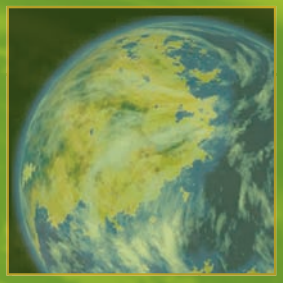
ACTIVITY REPORT 2005

and

ACTIVITY PLAN 2006

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SECTION 1

FACTS AND NUMBERS

Established in the University of Aveiro in 2002, the Centre for Research in Ceramics and Composite Materials (CICECO) is a national institute of excellence in the field of ceramics and composite materials, engaged in the development of the regional economy. CICECO is an Associate Laboratory of the Portuguese Ministry of Science, Technology and High Education.

This centre brings together the expertise of physicists, chemists, materials scientists and engineers from some of the best research groupes in the country. The evolution of CICECO in the past four years has been very positive: Research Staff and Scientific Output increased significantly (Table 1).

Table 1: CICECO's Research Team, 2002-2005

	2002	2003	2004	2005
Professors and Lecturers	47	47	*50*	***53***
Full Time Researchers	5	9	*12*	***16***
Post-Doctoral Associates	23	22	*29*	***40***
Collaborators	16	13	*12*	***11***
PhD Students	54	60	*61*	***54***
MSc Students and Other Students	26	44	*54*	***60***
Laboratory Technicians	4	8	*5*	***5***
Administrative Personel	1	3	*5*	***5***
	17U	**20U**	**228**	**244**

On 31ST December 2005 **CICECO** hosted 244 people, an increase of 7.0% over 2004 and 38.6% relatively to 2002. In particular, the number of Full-Time Researchers, Graduated Students and Post-Docs rose significantly.

A full list of the Research Team members is given at the end of Section 1.

A total of 94 PhD and MSc degrees have been awarded in the period 2002-2005. One third of these degrees were terminated during 2005. The number of papers published in this year in SCI journals was 306 (Table 2).

The number of SCI papers published in large Impact Factor Journals (IF≥5) has been increasing.

A complete list of CICECO's Scientific Production in 2005 is given in Section 4.

Table 2: CICECO's Scientific Output, 2002-2005

		2002	2003	2004	Corrigendum 2004	2005
Theses	MSc	10	8	*7*	-	***17***
	PhD	14	13	*11*	-	***14***
Books	Editions	0	0	*1*	-	***1***
	Chapters	4	14	*12*	-	***7***
SCI Papers	IF≥5	1	3	*4*	-	***6***
	IF<5	204	225	*284*	***4***	***296***

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Over the past four years 20 Patents have been filled.

The number of projects in progress in 2005 was 93, similar to 2004 (103) and 2003 (100). The main source of funding continues to be FCT (National Science Foundation) and the European Funding Agencies and Programmes (E. Commission, FEDER, INTERREG IIIB).

Section 5 lists all the Projects funded.

CDTM Centro de Design
e Tecnologia de Materiais

CENTRE FOR MATERIALS DESIGN & TECHNOLOGY (CDTM)

The Centre for Design and Technology of Materials (CDTM) is the CICECO unit responsible for the promotion of technology and knowledge transfer. CDTM works closely with researchers supporting them in: a) management of R&DT projects developed in partnership with companies; b) intellectual property protection; c) creation of new companies (spin-offs) based on technologies developed at CICECO, and d) commercialisation of technologies by licensing to existing companies. In addition, CDTM promotes training courses and technical workshops.

