# ACTIVITY REPORT 2006 and ACTIVITY PLAN 2007



# ACTIVITY REPORT 2006 and ACTIVITY PLAN 2007

ASSOCIATED LABORATORY

CENTRE FOR RESEARCH IN CERAMICS AND COMPOSITE MATERIALS

**Director:** Professor Doctor João Carlos Matias Celestino Gomes da Rocha

Vice-Director: Professor Doctor Joaquim Manuel Vieira

Universidade de Aveiro Campus Universitário de Santiago 3810-193 Aveiro Portugal 
 Phone: + 351 234 372 571

 Fax: + 351 234 370 004

 @: ciceco@ua.pt

 URL: <u>http://www.ciceco.ua.pt</u>

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# **INTRODUCTION**

The CENTRE FOR RESEARCH IN CERAMICS AND COMPOSITE MATERIALS (**CICECO**), is an interdisciplinary laboratory in the University of Aveiro (Portugal) with researchers from Chemistry, Ceramics and Glass Engineering and Physics Departments.

CICECO was created in 2000, as a result of the symbiosis of two research units of the University of Aveiro: *Centro de Química Inorgânica e de Materiais* and *Unidade de Investigação em Materiais Cerâmicos*.

On March 2002, CICECO received the status of Associated Laboratory from the Portuguese Ministry of Science, Technology and High Education, with the mission of:

Developing the scientific and technological knowledge necessary for the innovative production and transformation of ceramics and composite materials

Our main áreas of expertise are:

Area 1: Advanced micro- and nano-structured materials for communications technologies; Area 2: Advanced materials for industrial applications; chemistry and technology of polymeric; Area 3: Lignocellulosic materials and biomaterials.

In 2007 CICECO celebrates five years of activity as associated laboratory.

This document reports the main activities developed in 2006 as well as the actions aimed for 2007. More detailed lists of publications, theses, projects and activities are provided as annexes.

# **FACTS & NUMBERS**

CICECO is the largest Portuguese institute in the field of materials science and engineering. On 31<sup>st</sup> December 2006 CICECO hosted 270 people, an increase of 10.7% over 2005 and 53.4% relatively to 2002 (Table 1).

	2002	2003	2004	2005	2006
Professors and Lecturers	47	47	50	53	53
Full Time Researchers	5	9	12	16	20
<b>Post-Doctoral Associates</b>	23	22	29	40	46
Collaborators	16	13	12	11	23
PhD Students	54	60	61	54	67
MSc Students and Other Students	26	44	54	60	49
Laboratory Technicians	4	8	5	5	5
Administrative Personel	1	3	5	5	7
	176	206	228	244	270

## Table 1: CICECO's Research Team, 2002-2006

Almost 50% of the CICECO's Research Team in 2006 hold a PhD degree.

In 2006, our researchers were awarded to prizes and international recognition. Two of the youngest CICECO researchers, received the prestigious prizes '*Programa Gulbenkian de Estímulo a Investigação 2006*' and '*Prémio Celestino da Costa/ Jean*' - French Embassy.

We are also one of the most productive research institutes in the country in all scientific areas, with an average publication of 4.5-5 SCI papers per year per academic staff or full-time researcher:

\* over 1400 SCI papers published since 2002 (many in top journals);

\* 382 SCI papers published in 2006, ca. 5% of the total Portuguese papers (7686) quoted in the Web of Science;

\* 27 patents published;

\* 71 PhD theses terminated in the last 5 years (2002-06);

\* Aveiro University is at the top 1% of the most cited World institutions in the fields of Chemistry and Materials Science (Essential Science Indicators, Web of Knowledge).

Table 2 is a brief overview of the scientific activity outcome.

2002	2003	2004	2005	2006

Table 2: CICECO's Scientific Output, 2002-2006

		2002	2003	2004	2005	2006	Total
Theses	PhD	14	13	11	14	19	71
Theses	MSc	10	8	7	17	16	58
Books	Editions	0	0	1	1	1	3
DOORS	Chapters	4	14	12	7	5	42
	IF≥5	1	3	4	6	7	21
	IF<5	203	225	287	297	372	1384
SCI Papers	$2 \le IF < 5$	n.a.	n.a.	n.a.	n.a.	104	n.a.
	$0,6 \leq IF < 2$	n.a.	n.a.	n.a.	n.a.	184	n.a.
	IF<0,6	n.a.	n.a.	n.a.	n.a.	84	n.a.

The number of projects in progress in 2006 was 95, similar to the last five years. The main source of funding continues to be FCT, *National Science Foundation* (75%) and the European Funding Agencies and Programmes, *European Commission, FEDER*, (14%).

Section 5 list all projects, terminated and in progress.

CICECO is a truly international research centre:

\* 45% of full-time researchers and 65% of Post-docs are not Portuguese;

\* is part of the main stream materials research in Europe;

\* participates in Networks of Excellence.

We are actively involved in the Network of Excellence 'Functionalised Advanced materials and Engineering of Hybrids and Ceramics' - FAME, and in the INTERREG IIIB 'Materials Network for the Atlantic Area' – MNAA, or in Integrated Projects such as 'Innovation and Sustainable Development in the Fibre Based Packaging Value Chain'. We are running two Erasmus Mundus MSc courses, the 'Joint European Masters Programme in Materials Science' with the universities of Aalborg and Hamburg, and a FAME programme. We harboured one of the first and very few Portuguese Marie Curie Training sites, 'Advanced Ceramic Materials: Synthesis & Structure'.

CICECO is also committed to knowledge transfer to industry, through our CENTRE FOR MATERIALS DESIGN AND TECHNOLOGY - **CDTM**.

CICECO is probably the best equipped institute in the country to perform research in materials science. In particular, we are the focal point of the Portuguese Electron Microscopy Network and house the top solid-state

nuclear magnetic resonance facilities. We are also one of the best equipped national centres for X-ray diffraction.

X-Ray Diffraction Transmission Electron Microscopy (High resolution)







**CDTM** is CICECO's interface structure, responsible for promoting technology and knowledge transfer to society, and contributing to the economic and social growth of Portugal. CDTM works closely with researchers, in order to support them in four main areas: \*management of R&DT projects developed in partnership with companies; *\*intellectual property protection;* \*creation of new companies (spin-offs); \*commercialisation of technologies by licensing to existing companies.

To organise training courses and technical workshops in order to transfer knowledge and best practices to society is also a major objective of CDTM, whose initiatives in this area have proved successful.

With a total income of about 140 000€, CDTM has managed several R&DT projects in partnership with companies and research institutes, aiming at the development of new materials, process enhancement and resolution of specific industrial problems. In 2006 we have worked with the following companies and research institutes:

- Caima, Indústria de Celulose, SA, 'Determinação de pontos amostragem, análise de elementos não processuais e análises química e fisico-química de lenhosulfonatos';

- UNICER - Bebidas de Portugal SGPS, SA, 'Caracterização química de um tipo de cerveja por Espectroscopia de Ressonância Magnética Nuclear';

- Caima, Indústria de Celulose, SA, 'Estudo preliminar sobre a extracção dos componentes dos condensados em sistemas "líquido-líquido", utilizando diferentes solventes orgânicos. Desenvolvimento de metodologias de análise dos componentes em diferentes fases';

- *Caima, Indústria de Celulose, SA*, 'Análise preliminar de elementos não processuais, polissacarídeos e lenhosulfonados no licor de cozimento';

- Indasa - Indústria de Abrasivos SA, 'Estudo de novas resinas em base aquosa para lixas';

- Caima, Indústria de Celulose, SA, 'Biorefinaria - Aproveitamento de condensados';

- *Caima, Indústria de Celulose, SA*, 'Biorefinaria - Elementos não processuais no circuito de recuperação e potencialidades do licor de cozimento';

- *Centro Tecnológico da Cerâmica e do Vidro*, 'Duramater - Degradação de coberturas de edifícios sob acção da intempérie marítima: conhecimento sobre o desempenho e inovação de produtos';

- CERISOL - Isoladores Cerâmicos SA e MOTA - Pastas Cerâmicas SA, Contrato de assistência técnica;



CDTM is supporting the launching of a new company, *Tetracarbon*, specialized in developing new solutions for mechanical wear problems, by four researchers of the University of Aveiro, Rui Silva and Filipe Oliveira (CICECO - Dep. Ceramics and Glass Engineering) and Florinda Costa and António Fernandes (Physics Department).

Advanced ceramics and diamond coated materials are the basis of the solutions developed, which may be in the future adopted by various end-users, such as ceramic, moulding, chemical, wood, and metal industries, among others. The promoters hope that Tetracarbon will enter into force commercializing their solutions in 2008, associated with a company well-known in the market, with the proper distribution channels.



*FoodMetric* was legally created in 2006, with the support of CDTM. After a period of incubation at the University of Aveiro, this CICECO spin-off moved to the newly built infrastructures of the Incubator of Beira Atlântico Park. FoodMetric provides solutions to food and drink companies through

the implementation of fast and reliable methods to perform food analysis, replacing the conventional methods of analysis by instrumental ones. These solutions are systems composed by measurement instruments linked to software components able to meet exactly each customer's needs.

Several prizes in national entrepreneurial contests were awarded to FoodMetric:

- \* first prize at CONCURSO NACIONAL DE EMPREENDEDORES
- \* first prize at 2° CONCURSO DE CRIAÇÃO DE EMPRESAS DE BASE TECNOLÓGICA DE MIRA
- \* second prize at CONCURSO BIOEMPREENDEDOR

Concerning the external image of the Laboratory, there was an effort to improve the Promotion and Awareness of CICECO. Several technical workshops and training programmes were held in 2006, focusing mainly in the scientific areas of the laboratory, which have been giving an important contribution to the dissemination of the research results obtained by CICECO staff. In 2006 the following seminars, workshops and long-term training courses were organised:

- Revestimentos Rumo ao Futuro, Mechanical Engineering Department, March 31, 2006
- Nanotechnologies in Construction, Mechanical Engineering Department, July 3, 2006
- Lime Mortars, Past and Future, Mechanical Engineering Department, October 11, 2006
- Empreender: da Teoria à Prática, Environment Department, December 14, 2006

- *Synthesis, Properties and Application of Polymers*, an IDPoR 40- hours course, Chemistry Department, February 7, to July 18, 2006

- *Colloids, Interfaces and Surfaces*, an IDPoR 40-hours course, Chemistry Department, November 7, 2006 (to end in 2007)

These training sessions were attended by a total of 487 participants, 26% of which from SME's, which indicates how our objectives of promoting CICECO and exchanging know-how with companies were achieved.



**IDPoR** (Investigação e Desenvolvimento em Polímeros de Fontes Renováveis) Research and Development Platform on Polymers from

Renewable Resources, a cooperation platform between university and industry, was set up in September, 2006. Involving the creation of a consortium between a group of companies and the university research laboratories and departments, *IDPoR* focus on the area of Polymers from Renewable Resources, having the participation of major companies in this field, namely *CIN, CAIMA, Resiquímica, RAIZ and Euroresinas (Sonae Indústria). IDPoR* not only promotes a closer relation between these companies and the university, in the way that research may be driven by their specific industrial needs, but also encourages the development of training courses, workshops and, more importantly, an ambitious doctoral programme. The *IDPoR* associates have prior access to the research output of several PhD students, which will be available exclusively for them at the *IDPoR* webpage (http://idpor.ciceco.ua.pt). Associates are asked to pay a yearly fee to join *IDPoR* and to adhere for a period of five years. Its promoters are Professor Júlio Pedrosa, Professor Alessandro Gandini and Professor Carlos Pascoal Neto and *IDPoR* will hopefully respond to the needs of fundamental research and development in the area of Polymers from Renewable Sources, so as to improve technology transfer, offer training to its associates and develop a new idea of cooperation between University and Industry, increasing knowledge in this field and strengthening the Portuguese industrial competitiveness and innovation.

CDTM – CICECO perspective actions include the development of competitiveness poles between academia and industry, at national and international level, which will be of extreme importance in fostering the research and technology transfer among participants.

# **RESEARCH TEAM**

# **PROFESSORS & LECTURES**

ANA MARGARIDA M. V. BARROS TIMMONS ANA MARIA BASTOS COSTA SEGADÃES ANA MARIA DE OLIVEIRA ROCHA SENOS ANA MARIA PISSARRA COELHO GIL ANA MARIA VIEIRA SILVA VIANA CAVALEIRO ANTÓNIO TOMÁS DE FONSECA ARMANDO ANTÓNIO C. DOS SANTOS LOURENÇO ARMANDO JORGE DOMINGUES SILVESTRE ARTUR JORGE DE FARIA FERREIRA AUGUSTO LUÍS BARROS LOPES BRIAN JAMES GOODFELLOW CARLOS MANUEL SANTOS SILVA CARLOS PASCOAL NETO DMITRY VICTOROVITCH EVTYUGIN FERNANDO MANUEL BICO MARQUES FILIPE MIGUEL HENRIQUE L. R. FIGUEIREDO FRANCISCO AVELINO DA SILVA FREITAS HELENA ISABEL SEGURO NOGUEIRA HELENA MARIA C. SEIXAS CARAPUÇA ISABEL MARGARIDA MIRANDA SALVADO ISABEL MARIA BOAL PALHEIROS ISABEL MARIA DE SOUSA GONÇALVES ISABEL MARIA D. J. MARRUCHO FERREIRA JOÃO ANTÓNIO LABRINCHA BATISTA JOÃO CARLOS DE CASTRO ABRANTES JOÃO CARLOS MATIAS C. G. ROCHA

JOÃO MANUEL COSTA ARAÚJO P. COUTINHO JOAQUIM MANUEL VIEIRA JORGE RIBEIRO FRADE JOSÉ ANTÓNIO DA PURIFICAÇÃO MARTINS JOSÉ JOAQUIM COSTA CRUZ PINTO JOSÉ JOAQUIM CRISTINO TEIXEIRA DIAS JOSÉ MARIA DA FONTE FERREIRA JÚLIO DOMINGOS PEDROSA DA LUZ DE JESUS LEONEL MARQUES VITORINO JOAQUIM LUÍS ANTÓNIO FERREIRA M. DIAS CARLOS MARIA CLARA FERREIRA MAGALHÃES MARIA DE LOURDES GOMES PEREIRA MARIA ELISABETE JORGE VIEIRA COSTA MARIA GRACINDA FERREIRA DA SILVA MARIA HELENA FIGUEIRA VAZ FERNANDES MARIA INÊS PURCELL PORTUGAL BRANCO MARIA MARGARIDA TAVARES LOPES ALMEIDA MÁRIO GUERREIRO SILVA FERREIRA PAULA MARIA LOUSADA S. VILARINHO PAULO JORGE ALMEIDA RIBEIRO CLARO PEDRO MANUEL LIMA QUINTANILHA MANTAS RUI RAMOS FERREIRA E SILVA TERESA MARGARIDA DOS SANTOS TITO DA SILVA TRINDADE VICTOR MANUEL SOUSA FÉLIX VICTOR MIGUEL CARNEIRO S. FERREIRA VITOR BRÁS SEQUEIRA AMARAL

### **FULL TIME RESEARCHERS**

AIYING WU ALESSANDRO GANDINI ANA ISABEL DIAS DANIEL ANABELA TAVARES AGUIAR VALENTE ANDREI KHOLKIN CARMEN SOFIA DA ROCHA FREIRE BARROS FILIPE ALEXANDRE ALMEIDA PAZ FILIPE JOSÉ ALVES DE OLIVEIRA LUÍS MIGUEL MONTEIRO MAFRA MARC-GEORG WILLINGER MARIA RUTE DE AMORIM E SÁ F. ANDRÉ MÁRIO DE SOUZA REIS JUNIOR MARTYN PILLINGER NICOLA ALESSANDRO PINNA PAULA CELESTE DA SILVA FERREIRA SÉRGIO MANUEL DE SOUSA PEREIRA SUSANA ISABEL FONSECA A. S. BRAGA VLADISLAV KHARTON

YEVGENIY NAUMOVICH

ZHI LIN

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### **POST-DOCTORAL ASSOCIATES**

ALEKSEY YAREMCHENKO ALEXANDRE FERREIRA GALIO ANA LUÍSA DANIEL DA SILVA ANBALAGAN BALAMURUGAN ANDREI KAVALEUSKI ANTÓNIO ALEXANDRE DA CUNHA BASTOS ANTÓNIO FRANCISCO MOREIRA DOS SANTOS ARMANDINA MARIA LIMA LOPES BAOSHAN LI CLAUDIA CRISTINA LAGE PEREIRA COLLIN MAXIMILLIAN KOWALCHUK DILSHAT TULYAGANOV DUNCAN PAUL FAGG ERWAN RAUWEL FA NIAN SHI FENGYI LIU GARDAS RAMESH LAXMINARAYAN GONGBAO SONG IGOR RAEVSKYI IOLA MELISSA FERNANDES DUARTE JINGZHONG XIAO JOSÉ ANTÓNIO FERREIRA GAMELAS KUZHICHALIL PEETHAMBHARAN SURENDRAN LIANSHE FU LUÍS MANUEL CUNHA SILVA MARIA DE LA SALETE DA SILVA BALULA MARIA PAULA DA SILVA SEABRA MERCEDES VILA JUAREZ MIKALAI VYSHATKA

MIKHAIL ZHELUDKEVICH **OLEKSANDR TKACH** PAULA ALEXANDRINA DE AGUIAR P. MARQUES PAULA CRISTINA OLIVEIRA RODRIGUES PINTO PAULA CRISTINA RAMOS SOARES E SANTOS PROTIMA SINGH-RAUWEL RU ZHONG HOU SANDRA MARIA NUNES GAGO SANJEEVI KANNAN SERGEY YAKOVLEV SERGIO NUNO MARTINS LIMA STANISLAV FERDOV SVIATLANA V. LAMAKA VIVIANA POSSAMAI DELLA VLADIMIR ANATOLYEVICH KHOMCHENKO VLADIMIR BYSTROV WEIDONG ZHANG

# **PhD STUDENTS**

ANA CATARINA DE CARVALHO ESTEVES ANA CATARINA DIAS MARTINS COELHO ANA CRISTINA ESTRADA MORAIS G. SOUSA ANA GIZELA GUEDES NUNES DA CUNHA ANA LÚCIA HOROVISTIZ ANA SOFIA MADUREIRA BRUNO ANA SOFIA VAGUEIRO DE SOUSA DIAS ANA SOFIA VILA MONA SANTIAGO ANDRÉIA GERNISKI MACEDO ANDREIA MARIANA VALINHO DIAS ÂNGELA SOFIA DOS SANTOS PEREIRA ANTÓNIO PAULO CERQUEIRA DUARTE BÁRBARA JOANA MARTINS LEITE FERREIRA BERNARDO RAMOS BATISTA MONTEIRO CARLA MARIA BATISTA GONÇALVES CARLA PATRÍCIA ALVES FREIRE MADEIRA CRUZ CATARINA ALEXANDRA GONÇALVES PEREIRA CATARINA GONÇALVES CLÁUDIA MARIA BATISTA LOPES CLÁUDIA PEREIRA PASSOS CRISTINA MARIA DA SILVA FERNANDES CRISTIANO ALEXANDRE ANDRADE DIAS DMITRY KISELEV DORA SALOMÉ CORREIA COELHO FABIANE COSTA OLIVEIRA FÁBIO GABRIEL NAZÁRIO FIGUEIRAS FLÁVIA APARECIDA DE ALMEIDA FLORENTINA VIOLETA MAXIM FU ZHI **GUYLHAINE CLAVEL** HELENA CRISTINA CORREIA DE OLIVEIRA JOANNA ASZTEMBORSKA KRAKOVIAK JOÃO CUNHA DE SEQUEIRA AMARAL JOÃO EDUARDO ALEIXO RODRIGUES JOSÉ ANTÓNIO PEREZ DE LA TORRE JOSÉ MANUEL GONÇALVES VENTURA JOSÉ PEDRO TEIXEIRA DOMINGUES KIRYL YASAKAU LI JIAN LUCIANA SARABANDO DA ROCHA MARA GUADALUPE FREIRE MARTINS

MÁRCIA CARVALHO NEVES MARGARIDA ISABEL DOS SANTOS AMARAL MARIA GRÁCIA CORDEIRO COSTA MARIA MANUELA JORGE E. RODRIGUES MARIANA BELO DE OLIVEIRA MARIANA ISABEL COUTINHO R. SARDO MARIELA MARTINS NOLASCO MARIYA HRISTOVA KOSTOVA MOHAMED KARMAOUI NADIA KHALED ZURBA NELSON CHAMUSCA FONSECA NELSON SIMÕES OLIVEIRA NUNO MIGUEL DUARTE PEDROSA OLENA IVANIVNA OKHAY PATRÍCIA FERREIRA LITO PAULA ANDREIA FERNANDES DE SOUSA PENKA ILIEVA GIRGINOVA **RAJAMMA REJINI** RICARDO GIL HENRIQUES SERRA SANDRA CRISTINA ALMEIA PINA SANDRA CRISTINA PEREIRA CACHINHO SARA ALEXANDRA BRUNHETA LISBOA SÓNIA DE SOUSA NOBRE SUSANA CRISTINA DE MATOS FERNANDES SUSANA MARIA HENRIQUES OLHERO TETYANA VALENTYIVNA MALYSH

## **MSc STUDENTS**

**S1** 

ANA TERESA PAIVA AURORA BRANCA AMORIM DE ARAÚJO CARLOS MANUEL DOMINGUEZ MENDONÇA CATARINA FERREIRA DOS SANTOS EMMANUEL FRANCISCO CHIMA CHIMAMKPAM ERMELINDA DA CONCEIÇÃO P. SALGUEIREDO GERARDO GONZÁLEZ AGUILAR HELENA MARIA DA COSTA PAIVA JEAN CARLOS DE CONCEIÇÃO JIE GAO JÚLIO CÉSAR LONGO LUÍS MIGUEL ALMEIDA AMARAL PAULA MARIA DA COSTA TORRES RAFAEL DA SILVA MARTINS

## **OTHER STUDENTS**

ANA MARGARIDA BATISTA DA SILVA ANDREIA FILIPA GOUVEIA MACHADO ANA FILIPA SARAIVA DAS NEVES ASHUTOSH GOEL CARLA ANDREIA CUNHA VILELA CARLA PATRÍCIA GONÇALVES SILVA CARLOS MIGUEL CARDEAL ENES GRANADEIRO CARLOS VICENTE EDGAR PEREIRA NADAIS **EKATERINA TSIPIS** GABRIEL DUARTE ALMEIDA SOUSA GIL ALBERTO BATISTA GONÇALVES GONÇALO MIGUEL GOMES GRAÇA GUSTAVO ANDRÉ CASTRO FERREIRA MARQUES HELGA MARGARIDA CORREIA F. GRACIA HUGO ALEXANDRE G. R. FERNANDES

