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The 2nd NATIONAL CONFERENCE WITH
INTERNATIONAL PARTICIPATION

NANOSTRUCTURED MULTIFUNCTIONAL MATERIALS

BOOK OF ABSTRACTS

NMM 2010

November 4 - **5, 2010, Iași, ROMANIA**

Organized by the

Al. I. Cuza University of Iași, Faculty of Chemistry
Department of Chemistry, Laboratory of Materials Chemistry,
11 Bd. Carol I, 700506 Iași, ROMANIA

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NANOSTRUCTURED MULTIFUNCTIONAL MATERIALS

NMM – 2010

November 4 - 5, 2010, Iași, ROMANIA

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FOREWORD

On behalf of the Organizing Committee I would cordially like to welcome you to the National Conference with international participation entitled "*Nanostructured Multifunctional Materials 2010*", organized in the Cultural Capital of Moldavia, Iași. This is the second *Nanostructured Multifunctional Materials Conference*, since our inaugural event was in Sinaia in 2007.

We are particularly fortunate to have here some of the top scientists from Belgium, Italy, Sweden, Australia, Greece, Russian Federation and Republic of Moldova. And of course, together with our prestigious invited specialists, we have a lot of PhD students, young researchers and Roumanian specialists which have attending the Conference.

The NMM-2010 Conference hosted by the "A.I.Cuza" University of Iași - Romania, organized by the Faculty of Chemistry, Laboratory of Materials Chemistry, is a scientific event dedicated to the 150-th Anniversary of our University. The organization of the 2nd NMM-2010 Conference was possible thanks to the kind support of National Authority for Scientific Research (ANCS) and "A.I.Cuza" University of Iași by POSDRU nr. 89/15/S/49944 2010.

Based on their tunable structural and physico-chemical properties, nanostructured multifunctional materials have high potential for improving sustainable development across a wide range of industrial branches.

The Conference is aimed at promoting the fundamental and applied studies of nanostructured multifunctional materials. The scientific programme is set up around the following topics: nanostructured porous materials; natural and synthetic zeolites; mesostructured materials-synthesis and modification; cationic and anionic clays; environmental applications; novel inorganic and organic-inorganic hybrid nanostructured materials; nanobiomaterials and other aspects of nanomaterials science and technology.

Many of the key topics impacting on nanostructured materials will be lectured by our colleagues over the next two days and we are sure that all of you will be involved in many discussions to make this event more productive and having in mind that the nanostructured multifunctional materials are a key for "high tech" strategic materials.

The Conference will provide the opportunity for developing new scientific collaboration and represents an important meeting for those who are interested to share and disseminate their experience, knowledge and innovative ideas on nanostructured materials.

We are gratefull to the invited speakers for their effort to make our Conference more attractive, as well to the distinguished scientists, which accepted to chair the various sessions of NMM-2010.

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I would like to express my thanks to all young researchers for your oral and poster contributions.

Finally, on behalf of the Organising Committee, I wish you a fruitful scientific Conference and a pleasant stay in Iasi, Romania. The feedback from YOU is very important for planning the next conference in 2013, to which You are cordially invited.

Co-chairperson

Prof. univ.dr. Eveline POPOVICI



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CONFERENCE BRIEF SCHEDULE

Thursday, November 4 th		Friday, November 5 th
8 ⁰⁰ – 9 ⁰⁰ REGISTRATION		9 ⁰⁰ – 10 ⁵⁰ IL-5, IL-6, OP-8, OP-9
9 ⁰⁰ - 9 ¹⁵ OPENING CEREMONY		10 ⁵⁰ - 11 ²⁰ Coffee Break and Posters Exhibition Session 3
9 ¹⁵ - 11 ⁰⁵ IL-1, IL-2, OP-1, OP-2		11 ²⁰ - 13 ⁵⁰ IL-7, IL-8, OP-10, OP-11, OP-12, OP-13
11 ⁰⁵ - 11 ³⁵ Coffee Break and Posters Exhibition Session 1		13 ⁵⁰ - 15 ²⁰ LUNCH
11 ³⁵ - 13 ²⁵ IL-3, IL-4, OP-3, OP-4		15 ²⁰ - 17 ³⁰ OP-14, OP-15, IL-9, IL-10, OP-16
13 ²⁵ - 14 ⁵⁵ LUNCH		17 ³⁰ - 18 ⁰⁰ Coffee Break and Posters Exhibition Session 4
14 ⁵⁵ - 15 ⁵⁵ OP-5, OP-6, OP-7		18 ⁰⁰ CLOSING CEREMONY
15 ⁵⁵ - 16 ²⁵	Coffee Break and Posters Exhibition Session 2 Round Table Discussions	
20 ⁰⁰ Festive Dinner		

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ROUND TABLE

„EDUCATION AND PROFESIONAL FORMATION TO SUPPORT ECONOMIC GROWTH AND DEVELOPMENT BASED ON KNOWLEDGE”

1. **Prof. em. dr. ir. Etienne Vansant** – University of Antwerpen, Belgium, Doctor Honoris Causa of Al.I.Cuza University of Iasi. *The Importance of FP-7 Projects in Professional Growth of Young Researchers*
2. **Prof. dr. Anita Lloyd Spetz** - Linköping University, Dept of Physics, Chemistry and Biology, Sweden, *New Dimension of the Doctoral School Activity.*
3. **Prof.dr. Alfonso Nastro** - Calabria University, Italy, *Education and Student Research – Part of a Society Based on Knowledge*
4. **Lector dr. Mirela Petruta Suche**a - School of Applied Technology, Technological Educational Institute of Crete, Greece. *Ethics of Scientific Research.*

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CONFERENCE TOPICS

- 1. NANOSTRUCTURED POROUS MATERIALS**
- 2. NATURAL AND SYNTHETIC ZEOLITES**
- 3. MESOSTRUCTURED MATERIALS: SYNTHESIS AND MODIFICATION**
- 4. CATIONIC AND ANIONIC CLAYS**
- 5. ENVIRONMENTAL APPLICATIONS**
- 6. NOVEL INORGANIC AND ORGANIC-INORGANIC HYBRID
NANOSTRUCTURED MATERIALS**
- 7. NANOBIOMATERIALS**
- 8. OTHER ASPECTS OF NANOMATERIALS SCIENCE AND
TECHNOLOGY**

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INVITED LECTURES

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IL-1

NANOPOROUS MATERIALS: CHALLENGES FOR THE FUTURE

E.F. Vansant

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Zeolites are the best known nanoporous materials with a wide variety of applications in technology, industry and medicine. However, due to their microporous nature ($\varnothing < 2$ nm) large molecules and viscous liquids are excluded from their porous network. Therefore, mesoporous materials with large pore dimensions ($2 < \varnothing < 50$ nm) are gaining increasing interests due to their applicability in processes like in fine chemistry, biotechnology, biology, pharmacy, controlled drug release etc. They create accessibility for large molecules and the ability to allow and control high mass transfers. Nevertheless, they are inferior to zeolites with regards to stability, selectivity and activity. A wide range of modified materials were developed or modified in order to improve these properties.

In general, mesoporous materials are made by a synthetic route in which organic template molecules are used to direct the structural properties. However, also template free synthesis routes exist. By changes in the synthesis conditions or post-synthesis modifications, the structural characteristics can be altered. Therefore, knowledge on the synthesis mechanisms and influence of the synthesis parameters and utilized templates, can allow pore size engineering, control of the morphology and porous properties. This way, mesoporous and combined micro- and mesoporous silica and non-silica materials can be formed as well as materials that contain both silica and transition metal oxides.

Controlling the synthesis of the mesoporous materials and detailed knowledge on modification methods will allow the formation of designed materials for specific applications.

This way various materials can be made and modified:

a) *Mesoporous materials with internal nanoparticles*

Nanoparticles (zeolite precursors, silica, transition metal oxides) can be introduced into mesoporous materials by post-synthesis impregnation methods or in situ. The obtained materials have both open and narrowed sections in the mesopores which gives them unique properties (diffusion, stability, ...) that can be adjusted to the application (encapsulation, separation, controlled release, adsorption, catalysis etc.).

b) *Combined micro- and mesoporous materials*

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Combined micro- and mesoporous materials are claimed to have advantages towards diffusion, better hydrothermal stability, multifunctionality to process a wide variety of feeds, capabilities to encapsulate waste, controlled release etc. These materials can be made in a direct way by using specific triblock copolymers. On the other hand, new materials are developed in which zeolite precursor species are applied for the formation of these mesoporous materials. As a result, mesoporous materials containing these zeolitic precursor particles are obtained.

c) *Hybrid organic-inorganic materials*

The selectivity and stability of mesoporous materials can be changed by modification of the carrier materials with organic functional groups on the surface (grafting) or within the structural framework (co-condensation, periodic mesoporous organosilica's (PMO)). These materials find application in chromatography, catalysis (heterogenised homogeneous catalysts), controlled release, metal scavenging, solid state synthesis, etc. and can be adjusted to the environment in which they are utilized (hydrophobic, hydrophilic, ...)

Furthermore, considerable research was made on other hybrid materials, such as the metal-organic-frameworks (MOF), covalent-organic-frameworks (COF) and zeolitic-imidazolate-frameworks (ZIF).

Nanoporous materials play an important role in chemical processing in which they can be successfully used as adsorbents, catalysts, catalyst supports and membranes and form the basis of new technologies, mainly due to their unique structural or surface properties, which can be tailored to a certain extent to meet the process needs.

The synthesis and tuning the properties of nanoporous materials remain largely unexplored and constitute one of the main objectives in the future. Indeed, the understanding of the synthesis process is essential for developing novel active porous materials with well-defined properties and for enabling cost-, energy- and environmentally efficient processes. A challenge will be the development of synthesis routes to form predictive structures obtained from theoretical models. Furthermore, a self-restructuring or self-healing opportunity, taking place when the porous structure is damaged, becomes very important to increase the life-time of the porous materials.

Characterizations of the synthesized materials is crucial for the quantification and prediction of their physical, chemical and mechanical properties. Therefore, more detailed ex-situ and in-situ techniques have to be developed.

For the adsorption / separation applications, the main challenges are

- a) the development of high capacity CO₂ and CO-selective adsorbents, that can also operate in the presence of hydrogen and steam at elevated temperatures
- b) porous materials for the separation of paraffin / olefin mixtures

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- c) new adsorbents for the removal of radioactive nuclides
- d) porous materials with a combined removal and destruction of VOC's
- e) new porous materials for the storage of H₂, CH₄, natural gas, etc.
- f) sorbents for controlled drug delivery

Future challenges in the field of membranes are

- a) to improve the flux in combination of a longer-term stability
- b) H₂ removal / isolation from atmospheres containing steam at high temperatures
- c) removal of CO₂ from exhaust gases or natural gases
- d) development of catalytic membrane reactors

Catalysis is an indispensable tool, whenever chemical substances are to be converted into more valuable or environmentally more benign products.

The trends in the future in the field of catalysis (heterogeneous catalysis) are:

- a) catalysts for cleaner fuels from more-difficult-to-handle and lower-quality fossil raw materials
- b) catalysts for catalytic transformations starting from cheaper materials (dehydroalkylations of aromatics with alkanes)
- c) pushing catalysis beyond traditional limits: Process intensification
- d) unraveling mechanisms of heterogeneously catalyzed reactions by in-situ characterization techniques

The presentation aims to give a short summary of the most important challenges for the future in areas of synthesis, adsorption, membranes and catalysis using nanoporous materials.

IL-2

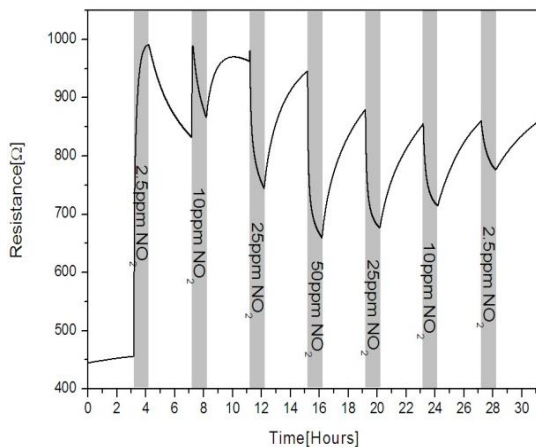
NANOSTRUCTURED & POROUS MATERIALS FOR FET SENSORS

Anita Lloyd Spetz, Ruth Pearce, Mike Andersson, Robert Bjorklund, Rositza Yakimova - Linköping University, Dept of Physics, Chemistry and Biology, SE 581 83 LINKÖPING, Sweden, e-mail: spetz@ifm.liu.se
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Chemical sensing has developed considerably from the remarkable development in nanoscience experienced in the research community all over the world. We have developed gas and biosensors based on wide band gap materials as transducers [1, 2]. Field effect devices based on silicon carbide, SiC, have been developed as chemical sensors with porous sensing materials for about 15 years with applications at high temperature and in harsh environment like exhaust and flue gases. Tailor made nanostructured materials have been developed e.g. for a selective oxygen sensor based on a SiC FET device. Zinc oxide nanoparticles as sensing material showed an interesting change in response direction for low and high concentration of e.g. NO₂ gas [3]. It is suggested that the conductivity of the material actually goes through an n- to p-type switch.

A similar phenomenon was experience for a sensing layer based on a monolayer graphene epitaxially grown on SiC [4]. Exposure to increasing concentration of NO₂ introduced a change in the direction of the response, see Figure 1. Also, the sensors showed very high sensitivity to NO₂ for monolayer graphene with a potential response to ppb levels of NO₂, which was a hundred fold increase of the sensitivity as compared to multilayer graphene.



The progress of nanomaterials and the use of e.g. nanoparticles also have given topical interest to investigate their toxicity. We have developed a soot sensor for diesel exhausts employing thermophoresis [5]. It is a simple finger electrode on a substrate and the resistivity of the soot is measured. By smart packaging the sensor surface is kept colder than the surrounding exhaust gases, which improves the sensitivity of the sensor and enables the on board diagnostics, OBD, application, see Figure 2.

Fig. 1. Response of epitaxial graphene/SiC to NO₂

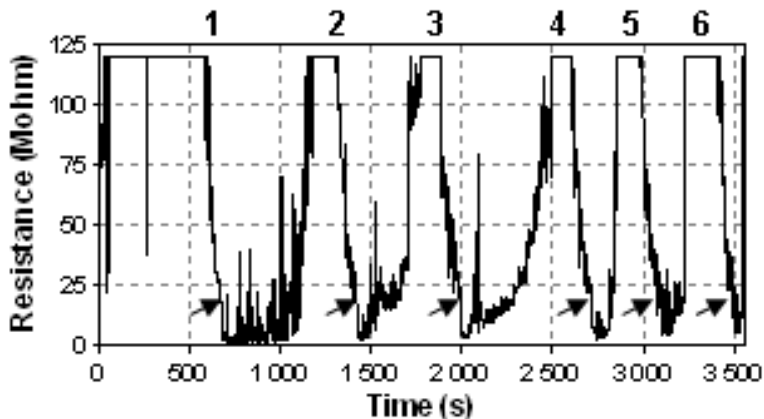


Fig. 2. Cyclic measurements of soot deposition and regeneration on resistive sensor

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ACKNOWLEDGEMENTS

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IL-3

PARTICULARITIES OF IN VITRO AND IN VIVO EVALUATION OF DRUG TOXICITY

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Abstract: Today, most developed countries have enacted laws and regulations to control the marketing of drugs and other substances of potential toxicological concern. Such regulations often prescribe a specific regimen of toxicity testing to determine whether the benefits of a particular substance outweigh its risks to human health and/or the environment. **Aim:** Theoretical meta-analysis regarding the methods used for new substances toxicity evaluation. A toxicity test, by extension, is designed to generate data concerning the adverse effects of a substance on human or animal health, or the environment. Many toxicity tests examine specific types of adverse effects. Other tests are more general in nature, ranging from single-exposure studies (acute) to multiple-exposure studies, in which animals are administered daily doses of a test substance, and determine whether one or more organ or system is adversely affected following subacute, subchronic or chronic exposures. Acute toxicity tests are generally the first tests conducted. They provide data on the relative toxicity likely to arise from a single or brief exposure. In addition to the LD50 (the dose lethal to half the animals), the results of the acute studies are used to set doses for longer-term, subchronic experiments. Subchronic toxicity tests are employed to determine toxicity likely to arise from repeated exposures of several weeks to several months. The purpose of the subchronic study is to determine target-organ toxicity, to determine the effects of prolonged dosing, and to help establish margins of safety for food additives and drugs. Chronic toxicity studies provide a thorough examination of the dose effect of a given chemical on homeostasis, bodily function, induced diseases, and the effect on lifespan. Chronic toxicity studies provide the bulk of the preclinical information used for assessing safety and risk. The alternative toxicity testing is used to describe any change from present procedures that will result in the replacement of animals. These cell and tissue-based methods have already achieved international acceptance as full or partial replacement methods for their animal-based counterparts. The advantages of in vitro tests are that they are quick, relatively inexpensive, and specific mechanisms of action can be tested. The disadvantage of these tests is that the homeostatic mechanisms and pathways found in animals are not present.

Keywords: toxicity, *in vivo*, *in vitro* evaluation.

IL-4

ELECTRON PARAMAGNETIC RESONANCE IN MESOPOROUS CARBON NANOTUBES

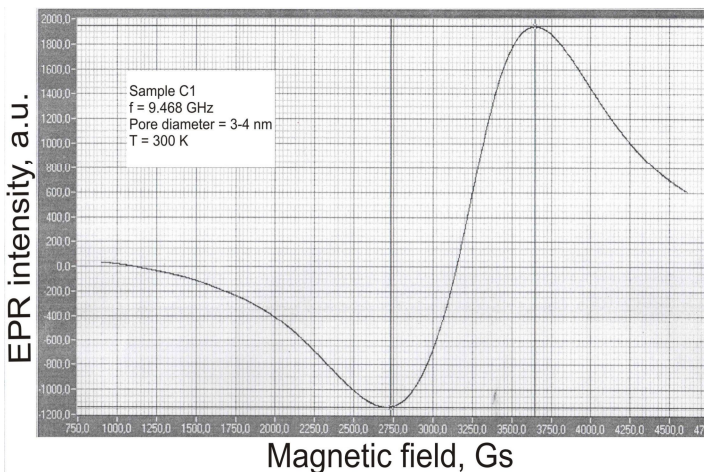
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The EPR spectrum of the mesoporous carbon nanotubes (CNTs) with pores diameter 3-4 nm was investigated. The CNT samples were prepared by using SBA-15 as template and glycerol as the carbon source. SBA-15 was synthesized using a tri-block copolymer (Pluronic P123). The structure of the CNTs was studied using low angle XRD. The carbon pattern shows three well resolved peaks indexed (100), (110) and (200) characteristic to the two-dimensional hexagonal space group $p6mm$. The calculated cell parameter of the CNT sample C1 is $a_0 = 9.8$ nm and the d -spacing is of 8.5 nm.

The EPR spectrum of mesoporous CNTs consistS of a width asymmetrical line with g-factor $g = 2.13$ and $\Delta H_{pp} = 912$ Gs. The main contribution into EPR line width is caused by localized magnetic moments including oxygen molecules with the triplet ground state (experiments were carried out at ambient conditions).



A detailed study of the adsorption of an O_2 molecule on a CNT, using spin-polarized local density approximation (LDA) as well as spin-polarized local generalized gradient approximation (GGA) shown a weak potential well of dept ~ 0.1 eV at a distance of ~ 3.0 Å between O_2

molecule and CNT for the LDA approximation and a very shallow well of ~ 0.03 eV at a distance ~ 3.5 Å for the GGA approximation [1]. For the triplet state of O_2 adsorbed on CNTs, two degenerated $p\pi\pi^*$ bands are split into four bands, with the two up-occupied $p\pi\pi^*(\downarrow)$ bands rising ~ 0.35 eV above the top of the valence band at the Γ point [2]. The sensitive response of CNT to the adsorption of O_2 molecules is due to the pinning of the Fermi level near the top of the valence band.

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IL-5

SMART NANOCOMPOSITES FOR FUNCTIONAL APPLICATIONS

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Smart materials have the ability to perform both sensing and actuating functions. Compared to smart materials, smart composite materials have many technical issues such as uniform mixing, interfacial adhesion, and property characterization [1-3]. Some smart nanocomposites of Ni-clinoptilolite, Ni-MCM-41, Ni- Al_2O_3 , Ni- Cr_2O_3 , Ni- SiO_2 , Ni- $(\text{Cr}_2\text{O}_3+\text{Al}_2\text{O}_3)$ were prepared and analysed by using X-Ray diffraction, scanning electron microscopy, N_2 adsorption-desorption measurements and electron spin resonance spectroscopy. The obtained nanocomposites can be used in different functional applications. An important one is the methanation reaction.

The catalytic activity of nanocomposites was investigated as a function of temperature (Fig.1).

