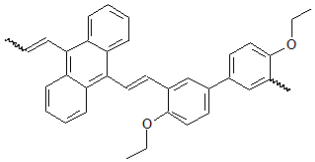

Figure 1: Polymer structure

The polymer has been synthesized via Wittig condensation using 9,10-bis(triphenylphosphoniomethyl)anthracène dichloride and new biphenol-derived dialdehyde. The chemical structure of the polymer was well defined by ^{1}H NMR, ^{13}C NMR. The polymer is fully soluble in common organic solvents and has a number-average molecular weight of 11400 $g.mol^{-1}$. The thin-film optical gap was about 2.69 eV make it possible to classify this polymer among the semiconducting materials. The polymer emits in green region both in dilute solution and in solid state. The HOMO and LUMO energy levels were determined by cyclic voltammetry and showed a p-type semi-conducting behavior. A single-layer device of the configuration [indium-tin oxide/**polymer**/aluminium] has been fabricated and has a relatively low turn-on voltage.

Keywords: Semi-conducting material; Anthracene; Photoluminescence; Thin solid film.

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Determination of affinity coefficient of cations exchange membrane at different ionic strengths

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Though it is known that the ion-exchange polymers have a variable affinity for the different counter-ions, This coefficient characterizes the difference in affinity of the membrane toward two counter-ions, so it allows to assess the competition between them to occupy fixed sites of the membrane. We have determined this characteristic for five cations pairs H^+/Na^+, H^+/Ag^+, $H^+/EDAH_2^{2+}$, $Ag^+/EDAH_2^{2+}$ $EDAH^+/EDAH_2^{2+}$ using commercial cation exchange membranes Nafion 324.

Equilibrium between Nafion 324 cationic membrane and solutions of cations was investigated at various ionic strengths (I=0.01M and I=0.03M). Membrane water content and ion exchange capacity were determined.

The experimental results obtained allow plotting the curves of variation of affinity coefficients depending on the composition of the external solution. It is defined with respect to counter-ion has the lowest hydration volume.

The obtained results show that the membrane appears more selective for polyvalent cations particularly for $EDAH_2^{2+}$.

Preparation and Physical Properties of Polymer for Photovoltaic Applications

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Polymer bulk heterojunction solar cells based on composites of electron-donating conjugated polymers and electron accepting molecules such as fullerenes, offer promise for the realisation of printable, portable and flexible photovoltaic devices with relatively low cost.

In recent years, organic solar cells utilizing conjugated polymers have attracted widespread interest. These polymers are promising in terms of their electronic properties, low cost, versatility of functionalization, thin film flexibility, and ease of processing. These factors indicate that organic solar cells, although currently producing relatively low power conversion efficiencies (~7%) [1-3] compared to inorganic solar cells, have the potential to compete effectively with alternative solar cell technologies. However, in order for this to be feasible, the efficiencies of organic solar cells need further improvement. This is the focus of extensive studies worldwide.

In this contribution, we report the preparation and characterisation of low band-gap semiconducting materials consisting of donor/acceptor type alternating copolymers of 2,7-linked carbazole units with 2,3,6,7-tetraphenyl-9,10-di(thien-2-yl)pyrazino[2,3-g]quinoxaline repeat units and 5,7-bis(5-bromothiophen-2-yl)-2,3-bis(4-(2-ethylhexyloxy) phenyl) thieno[3,4-b]pyrazine repeat units. The optical and electrochemical properties of these polymers are presented.

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ADSORPTION KINETICS OF TWO CATIONIC DYES ON STARCH FROM AQUEOUS SOLUTIONS.

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The textile industry uses toxic synthetic dyes that pollute surface waters, sometimes with important flow. Discharges from the textile industry establish enormous nuisance to human health.
Different dyes used in this type of industry, causing serious problems, due to their stability and low biodegradability. Thus, it is necessary to treat these rejections before they are discharged into the sewer system. Current methods of treatment of water polluted by dyes in the textile industry are bad and sometimes not even adapted to the depollution of these pollutants.
Looking for another source using natural and biological materials to be able to use as a new adsorbent is necessary in the treatment of effluents contaminated with dyes.
The objective of this work is the use of intrinsic properties of starch extracted from a chosen substrate (potato starch) for its application for removal of a dye to clean up the water.
The adsorption experiments were carried out batch in a stirred reactor.
The results of adsorption kinetics show a steady state is reached after 90min. They show a good affinity of the dye for these materials. And a chemical treatment will have an effect on the adsorption capacity.

Keywords: Dyes cationic textile, adsorption, water treatment, starch

COLORIMETRIC AND FLUORESCENT CHEMOSENSORS FOR THE TOXIC IONS DETECTION IN ENVORONMENTAL MEDIUMS

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There is interest in finding better and more efficient ways of detecting these ions that can be potentially harmful to the environment or human health. Among the anions and the heavy metals, cyanides, mercury and copper are the most dangerous which are lethal to humans at micromolar concentrations. Consequently, the presence of these anions in drinking water can give rise to a very serious risk to human health. Therefore, It is evident that reliable and efficient ways of detecting the presence of these ions in water, which are common in biological systems, are needed.
For this purpose, we are developed a new generation of water soluble chemosensors able to detect cyanide [1-7], mercury [8,9] and cupper [10] ions about 10-7 M in biological systems with high selectivity over other ions.

The water solubility of these new chemosensors generation was given by the incorporation of the saccharidic [1, 2, 3], glycerol [4] moiety on the starting organic chemosensor, or by the chemical grafting of the starting chemodosimeters on an adequate water soluble polymer such as polyvinyl alcohol [5], and on natural cellulose materials [6], or also by the organic chemosensor incorporation into polymeric plastic film based on starch as a biosourced biopolymer [7].

Recently, we developed a another strategy to detect the heavy metals in biological mediums by synthesizing a new water soluble fluorescents chemosensors [8] and nano hybrid materials based on silica [9] and silica coated magnetite [10] nanoparticles doped by an adequate fluorophore for the Hg (II) and Cu (II) colorimetric and fluorescent titrations.

In this conference, we will present our recent and the most significant results about the chemosensing of the cyanide and the heavy metals using a new chemodosimeter bio-based.

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NOVELS GEOTEXTILES FOR THE SELECTIVE CAPTURE OF THE HEAVY METALS FROM WASTEWATER BASED ON BIO-POLYMERS GRAFTED FUNCTIONALIZED CROWN ETHERS

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Transition metals are commonly found in industrial wastewater. For environmental and/or process concerns, it is often important to be able to detect and remove these before disposing or recycling the water. When this removal is carried out selectively, the burden of wastewater treatment can be transformed into an opportunity for metal recovery.

Biopolymers such as Chitosan (CTS) and alginate (Alg) can be used as ion exchange [1] material for the removal of heavy metal ions from industrial wastewaters. They present hydroxyl, amine and carboxylic groups respectively that can be modified to prepare CTS and Alg derivatives.

Chemical grafting of new functional groups improves the adsorption selectivity, as well as the sorption capacities of these biopolymers. In this sense, functionalized azathia crown ethers have good selectivity for its particular molecular structure and variety of ring cavities during chelation with metal ions. The chemical grafting of the functionalized azathia crown ethers onto a high molecular CTS and Alg backbone yield the CTS and Alg – functionalized azathia crown ethers (figure 1) containing the double structures and properties of CTS, Alg and functionalized azathia crown ethers. These novel CTS and Alg derivatives were synthesized by using the ultrasound technology in order to reduce the ecological impact of the classical organic synthesis methods.

In this conference, we will present our recent and the most significant results about the capture of different heavy metals, revealing the saturation level, kinetics of capture as well as the behavior of the geotextiles towards the calcium.

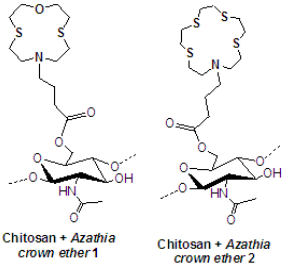

Figure 1: General structure of the modified chitosan, alginate- azathia crown ethers

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NEW SEMI-CONDUCTING POLY(ARYLENE SULFIDE)S FOR OPTO-ELECTRONIC APPLICATIONS

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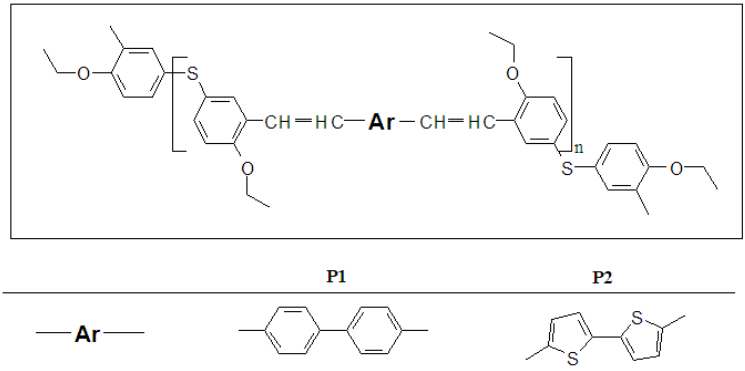
New semi-conducting poly(arylene sulfide)s were synthesized via Wittig polycondensation. The arylene moieties consist on distyrylbiphenyl (**P1**) and distyrylbithiophene (**P2**) π-conjugated systems. The polymers are soluble in common organic solvents and their molecular structures were confirmed by ^{1}H NMR, ^{13}C NMR and FT-IR spectroscopies. These organic materials were fully amorphous and show relatively high glass transition temperatures and good thermal stability. The optical properties of the polymers were investigated by UV-visible absorption and photoluminescence spectroscopies. The **P1** film has an optical gap of 2.95 eV and exhibits a green-blue fluorescence. The polymer **P2** shows a longer effective conjugation length with a gap of 2.42 eV and emits in the orange region. The HOMO and LUMO energy levels were estimated from cyclic voltammetry measurements and lower ionization potential and higher electron affinity were observed for **P2** film. Single-layer diode devices of [indium-tin oxide/polymer/aluminium] configuration have been fabricated and show relatively low turn-on voltages.

Keywords: organic semiconductors; poly(arylene sulfide)s ; optical properties ; thin films.