R&D projects in consortium

CDTM has managed several R&DT projects in partnership with companies aiming a developing new materials, process enhancement and resolution of specific industrial (12 months duration) problems. In 2005, CDTM worked with the folowing companies:

- Caima, Indústria de Celulose, SA, 'Improving the yield and the physico-mechanic characteristics of eucalyptus wood pulp obtained by the magnesium–based suphite;
- UNICER - Bebidas de Portugal SGPS, SA, "Chemical characterisation of beer by NMR';
- RAIZ, Instituto de Investigação da Floresta e Papel, 'Interaction between ink and paper';
- CERISOL - Isoladores Cerâmicos SA e MOTA - Pastas Cerâmicas SA, Technical assistance contract;

***FoodMetric*: a new CICECO spin-off**

A new spin-off company, *FoodMetric*, is being created with the support of CDTM, in collaboration with the Chemistry Department (Food Chemistry Group). This company will provide solutions to food and drink companies through the implementation of fast and reliable analytical methods for food analysis. The solutions encompass systems composed of spectrometers and software, which are able to meet customer's needs. Several prizes in national entrepreneurial contests have been awarded to *FoodMetric*: first prize at "Concurso Nacional de Empreendedores", first prize at "2º Concurso de Criação

de Empresas de Base Tecnológica de Mira" and second prize at "Concurso Bioempreendedor". *FoodMetric* has already obtained financial support from NEOTEC (programme promoted by AdI) and venture capital investors.

Technology transfer contracts

Two new contracts of technology transfer were established with Merck KGaA ('Synthesis and application of ionic liquids for the dehydration of mono- and poly- saccharides') and Alfama SA 'Inclusion and conjunction of CORMs with ciclodextrines molecules and with micro or mesoporous inorganic materials'). The main objective of these contracts is to develop new materials for industrial applications.

Marketing, promotion and awareness of CICECO

The promotion and awareness of CICECO in society has been improved by organising several technical workshops and training programmes, focused in the areas of polyurethanes, cements, ceramics, glasses and sustainable development. The following events gave an important contribution to the dissemination of the main results obtained by CICECO's researchers:

- 'Materials and cultural heritage', Complexo Pedagógico da Universidade de Aveiro, 20th March 2005
- 'Material for micro/nano-preparation of structural components', Complexo Pedagógico da Universidade de Aveiro, 23rd March 2005.

- 'Conforming in ceramics technology': development perspectives', Departamento de Engenharia Cerâmica e do Vidro da Universidade de Aveiro, 16th May 2005
- 'Drying and firing in ceramics technology: development perspectives', Departamento de Engenharia Cerâmica e do Vidro da Universidade de Aveiro, 19th May 2005
- 'Polyurethanes: fundamental aspects and manufacturing processes', Departamento de Mecânica da Universidade de Aveiro, 30th September 2005
- 'CIAT: $C0_2$ in high-temperature industries', Departamento de Mecânica da Universidade de Aveiro, 16th December 2005

These seminars were attended by a total of 350 participants (27% industrialists).

CDTM participated, with ten prototypes developed at CICECO, in the international exhibition of technologies and products for industry 'Evento de Investigação & Inovação Tecnológica', FIL - Feira Internacional de Lisboa, November 16-19, 2005. Around 15 000 visitors visited this exposition.

A 100 hours training programme on 'Environment and sustainable development' (legislation, recycling and development of new products) has been organized and had the participation of 16 industralists.

***IDPoR*: a new CICECO R&D platform**

CDTM is setting up a research and development platform aiming at the cooperation between university and industry. Polymer-based activities and the transformation of forest-derived materials (pulp-and-paper, cork, wood products) play a major role in the Portuguese industrial sector. Petrochemistry-derived polymers are slowly being replaced by polymers obtained from

renewable sources, but the lack of interest in these novel materials by the scientific community is a matter of concern. Furthermore, the transfer of knowledge from the academic to the industrial world is difficult to achieve. To fill in these gaps, CICECO and the Department of Chemistry of the University of Aveiro started a *Research and Development platform on Polymers from Renewable Sources, IDPoR*. A consortium has been created between a group of companies and CICECO, involving the major Portuguese companies in this field (eg. *CIN, CAIMA, Corticeira Amorim, Resiquímica, RAIZ, Sonae Indústria,* etc.). *IDPoR* promotes a closer relation between these companies and CICECO and encourages the development of training courses, workshops and, more importantly, an ambitious Ph.D. programme. *IDPoR* associates will have access to the research output of several Ph.D. students, which will be made available exclusively for them, on the *IDPoR* webpage (in preparation). The associate companies will pay a yearly fee to join *IDPoR*, for a period of five years. The promoters of *IDPoR* are Professors J. Pedrosa, A. Gandini and C. Pascoal Neto.