HUMBERTO FERREIRA PLÁCIDO JOÃO CARLOS ASCENÇÃO ALONSO JOÃO NUNO SANTOS GONÇALVES JOSÉ JOAQUIM BARROS MACHADO MARCOS GOMES GHISLANDI MICAELA FILIPA MOREIRA DE SOUSA MIGUEL JOSÉ LOPES MIRANDA CARRAPIÇO NUNO ALEXANDRE DIAS FERNANDES PATRÍCIA DOS SANTOS NEVES PAULO MANUEL MACHADO DE CARVALHO PRISCILA TATIANA DOS SANTOS GONÇALVES RICARDO JOÃO BORGES PINTO RUI MANUEL COUTINHO RODRIGUES **RUI MIGUEL DE ANDRADE DOMINGUES** SANDRA PEREIRA MAGINA SÓNIA PATRÍCIA MARQUES VENTURA THERESA OBIAGELI CHIMAMKPAM TSEVETELINA IVANOVA GERGANOVA YEVHENIY VOLODIMIROVICH PIVAK

# DIRECT COLLABORATORS

ANA MARGARIDA ALMEIDA JESUS ANTONIO ALEXANDRE MARTINS AVITO HERNÂNI DOS SANTOS REBELO CLÁUDIO JOSÉ DE ALMEIDA C. DA CRUZ DIANA MÓNICA DE MESQUITA S. FERNANDES DUARTE ANANIAS MARQUES ERIKA JUDITH CARDOSO RODRIGUES DAVIM FILIPE MIGUEL DE ALMEIDA M. DOS SANTOS JOAO MIGUEL MAIA CARRAPICHANO JOAQUIM MANUEL DA GRAÇA SACRAMENTO JOSE MARTINHO MARQUES DE OLIVEIRA LUIS PEDRO FARIA RIBEIRO S. ESTEVES MARIA ISABEL GOMES DE PINHO MARIA ARLETE CARNEIRO R. CARVALHO MARIA FERNANDA PEREIRA DA SILVA MOISES LUZIA GONCALVES PINTO NATÁLIA BRAZ BARROCA PRISCILLA FILOMENA FONSECA AMARAL RAQUEL SOFIA LINO FERREIRA DOS SANTOS RUI NUNES CORREIA SANDRA MANUEL SIMARIA DE O. LUCAS SUSANA RAQUEL DE MELO TIAGO TICIANA DE ASTRIOGILDO E TRÉZ

## LABORATORY TECHNICIANS

ANA PAULA FIGUEIREDO ESCULCAS ZHU MARIA CELESTE COIMBRA AZEVEDO MARIA DO ROSÁRIO TEIXEIRA SOARES MARTA ASCENSÃO CARMONA FERRO PAULA CRISTINA FERREIRA DA S. BRANDÃO

## ADMINISTRATIVE PERSONEL

CARLA PATRÍCIA COUTINHO RANITO MÓNICA SOFIA FERREIRA TAVARES DORA FÁTIMA DOS SANTOS VERA MÓNICA DE ALMEIDA FERNANDES MARIA ISABEL DE JESUS SANTOS MARINA KHOLKINA PAULA -CRISTINA DA SILVA PAIS <u>S1</u> 16



<u>S2</u> 18 CICECO focuses its activities on three distinct research areas and nine lines of study:

# Area 1 - ADVANCED MICRO- AND NANO-STRUCTURED MATERIALS FOR COMMUNICATIONS TECHNOLOGIES

Inorganic Multifunctional Materials and Organic-Inorganic Hybrids Electroceramics Magnetostructural Modulation of Strongly Correlated Electric Materials Advanced Molecular and Supramolecular Materials

# Area 2 - ADVANCED MATERIALS FOR INDUSTRIAL APPLICATIONS

Reactive Ceramic Components for Process Control Ceramic Composites and Ultra-Hard Coatings for Mechanical Applications

# Area 3 - CHEMISTRY AND TECHNOLOGY OF POLYMERIC AND LIGNOCELLULOSIC MATERIALS AND BIOMATERIALS

Macromolecular Materials and Lignocelullosics

Biomedical and Biomimetic Materials

Process Development and Optimisation

This secton will report the research activity of CICECO of 2006. An overwiev of the Actions for 2007 is exposed at section 3.

# AREA 1 – ADVANCED MICRO- AND NANO-STRUCTURED MATERIALS FOR COMMUNICATIONS TECHNOLOGIES

## INORGANIC MULTIFUNCTIONAL MATERIALS AND ORGANIC-INORGANIC HYBRIDS

*New Microporous Materials.* The first example of a photoluminescent chiral microporous Ln silicate system  $Na_3[(Y,Ln)Si_3O_9]\cdot 3H_2O$  (Ln=Eu, Tb, Er, Ce), in which discrimination between enantomeric domains was achieved by means of Eu<sup>3+</sup> photoluminescence spectroscopy, using unpolarised light and in the absence of external fields, has been reported. This enantioselectivity phenomenon may be of importance in the context of fundamental interactions between light and condensed matter.

Polycrystalline NASICON (sodium silicon conductor) fast-ion conductors ( $Na_5LnSi_4O_{12}$ , Ln = Eu, Tb) were prepared via solid-state synthesis and characterised by photoluminescence spectroscopy. Two  $Eu^{3+}$  sites were detected: (i) in regular framework positions, and (ii) replacing  $Na^+$  ions in the tunnels.

The hydrothermal synthesis of the small-pore framework sodium stannosilicate  $Na_2SnSi_4O_{11}$ ·2H<sub>2</sub>O (Sn-AV-14) possessing the structure of the rare titanosilicate mineral penkvilksite-1*M* were reported. The synthesis of this material was optimised and shown to be very sensitive to the composition of the precursor gel, particularly the Si and water contents and seeding.

The thermal transformation of  $Eu^{3+}$ -doped and undoped microporous titanosilicate AM-3 was reported. AM-3 is stable up to 600 °C and transforms into the analogue of the mineral narsarsukite at 800 °C. The narsarsukite obtained from AM-3 is purer than that prepared from the titanosilicates ETS-10, ETS-4 and AM-1, and is suitable for hosting optically-active Ln<sup>3+</sup> ions.

Eu and Eu-Gd silicates with apatite-type structure were synthesised under mild hydrothermal conditions. These materials are efficient room-temperature emitters with a maximum external quantum yield of ca. 21.4 %.

The hydrothermal synthesis and characterization of microporous mixed valent vanadium containing AM-2 have been reported. Titanium and silicon have been partly substituted by vanadium. The presence of octahedral  $V^{4+}$ ,  $V^{5+}$  and tetrahedral  $V^{5+}$  was revealed by diffuse reflectance ultraviolet-visible spectroscopy and solid-state NMR.

Membranes of microporous stannosilicate and titanosilicate ( $K_2MSi_3O_9$ , $H_2O$ , M= Sn and Si) with the umbite structure, prepared on porous alumina and TiO<sub>2</sub> tubular supports, are able to separate  $H_2$  from  $N_2$ , CO<sub>2</sub> and propane. After 640 h under stream, they separate a  $H_2/N_2$  mixture with selectivities larger than 30, and permeations of *ca*. 10<sup>-7</sup> mol/(m<sup>2</sup>·s·Pa).

A study wasperformed aimed at exploring the ability of very small sized N-bearing molecules to generate and stabilize microporous aluminophosphates. Two new AIPO4-n materials, named IST-1 and IST-2, have been

obtained in aqueous media using, as main template, methylamine (MA), directly added, or generated in situ from methylformamide degradation. While IST-1 topology is novel, IST-2 is structurally related to AlPO4-53(A). The structure of the microporous aluminophosphate IST-1 was revisited using high-resolution heteronuclear/homonuclear solid-state NMR techniques based on FS-LG homonuclear decoupling and <sup>31</sup>P-<sup>31</sup>P homonuclear recoupling allowed a complete assignment of the <sup>1</sup>H NMR spectra.

Aluminum methylphosphonate polymorphs (AlMePO's) are peculiar hybrid inorganic-organic porous materials with hydrophobic methyl groups on the inner surface and thus have low affinity for adsorbing highly polar molecules. A theoretical study of these materials was carried out to get some insight into the physicochemical characteristics of AlMePO- $\alpha$  by means of molecular simulations, and to provide test simulation tools to accurately describe the adsorption behaviour of nitrogen and water on this material.

*Mesoporous Materials.* MCM-48 membranes have been prepared on alumina supports. A battery of characterization techniques has been used to study the physical properties and the quality of these membranes. The membranes were tested in the separation of gas phase mixtures and a cyclohexane/ $O_2$  selectivity higher than 270 was obtained.

A comparison of the pore structural properties and catalytic activity of MCM-41 containing aluminium, prepared at room temperature with different aluminium sources, was reported. In addition, they were compared with those of MCM-41 obtained by two conventional hydrothermal procedures and room temperature synthesised Al-MCM-48 grades. The room-temperature synthesis resulted in the preparation in a short time of well structured MCM-41 materials containing mainly four-coordinated Al and presenting acidic catalytic activity similar to or better than those prepared by the two hydrothermal procedures. Aluminium sulfate is a good alternative to isopropoxide in the room temperature. The most active sample is a MCM-48 grade also prepared at room temperature with the same precursor.

The complex MoO<sub>2</sub>Cl<sub>2</sub>(THF)<sub>2</sub> and methyltrioxorhenium were immobilised in a mesoporous silica functionalised with a pyrazolylpyridine ligand (MCM-41-L) and characterised. In the former case, two supported materials were prepared, one of which involved the postsynthesis trimethylsilylation of MCM-41-L to remove the residual surface silanol groups. Model complexes containing the ligand ethyl[3-(2-pyridyl)-1-pyrazolyl]acetate were also prepared. The data showed the formation of tethered complexes of the type MoO<sub>2</sub>Cl<sub>2</sub>L and CH<sub>3</sub>ReO<sub>3</sub>L, involving bidentate and monodentate coordination of the pyrazolylpyridine ligand, respectively. The former materials were active and selective heterogeneous catalysts for the epoxidation of olefins using t-BuOOH as the oxidant. Some activity was lost from the first to second runs, but thereafter the solids could be recycled without significant loss of activity.

 $MX_2(CO)_3(DAB)$  (M = Mo, W; X= I, Br) complexes with the 1,4-diazobutadiene ligand with two Si(OEt)<sub>3</sub> groups were immobilised in MCM-41 mesoporous silica. The modified materials were extensively characterised by several techniques and were tested as catalysts on the reaction of ring-opening metathesis polymerization of norbornene and norbornadiene at 328 K.

Novel mesoporous MCM-41 type hybrid materials were synthesised by co-condensation of 1,4-diazobutadiene ligand bearing two  $Si(OEt)_3$  groups and tetraethyl orthosilicate in three different ratios (0.027, 0.05, and 0.20) and in the presence of cetyltrimethylammonium bromide. The surfact-free materials with low organic content present uniform 2D-hexagonal mesoporous arrays with pore diameter ranging from 2.0 to 4.0 nm and a surface area ranging from 693 to 803 m2/g. The increase of organic building block content on the materials reduces the order and the space available within the pore.

*Layered Materials*. The synthesis and characterisation of layered materials continued to be studied, as well as the intercalation of metallo-organic complexes in layered double hydroxides (Mg-Al and Zn-Al LDHs). The photofunctional and catalytic properties of some of these materials was tested. Oxomolybdenum(VI) and oxotungsten(VI) complexes of 3,4-dihydroxybenzoic acid, with the general formula  $[MO_2(3,4-dhb)_2]^{n-}$ , were incorporated into Mg-Al and Zn-Al layered double hydroxides by ion exchange with precursor materials in the nitrate form. Possible applications for these materials lie in the field of heterogeneous oxygen atom transfer reactions.

Two series of layered double hydroxides with compositions Mg,Al–CO<sub>3</sub> and Mg,Cr–CO<sub>3</sub> were prepared by microwave and conventional hydrothermal treatment to study the role of microwave radiation on their physicochemical properties. The hydrotalcite crystallinity is enhanced by microwave hydrothermal treatment. These samples also have larger amounts of interlayer water and smaller particle size than those prepared by conventional hydrothermal ageing. The textural properties of the solids are strongly modified during ageing, and

large specific surface areas are observed at shorter times for samples subjected to microwave radiation.

*Nanostructured Materials.* The chemistry related to colloidal nanocrystals consisting of ZnO doped with  $Ln^{3+}$  cations was researched. Introducing the Tb<sup>3+</sup> cations in ZnO nanosized hosts results in photoluminescent systems with wavelength emission which depends on the excitation line. Chemical surface modification of the doped ZnO nanocrystals and photoluminescence studies revealed that Tb<sup>3+</sup> cations are located in the ZnO core, although the exact crystalline environment is still uncertain. Surface modification methods were investigated to find evidence for the mechanisms of inclusion of these cations in nanophase semiconductors, using inorganic and polymeric phases as cappings.

Silica-coated  $Bi_2S_3$  nanofibers were synthesised and characterized. The nanofibers showed morphological properties which depend on synthesis conditions, such as temperature and solvent used. These nanomaterials were then investigated as nano-fillers in new nanocomposites prepared by in situ emulsion and suspension polymerization. TEM and SEM showed that in both cases the  $Bi_2S_3/SiO_2$  nanoparticles were densely coated with poly(styrene). In situ emulsion polymerization afforded nanocomposites in which the nanofibers were coated with polymer spheres whilst suspension polymerization gives rise to a homogeneous polymer layer coat. The morphology of the poly(styrene) coating observed was discussed considering the surface modification of the nanofibers and the polymerization technique involved.

The potential of Surface-Enhanced Raman Scattering to study the interaction of metal nanocrystals with molecular adsorbates was explored, in particular for the investigation of adsorption modes and orientation of molecules on the surfaces with relevance in heterogeneous catalysis and nanoparticle assembly studies.

Magnetic studies in natural ferritin and iron oxide nanoparticles (ferrihydrite and other phases) in organicinorganic hybrids and polymer matrices. Effect of partice size distributions on the magnetic properties/anisotropy. Simulation of magnetic properties (Monte-Carlo). Analysis of effect of magnetic dead layer. High Field (up to 50 Tesla) studies of Exchange Bias in nanoparticles (Grenoble, Niemegen and Toulouse).

The photoluminescence of di-ureasil hybrids containing homogeneously dispersed Ag nanoparticles, covered with silica shells was investigated and compared with emission of undoped hybrids. Metal-doped di-ureasils show a broad emission band in the blue spectral region similar to what has been observed in pure di-ureasils but with lower relative intensity. A broad minimum arises at *ca*. 438 nm for Ag@SiO<sub>2</sub>-containing di-ureasils, which coincides with the peak of plasmon resonance band in the absorption spectrum. This behaviour may be attributed to an inner filtering effect, manifesting itself as absorption by metal nanoparticles of light emitted from the matrix. The lifetimes for Ag@SiO<sub>2</sub>-doped di-ureasils are smaller than those measured for the undoped host, suggesting that energy transfer may occur between the hybrid's emitting centres (NH groups and siliceous nanodomains) and the nanoparticles.

The nitride light emitting backplanes (in the form of semiconductor p-n junctions) was used as a platform to integrate other material systems, such as luminescent polymers, metallic nanocrystals and semiconductor quantum dots, creating new hybrid nanostructures that may be excited by an electric current. Namely, the interactions between Mott-Wannier (M-W) and Frenkel excitons in a family of organic/inorganic hybrid structures consisting of thin organic (polyfluorene) films placed in close proximity (systematically adjusted by GaN cap layer thickness) to single inorganic InGaN/GaN QWs were studied. It was demonstrated that non-radiative energy transfer may occur between inorganic and organic counterparts.

A new general non-aqueous sol-gel route was used for the preparation of rare earth (RE) ordered nanocrystalline hybrid structures. In a simple one-pot reaction process, RE(III) isopropoxides (RE= Y, Er, Gd, Sm, Nd) were dissolved in different alcohols and reacted in an autoclave between 250 and 300°C. This approach leads to very thin (~0.6 nm) crystalline lanthanide oxide layers regularly separated from each other by organic layers of intercalated organic carboxylate molecules derived from the oxidation of the alcohol used as solvent (the thickness of the organic part is typically between 1.2 - 2.0 nm). The optical properties of such nanohybrids were evaluated. It was found that an efficient charge transfer from the organic molecules (benzoates and 4-biphenolates) to the metal emitting centres takes place. By following such an excitation path the energy required in order to sensitize the luminescent ions is significantly lower that the one needed by similar pure inorganic compounds.

 $\frac{S2}{24}$ 

*Polyoxometalates.* The synthesis and study of new hybrid compounds with polyoxometalates and organic moieties were continued. New compounds were obtained through solid state reaction of commercial Keggin heteropolyacids  $H_n[XM_{12}O_{40}] \cdot aq$  (M = Mo, W; X = P and n = 3; X = Si and n = 4) with the organic aromatic molecules 4-aminopyridine, 4-phenylpyridine e coumarin. The reactions were accompanied by loss of hydration water and probably by acid-base reaction between the oxonium cations and the basic organic moieties, with simultaneous change of the original lattice. A similar study was performed with  $H_3[PW_{12}O_{40}] \cdot 6H_2O$  and the above-mentioned organics, 1,10-phenanthroline and 2-amino-5-nitropyridine. The resulting compounds had very little hydration water and presented the original body centred cubic lattice typical of the hexa-hydrated heteropolyacids. In this case all evidence points to the isomorphic substitution of water by the organic molecules.

Studies were continued on the homogeneous catalytic oxidation of hydrocarbons with  $H_2O_2$  in the presence of the iron- or manganese-substituted polyoxotungstates  $[XW_{11}M(H_2O)O_{39}]^{n-}M = Mn^{III}$ ,  $Fe^{III}$ , X = P, Si and B, namely the oxidation of indan, indene, 1,2,3,4-tetrahydronaphtalene and 1,2-dihydronaphtalene. Identification of reaction products is now complete and the more convenient reaction conditions determined. The preparation of silica

supported transition metal-substituted polyoxotungstates to be evaluated as oxidative heterogeneous catalysts was started, but only preliminary results were obtained so far.

Preparation and structural characterization of lanthanide complexes of lacunary polyoxomolybdates or polyoxotungstates was continued. A new family of luminescent materials has been prepared by treatment of a Zn–Al layered double hydroxide in nitrate form with different polyoxotungstoeuropate anions,  $[EuW_{10}O_{36}]^{9}$ ,  $[Eu(BW_{11}O_{39})(H_2O)_3]^{6-}$  and  $[Eu(PW_{11}O_{39})_2]^{11-}$ . The host-guest interaction has a strong influence on the nature of the final intercalated species. The photoluminescence and structural studies afforded a detailed picture of the nature of the intercalated anions.

A layer-by-layer assembly method was employed to fabricate multilayered films containing the terbium heteropolyanions  $K_6[Tb(BW_{11}O_{39})(H_2O)_3]\cdot 32H_2O$  and  $K_y[Tb(XW_{11}O_{39})_2]\cdot nH_2O$  (X = Si and P) and the polyelectrolytes poly(4-styrenesulfonate) and poly(allylamine hydrochloride). A comparative study on the optical properties of the films containing distinct polyoxometalates and different types of multilayers was carried out. The photoluminescence of the solid polyoxometalates and of the resulting films was investigated.

New lanthanotungstocobaltates with the Keggin structure and general formula  $K_xH_y[Ln(CoW_{11}O_{39})(H_2O)_3]\cdot nH_2O$ or  $K_xH_yLn[Ln(CoW_{11}O_{39})(H_2O)_3]\cdot nH_2O$ , Ln(III) = Ce, La, Eu, Sm, Tb, were prepared and characterized. Hybrid silica materials, with the polyoxoanions anchored to spherical silica particles, were also prepared. Their characterisation suggests that the structure of the Keggin-type polyanions is maintained when they are immobilized in the silica material.

The first member of a new family of monolacunary Keggin-type polyoxotungstates containing Ag(I) metallic centres,  $H_2Ag_{0.33}K_{3.67}[AgPW_{11}O_{39}]\cdot 8.25H_2O\cdot CH3OH$ , was reported. A novel chain-like silver polyoxotungstophosphate forms when Ag(I) metal centres, exhibiting an unusual eight-coordination fashion, bridge a monolacunary  $[PW_{11}O_{39}]^{7-}$  anion to four bridging  $\mu_2$ -oxygen atoms of a neighbouring lacunary  $\alpha$ -Keggin anion.

The effects of organic ligands and POMs on the luminescent properties of hybrid materials have been investigated, in particular, considering the possibility that they may act as sensitizers of the lanthanide luminescence. Novel polynuclear tungsten and Mo(VI) complexes with 3-hydroxypicolinic acid (HpicOH) and Eu(III)  $[M_4O_{12}Eu(picOH)_3]$  (M(VI) = W, Mo) were prepared by hydrothermal methods. The photoluminescent properties of the materials were investigated, showing sensitization of the Eu(III) luminescence by both the 3-hydroxypicolinate ligand and the polyoxometalate moiety.

Novel ruthenium tetra-substituted polyoxometalate compounds  $K_6Na[SiW_9O_{37}Ru^{III}_4(H_2O)_3Cl_3]$  nH<sub>2</sub>O ( $\alpha$  and  $\beta$  SiW<sub>9</sub>O<sub>37</sub> isomers, **1** and **2**) were synthesised via the reaction of sodium salts of trilacunar Keggin  $\alpha$ - and  $\beta$ -

 $[SiW_9O_{34}]^{9}$  heteroanions with RuCl<sub>3</sub> in aqueous solution. The characterization of compounds **1** and **2** by wet chemistry, thermal analysis and spectroscopic techniques are in course. These POMs were preliminary considered as potentially interesting redox catalysts.

*Novel Luminescent Systems.* Research on novel lanthanide luminescent systems based on lanthanide complexes and its incorporation in nanomaterials was continued. Research concerned the coordination chemistry of lanthanides with aromatic ambidentate ligands, exploring the possibility of formation of multidimensional lanthanide compounds. The incorporation of those lanthanide compounds into nanosized SiO<sub>2</sub> and other substrates was explored. The luminescence and structural properties were studied.

Unusual full-colour phosphors, in the system  $Na_3LnSi_3O_9$ , have been prepared and their structure and photoluminescence properties characterized.  $Na_3(Y_{1-a}Ln_a)Si_3O_9$  (Ln = Eu, Tb, Tm) materials are primary emitters, with chromaticity colour coordinates comparable or better than properties of the standard phosphors recommended by EBU for display devices, resulting from the emission of red (Eu<sup>3+</sup>), green (Tb<sup>3+</sup>) and blue (Tm<sup>3+</sup>) light.