Structures of the π-conjugated polymers P1 and P2

CHARACTERIZATION OF PARTIALLY SULFONATED POLYOXADIAZOLES BEARING FURAN MOIETIES

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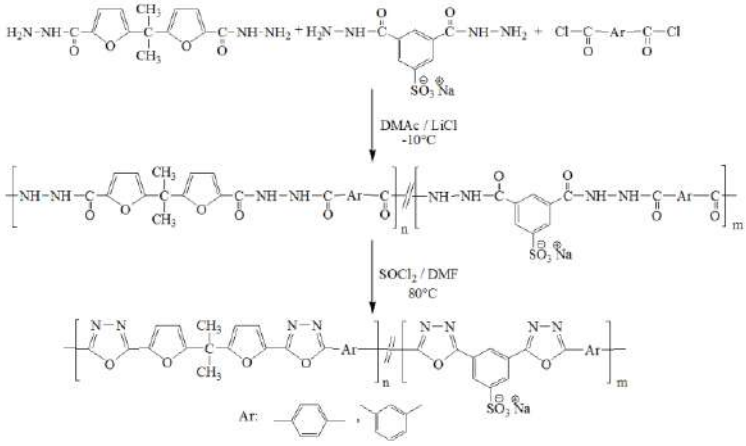
Polyoxadiazoles are a class of chemically and thermally stable heterocyclic polymers, they exhibit important application as polymer light-emitting diodes, acid sensors, gas separation, electron and proton conductive membranes due to the aromatic and basic character of the oxadiazole ring.[1] The sulfonation of polymers might improve wet ability, antifouling capacity, solubility in solvents for processing and gas permeation properties, which are relevant for other applications in membrane technology as fuel cells, electrodialysis and ultrafitration with reduced fouling susceptibility.[2]

We report in this communication our recent work devoted to the synthesis and characterization of new class of sulfonated polyoxadiazole incorporating 2,5-substituted furan moieties in their backbone which would be more flexible than their fully aromatic counterparts.



All furanic sulfonated polyoxadiazole showed good solubility in polar solvents and had glass transition temperatures close to 250 °C and good thermal stability up to 550 °C. Work is in progress to assess their physico-chemical and mechanical properties and to examine their biodegradability.

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DEVELOPMENT OF PHOTO-CROSS LINKED POLYETHYLENEIMINE NANOFIBERS AS CO_2 SENSOR

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The electrospinning process is a powerful tool to obtain polymeric fibers with diameter in the nano-micron range [1] This technique provides good mechanical properties and the process can also be carried out starting from small amounts of material [2]. The electrospun fibrous membranes with large specific surface used as sensing materials or templates would be an ideal candidate to replace the flat films. [3] In this study, polyethyleneimine was acrylated in the presence of triethylamine and was mixed with PVA solution. By utilizing UV and electrospinning technique at the same time, photo cross-linked polyethyleneimine based nanofibers were prepared. The thermal properties of nanofibers and polymers were examined by TGA and DSC. The morphology of nanofibers were investigated by SEM. The developed PEI/PVA nanofiber mats performed good selectivity toward CO_2 gas. These results demonstrate that PEI/PVA nanofiber is a promising material in development of a low-cost and high-performance CO_2 sensor.

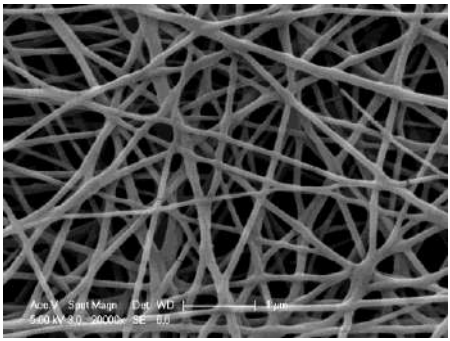

Figure 1: SEM micrograph of PEI/PVA nanofiber

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Acknowledgement
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NEW POLY(TRIAZOLO-IMIDE)S OBTAINED BY CLICK POLYADDITION

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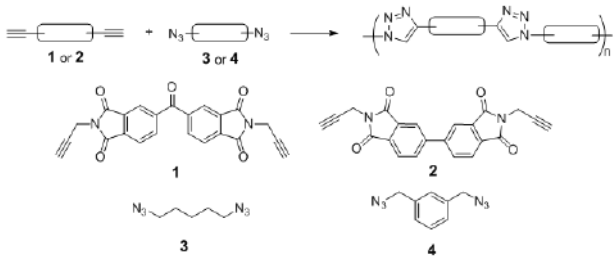
It is well known that aromatic polyimides have good thermal and mechanical properties and excellent resistance to solvents and chemical products allowing them to be ranked among very high performance polymers. They are employed for many applications such as electronics, microelectronics and aerospace industries [1]. Additionally, polyimides are used in a diverse range of applications, including the field of flexible electronics and displays, flexible and thin film photovoltaics, light emitting diodes, polymeric memories and membrane materials for separation [2]. Recently Ben Romdhane et al reported also their use for the storage and conversion of solar energy [3].

The usual approach to prepare polyimides consists on the polycondensation between bis-anhydrides and diamines via 2 steps. Polyamic acids produced in the first step give polyimides by thermal or chemical cyclization. However, in most of these reactions high temperatures are necessary and byproducts such as water are produced.
The aim of this work is to develop a new approach for polyimides synthesis by Cupper Catalyzed Azide Alkyne Polycycloaddition (CuAAC) . The general pathway adopted consists in reacting bis-(imide-alkyne)s and diazides monomers at low temperature by using Cu(I) as catalysts.(Scheme 1).



***Scheme 1** : Synthesis of poly(triazolo-imide)s via CuAAC of dialkynes (1,2) with diazides (3,4)*

Physical properties of the obtained poly(triazolo-imide)s were investigated by NMR, FT-IR and DSC. Inherent viscosities were carried out in NMP as solvent at 20°C.
This work conclusively shows that click polyaddition can be conducted in a simple and efficient way in view of making novel, high performance polyimides.

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Journal of Polymer Sciences Part A: Polymer Chemistry, 52, 223-231, 2014

ANALYSIS OF THE DOUBLE EFFECT OF THE COUPLING AGENT UPON THE STRUCTURE AND PROPERTIES OF SHORT GLASS FIBRE POLYPROPYLENE COMPOSITE

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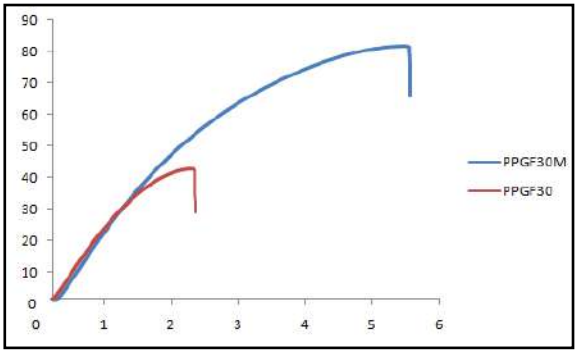
This research paper has an objective the exploration of glass fibre reinforced polypropylene which has been selected as an alternative to PA reinforced with the same rate and type of fibre usually employed in service dead-end clamps applications. As a matter of fact, PP is relatively cheaper and easier to process and it is for this reason that we have sought to optimize its mechanical properties by adding a coupling agent. To this end, we have undertaken a study of the structure and properties of modified and non modified 30 wt % glass fibre reinforced polypropylene.
Simultaneously, we have also conducted a comparative investigation of the composites in question to a commercially available 30 wt% glass fiber-reinforced and modified polypropylene.



Composites Stress- Strain curves, PPGF30: 30 wt% short glass fiber-reinforced polypropylene, PPGF30M: 30 wt% short glass fiber-reinforced and modified polypropylene.

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BIOCOMPOSITE BASED ON POLYLACTIDE ACID AND OLIVE SOLID WASTE: RHEOLOGICAL, PROCESSING AND MECHANICAL STUDIES

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The oil extraction industry generates two by-products, an aqueous effluent and a solid residue called the olive solid waste (OSW). The main problem regarding the disposal of OSW is to find an environmental and economical viable solution. A new valorization strategy has been carried out which consists in incorporating the OSW as a filler in biopolymer matrix.

The aim of the present work is to gain a fundamental understanding of the relationships between structure, processing conditions and final properties of these biocomposites. In this study, biocomposites based on poly(D,L-lactide) (PDLLA) and two OSW fillers were prepared by a twin screw extrusion under Argon inert gas with various filler contents and two kinds of OSW physicochemical treatments. The processing conditions were monitored to produce composites with well controlled physico-chemical, morphological and mechanical properties. It was highlighted that the inclusion of OSW under elevated temperatures resulted in the degradation of the matrix leading to a reduction of the viscoelastic properties and molar masses. Furthermore, we have demonstrated that the present degradation of PDLLA matrix can be attenuated using a chain extender agent named Joncryl and containing nine Glycidyl methacrylate (GMA) functions. Meanwhile, the effect of OSW with and without joncryl on the thermal properties as well the melt and the crystallization temperatures, the crystallization kinetic has been assessed. Furthermore, the rheological behaviour in linear viscoelasticity of the controlled PDLLA/OSW/ joncryl suspensions has been investigated in the molten state. The improvement of shear viscoelastic properties corroborates the mechanical ones. The created physicochemical interactions have to be taken into-account to explain the improved rheological and mechanical properties.

MODELING OF CONDENSED MODE FLUIDIZED BED REACTORS FOR POLYETHYLENE PRODUCTION AT STEADY STATE CONDITIONS

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In the gas phase olefin polymerization reactors the heat removal is of great importance. One efficient way is to use the condensed mode operation in which it is possible to increase the production rate as well. In this method, by using a non-polymerizing condensable material such as isopentane, it is possible to condense a portion of the recycle gas and return the liquid/gas mixture to the reactor. A larger amount of heat can be removed due to the latent heat of vaporization of condensed liquid entering to the reactor. In this study, the modeling of ethylene polymerization in Fluidized Bed Reactors (FBR) operating in the condensed mode at steady state is reported which can be used to predict to increase the production rate by altering operating conditions while the properties of the polymer remain constant.
Several aspects must be considered in the modeling of FBR operating in condensed mode. For kinetic modeling a multiple active sites mechanism for copolymerization of ethylene and 1-butene by Ziegler-Natta catalysts [1] was used. A simple two-phase model [2] with emulsion phase as a CSTR and bubble phase as a plug flow reactor with constant size of bubbles was employed for hydrodynamic modeling. For the average particle size, which has an influence on the prediction of hydrodynamic parameters, a population balance model [3] was chosen to predict the particle size distribution in the reactor. The Sanchez-Lacombe equation of state [4, 5] was also used for prediction of the concentration of reactants in the polymer phase, while the Peng Robinson equation of state was used for prediction of the concentration of reactants in the gas phase
To validate the developed model, its simulation results were compared with the industrial patent data [6]. The predicted polyethylene production rate and melt flow index (MFI) have good agreement versus actual data. Figure 1 shows the increase of the production rate versus the isopentane concentration in the reactor, as well as the weight fraction of liquid in the inlet stream. The change of the isopentane concentration around 1 mole percent leads to a variation of production rate about 8 weight percent.