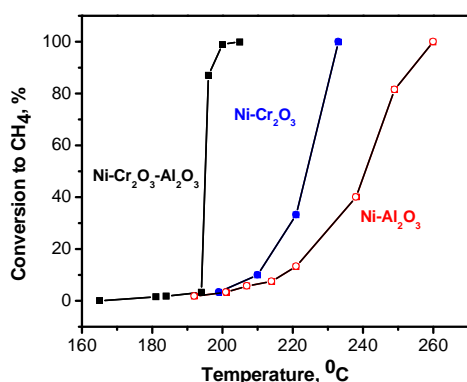


Fig.1. Conversion of CO and H₂ to CH₄ as a function of temperature, on different nanocomposites

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IL-6

TOWARD COMPLEX NANOARCHITECTURES: SELF-ASSEMBLED NANOSTRUCTURES BASED ON LDH ANIONIC CLAYS

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The ensembles of different nanostructures able to conjugate the characteristics of their components and generate materials with complex properties are largely studied because of their multifunctional capabilities. Layered double hydroxides (LDHs) are a class of anionic clays with a lamellar structure formed by positively charged brucite-like layers counterbalanced by exchangeable interlayer anions. The joining structure of LDHs layered matrix – nanoparticles of metal oxides ($\text{Me}_x\text{O}_y/\text{LDH}$) is able to combine the specific characteristics of nanoparticles of metal oxides and the surface/textural functionality of the clay into one single material. We present here a simple synthesis route in which the reconstruction process of the layered clay, the synthesis of the silver nanoparticles and their organization on the clay surface have taken place in one single step, at room temperature [1]. Results show that the evolved nanoarchitectures are composed of nanoparticles of Me_xO_y ($\text{Me} = \text{Fe}, \text{Ti}, \text{Zn}$) deposited on the larger particles of LDH anionic clay. The morphology, composition and phase structure of the as - prepared materials (studied by X-ray diffraction (XRD), IR spectroscopy (FTIR), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS) and N_2 adsorption) give rise to their multifunctional applications.

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IL-7

SOLID SORBENTS FOR CO₂ CAPTURE

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Excessive greenhouse gases (GHG) are considered the cause of world temperature increase and the consequent effects on climate change, as shown by the Intergovernmental Panel on Climate Change (IPCC). There is increased recognition by the world's scientific, industrial, and political communities that the concentration of GHG in the earth's atmosphere, particularly CO₂, is increasing: 280 ppm in the past 200 years (most of this in the past 50 years) and 380 ppm in the present (most of this after 1950). Anthropogenic source of GHG are considered responsible for this situation since human activities release $\approx 70\%$ of total emissions. The total amount of CO₂ emission into atmosphere is about 6.10^9 tons/year.

The capture and storage of CO₂ has been identified as one potential solution to GHG driven climate change. There are several methods to reduce the growth of CO₂ content in atmosphere:

1. to retard deforestation and to plant vegetation highly effective in CO₂ absorption;
2. to reduce the world production of thermal energy and to increase the alternative energy resources;
3. to extract CO₂ from the exhaust gases and to dispose it in ocean depths and in geological cavities;
4. to convert CO₂ chemically into useful products.

A wide variety of CO₂ removal technologies are available:

- reversible solvent absorption using amines: MEA, DEA, MDEA;
- cryogenic distillation;
- membrane gas separation;
- adsorption on solid adsorbents: oxides, zeolites, activated carbon, metal-organic frameworks (MOF's), organo-silicas and surface- modified silicas.

Adsorption separation is recognized as an efficient and economically attractive candidate to replace the solvent absorption with amines. The solid adsorbents for CO₂ separation are molecular sieve solid-supported amines obtained through grafting or impregnation. Intense research is currently focused on ordered mesoporous silicas (OMS), M41S, FSM-16, SBA-15, FDU, KIT, AMS functionalized with organic molecules containing amino-groups (diethylene-triamine, PEI, DEA, TEPA).

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IL-8

NANOSTRUCTURED METAL OXIDE SURFACES AS ACTIVE LAYERS FOR SMART WINDOWS AND SOLAR ENERGY DEVICES

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Technology requires today novel, advanced materials combining perfectly optimum functionality with low cost, reliability and preservation of the environment. Nanotechnology is an ideal approach towards this scope, since the size reduction of matter in the nano-scale can induce novel properties and functionalities. In parallel, the advances in materials science allow easy and excellent control of the materials properties, possibility leading to novel structures suitable for various applications. Metal oxide structures are a promising family of functional materials for photonic applications, since they are easily grown in various morphologies with various techniques, are semiconducting, having an energy gap in the UV/visible region of the light spectrum. Therefore, they are ideal candidates for processes involving light and charges, and the material engineering of the respective films and nanostructures is one of the most challenging research topics.

Considerable improvement of the physical properties and the functionality of metal oxide structures can be achieved via the optimization of their bulk and surface properties. Based on many years of research it was shown that the characteristics of metal oxide thin films and nanostructures, such as: thickness, grain size, porosity, grain faceting, agglomerations bulk conductivity, surface architecture, grain network, surface stoichiometry, catalytic reactivity, band gap and so on are playing a significant role with respect to a particular application. As a result, today there are various physical and chemical technological approaches, which can be used for the optimization of the above properties. Although physical growth techniques can be more reliable in the reproducibility of structure characteristics, the chemical ones seem to be more advantageous since they can be simpler and less expensive, allowing at the same time a very efficient control of the structure characteristics through the growth conditions.

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This contribution will focus on presentation of recent results related to the growth and characterization of metal oxide structure of different morphology, grown with different techniques, suitable for application such as: active and passive coatings in glass windows technology, active layer in energy production devices, photocatalytic layers for environmental applications, microelectronics and gas sensor applications. Emphasis is given on the functionality of these structures and its relation with their morphological characteristics.

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IL-9

ESR INVESTIGATION OF THE HYDROGEN ATOMS STABILIZATION IN GAMMA-IRRADIATED ZEOLITES CONTAINING VARIOUS ADSORBATES

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By means of electron spin resonance (ESR) spectroscopy the regularities of the hydrogen atom (H) low-temperature (77 K) stabilization in the nano- and microcavities of zeolites having contained a number of adsorbates (methanol, ethylene, acetylene, and ammonia) has been studied. The values of the H and the adsorbate radical radiation-chemical yields as well as stationary concentrations, and their ratio have been determined (table). It was concluded, that the necessary condition for the H atom stabilization created during low-temperature radiolysis of the hydrogen-containing adsorbate molecules is the presence of the H⁺ acid centers in zeolites. Possibly, hydrogen atoms may be stabilized on the surface of zeolite nano- and microcavities or in the traps located in the region of oxygen rings in the volume of the zeolite material.

Table Radiation-chemical yields, stationary concentrations and their ratio for hydrogen atoms and the adsorbate radicals formed during radiolysis of the methanol, ammonia, ethylene and acetylene molecules adsorbed on the HY zeolite

Sorbate	G(R), 1/100 eV	G(H), 1/100 eV	G(H)/G(R), arb. units	C(R), 10 ¹⁷ , 1/g	C(H), 10 ¹⁷ , 1/g	C(H)/C(R), arb. units
CH ₃ OH	0.65	0.10	0.155	8.0	1.6	0.2
NH ₃	0.5÷0.6	0.0014	0.0023	4.3÷6.0	0.05	0.0116
C ₂ H ₄	0.9	0.09	0.1	12.7	0.65	0.051
C ₂ H ₂	1.8	0.06	0.033	11.0	0.65	0.059

Note: the level of the surface filling was about 15 adsorbate molecules on the 10 "big" cavities in the HY zeolite

This investigation has been carried out with the help of Russian Federation Ministry of Science and Education (project No 02.740.11.0652).

IL-10

TITANOSILICATE ETS-10: SYNTHESIS, CHARACTERIZATION AND USES

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Zeolites are aluminosilicates with crystal structures forming channels and cavities in the nanometer range. These micropores show regular spatial arrangements and the pore dimensions are characteristic for each particular structure type.[1].

The microporous crystalline titanosilicates containing octahedral chains (nanowires) of TiO_6 (-O-Ti-O-Ti-O) connected to tetrahedral SiO_4 , the ETS-4 and ETS-10 structures (Engelhard Titanosilicate Structure no.), first synthesized by Kuznicki [2-4] in 1989, are the most important ones.

ETS-10 is the most interesting microporous titanosilicate because of the high thermal stability and of wide-pore structure (pore openings close to 0.8 nm).

The structure of ETS-10 consists of corner-sharing TiO_6^{2-} octahedral and SiO_4 tetrahedra linked through bridging oxygen atoms that construct the three-dimensional pore structure. The pore structure consists of 12-, 7-, 5- and 3-membered rings and has a three-dimensional wide-pore channel system whose minimum diameter defined by 12-membered ring is elliptical with $\sim 0.49 \times 0.76$ nm dimensions [5] (Fig. 1).

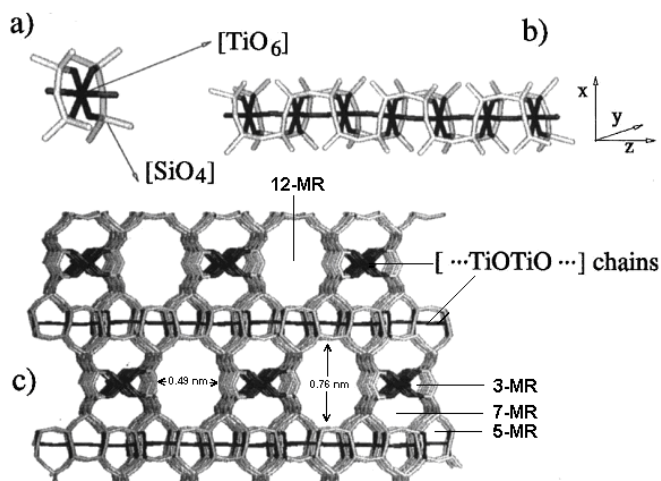


Fig. 1. The framework structure of ETS-10: (a) single element of the chain; (b) single chain; (c) three-dimensional view [5,6].

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Titanosilicate ETS-10 has attracted attention in adsorption, ion exchange, and shape selective catalysis for a number of reasons: (i) it has a three dimensional pore system that allows good diffusional access; (ii) it has a high cation-exchange capacity because exchangeable Na⁺ and K⁺ cations balance the two negative charges generated by the presence of [TiO₆]²⁻ octahedra; (iii) it possesses electronically isolated titanate (O-Ti-O-Ti) chains that may act as quantum wires with modified optoelectronic properties; and (iv) it contains a high concentration of defects as the result of the fusion of adjacent pores. These larger pores allow for easier access to the internal void space and furthermore provide active sites for catalysis[7,8]. Recently, ETS-10 became particularly interesting for photocatalytic applications. Examples are the oxidation of organic alcohols by ETS-10 within the pores of zeolitic supports;[9] the shape-selectivity effects in oxidative photodegradation of organic molecules (phenols) [10]; and the photocatalytic decomposition of acetaldehyde over ETS-10 and transition metals incorporated in ETS-10 [11]. Howe and Krisnandi concluded that the photocatalytic activity of ETS-10 is strongly influenced by stacking fault defects and ion exchange- induced external defects present in the structure[12,13].

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ORAL PRESENTATIONS

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OP-1

IRON CONTAINING ANIONIC CLAYS: AN XPS STUDIES OF THEIR SURFACE PROPERTIES

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Nanostructured assemblies of hydrotalcite-like clays supported with iron oxides have been obtained during the reconstruction process of the anionic clay layered structure by manipulating its structural “memory effect”. The surface characteristics of as-prepared nanoarchitectures were studied by X-ray photoelectron spectroscopy (XPS). The binding energy values of Fe 2p points out that Fe₂O₃ is present on the surface of Fe/LDH ensembles. For the parent matrix of iron substituted clay the curve fitting of the O1s profile indicates that the predominant signal contribution belongs to the energy band at 531.5 eV, assigned to the surface hydroxyl groups (OH⁻). The band situated at 529.58 eV is attributed to the lattice oxygen in the metal oxide while that situated at 532.5 eV corresponds to the oxygen from water. The increased contribution of the oxide component and a decrease in the hydroxyl – water components reveal the specific surface characteristics of the dual nanostructure of iron oxide on LDH clay.

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OP-2

ANODIC DETERMINATION OF PENTACHLOROPHENOL FROM WATER

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Pentachlorophenol (PCP) is a highly dangerous and persistent chemical widely that belongs to “priority pollutant list” and fast determination of its concentration is required. Carbon based electrodes are widely used in electroanalysis due to their low background current and wide potential window suitable for the investigation of an electrochemical oxidation process, chemical inertness, low-cost, and suitability for various sensing and detection application. The assessment of a carbon nanofiber-expanded graphite-epoxy composite (CNF-EG-Epoxy) electrode for the anodic determination of PCP is described. Cyclic voltammetry technique was used to characterize the electrochemical behavior of PCP at CNF-EG-Epoxy electrode in 0.1 M Na₂SO₄ supporting electrolytes in order to establish the parameters for amperometric/voltammetric determination of PCP. The linear dependence of the current versus PCP concentration was reached in the concentration range between 0.75 to 15 μM PCP using cyclic voltammetry (CV), differential-pulsed voltammetry (DPV), chronoamperometry (CA), and multiple-pulsed amperometry (MPA). Under the conditions of CA application, the fouling effect of the electrode occurred, which was avoided by MPA application that assures in-situ electrochemical cleaning of the electrode. The best electroanalytical parameters of the sensitivity, the relative standard deviation, the lowest limit of detection and the limit of quantification were obtained by MPA application, revealing that CNF-EG-Epoxy electrode exhibited useful characteristics for electrochemical determination of PCP from aqueous solution. In addition, some mechanistic aspects regarding PCP oxidation on CNF-EG-Epoxy electrode were discussed by performing CV at different scan rates.

OP-3

SYNTHESIS OF TI-CONTAINING COMBINED MICRO- AND MESOPOROUS SILICEOUS MATERIALS FOR THE CATALYTIC EPOXIDATION OF CYCLOHEXENE

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Different types of titanosilicates are synthesized, structurally characterized and subsequently catalytically tested in catalysis for the liquid-phase epoxidation of cyclohexene (figure 1). The performance of three types of combined zeolitic/mesoporous materials is compared with the one of widely studied Ti-grafted-MCM-41 molecular sieve and the TS-1 zeolite. The catalytic test results are correlated with the structural characteristics of the different catalysts. Moreover, immersion calorimetry with the same molecule as in the catalytic test reaction is for the first time applied as an extra means to interpret the catalytic results. The experiments show that the order of immersion enthalpy completely matches the order of catalytic activity for the different catalysts. It is demonstrated for the very first time that the combination of catalytic testing and immersion calorimetry while using the same molecule can lead to important insights in the influence of the material's characteristics on catalysis. Immersion calorimetry could therefore be used in combination with catalytic test reactions, as an auxiliary characterization tool of remarkable importance for a better understanding of the interaction between the solid catalyst and the reagent.

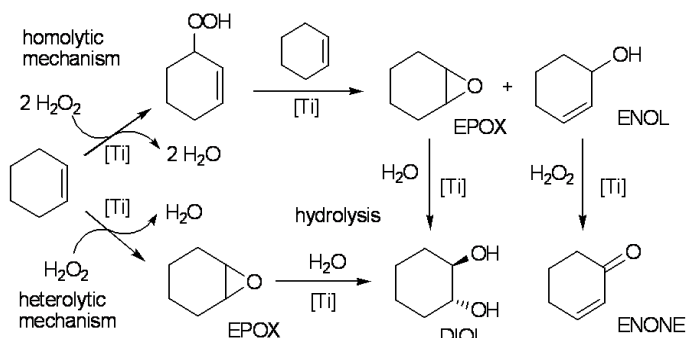


Figure 1: Schematic representation of the two different reaction pathways for the epoxidation of cyclohexene.

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OP-4

STUDY OF A NANOSTRUCTURED POROUS MATERIAL PATTERN OBTAINED THROUGH UV EXCIMER LASER IRRADIATION TREATMENT

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Wool processing technology is strongly dependent upon the monitoring of chemical and physical-structural properties of wool textile surfaces. The most important aim of the technologic processing of wool fibres is the increase of surface free energy hydrophilicity. Moreover, a high level of free movement of fibres in clothing is desirable to aid restoration of shape, after washing and to avoid shrinkage. Consequently, the above mentioned processes impose the modifying of the surface properties of wool for both subsequent textile applications and optimization of functionalisation/thermal-physiological comfort [1-6]. Modification of fibre surface chemistry at nanostructural level should affect particularly the lipid over layer which provides non-polarity and a small wettability. In textile industry, the method most widely used is an oxidation process, known as wet aqueous chlorination which involves treatment of wool fibre top or woven fabrics with an aqueous solution of sodium hypochlorite (NaOCl) or other oxidation agents. The process is aggressive and can cause some micro-destructions to the wool fibre but it does increase the wettability of the fibre surface and may also increase shrink resistance.

A series of modern irradiation treatments are applied at surface level of textiles by using of excimer UV laser, in order to increase the surface polarity and the wettability of the wool fabrics exposed to some technologic processes [7-10]. This kind of treatment does not induce both toxic effluents and waste water emissions with chemical or physical load which need denocivization. Moreover, the irradiation method has as result the loss of angularity at cuticle edges which leads to a more flexible and mobile network of fibres which allows relaxation back into shape before the irradiation.

Our present research is aiming the modification of some chemical and physical-structural properties of wool fibres surface exposed to excimer UV laser irradiation treatment, for enhance the functionalisation/thermal-physiological comfort of textile items, involving an experimental protocol between FTIR, XPS and reflexion colorimetry. The data obtained by means of these techniques have been correlated with the porosity of fibres, their

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wettability, the main characteristics used in the evaluation of the processability within some subsequent textile technologies suitable for some industrial purposes.

The second objective deriving from this study was to set-up a nanoscaled pattern of a reliable textile filter with other fibrous composition than wool fibres, providing properties as: photocatalytic action activated by UV-Vis light(i.e. sunlight); improved adsorption capacities; self-sterilizing and self-cleaning effects.

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OP-5

SOME APPLICATIONS OF POROUS MATERIALS IN GAS AND PARTICLE SENSING

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Various types of semiconductive oxides are used in gas sensing applications [1-3]. The sensing mechanism supposes the highlight of the changes occurring, for example, in the electrical resistance value of the solid layer while it works as a catalyst oxidizing and/or reducing the chemical species to be detected. One important aspect for a catalyst performance is the value of its specific surface area, since a big surface area assures a high number of molecules transformed per solid unit.

This presentation will display some examples of porous materials as: zeolites, mesoporous materials and layered anionic clays, well known as materials with high or relatively high ordered structure, which were tested and gave promising results as gas sensing species. Advantages and drawbacks can be found in this respect, since the tightness of the packing is a prime importance factor in the work function of solid sensitive layer and the thin layers are often preferred as regarded to thick layers. Tin, titanium, indium and zinc oxides and the mixtures thereof are the most investigated semiconductive species; obtaining ordered mesoporous and layered structures with these compositions is quite demanding.

Another important aspect is the possibility to mix semiconductive oxides with noble metals and beneficiate also from their catalytic potential and this is a quite easy task for the above mentioned classes of solids. Moreover, it is also possible to use these materials in the sensing of chemical inert species, as heavy hydrocarbons or carbon-rich nanoparticles (soot). The sensor is based on a layer of semiconductive oxide containing platinum deposited unevenly on a finger structure, which makes the adhesion of the soot better and helps to remove it easier in the regeneration step.

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OP-6

**ELECTROCHEMICAL MINERALIZATION OF DRIMAREN
BRILLIANT ROTH DYE ON BORON-DOPED DIAMOND
ELECTRODES**

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The electrochemical treatment of wastewaters containing dyes represents a promising treatment technique for the removal of pollutants that are resistant to biodegradation. This paper presents the results of the electrochemical decolourisation, aromatic ring degradation and mineralization of Drimaren Brilliant Roth (DBR) synthetic solutions using boron-doped diamond (BDD) electrodes. Electrolyses were carried out under galvanostatic conditions in 0.1 M Na₂SO₄ supporting electrolyte using undivided electrolytic cell. Prior to electrolysis application, the electrochemical behavior of BDD electrode was characterized in the presence of DBR dye by cyclic voltammetry in order to establish the working potential-current conditions. The influence of operating variables on the electrochemical process efficiency was studied as a function of the current density, the initial pH, the initial concentration of dye. The process performance was assessed in terms of decolourisation, aromatic ring degradation and mineralization efficiencies; electrochemical efficiencies; decolourization, aromatic ring degradation and mineralization rate; mineralization current efficiency and energy consumption. Under all applied conditions, the complete decolourisation of dye synthetic solution was achieved after several minutes of electrolysis (maximum 8 minutes). The total aromatic ring degradation occurred depending on current density, the pH and the dye concentration. The complete mineralization of dye synthetic solution was not achieved, and the maximum mineralization efficiency of 77.2 % occurred for initial DBR concentration of 50 mgL⁻¹, initial pH 5 and the current density of 10 mAcm⁻². The optimum current density parameter, which is one of most important for operating electrochemical processes, was established based on the best process efficiency achieved at low energy consumption.