*Crystal Engineering of Organic-Inorganic Hybrids.* A number of novel multi-dimensional magnetic materials combining N-(phosphonomethyl)iminodiacetic acid (H<sub>4</sub>pmida) and transition metal centres were isolated. The first example of a neutral 1D coordination polymer featuring Fe<sup>2+</sup> centres and H<sub>2</sub>pmida<sup>2-</sup> was reported, with this material being also the first of its kind with H<sub>4</sub>pmida residues exhibiting one uncoordinated carboxylic acid group. With 4,4'-bipyridine (2,2'-bpy) and H<sub>4</sub>pmida, two isostructural compounds were isolated in the solid-state, (4,4'-bpyH)<sub>2</sub>[M(4,4'-bpy)(H<sub>2</sub>O)<sub>4</sub>][V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]·2H<sub>2</sub>O (where M = Mn<sup>2+</sup> or Co<sup>2+</sup>). These compounds are formed by the close packing of 1D [M(4,4'-bpy)(H<sub>2</sub>O)<sub>4</sub>]<sub>n</sub><sup>2n+</sup> cationic polymers and 4,4'-bpyH<sup>+</sup> cations, along with dimeric centrosymmetric [V<sub>2</sub>O<sub>2</sub>(pmida)<sub>2</sub>]<sup>4</sup> anionic moieties located in the crystal interstices. From the reaction of Cu<sup>2+</sup> and H<sub>4</sub>pmida along with diethylenetriamine (det), a new complex-type material was isolated in the solid-state, [Cu(Hpmida)(Hdet)], and characterized by single-crystal XRD. The magnetic properties were also investigated, revealing that interactions between neighbouring metal centres are minimal with the bulk materials being best characterized by the magnetic properties of isolated metallic centres.

A series of three Sm(III) one-dimensional coordination polymers containing picolinic acid (Hpic), 3hydroxypicolinic acid (HpicOH) and 2-hydroxynicotinic acid (H<sub>2</sub>nicO) was isolated in the solid state and characterised structurally:  $K_2[Sm_2(pic)_6(\mu-pic)_2]\cdot7.5H_2O$  (1),  $[Sm(picOH)_2(\mu-HpicO)(H_2O)]\cdot3H_2O$  (2) and  $[Sm(HnicO)_2(\mu-HnicO)(H_2O)]\cdot5H_2O$ . All compounds show room-temperature photoluminescence which was investigated. A poorly crystalline organic-inorganic hybrid oxovanadium(V) phosphate complex,  $\{[V_2O_4(Phen)_2(PO_4)]_2VO(OH)\}_{3/4}\{[V_2O_4(Phen)_2-(HPO_4)]_2\}_{1/4}\cdot4.5H_2O$  (where Phen = 1,10-phenanthroline), was shown to contain two markedly similar binuclear dioxovanadium(V) moieties. <sup>51</sup>V and <sup>31</sup>P solid-state NMR proved unequivocally the presence of these two coordination environments. A simulation of the <sup>51</sup>V spectrum unveiled the relative populations of the <sup>51</sup>V sites plus additional information of their coordination geometries.

The anionic [Ge<sub>2</sub>(pmida)<sub>2</sub>(OH)<sub>2</sub>]<sup>2-</sup> complex, a potential secondary building block for the construction of metalorganic frameworks, was isolated in the solid state from hydrothermal synthesis as  $(C_4H_{12}N_2)[Ge_2(pmida)_2(OH)_2]\cdot 4H_2O$ pmida<sup>4-</sup> [where = N-(phosphonomethyl)iminodiacetate and  $C_4H_{12}N_2^{2+}$  = piperazinedium]. Several high-resolution solid-state <sup>1</sup>H NMR techniques, have been employed for the first time in the study of this type of hybrid crystalline materials.

The  $(HL)_6[Ge_6(OH)_6(hedp)_6]\cdot 2(L)\cdot nH_2O$  system, where L is 8-hydroxyquinoline (hqn) or 1,10-phenanthroline (phen), was shown to be composed by  $\pi$ - $\pi$  columns of the organic aromatic residues which pack with hexameric anionic  $[Ge_6(\mu_2-OH)_6(C_2H_4O_7P_2)_6]^{6-}$  moieties leading to the formation of porous supramolecular structures with large amounts of water filling up the available voids. The photoluminescence properties of these compounds were studied in the solid state. High-resolution solid-state <sup>1</sup>H NMR techniques have been used to study the complex hydrogen-bonding networks.

*Organic-Inorganic Hybrids Lacking Activating Centers.* Two organo-bridged photoluminescent silsequioxanes derived from the same precursor  $(EtO)_3Si(CH_2)_3-C_6H_4-(CH_2)_3Si(OEt)_3$  were synthesised under acid and basic hydrolysis. Crystalline lamellar bridged silsesquioxane with long-range ordering (acid hydrolysis) and amorphous hybrids (basic hydrolysis) were obtained. In both hybrids, the emission arises from a mixture of components due to the aromatic rings (short-lived and high-energetic band), and recombinations occurring at the NH groups (long-lived and low-energetic). A di-ureasil hybrid (d-U(600)) was successfully incorporated into the channels of mesoporous silica MCM-41, MCM-41, preserving therefore the hexagonal symmetry of the MCM-41 support. The emission spectrum of MCM-41-d-U(600) exhibits the intrinsic green emission of MCM-41, which is selectively excited in the UV spectral range. For excitation wavelengths lying in the visible the spectra is a convolution of the MCM-41 intrinsic emission with that characteristic of d-U(600). Time-resolved spectroscopy unequivocally demonstrates MCM-41-to-d-U(600) energy transfer and the di-ureasil contribution to the overall emission features.

*New Hybrid Materials.* Di-ureasils incorporating  $Eu(TPI)_3 \cdot 3H_2O$  or  $Eu(TPI)_3 \cdot 2TOPO$  (where TPI and TOPO stand for 3-phenyl- 4-(4-toluoyl)-5-isoxazolone and tri-*n*-octylphosphane oxide, respectively) were synthesised by both

acetic acid solvolysis and conventional hydrolysis sol-gel route. The photoluminescence spectra display the typical  $Eu^{3+}$  red emission, and a larger  ${}^{5}D_{0}$  quantum efficiency ( $\eta = 13\%$  vs. 32%) and  ${}^{5}D_{0}$  lifetime ( $\tau = 0.30$  vs. 0.42 ms) were measured for the hybrid incorporating the Eu(TPI)<sub>3</sub>·3H<sub>2</sub>O complex, compared with the isolated complex. The enhancement was explained by the coordination ability of the organic counter part of the host structure, which is strong enough to displace the water molecules of the Eu(TPI)<sub>3</sub>·3H<sub>2</sub>O complex from the Eu<sup>3+</sup> neighborhood in the hybrids. The ability of the di-ureasil host to coordinate tris- $\beta$ -diketonate complexes and the energy transfer processes involving the hybrid host emitting levels and those of the complexes were also studied in a series of di-ureasil hybrids incorporating Ln(btfa)3-(4,4'-bpy)(EtOH) (Ln=Eu, Gd; 4,4'-bpy=4,4'-bipyridine; btfa=4,4,4-trifluoro-1-phenyl-1,3-butanedione) complexes. The calculated quantum yield (*ca.* 46%) is in fair agreement with the experimental value (38 ±4%). For the Eu<sup>3+</sup>-based di-ureasils a 50% quantum yield enhancement compared to the Eu<sup>3+</sup> complex is observed, suggesting an effective hybrid host–metal ion interaction and an active energy-transfer channel between the hybrid host and the Eu<sup>3+</sup> complex. The Eu<sup>3+</sup>-based di-ureasils are photostable under UVA (360 nm) excitation, while degradation occurs under UVB (320 nm) and UVC (290 nm).

Materials with different concentration of zinc and lithium triflate incorporated into sol–gel derived di-ureasils were synthesised as transparent and flexible thin monolithic films. Depending on the salt concentration, the Zn-doped hybrids were produced as amorphous or crystalline POE/Zn(CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub> complexes of unknown stoichiometry. At 30 and 100 °C, the most conducting di-ureasils have ionic conductivities of  $3 \times 10^{-6}$  and  $7 \times 10^{-5}$  S.cm<sup>-1</sup>, respectively. The highest ionic conductivity found for Li-based di-ureasil is  $4.0 \times 10^{-6}$  and  $6.7 \times 10^{-5}$  Scm<sup>-1</sup> (at 35 and 104 °C, respectively) and the electrochemical stability domain spans 7 V versus Li<sup>+</sup>.

*Integrated Optical Devices.* The development of organic-inorganic di-ureasils modified by zirconium n-propoxide (ZPO) stabilized with metacrilic acid to produce cost effective integrated optics (IO) devices was continued. Waveguide channels with Gaussian profile and low surface damage rugosity (below 1 nm) were produced through the exposure to an Ar-ion laser (244 nm) of the di-ureasils modified with  $Zr(i-OPr)_4$ . The existence of guided modes within the channel was demonstrated. A diffraction grating was written using a modified Talbot interferometer. One phase mask was used to split the incident beam in the ±1 orders signals. The grating was characterised though atomic force microscopy revealing 500 nm pitch and low rugosity values (below 1 nm).

*Semiconductor Heterostructures.* The correlations between the nanostructure and optical properties on epitaxial thin films and low dimensional heterostructures of wide-bandgap semiconductors with technological interest for light emission were investigated. Device structures, which include single- and multiple-quantum wells (MQWs) were studied, in order to gain insight on the effects of strain and composition on various material physical properties which are relevant in terms of device performance and functionality.

The controlled integration of colloidal nanocrystals and semiconductor thin films was started. With such a dual approach, where flexible wet chemistry is combined with traditional semiconductor processing, this work aims to develop the fundamental knowledge to pursue alternative ways to create novel heterostructures that incorporate colloidal nanocrystals. Specifically, InGaN-based MQWs and various nanocrystals and quantum dots (QDs) were combined in such a way that the most favourable properties of each material system can be exploited. Preliminary results indicate that nanometer-scale control of the position of various colloidal nanocrystals at the surface of the nitride-based heterostructures can be achieved.

*C60 Phase Transitions Under High-Pressure.* The search for new C60 phases above 100 kBar, the characterisation of their corresponding pressure-temperature phase diagrams and the determination of their structures were carried out. Preliminary studies on metal-doped carbon clathrates and on the polymerisation of C60 molecules inside carbon nanotubes (peapods) were performed.

## ELECTROCERAMICS

*Microwave dielectric materials.* The crystal structure of dielectric ceramics in the  $(1-x)La(Mg1/2Ti_{1/2})O_3 - xBa(Mg_{1/2}W_{1/2})O_3$  [(1-x)LMT-xBMW] system was studied by powder XRD. LMT and BMW form solid solutions in the whole compositional range. Increase of BMW content results in two structural transformations: continuous

P21/n  $\rightarrow$  I2/m, at x about 0.2, and discontinuous I2/m  $\rightarrow Fm\overline{3}m$ , at x 0.4 - 0.5. The sequence of the phase transitions was compared with structural transformations in other solid solutions between dielectric complex perovskites.

The microwave dielectric characteristics of the LMT-BMW ceramics were evaluated as a function of their composition. The observed structure sequence and structure-dependent dielectric behaviour were considered and analyzed in respect to an order of the transition between anti-phase tilted and untilted configurations as well as a probable clustering in the system.

The  $(1-x)La(Mg_{1/2}Ti_{1/2})O_3 - x(Na_{1/2}Bi_{1/2})TiO_3$  perovskite ceramics, [(1-x)LMT-xNBT] ( $0 \le x \le 1$ ), were prepared by the conventional mixed oxide method and Pechini route, and their dielectric properties were investigated. The crystal structure of the solid solutions was investigated by XRD and the sequence of composition-driven structure transformation was revealed: P21/n ( $0 \le x \le 0.2$ ), a coexistence of *Pnma* and  $R\overline{3}c$  (in the vicinity of x=0.3), and  $R\overline{3}c$  ( $0.4 \le x \le 0.90$ ). The crystal structure symmetry of the NBT-rich compositions ( $0.95 \le x \le 1$ ) was described with

the polar R3c space group, which allows accounting for both antiphase oxygen octahedral tilting and A-site cation displacements.

The dielectric response of the ceramics was measured as a function of temperature and composition. The range between x=0.3 and 0.4, where the temperature coefficient of the resonant frequency passes zero, is associated with a discontinuous phase transition between orthorhombic and rhombohedral structure modifications. The compositional variation of the fundamental dielectric parameters, estimated at different frequency ranges, was analyzed in relation to the crystal chemistry of the system. The LMT-NBT solid solutions sided with sodium bismuth titanate exhibit the features typical of relaxor ferroelectrics. The temperature of their dielectric permittivity maximum does not change with increasing substitution rate. As the NBT content increases, the frequency-dependent dielectric peaks flatten. The compositional evolution of structure and dielectric characteristics of the ceramics were discussed in respect to size, charge and polarizability of the cations involved.

Dielectric properties of  $(1-x)La(Mg_{1/2}Ti_{1/2})O_3 - xLa_{2/3}TiO_3[(1-x)LMT-xLT]$  ceramics ( $0 \le x \le 0.52$ ) were studied at radio, microwave and far infrared (FIR) frequency ranges. The crystal structure sequence in (1-x)LMT-xLT reported by different authors was revisited. FIR spectroscopy was used to characterize the lattice contribution to
the dielectric response at microwave frequencies. The complex dielectric function was evaluated from the reflectivity data and extrapolated down to a gigahertz range. Compositional variations of the fundamental microwave dielectric parameters estimated by different methods were compared and discussed. The dependence of the quality factor on the composition in LMT-LT was interpreted in terms of the reduction of spatial phonon correlations originated from the increasing amount of La vacancies. This approach accounts for the compositional behaviour of the dielectric loss commonly observed in a number of microwave mixed systems.

The mixing of compounds with different crystalline structures and dielectric characteristics and its effect on the electrical properties was one of the targets last year. The perovskite structure adapts to all the performed manipulation of the cations, leading to complete solid solutions in the whole compositions range by reconstructing the crystal structures and, thus, the lectrical properties undergo smooth variations, except when phase transitions are first-order.

The other target was to explore compounds with modified crystalline structures. In this case, it was possible to prepare compounds with crystal structures with comparable characteristics (superstructures) and a new family of compounds was produced. La6Mg<sub>4</sub>Ta<sub>2</sub>W<sub>2</sub>O<sub>24</sub> is representative of this family,  $A_{3n}B'_{2n}B''_{2n}O_{12n}$  homologous series with n = 2.

*Ferroelectric Ceramics.* A model for the calculation of spontaneous polarization of Bi layered structure ferroelectric (BLSF) ceramics as a function of their texture degree was developed. The model uses a general formalism based on the texture analysis via an orientation distribution function. The March-Dollase equation fits the measured texture distribution because its fitting parameters can be related to experimentally-measurable stereological values obtained from SEM images. The results were applied to the SrBi<sub>2</sub>Ta<sub>2</sub>O<sub>9</sub> (SBT) system, a well known member of the BLSF family. For this purpose, textured SBT ceramics were produced by templated grain growth. Enhanced ferroelectric properties were measured in specific directions revealing the effects of texture development. The model predicted values for spontaneous polarization as a function of the degree of texture were compared with those measured from the hysteresis loops.

The dielectric response in the THz spectral range of the following Bi-layered ferroelectrics with Aurivillius structure was sutied:  $Bi_4Ti_3O_{12}$ ,  $SrBi_2Ta_2O_9$ ,  $SrBi_2Nb_2O_9$ , a relaxor ferroelectric  $BaBi_2Nb_2O_9$ , and intermediate-type compound  $Sr_{0.5}Ba_{0.5}Bi_2Ta_2O_9$ . The lowest-frequency polar phonons were studied by means of the time-domain THz transmission spectroscopy in the frequency range 0.1–2 THz at temperatures 10–950 K. Although previous structural studies suggested a displacive character of the structural phase transitions, no soft-mode anomalies were observed in our THz spectra near  $T_c$  in any of the investigated compounds. A gradual and only partial softening of

the lowest-frequency polar phonon was revealed during heating. Dielectric anomalies near  $T_c$  in all the compounds are caused by slowing down of relaxations, directly observed in some cases below the polar-phonon range. The ferroelectric transitions are, thus, not classically displacive. In analogy to other relaxor ferroelectrics, the existence of dynamic polar clusters is suggested to be the origin of such relaxations. Ferroelectric transitions are connected with an abrupt freezing and rise of these clusters into domains and the classical division of phase transitions into displacive and order-disorder is not sufficient.

*Ferroelectric Fibers, Single Crystals and Films.* The hybrid sol-gel method was used to deposit piezoelectric layers on optical fibers. To overcome the problem of mechanical stress, special buffer and electrode layers were designed that allow crack-free films to be deposited. The used hybrid sol-gel route allowed incorporating of presintered PZT particles that are 'glued' by the sol-gel solution. Multiple infiltration procedure was applied to remove undesired porosity and decrease the roughness of the coating. The films demonstrated high dielectric, ferroelectric and piezoelectric properties. Driving of the fibers by the applied electric field to the PZT film was demonstrated.

The domain structure of high-quality SBT single crystals obtained via a high-temperature self flux solution method was investigated by XRD and piezoelectric force microscopy. Two types of domains were revealed at room temperature, 180° domains and ferroelastic 90° domains (twins), forming a well-defined 'herringbone' structure with flat 90° walls. This SBT complex domain pattern was attributed to separate ferroelastic and ferroelectric phase transitions occurring as a two-stage process.

SBT seeds were used to produce SBT thin films with improved properties. The comparison of the results obtained by characterizing seeded and unseeded films via Rutherford Backscattering and Particle Induced X-Ray Emission techniques revealed an effective modification of the substrate-thin film interface by the presence of the seeds. The films synthesized without seeds show the formation of an interface of variable content and/or shape, whereas the use of seeds contributes to a higher stability of the film and electrode constituents, thus suggesting an inhibition effect of the seeds regarding interface reactions. The seeds also improve the homogeneity of the thin-film surface microstructure as well as the ferroelectric properties: the macroscopic P-E hysteresis loops and the local piezoloops showed that seeded films present larger remnant polarization and larger average remanent d33 than their unseeded counterparts. The barrier-like role of the seeds, while preventing the inter-diffusion between films/Pt-layer, helps preserving the thin film composition improving the electrical properties.

Thin PZT films 0.5-1.0  $\mu$ m thick were deposited on substrates of glass ceramics and silicon by magnetron sputtering, at a low temperature, and then annealed at 550 °C. The studies were carried out for films sputtered from

targets of stoichiometric composition and targets with an additional content of 10% PbO and 3% Nb<sub>2</sub>O<sub>5</sub>. Only films sputtered from non-stoichiometric targets possess polarization asymmetry and self-polarization, which was investigated by several techniques including SFM. These studies revealed the existence of an internal field created by negative charges on the lower interface with self-polarization vector directed toward the lower electrode. These data suggest that the PZT film has *n*-type conductivity, which is due to oxygen vacancies caused by the excess of either PbO or Nb in the film. An increase in the substrate temperature from 200 to 300 °C during the film deposition resulted in the reversal of self-polarization due to the change from the *n*- to *p*-type conductivity. The dependence of self-polarization with the composition was explained based on the space-charge model.

BaNd<sub>2</sub>Ti<sub>5</sub>O<sub>14</sub> (BNT) films 12 to 52 mm-thick were fabricated on platinum metallic foils by electrophoretic deposition (EDP). A 52  $\mu$ m-thick BNT film exhibit a dielectric constant and a loss tangent of 107 and 0.0006 (Q of 1600) at 1 MHz, respectively. The variation in permittivity was less than 0.02 % at a bias voltage ±8 kV/cm. The change of film permittivity with the temperature in the range 30-120 °C is below +58.5 ppm/°C, indicating a good thermal stability. The higher dielectric constant, high Q, good bias and temperature stability make EPD derived BNT thick films on metallic foils attractive candidates for new microwave communication devices.

*Incipient Ferroelectrics.* It was shown that the microscopic mechanism of the dielectric relaxation in Mn-doped strontium titanate (SrTiO<sub>3</sub>, ST) ceramics is associated with the off-center displacements of  $Mn^{2+}$  ions. This was accomplished by studying dielectric properties and electron spin resonance spectroscopy in combination with XRD and SEM of Sr<sub>1-x</sub>Mn<sub>x</sub>TiO<sub>3</sub> samples sintered in air, oxygen and nitrogen, at 1500 °C. It was shown that manganese is incorporated into the perovskite lattice of ST, preferably as  $Mn^{2+}$  at Sr sites. A small amount of  $Mn^{4+}$  at Ti sites is also detected when samples are fired in air or oxygen flow. The concentration of  $Mn^{4+}$ (Ti) is the highest for sintering in oxygen. Firing in a reducing atmosphere (nitrogen), results solely in incorporation of  $Mn^{2+}$  at Sr sites. Correspondingly, the dielectric relaxation observed in Sr<sub>1-x</sub>Mn<sub>x</sub>TiO<sub>3</sub> markedly increases in intensity and slightly shifts towards higher temperature for ceramics sintered in nitrogen as compared to those fired in air or oxygen.

The effect of Ca doping (x = 0.003-0.30) on the structural properties and dielectric response was investigated for ST ceramics, prepared by conventional method. No second phases and linear decrease of the lattice parameter with increasing x were detected for the studied samples. Dense microstructure with multimodal grain size distribution and monotonic increase of the average grain size with increasing x were observed by SEM. The dielectric anomaly at 22 K and enhanced tunability at 30-85 K were induced by Ca doping in Sr<sub>1-x</sub>Ca<sub>x</sub>TiO<sub>3</sub> ceramic samples with x = 0.10. For ceramics with Sr<sub>0.99</sub>Ca<sub>0.01</sub>TiO<sub>3</sub> composition the tunability was found to enhance and/or the driving electric field to reduce at 10-30 K. Samples with x = 0.003 and 0.01, show a monotonous increase of low temperature

dielectric permittivity, transforming to an anomaly just at x = 0.04. Such 'late' formation of the dielectric anomaly, comparing with  $Sr_{1-x}Ca_xTiO_3$  single crystals may be explained by the grain boundary effect on the dielectric properties. The boundary effect should decrease with increasing x, due to monotonous increase of the grain size, however more detailed sintering, microstructure and dielectric studies are required to confirm this hypothesis. Dense and homogeneous  $Sr_{1-1.5x}Bi_xTiO_3$  films were prepared by sol-gel on Pt/TiO<sub>2</sub>/SiO<sub>2</sub>/Si substrates. The solid solubility limit of Bi on ST lattice of thin films was determined by RX to be < 3%. For compositions with  $x \ge 0.0267$ ,  $Bi_4Ti_3O_{12}$  starts to appear as an extra-phase. This limit of solid solubility is similar to the one determined for identical  $Sr_{1-1.5x}Bi_xTiO_3$  ceramic compositions. The lattice parameter of Bi doped ST films was observed to increase with the increasing of the Bi content. The dielectric permittivity of  $Sr_{1-1.5x}Bi_xTiO_3$  films at room temperature increases with increasing of the dopant content from x=0.002 to 0.1. This suggests the appearance of a dielectric maximum for Bi doped ST films at low temperature. The loss tangent for Bi doped SrTiO<sub>3</sub> films is smaller than for undoped SrTiO<sub>3</sub> films. These preliminary results indicate that similar structural and dielectric behaviour at room temperature was observed between Bi doped ST films and ceramics.