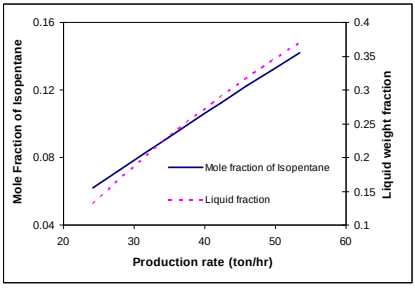

Fig. 1 effect of isopentane concentration on polymer production rate

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The Effect of the incorporation of the clay on Acrylic polymer

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The importance of the incorporation of clay as a filler in nanocomposite is encountered last twenty years when for the first time a research team of the laboratory Toyota, Okada et al. [1] showed many advantages of adding a low montmorillonite clay lamellar nanoscale in a polyamide matrix was late to improve their properties.

Our study is devoted to the synthesis of acrylic polymer and the development of new composite materials polyacrylate / clay which aims to improve intrinsic and extrinsic properties of these materials.

The clay used as filler in the matrix polyacrylate is completely dispersed after adding a small quantity of approximately 2%.

The results obtained by XRD, IR and NMR showed a good dispersion of the clay in the polyacrylic matrix, we can also say that the resin reacts well with the load in an exfoliated surface.

We also noticed an improvement in brightness, hardness and elasticity of the new composites. This composites can be used for various industrial applications such as water treatment as filtering in aeronautics (durability of adhesive joints) as an adhesive.

Keywords: Nanocomposite, Polyacrylate, Clay.

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Synthesis and characterization of graft copolymers of potato starch and acrylonitrile

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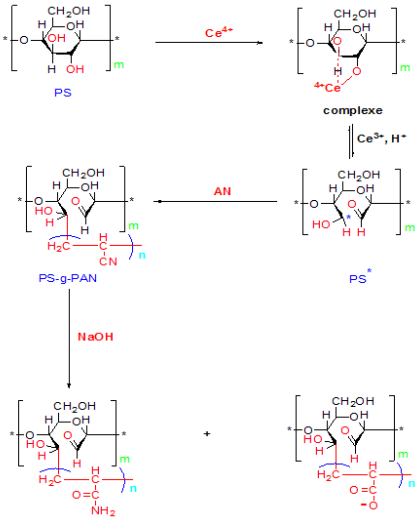
Chemical grafting is one the most popular methods for modifying structure and properties of biopolymers. Native starch granules are water insoluble containing two major polymers: amylose and amylopectin .Grafting of acrylonitrile (AN) on starch gives the possibility to perform numerous further chemical reactions and to synthesize new highly-added value materials for various application, such as: soil conditioners, additives for paper and textiles, adhesives, retention of heavy metals… [1-3].in the topic synthesis (Scheme 1) the copolymer of saponified acrylonitrile on starch was synthesize and characterize with RMN ^{1}H, ^{13}C, infra-rouge spectrum, SEM and DTA techniques.



Scheme 1: Mechanism of grafting AN on PS with Ce^{4+} as free radical initiator.

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CELLULOSE ACETYLATION BY FUNCTIONAL ANHYDRIDE - A POTENTIAL TOOL FOR THE DEVELOPMENT OF NON-BIOCIDAL WOOD TREATMENTS

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The use of wood as building material is increasing since many years. Although it is an ecological and renewable material, it is greatly competed by other materials such as concrete or plastic for outdoor use. A long lifespan is often required when wood is employed for outdoor applications and is difficult to ensure when constructions are little or not sheltered from weathering. When wood is used outdoors with repeated or continuous exposures to moisture, degradation by biotic and abiotic factors is high. Consequently, resorting to systems of protection is essential to ensure an optimal service life.

The increase of wood resistance against biological agents for outdoor use is conventionally performed by deep impregnation with preservatives. However, some of the traditional wood preservatives, used for decades for wood protection and regarded as the most effective (such as CCA), are currently subjected to severe restrictions because of their toxicity [1].

Increasing the hydrophobicity of wood can be an issue to reduce the continuous water exchange between wood and its environment which promotes fungal degradation and decreases wood's stability. Hydrophobic properties can be conferred by grafting functional monomers such as anhydrides in order to mask the hydroxyl groups responsible for the hygroscopic nature of wood [2]. Anhydride having a vinyl bond as itaconic anhydride allows a subsequent polymerization with acrylic monomers. This anhydride allows grafting non-biocidal polymers on the natural components of wood, such as cellulose, and can potentially limit their release to the environment [3]. In this study, grafting was optimized by taking into account various parameters such as catalyst, solvent or temperature. TGA, Infrared and NMR spectroscopy were used to investigate grafting in order to quantify and to identify the bonds established between wood components and the grafted compounds.

This research is a part of the TIMBIRDE project funded by the French FUI program (2012) and by France Bois Forêts, which the French Wood and Forestry Association.

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NEW POLYMERIC DISPERSANT FOR TITANIUM DIOXIDE PIGMENTS: SYNTHESIS AND CHARACTERIZATION

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The dispersion of pigments in fluid media is of great technological importance for coatings manufacturers who deal with pigmented systems[1]. Polymeric dispersants are claimed to be more effective dispersion stabilizers for non-aqueous systems. These substances have a two-part structure, one consisting of an anchoring functional group for pigments and the other one consisting of a polymeric solvatable chain[2].
In this work, several dispersants with different molecular weights named "Giral" were synthesized to improve the dispersion of titanium dioxide in a polyurethane system (PU). Their efficiency was investigated by particle size analyses in organic media and compared to a commercial dispersant commonly used in this field (figure 1a). Thus, the influence of these dispersants on the mechanical properties of paint film was evaluated (figure 1b).

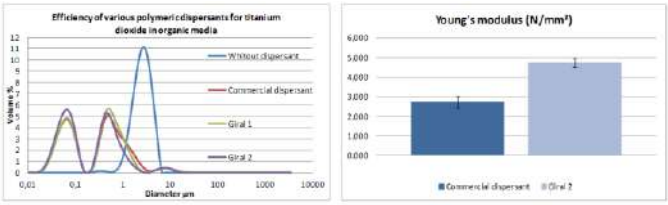

Figure 1: a) Particle size distribution of titanium dioxide in presence of dispersants (optimized mass percentage); b) Average Young's modulus for polyurethane paint formulated with Giral 2

Adding of commercial dispersant or "Giral 1" leads to the same particle size distribution of titanium dioxide. On the other side, the particle size distribution of TiO_2 with "Giral 2" is improved (compared to the commercial dispersant), leading to an improved dispersion. Furthermore, incorporation of "Giral 2" in PU system enhances mechanical properties, highlighting the best compatibility of "Giral" for this system.

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Study of the effects of the temperature and composition on dynamic viscosity and density of vinyl acrylic emulsions

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The emulsion copolymerization of vinyl acetate with unsaturated esters gives rise to products with high added values for adhesives, paints, cosmetics and pharmaceuticals.
For all those applications, the vinyl acrylic emulsions stability is important. Hence, it is necessary to build up the mathematical models linking the viscosity and the density to the experimental factors.
The aim of this investigation was to correlate the density and dynamic viscosity of industrial vinyl acrylic emulsions with the composition and the temperature
The results obtained revealed that the dynamic viscosity could be ascribed using Vogel-Fucher-Tamman equation where factors A and B depended on the industrial conditions and composition. Furthermore, the density follows an affine law depending on the composition, independently of the manufacturing conditions.

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STRUCTURAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF POLYANILINE / HUNTITE COMPOSITES

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Conducting polymers have become a popular basic material for advanced applications including plastic batteries, electromagnetic interference (EMI) shielding, electrochromic displays, and sensors. Among conducting polymers, polyaniline (PANI) has been extensively studied for its environmental stability in the conducting form, ease of synthesis, low cost and high conductivity [1-3].

The aim of this study was to improve thermal stability of polyaniline (PANI) by adding Turkish Huntite. PANI was synthesized and PANI/Turkish Huntite composites were prepared by adding various proportions of huntite to aniline. The chemical structures of the samples were investigated by Fourier transform infrared spectroscopy. Thermal properties of the composites were determined by thermogravimetric analysis and differential scanning calorimetry. The surface morphologies of the samples were investigated by a scanning electron microscopy. The obtained results proved that the composite system is more thermally stable than the pure PANI.

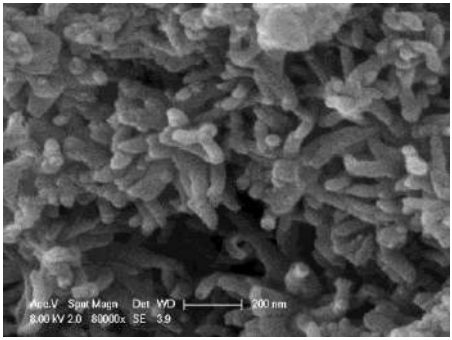

Figure 1. Sem Micrograph of Polyaniline/Turkish Huntite-Hydromagnesite Composites

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Acknowledgement
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POLY (ESTER-AMIDE)S AND POLYDEPSIPEPTIDES BASED ON AMINOACIDS

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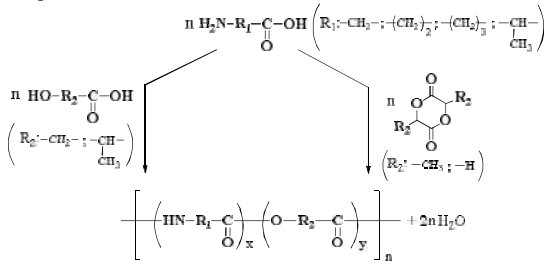
Poly (ester-amide)s (PEAs) can be generically defined as polymers with ester and amide linkages in their polymeric chain, with properties between those of polyesters and polyamides. The combination of ester and amide can open avenues in the design of new materials with different properties (*e.g.*, thermomechanical and degradability) for use in biomedical applications [1]. Different monomers (*e.g.*, α-amino acids, α-hydroxy acids, cyclic depsipeptides, fatty diacids, diols, α,ω-aminoalcohols, carbohydrates) have been used in the synthesis of PEAs to afford polymers with different properties.

It is in this context that this work was done. Our objective is the synthesis and characterization of novel PEAs and polydepsipeptides (PDPs) by two different methods: (i) By condensation between an α -hydroxy acids and amino acids. Different polymers have been synthesized by modifying both the nature of the amino acid (γ - aminobutyric acid (GABA), β -alanine (β -Ala) and glycine (Gly)), and the nature of α -hydroxy acid (glycolic acid (GA) and lactic acid (LA)) .These polymers were synthesized by the method of melt polycondensation in the presence of dibutyl tin oxide ($(Bu)_2$ SnO) as catalyst . The reaction was carried out in two phases, under flowing nitrogen and under reduced pressure. (ii) By Ring Opening Polymerization (ROP) of the cyclic ester (lactide, glycolide) in the presence of the amino acids. The polymerization is carried out in bulk and in three phases in the presence of stannous octoate (tin 2-ethylhexanoate) ($(Oct)_2Sn$) as catalyst , introduced during the second phase.