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SECTION 2

ACTIVITY REPORT 2005

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

INORGANIC MULTIFUNCTIONAL MATERIALS AND ORGANIC-INORGANIC HYBRIDS

New Microporous Materials. The hydrotherm l synthesis of sm ll-pore N $_2$SnSi$_4$O$_{11}$·2H$_2$O (Sn-AM-3), exhibiting the structure of miner l penkvilksite-2*O*, h s been reported. This is the first ex mple of synthetic microporous st nnosilic te whose structure is built up from silic te sheets. Solid solutions of synthetic n logues of the microporous miner l umbite K$_2$[(Zr$_{1-x}$Hf$_x$)Si$_3$O$_9$]·H$_2$O (x = 0, 0.17, 0.33, 0.5, 0.67, 0.83 nd 1) h ve been prep red nd ch r cterised. The second ex mple of microporous copper silic te N $_2$(Cu$_2$Si$_4$O$_{11}$)·2H$_2$O (AV-23) open-fr mework h s been reported. AV-23 possesses ch nnels running long the *a* direction, formed by eight-rings, with n effective cross-section of c . 4.2×2.5 . This m teri l exhibits singlet ground st te t low temper ture, which is conditioned by the topology of the isol ted edge-sh ring CuO$_6$ (or CuO$_5$) ch ins. The hydr ted nd dehydr ted forms exhibit strong ltern tion of the exch nge inter ction p r meter within these ch ins. M gnetic susceptibility nd specific he t me surements confirm spin g ps of Δ=96 nd 85 K for, respectively, the hydr ted nd dehydr ted forms. The reversible dehydr tion ffords n unique opportunity for tuning the m gnetic inter ction p r meter.

A new tit nosilic te umbite membr ne w s prep red on porous tit ni tubul r supports. This membr ne is ble to sep r te H$_2$/N$_2$ mixtures, with selectivities s high s 48, even in the presence of w ter nd, thus, it m y be of interest in the purific tion of H-cont ining stre ms nd H$_2$ proton-exch nge membr ne fuel cells. Pure tit nosilic te (ETS-10) nd v n dosilic te (AM-6) membr nes were synthesised on cer mic tubul r supports by seeded hydrotherm l synthesis. The membr nes h ve been ch r cterised by XRD, SEM nd temper ture progr mmed perme tion to me sure the single g s perme nces showing ctiv ted flow. At mbient temper ture, the membr nes exhibit promising propylene/prop ne sep r tion f ctors (between 3.8-6.5 for ETS-10 membr ne nd bout 2.3 for AM-6 membr nes). Eu^{3+}-ETS-10 powders nd films supported on dense α-lumin nd st inless steel substr tes were prep red vi seeded hydrotherm l synthesis, followed by convention l ion-exch nge. ETS-10 films nd powders exhibit different photoluminescence ch r cteristics, which were studied in det il. Possible re sons for this disp r te beh viour were discussed.

A series of zeolite-type silic tes cont ining fr mework l nth nides (N $_4$K$_2$)(Ln$_2$Si$_{16}$O$_{38}$)·10H$_2$O (Ln = Nd, Sm, Eu, Tb, Gd, Dy) h ve been synthesised. Aqueous suspensions of these m teri ls h ve been shown to be very effective in enh ncing tr nsverse rel x tion, p rticul rly t high m gnetic fields. They re, thus, ttr ctive s T$_2$ m gnetic reson nce im ging contr st gents.