<u>S2</u>

*Multifunctional Ceramic Films and Composites.* The effect of processing conditions on the phase separation and crystal structure of (*x*)La<sub>0.625</sub>Sr<sub>0.375</sub>MnO<sub>3</sub>–(1–*x*)LuMnO<sub>3</sub> multifunctional composite system was studied. The results confirmed a there is a solid solution of monoclinic phase (*P*1121/*a*), i.e. (La<sub>0.625</sub>Sr<sub>0.375</sub>)*x* Lu<sub>1-*x*</sub>MnO<sub>3</sub> is formed for *x* = 0.980–1.0. For  $0 < x \le 0.975$ , the immiscibility region shows a clear separation of La-rich and Lu-rich phases. The optimal preparation conditions are: sintering at 1250 °C and 1350 °C for samples of monoclinic La-rich phase and for the immiscibility region, respectively.

Another approach was to use SFM to observe mutual conversion of magnetic and electric variables by applying high electric field to the sharp tip. This allows the direct conversion of the mechanical/magnetic/electric energy and will pave the way for 'universal' memory applications. Consider the electric field writing on LSMO-based single crystals with low Sr content where the material is in the insulating antiferromagnetic state. In the initial state, no piezoelectric contrast is observed due to centrosymmetric structure of LSMO, but after the writing with the voltage +30 V a white stripe appears due to the locally induced piezoelectric effect. The hysteresis observed by sweeping the applied bias field attests the material to the ferroelectric class signifying effective local Jan-Teller phase transition into the polar phase. The existence of ferroelectricity was confirmed by the measurements of piezoresponse hysteresis.

*Nanoscale Properties of Ferroelectrics*. Local poling of ferroelectrics by a sharp conducting tip of the scanning force microscope was studied experimentally and theoretically. The formation of the inverse domains under the SFM tip, where the polarization is oriented in the direction *opposite* to that of the poling field, was observed for the first time in bulk ferroelectrics (single crystals of solid solutions PbZn<sub>1/3</sub>Nb<sub>2/3</sub>O<sub>3</sub>-PbTiO<sub>3</sub>). This result confirms earlier findings by others on ferroelectric films, thus proving the universality of the anomalous polarization inversion in ferroelectric media. It is shown that the inverse domain grows with the increase of the poling voltage and duration and remains stable for a long time after the removal of electric field. The growth process was described by a dynamic model assuming that the appearance of inverse domains is due to a local internal electric field directed against the poling one. This field was attributed to the space charge formed beneath the SFM tip due to the injection of charge carriers and their subsequent drift and trapping. Poling voltage and poling time dependences of the domain size were found to be correctly described by the presented model. The observed effect has important implications for the domain engineering and dense data storage in ferroelectric materials.

Epitaxial heterostructures YBa<sub>2</sub>Cu<sub>3</sub>Ox(YBCO)/CeO<sub>2</sub>/NdGaO<sub>3</sub> were prepared on tilted-axes NdGaO<sub>3</sub> substrates using laser ablation technique. Morphology, crystal structure and electrical properties of the obtained films were characterized. The seeding mechanisms are affected by the tilt angle, resulting in superior YBCO films on NdGaO<sub>3</sub> substrates in an intermediate range of tilt angles of 6–14°. The introduction of CeO<sub>2</sub> layer leads to change of the YBCO film orientation: at low deposition rate c-oriented films are formed, while at high deposition rates the film grows with c-axis tilted along the [110] NdGaO<sub>3</sub> direction. Bi-epitaxial films and structures were prepared by removal of part of the CeO<sub>2</sub> layer using ion-beam milling. SFM was used to characterize the surface morphology of the films.

Local piezoresponse of individual grains was studied in polycrystalline PLZT 9.75/65/35 relaxor ceramics using SFM technique. The piezoelectric contrast consisting of irregular (labyrinth-type) domain patterns was attributed to the compositional disorder and quenched random fields caused by high La concentration. A measure of this disorder, the correlation length is readily determined via an autocorrelation analysis function incorporated in SFM software. The analysis of experimental data showed that the correlation length determined at the mesoscale (~200×200 nm<sup>2</sup>) size varies as a function of the position within the grain notably decreasing upon approaching the grain boundary. As a result, the average correlation length was found to increase with increasing lateral grain size saturating at about 100 nm. The nature of the observed grain size effect and its relation with the macroscopic dielectric and ferroelectric properties of polycrystalline relaxors was delineated.

Nanoscale piezoelectric measurements were done on P(VDF-TrFE) films prepared by Langmuir-Blodgett (LB) technique. Polarization mapping, local switching, piezoelectric hysteresis and aging after poling were studied.

High quality ultra-thin P(VDF-TrFE) films with copolymer content of P(VDF-TrFE) 70:30 were fabricated using horizontal Schaefer monolayer transfer setup permitting precise control of the film microstructure. The thickness of the films as confirmed by ellipsometry was ~64 nm. Local switching resulted in the domain patterns with lateral size in the range 50-300 nm depending on the applied voltage and time. Local hysteresis loop (at fixed tip position) demonstrated clear ferroelectric switching with the coercive voltages in the range 10-15 V. Relatively slow aging after poling was observed with the characteristic relaxation time of the order of 1000 s. The obtained results clearly demonstrate that the stable polarization patterns can be created in LB P(VDF-TrFE) films that can be used in memory devices and as nanopatterned templates.

#### MAGNETOSTRUCTURAL MODULATION OF STRONGLY CORRELATED ELECTRIC MATERIALS

*Colossal Magnetoresistive Materials.* A main topic of study is the complex interplay of lattice structure, oxygen vacancy, defects and doping on the properties of CMR manganites. This leads to phase segregation at different length scales: charge or orbital ordered; insulator vs metallic. Ferroelectricity driven by magnetic order or charge/orbital order: multiferroic hexagonal manganites: REMnO<sub>3</sub> (RE=Er, Lu, Eu ferroelectric) and composites LaSrMnO<sub>3</sub>-REMnO<sub>3</sub> (ferromagnetic/ferroelectric). Thin film preparation (sputtering) and structural studies (XRD) for phase purity, lattice parameters and temperature study of structural phase transitions were carried out. Hyperfine local probe using implanted radioactive isotopes at ISOLDE-CERN, with Perturbed Angular Correlation Spectroscopy and Emission Channeling, was performed to provide local and element selective information on doping mechanisms. Other studies include: magnetic properties in the vicinity of phase transitions; application of Landau theory of phase transitions; magnetostructural coupling and application of mean field approaches to the study of magnetic interactions.

Magnetocaloric effect and application to magnetic cooling on manganite samples as La-(Ca,Sr)MnO<sub>3</sub> and Er, Eu doped and derived with vacancies in the A and B sites; effect of lanthanides substitution on the cooling power for near-room-temperature applications. Study of magnetic entropy in competing phase systems (ferromagnetic and charge-order). Intermetallic alloys: Pr (Ni-Co), NiMnGa and metal/metalloid Gd-Si-Ge martensitic transitions and electronic density coupled to magnetic entropy changes. Modelling of magnetocaloric properties with mean field theory.

*Theoretical Approaches to Magnetic Materials Using Generalized Thermodynamics.* Description of complex systems with long-range interactions or distributed characteristic parameters using Tsallis non-extensive statistics. Connection to Griffiths phase and disordered inhomogeneous systems.

*High Temperature Superconductors.* Magnetic studies of oxide superconductor materials prepared by LFZ, phase studies, critical current and their relation with structural and phase characteristics in BSCCO fibres. Magnetic and electrical studies in new MgB2-type superconductors, superconducting fraction and critical currents. Hyperfine studies on Hg-HighTc superconductors and role of oxygen defects in fluorinated compounds.

## ADVANCED MOLECULAR AND SUPRAMOLECULAR MATERIALS

*Cyclodextrins.* New inclusion systems were produced from the reaction of previously synthesised and characterised Ru(II)-thioether/polypirydilic complexes and cyclodextrins. Synthesis of new Ru(II) compounds with aminoacids and/or organometallic fragments was also attempted, in order to obtain new compounds to be tested with cyclodextrins.

A 1:1 inclusion compound between Eu(NTA)<sub>3</sub>·2H<sub>2</sub>O (NTA=1-(2-naphthoyl)-3,3,3-trifluoroacetonate) and octakis(2,3,6-tri-*O*-methyl)- $\gamma$ -cyclodextrin (TRIMEG) was prepared and characterised. The quantum efficiency of the ligand-to-metal energy transfer pathway increased upon inclusion complexation. Although a similar increase was previously found for the corresponding native  $\gamma$ -CD adduct, the efficiency of the Eu<sup>3+</sup> sensitisation was significantly higher with the TRIMEG host. Replacement of the water molecules in Eu(NTA)<sub>3</sub>·2H<sub>2</sub>O by other ligands may help to reduce the non-radiative relaxation of the Eu<sup>3+</sup> centres. The complex Eu(NTA)<sub>3</sub>·(2,2'-bipyridine) was prepared and encapsulated in  $\beta$ -CD to give a 2:1 (host:guest) inclusion compound. Photoluminescence studies showed a change in the emission features upon inclusion complexation, possibly due to a reorganisation of the ligands into a geometry more favourable for interaction with two host molecules.

The carbonyl complexes CpFe(CO)<sub>2</sub>Cl, CpMo(CO)<sub>3</sub>Cl, CpMo(CO)<sub>3</sub>CH<sub>2</sub>CONH<sub>2</sub>, [CpMo(NCMe)<sub>2</sub>(CO)<sub>2</sub>](BF<sub>4</sub>) and [CpMo(2,2'-biimidazole)(CO)<sub>2</sub>](BF<sub>4</sub>) were immobilised in  $\beta$ -cyclodextrin ( $\beta$ -CD) and permethylated  $\beta$ -CD (TRIMEB) by tailored methods. In general, adducts with a 1:1 (host:guest) stoichiometry were obtained. Powder XRD indicated that the crystal packing arrangements for the TRIMEB adducts of CpMo(CO)<sub>3</sub>X and [CpMo(NCMe)<sub>2</sub>(CO)<sub>2</sub>](BF<sub>4</sub>) were similar. A hypothetical structural model of TRIMEB·CpMo(CO)<sub>3</sub>Cl was obtained by global optimisation using simulated annealing. The inclusion compounds containing CpMo(CO)<sub>3</sub>CH<sub>2</sub>CONH<sub>2</sub> were used as precursors to catalysts for the liquid-phase epoxidation of cyclooctene using t-BuOOH. The results indicate that the  $\beta$ -CD adduct has potential to be used in heterogeneous solid-liquid systems, while the TRIMEB inclusion compound is more suited to homogeneous or liquid-liquid biphasic systems.

Research was carried out on the competition between the hydrated fluoride anion and hexanoic acid for inclusion in  $\beta$ -Cyclodextrin in aqueous solution using <sup>1</sup>H-NMR. How  $\beta$ -cyclodextrin affects oxygen solubility in aqueous solutions of sodium perfluoroheptanoate was an issue also studied.

Oxomolybdenum Molybdenum(VI) oxides Catalysts. bearing 1,4,7-triazacyclononane and 1.1.1tris(aminomethyl)ethane ligands with the general formula  $[MoO_2Cl(L)]Y$  (Y = Cl, BF<sub>4</sub>) were prepared and examined as catalysts for the liquid phase epoxidation of various olefins, using *tert*-butyl hydroperoxide as the oxidant. Tricarbonyl complexes of the type  $LM(CO)_3$  (M = Mo, Cr) bearing tridentate ligands (L) were also prepared and evaluated as catalyst precursors for the epoxidation reaction. In related studies, the 4-coordinate (tetrahedral) compound  $MoO_2(OSiPh_3)_2$  and the 6-coordinate (distorted octahedral) complexes  $MoO_2X_2L_2$  [X = Cl,  $OSiPh_3$ ;  $L_2 = 2,2$ '-bipyridine or a chiral bis(oxazoline) ligand] were prepared and examined as catalysts for the epoxidation of olefins such as cyclooctene and *trans*-β-methylstyrene. Direct grafting of the bis(chloro) complex containing the chiral bis(oxazoline) ligand onto the ordered mesoporous silica MCM-41 gave a recyclable heterogeneous catalyst for the epoxidation of cyclooctene.

*Metal Complexes of Bio-Inorganic Interest.* The fragmentation patterns and the identification of the hapticity changes in solution of Ru(II) complexes, when N-N = trispyrazolylmethane, were studied by mass spectrometry, mainly by ESI-MS, and a mechanism was proposed. The interaction of different Ru(II) complexes with tetramer duplexes have been also studied and the type of DNA-bases interactions has been assigned.

In order to assess the mechanisms of the carcinogenicity and mutagenicity of chromium, some chromium compounds were synthesised, characterized and in vivo tested.

#### AREA 2 – ADVANCED MATERIALS FOR INDUSTRIAL APPLICATIONS

# **REACTIVE CERAMIC COMPONENTS FOR PROCESS CONTROL**

*Materials for High-Temperature Electrochemical Applications.* The activities were focused on studies of solid electrolytes, electrodes and other materials for fuel cells, and other high temperature electrochemical applications. Mechanisms of ionic conduction on oxide materials were examined with an emphasis on oxygen ion conductors with fluorite, pyrochlore and apatite types, and also cerate protonic conductors. The work on fluorites was mainly dedicated to ceria-based materials with different lanthanide additives and sintering additives to lower the sintering temperature and change the grain boundary behaviour. For silicate-based apatites we attempted to prevent long term degradation in fuel atmospheres by partial substitution in the La-site (*e.g.* Ce) or Si-site (Al, Fe, Ti, Zr). The use of protecting layers is being considered. Different experimental methods were used to re-examine the transport properties of protonic conductors, namely the co-existence of protonic and oxygen ion conduction, and the onset of electronic contributions. Work on potential electrodes for solid electrolyte cells included novel concepts of cathodes with high oxygen storage ability (YBa(Co,Fe)<sub>4</sub>O<sub>7</sub>, Ce(Pr,Zr)O<sub>2-d</sub>), and materials with two co-existing redox pairs (Sr<sub>1-x</sub>Ce<sub>x</sub>Mn<sub>1-y</sub>Al<sub>y</sub>O<sub>3-d</sub>). Work on mixed conducting components of anodes included nanostructured materials. Glass-ceramic seals were prepared and characterized in terms of crystallization and sealing ability, electrical short-circuiting and electrochemical permeation.

*Mixed Conducting Materials and Catalysts For Oxygen Separation or Partial Oxidation of Hydrocarbons.* Work comprised a wide range of mixed conducting materials based on  $K_2NiF_4$ -type nickelates and perovskite-type LaFeO<sub>3</sub>, LaCoO<sub>3</sub>, SrFeO<sub>3-d</sub>, LaAlO<sub>3</sub>, LaGaO<sub>3</sub>, with partial substitution in A- and/or B-sites by acceptor or donor-type additives, including co-additives. The main effort was focused on interrelations between transport properties, redox behaviour, chemical and thermomechanical stability, phase transformations, and electrocatalytic activity. A wide range of experimental techniques was used, as well as structural refinement and atomistic simulations for selected materials. Less common features were also explored in mixed conductors and potential catalysts, including off-stoichiometry to the onset of minor second phases (e.g La<sub>1-x</sub>Ni<sub>0.5</sub>Ti<sub>0.5</sub>O<sub>3-d</sub>) and interactions between different co-existing mixed valency species (e.g CeNbO<sub>4+d</sub>).

*Microsctructural Effects.* Microstructural effects in solid electrolyte materials and mixed conductors were evaluated both as single phase and composite materials (e.g.  $Ce_{0.8}Gd_{0.2}O_{2-d} + La_{1-x}Sr_xMnO_{3-\delta}$ ). Double layer dense/porous membranes were also prepared, (e.g.  $SrFeO_{3-d}$ - $SrAl_2O_4$ ,  $La_{0.5}Sr_{0.5}FeO_3$ - $SrAl_2O_4$ ,  $La_{0.5}Sr_{0.5}FeO_3$ - $SrAl_2O_4$ ,  $La_{0.5}Sr_{0.5}FeO_3$ - $Sr_{1-2}Sr_$ 

 $_{x}$ Fe(Al)O<sub>3</sub>). Different powder preparation methods (glycine-nirate and spray pyrolysis), ceramic processing or layers with different sinterability were used for this purpose. We also attempted to improve the thermomechanical behaviour (thermal shock, toughness, hardness) and stability under reducing conditions, without affecting the permeability and electrocatalytic activity. Materials interactions in composites were also studied, from modest compositional changes (addition of sintering additives) to formation of new phases with major changes in composition. One analysed effects of powder or ceramics processing conditions on phase preservation, and ability to adjust compositional gradients at grain size level (e.g. core-shell structures).

*Materials For Other Electrochemical Technologies.* Electronic ceramic conductors such as  $Ln_{1-x}A_xCo_{1-y}M_yO_3O_{3-d}$  cobaltites, with Ln = Pr, Nd, Sm and A= Sr, Ca, Ba, M=Fe, Ni, Ti and nickelates  $La_2Ni_{1-y}M_yO_{4+d}$  have been studied as potential electrodes for aqueous electrochemical applications in alkaline conditions. One attempted to establish correlations between the electrode performance and the transport properties, oxygen stoichiometry, and stability of these materials. One also studied the oxygen evolution activity on hydroxide or oxy-hydroxide layers deposited onto metallic substrates.

#### CERAMIC COMPOSITES AND ULTRA-HARD COATINGS FOR MECHANICAL APPLICATIONS

*Diamond Cotings.* The work was directed towards the production and characterization of nanocrystalline diamond coatings (NCD) for tribological and bio-tribological applications. These studies were based on the optimization of NCD, physical and tribological characterization. Some work was also done on CVD microcrystalline diamond films for mechanical applications and on ceramic-metal composites produced by melt infiltration.

NCD was produced both by microwave plasma-assisted chemical vapour deposition (MPCVD) using Ar/H<sub>2</sub>/CH<sub>4</sub> gas mixtures on Si<sub>3</sub>N<sub>4</sub> by a conventional continuous, and by a new pulsed regime and by hot-filament chemical vapour deposition (HFCVD). Optimization of NCD growth parameters was done for commercial MPCVD and for an in-house built HFCVD reactor, for coating Si<sub>3</sub>N<sub>4</sub> ceramics. Growth rates of up to 1.6  $\mu$ m h<sup>-1</sup> were obtained for NCD coatings with 28 nm crystallite size. The influence of argon content on the deposition atmosphere was directly related to the formation of nondiamond phases, trans-polyacetylene and graphite at the grain boundaries. These lowered tensile stresses, diminishing the risk of delamination.

Tribological characterization of the NCD/Si<sub>3</sub>N<sub>4</sub> samples was carried out using self-mated pairs without lubrication, in samples obtained by MPCVD and HFCVD. Friction values reached a steady-state minimum of 0.02 following a short period with a peak at 0.44. Calculated wear coefficient denoted a very mild regime (K~ $10^{-8}$  mm<sup>3</sup> N<sup>-1</sup> m<sup>-1</sup>) for the self-mated NCD coatings. The predominant wear mechanism was identified as self-polishing by micro-abrasion. The

critical loads of 35-40 N were further optimized by hydrogen plasma etching of the substrates prior to NCD growth by HFCVD, as was demonstrated by the high threshold loads (60 N; 3.5 GPa) for film delamination. A bioactive  $Si_3N_4$ -bioglass composite was also combined with NCD of low intrinsic surface roughness in the range 5 to 15 nm and crystallite sizes of 1 to 30 nm. Depositions were conducted below the bioglass crystallization temperature (637 °C), allowing a highest growth rate of 1  $\mu$ m h<sup>-1</sup>, with Ar /H<sub>2</sub>=0.1. Slow cooling rates after deposition (2 °C min<sup>-1</sup>) prevented film delamination under Brale tip indentations up to 200 N.

CVD diamond was also studied for sharpenable directly coated cutting tool for hard materials. Thick CVD diamond films (150  $\mu$ m) could be sharpened to the desired geometry using grinding wheels. The tools were tested in dry turning of three types of hardmetal at a cutting speed of 20 m min<sup>-1</sup>, feed of 0.1 mm rev<sup>-1</sup> and DOC of 0.2 mm. Wear by micro-chipping of the CVD diamond, results in abrasion of the rake and flank faces by diamond debris changing the cutting edge geometry and increasing the cutting force to values above which the tool fails (F<sub>d</sub>~700 N). Up to 2000 m dry machining length per tool could be achieved before the re-sharpening operation had to be performed. The use of a cutting fluid increases tool life due to reduction of Co adhesion and enhancement of diamond debris removal from the cutting edge. When using smoother CVD diamond films as direct coatings on Si<sub>3</sub>N<sub>4</sub> tool substrates, the turning performance is the best for the more even surface, as a consequence of morphology and surface roughness characteristics, with reduced cutting forces being obtained.

*Other Hard and Ultra-Hard Materials.* Most work on hard materials was related to processing of nitride, carbide or corresponding composite materials as detailed below. The tribological properties of selected materials were evaluated by using sliding tests against steel under dry and unlubricated conditions qualified these materials for wear applications. Some work was performed on ceramic-metal composites produced by melt infiltration.

*Processing Methods.* Colloidal processing studies on oxide ceramics focused on the dispersing ability of  $Cu_2O$ , CuO and  $Al_2O_3$  powders to access the aqueous slip casting processing conditions of the oxides. Some effort were also put on optimising the tape casting process of cordierite-based glass ceramic substrates of low dielectric constant. A significant research activity was devoted to glass-ceramics of different compositions. Anorthite-diopside-apatite materials were studied as multilayer substrates for microelectronic applications, co-fired at low temperatures. CaO-MgO-SiO<sub>2</sub> materials with B<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>O, CaF<sub>2</sub> and P<sub>2</sub>O<sub>5</sub> were prepared as candidates for biomaterials applications. Monomineral glass-ceramics of akermanite were also developed.