These different PEAs and PDPs were obtained by varying the initial weight ratio of the two comonomers from 10/90 to 90/10. All polymers were characterized by FTIR spectroscopic techniques, ^{1}H NMR, ^{13}C NMR. These analyzes confirm clearly the expected polymers, synonymous with the smooth running of the copolymerization reaction. The thermal study (DSC and TGA) of the PEAs shows that the glass transition temperatures and degradation temperatures are closely linked to the amide moiety in these PEAs. And to evaluate the molar masses of the samples, these polymers were characterized by Size Exclusion Chromatography (SEC).

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MANIPULATION OF BIO-ADHESION BY THERMOPLASMONIC ACTIVATED PHASE-CHANGES

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In cell biology and tissue engineering, the control of when and where cells can attach or migrate on a given substrate is a challenging task. A sub-micron cell adhesion pattern in combination with the ability to reversibly adsorb or detach proteins or cells would enable interesting investigations in this field.

Here we propose a concept for realization of active, dynamic and reversibly controlled adhesion landscapes for bio-objects (proteins or cells). It is based on the local optical heating of homogeneous layers of the thermo-responsive polymer poly(N-isopropylacrylamide) pNIPAM, which undergoes a reversible hydrophilic/hydrophobic transition at roughly 32°C [1]. By this transition, the adhesion of proteins or cells can be strongly modified and it was demonstrated that these bio-objects can be reversibly attached / detached from such a substrate [2]. By a local and temporal modification of substrate temperature, arbitrary and time dependent landscapes of biomolecule affinity could be created. This was realized in the present work by optical heating with the help of gold nanoparticle arrays beneath the pNIPAM. The light absorbed by the gold nanoparticles is converted to heat by the thermoplasmonic effect, which allows for a precise control of spatial and temporal temperature distribution by designing illumination pattern, plasmonic resonances and laser power. Such substrates open new opportunities for the realization of spatially and temporally controllable bioadhesion landscapes.

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Electrosynthesis, Characterization and Electrocatalytic Behaviour of Organic–Metal thin-Film based on 2,2'-Bithiophene-5-carboxylic Acid and Metallic Ions

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2,2'-Bithiophene-5-carboxylic Acid (BTA) thin films on Pt electrodes were electrochemically prepared in a CH_3CN solution containing 0.1 M tetrabutylammonium perchlorate (TBAP) and 0.05 M BTA. These films were complexed with several metal ions such as Cu^{2+}, Ag^{+} and Co^{2+} after their incubation in metallic solutions. A comparison of their structural characteristics with those of powder complexes chemically prepared from BTA and corresponding metal ion was made. IR and XPS techniques reveal that the films complexed with metal ions have the same structures that those of corresponding powder complexes.

The electrocatalytic activity of BTA films-metal ions has been investigated toward Ascorbic acid (AA) oxidation and compared to that obtained on a free BTA-film. We have shown that BTA films-metal ions exhibit good catalytic proprieties and high performances to detection of AA than that a free BTA-film. This new propriety allows using these films as electrochemical sensors.

Keywords: 2,2'-Bithiophene-5-carboxylic acid, metallic ions; cyclic voltammetry, IR and XPS techniques; electrocatalysis; Ascorbic acid.

Decomposition of Yttrium acetate in the form of films

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The processes involved in the thermal decomposition of thin films of yttrium acetate tetrahydrate, $Y(CH_3COO)_3 \cdot 4H_2O$, in air and in an inert atmosphere have been analyzed by thermoanalytical techniques (thermogravimetry, differential thermal analysis and evolved gas analysis) and by the structural characterization (X-ray diffraction, infrared spectroscopy, elemental analysis and scanning electron microscopy) of intermediates and final products. Decomposition of yttrium acetate is an endothermic transformation that takes place in a temperature range between 350 and 580°C.The evolution of the mass during the decomposition process is not affected by the presence of oxygen. The process is initiated by the rupture of the bond between the metallic cation and the acetate ligand. This initial step (350–450°C) involves the formation of amorphous yttrium hydroxide and yttrium carbonate and is characterized by a fast mass loss rate.

A sudden decrease of the mass loss rate indicates a change in the decomposition kinetics that continues with the decomposition of yttrium hydroxide and yttrium carbonate. The main effect of an oxygen atmosphere is an intense exothermic process due to the combustion of organic species in the gas phase.

Keywords: Yttrium acetate tetrahydrate, thermoanalytical, X-ray diffraction and Decomposition.

Elaboration of biocomposite materials PLA with celluloses fibers

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As part of the industrial and technological developments, new materials have to be developed. Many researches show that composites play an increasingly important role (lighter, more efficient or both) [1]. In addition, many studies in the area seek to prevent the shortage of petrochemical resources in developing the use of natural fibers and the use of new biopolymer [2]. Polymer blending constitutes a most useful method for the improvement properties of polymeric materials. In order to elaboration a new biocomposite material between PLA and cellulose fibre, extracted by Tunisian flora [3]. The main objective of this work is is the shaping of new bio composite materials. The way in which we focused, is the use of polymers from renewable resources [ ] and elaborating this material with mixtures PLA and cellulose fiber by melt extrusion [4]. The presence of 10 % of the fibers leads to improve mechanical and thermal properties which attest the crystallinity growth of the studied material. Finally, we investigate the biodegradability of the material [[5]].

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CHEMICAL MODIFICATION OF POLYVINYL CHLORIDE WITH AROMATIC AND ALIPHATIC AMINES. SYNTHESIS, CHARACTERIZATION AND EXTRACTANTING PROPERTIES FOR SOME METALS.

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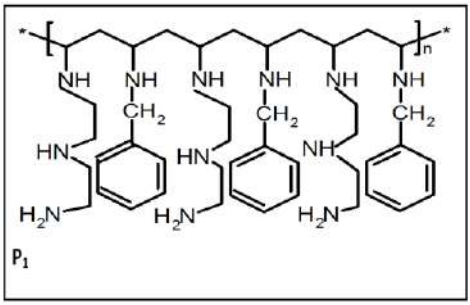
The polyvinyl chloride is the most used plastics in the world thanks to their thermal and physical properties [1-2]. The polymer has been subjected to many changes, mainly chemical modification [3]. In this work we substituted PVC with amino groups (aromatic and aliphatic). The obtained structure of P_1 was proved by the different techniques of physicochemical analysis: IR spectroscopy, DSC and elemental analysis. The modified polymer by different groups was tested for his extracting properties of some metals (Cu, Zn, Cd, Pb). The study of this extraction with modified PVC was followed by measuring electrical conductivity and atomic absorption.



P_1

Key words: PVC, chemical modification, extraction.

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DELAY EFFECT EVALUATION OF LIBERATION OF ANTIPYRINE DISPERSED IN MATRIX COPOLYMERE BASED ON N-2VINYLPYRROLIDONE

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In the galenic industry, the use of copolymers synthesized as carriers of active principles was the object of several works because of their very numerous functional properties considered very interesting [1]. These last years, the delayed effect of active principles constituted the main axis of search for numerous laboratories [2-3].
In this objective, we were interested to co-polymerize by radical way N-2-vinylpyrrolidone with the methacrylate of methyl, in the presence of AIBN. This last one was characterized by FTIR, DSC and the mass were determined by viscosimetry.
The active agent Antipyrine AP with pharmacological property, was scattered in a matrix of copolymer poly (N-2-vinylpyrrolidone co-methacrylate of methyl), forming a disk with mass composition 80/20. The liberation of the AP was established in the gastric environment of pH = 1.2.
The process of liberation of the AP is controlled by the distribution of the liquid inside the galenic shape and the active agent towards the outside. The rates of active agent freed after 8 hours is slightly strong overtake the 65%. This diffusion speed of the active principle through the disk is also influenced by several parameters due to the nature of the active principle; in the pH, the molar mass of the copolymer. Finally the diffusivities of the active agent and the liquid transferred in the galenic shape were calculated.

Keywords: N-vinyl-2-pyrrolidone, copolymer, Antipyrine liberation, delayed effect.

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New organic-inorganic hybrid crystal structure of of 1,3,5-triazinidium-2,4,6-triamine hexachlorodicuprate (II)

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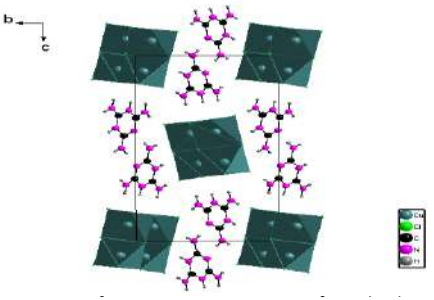
The crystal structure of $(C_3N_6H_8)Cu_2Cl_6$ has been determined at room temperature by single crystal X-ray diffraction. The compound crystallizes in the monoclinic system with $P2_1/C$ (N°14) space group. At 293 K, the unit-cell dimension are a=6.6586 (3) Å; b=10.0556 (4) Å; c=19.2638(7) Å; β=96.040 (3) °; V=1282.67(9) $Å^{-3}$, Z=4 and D_x= 2.423 $g.cm^{-3}$. The crystal structure consists of two asymmetric inequivalent molecules of 1,3,5-triaminidium-2,4,6-triamine and anionic Cu_2Cl_6 chains. In the structure $(C_3N_6H_8)$ Cu_2Cl_6 the hydrogen bonds are of two type N–H...Cl and N-H…N. X-ray data were collected at 293 K transparent and dark brown crystal of dimensions 0.166x0.169x0.360 mm^3 using a Brucker Kappa CCD area detector diffractometer, with MoKα radiation (λ=0.71070 Å). The final cycle of refinement of all isotropic and anisotropic thermal parameters led to R= 0.035 and Rw = 0.088. The molecular structure of the composite is one-dimensional chains of octahedral dimmers $[Cu_2Cl_6]^{2-}$ extending the axis a, being placed approximately at the vertices and center of the face (b,c) of the mesh and into the gaps these chains are housed cationic dimmers $(C_3N_6H_8)^{2+}$.