OP-7

HYBRID ORGANIC-INORGANIC NANOSTRUCTURED CATALYSTS FOR ALKYLATION OF ISOBUTANE BY OLEFINS

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In present study the elimination of some shortcomings of the alkylation process by means of homogeneous catalysis has been studied. The necessary catalytic systems would to have the properties of a liquid acid and they have to be environmentally safe. One of these areas is the ionic liquids. These ones are in liquid state at temperatures lower than 100°C and consisting of organic cations and inorganic anions. Well-known superacidity of the ionic liquids containing aluminum chloride is most often associated with the presence of dry HCl, dissolved in the ionic liquid, which involved in the formation of delocalized and highly reactive protons.

In our experiments the concerned ionic liquid (1-n-butyl-3-methylimidazoline chloride - AlCl₃) has been distributed on the surface of the acid-activated montmorillonite (MM) by chemical grafting. MM has been used as the matrix and the grafted organic film provided the catalytic activity, created new hybrid organic-inorganic nanostructured catalytic systems (ionic liquid/MM catalytic system).

Isobutane alkylation by interaction with butane has been promoted by “ionic liquid/MM” catalytic system. This process has been carried out at the following conditions: temperature - 70°C, pressure - 1.5 MPa, the raw flow rate ~1 h⁻¹, isobutane/olefin ratio ~50/1. Composition of the reaction products is showing in the table.

Table Investigation the composition of the reaction products of the isobutane catalytic alkylation by olefins, promoted by ionic liquid/MM catalyst

Hydrocarbons	Alkylate composition, % wt.
C ₅ - C ₇	19.1
Total: isomers dimethylhexane	6.5
Total: isomers trimethylpentane	72.1
C ₉	2.3
Total:	100

OP-8

KINETICS AND EQUILIBRIUM STUDIES OF REACTIVE YELLOW 125 ADSORPTION ON A NITROGEN DOPED TiO₂ MODIFIED ZEOLITE

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Azo dyes, as an important class of dyes present in textile effluents, with different functionalities are difficult to be treated effectively by classical methods such as chemical coagulation/flocculation and biological methods because of their non-degradable, resistant and carcinogenic character.

The removal of colorants from industrial wastewater is considered an important application of the adsorption process using suitable adsorbents. For this, natural zeolites and their modified forms have a special value due to their characteristic properties including adsorption, catalysis and ion-exchange capacity.

In the present paper we examined the adsorption mechanism of Reactive Yellow 125 azo dye with the aim of identifying the ability of a nitrogen doped TiO₂ modified zeolite to remove the dye from aqueous solutions.

Towards this aim, a series of adsorption experiments were carried out, in the dark, under magnetic stirring at 20°C. The UV spectra of Reactive Yellow 125 solutions were measured on a Varian Cary 100 UV-VIS Spectrophotometer and the efficiency of the catalyst adsorption of the dye was determined in relation with Zeta potential analysis.

The adsorption data obtained were fitted with different equilibrium isotherm equations namely Langmuir, Freundlich and Dubinin-Radushkevich (D-R) where the best description of adsorption process was given by the Freundlich model with a correlation coefficient of 0.8963. For the adsorption kinetics we used three simplified kinetic models: first-order, pseudo-second-order and intraparticle diffusion model in order to predict the adsorption rate constants.

Taking into account that the modified zeolite framework has a net negative charge for the whole pH range, resulted from Zeta potential analysis, and that the structure of the investigated azo dye is the one of an anionic dye, all equilibrium data come to underline the difficulty by which the adsorption process takes place.

OP-9

SYNTHESIS OF MICROCAPSULES BASED ON NATURAL POLYMERS

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Introduction. Microparticulated systems are promising as vectors for biological active principles, due to their ability to release drugs in a controlled manner, to improve the stability of entrapped active substances and their biocompatibility or biodegradability, shell protection against degradation factors and reduction of tissue irritation [1,2], especially when prepared from natural polymers. Our purpose is to prepare core shell chitosan/gelatin microcapsules using a double emulsion technique and a double crosslinking (ionic and covalent) original method.

Materials and Methods. Chitosan (CS) of low molecular weight, gelatin type A (G), toluene, Span80, Tween 80, sodium sulphate, glutaraldehyde were purchased from Sigma-Aldrich. The microparticles have been prepared by double crosslinking, using a double emulsion (O/W/O) technique. The microparticles are characterized by FT-IR, particle size analysis, SEM, particle morphology, evaluation of swelling degree, drug loading/release efficiency and *in vitro* drug release kinetics.

Results and Discussions. The prepared microparticles present good stability due to double crosslinking. The presence of gelatin into the particles matrix determines high swelling degree and drug loading/releasing efficacy. The polymers crosslinking is proved by IR spectra. Particle size and drug loading/release capacity depend on the ionic crosslinker/polymer molar ratio and the CS/G weight ratio.

Conclusions. The above described original method for preparation of core shell microparticles with interesting properties shows a promising wide range of applications.

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OP-10

FROM $\text{Fe}_2\text{O}_3@ \text{BaTiO}_3$ CORE-SHELL PARTICLES TO MULTIFUNCTIONAL COMPOSITES

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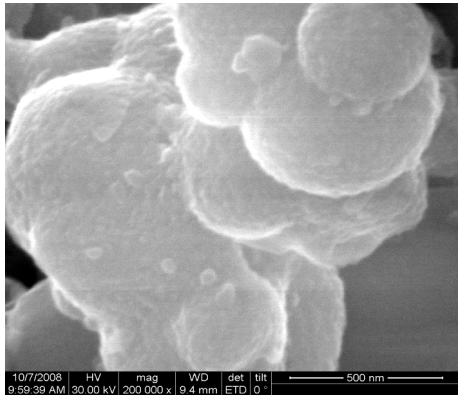
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In the present study we describe the synthesis of $\text{Fe}_2\text{O}_3@ \text{BaTiO}_3$ core-shell particles and their use as reactive templates for the preparation of multifunctional composites containing one dielectric/ferroelectric phase and two magnetic phases with contrasting coercivities ($\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4$, $\text{BaFe}_{12}\text{O}_{19}/\text{Ba}_{12}\text{Fe}_{28}\text{Ti}_{15}\text{O}_{84}$) [1]. This combination leads to limited dielectric losses and peculiar magnetic properties. Formation of new magnetic phases occurs during sintering or post-annealing by reaction between BaTiO_3 and Fe_2O_3 (Fig. 1a). The starting powders have been prepared by a multi-step process combining colloidal chemistry methods and a solid-state reaction. The magnetic properties of the composites can be tuned by controlling the amount of Fe_2O_3 in the initial particles, the densification method (conventional or spark plasma sintering) and the temperature of the thermal treatments. The composites show constricted magnetic hysteresis loops with coercivity of 0.1-2.5 kOe and saturation magnetization of 5-16 emu/g (Fig.1b). Composites obtained from powders containing 30 vol.% Fe_2O_3 show at 20-80°C and 10kHz-1MHz a relative dielectric constant of 50 and dielectric losses <10%. Our results provide evidence of the possibility of fabricating multifunctional composites by in situ solid-state reactions using suitable reactive templates. Well designed reactive templates allow for a careful control of the solid-state reaction at the nanoscale, thus enabling the fabrication of materials and composites with specific morphologies, micro/nanostructure and functional properties using a knowledge driven procedure rather than a trial and error process.

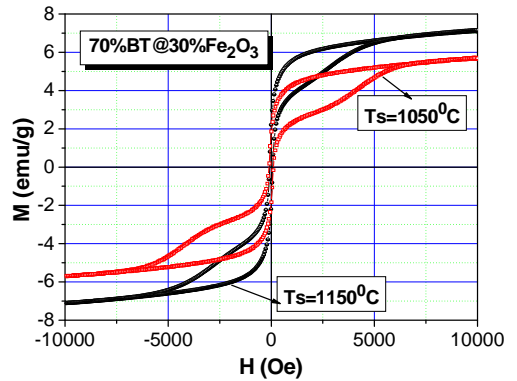
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(a)



(b)

Fig.1 (a) SEM images of 0.70BaTiO₃ @ 0.30Fe₂O₃ core-shell nanocomposite powder; (b) magnetic properties for same composition sintered at two temperatures.

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ACKNOWLEDGEMENTS

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OP- 11

**MODULATION OF THE CAPTOPRIL INTERFERENCE WITH THE
ACTIVITY OF SOME ENZYMATIC BIOMOLECULES IN MONKEY
KIDNEY VERO CELLS BY MESOPOROUS SILICA DRUG
DELIVERY SYSTEMS**

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Keywords: *mesoporous silica, captopril, nanocomposites, monkey kidney Vero cells, membrane bound and intracellular enzymes, oxidative stress, enzymatic activity.*

Abstract: The present research investigated the effect of different formulations of captopril on some cellular enzymatic equipments activities of monkey kidney Vero cells. The preparation of the samples of the mesoporous silica nanocomposites loaded or not with captopril was described and their effects on membranary Na⁺-K⁺-ATP-ase, cell Mg²⁺-ATP-ase, lactat dehydrogenase (LDH), peroxidase (Px), glutathion-peroxidase (GSH-Px), superoxid dismutase (SOD), catalase (CAT), acid (ACP) and alkaline (ALP) phosphatase activities were studied. The cells were incubated for a period of 144 hours, with growing medium renewed twice. When the cells reached confluence in the monolayer stage, the cultures were divided into control and other 4 groups of treated cell cultures. Control Vero cells were considered for evaluation of the preparations action upon cell enzymes activities. To the 12 hours treated cells the following materials were added: captopril (Cap H₂), mesoporous silica matrix (SBA-15), unfunctionalized (SBA-15_CapH2_RT) and functionalized (SBA-15_APTES_CapH2_80°C) nanocomposites, each of them in a dose of 0.4μg./flask, As compared with the control Vero cells, which are characterized by a specific level of the enzymatic activities, the cultures treated with SBA-15 have not presented significant alterations of the cytophysiologic process.

The comparative study of captopril formulations interacting with some membrane bound and intracellular enzymatic biomolecules of monkey kidney Vero cells, revealed an enhancement of membranary Na⁺-K⁺-ATP-ase, intracell total ATP- ase, LDH, ACP and GSH-Px activities, as well as a repression of cellular CAT, Px and SOD functioning intensity for the SBA-15_APTES_CapH2_80°C. These variations of the enzymatic activities –

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which induce modifications of metabolic processes - could be due to a direct or indirect interaction of captopril with cellular (plasmalemma) or subcellular (organelles) structures and with intracellular biomolecules (enzymes, DNA, RNA etc.).

The association of captopril with SBA – 15 or SBA – 15 _ APTES mesoporous silica matrices and treatment of Vero cells with these nanocomposites was correlated with modulation of the captopril interference at cellular level. The activity of investigated enzymes, the sense (stimulation or inhibition of enzymatic activity) and amplitude (high or small modulation) of their reactivity were dependent of carrier type and particularities of formulation. Interestingly, the enzymatic effects were induced with a lower quantity of captopril loaded per sample than in the non functionalized formulation.

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OP-12

REMOVAL OF RY 125 FROM AQUEOUS SOLUTION USING A SILVER DOPED TiO₂ MODIFIED CLINOPTILOLITE. PRELIMINARY DARK ADSORPTION STUDIES

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Pollution caused by industrial wastewaters has become a major problem for many countries; especially, organic, inorganic and dye pollution from industrial effluents disturb human health and ecological equilibrium.

Reactive dyes are widely used in dyeing process in textile industry, of which about 20-40% remain in the effluents.

Removal of color from textile effluents by conventional methods such as: coagulation, biological treatment, chemical oxidation, membrane filtration, osmosis, etc., are ineffective because the reactive dyes are resistant to biodegradation, oxidizing agents, high capital and operational costs or secondary sludge disposal.

Adsorption is an excellent way to treat textile waste effluents, offering significant advantages such as cheapest, profitability, easy of operation and most efficient, compared with conventional methods, especially from economical and environmental point of view.

The characteristics of natural zeolites and modified forms, such as high adsorption capacity, ion-exchange capacity, catalytic activity make it a powerful material for environmental remediation processes.

The aim of the present work was to study the dark adsorption of Reactive Yellow 125 using a silver doped TiO₂ modified clinoptilolite as preliminary stage in the removal process by heterogeneous photocatalytic oxidation.

The solid-liquid equilibrium experimental data have been correlated with six mathematical models: Langmuir, Freundlich, Dubinin-Radushkevich, Temkin, Harkins Jura, Halsey. The obtained results were correlated with the Zeta potential measurements.

Several kinetic models (Lagergren, Ho-McKay, Weber-Morris intraparticle diffusion model, Dumwald-Wagner intraparticle model) have been proposed to check the behavior of the experimental data.

OP-13

**NANOCATALYSIS AND THE WAYS OF THE NANOCATALYTICS
FORMATION FOR CONVERSION OF LIGHT HYDRCARBONS
INTO MOTOR FUELS**

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With the aim to create the new high-performance catalysts for conversion of light hydrocarbons by means of the Fisher-Tropsch synthesis as well as alkylation of the isobutane by olefins the number of nanostructured carriers have been synthesized on the basis of acid-activated montmorillonite and then studied by forming the nano-sized catalytic sites both on the surface of the proposed speakers and existing industrial samples of zeolites by following methods:

Mechanical activation by nanosized metal powders (NSP).

Surface grafting of the organometallsiloxanes (OMS) containing OH-groups and phenyl radicals. Interaction with OH-groups of the catalytic surface promotes OMS grafting with the allocation of water molecules and benzene, and then formed on the catalytic surface the active structural composition having the polyhedron form which contains different molecular ensembles determining increase the catalytic activity of the new material.

Given results have been showed following:

Nanostructured bifunctional catalysts prepared on the base of acid-activated montmorillonite, coated by cobalt atoms and modified by ruthenium-zirconium-phenyl-siloxan up to 1% wt., can convert synthesis gas into isoalkanes with the yield $\sim 80-90 \text{ g/nm}^3$.

Inoculation of the ruthenium-phenyl-siloxan into zeolite type Y mechanically activated by the cobalt NSP increased $\text{C}_5\text{-C}_{10}$ iso-alkanes yield up to 98 g/nm^3 .

Mechanical activation by nickel/rare-earth elements NSP's or by inoculation of zirconium- lanthanum-phenyl-siloxan increases the selectivity the isomers C_8 generation and duration of the isobutene alkylation by olefins on the surface of HCaLaUSY/Si-O-Zr-zeolite catalyst up to 50 hours accompanied by high-level preservation of the main indicators.

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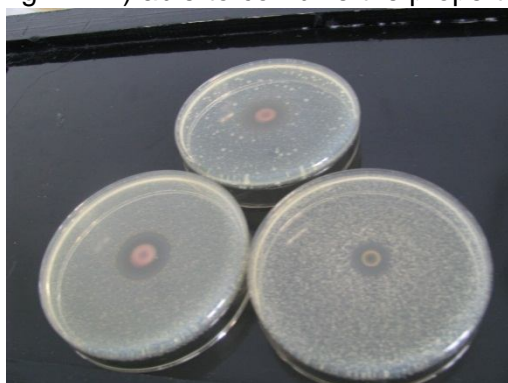
OP-14

NANOSIZED SILVER ON HYDROTALCITE-LIKE ANIONIC CLAY AS A NEW ANTIMICROBIAL FORMULATION

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Ensembles of different nanostructures which are able to combine their characteristics and to organize themselves for creating materials owning complex properties have a great potential in biomedicine applications due to the particular interactions that can be established between nanoscaled architectures and biological interfaces. We present here nanostructured assemblies of nanoparticles of silver and hydrotalcite-like anionic clay matrix (MgAILDH) able to combine the properties of the biocompatible matrix of the



anionic clay and the specific induced characteristics of the nanosized silver (Ag) into one single material. The antibacterial activities of Ag/MgLDH against *E. coli* and *S. aureus* were qualitatively and quantitatively assessed by agar diffusion tests and MIC (minimal inhibitory concentration) values, respectively.

Figure1. Symptoms of clear zones of Ag/MgAILDH against (a) *E. coli*.

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OP-15

PHOTOCATALYTIC APPLICATION OF SOME NEW HT-BASED NANOCOMPOSITES

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Titania (TiO₂) colloid particles have been widely used for the photocatalytic degradation of organic molecules due to the semiconductor property. It is especially interesting to support TiO₂ on different substrates for wastewater and atmosphere remediation. The immobilization of TiO₂ in the form of a nanocomposite provides an advantage over the drawbacks encountered with pure TiO₂ powders: (i) difficulties in separation from suspensions when they are used in aqueous systems and (ii) the suspended TiO₂ particles tend to agglomerate into larger particles especially at high concentrations reducing the photocatalytic performances. Cationic clays were used to obtain nanocomposite systems type TiO₂/clays having the advantage of a better separation from the aqueous systems than the pure TiO₂ powder [1]. HT solids (anionic clays) may be used to obtain nanocomposite systems like HT/TiO₂ having the advantage in separation. Up to now the HT solids were not used to create such systems. In this study we report the synthesis of some composite nanostructures comprising HT structures embedded in anatase matrix. The final nanocomposites were tested for the photodegradation of methylene-blue dye under UV light.

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OP-16

NANOTECHNOLOGY – A RAPIDLY GROWING FIELD OF RESEARCH AND DEVELOPMENT

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**"Knowing is not enough; we must apply.
Willing is not enough; we must do."**

Goethe

Nanotechnology first gained recognition after Nobel Laureate, Richard Feynman, presented his talk, *"There's Plenty of Room at the Bottom"* to the American Physical Society in 1959. Activity surrounding nanotechnology began to slowly increase over the next few decades. In 1988, Eric Drexler taught the first course on nanotechnology. In that program, he suggested the possibility of nanosized objects that were self-replicating. Slow growth in this area of scientific investigation continued. The next major milestone was when Rice University Professor Richard Smalley won the 1996 Nobel Prize for discovering a new form of carbon: a molecule of sixty carbon atoms (referred to as C₆₀)

The idea of manipulating and controlling things on a small scale and predicted that there is plenty of room at the bottom is the central idea of nanoscience and nanotechnology.

It is known that a nanometer is roughly 1,000 times smaller than the wavelength of visible light and at this scale - the level of individual atoms and molecules - the properties of matter are fundamentally different. Governed by quantum mechanics, these properties are profoundly interesting to basic science and also extremely useful in practical applications. Nanotechnology has created a growing sense of excitement due to the ability to create and utilize materials, devices, and systems through the control of matter on the nanometer scale (1 to 50 nm).

Nanotechnology - the application of nanoscience research - is becoming more and more popular everyday and refers to the ability to employ scientific principles in deliberately creating structures with nanoscale features that deliver unique functionality and utility for target applications. The advancements in nanotechnology really began to accelerate in the late 1990s. nanotechnology captured the thoughts and imagination of scientists. Nanotechnology is of key importance to realize a sustainable society. In order to fulfill its role, it is essential to establish new science and technology that will enable us to create nanomaterials and to employ them into practical uses.

In recent years, our ability to explore this new frontier has increased spectacularly. Advances in imaging, measurement, simulation, and

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manipulation have given researchers unprecedented understanding and control of molecules, atoms, and photons. The refined analytical techniques developed in the middle of the last century have allowed chemists to understand the true nature of many materials both at the atomic and molecular scales. Scientists and engineers can now work with both living and nonliving materials that exhibit nanoscale properties, even creating artificial atoms and building devices smaller than DNA.

The application of nanotechnology has enormous potential to greatly influence the world in which we live and holds great promise for improving our lives and effecting profound scientific, medical, economic and cultural change on society. From consumer goods, electronics, computers, information and biotechnology, to aerospace defense, energy, environment, and medicine, all sectors of the economy are to be profoundly impacted by nanotechnology.