Innovative methods for protecting AlN powders against hydrolysis and for preparing stable and high concentrated ( $\geq$  50-vol.% solids) aqueous suspensions for colloidal processing (slip casting, tape casting, pressure casting, etc.)

and for granulation of powders for dry pressing technologies have been developed. The formulations include the AlN powders and sintering additives, types and amounts of the processing aids (dispersants, binders, plasticizers) required for each consolidation technique. Green densities higher than 70% of the theoretical density was obtained by slip casting, and full dense ceramics were obtained by pressureless sintering at 1750 °C for 2 hours.

Ceramic bodies of  $\alpha$ - and  $\beta$ -sialon were colloidally processed in organic media. The same organic media proved to be suitable for the fabrication of Si<sub>3</sub>N<sub>4</sub>-SiC nano-composite ceramics through temperature induced gelation and for enabling liquid phase sintering at 1850 °C. Rod-like  $\alpha$ -SiAlON and of  $\beta$ -SiAlON powders stabilised with single and multi-cations were prepared by combustion synthesis (CS) and used as reinforcing elements of structural sialon-based ceramics densified by pressureless sintering. CS was also used to study the factors affecting the morphology of AlN and of TiN microcrystals.

Additional contributions were related to processing of traditional ceramics and recycling of industrial wastes. This comprised effects of sodium hexametaphosphate on the rheological behaviour of kaolin dispersions, the influence of  $Li_2O$ -doping as auxiliary flux on the properties and firing of triaxial porcelain bodies, formulation of cordierite ceramics based on Al-rich anodising sludge, and insulating foams based on glass residues.

*Corrosion Protection Methods.* The activity was devoted to different aspects of the corrosion protection for various metallic products. Different components of the protective system, such as surface pre-treatments, anti-corrosion inhibitors, organic polymer coatings and the surface modification top layers were investigated in terms of their applicability for specific metals and alloys. Special attention was paid to the deeper mechanistic understanding of the coatings degradation and the corrosion and inhibition processes. Another topic started last year was the application of localized techniques to study the corrosion processes on nano- and micro-scale.

The main direction of the research activity of the group was focused on the development of novel environmentalfriendly active corrosion protection systems based on the self-healing provided by intelligent release systems incorporated into the coatings. The polyelectrolyte, in-situ formed oxidenanoparticles and nanostructured porous layers impregnated with corrosion inhibitors were used to produce controllable nano-containers preventing a negative effect of the inhibitor species on the stability of the coating matrix. The use of nano-scaled reservoirs helps in uniform distribution and controllable release of the corrosion inhibiting species. This allows the introduction of different inhibitors in the polymer matrixes without the negative effect of the inhibitor on the stability of the coating and without the deactivation of inhibitor originated from its interaction with polymer. Much attentioon was payed to the development of 'intelligent' reservoir systems which may be used in different coatings. The permeability of polyelectrolyte shells strongly depend on pH and can confer the controllable release of the inhibitor triggered by the corrosion induced change of pH.

Hybrid organosilane and the sol-gel derived composite thin films were investigated as pre-treatments for aluminium alloys and galvanized steel. Sol-gel films were doped by the in-situ formed oxide nanoparticles which enhance the corrosion protection of hybrid coatings. Organosilane and hybrid sol-gel films developed provide long-term corrosion protection. Doping of the hybrid films with different active components offers additionally the active corrosion protection component to the developed coatings. These coatings are promising candidates to substitute chromates in many corrosion protection systems.

A set of organic compounds was investigated for corrosion inhibition of aluminum and magnesium alloys, to replace carcinogenic chromate, which will be banned in Europe by 2007. 8-hydroxyquinoline was found to be very effective corrosion inhibitor for aluminum alloy 2024 and magnesium alloy AZ31. Corrosion mechanisms of aluminum alloys demonstrated crucial role of cathodic intermetallic inclusions in the localized corrosion attack, and contributes to the search of new corrosion protection approaches.

The weathering stability of the plasma polymer films deposited to tailor the surface properties of coil coatings was investigated demonstrating strong dependence of the plasma polymer films stability on the film composition. The plasma parameters such as pressure and power play a role in the stability of the obtained layers. Deposition of thin plasma polymer layer leads to partial degradation of barrier properties of coil coatings. Atomic oxygen in plasma plays a critical role on barrier degradation.

Weldable primers for automotive applications were developed. One studied effect of organic corrosion inhibitors on corrosion processes on the galvanized steel coated with zinc-rich weldable primers. Benzotriazole and mercaptobenzothiazole were very effective corrosion inhibitors and will be introduced to primer formulations to improve self-healing ability.

# AREA 3 – CHEMISTRY AND TECHNOLOGY OF POLYMER AND LIGNOCELLULOSIC MATERIALS AND BIOPOLYMERS

#### MACROMOLECULAR MATERIALS AND LIGNOCELLULOSICS

*Lignocellulosics.* A study was performed on the use of chitosan, with different degrees of deacetylation and different molecular weights, in the processing of polymer blends (*e.g.* with other natural polymers or their derivatives), complexes (*e.g.* with paper) and composites (*e.g.* with natural fibres) and on the preparation of unexplored chitosan derivatives. The inclusion of commercial chitosans in paper through diffusion from aqueous solution, was carried out and the properties of the ensuing complexes assessed. In order to establish the extent of insertion and actual mapping of the chitosan molecules in the paper sheet, a fluorescent material was synthesized by appending an appropriate chromophore to about 4% of the primary amino groups of chitosan. In a different vein, chitosan was oxypropylated in a bulk heterogeneous process, yielding a viscous polyol mixture whose structure and properties are being evaluated.

Another new project focuses on the application of the reversible Diels-Alder reaction to the synthesis of novel polymeric materials bearing furan moieties. The working hypothesis behind this approach is that a number of promising macromolecular architectures may be built using monomers incorporating complementary diene (furan) and dienophile (maleimide) functions through multiple adduct formation and that all of them can then either be turned into thermally stable structures by the aromatization of the adduct or, more interestingly, be subject to thermal reversibility (i.e. back to the starting monomeric entities) for recycling or other more technical applications like solvent-free printing inks, self-mendable materials, etc. Bifunctional monomers have thus been synthesized, both of the A-A (difuran) and B-B (bismaleimide) and of the A-B type. Their structure has been confirmed in all instances and their larger scale synthesis and purification are in progress.

The search for original ways of valorizing suberin has entered a critical phase with a systematic approach of the different routes leading to polyesters based on the long-chain components bearing OH and COOH moieties in different numbers and molecular distribution. Suberin components can be isolated with free COOH groups or their corresponding methyl esters, depending on the fragmentation technique adopted. The polycondensation methods tested were therefore based on both direct esterification and transesterification. Model compounds, *viz.* long-chain diacids, diols and hydroxyacids, were used to assess the viability of each method before switching to the actual suberin monomer mixture. It was thus found that a number of procedures, like polytransesterification, direct polycondensation catalyzed by acidic surfactants or enzymes, gave good results, albeit with polyesters of modest molecular weights of a few thousand. The chemical characterization of other by-products from cork processing

was pursued, including low molecular mass components (mostly terpenes and polyphenols), lignin and polysaccharides.

The studies on the controlled heterogeneous modification of cellulose fibers with fluorinated reagents (eg. trifluoroacetic anhydride, pentafluorobenzoyl chloride and trifluoropropanoyl chloride) for the development of highly hydrophobic cellulose-based materials were pursued. All reaction systems were optimized and the new fluorinated cellulose fibers derivatives were extensively characterised. The hydrolytic stability of these new cellulose derivates was also assessed. New routes for the controlled heterogeneous modification of cellulose fibers were also initiated, including reaction with reagents bearing isocyanate and silane groups and the enzymatic esterification with fatty acids.

Research on the synthesis, characterization and assessment of properties of nanoparticles/cellulose hybrids was pursued. The photocatalytic activity of  $TiO_2$  based materials started to be evaluated. The degradation of some dyes (methylene blue, methyl orange) in aqueous solution in the presence of the hybrids under UV radiation was evaluated. Preliminary results show that these materials are effective in the dyes degradation. The potential antibacterial activity of these materials started to be studied in cooperation. Due to the photoactive semiconductor nature of  $TiO_2$ , the direct contact between this oxide and cellulose may limit the applications of these materials for long periods, in particular if they are exposed to light sources. Because of this, a new strategy for the preparation of these hybrid materials was initiated. Cellulose fibres were coated using silane coupling agents (PTMS and OTMS) followed by deposition of  $TiO_2$  nanoparticles. These new hybrids showed a high hydrophobicity, opening the possibility for higher compatibility between hybrids and polymeric matrixes in composites preparation.

Silver nanoparticles generated *in situ* in the presence of cellulose (vegetal or bacterial) were prepared, with different particle sizes and loads. These materials were tested for surface enhanced Raman spectroscopy and antibacterial applications, with very promising results.

The optimization of  $CaCO_3$ /cellulose hybrids preparation is also under investigation. The control of synthesis parameters and the influence of the carboxylic content in the modified cellulose fibres are being assessed. Scale-up procedures are being implemented in order to test the possible application of these hybrids in paper and polymer-based materials.

The work on polyoxometalate (POM) catalysis in oxygen delignification of kraft pulp using the laccase as biocatalyst for the POMs re-oxidation was continued. New polyoxometalate-laccase integrated system (PLIDS) employing polyoxometalate  $[SiW_{11}V^{V}O_{40}]^{5-}$  and laccase of *Trametes versicolor* for the continuous delignification of eucalypt kraft pulp was developed. Pulp was delignified in a batch reactor containing catalytic amounts of  $[SiW_{11}V^{V}O_{40}]^{5-}$  at *ca.* 90 °C under atmospheric pressure. Re-oxidation of reduced POM with laccase was carried

out at 45 °C in a separate aerated bioreactor coupled with an ultrafiltration tubular ceramic membrane. The later allowed separation of laccase from re-oxidized POM, which was supplied in turn continuously to the delignification reactor. PLIDS allowed sustainable pulp delignification with minimal degradation of polysaccharides. The implementation of PLIDS, replacing the first chlorine dioxide stage (D) in conventional DEDED bleaching sequence, showed almost 60 % of chlorine dioxide savings with strength properties of the bleached pulp (90% ISO) similar to those obtained after the conventional bleaching.

The preliminary research work was performed on the kinetics of the oxidation of substituted phenols with either vanadium polyoxotungstate,  $[\alpha-\text{SiV}^{V}W_{11}O_{40}]^{5-}$  (viz.  $SiW_{11}V$ ), or manganese polyoxotungstate,  $[\alpha-\text{SiMn}^{III}W_{11}(\text{H}_2\text{O})O_{39}]^{5-}$  (viz.  $SiW_{11}Mn$ ), in aqueous solution at pH 4.

Aiming to promote a better understanding of the oxidative delignification mechanisms with polyoxometalates and its re-oxidation mechanisms, the electronic tongue multisensor system wasproposed for the detection of metal-oxygen cluster anions (polyoxometalates) containing vanadium (IV/V) atoms. Sensitivity of a variety of potentiometric chemical sensors with plasticized polyvinyl chloride and chalcogenide glass membranes was evaluated with respect to vanadyl/vanadate anions, decavanadates and a series of Keggin-type polyoxometalates (POM) such as  $\alpha$ -[SiW<sub>11</sub>V<sup>IV</sup>O<sub>40</sub>]<sup>6-</sup>,  $\alpha$ -[SiW<sub>11</sub>V<sup>V</sup>O<sub>40</sub>]<sup>5-</sup>,  $\alpha$ -[BW<sub>11</sub>V<sup>IV</sup>O<sub>40</sub>]<sup>7-</sup>,  $\alpha$ -[BW<sub>11</sub>V<sup>V</sup>O<sub>40</sub>]<sup>6-</sup>,  $\alpha$ -[PW<sub>11</sub>V<sup>IV</sup>O<sub>40</sub>]<sup>5-</sup> and  $\alpha$ -[PW<sub>12-n</sub>V<sup>V</sup><sub>n</sub>O<sub>40</sub>]<sup>(3+n)-</sup> (n=1,2,3). Such sensor array was able to distinguish different vanadium complexes allowing their simultaneous quantification in binary (V(IV)/V(V)) mixtures.

The study on the paper surface modification by deposition of *in situ* formed inorganic pre-polymers obtained with sol-gel process was initiated. The main attention was paid to the optimization of synthesis of sol-gel formulations composed of tetraethylortosilicate (TEOS) and a series of its monosubstituted analogs (alkyl and amine moieties among others) in aqueous solutions and to the characterization of organic-inorganic hydrids (OIH) obtained. The methods for the deposition of these formulations on the paper surface were developed (using impregnation press, by spray and using sizing press) and treated papers were analysed (image analyses for ink jet printing, contact angle, surface energy, etc.).

Work was done on the chemical analysis and structural characterization of macromolecular components and extractives from different morphological parts of banana plant (*Musa acuminata* Colla var. *cavendish*) aiming to promote the valorisation of this important regional (Madeira) agricultural waste material. Particularly, the study on chemical composition and structure of components from different morphological parts of 'Dwarf Cavendish' banana plant (petioles/midrib, leaf blades, floral stalk, leaf sheaths and rachis) was carried out aiming to evaluate their potential as eventual raw materials for the chemical processing.

A study was carried out on the decolourisation and detoxification of kraft effluent by *Trametes versicolor*. It was proved that this fungus possess an excellent capacity of development in toxic environments once its cell growth was observed and oxidative enzymatic activity was remarkably increased in presence of effluent and both high decolourization and detoxification parameters were attained.

The studies on ESI-MS application for the structural characterization of lignin were continued. Several fractions of synthetic dehydrogenation polymer (DHP) were fractionated by GPC and analysed by ESI-MS/MS aiming to provide a MS/MS spectra database on the lignin oligomeric structures. ESI-MS/MS technique was used for the first time to assess the molecular mass of hardwood (eucalypt) lignosulphonates (LS) and for their structural characterization.

The investigation of different approaches or process modifications to improve the retention of polysaccharides during *Eucalyptus globulus* kraft pulping was pursued. Particular attention was devoted to the rational addition of antraquinone to the kraft process as a mean to improve pulping yield and, simultaneously, decrease the amount of hydrogen sulphide in the process. Pulps produced with such process modifications were fully bleached and tested for their mechanical properties.

Investigations aiming to understand/improve the retention of polysaccharides during *Eucalyptus globulus* kraft pulping on the pulp fibres surface have been performed. Current research was focused on the analysis of pulps obtained after cooking under different pulping conditions (active alkali (12-17%), sulfidity, etc.) along the last delignification period aiming to estimate the proportion of xylan adsorbed on pulp surface and how this xylan retention is affected by pulping conditions. The amount of glucans in *Eucalyptus globulus* wood (about 4.5 %) was estimated using sawdust extraction with 0.5M NaOH solution at 90 - 120 °C and the structural features were elucidated in glucans-enriched polysaccharide fraction isolated by hot water extraction (120 °C, 2 h). Isolated glucans were characterized by wet chemistry methods and by different 1D and 2D NMR techniques. The major proportion of glucans was comprised by linear (amylose, 20 %) or ramified at *O*-6 (amylopectin, 80 %)  $\tilde{\alpha}(1 \rightarrow 4)$ linked glucans though a small proportion of  $\beta$ -(1 $\rightarrow$ 3)-glucan (< 0.2% on wood weight) have been also detected.

Two new research projects related to the utilization of by-products from the acid sulphite pulping of *E. globulus* wood were started this year. One is related with the production of acetic acid and furfural from condensate of pulping liquor evaporation, while the other deals with the analysis of industrial circuits and utilization of lignosulphonates.

The study on the chemical composition of macromolecular components of the natural hybrid of *Paulownia elongata* and *Paulownia fortunei* was continued. The work concentrated on the study of hemicelluloses structure

(xylan and glucomannan). The preliminary kraft pulping experiments on *Paulownia* wood were carried out aiming to optimize the conditions.

Work was performed on the structural features of pectic polysaccharides of olive pulp cell walls that remained entrapped in the cellulosic residue after sequential extraction of the cell wall material (CWM) with imidazole, carbonate and KOH aqueous solutions. These polymers, obtained after neutralisation and dialysis of an aqueous suspension of the residue (sn-CR fraction), were arabinan-rich pectic polysaccharides. Size-exclusion chromatography showed that sn-CR fractions possessed a bimodal molecular weight distribution. The smaller molecular weight fraction of sn-CR ( $M_w$ =82-135 kD) was independent on the ripening stage of olive fruit, whereas the higher molecular weight fraction showed values of 1.1, 0.9 and 0.7 MDa, respectively, for green, cherry and black olives. De-complexation of sn-CR pectic polysaccharides with a 2 M imidazole solution disrupted the CPPC crystalline network showing the molecular weight decrease to half thus indicating that pectic polysaccharides of sn-CR fraction of sn-CR molecular weight decrease to half thus indicating that pectic polysaccharides of sn-CR fraction of sn-CR molecular weight decrease to half thus indicating that pectic polysaccharides of sn-CR fraction of sn-CR molecular weight decrease to half thus indicating that pectic polysaccharides of sn-CR fraction occurred in olive pulp cell walls as calcium bridged macrodimers.

Work was started on the elucidation of minor structural features in wood hemicelluloses employing ESI-MS/MS and MALDI-TOF/TOF. The main objects were glucomannan and glucuronoxylan from *E. globulus* wood and glucoronoxylan from the natural hybrid of *Paulownia elongata* and *Paulownia fortunei*.

A PhD thesis was completed on the oxypropylation of cork residual powder to obtain polyols to be used in the synthesis of polyurethanes.

*Other Polymer Systems and Materials.* The PhD programme on the preparation of functional polymer based nanocomposites (NCs) using *grafting from* strategies has been concluded including the preparation of block copolymers. The PhD programme on polysaccharides NCs has been extended to chitosan and locust bean gum (LBG). The preparation of films has started.

A joint (UA/TUHH) MSc thesis on the preparation of DWCNTs/ poly(methyl methacrylate) NCs and the study of the mechanical properties was concluded. The results obtained were superior to those reported in the literature due to the *grafting from* strategy followed. Three new joint (UA/TUHH) MSc theses started: (i) on the preparation of SiO<sub>2</sub> @Fe<sub>2</sub>O<sub>3</sub>/PANI NCs; (ii) on CNFs/poly(acrylic acid) and (*iii*) a thesis in collaboration with Weber Cimenfix which aims at studying the influence of polymer ageing on the properties of cement mortars. One MEng thesis has been started on dimensional stability and permeability studies of flexible PUs.

Contributions were made to (i) the demonstration that crystallization from sheared polymer melts saturates; (ii) the demonstration that the polymer melt state responsible for this saturation is the steady state in steady shear flows; (iii) the demonstration of a significant entanglement loss during the transition to the steady state and iv) the

demonstration that a basic assumption of current flow theories is lacking experimental support and that these theories need to be revised.

The predictive, analytical (non-simulative) cooperative segmental theory of materials dynamics continued to be applied to the dynamic behaviour of polymers. The dynamic crossover and the corresponding temperature were shown to be predictable by the theory for amorphous polymers from creep data. The installation of the Laboratory of Thermal Analyses was continued, namely in what concerns the facilities for dynamic mechanical analysis (DMTA) and simultaneous calorimetry and thermogravimetry (DSC/TG).

#### **BIOMEDICAL AND BOIOMIMETIC MATERIALS**

*Biological, Structural and Identification FTIR, NMR and Other Studies.* On the mechanisms of chromium toxicity, chromium compounds, including Cr(V), with biologically relevant ligands were synthesised and characterised. Mice, previously intoxicated with controlled levels of different metal complexes, were used as preclinical models, in order to evaluate the role of the chromium compounds, and the intermediates, following their routes in the so-called 'chromium reduction toxicity mechanisms'. The adverse effects of these compounds were investigated using slices of target organs (kidney, liver and testis), prepared for histological, histochemical and ultrastructural methods. Complementary approaches, such as flow cytometry that allows, in real time, a multiparametric assay of cells, were also used to get more complete insights into the underlying mechanisms of toxicity. The possibility of recovery of these lesions was also investigated. Lesions induced by those compounds were described in mice organs, obviously reflecting changes on the physiology. For example, some hyaline and granular casts and desquamation of tubular epithelium within the renal cortex and hyaline casts in the medulla were revealed in these groups of mice.

The NMR characterisation of biofluids and biological tissues waspursued, involving the construction of compositional databases and determination of 'healthy' profiles for subsequent detection of several disorders under study. Potential biomaterials of interest based on magnetic nanoparticles (magnetite/carrageenan) and on biopolymer/inorganic scaffolds have been prepared and characterised.

NMR-based models for the routine control of beers and brewing process were developed and should be shortly implemented as a service for a national brewing company. Projects on detection of beer ageing and contamination were initiated. Solid state NMR and FTIR have been used to determine the degree of polymerisation and hydrolysis in hydrogels based on vinylacrylate grafted dextrin (highly biocompatible gels). The role of several depolymerising enzymes on the nanostructure of cork was studied by NMR and FTIR methods.

Using NMR pseudocontact shifts for the Ni and Zn/Ni forms of Desulforedoxin the residues involved in structural changes and in the H-bonding network at the metal centre were identified. Using this approach contact shift contributions were estimated and the importance of the H-bonding network at the metal centres for distribution of the unpaired electron density was highlighted. It was found that the DvRd(Ni) PCS tensor is more isotropic than that for Dx(Ni) possibly be due to the higher symmetry of the DvRd centre compared to Dx.

The 3D structure of the novel Heme Binding Protein p22HBP was determined by NMR. Previous work indicating that the protein does not undergo large structural modification on binding was confirmed by the determination of the structure of the bound form.

*Glass and Ceramic-Based Biomaterials.* The work carried out along the past year was focused on the wet chemical preparation and characterisation of pure calcium phosphate powders, namely hydroxyapatite (HAP) and its derivatives (F-HAP, Cl-HAP,  $\beta$ -TCP), and HAP doped with several cations (sodium, magnesium, strontium, and anions, fluorine, chlorine) as well as their biphasic mixtures in different ratios, including nano-sized powders and their in-situ stabilization. Hydrothermal treatments were also used to transform nacreous materials into HAP-based materials. The as prepared powders were used to produce scaffolds with tailored pore size distributions. Following a different route, scaffolds for bone restoration were also produced from cuttlefish bones hydrothermally transformed into HAP-based biomaterials. A new model formulation of a biocompatible glass of the system SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-B<sub>2</sub>O<sub>3</sub>-MgO-CaO-Na<sub>2</sub>O-F was developed and revealed to have interesting bioactive properties *in vitro*.