Crystal data			
Empirical formula	$[C_3N_6H_8]$ Cu_2Cl_6	Data collection	
Formula weight	467. 95	Reflections collected	10403
Crystal system	Monoclinic	Independent reflections	[R_{int} = 0.0449]
Space group	$P2_1/c$	Reflection with I>2σ(I)	2001
Hall symbol	-P 2ybc	Limiting indices	h= -8→8
Unit cell dimensions			k= -12→11
a(Å)	6.6586 (3)		l= -23→23
b(Å)	10.0556 (4)	CCD area detector diffractometer	T_{min}=0.410, T_{max}=0.467
c(Å)	19.2638(7)	Radiation source: fine-focus sealed	θ_{max}= 25.38 °, θ_{min}=2.29 °
β(º)	96.040 (3)	Tube graphite φ and ω scans	
Volume($Å^3$)	1282.67(9)	Absorption correction: multi-scan	
Z	4	North et al. (1968)	
D_{cal} ($mg\ m^{-3}$)		Refinement	
Absorption coefficient (mm^{-1})	4.548	Refinement method	Full-matrix least-squares on F^2
F (000)	912	R [F^2>2σ (F^2)]	0.0353
Crystal dimensions (mm^{-1})	0.166×0.169×0.360	WR (F^2)	0.0887
Crystal color	Needle-shaped/Brown	Goodness-of-fit on F^2	S=1.167
ΘRange for data collection (º)	3.44 - 22.21	Data/restraints/parameters	
		Extinction coefficient	0.0000

Characterization of geopolymer synthesised from Tunisian illitic clay

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Geopolymer has been synthesized from Tunisian raw and calcined illite by activation with NaOH, KOH and sodium silicate solution. The reaction product was compressed in cylindrical mould and then cured at 60°C in oven. Compressive strength and apparent density and porosity of the material were systematically measured. The effect of concentration of NaOH and KOH solution on the mechanical and chemical properties of the geopolymer was investigated by means of X-ray diffraction (XRD) and infrared spectrometry (IR). Results show compressive strength and apparent density of the geopolymer increase along with the increase of concentration of NaOH solution within 8–15 mol/L. XRD and IR analyses indicate the géopolymère material is composed of amorphous phase and semi-crystalline phase, containing adsorbed atmospheric water and small amount of inert ingredients originated from the illite. Content of the amorphous phase in the geopolymer increased along with the increase of NaOH and KOH concentration. Strength data of the material show such a colloids reaction based on calcined illite clay could make building materials with good quality, the 28-day compressive strengths up to 34 MPa.

Hybrid Nanoparticles: Plasmonic, SERS and phase transition properties

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The ability to synthesize and assemble monodisperse core-shell nanoparticles is important for exploring and tailoring their unique properties at the nanoscale and for technological applications in various fields (optoelectronics, photovoltaics, solar water splitting) including in nanomedecine (cancer cells imaging and treatment).

In this paper, we report on a simple, facile, and efficient new approach to synthesize core-shell Au@Fe_3O_4 nanocomposites (NCs) owing to a modified polyol process that makes use of triethyleneglycol as a solvent.

The preparation of bifunctional Au@Fe_3O_4 (NCs) via "One-pot" synthesis involved an initial synthesis of Au nanoparticles by thermal reduction of the Au precursor as the cores and a subsequent thermal decomposition of $Fe(acac)_3$ onto the surface of the preformed Au nanoparticles at high temperature.

Au@Fe_3O_4 NPs have been characterized by Transmission Electron Microscopy (TEM) and Energy Dispersive X-rays Spectroscopy (EDX). Au@Fe3O4 appear as formed of Au nanoparticles cores 90 nm in diameter decorated with magnetite nanoparticles 7nm in diameter. The optical properties were investigated using absorption spectroscopy and Surface Enhanced Raman Scattering (SERS). The latter evidenced that the NP shell was formed by Fe_3O_4 (magnetite). The surface plasmon absorption band of Au@Fe_3O_4 NCs is broadened and red-shifted relative to monometallic Au nanoparticles, these observations reflect the strong interaction between Fe_3O_4 and Au. This is confirmed by the simulations of the extinction spectra performed using the discrete dipole approximation (DDA). These heterostructures exhibit both plasmonic and magnetic properties and may give rise to a new class of multifunctional materials.

The synthetic strategy paves the way for facile one-pot preparation of Au-Fe_3O_4 nanoparticles without addition of any capping agent, template or complex metal ligands.

Facile synthesis of monodisperse triangular gold nanoplates based on a polyol process

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A simple and low-cost chemical synthesis method of monodisperse triangular gold nanoplates (Tr-AuNPs) is reported. This novel approach allows one to produce Tr-AuNPs owing to a modified polyol process that makes use of Triethyleneglycol (TREG) as a solvent in the presence of the polymer capping agent poly(vinylpyrrolidone) (PVP). Both the solvent and the surfactant play important roles in the formation of the Tr-AuNPs. The size and the shape of the particles could be tuned by changing the molar ratio of PVP to metal salts. The formation of, single-crystal Tr-AuNPs is explained by the preferential adsorption of some species of molecules from the solution onto the {111} planes of Au nuclei, and the connection of small, triangular nanoparticles. Due to their rather large size and to their triangular shape, the surface plasmon absorption band of the nanoparticles is strong and red-shifted to 720 nm. Near infrared spectral region of these nanoparticles might find use in areas that include photonics, optoelectronics, optical sensing and also in nanomedicine.

SYNTHESIS, CHARACTERIZATION AND HYDROLYTIC DEGRADATION OF NEW BIOBASED (FURANIC-SULFONATED COPOLY(ESTER-AMIDE)S

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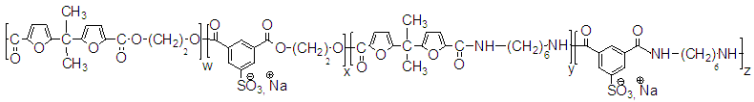
Polymers from renewable resources have attracted an increasing amount of attention over the last two decades, predominantly due to two major reasons (i) firstly environmental concerns, and (ii) secondly the realization that our petroleum resources are finite. Furanic polymers have been considered as alternative environmentally friendly polymers [1]. In our earlier work, modifying furanic polyesters by incorporation of amide functions along their backbone, lead to a particular class of polymer "poly(ester-amide)s", was investigated to combine the excellent mechanical properties of polyamides and the biodegradability of polyesters [2]. As a continuation of our studies on this family of polymer, a series of furanic poly(ester-amide)s bearing sulfonate groups in the main chain were synthesized from 5,5'-Isopropylidene-bis(ethyl 2-furoate), dimethyl 5-sodiosulfoisophthalate, ethylene glycol and hexamethylene diamine by melt polycondensation using zinc acetate as a catalyst. In view of the complexity of the NMR spectrum analysis of the resulting sulfonated poly(ester-amide)s, we found that it is useful to prepare initially the corresponding homopolymers: sulfonated polyesters and polyamides. Structural data of these polymers will be used as a basic element in ^{1}H NMR characterization. The hydrolytic degradation in acidic aqueous conditions (pH= 4,35) at 37°C over the period of four weeks show that the mechanism of the hydrolysis of poly(ester-amide)s was elucidated in relation with the microstructure.

The strong intermolecular hydrogen bonding interactions between amide functions and water molecules increases the hydrophilicity of the macromolecular chains and consequently their hydrolytic degradation.



Furanic poly(ester-amide) bearing sulfonated groups

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Theoretical Study of New Conjugated Polymers Containing Thiophene and 1,3,4-Oxadiazole or 1,3,4-Thiadiazole in the Main Chain

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We studied the structural and optoelectronic properties of the model polymer Poly-Phenylene-Vinylene (PPV) based on 2,5-bis(5-phosphonate-2-thienyl)-1,3,4-Oxadiazole ; 2,5-bis(5-phosphonate-2-thienyl)-1,3,4-thiadiazole and 1,3-benzenedialdehyde ; 1,4-benzenedialdehyde using Density Functional Theory (DFT) with B3LYP/ 6-31G* calculations and SCF with base standard 3-21G.

With :
X= O : 2,5-bis(5-phosphonate-2-thienyl)-1,3,4-Oxadiazole
X= S : 2,5-bis(5-phosphonate-2-thienyl)-1,3,4-thiadiazole

1,3-benzenedialdehyde (m-linkage)
1,4-benzenedialdehyde (p-linkage)

PREPARATION OF THE POLY (N-2-VINYLPYRROLIDONE-CO-VINYL ACETATE) FOR APPLICATION MATRIX-TYPE LOADED MICROSPHERES OF 2-AMINOBENZOTHIAZOLE.

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The aim of this study is to prepare and characterize the poly (N-2-vinyl pyrrolidone-co-vinyl acetate) P(VP/VAC) copolymers that were used in combination with ethyl cellulose EC for application in the preparation of matrix-type microspheres of 2-aminobenzothiazole (ABT).

It has been prepared and characterized two vinyl copolymers (CP_a) and (CP_b) by radical copolymerization using the couple of comonomers N-2-vinyl pyrrolidone (VP) /vinyl acetate (VAC). Mass copolymerization (AIBN 1%) leads to white solid copolymer. The slightly yellowish solid copolymer was prepared in solution in dioxane (0.3% AIBN). Copolymers were recristallised from petroleum ether. These copolymers were characterized by IR, RMN ^{1}H, ^{13}C,DSC and the $\overline{M}_v$ molecular masses were determined.

Microspheres of ABT were prepared by the emulsion solvent diffusion method [1-3]. Formulation microspheres were prepared by using P(VP/VAC)/EC as polymeric matrix retardant materials. Scanning electron microscopy (SEM), Encapsulation efficiency, the yield, particle size, morphology of microspheres, and DSC were evaluated.

Key words: Radical Copolymerization, P(VP/VAC), Ethyl Cellulose, Emulsion Evaporation Technique.

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SYNTHESIS AND CHARACTERIZATION OF NOVEL BIO-BASED PLATFORM FROM DIANHYDROHEXITOLS FOR APPLICATION IN POLYMERS SYNTHESIS.

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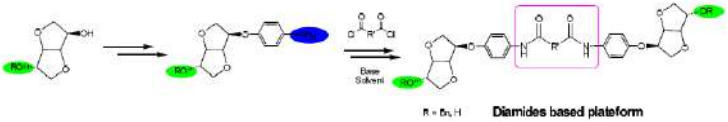
Campus unversitaire, 2092 , (Tunisie).

The development of renewable resources has become a main field of research in both academic and industrial scale in recent years. 1,4:3,6-dianhydrohexitols (**DAH**) derivatives were used as monomers and building blocks for new polymeric and functional materials, medical and pharmaceutical applications, new organic solvents and even as fuels or fuel additives[1]. The low reactivity of the secondary alcohol groups of **DAH** has encouraged the researchers to design more reactive functionalities such as diamines, diacides, dinitriles and diazides…[2,3,4,5] . In this work we report a new platform of diamides based on 1,4:3,6-dianhydrosorbitol, commercially known as isosorbide (**Is**), which can be used for the synthesis of co-poly(ether)-amides, co-poly(ether)esters-amides and co-poly(ether)urethane-amides. At first, the protected **Is** allowed via SNAr in solvent free conditions, to obtain
the nitroaromatic compound on the *endo* hydroxyl group. A selective reduction of the nitro group, gave rise to the corresponding amino compound which was reacted with several aliphatic and aromatic diacyl chlorides. This versatile strategy allowed obtaining the desired compounds with good yields. Characterization of this novel class of monomers and identification of signals was ascertained by 1D and 2D liquid-state NMR and confirmed complementary analytical techniques. These building blocks are expected to afford a variety of co polymers with interesting properties.