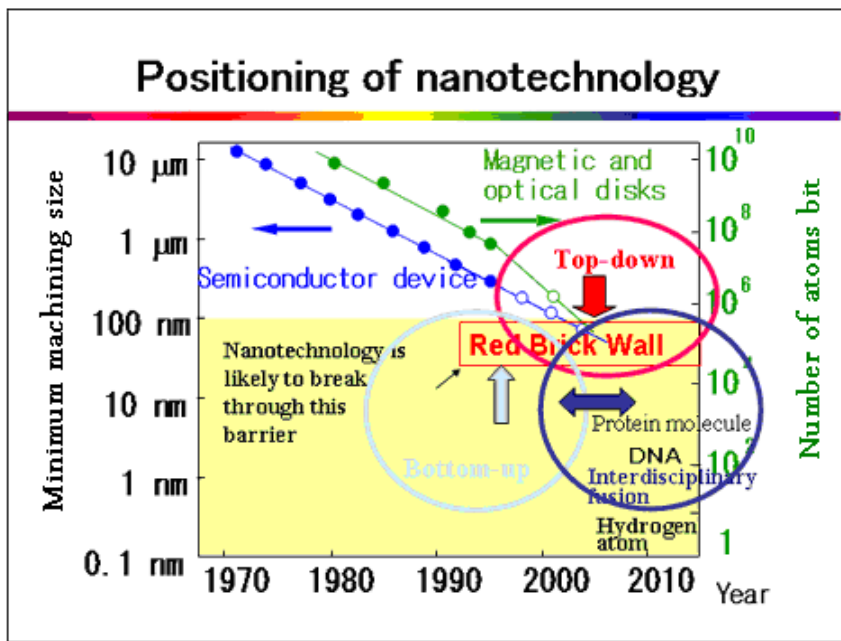


Fig.1. The first generation of nanotechnology.
(A. Wada ,RIKEN, Dec 18,2001)

Nanotechnology pulls theories and conceptions from disciplines not only comprising engineering and physics but also chemistry, biology, mathematics and computer science. Moreover, the emerging field of nanoscience and nanotechnology is leading to a technological revolution in the new millennium.



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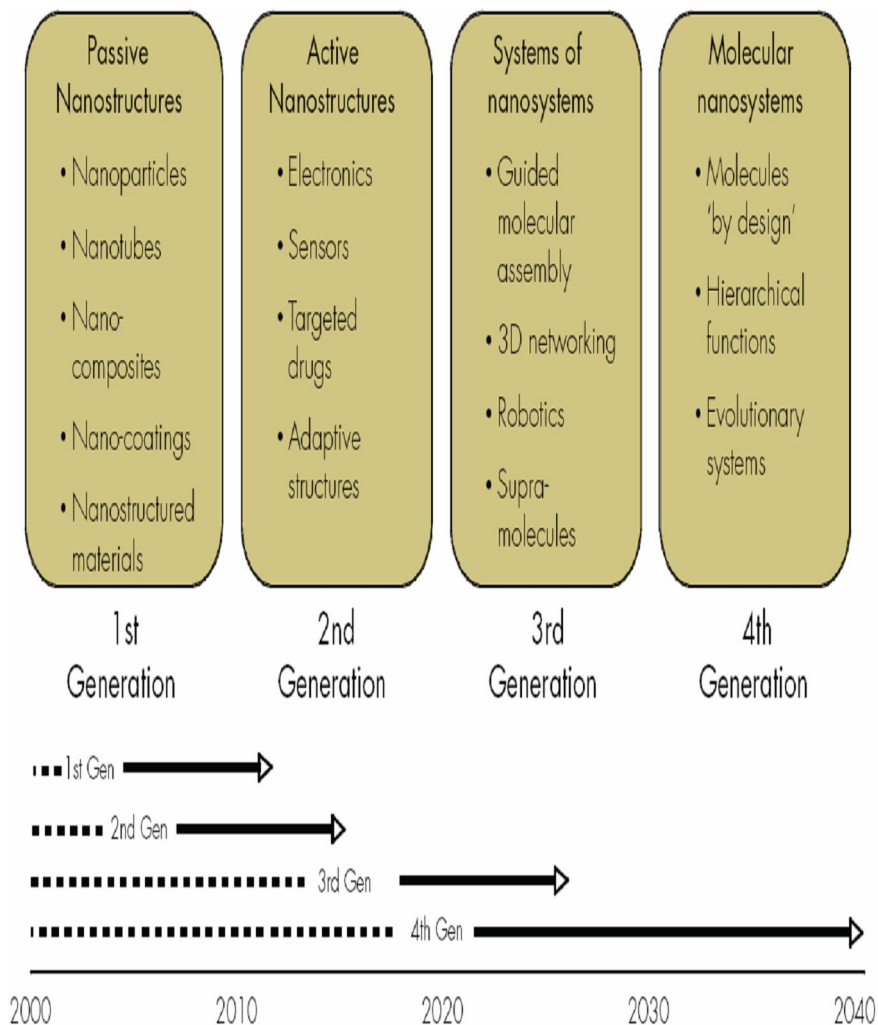


Fig. 2. Development of nanotechnology 2000-2040
(Roco, M.C., *AIChE. J.* 50(5), 890-897, 2004)

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POSTERS

NANOSTRUCTURED MULTIFUNCTIONAL MATERIALS

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1. NANOSTRUCTURED POROUS MATERIALS

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1-P-1

DIELECTRIC PROPERTIES AND NON-LINEAR CHARACTERISTICS OF BaTiO₃ CERAMICS DERIVED FROM NANOPOWDERS PREPARED BY THE MODIFIED PECHINI METHOD

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The ferroelectrics show a strong nonlinearity under the electrical field. In the last few years, electric field-tunable dielectrics have attracted much interest for their potential applications as variable capacitors, phase shifters, tunable filters and voltage-controlled oscillators [1], particularly in circuits and devices needed by the wireless communications industry, for scientific, space, commercial and military use. The electric field-induced tunability describes the ability of a material to change its permittivity by the electric field.

In this work, BaTiO₃ nanopowders were prepared by the modified Pechini. TEM and HRTEM investigations pointed out that the as-prepared BaTiO₃ powders consist of well crystallized, small nanoparticles (of ~ 30 – 60 nm) (Fig.1a). Ceramic samples were obtained from these powders after spark plasma sintering. Unlike the coarse-grained ceramics that resulted after classical sintering dense, microstructurally homogeneous and fine-grained ceramics were obtained by spark plasma sintering (SPS) in the temperature range of 1000 – 1050°C. In this non-conventional sintering process grain growth is strongly inhibited, so that the particle size from the precursor oxide powders is almost preserved in the nanocrystalline ceramics.

Impedance spectroscopy in the temperature range of (20, 150)^oC demonstrated a diffuse phase transition and a shift of the ferro-para phase transition to lower temperatures.

The dc-tunability at room temperature were experimentally investigated and described within a few models. The Landau-Ginzburg-Devonshire (LGD)

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theory and its approximate treatments (Johnson relation) approach in case of a single polarization mechanism was used in the polar state of the ceramics to describe tunability data and it was a good correlation between theory and experiment.

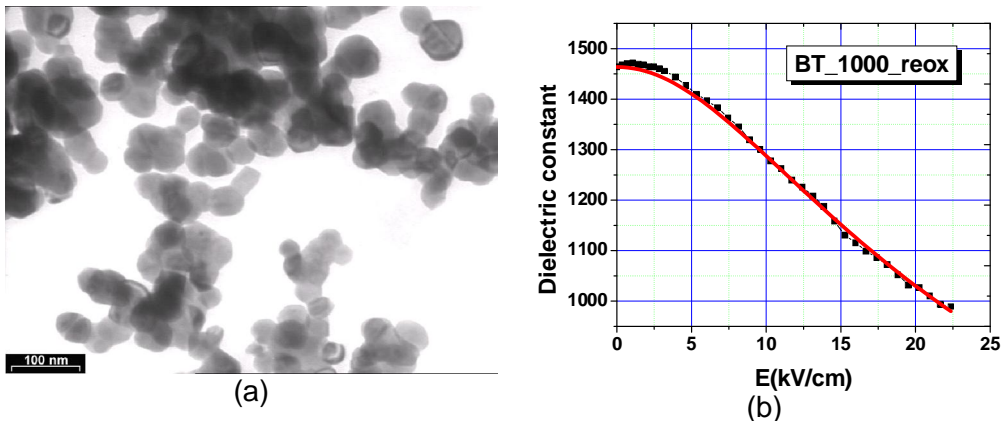


Fig. 1 (a) TEM image of BaTiO₃ powders prepared by Pechini method; (b) The dc-tunability measurements and fitting of experimental data with Johnson equation.

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1-P-2

TRI-NUCLEAR CARBOXYLATE IMMOBILIZED ON SILICA

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This paper presents the result of attempts to immobilize the tri-nuclear carboxylate of transition elements on inorganic supports. The tri-nuclear carboxylate can be used as catalysts in some oxidation reactions. The immobilization process on inorganic supports combines the advantages of homogeneous catalysis with those of heterogeneous catalysis.

The inorganic material used was silica functionalized with (3-Mercaptopropyl)-trimetoxysilane.

The coordinative compound synthesized, the functionalized support and the obtained catalyst, by immobilization of the coordinative compound, were characterized by UV-VIS and IR spectroscopy, diffuse reflection spectroscopy and derivatography.

The potential catalytic activity was evaluated in the process of decomposing of hydrogen peroxide.

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1-P-3

LAYERED SILICONE-SILVER COMPOSITES

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The obtaining of the composites based on metallic nanoparticles with well-defined dimensions and structures is of high interest for sensors, electrodes, biosensors, catalysis, and optical application [1-3]. Some composites based on silicone matrix and silver particles have been prepared by mixing a prehydrolysate based on three silanes (methyltrimethoxysilane, dimethyldiethoxysilane, and 3-aminopropyltriethoxysilane) with a freshly prepared AgNO_3 solution in DMF. The resulted sols were layered on the glass substrate and kept in the laboratory environment to harden as a result of finalizing the hydrolysis and condensation of the silanes. In order to investigate structural properties of composite films, for different silver contents, X-ray diffraction, and scanning electron microscopy measurements were performed. Electrical studies, made as a function of temperature, evidenced the importance of the content of silver nanoparticles on the conduction mechanism.

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1-P-4

SELECTIVE RING-OPENING OF NAPHTHENIC COMPOUNDS ON SUPPORTED BIMETALLIC Pt-Rh CATALYSTS

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To answer to the increasingly strict regulations for the protection of environment, it is necessary to produce cleaner and cleaner automobile fuels. In particular, aromatic compounds must be reduced and replaced by paraffins, substituted in the case of the gasoline, or linear in the case of diesel oil. The production of such fuels supposes the total hydrogenation of the aromatic compounds in naphthenic compounds, then the selective opening of the latter to paraffins while avoiding the secondary reactions in particular deep hydrogenolysis leading to light hydrocarbons [1-4].

In this research field, the ring opening of methylcyclopentane (MCP) is often used as a probe reaction in the investigation of the structure sensitivity of several hydrocarbon conversions catalyzed by noble metal-based catalysts. Among the monometallic catalysts tested (Ru, Re, Rh, Pt and Ir), iridium was by far the most selective in ring-opening (RO) products (2-methylpentane, 3-methylpentane and n-hexane), the formation of C1–C5 products being negligible, as already reported in the literature [4].

Thereby, the aim of the present study is to modify a Pt/Al₂O₃ catalyst by addition of another metal in order to obtain Pt-based bimetallic catalysts with selectivity comparable to those of Ir catalysts for MCP hydrogenolysis in industrial conditions (P = 28,5 bar, PPH = 15 h⁻¹, H₂/MCP = 7.5, 200 ≤ T ≤ 425°C).

Among the tested series, only Pt-Rh showed interesting properties: whereas platinum is not very active for this reaction, the addition of rhodium by surface redox reaction allowed us to increase its activity. More surprising, whereas neither platinum nor rhodium were as selective as iridium, both favoring the production of C1–C5 products, the combination of the two metals in the same metal particles led to completely different selectivity, which was similar to that of iridium.

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1-P-5

A COMPARATIVE STUDY OF TiO₂ DEPOSITION ON SBA-15 BY IMPREGNATION WITH MAGNETIC STIRRING AND ULTRASOUND RADIATION

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In recent years, photocatalytic degradation attracts increasing attention as a promising technology for the removal of toxic organic and inorganic contaminants that are recalcitrant to biodegradation from water and wastewater [1]. As an efficient heterogeneous photocatalyst, high performance of titania strongly depends on the surface area, thermal stability and phase composition. SBA-15 is a mesoporous silica with uniform channels varying from 50 to 300 Å, high surface area, thick walls and hence a high hydrothermal and thermal stability [2]. TiO₂/SBA-15 materials could be obtained by different conventional post-synthesis methods [3, 4]. However, those techniques involve long processes time for TiO₂ deposition. Ultrasound radiation used as materials synthesis procedure allows reducing the time and favors a good dispersion of metals or metal oxides [5]. The very high local temperature, pressure and cooling rates due to acoustic cavitation phenomena give sonicated solutions unique properties.

In this work we prepared nanosized TiO₂ particles in SBA-15 material by impregnation with magnetic stirring and ultrasound radiation. We describe the structural differences between both materials by using several techniques: Powder X-ray diffraction pattern, N₂ sorption, Raman and UV-Vis DR spectroscopies. Both TiO₂/SBA-15 materials were used as photocatalysts in the degradation of methylene blue in aqueous solutions. Ultrasound technique demonstrated to be useful and shorter in TiO₂ nanoparticles formation on mesoporous SBA-15. On the other hand, the TiO₂/SBA-15 prepared via impregnation with magnetic stirring had a higher efficiency in MB degradation under the UV light than the other one prepared by ultrasound assisted treatment.

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1-P-6

OPTICAL, ELECTRICAL AND MORPHOLOGICAL STUDIES ON WO₃ THIN FILMS USING SURFACTANT MEDIATED SPRAY PYROLYSIS

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Finding a method for obtaining uniform thin layers with size controllable particles and narrow size distribution is the key in controlling the optical and electrical properties of materials in order to suit the desired application [1,2]. In this study thin films of WO₃ were prepared by spray pyrolysis deposition on F doped SnO₂ (FTO) conductive glass using a cationic surfactant as structure-directing agent. The effect of cetyltrimethylammonium bromide (CTAB) addition on crystalline structure, topography, conducting behavior and optical properties was investigated. I-V curves were registered in order to establish the semiconducting behavior of the layers as function of surfactant content. Reflectance and transmittance spectra of WO₃ layers were recorded using UV-VIS spectrometry and used for the calculation of film thickness and band gap. The morphological and structural characterization of resulting WO₃ thin films as function of surfactant addition levels has been examined using atomic force microscopy and X-ray diffraction.

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2. NATURAL AND SYNTHETIC ZEOLITES

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2-P-1

THE EXTERNAL CATIONIC EXCHANGE CAPACITY DETERMINATION AND ITS RATIO IN THE TOTAL CATIONIC EXCHANGE CAPACITY VALUES OF THE CLINOPTILOLITE FROM PIATRA-VERDE SLANIC-PRAHOVA

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The work presents the results of the performed analyses in order to obtain the External Cationic Exchange Capacity (ECEC) values of the clinoptilolite type volcanic tuff from Piatra-Verde Slanic –Prahova quarry.

The ECEC determination and its ratio in the total cationic exchange capacity was performed by a procedure inspired after Ming & Dixon method, which is based on the shape selectivity of the zeolite particles regarding to the tert butyl ammonium ions.

The determination of the clinoptilolite type features from Piatra-Verde is a starting point for obtaining the organo-zeolites with various applications.

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3. MESOSTRUCTURED MATERIALS - SYNTHESIS AND MODIFICATION

3-P-1

VISIBLE LIGHT RESPONSE OF N-DOPED MESOPOROUS TiO₂ PHOTOCATALYST.

THE EFFECT OF THE SYNTHESIS PROCEDURE

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Titanium dioxide has become the most frequently employed photocatalyst in realising complete mineralization of various organic pollutants in water treatment, owing to its low cost, non-toxicity, and structural stability. Its large bandgap energy (3.2 eV) necessitates though UV excitation to induce charge separation within the particle. Nitrogen doped into substitutional sites of TiO₂ has shown bandgap narrowing as well as photocatalytic activity in the visible light. N-doped mesoporous titania was synthesized using hydrothermal and ultrasound methods. Blockcopolymers Pluronic P123 and F127 were used as surfactants and titanium-tetraisopropoxide as Ti precursor. Different types of N-doped and non-doped mesoporous titania were synthesized by varying composition of surfactant and nitrogen precursor. UV absorption spectrum and N₂ adsorption-desorption techniques have been used to investigate the structure, morphology and optical properties of these photocatalysts. The photocatalytic activity of mesoporous

titania was studied by photoreactions of different dyes used in textile industry. From the experimental results it was observed that the N-doped mesoporous titania obtained by ultrasound method has higher photocatalytic activity as compared to N-doped mesoporous titania prepared by hydrothermal method. Also, as expected, N-doped titania has proven to have higher photocatalytic activity than the undoped mesoporous TiO₂.

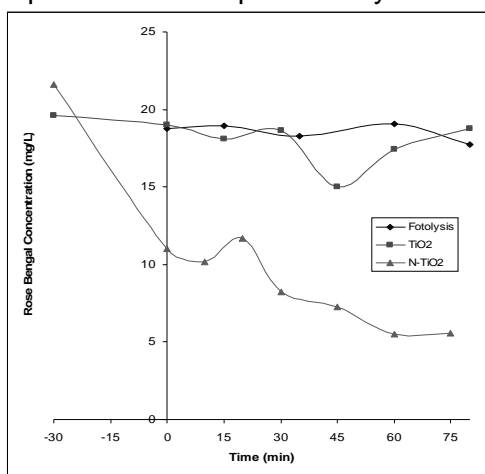


Fig. 1. Photocatalytic activity of N-

doped

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3-P-2

SYNTHESIS AND CHARACTERIZATION OF NOVEL COMPATIBILIZED CNTs-BASED NANOCOMPOSITES FOR EMI SHIELDING

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Carbon nanotubes (CNTs) have unequal physical properties that recommend them for various applications in nanocomposite materials for electronic devices, nano(bio)sensors or gas storage materials, etc., all of them intensively explored. Still, even when a good dispersion has been achieved in a specific polymer matrix by melt blending, co-precipitation or *in-situ* polymerization using unmodified CNTs, they are generally poorly dispersed within polymeric matrices, resulting in a slight improvement of nanocomposites properties.

The most widely used method to overcome this disadvantage is CNTs functionalization. Non-covalent and covalent modifications by organic molecules, including polymer chains, are widely used to improve CNTs dispersion. Non-covalent functionalization of CNTs resides in the use of polymers bearing aromatic molecules able to undergo π - π stacking with the CNTs surface. This technique allows the nanotube to preserve its structure and electronic properties unmodified.

This paper presents a new series of CNTs-polypropylene nanocomposites obtained using PP-g-MA grafted with aromatic moieties from aminomethylpyridine (AMP) and an amino-pyrene derivative (Py-NH₂). These compatibilizers are able to interact by π - π stacking with the surface of CNTs in order to improve their dispersion in polypropylene and the amine group serves to anchor the molecules to PP by reaction with maleic anhydride functions of PP-g-MA. The extent of the reaction between amino groups and anhydride functions was assessed by FTIR. The effect of the dispersion method (melt blending or co-precipitation) on the properties of the nanocomposites was studied by rheologic and conductivity measurements. It appeared that the co-precipitation is the most efficient technique, most likely because it allows ultrasonication. In contrast to unmodified PP-g-MA, the two compatibilizers were able to significantly increase the conductivity of PP/CNTs nanocomposites within limits that enable their use for EMI shielding applications.

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3-P-3

PREPARATION OF THE $BaTi_{1-x}Sn_xO_3$ CERAMICS BY THE MODIFIED PECHINI METHOD

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$BaTi_{1-x}Sn_xO_3$ ($x=0; 0.10; 0.15$ and 0.20) nanopowders were prepared by the modified Pechini method from titanium isopropoxide, tin isopropoxide and barium carbonate using acid as chelating agent and ethylene glycol as esterification agent.

X-ray diffraction data show the formation of $Ba(Ti,Sn)O_3$ solid solutions, free from secondary phases as $BaCO_3$ or Ti-rich oxides, when the polymeric precursors were thermally treated in air at $900^\circ C$ for 2 hours. As the Sn content, x , increases, the tetragonality degree decreases, so that the unit cell symmetry changes from a tetragonal one, specific to the pure $BaTiO_3$ powder towards a cubic form, for powder with $x=0.15$. The particle average size of the as-prepared powders decreases from 44 nm, for pure $BaTiO_3$ to 22-26 nm for the $BaTi_{1-x}Sn_xO_3$ solid solutions. The microstructural features (density, porosity, grain size and grain size distribution) of the ceramic pellets obtained after shaping and classical sintering at $1300^\circ C/8$ hours and $1400^\circ C/4$ hours obviously varied as a function of the sintering strategy.

The dielectric and ferroelectric properties of the $BaTi_{1-x}Sn_xO_3$ ceramics are strongly dependent on the Sn addition and on the grain size. A ferroelectric-relaxor crossover is induced when increasing the Sn concentration.