165 different structur l types of zeolitic m teri ls h ve been identified, nd every ye r m ny new structures re considered by the *International Zeolite Association* for inclusion in their d t b se. We h ve used comput tion l system tic enumer tion (b sed on dv nces in combin tori l tiling theory) to derive ll possible binod l structures (*i.e*, with 2 cryst llogr phic lly-independent tetr hedr l sites) b sed on simple tilings. E ch of the 109 refine ble topologies h s been converted into silic

polymorph, its energy minimised and compared with that of α-quartz, and the volumes accessible to sorption, determined. 11 of the 30 known bimodal topologies listed in the Atlas of Zeolite Framework Types have been found, leaving 98 unknown topologies, many of which are chemically feasible.

Layered Materials. The study on photoluminescent layered silicates known as AV-22 materials $K_3[M_{1-a}LnaSi_3O_8(OH)_2]$ ($M=Y^{3+}$, Tb^{3+}; $Ln=Eu^{3+}$, Er^{3+}, Tb^{3+}, and Gd^{3+}, has been continued.

Even though the isolation of γ-titanium phosphate intercalation compounds with dialkylamines was already known, the use of monoalkylamines as templates have been reported for the first time. The first member of this family, $(C_6H_{13}NH_3)[Ti(HPO_4)(PO_4)]\cdot H_2O$, has been prepared under hydrothermal conditions and characterized. The layered nature of the material is clearly shown by the powder XRD patten via strong preferential orientation, which avoided a reliable *ab initio* structure solution. Also using hydrothermal synthesis, another hybrid layered material containing V^{4+} centres, $(C_{10}H_{10}N_2)[(VO)(HPO_4)]_2(C_2O_4)$, has been isolated and structurally characterised by using X-ray diffraction methods. The compound is formed by two-dimensional $\{[(VO)(HPO_4)]_2(C_2O_4)\}_n^{2n-}$ anionic layers exhibiting small and distorted rectangular pores having a cross-section of 2.5×2.0 Å. These layers are intercalated by 4,4'-bipyridinium cations ($C_{10}H_{10}N_2^{2+}$), which are strongly hydrogen-bonded to the inorganic two-dimensional skeleton via very strong N^+─H···O interactions.

The synthesis and the characterisation of layered materials have been performed and the intercalation of metallo-organic complexes into the prepared layered double hydroxides (Mg-Al and Zn-Al LDHs) has been accomplished. The photofunctional and/or catalytic properties of several of these materials have been tested.

Nanostructured Materials. Cadmium sulfide and cadmium selenide/polymer nanocomposites have been prepared via in-situ radical polymerisation in a miniemulsion. Organicall-capped CdE (E =S,Se) quantum dots (QDs) have been used as the starting materials, and ensembles of these dots have been encapsulated with no need of further surface treatment. The use of two polymer matrices have been investigated: poly(styrene) and poly(n-butylacrylate). In both cases, homogenous nanocomposites have been obtained and their optical properties were studied by visible absorption and photoluminescence spectroscopy. Quantum-size effects have been assigned to the nanocomposites, indicating the integrity of the individual QDs upon polymer encapsulation using the miniemulsion process. The hybrid nanomaterial is very stable and presents a bright green photoluminescence at 2.29 eV under ultraviolet excitation. With the excitation conditions used the intensity of the emission band remains nearly constant from 7 K to room temperature. The morphological, structural and room-temperature electrical properties of the CdSe/poly(butylacrylate) nanocomposite have been investigated. Studies concerning the potential use of these nanoparticles as bio-markers for in vitro bioapplications have started.

Cadmium selenide nanocrystals have been grown over silica surfaces by a one-step synthetic method involving the thermal degradation of the single-molecule precursor $[Cd(Se_2CNEt_2)_2]$.The powder XRD patterns of the SiO_2/CdSe nanocomposites

show diffraction peaks consistent with the presence of hexagonal-CdSe. SEM and TEM show inorganic composite particles formed by islands of CdSe nanocrystals at the SiO_2 surfaces. A series of SiO_2/CdSe particles have been obtained, showing quantum size effects in their optical spectra.