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NEW BIOSOURCED AA AND AB MONOMERS FROM 1,4:3,6 DIANHYDROHEXITOLS

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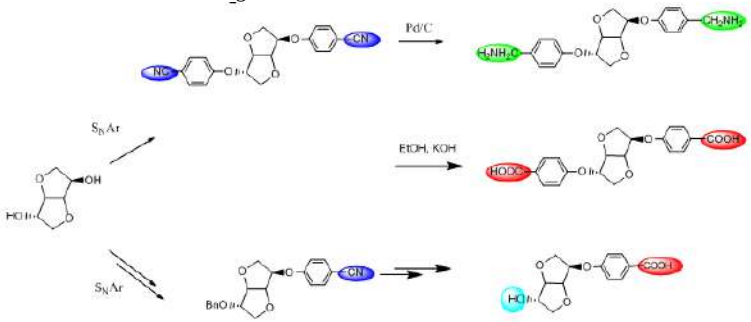
The majority of polymers used for commercial purposes originate from oil-based monomers. Polymers derived from starch or other carbohydrates are of great interest since these materials are made from entirely renewable resources. The direct use of monomeric carbohydrate derivatives is extremely difficult because of a number of hydroxyl functionalities in these compounds rendering the synthesis of well-defined products a very difficult task. This problem can be circumvented by using 1, 4:3,6- dianhydrohexitols for the synthesis of numerous polymers.[1,2,3]

In this context we propose herein unprecedented biosourced monomers which exhibit AA or AB unsymmetrical functions suitable for high performance composite materials.

We have prepared three new monomers from isosorbide starting from versatile phenyl nitrile compounds. The latter could be obtained by S_NAr reaction between isosorbide and *p*-fluorobenzonitrile in basic medium and solvent free conditions with 18-C-6 as phase transfer agent. The dinitrile lead to the obtention of diamine compound as previously reported.[4] As an extention the hydrolysis of the latter lead to the quantitative obtention of the corresponding diacid monomer by simple precipitation and filtration. The protected isosorbide diol would further give rise to the desired AB monomer.



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Theoretical study of the copolymer based on carbazole and 4,7-di(thiophen-2-yl)-2,1,3-benzothiadiazole

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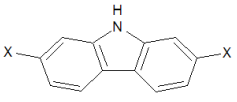
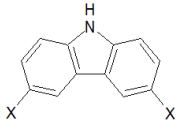
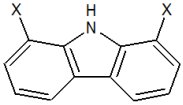
In this work we are studied the possibility and the regioselectivity of reaction of polymerization between a 2,7 carbazole ;1,8 carbazole ; 3,6 carbazole and 4,7-di(thiophen-2-yl)-2,1,3-benzothiadiazole.In addition we studied the structural and the electronic properties of the molecules ,HOMO,LUMO and E_g (HOMO-LUMO) using DFT (B3LYP/6-31G*) , SCF with base standard 3-21G* and .



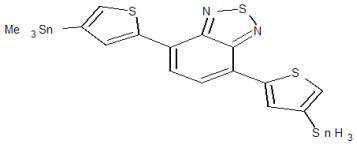
2,7 carbazole



3,6 carbazole

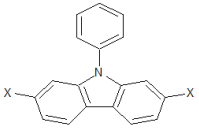


1,8 carbazole

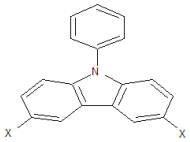


4,7-di(2'-triméthylstannylthièn-5'-yl)-2,1,3benzothiadiazole

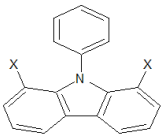
Figure 1



2,7 carbazole



3,6 carbazole



1,8 carbazole

Figure 2

RANDOM AND ALTERNATED POLYESTERAMIDES DERIVED FROM β-ALANINE AND ε-CAPROLACTONE

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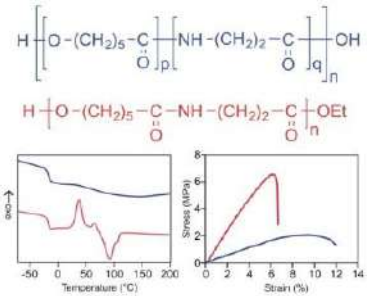
A series of polyesteramides (PEA1) were successfully synthesized by the melt polymerization of various ε-caprolactone and β-alanine mixtures. Model reactions carried out between ε-caprolactone and dodecanoic acid or dodecylamine in the bulk show that the copolymerization likely occurs in two steps: In the first step, CL reacts with the amine group of β-alanine leading to a hydroxy- and carboxy-terminated dimer. The hydroxyl groups of this dimer can then initiate the ring opening polymerization of excess CL, if any. In the second step, slower polycondensation reactions take place: Polyesterification of the dimer, polyamidation of β-alanine and copolycondensation of the dimer with β-alanine, leading to high-molar-mass random polyesteramides (Mw =36400 to 92800 g.mol^{-1}). As expected, the glass transition temperature of these PEAs decreases when CL content increases. These copolymers are semi-crystalline for CL contents of 60 mol-% and above (Tm =29 to 56 °C). A PEA with high amount of alternating CL and β-Ala units (degree of randomness = 1.63), containing 50 mol-% of CL units, was prepared by the melt polycondensation of a hydroxyl and ethyl ester-terminated amide. In opposition to the random PEA1-50/50 of similar composition, which is amorphous, the quasi-alternating PEA2-50/50 is semi-crystalline (Tm = 92 °C) and exhibits notably higher Young's modulus and ultimate tensile strength than its random counterpart.



Keywords: Polyesteramide, ε-caprolactone, β-alanine, polycondensation, microstructure, NMR.

DOUBLE-WALLED CARBON NANOTUBE DISPERSION VIA BIOPOLYMERS

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Reaching a stable suspension of carbon nanotubes (CNT) in water is still a challenging and time consuming research task. Addition of surfactants is desirable as it allows keeping intact the intrinsic properties of the CNT. However, as demonstrated in different applications, the potential toxicity of the surfactant is a key concern. The biopolymers in nontoxic amphiphilic matter have the potential to providing an alternative.

In this research, we are investigation the aqueous dispersions by bio molecules and polymers nontoxic and natural origin. The objective is to pave the road for an innovative family of bimolecular that can increase the dispersion of CNT in aqueous media. This could be very useful for research on their toxicity in the biological systems.

Accordingly, our ongoing study consists of the formulation and the characterization of suspensions of double-walled carbon nanotubes using some biopolymers such as sodium caseinate, the lecithin of soya and the bovine serum albumin**.**

The operating conditions of dispersion such as the power and the duration of sonication as well as the dispersant/NTC report/ ratio, were optimized.

The stability was demonstrated using measurements of potential zeta in the course of time and by the technique at the Turbiscan. The interactions between biopolymers and CNT were confirmed using spectroscopy FTIR. The characterization was supplemented by a study of the influence of the pH on the parameters such as viscosity and the zêta potential.

The sodium caseinate for a very weak concentration shows the best results in terms of duration and homogeneity of dispersion. This is a very promising result for the continuation of our research, that consists to investigate ecotoxicity.

Doping polypyrrole particles by removing of Cd^{2+}, Co^{2+} from aqueous solutions

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The development of simple methods for removing heavy metals from aqueous samples is a relevant field of research. For this, various techniques are used such as chemical sedimentation [1], surface absorption [2, 3] ions-exchanger [4, 5] and reverse osmosis [6].
In the same aims, the objective of this study is to investigate the removal of Cd^{2+} and Co^{2+} ions, from aerated aqueous solutions by complexing them with polypyrrole particles. A mass of polypyrrole powder was immersed in solutions of metallic salts ($CoSO_4$, $CdSO_4$), with concentration of 10^{-2} M for 48 h, at room temperature, then the mixture was filtered and dried. The obtained complexes were characterized by analytical methods**:** FTIR spectroscopy, cyclic voltammetry, X-ray diffraction, atomic force microscopy. Conductivity measurements of the polypyrrole and complexes were made using the four-points method.
The results show that the FTIR comfirmed the metal coordination with azomesthine nitrogen of polypyrrole [M-N] and the complexes present an amorphous structure which was determined by XRD. The voltammetric reponse of PPy modified with metallic microparticles showed higher current and a shift in the peaks potentials compared to unmodified polymer electrode. the conductivity of complexes are very important in presence of metallic cations, particullarly in the case of Co.
From economic view, the new composites open us many application in different fields as catalysis, batteries, fuel cells or microelectronics.

Keywords: heavy metals, polypyrrole, complexes, doping, conductivity

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POLYPYRROLE / HEXAGONAL BORON NİTRİDE NANOCOMPOSITES

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PPy is one of the most popular ECPs for various electronic applications such as the secondary battery, EMI shielding, light emitting diode, chemical and biochemical sensors and conducting composite materials, because of its high conductivity, environmental stability to water and oxygen and ease of synthesis [1]. Hexagonal boron nitride (h-BN) is the most preferred polymorph among other boron nitrides. It is an isoelectronic analog of graphite in which boron and nitrogen atoms are positioned alternatively to form planar conjugated layers. It is a white colored powder with a melting point of approximately 3000 °C [2].

The aim of this study was to improve thermal stability of polypyrrole (PPy) by adding hexagonal boron nitride (h-BN). Polypyrrole (PPy) was synthesized and PPy / h-BN nanocomposites were prepared by adding various proportions of h-BN to polypyrrole. The chemical structures of the samples were investigated by fourier transform infrared spectroscopy (FT-IR). Thermal properties the nanocomposites were determined by thermogravimetric analysis (TGA), differential scanning calorimetry (DSC). X-ray diffraction (XRD) analysis of the samples was carried. The surface morphologies of the samples were investigated by a scanning electron microscopy (SEM-EDS). The obtained results prove that the nanocomposite system is more thermally stable than the pure PPy.