All glasses and glass-ceramics studied have compositions within the 3 main Si-based systems  $SiO_2-CaO-P_2O_5-MgO, SiO_2-P_2O_5-CaO-MgO-K_2O$  and  $SiO_2-Na_2O-MgO$  and in the Si-free system  $TiO_2-CaO-P_2O_5$ . The research carried out on the above systems had the purposes of (i) studying their structure and establish the relationship with surface reactivity in simulated physiological plasma (ii) understanding the mechanisms of adhesion of apatite layers in acellular media and (iii) controlling the *in vitro* mineralization in physiological fluids. The development of porous glass scaffolds by the salt sintering method was started in a project where the study of the processing variables is one of the key tasks.

Si-based glasses that indicated *in vitro* bioactive behaviour were added to different polymeric matrices (PMMA, PE, SEVA, PHB, PLLA) to obtain novel composites for bone regeneration, cement substitution and drug delivery systems. Organic-inorganic Si-based materials were prepared by the sol-gel process using different silicon precursors. The structure of the hybrid gels and the resulting materials were studied with the aim of obtaining nanocomposites to be used as carriers for cell immobilisation.

A new system for the local delivery of chemotherapy to malignant solid tumours was developed based on apatite particles. The adsorption of the drug 5-Flourouracil (5-FU) on hydroxyapatite (Hap) porous granules produced by spray drying suspensions of Hap nanoparticles was studied at 37 °C. Nanostructured and porous apatite granules produced by spray drying showed a good ability for adsorbing the chemotherapeutic drug 5-FU. The dynamics and equilibrium of the 5-FU adsorption process were suitably described by pseudo-second order kinetic model and Langmuir isotherm respectively. Porous chitosan/brushite composite scaffolds prepared by a freeze-drying technique, starting from brushite suspensions in chitosan solutions showed a regular macroporous and interconnected structure with brushite particles uniformly distributed in the chitosan matrix. Their in vitro bioactivity was revealed by the formation of a hydroxyapatite layer after 24 h of ageing in SBF (simulated body fluid) solutions.

*Other New Biomaterials.* Our activities addressed the enhanced production of lipase in two phase reactors. This is a project related with our activities in studying non conventional solvents, namely fluorocarbon compounds that are here used for oxygenation of bioreactors.

Using a particular type of *Yarrowia lipolytica* it was possible to identify an anomalous behaviour on its adhesion to organic phases and a production of a biosurfactant. The biosurfactant was characterized and studies of surface that lead to the characterization of the Yeast surface were lead.

### PROCESS DEVELOPMENT AND OPTIMISATION

A new UNIQUAC based model was proposed for the description of wax formation in conventional petroleum based fuels and crudes. This model was shown to be superior to the previous versions proposed by us. Other fluid properties where also addressed and a corresponding states model was used to describe speeds of sound, viscosities and surface tensions of hydrocarbons with success. Together with a British consulting company, INFOCHEM UK, a new approach to the prediction of wax formation for flow assurance was developed and reported. A new focus was placed on biofuels. Water solubilities on these fules and their behaviour under low temperatures were investigated.

The thermal characterization of phase change materials (PCM) was carried out. A patent for composites of paraffins and cellulose or cork derivatives was filled. Studies concerning other phase change materials than n-paraffins, such as fatty acids and esters have been conducted.

The studies of unconventional solvents for the development of chemical products and processes focusing on the fluorinated compounds were extended to ionic liquids. Particular emphasis was given to the surface properties, gas solubility and mutual solubilities with water or aqueous solutions.

On polymers and polymer solutions, two main distinct areas were studied: in the laboratory, gas solubility in biodegradable polymer films data were measured in order to define possible modifications to use them in packaging films; a new molecular level model for polymer systems and polymeric solutions was programmed and tested with excellent results.

Several perfluorocarbon systems were experimentally characterized: liquid-liquid equilibria of perfluorocarbons + n-alkanes and perfluorcarbons + water, vapour-liquid equilibria of carbon dioxide + perfluoroalkanes. The structure of the perfluorocarbon liquid phase was addressed by measuring surface tensions and by ab initio calculations to pin point specific interactions. The study of the stability of perfluorocarbon emulsions with several surfactants was also continued.

Furfural is a basic nonpetroleum chemical readily accessible from renewable resources, arising from the acidcatalysed dehydration of pentoses, and has a broad industrial application profile, both as a solvent and as a building block for the preparation of tetrahydrofuran, pharmaceuticals, agrochemicals, fragrances and furan based chemicals. The industrial use of sulfuric acid as the catalyst poses serious operational, safety and environmental problems, and therefore its replacement by alternative 'green' non-toxic catalysts is of high priority. Our recent research has been directed towards finding solutions to this problem.

Aggregates of acid nanosheets prepared by exfoliating crystalline layered metal oxide cation exchangers, such as titanates, niobates and titanoniobates, were found to be more active and somewhat more selective catalysts than microporous AM-11 crystalline niobium silicates, which in turn yielded more furfural than zeolites, such as HY and mordenite (Si/Al  $\approx$  6), under similar reaction conditions. Furfural yields remained practically the same in recycling runs and no water leaching of metals from these catalysts to the reaction mixtures was detected.

On the hydrodynamic behaviour of process-relevant liquid-liquid dispersions, a detailed quantitative description of the drop interaction (breakage and coalescence) processes in a real column extractor was coupled with the flow of both phases, to accurately describe its experimental behaviour.



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# AREA 1 – ADVANCED MICRO- AND NANO-STRUCTURED MATERIALS FOR COMMUNICATIONS TECHNOLOGIES

# MULTIFUNCTIONAL MATERIALS AND ORGANIC-INORGANIC HYBRIDS

*Novel Microporous Materials.* The M-doped (M=Y, Eu) microporous zirconosilicate AV-3 materials, possessing the structure of mineral petarasite and with Zr/M ratio of 4, will be prepared via hydrothermal synthesis and their structure determined. This is expected to be a suitable system to create new multifunctional photoluminescent (PL) materials.

The potential of synthetic microporous (ETS-10, ETS-4 and AM-2) and layered (AM-4) materials will be evaluated for the decontamination of natural waters polluted with low  $Hg^{2+}$  levels, in the presence of the competitive Cl<sup>-</sup>, Ca<sup>2+</sup>, Na<sup>+</sup> and Mg<sup>2+</sup> ions.

Microporous lanthanide silicates  $K_7Ln_3Si_{12}O_{32}\cdot 3H_2O$  (Ln = Sm, Eu, Gd and Tb) will be prepared and characterised (structure and PL properties). Preliminary work suggests that these materials are unique because they possess Ln-O-Ln dimmers, which confers them unusual PL properties.

Vinyl chloride (VC) is the primal compound in the production of polyvinylchloride. Normally, ethyl chloride (EC) is an impurity present in low levels in the VC effluent stream, inhibiting the polymerization reaction. The challenge is to come out with a clean and safe technology, which empowers a cheaper and efficient separation of these compounds in gas phase by means of an appropriate adsorbent that also minimizes undesired emissions. The adsorption of EC and VC on microporous aluminum methylphosphonate polymorph- $\alpha$  will be investigated by performing Grand Canonical Monte Carlo simulations in order to explore the possibility of using this adsorbent for the purification of VC, upholding the selective adsorption of the EC, and eventually to find the best conditions for EC/VC separation. Experimental data will be used to validate the molecular model of the fluid used in the simulations, with the model for this adsorbent taken from our previous work.

Work on zeolitic membranes will continue. The optimization of ETS-10 and AM-2 membranes will be studied in order to improve membrane quality. The work will also be extended to aluminosilicates. Zeolite Y membranes and their silver-modified form will be prepared for the selective separation of olefin and paraffin.

*Mesoporous Materials.* In previous work carried out at CICECO, the ordered mesoporous silica MCM-41 was derivatised with dioxomolybdenum(VI) species by direct grafting with the complexes  $MoO_2X_2$ (tetrahydrofuran)<sub>2</sub> (X = Cl, Br). Depending on the synthesis conditions used, the materials were effective as catalysts for the oxidation of either olefins or alcohols. In an extension of this work, materials prepared using  $MoO_2X_2$ (dimethylformamide)<sub>2</sub>

as the grafting agent will be characterised by various techniques including EXAFS, and examined as heterogeneous catalysts for the liquid-phase oxidation of organic compounds. The synthesis and characterisation of periodic mesoporous organosilicas containing organometallic groups as an integral part of the structure will also be undertaken. Fragments such as  $(\eta^6-C_6H_4)Cr(CO)_3$  will be introduced into these structures by either one-step surfactant-templated hydrolytic polycondensation of functionalised trialkoxysilane precursors or post-synthesis derivatisation.

Work will continue on the preparation of new organic-inorganic precursors aiming the development and design of new periodic mesoporous organosilica (PMO) hybrids with molecular-scale periodicity in the walls. The thermostability, energy and length of the Si-C bond will be studied as a function of the organic group. PMO materials will be prepared, in which the silicon atoms are bounded to one or two organic groups in both powder and oriented film morphology for low dielectric applications.

*Layered Materials.* Work will proceed on the dehydratation of layered lanthanide silicates K<sub>3</sub>[LnSi<sub>3</sub>O<sub>8</sub>(OH)<sub>2</sub>], Ln=Y, Eu, Tb, Er, and the structural characterisation of the obtained small-pore framework (K<sub>3</sub>LnSi<sub>3</sub>O<sub>9</sub>), Ln=Y, Eu, Tb, Er solids, named AV-23, will be been reported. Both materials have a similar chemical makeup and structures sharing analogous building blocks, hence providing an unique opportunity for rationalising the evolution of the PL properties of lanthanide silicates across dimensionality.

Nanoparticles of layered  $Ln_2(SiO_4H)(OH)_2(H_2O)Cl$ , Ln= Eu, Gd will be prepared and characterised. These materials are expected to display efficient and tuneable PL properties, for example energy transfer between different  $Ln^{3+}$  centres, such as  $Gd^{3+}$  and  $Eu^{3+}$ .

In an extension of previous work, the intercalation of oxomolybdenum complexes of dihydroxybenzoic acids into the layered double hydroxide (LDH) [LiAl<sub>2</sub>(OH)<sub>6</sub>]Cl will be studied. A dioxomolybdenum(VI) complex of 2,5pyridinedicarboxylic acid will also be prepared and incorporated into LDHs by ion-exchange reactions with precursor materials in nitrate or chloride form. In related work, LDHs pillared by 2,2'-bipyridine-5,5'-dicarboxylate anions will be examined as solid-state complexation materials for the compounds  $MoO_2(OSiR_3)_2$  (R = methyl, phenyl). Selected materials will be tested as catalysts for the oxidation of organic compounds. In another study, LDHs intercalated by anionic pyrene derivatives will be prepared and their photophysical properties studied.

Study the potential catalytic application of the molybdenum eta3-allyldicarbonyl complexes on the cyclooctene epoxidation with tert-butyl hydroperoxide in homogeneous phase and after immobilisation in pillared clays. We will prepare porous clay heterostructures with organosilica precursors. The solids will be characterised from the point of view of structure and application as adsorbents.

*Nanostructured Materials.* Research on the synthesis and surface modification of quantum dots will be continued, now extended our work to the synthesis of doped nanocrystals of ZnO and ZnS. Also the optical/magnetic properties of several inorganic nanoparticles will be investigated as isolated nanostructures or incorporated in several polymers (natural and synthetic) having in mind diverse applications such as in the paper industry and nanomedicine.

The preparation of novel lanthanide luminescent systems at the molecular or supramolecular level together with systems supported on a nanosized material will be performed. Research will be continued on the synthesis of photoactive lanthanide complexes with aromatic ambidentate ligands, exploring the possibility of formation of multidimensional coordination compounds. The luminescence and structural properties will be thoroughly studied. The possibility of the ligands to sensitize the lanthanide emission will be investigated. The incorporation of those lanthanide compounds into nanosized SiO<sub>2</sub> and other substrates will be explored.

The use of SERS (Surface-Enhanced Raman Scattering) in the study of the interaction of metal nanocrystals with molecular adsorbates will be continued, in particular to explore it as a tool for trace detection using natural substrates. SERS will also be used to characterize systems with potential interest in heterogeneous catalysis. Techniques of infrared spectroscopy applied to surface studies will also be explored.

*Polyoxometalates.* The synthesis by several synthetic procedures and the study of new hybrid compounds with polyoxometalates and organic aromatic moieties, either as cationic species or incorporated in metal complexes, will be continued. Following the studies with pyridine derivatives and aminoacids performed in the last years, the work will proceed with other N,O supporting molecules, like caffeine and others. The obtained compounds will be assessed for their electrochemical or optical properties. Solid-solid solvent free reactions will be extended to the incorporation of inorganic cations (namely group 2 or lanthanide cations) in salts with Keggin polyoxometalates. Studies on homogeneous catalysis will be continued with new substrates, namely 1-ethylnaphtalene, 2-ethylnaphtalene, *p*-cymene, cumene and sec-butylbenzene. The studies on the preparation of silica supported transition metal-substituted polyoxotungstates, to be evaluated as oxidative heterogeneous catalysts, will be continued.

We will continue to investigate the preparation of new organic-inorganic hybrid coordination compounds containing lanthanides, POMs and an organic ligand. The effects of the organic ligand and the POMs on the luminescent properties will be investigated in particular, considering the possibility that they might act as sensitizers of the lanthanide luminescence. The application of the compounds in the preparation of POM based

materials will be explored, namely by the preparation of mono or multilayered nanostructured films, incorporation into nanosized  $SiO_2$  and preparation of polyoxometalate-anion-pillared layered double hydroxides.

The characterization of novel ruthenium tetra-substituted polyoxometalate compounds of general formulae  $K_6Na[SiW_9O_{37}Ru^{III}_4(H_2O)_3Cl_3]$  nH<sub>2</sub>O ( $\alpha$  and  $\beta$  SiW<sub>9</sub>O<sub>37</sub> isomers, **1** and **2**) synthesised via the reaction of sodium salts of trilacunar Keggin  $\alpha$ - and  $\beta$ -[SiW<sub>9</sub>O<sub>34</sub>]<sup>9-</sup> heteroanions with RuCl<sub>3</sub> in aqueous solution will be accomplished.

Crystal Engineering of Organic-Inorganic Hybrids. Research efforts towards the isolation of novel multidimensional organic-inorganic frameworks will be continued, with the main synthetic strategy still being hydrothermal synthesis. Future work will contemplate a change in the ideology of the metallic nodes. While in previous years the selected metal centres were mainly d-block elements, from now on lanthanide centres will be preferred so to introduce functionality at the basic level of the primary building blocks of the networks. These cations will be combined with organic ligands which contain carboxylic acid groups (such as N-(phosphonomethyl)iminodiacetic acid, glutaric acid, 2,6-pyridinedicarboxylic acid and picolinic acid), and with molecules composed essentially by phosphonic acid groups [for example, etidronic acid, N-(Carboxymethyl)iminodi(methylphosphinic acid), and nitrilotri(methylphosphonic acid)]. Particular attention will be given to materials which can only be isolated as microcrystalline powders, and structure elucidation will be attempted from systematic combined studies of powder X-ray data (either collected at a high-resolution station such as a synchrotron source, or at the laboratory scale) with structural information derived from high-resolution solid-state NMR investigations. Particular attention will be given to the information which this latter technique can provide concerning the location of the hydrogen atoms. Lanthanide-containing materials will be further studied for their photoluminescence properties.

In previous work carried out at CICECO, the structural and catalytic properties of organotin metalates with the general formula  $[(R_3Sn)_2MO_4]$  (M = Mo or W; R = methyl, n-butyl, cyclohexyl, phenyl and benzyl) were studied. The catalytic results with these materials depend on the nature of the tin-bound R groups. In future work, the introduction of chirality into the system  $[(Me_3Sn)_2MO_4]$  will be attempted by the substitution of one of the methyl groups by the (–)-menthyl group. The resultant materials will be characterised by vibrational spectroscopy and EXAFS, and tested as catalysts for the liquid-phase epoxidation of model substrates such as cyclooctene and prochiral olefins such as *trans*- $\beta$ -methylstyrene. The synthesis of organotin vanadates of the type [R<sub>3</sub>SnVO<sub>3</sub>] will also be undertaken. To date, only the trimethyltin derivative [Me<sub>3</sub>SnVO<sub>3</sub>] has been reported in the literature, and therefore a considerable effort will be made to prepare and structurally characterise new derivatives.

*Organic-Inorganic Hybrids Lacking Activating Centers.* Development of new functional hybrids hierarchically ordered with potential applications in integrated optics devices. In particular, highly ordered mono-amide cross-linked alkylene/siloxane hybrid (mono-amidosil) will be synthesised and its local structure and photoluminescence characterised. These hybrids consist of a highly organized bilayer of 2D siliceous domains, separated by perpendicularly oriented alkyl chains, self-assembled through (i) intermolecular hydrogen bonding; (ii) partially interdigitated van der Waals packing, and (iii) an entropic term related to the phase separation. The investigation of the mechanism of self-organization and the relationship between the ordered nanostructures and the corresponding emission properties will be addressed. The emission decay curves of several organic-inorganic hybrids will be modeled as a function of the temperature, emission and excitation wavelengths. Several decay models typical of disordered structures (*e.g.* stretched exponential) and semiconductor materials involving mechanisms typical of donor-acceptor pairs will be employed.

*New Hybrid Materials*. With the goal of enhancing the luminescence features and the chemical stability under ultraviolet (UV) radiation exposure, different lanthanide based complexes will be incorporated in organicinorganic hybrids, such as di-ureasils and poly(beta-caprolactone)siloxane biohybrids. The ability of the hybrid host to efficiently incorporate different lanthanide complexes will be investigated together with the hybrid host contribution in the decrease of the non-radiative paths accessible to the lanthanide ions. The absolute emission quantum efficiency will be quantified. The chemical mechanisms behind the typical photobleaching of the isolated lanthanide based tris-beta-diketonate complexes will be addressed. The quantitative description of the energy transfer processes occurring in the organic-inorganic hybrids incorporating different concentrations of lanthanide ions will be addressed. In particular, for di-ureasils, the energy transfer between the hybrid host emitting centers (urea-cross linkages and siliceous nanodomais) and the intra-4f levels of the Ln<sup>3+</sup> ions and the triplet excited levels of the organic ligands will be quantitatively estimated generalizing the ideas proposed recently for the intramolecular energy transfer between singlet and triplet ligand levels and ligand-to-metal charge transfer states in lanthanide coordination compounds.

The magnetic and structural properties of organic-inorganic nanostructured hybrids or polymer matrices incorporating Fe-based nanoparticles will continue. The effects of inhomogeneous distributions will be also addressed.

The possibility of integrating luminescent polymers and nitride heterostructures will be studied in detail. The ultimate goal is create novel inorganic/organic electroluminescent heterostructures where the flexibility and low

cost of the polymeric materials is combined with the ease of electric excitation via bipolar semiconductor structures.

The work on lanthanide-based ordered nanocrystalline hybrid structures will continue especially by intercalating new organic moieties, performing advanced structural and optical characterization. By changing the intercalated species we expect to add new functionalities to these materials. Furthermore, other types of luminescent hybrids and inorganic nanoparticles will be synthesized by similar methods. For example, lanthanide doped strontium aluminates are a promising class of luminescent materials for application ranging from luminescent displays to biological labeling.

*Integrated Optical Devices.* The studies on organic-inorganic hybrids modified by zirconium (IV) n-proposide stabilized with methacrylic acid to produce low cost effective integrated optics devices will continue, namely: (i) the structure-properties relationship; (ii) the investigation of the influence of the processing of the materials as thin films and monoliths on the optical features and (iii) the implementing enabling solutions for access optical networks, such as low cost optical power splitters and optical filters.

*Semiconductor Heterostructures.* The manipulation of various nanocrystals (NC) at the surface of In-containing nitride heterostructures will continue. This platform will be exploits to integrate various nanomaterials and experiments at a single NC or quantum dot level and study cross coupling effects among different material systems will be attempted. The development of novel multifunctional hybrid nanomaterials (ex. magnetic-optical-electric) and new device concepts is a medium term objective.

The correlations between the nanostructure and the optical properties on epitaxial thin films and low-dimensional heterostructures will be done. Here the focus will be on characterization and modelling of the strain relaxation phenomena, and its relation to the compositional uniformity in thins films and device structures. This know-how will be extended to other material systems, namely towards oxide semiconductors, multiferroic and ferroelectric thin films.

*C60 Phase Transitions Under High-Pressure.* The structural mechanism by which C60 amorphises to carbon  $sp^2$ -phase will be addressed and new attempts to determine the structure of new carbon clathrates will be done, including complementary computer simulations. Also metal-doped carbon clathrates will be studied.

*Development of Spectroscopic Techniques.* The structure of microporous alumino-phosphate AlPO4-40 will be revisited using advanced one- to two- and three-dimensional MAS NMR solid state experiments involving <sup>31</sup>P and <sup>27</sup>Al (particularly <sup>27</sup>Al-<sup>31</sup>P MQ-HMQC).

#### ELECTROCERAMICS

*Microwave Ceramic Dielectrics.* The temperature variations of the dielectric permittivity and loss of the perovskite ceramics  $(1-x)La(Mg_{1/2}Ti_{1/2})O_3 - x(Na_{1/2}Bi_{1/2})TiO_3$  [(1-x)LMT-xNBT] ( $0 \le x \le 0.6$ ) estimated by different methods (radio-frequency, microwave and far infrared measurements) will be compared and analyzed. Contributions of extrinsic and intrinsic factors to the microwave dielectric loss of the ceramics will be evaluated. The low-temperature dielectric relaxation recently revealed in LMT-NBT will be considered in the context of similar effects observed in other Bi-containing, *A*-site disordered oxygen-octahedral compositions.

Perovskite ceramics in the solid solution system  $(1-x)La(Mg_{1/2}Ti_{1/2})O_3 - xBi(Mg_{1/2}Ti_{1/2})O_3$  [(1-x)LMT-xBMT] (0<x<0.3) will be examined using an Evanescent Microwave Probe. The maps of distribution of dielectric permittivity and quality factor will be obtained and compared with the surface topography. In order to estimate the lattice (intrinsic) contribution to the permittivity and loss observed in LMT-BMT, far infrared spectroscopy will be performed. These experiments allow ascertain if the observed microwave dielectric properties of the LMT-BMT ceramics are determined by the crystal chemistry or their microstructure.

The characterisation of the electrical properties of the compounds of the new family,  $La_6Mg_4Ta_2W_2O_{24}$ , will be performed. Using the mixing of compounds methodology, a new set of mixtures with cation ordering will be explored, in order to understand the contribution of this ordering to the dielectric characteristics, particularly the dielectric losses.