Novel Pigments. Studies on the incorporation of d-metals in $BiVO_4$ pigment particles have been carried out, using a precipitation method based on the controlled rellease of cations in aqueous solution. NMR and Raman have been employed to monitor the synthesis of the pigment particles.

Polyoxometalates. Research on novel luminescent systems based on lanthanide complexes and their incorporation in nanomaterials has been continued. Research concerned the coordination chemistry of lanthanides with derivatised [60]fullerene ligands and aromatic ambidentate ligands, exploring the possibility of formation of multidimensional compounds. The incorporation of those lanthanide compounds into nanosized SiO_2 and other substrates has been explored. The luminescence and structural properties have been studied.

New compounds have been obtained with: $[PM_{12}O_{40}]^{3-}$ or $[SiM_{12}O_{40}]^{4-}$ (M = Mo, W) and *S*-histidine, *S*-tryptophan, 4-aminopyridine, 4-phenylpyridine, 2,3-dihydroxypyridine, 8-hydroxyquinoline and 2-aminoethyiamino-5-nitropyridine; and $[BW_{11}Fe(H_2O)]^{6-}$ or $[SiW_{11}Mn(H_2O)O_{39}]^{5-}$ with aminoacid derivatives, namely *L*-arginine, *L*-aspartic acid dimethyl ester and *L*-leucine methyl ester. The first set of compounds has been obtained in different forms by two different synthetic procedures. Their non-linear optic properties have been evaluated by the Kurz-powder method, but only a few presented second-harmonic generation. The second compounds set has been used in studies of catalytic oxidation of terpenes. Catalytic oxidation work with H_2O_2 of cycloalkanes and fused cycloalkylaromatics continued. Interesting results have been obtained in the oxidation of cycloalkanes (cyclohexane, cyclooctane and cyclododecane) using as catalysts the tetrabutylammonium salts of the sandwich type polyoxotungstates $[M_4(H_2O)_2(PW_9O_{34})_2]^{n-}$, M = Co(II), Mn(II) and Fe(III). Other studies concerned the oxidation of indane, tetralin and cyclododecane in the presence of the Fe-substituted polyoxotungstates, $[XW_{11}Fe(H_2O)O_{39}]^{n-}$.

Preparation and characterisation of lanthanide complexes of lacunary polyoxomolybdates or polyoxotungstates has been continued. The crystal structure of a novel compound, $H_2(NH_4)_{10}[Ce_2(BW_{11}O_{39})_2(H_2O)_6]\cdot 21H_2O$ has been solved. This is the first example of a one-dimensional polymer where Keggin-type anions containing a central boron atom are bridged by Ce^{3+} cations. The crystal structure of a compound with a lanthanum polyoxotungstate complex, $Na_2(NH_4)_7[La(W_5O_{18})_2].16H_2O$, has been determined. The application of the compounds in the preparation of polyoxometalate-based materials has been explored, namely the preparation of mono- or multi-layered nanostructured films and polyoxometalate-anion-pillared layered double hydroxides. Investigation started on the preparation and structural characterization of new organic/inorganic hybrid coordination networks using lanthanopolyoxometalates and aromatic ambidentate ligand.