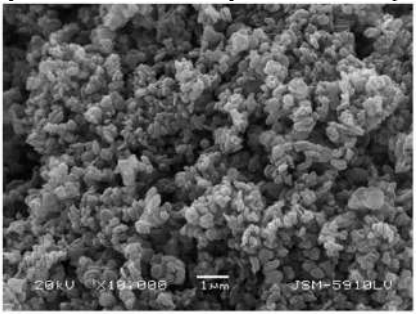

Figure 1. Sem Micrograph of Polypyrrole / H-Bn Nanocomposites

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Host-guest inclusion complex between β-cyclodextrin and sulfonamide: A theoretical approach

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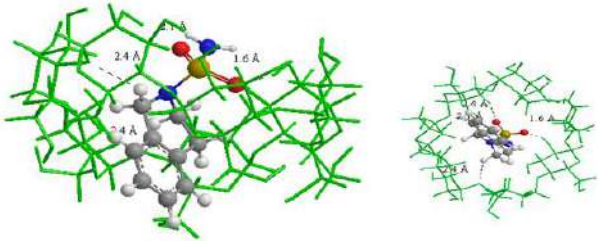
Cyclodextrin (CD) chemistry has caused much interest, not only due to its applications in pharmaceutical science and technology but also because the inclusion represents an ideal model mimicking enzyme–substrate interactions [1,2], Furthermore, The resultant inclusion complexes can induce modification of the physicochemical properties of the 'guest' molecules, particularly in terms of water solubility and solution stability [3–5]. The sulfonamide group is considered as a pharmacophore, which is present in number of biologically active molecules, particularly antimicrobial agents [6]. Hence, this work is designed to investigate the complexation of sulfonamide/β-CD through molecular modeling, using semi-empirical PM3 and PM6 methods, quantum hybrid ONIO2, in vacuum and in water. Two different docking orientations were considered. The energetically more favorable structure obtained with the ONIOM2 method leads to the formation of intermolecular hydrogen bonds between sulfonamide and β-cyclodextrin. These interactions were investigated using the Natural Bond Orbital (NBO). Moreover, the statistical thermodynamic calculations at 1 atm and 298.15K showed that the inclusion reaction is an exothermic process [7].



Keywords: 3,4-Dihydroisoquinoline-2(1H)-sulfonamide, β-Cyclodextrin, Inclusion complex, PM6.

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Valorization of polystyrene waste foam as polymer/clay nanocomposite: Application as methanol fuel cell membrane

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Polymer clay nanocomposites can be prepared via several differents methods, including in situ polymerization, melt compounding, or solution intercalation. In this paper, we investigate the solution intercalation method to valorize polystyrene foam waste as nanocomposite. A second step of this work is using this new valorized material as methanol fuel cell membrane. In this context, we have tested thepermeability behaviour toward methanol and the water uptake.

The sulfonation of polystyrene has been carried out using H_2SO_4 as sulfonating agent and ($CHCl_3$) as a solvent to dissolve polystyrene waste and to disperse the clay pallets. We have tested naturel clay without modification and an acid treated one while we have varied the proportion of clay to polymer from 1 to 5 wt%. These nanocomposites were characterized by X-ray diffraction, scan electron microscopy, thermogravimetric analysis (TGA) and calorimetry.

The water absorption of the membranes is usually defined in weight percent with respect to the weight of the dry membrane. The membranes initially absorbed large amount of water, which was followed by only a gradual raise in absorption towards the saturation point.

The effect of montmorillonite clay on exfoliatione behavior, degree of sulfonation and cristallinity of sulfonated ionomer nanocomposites was systematically studied. X-ray diffraction and SEM were used to evaluate the dispersion of clay platelets within sulfonated ionomer matrices. Experimental results obtained from XRD and SEM revealed a predominately exfoliated morphology within the sulfonated polystyrene ionomer containing 1-5 wt% of clay and acid treated clay.

Keywords: chemical modification;In situ Polymerization; polystyrene; sulfonated of polystyrene; Nanocomposites; Physico-chimique and properties applications; Solvent absorption.

SYNTHESIS AND CHARACTERIZATION OF SEMICONDUCTING POLYROTAXANE BASED ON ANTHRACENE AND β-CYCLODEXTRIN

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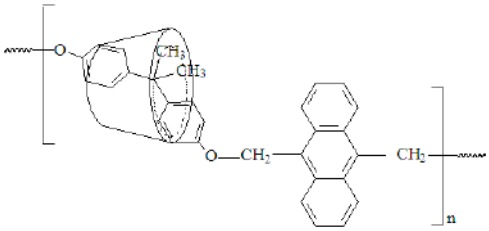
A new anthracene-based polether with rotaxane architecture (**PBPAAn/β-CD**), containing non-covalently bounded β-cyclodextrin (β-CD) in the main-chain, was synthesized and characterized. The chemical structure of the polyrotaxane was confirmed by NMR and FT-IR spectroscopies and its optical properties were investigated by UV-visible absorption and photoluminescence analyses. An optical gap of 2.70 eV was estimated from the absorption-onset of the polyrotaxane thin film. Blue photoluminescence was observed in dilute solution and in solid state. The emission spectrum of the polyrotaxane is blue-shifted compared with non-encapsulated polymer (**PBPAAn**). The HOMO and LUMO levels were estimated using cyclic voltammetry analysis. The thermal properties of the polyrotaxane were investigated by differential scanning calorimetry and thermogravimetry analysis; the results show a fully amorphous morphology and a thermal stability up to 350 °C.



Structure of the polyrotaxane (PBPAAn/β-CD)

Keywords: Polyrotaxane; β-Cyclodextin; Semi-conducting polymers; Photoluminescence; Thin solid film.

Synthesis and characterization of copper/ poly (5- aldehyde-2,2',5',2"-terthiophjene) composite material and its application for electrocatalysis

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Electrochemical modification of electrodes surfaces with electrodeposition of conducting polymers plays an important role in electroanalysis and electrocatalysis [1-3]. Among of these conducting polymers; oligothiophenes which are the most studied and synthetized, in raison of their chemical stability, and electrochemical properties.

In this work copper/poly (5-aldehyde-2,2',5',2"-terthiophene (3TCHO)) composite material (Cu/3TCHO) have been prepared electrochemically. The poly(3TCHO) films were electropolymerized by cyclic voltammetry from deaerated solution of CH_3CN which contained (10^{-2}M) of monomer (3TCHO) and 0.1 M TBAP as supporting electrolyte, by cycling the potential between 0V and 1.5V (versus SCE) at 50 mVs−1. Then the formed film was conditioned in metallic solution of copper.

The resulting materials were characterized by cyclic voltammetry, X-ray Photoelectron Spectroscopy (XPS), and Scanning Electron Microscopy (SEM).

The electrochemical analysis and XPS measurements show that copper exists in the final products. A SEM image shows the copper particles dispersion which are spherical in nature and seem to be microsized. Finally, the composite material was tested for the electrocatalysis of ascorbic acid and it's exhibited a best signal compared with the simple film of poly(3TCHO). This work provides a simple route for the synthesis of Cu/polyoligothiophenes microcomposites materials.

Keywords: Composite materials; Electropolymerisation; 5-aldehyde-2,2',5',2"-terthiophene; Ascorbic acid.

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Crystal structure, thermal analyze and vibrational spectroscopy of the new compound $[(C_4H_9)_4N]_3Bi_2Cl_9$

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The new organic-inorganic compound tri-tetrabutylammonium nonachlorodibismuthate(III) , $[(C_4H_9)_4N]_3Bi_2Cl_9$, crystallizes at room temperature in the monoclinic system $P2_1/n$ space group with the following lattice parameters a = 11.317(5) Å, b = 22.303(1) Å, c = 28.529(1) Å, α = 90.00 (3)°,β = 96.52 (2) ° and γ = 90.00 (3)°. The crystal packing can be described by stacking of organic-inorganic layers in the [010] direction. Each layer is formed by one type of isolate $[Bi_2Cl_9]^{3-}$ anions and three types of $[(C_4H_9)_4N]^+$ cations. The differential scanning calorimetry (DSC), disclosed four reversible phase transitions at 214, 238, 434 and 477 K. This compound has been studied by Raman scattering on single crystals, in the frequency range 10 - 400 cm^{-1} for temperatures ranging between 183 and 453 K. The temperature-dependant Raman spectrum shows the wave-number variation of several vibrational modes at the transition temperature detected by DSC.

Keywords: Tri-tetrabutylammonium nonachlorodibismuthate(III); X-rays diffraction; DSC ; vibrational spectroscopy.

Table: Crystal data and structure refinement for $[(C_4H_9)_4N]_3Bi_2Cl_9$ crystal.

Formula	$[(C_4H_9)_4N]_3Bi_2Cl_9$
Molecular weight (g. mol^{-1})	1452.94
Color/shape	colorless / prismatic
Space group	P $2_1/n$
Crystal system	Monoclinic
Temperature (K)	293(2)
Unit cell dimensions	
a (Å)	11.3174(5)
b (Å)	22.3031(1)
c (Å)	28.5295(1)
α (°)	90.000(3)
β (°)	96.516(2)
γ (°)	90.000(3)
Volume ($Å^3$)	7154.71(6)
Z	4
Absorption coefficient (mm^{-1})	5.270
$\rho_{calc.}$ (g/cm^3)	1.204
Radiation type, λ (Å)	MoK_α, 0.71073
Monochromateur	Graphite
Crystal size (mm^3)	0.32 x 0.28 x 0.25
θ range (°)	$1.44 \leq \theta \leq 29.99$
Range of h. k. l	-8→ 13, -25→ 25, -33→ 35
Independent reflections	10608
Observed reflections (I>2σ(I))	5094
R_{int}	0.0465
Refinement on	F^2
Goodness of fit	1.504
R / wR	0.0462/ 0.1571
Δρ (max)/Δρ (min) (e $Å^{-3}$)	-0.900/ 1.228

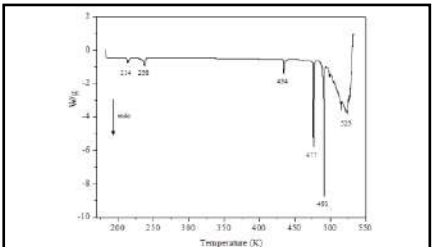

Figure: Differential scanning calorimetry curve of $[(C_4H_9)_4N]_3Bi_2Cl_9$ compound.

Characterization of a new composite bone implant : polyhydroxybutyrate -co -3- hydroxyvalerate - phosphate ceramic (CPC- PHBV)

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Currently biopolymers are required for many biomedical and pharmaceutical clinical applications, including, in particular the implementation of artificial prostheses organs including bone implants.

In this work, we present a new biomaterial based on a biodegradable polymer (PHBV) and a calcium phosphate ceramic (CPC) and its application in an animal experimentation.

The principle of synthesis of this compound is based on the interpenetration of calcium phosphate ceramic crystals in the polymer chains of PHBV.

Before clinical application, physicochemical analyzes should be performed and the properties of the biomaterial should be identified. In this work, we adopted the granulometric and morphological analyses by SEM (JEOL JSM 6400)

In-bone bio-ceramics implants were performed in 7 rabbits where a 1cm bone defect was created at the tibial shaft.

Histological studies were performed to evaluate the functionality of the elaborated biomaterial.

Composition and morphological structure of the biomaterial (CPC- PHBV) carry the main properties required of substitutes for bone repair. In particular, these properties are the bioactivity, biocompatibility and resorbability.

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MORPHOLOGICAL, RHEOLOGICAL AND MECHANICAL STUDIES OF BIO-SOURCED POLYMER BLENDS BASED ON POLYLACTIDE-ACIDE AND POLYAMIDE 11

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The focus of this paper is to gain a true understanding of the effect of the composition and compatibilization on the properties of the of entirely biosourced biopolymer blends based on PLA and PA11.