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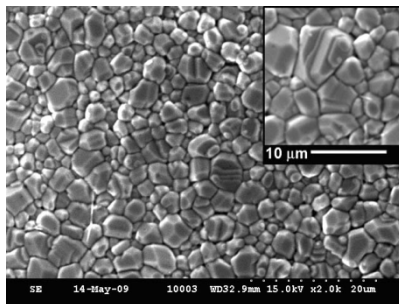


Fig.1. SEM images of the $\text{BaTi}_{1-x}\text{Sn}_x\text{O}_3$ $x=0.2$ sintering at $1300^\circ\text{C}/8$ hours

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3-P-4

POLYMER-METAL COMPOSITE MATERIALS AS ELECTROMAGNETIC RADIATION ABSORBERS

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A composite material suitable for electromagnetic radiation absorption is obtained using two-step technology. In first step, a Loeb-Surirajan-type polyethylene membrane was obtained using the technology of drawing and stretching the melting of extruded polymer. In a second step, metallic micro- and nano-particles was included using electrodeposition. Morphology and structure of materials was studied using x-ray diffraction technique and scanning electron microscopy. Electromagnetic field was created using a pair of Helmholtz coils powered using a power broad-band amplifier and a 0-3 MHz signal generator. Electromagnetic radiation absorption efficiency was measured using a digital gaussmeter in AC mode. The nanostructured material can be used in electromagnetic protection suit design.

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3-P-5

AROMATIZATION OF C₄/C₄⁻ TECHNICAL FRACTION OVER ZnO/HZSM-5 CATALYST PREPARED BY MECHANICAL MIXTURE

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The conversion of a technical fraction butanes /butenes in presence of a ZnO/HZSM-5 (SiO₂ /Al₂O₃ = 35,85; 5 % ZnO) prepared by mechanical mixture was studied. Pentasil zeolite ZSM-5 due to its special structure has been generally chosen. Synthesis of the ZSM-5 type zeolite in alkaline media has been performed, hydrotermaly. Etylene glycol has been employed as template. The catalytic tests as butanes /butanes fraction aromatization was carried out in a continuous fixed-bed flow stainless-steel reactor (Twin Reactor System Naky) reaction temperature 450 °C, and 4 atm pressure. Products were analyzed through by a capillary gas chromatograph. Catalytic activity and selectivity towards aromatic hydrocarbons BTX variation on time was studied.

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3-P-6

PREPARATION AND CHARACTERIZATION OF $Ba_{12}Fe_{28}Ti_{15}O_{84}$ NATURALLY SELF-ASSEMBLED LAYERED SYSTEM

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A recent study of the $BaO-Fe_2O_3-TiO_2$ phase diagram found a large number of new phases, with structures featuring moieties from both the polytitanates and hexaferrites, as well as new building blocks showing a mixture of both phases. One of this mixture phases is the ternary compound $Ba_{12}Fe_{14}Ti_{15}O_{84}$. The naturally self-assembled layered system prepared by two different methods: solid state reaction and combined wet chemistry method were presented in the present paper. Phase and crystalline structure analyses carried out by X-ray diffraction indicated a pure monoclinic structure for powders calcined at $1200^{\circ}C/48$ h. The HRTEM investigations confirmed the layered nature of the compound and the monoclinic unit cell parameters resulted from XRD analysis. The sintered ceramics show an intrinsic dielectric permittivity of the order of 40-60 at 10^8-10^9 Hz and $\tan\delta=0.35$ at 5×10^8 Hz, while at low frequency the apparent permittivity reaches values of 10^5 due to conduction phenomena, and are presented in Fig.1. A ferri/ferromagnetic order with coercivity of ~ 40 Oe, remnant magnetization of ~ 2.5 emu/g and saturation magnetization of ~ 13 emu/g was determined at room temperature, while the thermomagnetic data showed the cancellation of magnetisation at $T_C \sim 700K$.

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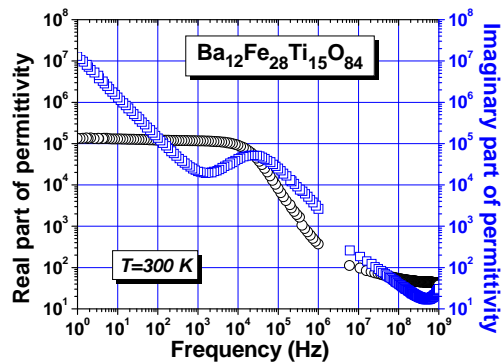


Figure 1. Real and imaginary part of permittivity vs. frequency measured at room temperature

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3-P-7

NOVEL PREPARATION OF NANOSIZED ZnO-SnO₂ WITH PHOTOCATALYTIC ACTIVITY

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Research studies on the coupled semiconductor nanocomposites reveal that they play a key role in the photocatalysis processes. In the last decade, there are many questions on the mechanism interaction between coupled particles. There is most unlikely the effectiveness of the coupling of the photocatalytic semiconductor nanoparticles, which is correlated with the interfacial charge transfer between two different semiconductors with different favorable band gap energy levels [1].

Our study is focused on a typical n-type semiconductor with a wide band gap, as tin oxide [2-6]. Due to their chemical and mechanical stability, band gap energy of 3.6 eV, the SnO₂ have been used in many photocatalytic reactions.

In this study, an innovative ZnO/SnO₂ photocatalysts active in UV light were prepared through one-step synthesis. Their properties were characterized by X-ray diffraction (XRD), N₂ adsorption-desorption isotherms and UV-Vis diffuse reflectance spectroscopy. The phase composition, surface properties and optical absorption of the samples were found to vary freely with zinc oxide to tin oxide ratio (table 1).

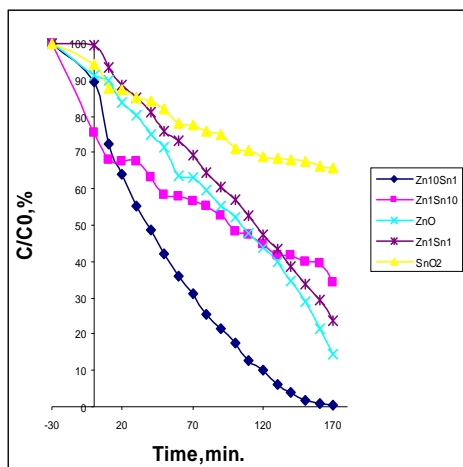


Table 1. The experimental data.

Sample	Molar ratio Zn/Sn	Composition (mol%)	
		ZnO	SnO ₂
ZnO		100	0
Zn ₁₀ Sn ₁	10/1	91	9
Zn ₁ Sn ₁	1/1	50	50
Zn ₁ Sn ₁₀	1/10	9	91
SnO ₂		0	100

Fig. 1: Photocatalytic activities of the samples.

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The photocatalytic process, using ZnO/SnO₂ semiconductors as photocatalysts, was applied on photocatalytic degradation of Eosin Y dye, as a reference reaction. The optimum molar ratio combination between Zn and Sn oxides was found to be 10:1. This sample exhibits high photodegradation efficiency, as can be observed from the figure. As conclusion, an efficient electron-hole separation at the photocatalyst interface occurs, in order to improve the photodegradation efficiency (fig.1).

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4. CATIONIC AND ANIONIC CLAYS

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4-P-1

REMOVAL OF P-CLORO-PHENOL IN SYNTHETIC WASTEWATER SYSTEMS Fe₂O₃-CLAY NANOCOMPOSITES

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Currently there is a substantial interest in the preparation and characterization of semiconductor nanoparticles, which is explained by a real potential for their application in solar energy conversion, photocatalysis and optoelectronics fields. Semiconductor nanoparticles have chemical properties, magnetic, optical and electronic dependent on their size. This is the point generating the idea of synthesis of oxide nanoparticles directly into interlayer's gallery belonging to a cationic clay. Thus, we made clay-Fe₂O₃ nanocomposite systems.

A-Fe₂O₃ oxide is an antiferromagnetic oxide, with high and unstable properties capable to use in semiconductor photocatalysis processes. The fundamentals of energy on this oxide photocatalysis are based on its electronic structure.

The presence of chlorinated phenolic compounds in surface waters is prohibited because they are toxic, carcinogenic, tend to bioaccumulation and are persistent. Severe limits required by international standards (ISO 14001) to the discharge of effluents has led to development of physical, biological and chemical processes (biological degradation - Biodegradation), coagulation, solvent extraction, incineration, reverse osmosis, membrane separation, adsorption), the introduction of oxidative processes and especially advanced oxidation processes (AOPs - Advanced Oxidation Process).

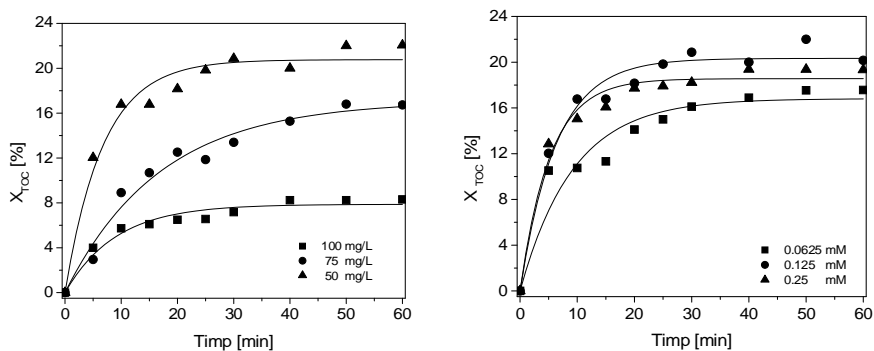
Results highlight the contribution provided by the use of clay samples pillared with Fe₂O₃, in processes at laboratory level, in order to remove p-chloro-phenol from synthetic wastewaters by advanced oxidation processes. Study of 4-chlorophenol degradation by Fenton process (H₂O₂/Fe²⁺) was carried out aiming at the following influences: initial concentration of p-chlorophenol, the concentration of Fe²⁺ (from Fenton reactive), the degree of mineralization, influence of H₂O₂ concentration (from Fenton reactive) the degree of mineralization of p-chlorophenol and the influence of H₂O₂ concentration.

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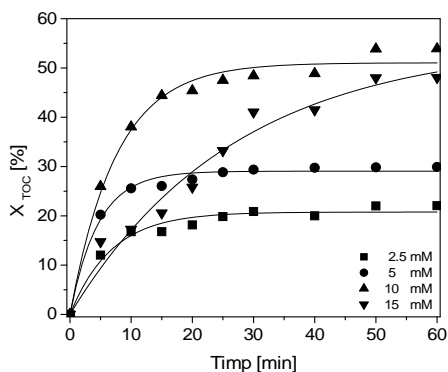
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Using native clays as host, the preparation of effective systems of advanced oxidation processes of phenolic polluted priority in the hazardous wastewaters has been performed.



(b)



(c)

Figure: The correlation of the mineralization degree with (a) the initial concentration of p-chlorophenol, (b) the concentration of Fe^{2+} , (c) the concentration of H_2O_2 .

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4-P-2

SORPTION OF RED ACID G DYE FROM AQUEOUS SOLUTION BY LAYERED DOUBLE HYDROXIDES

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Textile industry waste water contains a wide variety of dyes which are toxic and have a great influence on photosynthetic activity in aquatic medium. Generally, the dyes are characterized by low biodegradability and, preferably they might be removed by using less expensive materials, such as activated carbon, bentonite, layered double hydroxides etc. Layered double hydroxides (LDHs) are an important class of anionic lamellar solids which have received considerable attention in recent years because of their potential applications such as ion-exchangers, catalyst supports, antiacids and sorbents for organic solutes and, particularly for negatively charged species [1]. By heating LDHs at appropriate temperatures, their decomposition leads to mixed metal oxides which are characterized by large specific surface areas and homogenous dispersion of metal cations. The calcined LDHs can uptake anions from aqueous solution, with concomitant reconstruction of the original layered structure, due to the “memory effect” [2].

In the present work, we describe the removal process of azo dye Red acid G from the wastewater, using calcined Zn₂AlLDH. The anionic clay was prepared by coprecipitation method and calcined at 500°C for 4 h. The equilibrium isotherms were determined by putting in contact a constant mass of LDHs with equal volumes of dye solutions at different concentrations. The obtained suspensions were stirred under argon atmosphere with a rate of 200 rpm for 24 h. The concentration of the dye from supernatant was determined by means a spectrophotometer using a wavelength of 505.7 nm, and the quantity uptaken by sorbent was calculated by subtraction from the initial concentration. The shape of isotherms indicates that the sorption process does not occur on the surface of the LDHs only, but an intercalated process is also occurring concomitantly. The intercalation of the azo dye was proven by FT-IR spectroscopy measurements. A contact time of 60 min between the dye solution and calcined LDHs (2g/L) assures the removal of 99.12% dye from the solution of 50 mg/L concentration, and 96.84% from the solution of 200 mg/L concentration.

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4-P-3

EFFECT OF ANNEALING AND GAMMA IRRADIATION ON CLAY MINERAL PROPERTIES

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Most clays are chemically and structurally analogous to other phyllosilicates but contain varying amounts of water and allow more substitution of their cations. There are many industrial processes in which raw or modified clays have been used [1,2]. In this paper, the effect of annealing and gamma irradiation on the structural, electrical and magnetic properties of some green and grey clay minerals, collected from different sites (Dolhesti, Bradicesti, Iasi-Frumoasa) is investigated. X-ray diffraction evidenced a multiphase mineralogical composition; muscovite being the predominant phase. The annealing (at 500°C for 4h) and gamma irradiation procedures (0.126 mGy/h) induced some structural changes: some impurity phases are destroyed, some oxide phases are formed. Electron spin resonance spectra (room temperature, 9.4GHz) showed the presence of some paramagnetic species (Fe^{3+} , Mn^{2+}), whose neighborhood was changed by the thermal treatment. The electrical conductivity of clay minerals was investigated as a function of temperature and was found to be dependent on the sample composition and water content. The measurements made with ultra high resolution gamma spectrometry showed that concentration of natural radionuclide series and primarily descendants of K-40 were not changed by the annealing and gamma irradiation procedures.

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5. ENVIRONMENTAL APPLICATIONS

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5-P-1

MODIFIED MESOPOROUS SILICA MATERIALS FOR COPPER RECOVERY FROM WASTEWATER

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Copper is a significant metal pollutant due to its widespread use, but also is an essential trace element that plays a pivotal role in a variety of fundamental physiological processes in organisms ranging from bacteria to mammals [1]. However, exposure to a high level of Cu^{2+} can cause major disturbances in the physiology of living organisms. Removal of copper ions from wastewater was extensively studied. Sorption of copper ions on nanoporous materials is considered to be an effective method for improving the environment. Mesoporous materials (with pore size in the range 2-50 nm) have great potential in applications like environmental technologies. The tunable pore sizes of 2-10 nm and superficial large surface areas extending up to 1000 m^2/g makes MCM-41 an interesting material for environment remediation processes.

A mesoporous silica material, MCM-41, was functionalized with 1-thiocarbamylid-formyl-4-aryl-thiosemicarbazide (TSC) by co-condensation method in the presence of cetyltrimethylammonium bromide (CTABr) as a template under basic conditions. Post synthetic functionalization method was also applied. The synthesized materials were characterized by XRD, FTIR, UV-DR, and nitrogen adsorption-desorption isotherm as well as elemental analysis. The influence of the functionalization level of mesoporous silica MCM-41 on the equilibrium and kinetics of copper ions sorption from wastewater was studied. It was also established the method for the recovery copper from the saturated mesoporous material. The results show that the MCM-41 organized structure is preserved after recovery copper. The functionalization process modified the surface properties of the mesoporous silica materials, making it a promising material for recovery of copper ions from waste water.

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5-P-2

REMOVAL OF CHROMATE FROM AQUEOUS SOLUTIONS USING SURFACTANT- MODIFIED NATURAL ZEOLITES

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A promising application of purified and modified Romanian natural clinoptilolite has been studied. Natural zeolites have been widely used to remove cationic heavy metals from aqueous solutions, but they are not useful for removing inorganic anions such as chromate (CrO_4^{2-}). Due to the net negative charge on the framework, zeolites have little or no affinity for anions. It is known that the external surface charge of natural zeolites can be modified with a surfactant, thereby resulting in a positively charged external surface suitable for adsorbing anionic ions [1-3].

In the present study two kinds of surfactants (cationic and non-ionic) have been employed for the obtainment of different surfactant - modified natural zeolite samples. The obtained surfactant - modified clinoptilolite samples were used to remove chromate from aqueous solutions. The structures of purified clinoptilolite and surfactant - modified clinoptilolite samples were characterized by XRD, FTIR and nitrogen adsorption-desorption isotherm. Kinetic curves and sorption isotherms for the two type surfactants on purified clinoptilolite were determined. The effects of contact time, initial chromate ions concentration, pH of solution on the chromate sorption were evaluated. Chromate sorption was determined from the difference between initial and final solution concentrations, with appropriate corrections based on blanks. The chromate ions sorption capacity of cationic surfactant-modified clinoptilolite was significantly higher than that of non-ionic surfactant-modified clinoptilolite. Also, surfactant modified clinoptilolite saturated with chromate ions regeneration was studied using solvent extraction process. Promising results were obtained for extraction with sodium bicarbonate solution.

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5-P-3

AMMONIUM REMOVAL BY MODIFIED VOLCANIC CLINOPTILOLITE TUFFS

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The presence of ammoniacal nitrogen (sum of unionized ammonia, NH_3 and ammonium ion, NH_4^+) in various types of agricultural, municipal and industrial waste waters can be detrimental to a variety of living species. The maximum level for drinking water set by the Council of European Community is of 0.5 mg NH_4^+/L [1]. A high concentration of ammoniacal nitrogen in aquaculture water can be fatal for most fish species. [2]

Objectives of this study were to estimate the ability to remove ammonium ions by natural zeolites tuff collected Mârșid area.

Among to natural zeolites, clinoptilolite has shown a great affinity for ammonium ions. Ammonium exchange capacity depends of the purity to the natural zeolite, the nature of cation exchange, the tuff treatment of and the experimental conditions used to remove ammonium.

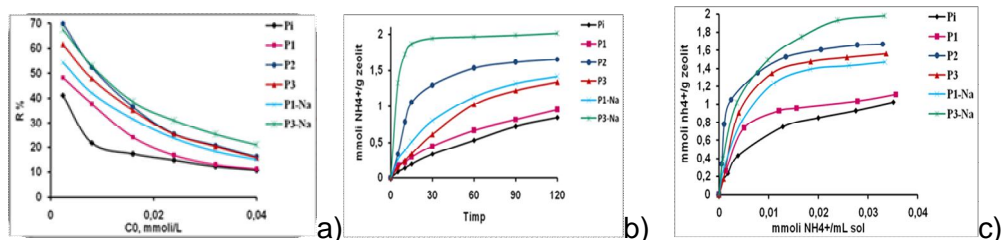


Fig.1. Degree (%) of ammonium ions retained on natural and modified tuff (a), evolution with time of the removal degree of ammonium on natural and modified tuff (b) and the ammonium ions distribution between aqueous solution and zeolite tuff(c)

The sorption of ammonium from aqueous solutions on natural and modified tuffs depends the initial ion metal concentration, contact time and chemical treatment (acid treatment, with HCl and $\text{H}_2\text{C}_2\text{O}_4$, and alkaline treatment, with NaOH). Solid affinity for ammonium ions decreases in order: $\text{P3-Na} > \text{P2} > \text{P3} > \text{P1-Na} > \text{P1} > \text{Pi}$.

Results obtained from the adsorption experiments showed a good efficiency in removing ammonium from water.

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5-P-4

FUNCTIONAL COATING IN LINEN/COTTON FIBROUS SUPPORTS USING ZINC OXIDE NANO PARTICLES FOR UV PROTECTION

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Protective treatment of fabrics and textiles using the latest results of nanotechnology like nanoparticles, nanostructured coatings, smart materials etc is one of the newest textile finishing techniques with wide application in improving the performance of textiles [1- 4]. The present work is directed to develop a safe transfer technique of zinc oxide nanostructures onto textile supports as well as detailed study of physical/chemical properties of nanostructures onto textile substrates. The preparation of the ZnO colloids and the cotton surface functionalization are described in a detailed way. Thus, zinc oxide nanoparticles were prepared by wet chemical method using zinc nitrate and sodium hydroxide as precursors and Cavatex W7 MCT (Monochlor-triazinyil Beta – Cyclodextrine Na salt) as stabilizing agent. These nanoparticles were coated on the linen fabrics (plain weave, 30 s count) using acrylic binder and functional properties of coated fabrics were studied. The topographical analysis of the treated fabric and untreated fabric were studied and compared. Some microscopic investigations (SEM/AFM) analysis revealed the embedding of ZnO nanoparticles in treated fabrics. Surface Electron Microscopy showed the homogenous coating of fibers. FTIR ATR spectroscopy was also tested for coated samples. UV-vis with diffuse reflectance also revealed the fact that ZnO, induces the UV-blocking properties.