 $MgTiO_3$  thin films will be prepared by sol gel. Different precursors and stabilizers will be used in order to prepare dense, crack free and monophasic  $MgTiO_3$  films.

*Ferroelectric Fibres, Single Crystals and Films.* The interface characterization studies of SBT thin films have revealed significant differences between seeded and unseeded thin films. A similar study will be undertaken using SBN thin films. For obtaining the bottom electrode and/or buffer layer elements profiles and film components (Bi, Sr, Nb) in seeded and unseeded SBN films techniques such as Rutherford Backscattering and Particle Induced X-Ray Emission will be used. This interface thematics is expected to contribute to a better understanding of SBN ferroelectric properties.

The work on PZT thick films on Cu foils will continue aiming at decreasing the sintering temperature on the films. These studies will be focused on the selection of ideal sintering aids and their effects on the properties.

The fabrication of thick films of  $BaNd_2Ti_5O_{14}$ -based high Q dielectrics prepared by electrophoretic deposition for microwave communications will continue. The main objectives are the characterization of the dielectric properties at high frequencies and the improvement of tunability. The origin of the microstructure differences between BNT thick films and BNT ceramics will also be investigated.

The preparation of thick films of Bi based compositions for microwave applications will be initiated.

*Incipient Ferroelectrics.* The work on incipient ferroelectrics will continue along the following lines. Structural and dielectric properties of Y and Cr doped  $SrTiO_3$  ceramics, prepared by conventional mixed oxide method. The possible mechanisms of dielectric relaxations and the structure-microstructure-dielectric properties relation in SYT and SCT ceramics will be addressed in detail. Sintering and dielectric studies of the effect of non stoichiometry on the incipient ferroelectric behaviour of ST. Continuation of the studies of the stress effect on the dielectric response of doped and undoped ST thin films.

*Nanoscale Properties of Ferroelectrics.* PZT seeded films show improved macroscopic dielectric properties. Systematic studies of the local switching polarization during fatigue in unseeded and seeded films PZT thin films will be conducted by piezoresponse force microscopy (PFM). Local PFM loops which characterize domain switching will be acquired and compared with macroscopic hysteresis loops. The observed fatigue behaviour will be discussed based on domain wall pinning induced by injected electrons. The role of nano-seeds in the fatigue behaviour will be analised.

nanodomain switching of PZT thin films at reveals different features of the polarization behavior. The piezoresponse force microscopy (PFM) observation of abnormal domain switching of PZT seeded and unseeded thin films of morphotropic boundary composition will be studied.

The study of the local properties of ferroelectric thin films, single crystals and ceramics will be continued, including also PZT single crystals and commercial PZT ceramics. We plan to investigate the local properties under varying electric field and temperature (up to 250 °C). Polarization patterns prepared by nanolithography will be studied as a function of temperature and time to verify their stability for memory applications. The investigations will be extended to lead-free KNN and NBT systems. Multilayer actuators based on the commercial PZT ceramics will be investigated at the nanoscale with the aim of improving their electromechanical performance (with Siemens). The domain wall motion studies (creep and activation mechanisms) will be studied in detail. The SFM

programs to acquire piezoelectric signal will be modified to measure additional parameters (inaccessible so far). This work will be done in collaboration with Agilent Technologies.

Work on ferroelectric relaxors will be extende to new materials, such as lead-free relaxors to understand the nature of ferroelectric disorder at the nanoscale. PMN and PZN single crystals and their solid solutions with  $PbTiO_3$  will be studied to determine the role of the nanoscale domains in high piezoelectric performance of these crystals. Pure PMN and PLZT that freeze at low temperatures will be investigated using new AFM setup. The comparison with the results of macroscopic measurements will be done in order to reveal information delivered by AFM. The study will be extended to the smallest domains created by nature using a liquid measurement cell available in the new setup.

*Multifunctional Ceramic Films and Composites.* The magnetoelectric coupling, conductivity and local PFM/MFM measurements of the LSMO-LuMnO<sub>3</sub> system will be studied. Optimal composition will be scaled down in order to transfer useful ceramics properties to the thick film level using a hybrid technique. BiFeO<sub>3</sub> with different dopants (Ca, Pb, Sr, Zn, etc) will be sintered to reduce the amount of second phases and to cause uncompensated magnetization of canted magnetic moments. Novel composites such as PZN-PT/Terfenol D will be sintered and investigated. Novel composite thick film materials (BiFeO<sub>3</sub>-PZT) will be fabricated by a hybrid solgel method. Magnetoelectric measurement setup will be built. The preparation by sol gel of nanosized nickel ferrite and bismuth ferrite powders and thin films will be undertaken. Different metal alkoxides and nitrates or acetates as Ni, Bi and Fe sources, together with the use of alkoxides stabilizers and nitrates and acetates solvents will be evaluate to obtain stable, transparent sols. Subsequent chemical and heat treatments will be performed for the preparation of powders. Alternatively, the sols will be used to thin films deposition. The heat treated powders and films will be characterized in terms of the desired properties.

*Bioferroelecticity and Polarization-Induced Self-Assembly.* We will explore an innovative approach that allows controlling the local reactivity and physico-chemical absorption of organic molecules on the ferroelectric surface due to variation of atomic polarization and electronic structure of ferroelectric polymers. The method consists on the nanoscale polarization patterning of ultrathin Langmuir-Blodgett films based on P(VDF-TrFE) copolymer by scanning force microscopy and subsequent exposure of the patterned surface to several chemical species including amphipilic merocyanine molecules and different glicolipids that possess glucose polar hydrophobic head group and a hydrophobic alkyl chain. The obtained complex self-assembled nanostructures will be investigated by a variety of analytical and spectroscopic techniques including macroscopic (dielectric constant, polarization, switching current, pyrocoefficient, optical spectroscopy, exoelectron emission, XPS, etc) and local ones (electric potential,

charge, electric field, conductivity, piezoresponse). The switching and recognition phenomena related with the dynamics of ferroelectric domains during application of external electric field will be also studied. The measurements will be complemented with extensive modelling of the nanostructures including configuration of molecular units, cooperative phenomena, molecular dynamics, and local electronic structure.

*Nanoscale Materials.* Work will continue on the laboratory scale preparation of a broad range of ferroelectric, piezoelectric and quantum paraelectric perovskites 1D nanostructures, namely, nanotubes, nanorods and nanowires, by soft chemical methods. The optimal processing conditions, thermodynamics and kinetics for the hydrothermal synthesis of each crystallographic phase in a certain 1D morphology will be studied. The method of synthesis of mesoporous perovkites will be investigated.

#### MAGNETOSTRUCTURAL MODULATION OF STRONGLY CORRELATED ELECTRIC MATERIALS

*Colossal Magnetoresistive Materials.* Work on thin film prepared with the new RF sputtering deposition system for oxide thin films, structural (X-ray), magnetic and electrical properties. Studies using implanted radioactive isotopes at ISOLDE-CERN. Charge/orbital order electronic phase segregated systems: competition and interface effects. Multiferroic materials (as hexagonal manganites REMnO<sub>3</sub>, RE=Er, Lu) and composites LaSrMnO<sub>3</sub>-REMnO<sub>3</sub> and BaTiO<sub>3</sub>-LaBaMnO<sub>3</sub> manganites.

*Theoretical Approaches to Magnetic Materials Using Generalised Thermodynamics.* Application of nonextensive statistics to manganites and other complex inhomogeneous magnetic systems. Critical phenomena, Griffiths phase, Yang-Lee zeros, diluted Ising model, Monte-Carlo simulations and non-extensive statistics. Specific heat of fractal systems. Thermodynamics of two-level magnetic system. Magneto-electro-elastic coupling effects. Mean-field approaches for data analysis.

*High Temperature Superconductors.* Physical characterization studies of oxide superconductor materials prepared by LFZ. Studies on new MgB<sub>2</sub>-type superconductors, preparation and processing. Superconducting thin films. Epitaxy and Microstructure. Magnetic and electrical properties studies.

*Other Developments.* RE-metalloid (RE-Si-Ge and others, RE stands for rare-earth metals), intermetallic compounds (RE-M, M is a transition metal) and manganite systems. Influence of interstitial hydrogen and nitrogen on the magnetocaloric properties of metals. Modelling of magnetic entropy contributions.

Development of new experimental techniques: VSM magnetometer, AC susceptibility, electrical measurements, specific heat and thermal-magneto-expansion up to 10 T and temperature 1.9-300 K. Development of a new process to optimize the thermomagnetic Brayton cycle, in the sense of the magnetocaloric effect. Construction of a prototype for magnetocaloric refrigeration near room temperature. Use of a second rf power supply in the sputtering system for sequential and/or co-deposition of thin films. Experimental set-up for electric measurements, with thermal-magneto expansion and magneto-resistance, from 77 to 473 K (and magnetic field up to 1 T, rotating from 0° to 360°).

#### ADVANCED MOLECULAR AND SUPRAMOLECULAR MATERIALS

*Cyclodextrins.* Studies on cyclodextrins (CDs) as second sphere ligands for organometallic compounds with interesting catalytic properties or biological chemistry will continue. The complexes  $CpFe(CO)_2Cl$  and  $CpMo(CO)_3Cl$  encapsulated in native  $\beta$ -CD and permethylated  $\beta$ -CD (TRIMEB) during 2006 will be tested as catalysts for the liquid-phase epoxidation of cyclooctene.

Metallocene dichlorides  $Cp_2MCl_2$  (M = Ti, V, Nb, Mo) have attracted considerable interest due to their antitumour activities against a range of tumour cell lines. While the encapsulation of the Ti, V and Mo derivatives in CDs has already been studied, CD inclusion compounds containing  $Cp_2NbCl_2$  have not yet been described. In another study, the inclusion of the biomimetic complex  $Mo(\eta^3-C_3H_5)CpCOO-Phen-CH_3$  (Phen = Phenylalanine) and its precursor  $Mo(\eta^3-C_3H_5)CpCOOH$  in the hosts  $\beta$ –CD and TRIMEB will also be attempted. The cytotoxic and antiproliferative activity of these compounds towards human adenocarcinoma cells will be screened. All of the inclusion compounds prepared will be characterised in detail. Powder XRD studies will be complemented by *ab initio* calculations to elucidate preferential organometallic-cyclodextrin inclusion geometries. Finally, in an extension of previous work, the encapsulation of  $[CpMo(NCMe)_2(CO)_2](BF_4)$  and [CpMo(2,2' $biimidazole)(CO)_2](BF_4)$  in heptakis-2,6-di-*O*-methyl- $\beta$ -CD (DIMEB) will be performed.

New compounds with potential anti-tumoral activity find, in general, difficulties to reach their targets, mainly due to non-compatibility with the physiological medium (hydrolysis or enzymatic inactivation) or to toxicity. The inclusion of those compounds may permit to overcome these problems. Inclusion into cyclodextrins will be attempted using previously synthesised and characterized ruthenium(II)-thioether/polypirydilic complexes or new synthesised ones. The newly designed complexes will possess suitable ligands for in vivo media, such as aminoacids, or organometallic fragments, which efficacy has already been demonstrated.

Studies are planned to investigate the influence of the cyclodextrins concentration in inclusion/micellization processes in the system  $\beta$ -cyclodextrin and TRIMEB with sodium decanoate in water.

*Metal Complexes of Bio-Inorganic Interest.* New binuclear Ru(II)-thioether systems will continue to be studied by mass spectrometry, mainly ESI-MS, in order to elucidate their fragmentation mechanisms. The interaction of mononuclear Ru(II)-thioether-polypirydylic complexes with DNA-base sequences will be completed and their intercalation modes assigned.

New chromium compounds taken as models for relevant intermediates in the intracellular-Cr(VI) reduction will continue to be studied by several techniques (EPR, for example). Additionally, other chromium compounds, considered important from a nutritional point of view, will be synthesised and tested (in vivo), as they have become suspicious to provoke damages *in vivo*.

New compounds for DNA molecular recognition, with transition metal and polypyridyl and/or thioether ligands will continue to be synthesised and characterised. Studies directed to the syntheses of 'square' metal transition systems will continue to be undertaken.

*Oxomolybdenum Catalysts.* Novel cis-dioxomolybdenum(VI) complexes of the type MoO<sub>2</sub>L and MoO<sub>2</sub>Cl<sub>2</sub>L, with tetradentate [N<sub>2</sub>(imine)O<sub>2</sub>] and bidentate [N<sub>2</sub>(imine)] salen-type ligands (L), respectively, will be synthesised and characterised. The complexes will be examined as catalysts for the epoxidation of a series of olefins using *tert*-butyl hydroperoxide as the oxidant. In a related study, cyclopentadienyl molybdenum(II) carbonyl complexes with the general formula Cp'Mo(CO)<sub>2</sub>( $\eta^3$ -C<sub>3</sub>H<sub>5</sub>) (Cp' = Cp, CpMe, Cp\*) will be prepared using an optimised method. Previous work carried out at CICECO has shown that these types of compounds can be used directly as precursors to oxomolybdenum(VI) catalysts for the epoxidation of olefins because they undergo oxidative decarbonylation in the presence of the oxidant t-BuOOH. The new Cp'Mo(CO)<sub>2</sub>( $\eta^3$ -C<sub>3</sub>H<sub>5</sub>) compounds will therefore also be tested as precatalysts for the epoxidation reaction.
#### AREA 2 – ADVANCED MATERIALS FOR INDUSTRIAL APPLICATIONS

### COMPONENTS FOR PROCESSING CONTROL

*Materials For High-Temperature Electrochemical Applications.* The main activities will be dedicated to novel ionic conductors, mixed conductors and electrocatalysts for fuel cells and other high- or intermediate-temperature electrochemical applications. Some of these studies will be directed to detailed assessment of limitations of known materials in terms of insufficient or non-optimized transport properties, stability and other requirements imposed by processing, or operation conditions, including electrocatalytic effects. Stability issues will be directed to apatite and other novel electrolyte types and their interactions with prospective electrode materials. Attention will be given to stability issues related to reducing conditions, high gradients of chemical potential, materials interactions, chemically or thermally induced changes and stresses developed from processing to operating conditions and to maintenance at room temperature. One will investigate the advantages and restrictions imposed by transport properties of electrolyte and electrode materials on prospective applications under OCV (gas sensors), power generation (fuel cells), and electrochemically driven conditions (electrolysers).

Continuing our previous work on electrodes and electrocatalysts we will extend the studies of materials with oxygen storage ability and its effects on electrode performance; this will be extended to include a new concept of incipient phase transformation to boost oxygen storage/release, and corresponding effects on electrode kinetics, under OCV conditions and also under cathodic or anodic polarization. A number of potential anode materials are being considered for silicate based electrolytes, including some mixed conducting materials containing silica.

*Microstructural Effects.* Studies of processing and microstructural effects will comprise work on single phase and composite materials. Work on single-phase materials will be mainly related to improving the grain boundary behaviour of solid electrolytes, especially for intermediate temperature applications. Different types of ionic conductors will be studied, including materials with the fluorite structure (*e.g.* cerias), perovskites, apatites and pyrochlore materials. A revision of space charge effects and their dependence on temperature, composition and sintering additives will be considered. We will also attempt to distinguish the ionic and electronic contributions of grain boundaries and to attain a better understanding of their role on bulk transport properties and surface processes, including electrocatalytic effects or surface exchange.

Mixed Conducting Materials and Catalysts for Oxygen Separation or partial Oxidation of Hydrocarbons. The work on mixed conducting materials and their potential use in oxygen separation and fuel processing will continue,

taking advantage of a very wide range of materials studied by our group. We will continue the assessment of materials characteristics (composition, structure, incipient phase change, and other factors) determining preferential formation of partially or fully oxidized species, even in oxygen lean conditions. New studies of fuel processing or conversion directed to lower grade fuels (biogas) requiring specific methods (reforming of methane+carbon dioxide), and/or with very demanding requirements of tolerance to contaminants (sulphur tolerance) will be started. This work will involve novel material formulations, possibly including potential non-oxide electrocatalysts (carbides ou nitrides).

*Materials for Other Electrochemical Processes*. We will also extend our work on electrode materials for low temperature electrochemical technologies, with emphasis on electrolytic processes under alkaline aqueous media. The work will include the development of ceramic and metal based anodes and their surface modification with electroactive layers (hydroxide or oxy-hydroxide layers). We will attempt to find a comprehensive interpretation for the similarities between ceramic and metal-based anodes, including a detailed examination of the role of different hydroxide layers (single component or binary layers) obtained by chemical or electrochemical methods on ceramic and metallic substrates. The range of working conditions will be extended to more extreme conditions, such as temperatures approaching boiling conditions, higher alkalinity in aqueous solutions or suspensions, and stirring, to obtain guidelines for optimized materials and operation conditions in terms of electrocatalytic activity, degradation and prospective regeneration.

#### CERAMIC COMPOSITES AND ULTRA-HARD COATINGS FOR MECHANICAL APPLICATIONS

*Diamond Coatings.* The tribological characterization of nanocrystalline diamond (NCD) will be completed by the use of lubricant fluids, namely for bio-tribological purposes using adequate loads and fluids for screening tests aiming hipjoint implant applications. The machining of abrasive materials such as sintered ceramics, graphite and aluminium alloys will be another line of development for the CVD diamond coated silicon nitride ceramics. The NCD coating of other materials such as ultra-fine or nano-sized hardmetals will also be started. This work will include the production of such substrate materials and the study of the surface treatments needed to enhance the adhesion of CVD diamond coatings. A new pilot plant size HFCVD reactor for the deposition of areas up to  $25 \times 25$  cm<sup>2</sup> will be designed and constructed, aiming its use for proof of concept of products for the international hard tools market.

Other Hard and Ultra-Hard Materials. Another research area, also related to functional coatings, is being carried out, aiming the aluminium injection industry. 'Duplex hard coatings on steels for aluminium injection' is an industry-

oriented research subject where the adhesion, thermochemical and thermomechanical properties of the new materials will be tested and improved. Similarly, industry related research will be focused on technologies and business opportunities, namely unconventional heat treating to improve the wear resistance and on-service dimensional stability of tools and mechanical components such as gears.

*Processing Methods.* The work plan for the next year is a natural continuation of the research lines started before and will be focussed on the following topics:

(i) Colloidal processing of advanced ceramics in aqueous and non-aqueous environments. New direct shaping methodologies are under development, which will enable de consolidation of large and homogeneous ceramic parts. Well dispersed systems of nanoparticles will be developed aiming at rapid manufacturing and ink-jet printing.

(ii) Development of new glass and glass-ceramic compositions for different applications (structural, optical, biomedicine, electronics, sealants for SOFC, etc.), and study the nucleation and crystallization processes and the properties of the resulting glass-ceramics.

(iii) Preparation of macroporous ceramic bodies with tailored porous microstructures for bone-ingrowth in biomedical applications. Mechanical and *in vitro* and *in vivo*, characterization of the macroporous materials will be carried out in collaboration with other specialized research centres and hospitals.

(iv) Studies of recycling industrial wastes and by-products to prepare new added-value materials. The current research works will be extended to other types of residues in order to find suitable solutions to clean the environment, preserve natural and non-renewable resources, save energy, while improving some of the actually existing products or developing new ones with new functionalities.

A new non-aqueous sol-gel approach applied to the atomic layer deposition technique will be pursued in order to synthesize high-K dielectric materials on different substrates. Such a novel approach will bring several advantages compared to ordinary deposition techniques namely a very low deposition temperature, the ability to avoid a  $SiO_2$  interfacial layer during the growth of an oxide on a silicon substrate and the possibility to precisely control the film thickness. Furthermore, this approach will be extended in order to grow multiferroic nanostructure.

*Corrosion Protection Methods.* The work will be a continuation of the liness started in recent years. The main studies will be concentrated on the development of active corrosion protective coatings with self-healing ability for different metallic substrates. Novel nanocontainers will be developed in order to incorporate organic corrosion inhibitors into the different coatings. The new nanoreservoirs using layer-by-layer assembly of polyelectrolyte

shells will be developed. Another approach for creation of 'smart' nanocontainers will based on employment of layered double hydroxides as controllable release systems for corrosion inhibitors. The new effective corrosion inhibitor explored during last year will be impregnated to the different developed nanocontainers and then introduced to the coatings.

The work on the development of novel nanostructured hybrid sol-gel films for corrosion protection of aluminum and magnesium alloys will be continued. The new more effective sol-gel films for magnesium alloys will be developed. Although magnesium alloys are becoming more important for many industrial applications their high susceptibility to corrosion limits their present use. Therefore, development of new corrosion protection approaches for magnesium alloys is an issue of primer importance. The new functional organosiloxane with high affinity to the magnesium will be used to create novel hybrid sol-gel films.

The work on investigation of the mechanism of corrosion processes and corrosion inhibition will be continued. The aluminum alloy 5083 and magnesium alloys AZ31 and AZ91 will be in main focus. New organic and inorganic corrosion inhibitors will be tested.

The Scanning Vibrating Electrode Technique (SVET) and related techniques (SIET) will be employed to get a fundamental understanding of the localized corrosion processes in the micro-scale. The corrosion processes will be studied in the induced microdefects in the coatings as well as on the bare alloys. The effect of corrosion inhibitors using SVET and SIET methods will be also investigated. Active corrosion protection of weldable primers doped with corrosion inhibitors will be investigated using a wide range of the experimental techniques in the macro- and micro-scale.

# AREA 3 – CHEMISTRY AND TECHNOLOGY OF POLYMERIC AND LIGNOCELLULOSIC MATERIALS AND BIOMATERIALS

#### MACROMOLECULAR MATERIALS AND LIGNOCELLULOSICS

*Lignocellulosics.* The chitosan project will focus on the study of the use of the polyols arising from its oxypropylation as macromonomer in the synthesis of polyurethanes and polyesters. Modified chitosans bearing furan and maleimide moieties in modest percentages will be prepared and characterized with the aim of studying the formation of thermally reversible chitosan networks. Work will also begin on the self-assembly of chitosan monolayers with complementary polyelectrolytes bearing negative charges, *e.g.* carboxymethylcellulose.

The Diels-Alder study will enter its 'macromolecular' phase with the synthesis of linear and hyperbranched polymers, the study of the equilibria associated with forward and retro-reactions and the characterization of the materials. The systematic use of UV and NMR spectroscopy, as well as of viscosity and GPC measurements will provide precious information about the physical chemistry of these systems. Among the different points of interest here, the growth and backward steps in the synthesis of dendrimers, applied to each generation, will represent an important aspect of this project.