PLA cannot challenge usual commodity polymers regarding to the weak thermo-mechanical properties of this polyester wish reduces its use to very limited applications. Moreover the enhancement of the rheological properties presents several challenges mainly due to the poor shear and elongation properties of this biopolymer. Through this work, we present one promising routes to enhance processing ability of PLA for blown extrusion as well it's blend with various composition of PA11 and multi-functionalized epoxyde to improve both barrier and melt strength properties, respectively. Hence, various amounts of a chain extender, named Joncryl ADR®-4368, containing reactive epoxy functions have been incorporated into PLA/PA11 blends through mixing in a laboratory-scale twin-screw extruder. Their effects on the thermal, rheological, morphological and mechanical properties of PLA/PA11 blends were investigated systematically. Nodular and co-continuous morphologies were observed depending on the composition. The results showed an important role of Joncryl in the improvement of viscosity and storage modulus at low angular frequencies. The relaxations of matrix, dispersed and shape have been detected using a relaxation spectrum and Cole-Cole diagram. Mechanical properties showed that the Young modulus of the blends roughly obeys an additive mixture law. Moreover, the elongation at break of the PLA was greatly improved with the addition of PA11. The role of Joncryl as a compatibilizer has been demonstrated. The co-existence of chain extension/branching chains coupled to the PLA-copo-PA11 formation have to be taken into-account to explain the improved mechanical properties.

Synthesis, crystal structure, thermal analyze and Raman spectroscopy of the new compound $[(C_3H_7)_4N]SbCl_4$

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The new organic inorganic compound, tetrapropylammonium tetrachloroantimonate, $[N(C_3H_7)_4][SbCl_4]$, crystallizes at room temperature in the monoclinic system ($P2_1/c$ space group) with the following unit cell parameters: a=18.1973(5) Å, b=15.7225(4) Å, c=13.6491(3) Å, β=91.65(1)° and Z = 4. The atomic arrangement can be described by a stacking of organic-inorganic layers along a direction. Differential scanning calorimetry studies indicate a presence of two order-disorder phase transitions located at 343K and 363K.
Besides, an analysis of the Raman spectra of a monocrystalline sample, $[(C_3H_7)_4N]SbCl_4$ have been recorded at different temperatures within the interval 300–400 K in order to identify the structural changes in crystalline state and to understand the molecular alignment. The thermal evolution of Raman spectra reveals an order-disorder phase transition at about 343K and 363K.

Keywords: tetrapropylammonium tetrachloroantimonate; X-rays diffraction; phase transition; vibrational spectroscopy.

Table: Summary with data collection and structure refinement.

Compound	$[(C_3H_7)_4N]SbCl_4$
Color	Colorless/ parallelepiped
System	Monoclinic
Space group	$P2_1/c$
Temperature (K)	293(2)
Cell dimensions	
a (Å)	18.1973(5)
b (Å)	15.7225(4)
c (Å)	13.6491(3)
β (°)	91.650(1)°
Cell volume (Å³)	3903.48
Z	4
D_{calc} (gcm^{-3})	0.879
Diffractometer/scan	Bruker AXS CCD
Radiation, *λ*(Å)	Mo *Kα*, 0.71073
Crystal dimensions (mm)	0.38 ×0.28×0.16
μ_{calc} (mm^{-1})	1.945
R (int)	0.042
Unique reflections	12298
θ range (deg)	1.7-31.16
Reflections with $I > 2\sigma(I)$	6992
Range of *h*, *k*, *l*	-24/26, -22/21, -17/19
F(000)	1619
R/Rw	0.055 / 0.15
GOOF	1.055
$\Delta\rho_{min}/\Delta\rho_{max}$ (é Å^{-3})	-0.75/1.58

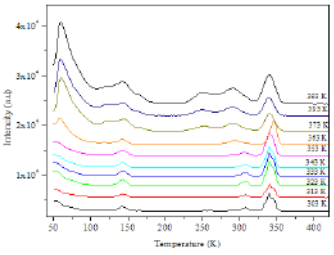

Figure: Temperature evolution of the vibrations modes for the $[(C_3H_7)_4N]SbCl_4$ crystals in the range (50-450 cm^{-1}).

NOUVEAUX COMPLEXES DE COBALT(III) AVEC DES LIGANDS AMIDES. SYNTHESE ET CARACTERISATION

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Les ligands amidiques jouent un rôle fondamental dans les processus biologiques [1], présentant en coordination dans les complexes, des propriétés spécifiques en catalyse [2], et comme agents thérapeutiques divers [3].

L'objectif de notre présent travail est de synthétiser des modèles de complexes de Cobalt(III) afin de pouvoir accéder à des catalyseurs.

Les nouveaux complexes du Cobalt (III), dérivés de ligands amides ont été isolés à l'état solides et caractérisés par analyse élémentaire (C, H, N, M, solvant), analyses spectroscopiques (IR, UV- visible) et par la mesure de la conductance molaire.

Tous les complexes possèdent des points de fusion élevés, ils sont stables à l'air libre insolubles dans l'eau, dans la plupart des solvants organiques mais solubles dans le dimétylsulfoxide.

L'exploitation des résultats nous a permis de déterminer les structures de ces complexes qui sont de nature dimère dans laquelle chacun des deux ligands penta dentés de type (N_3O_4) est coordiné par deux atomes donneurs au premier centre métallique et par trois au deuxième centre.

Mots clés : synthèse, caractérisation, complexe, ligand amide, catalyse.

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Anticorrosive and dielectric properties of polypyrrole/ Na^+ –montmorillonite (PPy/Na^+ –MMT) clay nanocomposites

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Frequency dependent ac conductivity (σac), dielectric constant (ε') and loss factor (ε") have been measured in the frequency range 100–10^4Hz in polypyrrole/Na^+ –montmorillonite (PPy/Na^+–MMT) clay nanocomposites. The nanocomposites were synthesized by in situ polymerization of pyrrole with varying the molar ratio of oxidant (peroxydisulfate) to pyrrole from 0.25 to 4 in an aqueous medium in order to study the influence of this parameter to the dielectric properties of polypyrrole/Na-montmorillonite nanocomposites.
All three measured quantities; σac, ε' and ε" depends with the variation of the molar ratio of oxidant (peroxydisulfate) to pyrrole in the composites at all frequencies.
In other hand polypyrrole clay nanocomposites in the form of coatings were found much superior in corrosion protection over those of conventional polypyrrole and clay only, based on the series of electrochemical measurement of corrosion potential, polarization resistance and corrosion current in 3.5% aqueous NaCl electrolyte.

Keywords: nanocomposites, polypyrrole, montmorillonite, polymerization, dielectric parameters, anticorrosive properties.

ADSORPTION OF HEAVY METALS ONTO OXIDISED AND CROSS-LINKED CORN STARCH

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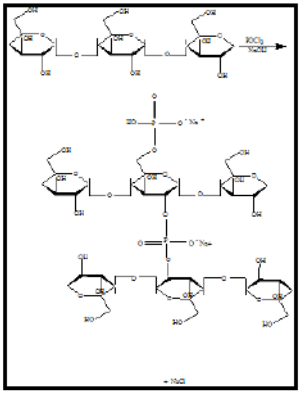
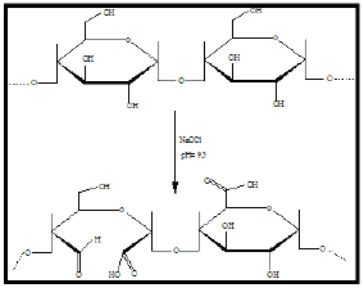
The chemical contamination of water from a wide range of toxic derivatives in particular heavy metals is a serious environmental problem owing to their potential human toxicity. Therefore, there is need to develop technologies that can remove toxic polluants found in waste water. In this work, the effect of some common chemical modifications such as oxidation with sodium hypochlorite NaOCl and cross-linking with phosphorous oxychloride $POCl_3$ on the physicochemical, morphological, thermal and rheological properties of corn starch have been investigated and characterized with (FT-IR) spectroscopy, optical microscopy, X-ray diffraction (XRD), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The adsorption properties of native and chemically modified starch for Cu^{2+}, Hg^{+}, Pb^{2+}, Cd^{2+}, Fe^{3+}, Ni^{2+} and Co^{2+} in their aqueous salt solutions were investigated under time condition. Regardless of the metal type, the adsorption capacity of both ionic starches reached a state of equilibrium within 24 h for starch dispersion in metals solutions.



Scheme1: Crosslinked starch with $POCl_3$



Sheme2: Oxidised starch with NaOCl

Keywords : Corn starch, Oxidation, Cross-linking, Heavy metals.

NEW ANTHRACENE-BASED II-CONJUGATED POLYMER: SYNTHESIS AND OPTICAL PROPRIETIES

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Organic π-conjugated materials with semi-conducting properties have attracted immense interest from both the scientific community and industrialists, because of their potential application as active layer in electronic devices.[1] The first organic light-emitting diode (OLED) was fabricated with anthracene crystals in 1965 but failed to attract attention because of poor performance.[2] Good quantum yields had thus been obtained for an emission in blue, but under high tensions (higher than 100 V), which led to very weak energetic efficiencies. In this communication, we report the synthesis and characterization of new soluble anthracene-based π-conjugated polymer (Figure1) for organic thin-layer electronic applications.

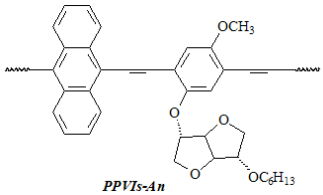

Figure1: Polymer structure

The polymer **PPVIs-An** has been synthesized via Wittig polycondensation using 9,10-bis(triphenylphosphoniomethyl)anthracene dichloride and new isosorbide-derived aromatic dialdehyde. The chemical structure of the polymer was defined by ^{1}H-NMR, ^{13}C-NMR, and FT-IR spectroscopic analysis. **PPVIs-An** containing isosorbide in the side chain linked with flexible hexyl chain; it is fully soluble in common organic solvents and has a number-average molecular weight of 10300 g mol^{-1}. Differential scanning calorimetry indicates that **PPVIs-An** is amorphous and displays a glass transition temperature of 50°C. The thin-film optical gap was about 2.56 eV make it possible to classify this polymer among the semiconducting materials. A high photoluminescence quantum efficiency of about 72% was determined in dilute solution. The polymer emits in yellow region in solid state. The HOMO and LUMO energy levels were determined by cyclic voltammetry and showed a p-type semi-conducting behavior. A single-layer device of the configuration [indium-tin oxide/**PPVIs-An** /aluminium] has been fabricated and has a relatively low turn-on voltage.

Keywords: π-conjugated polymer; anthracene; photoluminescence; thin film; isosorbide side-chain effect.

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