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Keywords: fibrous materials, functionalization, optic investigations, FTIR-ATR, ZnO nanoparticles, UV protection

5-P-5

USE OF NATURAL AND MODIFIED CLAY FOR THE REMOVAL Zn(II)

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Heavy metals cations are released in the environment by natural phenomena and human activities, such as agricultural practices, transport, industrial activities and waste disposal.

Due to their high negative impact as environmental contaminants, in this research the removal of Zn(II) from wastewaters was studied. Clays from Valea Chioarului and Razoare areas - Romania as well as Na-clays were investigated as sorbents.

The effect of initial concentration of Zn(II), temperature and time on removal efficiency was studied. The equilibrium data were fitted with two standard isotherms, the Langmuir and Freundlich models. The Langmuir model was more suitable for these systems. The highest ion exchange capacity based on Langmuir constants was displayed by the Na-exchanged clay.

The kinetic studies showed that the sorption rates could be described by a second-order Lagergren equation.

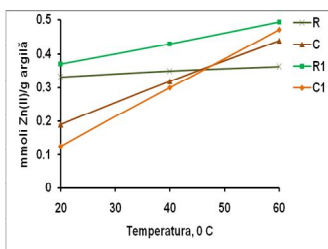


Figure 1. The influence of temperature on the metal ions sorption

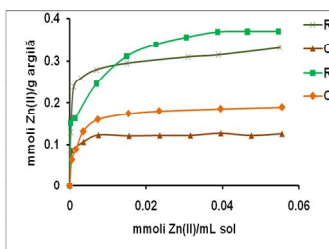


Figure 2. The Zn(II) ions distribution between aqueous solution and clay

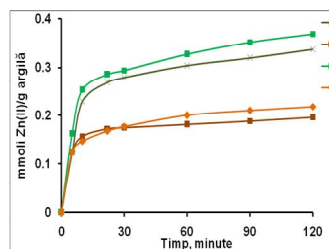


Figure 3. Zn(II) removal on natural and modified clay in relation to time

The sorption of Zn(II) from aqueous solutions on original and modified clays depends on the initial ion metal concentration, temperature and contact time. The equilibrium sorption data fitted well to the Langmuir model. The apparent thermodynamic parameters of sorption suggest an entropy-driven endothermic and exothermic sorption process. The kinetic sorption data fitted well to the second-order kinetic model, indicating an intraparticle diffusion mechanism.

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5-P-6

ASPECTS CONCERNING THE EVALUATION OF ENVIRONMENTAL IMPACT AND MANAGEMENT OF TEXTILE INDUSTRY DERIVING WASTEWATERS

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The study provides comprehensive and informative data on the main issues of environment induced by industrial technologies and processes performed by textile companies, as well as alternatives of decreasing the pollution generated by the wastewater emissions, using a rational and efficient management.

The hazardous topics deriving from wet textile processing pollutants for environment, as well as some prevention measures and monitoring of confirming in this field are approached.

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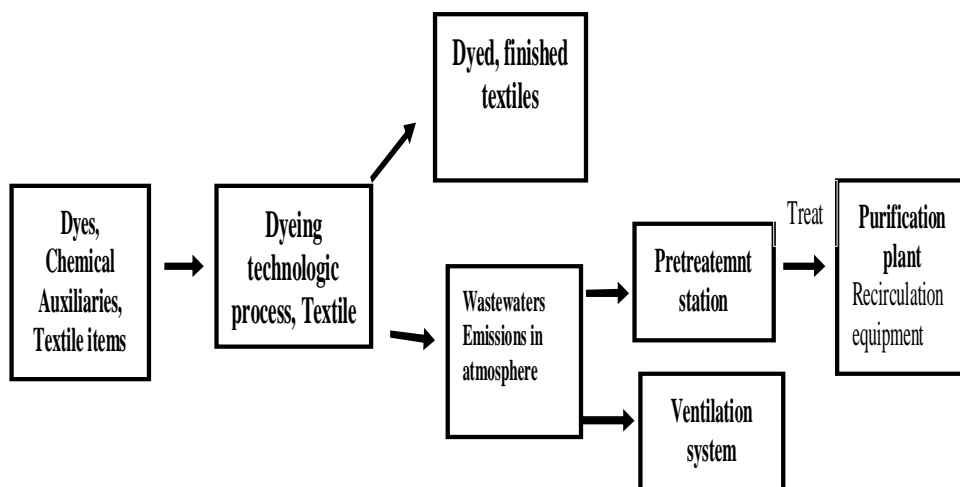
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Key-words: textile, pollution, wastewaters, chemical reagents, treatment, prevention



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5-P-7

ADSORPTION FOR METHYL ORANGE REMOVAL FROM WASTEWATER USING SAWDUST AND SAWDUST-FLY ASH SUBSTRATES

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Methyl orange is used in textile, printing, paper, manufacturing, pharmaceutical, food industries and in research laboratories. Because of its structure, this dye is rather difficult to degrade and is used as reference for advanced removal of dyes from the textile industry. Wastewater removal using second raw materials for substrates development represents a novel trend that complies with the sustainability requirements. As substrates ash, especially fly ash is investigated but attempts were done also for the use of raw wood wastes.

The potential capacity of sawdust as adsorbent the methyloange (MO) dye was tested in our work. Three types of sawdust were tested: oak wood (*Quercus robur*), white poplar (*Populus alba*) and willow (*Salix alba* L.) along with their mixtures with fly ash. The adsorption efficiency of MO was tested from 0.1mMol/L solution. We evaluate the effects of various parameters such as contact time, initial dye concentration, pH. The equilibrium data were modeled using the Langmuir and Freundlich isotherms and the adsorption mechanisms were discussed, correlated with the substrates structure and morphology. Kinetic studies showed that the adsorption followed a pseudo-second order kinetic model.

Keywords: biosorption, dye removal, sawdust, wastewater treatment

5-P-8

INVESTIGATION OF PHENOLIC COMPOUNDS ADSORPTION ON SBA-15 USING A HPLC METHOD

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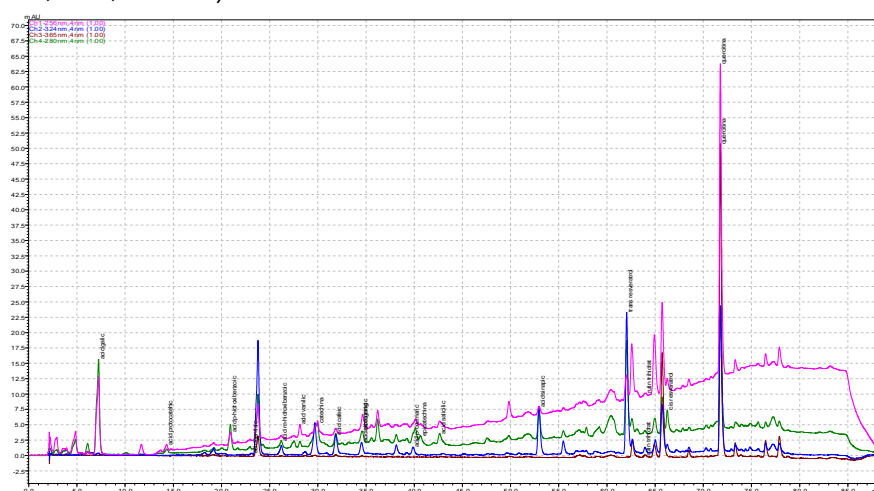
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The aim of this research was to investigate the use of the mesoporous silica material SBA-15 as adsorbent for phenolic compounds from wine, material not yet employed in oenology.

The purely siliceous molecular sieve SBA-15 was synthesized hydrothermally in acidic media via a hydrogen bonding (S^0H^+) (X^-I^+) templating route according to a procedure reported earlier, with some modifications. The typical gel molar composition was 1TEOS:0,017P123:5,95HCl:194H₂O.

HPLC method has been used in order to separate and quantify in a very short time a number of 19 phenolic compounds [1]. The results show that SBA-15 is an adsorbent for monomeric phenolic compounds. Quercitine is the phenolic compound adsorbed in the largest quantity.

Fig. 1. Chromatograms of red wine sample (at four different wavelengths: 256, 324, 365,280 nm)



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5-P-9

USE OF DI-(2-ETHYL HEXYL) PHOSPHORIC ACID (DEHPA) IMPREGNATED XAD-7 COPOLYMER RESIN FOR THE SEPARATION OF CHROMIUM (III) FROM WATER

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The paper presents a novel support for Cr(III) sorption and its removal from wastewaters. The support is new solvent-impregnated resins (SIR) and can be considered as alternative adsorbent materials since they are similarly capable of selective sorption. SIRs have been formed by impregnating Amberlite XAD-7 with DEHPA as extractant and ethylic alcohol as solvent by dry impregnation method. Adsorption on the XAD-7 support macro and interaction between the extractant and support has been emphasized by physical chemical methods of analysis (FTIR spectroscopy, EDAX). In order to establish the sorption performance of the impregnated XAD-7, the influence of different physical chemical parameters (pH, contact time and initial concentration of Cr) upon the Cr(III) sorption onto XAD-7 was investigated. The optimum pH for Cr(III) sorption was found 3.0 for this material. To express the kinetics of chromium sorption onto XAD-7 the results were analysed using the pseudo-first order and pseudo-second order models. The sorption process is best described by the pseudo-second order kinetic model. Langmuir and Freundlich isotherm studies were conducted in order to determine the maximum adsorption capacity of XAD-7 towards chromium. Langmuir adsorption isotherm gave a satisfactory fit of the equilibrium data. The maximum adsorption capacity is of ~5 mg Cr(III)/g SIR.

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5-P-10

SYNTHESIS AND CHARACTERIZATION OF POLYMETALLIC OXIDE USED IN ENVIRONMENTAL PROTECTION

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To remove the effects of pollution and to be consistent with international standards on environmental issues is necessary to obtain the materials with special properties, able to identify, retain pollutants or convert them into clean compounds. Studies performed in the past decade have shown that polymetallic oxides are materials which meet this goal.

Polymetallic oxides are materials characterized by chemical and thermal stability, used as sensors for identify and adsorb of gaseous pollutants, catalysts for total oxidation of volatile organic compounds respectively, substances with stability and high pollution risk. Catalytic properties depend on the oxidic catalyst structure, size, shape and particles distribution, parameters which are determined by the synthesis methods used to obtain oxides – sol-gel method, co-precipitation, microemulsion, unconventional methods (plasma, microwave), host template, hydrothermal treatment, mechanical chemical methods.

In this paper the results obtained following the synthesis of polymetallic oxides containing nickel ions in their structure are presented. Polymetallic oxides with spinel structure, with general formula AB_2O_4 , were $A = Ni, Zn, Mg$ and $B = Fe$ are metals with different oxidation state; they have been synthesized by sol-gel method using different fuel/complexation agents. Sol-gel method has some advantages such as high degree of homogeneity, reproducibility of results, low atomic diffusion during sintering processes, avoiding phase segregation and the possibility of obtaining nanostructured compounds that provide greater contact area and therefore high capacity to retain pollutants. Spinel-type polymetallic oxides synthesized were characterized by electronic microscopy, FTIR spectroscopy and X-ray diffraction. Catalytic activity of the synthesized compounds has been highlighted by the hydrogen peroxide decomposition tests.

The results confirm that polymetallic oxides synthesized showed catalytic properties.

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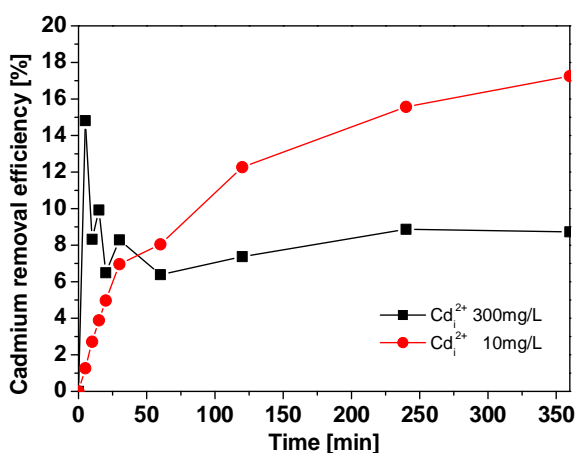
5-P-11

IMMOBILIZED FILMS OF MODIFIED FLY ASH FOR DEPURATING Cd^{2+} AND Cu^{2+} CONTAMINATED INDUSTRIAL WASTEWATER

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Heavy metals represent dangerous pollutants that are present in large quantities in the textile finishing industry. Advanced removal of heavy metals via adsorption on modified fly ash represents a sustainable solution for treating these wastewaters. Adsorption processes over powder suspension present high removal efficiencies but are less suitable for industrial implementation because of the complexity and cost of the recovering step, thus immobilizing the fly ash on a substrate represents a feasible alternative to design an up-scalable process resulting waters that comply with the discharge regulations. The study presents the results obtained on immobilized modified fly ash films in Cd^{2+} and Cu^{2+} removal (300 and 10mg/L initial concentrations), using fly ash captured from the electro-filters of the CPH plant in Braşov - România with particle size $\sim 100\mu\text{m}$ and type F according to the ASTM standards (sum of the SiO_2 , Al_2O_3 and Fe_2O_3 above 70%). AFM and XRD techniques are used to characterize the fly ash morphology and crystallinity. The cadmium removal efficiency is presented in the following figure:



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5-P-12

THE REMOVAL OF THE Zn(II) IONS FROM GROUNDWATER USING HYDROXIAPATITE NANOPARTICLES

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Due to their properties as reactivity, high surface area and the ability to be dispersed in aqueous solution, nanomaterials represent a promising application in different areas. Usually, a nanomaterial displays higher reactivity and sorption ability than the same material of normal size. Several heavy metals arising from mining wastes and industrial discharges contaminate natural waters. The increase in the use of the heavy metals over the past decades has eventually resulted in an increased flux of metallic substances in the environment. The heavy metals are of special concern because they are non-degradable and therefore persistent. Cationic metals usually have limited mobility in soil and groundwater with high clay and organic content, high alkalinity and low permeability. The complexing agents such as carbonates, hydroxides, sulphates, phosphates, which are present in natural waters, increase the solubility of metal ions [1]. A possible reaction to decrease the concentration of heavy metals in groundwater is precipitation. Other possibilities are sorption, precipitation subsequent to a chemical reduction or a combination of the different processes.

Actually, is difficult to remove heavy metals from soil and sediment and it costs a lot [2]. So, as an alternative, many researchers have stabilize heavy metals in soil or sediment using some materials to make them less mobile and bioavailable, thus reducing their ecological risk. It has been proved that this alternative is effective in the remediation of polluted waters by organic pollutants and heavy metals. Among the materials that immobilize heavy metals, such as activated carbon [3], zeolite, clay, apatite were proven effective to immobilize heavy metals during the past several years.

Because of its high sorption capacity for heavy metals, low water solubility, high stability under reducing and oxidizing conditions, availability, and low cost, hydroxyapatite became an ideal material for ion retention from solutions [4]. Therefore, the hydroxyapatite was investigated for its possible application in the removal of Zn(II) ions from aqueous solutions. Effects of contact time, amount of adsorbent and initial concentration of metal ions were studied. The possible mechanism of metal ions removal by hydroxyapatite was found to be by its adsorption or retention.

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5-P-13

PREPARATION AND CHARACTERIZATION OF NEW HYBRID LAYERED DOUBLE HYDROXIDES-PANS NANOCOMPOSITES BY IN SITU POLYMERIZATION

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The field of organic polymer-inorganic nanomaterials has attracted considerable attention for their novel or enhanced properties due to nanometer dispersion of the inorganic compound in the polymer matrix. Clays are suitable materials for the preparation of polymer nanocomposites due to their ability to be exfoliated by polymer molecules, producing a material with excellent properties, because of the high polymer/filler interfacial area. Up to now, modified cationic clays have been used to prepare polymer composites. However, recently, attention is being focused on anionic layered double hydroxides (LDHs) of the hydrotalcite type [1, 2]. Layered double hydroxides (LDHs) are interesting materials for nanocomposite formation because one can vary the identity of the metals, the anions and the stoichiometry to obtain a wide range of new multifunctional materials based on LDHs.

In the present study a new hybrid material based on poly (aniline-N-propane sulfonic acid) and Zn Al-LDH was obtained by a two-stage process with an *in situ* bulk polymerization. Sulfonate-derived aniline was preferred rather than the sulfonation on the formed polymer because the latter method gives rise generally to partially sulfonated polymer [3] and incorporation of performed polymer may be hampered by diffusion. Furthermore, the oxidative polymerization of bulk aniline sulfonate requires high pressure (19kbar) and the presence of external oxidative agent. These underlined the importance of the confinement provided by LDH structure. X-ray diffraction, SEM and UV-vis transmission spectroscopy were used to characterize the morphology of the nanocomposites, while the thermal behavior was studied by thermogravimetric analysis.

Keywords: Layered double hydroxides ,nanocomposites, polyl(aniline-N-propane sulfonic acid).

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6. NOVEL INORGANIC AND ORGANIC-INORGANIC HYBRID NANOSTRUCTURED MATERIALS

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6-P-1

NOVEL HYBRID MATERIALS BASED ON POLYURETHANE-SILICA NANOCOMPOSITES: MORPHOLOGY, ELECTRICAL AND THERMO-MECHANICAL PROPERTIES

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Organic-inorganic composites (OICs) are recognized as a fascinating class of advanced materials, that synergistically combine properties of organic polymers and inorganic materials. OICs already have a wide range of practical applications due to their unique characteristics. The most important factors are the selection of a suitable organic-inorganic component pair and the close control of the organic-inorganic structure of the composites. Changing the chemical composition of the organic and inorganic phases can modify the properties of the final compound, and, as a result, new materials with desirable properties can be obtained.

Polyurethanes (PUs) are probably the most versatile class of polymers, which makes them very attractive as matrices for OICs with superior characteristics. Their properties can be improved and material costs may be reduced by dispersing inorganic fillers into the pure PU.

Silica, one of the most important inorganic components for composites used in electric applications, is also preferred as reactive filler since its –OH groups may generate hydrogen bonds with various functional groups (–NH– or –C=O) from polymers.

In this paper, novel OICs with various structure and properties are presented. A series of novel organic–inorganic hybrid composite materials (OICs), consisting of a polyurethane organic phase (macrodiisocyanate + polyisocyanate, MDI+PIC) and a mineral inorganic phase (sodium silicate, SS) has been prepared by a modified sol-gel method. Thermo-mechanical and electric properties of these new hybrid materials were evaluated in correlation with their composition. It was found that the glass transition temperature (T_g) shifts toward higher temperature with the increase of NCO group content. All results showed that increasing the NCO group content, can affect the main properties of these novel OICs.

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6-P-2

IN VITRO BEHAVIOR OF ANTIOXIDANT COMPOUNDS INTERCALATED INTO LAYERED DOUBLE HYDROXIDES

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The layered double hydroxides (LDHs) are a class of lamellar synthetic materials. Due to their unique anion exchange capability, controlled release and protection of the payload, they can protect various biomolecules from decomposition and could act as a stable matrix for storage and drug delivery systems. [1,2] The antioxidant compounds reduce or retard free radical generation and prevent the oxidation of cellular oxidizable substrates but unfortunately, they are sensitive to light and show sensitivity to pH, which decreases their biofunctionality. To overcome this limitation, a useful approach is the linkage of these molecules on a polymeric or inorganic matrix. [3,4]. Therefore, in the present study we report the design of new composites based on ZnAlLDH-NO₃ intercalated with gallic acid (GA) and vanillic acid (VA) by coprecipitation and ion exchange methods. X-ray powder diffraction (XRD), infrared spectroscopy (FTIR) and thermogravimetric analysis (TG-DTG) confirmed that antioxidant molecules were intercalated into interlayer spaces of LDHs. The *in vitro* experiments were performed at pH=2.0 and pH=7.4, conditions similar to physiological environment and the release mechanism of the antioxidant drugs from the hybrid composites have been described following a kinetic model based on empirical equation developed by Ritger and Peppas. The diffusional exponents obtained in our study are characteristic for a non-Fickian diffusion process and the release behavior of antioxidant molecules intercalated into ZnAlLDH layers was found to be very interesting and promising for drug delivery system.

Keywords: layered double hydroxides, antioxidants, hybrid materials, controlled release

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7. NANOBIMATERIALS

7-P-1

SYNTHESIS AND ANTIMICROBIAL ACTIVITY OF ZnO/CLINOPTILOLITE AND ZnO/SBA-15

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Outbreaks of foodborne pathogens such as *Escherichia coli* continue to draw public attention to food safety. There is a need to develop new antimicrobials to ensure food safety and extend shelf life. The use of antimicrobial agents directly added to foods or through antimicrobial packaging is one effective approach. In recent years, the use of inorganic antimicrobial agents in nonfood applications has attracted interest for the control of microbes (Okouchi *et al.*, 1995; Wilczynski 2000). [1,2]

In this study, the possible use of ZnO/clinoptilolite and ZnO/SBA-15 as antimicrobial agents was investigated. We demonstrate their antibacterial activity in solid (LB agar) and liquid media (LB). For this purpose the *Escherichia coli* cells (DH5 α , New England, BioLabs) were treated at 37 °C with different concentrations of ZnO/clinoptilolite and ZnO/SBA-15.