The polymerization of the suberin monomer mixture will enter an intensive phase with the extension of the catalytic systems to be tested and the optimization of those giving the most promising results. The ensuing materials will receive a thorough characterization in terms of both structure and physical properties. The applications of these polyesters are also a key point of this phase, notably as adhesives and coatings, with particular emphasis on natural substrates like cellulose fibres, cork particle and panels. The study of polymer syntheses involving suberin in the presence of these substrates will also be explored with the aim of generating covalently bound supramolecular architecture.

Within the scope of the controlled heterogeneous modification of cellulose fibers, future studies will focus on the investigation of new strategies and on the application of the new cellulose derivatives already obtained. Composites materials with fatty acids modified cellulose fibers and different polymeric matrices (polyethylene and biodegradable polymers namely polylactic acid and polycaprolactone) will be prepared and characterized in collaboration. The approaches developed and optimized for the modification of plant cellulose fibers will be extended to other natural polymers (chitin, chitosan and starch) and as well as to other cellulose substrates (bacterial cellulose and microfibrillated cellulose).

As far as inorganic nanoparticles/cellulose hybrids are concerned, future work will be focused on the study of possible applications including photocatalysis, antibacterial activity and incorporation in polymeric matrixes.

Depending on the results obtained, different strategies such as the deposition of silver nanoparticles at  $TiO_2$ /cellulose surfaces to enhance the photocalytic behaviour and antibacterial properties will be investigated. The use of bacterial cellulose as template for the controlled growth of metallic nanoparticles will be assessed.

The work on polyoxometalate (POM) catalysis in oxygen delignification of kraft pulp, using POMs with relatively high redox potential, will be continued. The special emphasis will be placed on the study of the electrochemical re-oxidation of POMs instead of biocatalytic re-oxidation with laccase aiming to improve the process efficiency.

The study of the detail mechanisms of substituted phenols oxidation with  $[\alpha-SiV^VW_{11}O_{40}]^{5-}$  and  $[\alpha-SiMn^{III}W_{11}(H_2O)O_{39}]^{5-}$  will de carried out..

The development of a new electronic tongue (ET) multisensor system for the detection of particular structures of polyoxometalates containing vanadium (IV/V) atoms and for the monitoring of their behaviour in redox catalysis will be continued.

The study of the paper surface modification with perspective formulations prepared by sol-gel will continue. The formulations of optimised composition will be deposited on different types of papers using novel sophisticated equipment installed in RAIZ (double roll sizing press) and the printing properties of coated paper will be evaluated (ink jet and off set printing). The synthesis of new formulations with specific catalytic properties for the pulp/paper coating (functional materials) will be started this year.

The ESI-MS studies for the structural characterization of lignin will continue. The studies on specific structural features of hardwood hemicelluloses employing ESI-MS/MS and MALDI-TOF/TOF will be continued.

The study of the chemical composition of macromolecular components of the natural hybrid of *Paulownia elongata* and *Paulownia fortunei* will be further developed. Special attention will be paid to the evaluation of pulp properties and papermaking potential of this new fast growing wood.

Two research projects on the utilization of by-products from the acid sulphite pulping of *E. globulus* wood (condensates from pulping liquor evaporation and the components of sulphite spent liquor) will be carried out. Special attention will be paid to the bioprocessing of sulphite spent liquor for the production of ethanol, xylitol and microbial proteins.

Work on the preparation of polymer based NCs via living polymerisation mechanisms in miniemulsion will be developed. Attempts will be made to prepare block copolymers and to functionalise end groups for use as bio labels as well as to investigate further the photoluminescent properties of those materials.

As regards the polysaccharide NC project, exopolysaccharides produced by *Rhyzobium sp* will be tested and the preparation of thin films from such composites will receive significant attention. The effect of  $SiO_2@Fe_2O_3$  nanoparticles will also be investigated.

The modelling and scale-up studies of the oxypropylation reaction of cork residual powder will be continued. The joint (UA/TUHH) MSc theses will continue along the following lines: (i) the conducting properties of the NCs prepared will be investigated; (ii) the modified CNFs will be dispersed in a polyamide matrix and the mechanical properties studied; (iii) upon understanding the role of the polymer in cement mortars new formulations will be tested including CNTs in collaboration with Weber cimenfix.

*Other Polymer Systems and Materials.* Work will continue on the shear-induced solidification effect on melt morphology developed by amorphous and semicrystalline polymers. Three papers will be submitted soon on: size of precursor structures in critically sheared polymers melts; effect of melt memory on the shear-induced non-isothermal crystallization of polypropylene and evaluation of the size of precursor structures; shear induced non-isothermal crystallization of low density polyethylene. Other work dealing with the physical meaning of oscillatory shear and steady shear flow activation energies, the estimation of chain dimensions at the steady-state and on the physical nature of entanglements is in progress.

The predictive, analytical (non-simulative) cooperative segmental theory of materials dynamics (CSTMD) will continue to be developed and applied to the dynamic behaviour of polymers. The installation of the Hyper DSC instrumentation in the Laboratory of Thermal Analyses will be carried out.

### **BIOMEDICAL AND BIOMIMETIC MATERIALS**

*Biological, Structural and Identification FTIR, NMR and Other Studies.* Chromium compounds, in different oxidation states, will continued to be tested in *in vivo* studies with mice in order to localise degenerative damages in animal target organs and quantify the amounts of this element in those organs, using. histology, histochemistry and ultra-structural techniques. Chromium picolinate, a dietary supplement suspict of causing health damage, will be used for *in vivo* studies with mice, in order to evaluate possible effects on target organs. The toxic effects of titanium compounds frequently used as chemotherapeutic agents will be investigated, namely on the male mice reproductive system. Traditional diagnostic techniques (biopsies) will continued to be compared with faster, reliable and cheaper ones (flow citometry and fluorescence microscopy) in order to quantify 'dose-effect' relationships.

The NMR characterisation of biofluids and biological tissues will be pursued, with extension and applications to (i) study of pregnancy disorders, (ii) breast and thyroid cancer, (iii) heart failure and diabetes incidence, (iv) detection of inborn errors.

Recently developed biomaterials based on magnetic nanoparticles (magnetite/carrageenan) will be chemically derivatised and tested in near-physiological conditions.

NMR-based models for the routine control of beers and brewing process should be implemented as service for the brewing company. Projects on spectroscopic detection of beer ageing and contamination will be carried out.

The role of several depolymerising enzymes and fungi on the detailed nanostructure of cork will be continued, with strong emphasis on the direct characterisation of the complex material by NMR and FTIR methods.

The interaction of the heme-binding proteins murine p22HBP and human SOUL with functional porphyrins will be studied using NMR and fluorescence quenching in order to assess their binding characteristics. The X-ray and NMR structures of hSOUL will be initiated. Functional studies of mHBP and hSOUL will be started. Molecular modelling of these systems will also be initiated.

*Glass and Ceramic-Based Biomaterials.* Continuing current research lines, we will address the colloidal processing of calcium phosphate-base ceramics, including well dispersed systems of nanoparticles aiming at rapid manufacturing techniques, and the development of new glass and glass-ceramic compositions for biomedical applications, and study of the nucleation and crystallization processes and the properties of the resulting glass-ceramics. The preparation of macroporous ceramic bodies and ceramic-biopolymer composites with tailored porous microstructures for bone-ingrowth in biomedical applications, and the mechanical, *in vitro* and *in vivo* characterization of the macroporous materials will be carried out in collaboration with other specialized research centres and hospitals.

Research on the developed glasses and glass-ceramics will progress this year with a main emphasis on the study of the polymer-based composites where those materials provide the reinforcing phase. *In vitro* tests in simulated physiological fluids will be performed in all composites to assess their mineralization capability. A number of selected composites will be loaded with therapeutic agents to study the drug leaching rate of various medicals. *In vitro* tests in osteoblast cell cultures will also start this year, aiming at studying cell adhesion and proliferation on those materials.

The releasing behaviour of the spray dried porous granules for drugs and bioactive substances in solutions will be studied at physiological conditions, as well as the evaluation of the influence of the porosity of the material on the release profiles. The in-vitro release of 5-Flourouracil will be tested by immersing the granules in phosphate buffered solution with pH 7.4 at 37 °C. The measurement of the released amount of 5-FU will be performed by UV-Vis spectroscopy.

*Other New Biomaterials.* Using the surfactant production characteristic of the *Yarrowia lipolytica* and its ability to degrade paraffins a study concerning its use on biodegradation of fuels will be conducted.

Studies of the biodegradation of non-conventional solvents such as ionic liquids and studies of their toxicity on model organisms will also be conducted.

### PROCESS DEVELOPMENT AND OPTIMISATION

*Phase Behaviour and Transport Properties Relevant in Environmental Protection, Chemical Processing and New Materials Production*. On fuels and biofuels, our work will be moving from conventional fuels towards biofuels or blends of biofuels with conventional fuels. Modelling phase equilibria during conventional biodiesel production by transesterification will be carried using the CPA-EoS. Measurements of water solubility in fuels and ternary liquid liquid equilibria of oils alcohols and water or glycerol, esters and alcohols have been carried and will be modelled. Concerning the formulation of biodiesels studies will be lead to study the acceptable regions of mixture of different types of oil to meet the iodine index and the cloud point specifications.

The models previously developed for the wax formation in conventional fuels will be extended to the description of biofuels. Studies of wax formation in biodiesels and conventional diesel plus biodiesel mixtures will be carried out.

Concerning bioethanol, studies will be carried out of their blends with hydrocarbons to evaluate the limits of their mixture in the formulation of new fuels. Surface tensions and liquid liquid equilibria will be addressed.

On phase change materials, alternatives to paraffins from renewable sources will be investigated. Special emphasis will be given to fatty acids and their esters as secondary products from biofuels production. Thermophysical characterization of these compounds and mixtures will be carried out.

The studies with ionic liquids will be continued into two different directions. One is the attempting to better understand the liquid structure of these compounds. Studies of enthalpies of vaporization will be published and mixtures with other solvents such as water and alcohols will also be investigated to reveal about the molecular interactions in the liquid phase. The second direction is their environmental impact. The new REACH regulations require that producers have a good understanding of the toxicity, biodegradability and dispersion into the environment of new chemicals. Since these compounds have negligible vapour pressures their release into the environment can only take place through liquid effluents. Their solubility in water and in aqueous solutions will be measured. Studies will also be conducted of the octanol-water partition coefficients and of their toxicity towards industrial microorganisms.

On the field of polymers and polymeric solutions, the encapsulation of hydrophilic and hydrophobic drugs using biodegradable polymers will be addressed. The study of biodegradable polymers in non conventional solvents such as ionic liquids will be measured in the laboratory and modelled with the models developed in the previous year. Films with oxygen scavengers will also be prepared and the barrier properties (diffusion and sorption) will be measured.

The systematic characterization of the perflurocarbon systems will be continued, by measuring viscosities. The study of the water in fluorocarbon (cyclic and aromatics) will be performed by both measuring the liquid-liquid equilibria and modelling it with COSMO-RS. A study of the phase equilibria of systems involving flurocarbons and ionic liquids will be carried out.

*New Chemical Processes.* We will continue to study the transformation of carbohydrates into furfural and 5hydromethyl furfural using nanoporous acid catalysts. Several bulk and ordered mesoporous silica-supported zirconias will be prepared and examined as acid catalysts for the dehydrocyclisation of xylose into furfural. Concerning the catalyst preparation we will investigate the influence of promoters (*e.g.* Al) and the sulfating agent. Attempting to improve the textural properties and acid site distribution, modified zirconias will be supported on ordered mesoporous silica or prepared using surfactants as structure-directing agents. These catalytic results will be compared to those obtained with  $H_2SO_4$ . In a further study, zeolite-like materials will be used as precursors to prepare materials presenting strong acidity in an open porosity for the dehydrocyclisation of hexoses and pentoses. The preparation method will involve the exfoliation of lamellar aluminosilicates.

Glucose is an important monomer unit of the abundant natural polysaccharide cellulose and is likely to become one of the most important starting chemicals as a result of the increasing dependence of society on biomass resources for sustainability. One of the primary reactions of glucose is base-catalyzed isomerization into fructose. Work will be initiated on the isomerization of glucose over microporous zeolite-like solids containing non-framework and/or framework alkali metals, including some materials that were discovered at CICECO. The catalytic results will be compared to those obtained with commercial zeolite Na-X or aqueous NaOH.



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## **CONGRESS ORGANISATION**

11 OLIMPÍADA IBEROAMERICANA DE QUÍMICA MAGALHÃES, MCF 08-15 SEPT 2006, UNIVERSITY OF AVEIRO, PORTUGAL

12TH INTERNATIONAL SYMPOSIUM ON SOLUBILITY PHENOMENA AND RELATED EQUILIBRIUM PROCESSES\_IN MAGALHÃES, MCF 23-28 JUL 2006, TU BERGAKADEMIE FREIBERG, GERMANY

25 ANOS DE MATERIAIS EM PORTUGAL FERNANDES, MH; VILARINHO, PM 02-04 NOV 2006, UNIVERSITY OF AVEIRO, PORTUGAL

2ND REPSOL MEETING ON FLOW ASSURANCE COUTINHO, JAP 10 NOV 2006, UNIVERSITY OF AVEIRO, PORTUGAL

6TH IBERIAN VACUUM MEETING ETCHC-4 SILVA, RF: INTERNATIONAL ADVISORY COMMITTEE 26-28 JUN 2006, UNIVERSITY OF SALAMANCA, SPAIN

6TH INTERNATIONAL CONFERENCE ON F-ELEMENTS CARLOS, LD: INTERNATIONAL SCIENTIFIC ADVISORY COMMITTEE 04-09 SEPT 2006, WROCŁAW, POLAND

8TH EUROPEAN CONFERENCE ON APPLICATIONS OF POLAR DIELECTRICS KHOLKINE, A 06-08 SEPT 2006, METZ

9TH EUROPEAN WORKSHOP ON LIGNOCELLULOSICS AND PULP – EWLP NETO, CP: INTERNATIONAL ADVISORY COMMITTEE 27-30 AUG 2006, VIENNA, AUSTRIA

JORNADAS DE ELECTROQUÍMICA E INOVAÇÃO 2006 (E-INOV 2006) CARAPUÇA, H 20 OCT 2006, FUNDAÇÃO CUPERTINO DE MIRANDA, PORTO, PORTUGAL

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*EMPREENDER: DA TEORIA À PRÁTICA* DANIEL, A 16 DEC 2006, UNIVERSITY OF AVEIRO, PORTUGAL

*E-MRS 2006 - SPRING MEETING* MARQUES, FMB: SCIENTIFIC COMMITTEE 29 MAY-02JUN 2006, NICE, FRANCE *FÍSICA 2006: 15ª CONFERÊNCIA NACIONAL DE FÍSICA* CARLOS, LD: SCIENTIFIC COMMITTEE 04-07 SEPT 2006, UNIVERSITY OF AVEIRO, PORTUGAL

*III JORNADAS CICECO* FERREIRA P: ORGANISING COMMITTEE 13-14 JAN 2006, UNIVERSITY OF AVEIRO, PORTUGAL

IV CONGRESSO IBEROAMERICANO DE INVESTIGACION EN CELULOSA Y PAPEL NETO CP: SCIENTIFIC ADVISORY COMMITTEE 23-27 OCT 2006, SANTIAGO E VALDIVIA, CHILE

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*OLIMPÍADAS IBEROAMERICANAS DE QUÍMICA* DA SILVA, F; FRANCISCO, A: SCIENTIFIC ADVISORY COMMITTEE 07-15 SEPT 2006, UNIVERSITY OF AVEIRO, PORTUGAL

SEMINÁRIO ACERCA DA UTILIZAÇÃO DA DIFRACÇÃO DE RAIOS-X APLICADA A CARACTERIZAÇÃO DE HETEROESTRUTURS ELECTRO-CERÂMICAS PEREIRA, S UNIVERSITY OF AVEIRO, PORTUGAL

WORKSHOP – NANOTECHNOLOGIES IN CONSTRUCTION BARROS-TIMMONS, A 03 JUL 2006, UNIVERSITY OF AVEIRO, PORTUGAL

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XII OLIMPIADAS IBERO-AMERICANAS SANTOS T M: SCIENTIFIC ADVISORY COMMITTEE 07-11 SEPT 2006, UNIVERSITY OF AVEIRO, PORTUGAL

XVTH NATIONAL CONGRESS OF BIOCHEMISTRY PEREIRA, ML: ORGANISING COMMITTEE 08-10 DEC 2006, UNIVERSITY OF AVEIRO, PORTUGAL

# **COURSES, SEMINARS AND TRAINING PROGRAMMES**

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*FAME - ERASMUS MUNDUS MASTER PROGRAMME IN FUNCTIONALIZED ADVANCED MATERIALS AND ENGINEERING* BARROS-TIMMONS, A UNIVERSITIES OF AUSBURG, GRENOBLE, AVEIRO, BORDEAUX, DARMSTADT, LIÉGE, LOUVAIN, 2006

*1° CURSO DE TRABALHOS PRÁTICOS DO 12° ANO* RIBEIRO-CLARO, P UNIVERSITY OF AVEIRO, PORTUGAL, 14 JAN - 11 APR 2006

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### **REACHING OUT ACTIVITIES**

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*COMUNICAÇÕES EM EVENTOS PÚBLICOS KINETICS OF SOLID STATE PROCESSES UNDER VARIABLE TEMPERATURE* FRADE, JR

UNIVERSIDAD DE LA LAGUNA/SPAIN/TENERIFE, 05 APR 2006

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### **PROJECTS TERMINATED**

AERATION OF MULTIPHASE BIOLOGICAL REACTORS POCTI/EQU/44427/2002

E-ECORISK – A REGIONAL ENTERPRISE NETWORK DECISION SUPPORT SYSTEM FOR ENVIRONMENTAL RISK AND DISASTER MANAGEMENT OF LARGE SCALE INDUSTRIAL SPILLS EVG1-CT-2002-00068

HYBRID MATERIALS PREPARED BY IRRADIATION POCTI/CTM/44150/2002

NETWORKED CENTRE OF EXCELLENCE IN MATERIALS FOR THE ECONOMIC DEVELOPMENT OF THE ATLANTIC AREA – MNAA

OPTICAL WAVEGUIDE LASERS BASED ON HYBRID SOL-GEL DERIVED MATERIALS DOPED WITH LANTHANIDE IONS POCTI/CTM/42478/2001

BIOMATERIAIS DE SI3N4 REVESTIDOS A DLC E DIAMANTE CVD – BIODIAM POCTI/CTM/45423/2002

CONTROLO DE QUALIDADE NA ÁREA DA TECNOLOGIA DE FUNDIÇÃO

DEPOSIÇÃO DE CERAS PARAFÍNICAS EM OLEODUTOS

ESTUDO COMPARATIVO DO COMPORTAMENTO "IN VITRO" DE VIDROS DO SISTEMA SI-CA-P-MG, EM ESPÉCIMES MONOLÍTICOS E NA FORMA DE FILMES

LABORATÓRIO DE MICROSCOPIA ELECTRÓNICA ANALÍTICA DE RESOLUÇÃO ATÓMICA - GRANDES EQUIPAMENTOS DE USO COMUM REEQ

NANOCOMPÓSITOS DE MATRIZ CARRAGENANO PARA APLICAÇÕES BIOMÉDICAS

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### **PROJECTS IN PROGRESS**

ADOPTIC - ADDITIVE OPTIMISATION FOR IMPROVED CERAMICS SIXTH FRAMEWORK PROGRAMME (FP6).

ADVANCED ENVIRONMENTALLY FRIENDLY MULTIFUNCTIONAL CORROSION PROTECTION BY NANOTECHNOLOGY 11783-2 MULTIPROTECT

APPLICATION OF NMR METHODS FOR THE CHARACTERIZATION AND QUALITY CONTROL OF BEER UNICER-BEBIDAS DE PORTUGAL, SGPS, S.A.

BIOACTIVE TEXTILES USING FUNCTIONAL BIOPOLYMERS POCI/CTM/58312/2004

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CARACTERIZAÇÃO E DESENVOLVIMENTO DE AGLOMERANTES PARA A INDÚSTRIA DE MÓS ABRASIVOS ACORDO DE COOPERAÇÃO

CARBIFINO – CONCEPÇÃO DE NOVOS GRAUS DE METAL DURO DE GRANULOMETRIA SUB- A NANO-MÉTRICA COM ELEVADA RESISTÊNCIA AO DESGASTE EROSIVO AL PRIME IDEIA 70/00090

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COMPLEXOS DE METAIS DE TRANSIÇÃO(III) E LANTANÍDEOS(III) COMO CATALISADORES NA POLIMERIZAÇÃO DE OLEFINAS PTDC/CTM/67409/2006 CONTROLO DE QUALIDADE NA ÁREA DA TECNOLOGIA DE FUNDIÇÃO

PROTOCOLO DE COLABORAÇÃO ENTRE FUNFRAP – FUNDIÇÃO PORTUGUESA E A U.AVEIRO

CONVERSÃO ELECTROQUÍMICA DE COMBUSTÍVEIS (ECOFUELS) REEQ

CORE-SHELL CERAMIC MEMBRANES WITH NANO-SIZED GRAINS FOR OXIGEN SEPARATION AND SYN-GAS PRODUCTION POCI/CTM/59727/2004

DESENVOLVIMENTO DE UMA SONDA DE MICROSCOPIA DE FORÇA ATÓMICA PARA MAPEAMENTO DE PH À NANOESCALA EM SUPERFÍCIES PTDC/CTM/74290/2006

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DETECTANDO A MIRCO-DISTRIBUIÇÃO DE ESPÉCIES QUÍMICAS EM SOLUÇÃO NA VIZINHANÇA DE METAIS ACTIVOS PTDC/CTM/66041/2006

DEVELOPMENT AND CHARACTERIZATION OF CERAMIC COMPOSITE MATERIALS FOR THERMOMECHANICAL APPLICATIONS REEQ

DEVELOPMENT OF A FIXED SITE CARRIER CERAMIC ULTRAMICROPOROUS MEMBRANE AND CATALYTIC MEMBRANE REACTOR FOR OLEFINS SEPARATION/PURIFICATION POCI/EQU/59344/2004

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DEVELOPMENT OF A NEW PHASE CHANGE MATERIAL COMPOSITE FOR ENERGY STORAGE AND THERMAL INSULATION POCI/CTM/60288/2004

DEVELOPMENT OF NEW INTERSTITIAL OXIDE-ION CONDUCTORS FOR EFFECTIVE OXYGEN SEPARATION AND CONVERSION OF THE HYDROCARBONS POCI/CTM/59197/2004 DEVELOPMENT OF NEW NANOSTRUCTURED HYBRID SOL-GEL COATINGS MODIFIED WITH ORGANIC INHIBITORS FOR CORROSION PROTECTION OF METALLIC SUBSTRATES POCI/CTM/59234/2004

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