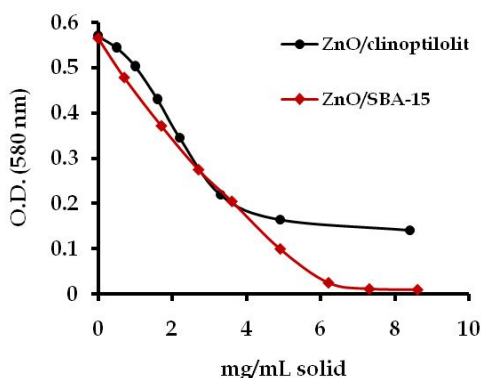


Fig. 1: The growth inhibition of *Escherichia coli* (DH5 α) by ZnO/clinoptilolite and ZnO/SBA-15

Our results clearly show that the ZnO coated zeolite supports exhibited a good inhibition effect on the growth of *Escherichia coli* (DH5 α , New England, BioLabs) and its ability was attributed mainly to the ZnO nanoparticles. Moreover, the inhibitory effects of ZnO nanoparticles were found to be concentration-dependent, related to the incubation temperature and the type of application.

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7-P-2

FUNCTIONALISATION OF Fe-Pd NANOPARTICLES FOR BIOMEDICAL APPLICATIONS

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The nanocrystalline Fe-Pd powders in 1:1 ratio were synthesized by the reduction of palladium chloride with hydrazine in the cationic water-in-oil microemulsions of water/CTAB (cetyltrimethylammonium bromide)/n-hexanol at 80 °C [1]. The functionalization of the nanoparticles has been performed with SiO₂ and polyvinyl alcohol in nitrogen atmosphere [2]. X-ray diffraction data sets have revealed a disordered face centered cubic structures. Transmission electron micrographs have indicated the presence of spherical particles with mean diameter in the range of 9-15 nm. The magnetic measurements at room temperature have demonstrated a superparamagnetic behavior of the all samples. The saturation magnetization (12-20 emu/g), remnant magnetization and the coercivity were significantly smaller than those of the bulk Fe-Pd, reflecting the nanoparticle nature. Fe-Pd functionalized nanoparticles have potential for various biomedical applications, including biological separation, protein immobilization, and biosensors [3].

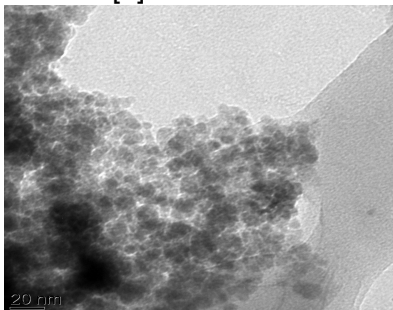


Figure 1. TEM image of the Fe-Pd/PVA functionalized nanoparticles.

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7-P-3

NANOSCALE SURFACE TREATMENTS OF PAPER SUPPORTS FOR ANTIMICROBIAL FINISHES

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Heritage documents are largely affected by the influence of environmental conditions, namely light, temperature, moisture, microorganisms. In order to prevent the damaging activity of these factors, one relatively simple and non-invasive conservation method is the treatment of the paper support with different protective substances. This study presents the investigation on the efficiency of a grafting treatment using the reactive derivative of a cyclic oligosaccharide, β -cyclodextrin (i.e. monochlorotriazinyl- β -cyclodextrin). Due to their characteristic molecular structure, cyclodextrin and their compounds have the ability of forming inclusion compounds with a wide range of guest compounds. For the present application three guests with antimicrobial protective properties were chosen: ferulic acid, Michler ketone, allantoin. After the grafting treatment, the guest compounds were loaded by spraying and by brushing their solutions on the CD-grafted substratum. The cellulosic supports that have been used were: Japanese veil, filter paper, writing paper etc.

The grafting of the reactive compound on the cellulosic paper supports was realized by a relatively simple pad-dry-cure treatment in mild conditions and the inclusion of the protective substances-by wet treatment with a guest solution. The untreated and treated supports were analysed by FT-IR-ATR spectroscopy, SEM, antimicrobial tests.

Our results have indicated that the included components are efficiently hosted in the CD cavities and the paper surface properties are significantly modified by the chemical treatment in the desired direction.

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7-P-4

MTX-MESOPOROUS MATRIX NANOCOMPOSITES FOR NANOMEDICINE VALORIZATION.

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INTRODUCTION: Arrays of nanometer-scale needles could be used to deliver drugs, proteins, and particles across skin in a minimally invasive manner.

In this paper, the feasibility of some mesoporous materials, based on SBA-15, MCM-41, LDH (Mg_3Al-NO_3) and MC (carbon mesoporous) were comparatively evaluated for oral drug delivery applications. Methotrexate (MTX) is an anticancer drug, employed in the therapy of solid tumors and leukemia. Intercalation reactions are particularly important as they can produce big changes in the chemical, electronic, optical, and magnetic properties of a host matrix. The nanocomposite systems have remarkable intercalation properties.

METHODS: In all experiments, the drug loading was carried out by impregnation method. The loading of methotrexate molecules was found to be influenced by the initial synthetic procedure. The drug release in synthetic stomach media (PBS), compared with the release from the pharmaceutical formulations, proved that the methotrexate encapsulated-LDH, SBA-15, MCM-41 and MC can be considered efficient controlled drug delivery systems. The obtained materials were analyzed by N_2 sorption, UV-Vis DR, TOC/TN and FTIR Spectroscopy.

Sample	S, m^2/g	Vp, cc/g	Pore diam, nm	mg of MTX/g
LDH	60.33	0.01	5.33	-
LDH- MTX	42.52	0.06	5.32	339
SBA-15	794.5	1.07	6.84	-
SBA-15- MTX	466.3	0.68	6.65	348
MCM-41	1310.1	0.31	3.30	-
MCM-41- MTX	583.6	0.27	2.38	349
CM	1122.6	1.15	3.51	-
CM- MTX	964.3	1.14	3.28	365

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RESULTS:

In the attached table, textural properties and adsorbed quantities of methotrexat calculated from TOC results are presented.

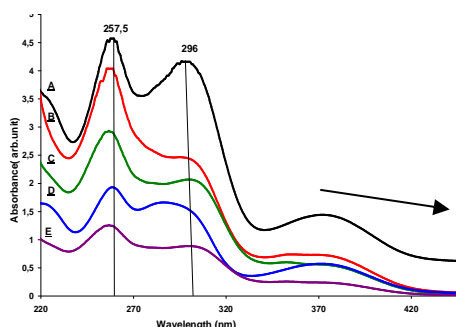


Fig.1. UV-Vis absorption spectra for the obtained nanocomposite systems, A. MTX-commercial drug, B. SBA-15-MTX, C. MCM-41-MTX, D. LDH-MTX, E. MC-MTX

CONCLUSIONS:

From UV-Vis absorption these results also confirm that drug molecules are stabilized by electrostatic interactions due to the positively charged nanostructures systems. The adsorption capacity of SBA-15 and MCM-41 for MTX, also studied in this work, was significantly higher than that achieved with LDH and MC.

The aim of the study was to determine the feasibility of obtained nanocomposites application as intercalated materials storage and transport, and mainly as drug delivery systems. We concluded that the nanocomposite systems (LDH, SBA-15, MCM-41 and MC) can be used as an excellent inorganic carrier for an advanced biocompatible drug delivery system.

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8. OTHER ASPECTS OF NANOMATERIALS SCIENCE AND TECHNOLOGY

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8-P-1

PREPARATION OF BiFeO₃ – BASED MULTIFERROIC NANOSTRUCTURES BY HYDROTHERMAL SYNTHESIS

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BiFeO₃ (BFO) is one of the few single-phase magnetoelectric multiferroics at room temperature and one of the most studied and controversial systems in the last years [1]. Recently, researchers have attempted to prepare phase pure BFO via a variety of routes [2]. In the present work, different BiFeO₃ – based multiferroic nanopowders with particular geometries and microstructural characteristics obtained by hydrothermal synthesis were investigated. The influence of the processing parameters (NaOH concentration, reaction temperature and reaction duration time) on the phase formation and morphology are presented. Single phase perovskite BiFeO₃ with small amounts of secondary phases has been formed using 6M NaOH at

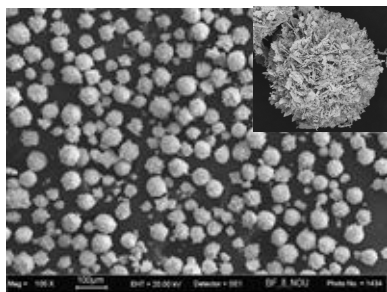


Fig.1. SEM images of the BiFeO₃ powders.

a temperature of 180°C in 48h reaction period. With increasing of reaction temperature, impurity phases were detected in addition to the major phases BFO, for example: Bi₂O₃, Bi₂Fe₄O₉ and some unknown phases. The powders exhibit hierarchical microstructures (Fig.1) constituted of spherical larger aggregates of around 70-80μm diameter formed by platelets crystalline particles.

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ACKNOWLEDGEMENTS

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8-P-2

IMPACT OF A CATION SIZE ON THE STRUCTURE AND PROPERTIES IN THE DOUBLE PEROVSKITE A_2AlTaO_6

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The crystal structure of A_2AlTaO_6 perovskites depends upon the size of the A- or B-site cations. In this paper we purpose to study the influence of the A cation size in the crystal structure and properties on the double perovskites A_2AlTaO_6 (A = Ca, Sr, Ba).

Polycrystalline samples A_2AlTaO_6 (A = Ca, Sr, Ba) were obtained by conventional solid state reaction, from a stoichiometric mixture of ACO_3 (A = Sr, Ba), CaO, Al_2O_3 and Ta_2O_5 . The resulting powders were sintered at 1300°C /48h.

X-ray diffraction measurements at room temperature, used to investigate the purity of the perovskite powders were performed with a SHIMADZU XRD 6000 diffractometer using Ni-filtered $CuK\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). Note that with increasing ionic radius in the series $Ca < Sr < Ba$, tolerance factor and the symmetry of the crystalline cell increases, passing from monoclinic cell to cubic cell.

FT-IR transmission spectra were obtained using a JASCO 660 PLUS spectrophotometer used to complete the structure studies. All the spectra present the typical two band pattern characteristic of the perovskite structure: the strong high energy band centered at about 660 cm^{-1} can be assigned to the asymmetric stretching mode of TaO_6 octahedral and a band at 840 cm^{-1} which can be assigned to the symmetric stretching vibration of these octahedral and the strong IR-band at around 450 cm^{-1} which can be assigned to the $Ta(Al)O_6$ deformation.

The complex impedance at room temperature was determined by using an impedance bridge type Agilent E4980A. Size of A cation has influenced the dielectric properties : compound with the distorted structure (A = Ca) has low dielectric losses and low relative permittivity and the compound with the largest cation (A = Ba) has high dielectric losses and high relative permittivity. For the Sr_2AlTaO_6 are the best dielectric properties.

We investigated the catalytic properties of A_2AlTaO_6 perovskites in water splitting process, under gamma-rays irradiation emitted by a ^{60}Co source. It was noticed that the double perovskite Ca_2AlTaO_6 had a higher catalytic effect comparing with Ba_2AlTaO_6 and Sr_2AlTaO_6 . A smaller ionic radius will determine a higher number of anionic vacancies.

8-P-3

SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE
Ni-Zn FERRITE DOPED WITH DY

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Extensive research has focused on investigating the effect of cation doping on the spinel ferrite lattice. Magnetic properties could be improved by adding small quantities of rare earth ions into Ni-Zn ferrite lattice.

The rare earth ions were chosen because of their electronic configuration with unpaired 4f electrons and also because of their strong spin-orbit coupling of the angular momentum. Doping rare earth ions into spinel-type ferrites, the occurrence of 4f-3d couplings which determine the magnetic anisotropy in ferrites can also improve the electrical and magnetic properties of Ni-Zn ferrites. Dy³⁺ doped Ni-Zn ferrites with the general formula Ni_{0.8}Zn_{0.2}Fe_{2-x}Dy_xO₄ (x = 0; 0.02) were prepared by the sol-gel auto-combustion method using tartaric acid. The crystal structure and surface morphology of the calcined samples at 973 K and 1173 K were characterized by infrared spectroscopy (IR), X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). The particle size lied in the nanometric range (27 ÷ 58 nm) and the particles have spherical agglomerated shape.

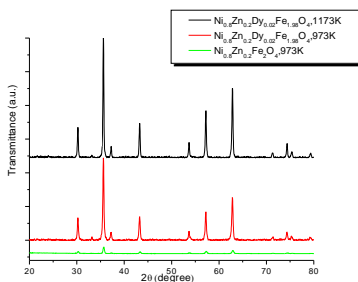


Fig.1.XRD results

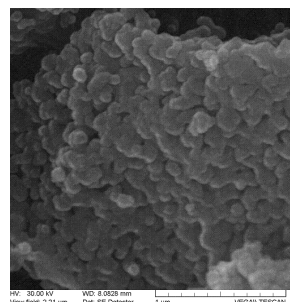


Fig.2. SEM result for
Ni_{0.8}Zn_{0.2}Fe_{1.98}Dy_{0.02}O₄ calcined at
973K

The magnetic properties of the samples were tested by a vibrating sample magnetometer (VSM) at room temperature.

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8-P-4

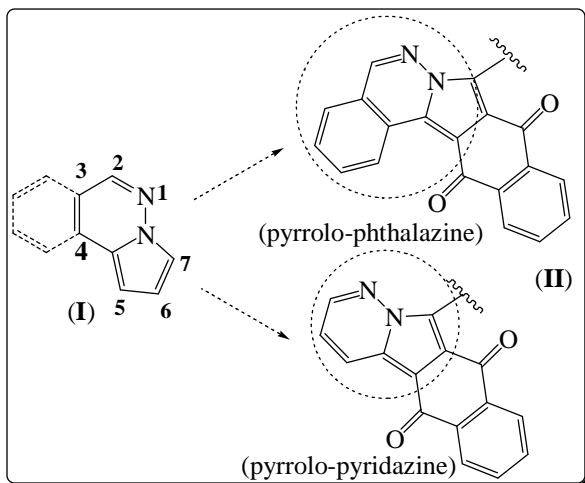
UV-VIS STUDIES OF NEW PYRROLO-DIAZINIC COMPOUNDS WITH FLUORESCENT PROPERTIES

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Highly fluorescent derivatives with extended π -conjugation continues to be an interesting field in the research area¹, especially to their applications assensors and biosensors, electroluminescent materials, lasers, and other optoelectronic devices.

Considering the pyrrolo-pyridazine moiety (I) responsible for blue fluorescent properties, we rationalize the syntheses in order to extend the π -conjugation by introducing in 3,4-position a benzene ring and in 5,6-position a naphthoquinone moiety. Also we were interested to study the influence of introducing different substituents in the 7-position (ester, amide, ketone).



In order to synthesize the desired compounds with pyrrolo-phthalazine and pyrrolo-pyridazine moiety (II) we adopted a general and straightforward strategy, involving two steps only: quaternization and cycloaddition. These reactions were performed under ultrasound and classical conditions². UV-VIS spectra were recorded with a Shimadzu UV-1800 spectrometer in acetonitrile.

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NOVEL FORMULATIONS FOR COMPATIBILIZED POLYMERIC BLENDS BASED ON RECYCLED PE AND PA. STUDY ON THE COMPOSITION-PROPERTIES RELATIONSHIP

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Nowadays, because most of the synthetic polymers are derived from non-renewable fossil resources, for keeping in step with societal concerns on energy crisis and ecological problems, reduction in the usage of fossil-based materials becomes more and more important. Some cardinal benefits of polymer recycling are the reduction of waste generation, less need for landfills and a reduction in consumption of resources [1]. Therefore, polymer wastes are a tremendous, although relatively little studied, source of raw materials for industry.

Block copolymers (BCPs) as compatibilizers are valuable components because, due to a wise choice of components and method of synthesis, offer the possibility to tailor the properties of the final material.

Our research is focused on the possibility to upcycle some polymer waste and obtain new composite materials with tailored properties, to identify new potential applications or to expand the limits of the classic ones. This paper presents the preparation and characterization of new polymeric materials obtained using polymeric wastes, recycled polyethylene (RPE) from agriculture foils and polyamide fibers (PA) recovered from scrap automobile tires, and two novel polyamide–dienic rubber block copolymers (AB and ABA) as compatibilizers. The main purpose of this paper is the comprehension of the effect of the AB and ABA content and structure on the morphology and properties of these blends.

Another factor affecting the properties of these new materials is the amorphous/crystalline ratio. The increase of amorphous structures in the blends structure favors their impact strength, but it has an unfavorable effect on the tensile strength.

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8-P-6

THE EFFECT OF POSTANNEALING ON THE PHOTOLUMINESCENCE RESPONSE AND PIEZOELECTRIC PROPERTIES OF ZnO FILMS PREPARED BY PULSED MAGNETRON SPUTTERING

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In the recent years, there has been increased interest in zinc oxide (ZnO) in terms of potential applications as piezoelectric films for surface acoustic wave devices, gas sensor and transparent conducting electrodes of optoelectronic devices, for light emitting devices and UV sensing.

Zinc oxide thin films were deposited on silicon and quartz substrates by high power impulse magnetron sputtering system. Thermal annealing was performed at up to 800 °C in nitrogen atmosphere for 20 min. The effect of annealing on the crystal quality, photoluminescence response and piezoelectric properties was investigated using X-ray diffraction, photoluminescence (PL) spectra, atomic force microscopy and piezo force microscopy (PFM). The photoluminescence response improved considerably during annealing, while the piezoelectric constant becomes lower at high annealing temperature. The sputtered ZnO thin film has been optimized for both piezoelectric properties and UV sensing ability. A film with highly piezoelectric properties as well as good photoluminescence response was obtained in order to fabricate UV sensor based on surface acoustic wave (SAW) device.

8-P-7

TRANSPORT PHENOMENA IN $\text{La}_{0.54}\text{Ho}_{0.11}\text{Sr}_{0.35}\text{Mn}_{1-x}\text{Cu}_x\text{O}_3$
MANGANITES

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The colossal magnetoresistance (CMR), which was observed in perovskite manganites, involves, besides the double exchange mechanism, electron-phonon interactions.

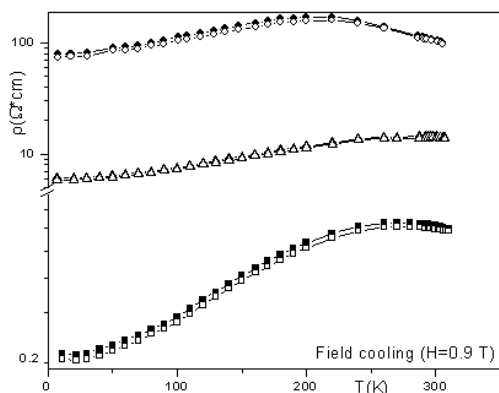


Figure 1. Variation of resistivity with temperature, magnetic field and Cu concentration (x) in (LHMCO) manganites (for $H=0$, $x = 0.03$ (■), 0.06 (●) and 0.15 (▲); for $H=0.9$ T, $x = 0.03$ (□), 0.06 (○) and 0.15 (Δ); field cooling).

The sizes of magnetic clusters have been determined at room temperature. The variation of the specific/molecular magnetization with temperature was determined by using a Foner-type magnetometer with a data acquisition system, between 77 and 350 K. The electric measurements were made between 7 and 330 K at $H_{\max}=0.9$ T using a four-probe method and a data acquisition system. The substitution of the Mn cation with Cu leads to a nonmonotonous variation of the resistivity and magnetoresistance (s. Fig.1). Small Cu concentration in the $\text{La}_{0.54}\text{Ho}_{0.11}\text{Sr}_{0.35}\text{Mn}_{1-x}\text{Cu}_x\text{O}_3$ manganites contributes to an important value of the magnetoresistance near room temperature. This behaviour was attributed to the presence of the small nanomagnetic domains, present in the sample near room temperature.

The $\text{La}_{0.54}\text{Ho}_{0.11}\text{Sr}_{0.35}\text{Mn}_{1-x}\text{Cu}_x\text{O}_3$ (LHMCO) manganites were obtained by sol-gel method using oxides and acetates and sintered in air at 1200°C for 15 h. Chemical composition was obtained by means of EDX method. The samples were investigated by X-ray diffraction using a HUBER diffractometer with GUINIER CAMERA 670 with a $\text{CuK}\alpha_1$ radiation, magnetic and electric methods. SANS measurement were performed on the SANS-1 instrument at the FRG-1 reactor. The samples contain only a perovskite phase, with orthorhombic structure (S. G. 62 – Pnma).

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FLUORESCENT COMPOUNDS WITH DIAZINIC SKELETON

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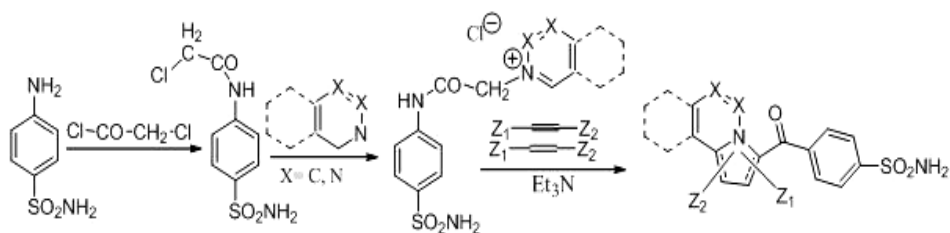
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The antimicrobial, diuretic and anticancerous activity of sulfonamides, as well as the antimicrobial and anticancerous activity of quaternary cycloimmonium salts and fused diazine are well known. The emphasis of this work was to synthesise a new class of sulfonamides with diazinic skeleton of interest as antimicrobial and anticancerous derivatives, which also possess fluorescent properties. The fluorescent ability of the new compounds, which appears as a result of an extended π -conjugation, might also find application as biosensors, electroluminescent materials, lasers or optoelectronics devices. The reaction pathway is generally, straightforward, efficient and general and consists in three steps: acylation of sulfonamide, quaternization of diazine and 1,3-dipolar cycloaddition of ylides to various dipolarophiles. The structure of compounds was proved through elemental and spectral analysis.



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8-P-9

THE STRUCTURAL, ELECTRICAL AND MAGNETIC PROPERTIES OF Ni-Mg FERRITES

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Nanosized spinel ferrite particles have attracted considerable attention and continued efforts to investigate them for their technological importance to the microwave industries, high speed digital tap or disk recording, repulsive suspension for use in levitated railway systems, ferrofluids, catalysis and magnetic refrigeration systems. Among different ferrites, magnesium ferrite $MgFe_2O_4$ enjoys a special attention because of its vast applications in high-density recording media, heterogeneous catalysis, adsorption, sensors and magnetic technologies. The nickel ferrites $NiFe_2O_4$ are one of the most important ferrites with reversed spinel structure having ferromagnetic properties. This material is widely used in electric and electronic devices and in catalysis.

The purpose for this work was the study of the structural, magnetic and dielectric properties of the nickel ferrites substituted by magnesium: $Ni_{1-x}Mg_xFe_2O_4$ with compositions $x=0; 0.17; 0.34; 0.5; 0.66; 0.83; 1$. The materials have been prepared by sol-gel autocombustion method using metal nitrate salts as a cation precursors and citric acid as combustion/chelating agents.

The FTIR spectra were recorded in the range $400-1000\text{ cm}^{-1}$. The spectra show two absorption bands ν_1 and ν_2 , corresponding to the stretching vibration of the tetrahedral and octahedral sites around 600 and 400 cm^{-1} , respectively. The powder X-ray diffraction pattern confirms the spinel structure for the synthesized compound. The crystallite size diminishes when increasing the Mg substitution from $\sim 71\text{ nm}$ for $x=0$ to $\sim 43\text{ nm}$ for $x=1$. The lattice parameter increases with the increase in Mg concentration and this may be attributed due to the larger ionic radius 0.72 \AA of Mg^{2+} than that of the Ni^{2+} ion 0.69 \AA . The electrical and magnetic properties at room temperature were also studied.

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8-P-10

SOL-GEL SYNTHESIS AND CHARACTERIZATION OF
Ba₂Co₉O₁₄ FOR SOFCs APPLICATION

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The purpose of the present work is to obtain powders of new cobalt-based oxide of type Ba₂Co₉O₁₄. The first crystal of Ba₂Co₉O₁₄ had obtained by solid state reaction [1]. Its original structure is close to those of ferrite, but mostly it is the term n = 1 of a new family compounds Ba_nCo_nO_{3n+3}(Co₈O₈). The discovery of the new Ba₂Co₉O₁₄ is particularly interesting from the structural point of view and its possibility to be used as anode for SOFC cells.

The new mixed oxide Ba₂Co₉O₁₄ was prepared by derived sol-gel method using nitrates as precursors and citric acid and ethylene glycol as chelating agent. The obtained powder was annealed at different temperatures (750 to 900 °C) for 4 h in air atmosphere. Chemical composition was obtained by means of Energy Dispersive X-ray method (EDX). The structure of sintered samples was also investigated by means of scanning electron microscopy (SEM), FT-IR Spectroscopy and X- ray Diffraction (XRD) analysis.

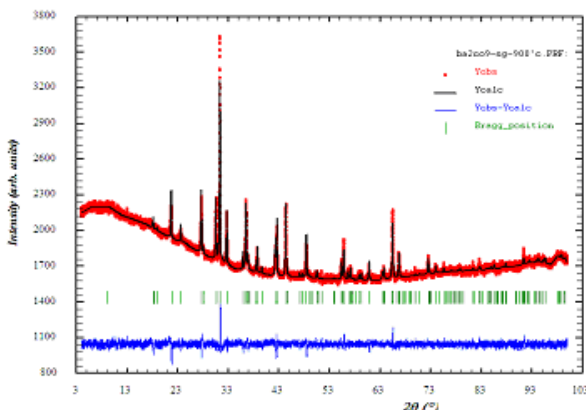


Figure 1. XRD powders of Ba₂Co₉O₁₄ (calculated and observed), annealed at 900 °C.

The results of X-ray diffraction indicated that Ba₂Co₉O₁₄ begun to form at 800 °C and had a hexagonal structure (space group: R-3m, parameters a = 5.6964 Å, c = 28.923 Å), in according with literature data [1] (fig.1). The results of SEM analysis show that particle sizes are between 0.1-2 μm. Distribution of crystallites size and existence of cobalt cations in multiples oxidation state are qualitatively confirmed by the FT-IR spectra.

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8-P-11

ON MAGNETIC/CRYSTALLINE STRUCTURE OF
 $\text{La}_{0.54}(\text{Sm}/\text{Nd})_{0.11}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{Cu}_x\text{O}_3$ NANOMANGANITES

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$(\text{La},\text{Re})_{1-x}\text{Alk}_x\text{MnO}_3$ manganites, where Re is a trivalent rare-earth ion and Alk - a divalent alkaline-earth metal, have interesting structural, magnetic and transport properties for $x \approx 0.35$. Substitution of Mn with other transition cations produces a change of Mn-O-Mn interactions, followed by corresponding change in the magnetic and transport properties. Our aim is to investigate the modifications induced by the Mn substitution with Cu and of the La with Sm or Nd on the crystalline and electronic state of the $\text{La}_{0.54}\text{Re}_{0.11}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{Cu}_x\text{O}_3$ nanomanganites. The compounds were prepared as nanometric powders using a sol-gel method. All synthesized compounds contain only perovskite phases, the volume of the lattice cell increase with the increase of the size of the transitional metal in the samples.

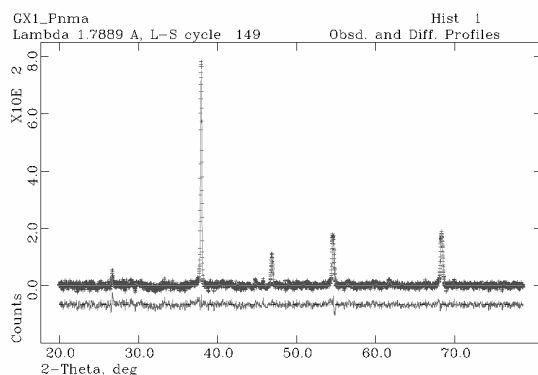


Figure 1. Diffractogram of LNCMCO ($x=0.03$), manganite (GSAS method)

manganites doped with Nd can be explained by the presence of a mixture of ferromagnetic metallic and an antiferromagnetic (ferrimagnetic) insulator phases. The presence of Cu on the B sites destroys the DE interaction. Cu cation enhances the superexchange interaction and the appearance of a larger amount of insulator phase. Charge transport takes place by a small polaron hopping mechanism. Activation energy of charge transport decrease with about two magnitude order in Nd as comparing with Sm substituted manganites.

Crystalline structure, implicitly Mn-O distances, Mn-O-Mn bonds, average size of the mosaic blocks and microdistortions, are strongly influenced by the substitution of La with Sm or Nd. Maximum of the resistivity decrease with the increase of Cu concentration in the samples. The SPH process can describe the behavior of the samples at temperatures around Curie temperature. The magnetoresistance behavior of

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OPTICAL CHARACTERIZATION OF DC MAGNETRON SPUTTERED TiO₂:N THIN FILMS

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Titanium dioxide (TiO₂) has attracted the interest of many researchers due to its exceptional properties like high refractive index, low absorption coefficient, wide band gap, chemical stability and long time corrosion resistance. TiO₂ thin films have been studied for numerous optical applications, like photocatalysis, solar cells etc. Different growth techniques have been used to prepare films of this class of materials, such as PVD, laser ablation, thermal evaporation, spin/dip coating, or various chemically - assisted methods. Magnetron sputtering allows the preparation of densely packed, homogenous and smooth-surface films.

We propose here a new approach to synthesize nitrogen-doped TiO₂ (TiO₂:N) thin films of controllable composition by using a sintered TiN target in a reactive DC magnetron sputtering discharge running in an Ar-O₂ gas mixture. Films with different dopant concentrations were prepared by varying the mass flow rate (MFR) of oxygen in the discharge.

Results reported in this contribution are aiming the characterization of ~ 300 nm thick TiO₂:N thin films by UV-VIS spectrometry and ellipsometry. The band gap of N-doped TiO₂ was found to be about 3.20 eV. The values of the refractive index ranged between 2.12 - 2.36, as determined from the transmittance spectra. The dependence of the ellipsometric parameters (Δ , Ψ) of the incidence angle has been determined by ellipsometry. The results have been interpreted in relation with crystallographic data (X-ray diffraction patterns exhibited diffraction peaks at 25.3°, 38.6° and 55.1° indicating TiO₂ in anatase phase) and elemental composition (derived from the X-ray photoelectron spectroscopy measurements). The highest concentration of nitrogen (~ 4.6 at. %) in the films was obtained for an O₂ MFR of 0.7 sccm. The statistics of the atomic force microscopy data showed typical RMS surface roughness ranging between 3 - 5 nm.

The DC magnetron sputtering discharge used in the described configuration proved to be a versatile technique that allowed the deposition of TiO₂:N thin films with remarkable optical properties, crystalline structure, homogeneous and smooth surface, and high range controllable dopant concentrations.

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THE INFLUENCE OF SUBSTRATE TEMPERATURE AND POST DEPOSITION TREATMENT ON THE PROPERTIES OF SPRAYED In_2S_3 THIN FILMS

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Thin film solar cells based on solid state semiconductors are key elements in the development of non-silicon photovoltaics. Recently developed solar cells using Cu(In,Ga)Se,S absorber layers have reached efficiencies comparable to silicon based cells with the use of cheaper materials and fabrication techniques.

However the use of highly toxic Cd based intermediate layers such as CdS raises a set of environmental problems which need to be addressed. The intermediate layers have to allow for the electrical conduction and the transmission of solar photons between the window and the absorber layers. These requirements can be achieved by thin films of cadmium-free materials such as ZnS, ZnSe and In_2S_3 .

The aim of the paper is to study the effect of the substrate temperature and post-deposition heat treatment on the electrical, optical and structural properties of In_2S_3 thin films deposited on FTO substrates using spray pyrolysis.

X-ray diffraction and Atomic Force Microscopy were used to investigate the structural and morphological properties of the deposited films; electrical and optical properties have been studied by current-voltage measurements and UV-VIS spectroscopy.

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PREPARATION OF NiFe₂O₄ ON Nb DOPED PZT-BASED TEMPLATES FOR OBTAINING *IN-SITU* MULTIFERROIC COMPOSITES

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In the last years, artificial multiferroic structures formed by ferroelectric and magnetic components have been prepared, in the view of obtaining magnetoelectrics, by exploiting their piezoelectric and magnetostrictive characteristics, according to the principle of “product property” [1]. By simple mixing the two phases, no satisfactory magnetoelectric coupling was reported for the large majority of ceramic composites [2]. In order to ensure a better connectivity of dissimilar phases, more innovative methods for preparation in-situ composites by chemical methods, followed by adequate sintering procedure is needed. In the present paper, Nb-doped PbZr_{0.52}Ti_{0.48}O₃ (PZT) and NiFe₂O₄ (NF) were chosen, due to their excellent piezoelectric and piezomagnetic properties, respectively.

Nb-doped PZT powders were firstly prepared by mixed oxides method. The PZTN powders were used as templates for growing the ferrites obtained by citrate method. After gelation the samples were dried at 150°C and calcined at 300°C. Finally the samples were sintered at 1200°C for 1h.

Only the peaks characteristics to the perovskite PZTN and spinel NF were detected by XRD analysis, demonstrating the synthesis of pure ferrite phase directly on the ferroelectric templates. An excellent mixing was obtained in the composite powders, as proved by a detailed SEM-EDX analysis. The sintering parameters were optimized in order to obtain a high density and to avoid the formation of secondary phases at interfaces. The dielectric, piezoelectric and magnetic properties are strongly dependent on composition, microstructures (degree of phase interconnectivity). By comparison with other preparation methods reported in literature, the in-situ preparation of nanocomposites leads to a much better local homogeneity and the highest dielectric quality of the composite samples.

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8-P-15

STRUCTURAL PROPERTIES OF CuInGa-ZnS HETEROSTRUCTURE DEPOSITED ONTO POLYAMIDE SUBSTRATE

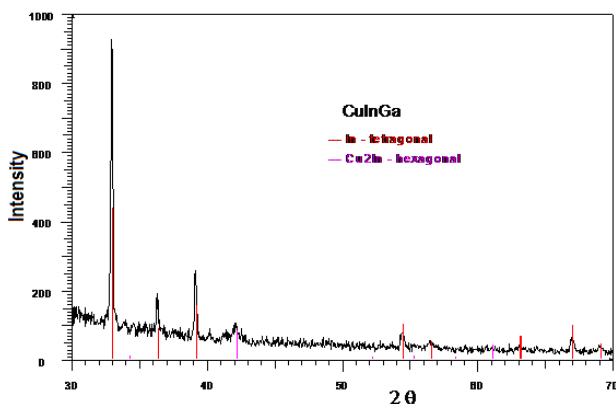
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The heterostructure is realized by successive deposition of copper-indium-gallium (CuInGa target) and zinc sulphide (ZnS target) thin films onto polyamide (e.g. kapton) substrate, using the rf magnetron sputtering technique. At the same time individual deposition of each component element of the heterostructure was realized onto the same flexible kapton substrate. The 125 μm thick kapton substrate is stable with temperature, as demonstrated by the vitreous transition point T_g at 202°C. From atomic force microscopy studies resulted that the heterostructure deposited onto such a substrate shows a high porosity with roughness values of 300-350 nm. The CuInGa control sample (Figure) exhibits two crystalline structures, In tetragonal and Cu_2In hexagonal. Even though gallium cannot be seen separately, it may be present in the indium structure. Both In and Ga have tetragonal structures with the same symmetry. In the spectrum of the sample, In XRD peaks seem to be shifted towards smaller angles, corresponding to bigger interplanar distances. It is possible that Ga entered the In lattice.



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8-P-16

BEHAVIOR OF POLYIMIDE THIN FILMS UPON LASER IRRADIATION

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Laser ablation of polymers is a routine part in micro/nanoelectronic packaging and fabrication of devices. Polyimides possess outstanding key properties, such as thermo-oxidative stability, high mechanical strength, high modulus, excellent electrical and optical properties and superior chemical resistance. Here, we present a study of thin films made from new fluorinated polyimides containing oxadiazole and naphthyl rings upon irradiation with UV laser and the investigation of their morphology before and after laser ablation. Thin films were prepared by casting polyimide solutions onto glass plates, followed by gradual heating up to 210°C. Static ablation of polyimide films was performed by using a COMPexPRO (Coherent) excimer laser operating at 248 nm (KrF). The experiments were performed after 30 pulses at laser fluence of 57 and 240 mJ/cm². The quality of films before and after ablation was investigated by atomic force microscopy (AFM), time-resolved emission, FTIR and X-photoelectron spectroscopy, and contact angle measurements.

FTIR spectroscopy evidenced a decomposition of imide rings under laser radiation and accumulation of carbonaceous species in/and around the irradiated area. The absorption bands characteristic to oxadiazole rings remained unchanged after irradiation. AFM showed that the film surface changed from a smooth morphology to one having cone-like structure: at low laser fluence, of 57 mJ/cm², the film surface looked like a densely packed cone-shaped long periodic structure; at higher laser fluence, of 240 mJ/cm², the number of cones decreased while their height and base diameter increased. The formation of cone structure at low laser fluence is explained by the presence of opaque impurities which shade the material underneath from laser radiation, while the surrounding polyimide molecules self-organize under UV-laser radiation and lead to the formation of long periodic structure. The long periodic structure was confirmed by time-resolved spectroscopy which revealed the presence of impurities like calcium and phosphorous atomic species. The random distribution of cones and the increase of their height and base diameter at higher laser fluence (240 mJ/cm²) is explained by the formation of a carbon layer on the cone surface and by ablation of impurities which made the density of cones lower.

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PODE Rodica	OP-2	27
	OP-6	34
	OP-8	36
	OP-12	42
	4-P-1	74
POP A.	OP-6	34
POPA A.	5-P-9	90
POPA Gheorghe	8-P-6	113
POPA Ionel Marcel	OP-9	37
	6-P-2	99
POPOVICI Evelini	IL-4	15
	OP-4	30
	OP-15	45
	OP-16	46
	1-P-5	57
	3-P-1	63
	3-P-7	71
	4-P-1	74
	5-P-3	81
	5-P-4	83
5-P-8	89	
7-P-4	104	
POPOVICI Ionut	8-P-13	120
POPOVICI Roxana F	OP-11	40
POSTOLACHE Petronel	OP-10	38
PREPELITA Petronela	8-P-15	122
PRISECARU (MIHAI) Gina D.	OP-5	32
	1-P-5	57
PSARO Rinaldo	OP-3	28
PUICA Melniciuc N.	5-P-10	91
	8-P-9	116
RUSU Dan Radu	8-P-16	123
REINOSO Francisco R.	OP-3	28
REMES A.	OP-2	27
ROLLE Aurelie	8-P-10	117

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ROTINBERG P.	OP-11	40
SAVVAJIS K.	IL-8	20
SAMOILA Petrisor Mugurel	1-P-4	55
SANDU Andrei V.	4-P-3	77
SANDU Ion	OP-4	30
SEFTEL Elena Mihaela	OP-15	45
	1-P-5	57
	3-P-1	63
	5-P-8	89
SHIRIYAZDANOV Rail.R.	OP-7	35
	OP-13	43
SILION Mihaela	6-P-2	99
SIRGHI Lucel	8-P-6	113
SLATINEANU Tamara	5-P-10	91
	8-P-3	109
SMOLYANSKII A.S	IL-9	22
SOROKIN Yu. A.	IL-9	22
SPETZ Anita Lloyd	IL-2	12
	OP-5	32
SUNEL Valeriu	OP-9	37
SUPURAN Claudiu	8-P-8	115
SUCHEA Mirela	IL-8	20
STURZA Mihai	8-P-10	117
TABACARU Claudia	1-P3	54
	4-P-3	77
TANASA Fulga	3-P-2	64
	6-P-1	98
	8-P-5	112
TANASA Diana Ecaterina	3-P-7	71
TANCRET Nathalie	8-P10	117
TIRON V	8-P-6	113
TUDOSE I.V.	IL-8	20

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TUDORACHE Florin	1-P-1	51
TRUTA E.	OP-11	40
TUPIKOV V.I	IL-9	22
VANSANT Etienne F.	IL-1	9
	OP-15	45
VASILE Aurelia	IL-5	17
	5-P-1	79
	5-P-2	80
	5-P-8	89
VERNARDOU D.	IL-8	20
VERNIMMEN Jarian	OP-3	28
VISA Maria	5-P-11	93
VOICHITA G	OP-11	40
VOICU V.A	OP-11	40
VRINCEANU Narcisa	OP-4	30
	4-P-1	74
	5-P-4	83
	5-P-6	86
YIAKIMOVA Rositza	IL-2	12
ZANOAGA Madalina	3-P-2	64
	6-P-1	98
	8-P-5	112
ZBANCIOC Gheorghita	8-P-4	111
	8-P-8	115
ZHU H.Y.	OP-15	45
URSACHI Irina	5-P-1	79

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