Novel Luminescent Systems. The Lewis base adducts $Ln(NTA)_3L$ and $[Ln(NTA)_3]_2\cdot$bpym [Ln = Eu, Gd; NTA = 1-(2-naphthoyl)-3,3,3-trifluoroacetone; L = 1,10-phenanthroline or ethyl[3-(2-pyridyl)-1-pyrazolyl]acetate, bpym = 2,2'-

bipyrimidine] have been prepared and characterised. Similar complexes of the type $Ln(NTA)_3L$ have been immobilised in the ordered mesoporous silica MCM-41 by complexation of $Ln(NTA)_3$ with a pyrazolylpyridine ligand covalently anchored to the support. Evidence for tethering of coordinatively saturated complexes has been obtained. An unusual two-step intermolecular energy transfer between 'free' and complexed ligands, enhancing the Eu^{3+} luminescence, is observed. Layered double hydroxide pillared by 2,2'-bipyridine-5,5'-dicarboxylate anions has been used as a porous matrix to intercalate $LnCl_3$ (Ln = Eu, Gd). The photoluminescence studies for the Eu-containing material showed the existence of only one type of Eu^{3+} binding site, probably involving four-coordinated water molecules. Taking into account Eu L_3-edge EXAFS results, it was concluded that the europium ions are six-coordinate, with one bidentate bipyridyl ligand.

A covalency scale has been proposed for lanthanide compounds using an analytical function (the covalent fraction) of the overlap polarizability. The inverse of a parameter appearing in this function is interpreted as a typical average volume of the overlap region. The red shift in the nephelauxetic effect for the $^5D_0 \rightarrow {}^7F_0$ transition has been investigated, for 15 europium compounds, in terms of the ligand field strength parameter, N_v, and the covalent fraction. This shift o increases with the covalent fraction.

Crystal Engineering of Organic-Inorganic Hybrids. The isolation of novel crystalline hybrid materials has been based on the use of N-(phosphonomethyl)iminodiacetic acid (H_4pmida, a highly flexible organic precursor of a multidentate chelating ligand) and 3-hydroxypicolinic acid (HpicOH). H_4pmida self-assembles with V^{4+}, under mild hydrothermal synthetic conditions, to form centrosymmetric anionic $[V_2O_2(pmida)_2]^{4-}$ units, which have been used as robust Secondary Building Units to construct the first 2D hybrid framework, $[Co(H_2O)_6]\{[Co(pyr)(H_2O)_2][V_2O_2(pmida)_2]\}\cdot 2(H_2O)$ (where pyr stands for the pyrazine). During the course of the year we have taken advantage of the coordinating flexibility of H_4pmida to isolate materials with other transition-metal centres. For instance, $[M(pyr)(H_2O)_4][M_2(Hpmida)_2(pyr)(H_2O)_2]\cdot 2(H_2O)$ (py =pyrazine and M = Co^{2+} or Ni^{2+}) have been isolated and characterised structurally. The magnetic behaviour of these compounds deviates from a simple paramagnetic Curie-like behaviour, and has been attributed to antiferromagnetic interactions between the various metallic centres or single-ion anisotropy and crystal-field effects. During our incursion into the use of transition metal centres, the $[Cu(H_2pmida)(phen)]$ (phen=1,10-phenanthroline) complex has been isolated in a highly crystalline form, and its crystal structure re-determined at 180 K.

Despite its aromaticity, HpicOH exhibits a rich coordination chemistry, with O,O-chelation (achieved via deprotonation of the carboxylic acid and hydroxyl groups) being observed for complexes with Cu^{2+} and Co^{2+}. After isolating $[Cu(picOH)_2]$ complexes, which exhibit a typical square-planar coordination geometry, 1,2-bis(4-pyridyl)ethane (BPE) molecules have been employed to establish physical links between adjacent complexes. A novel material, $[Cu(picOH)_2(BPE)]_2\cdot[Cu(picOH)_2(BPE)_2]\cdot 8H_2O$, havs been isolated, containing 1D neutral $[Cu(picOH)_2(BPE)]$ coordination polymers close-packing with individual $[Cu(picOH)_2]$ neutral complexes. The use of BPE imposes a relatively long inter-metallic distance, leading to negligible magnetic interactions. The reaction of HpicOH with Co^{2+} centres leads to the isolation

$[Co(picOH)_2(H_2O)_2]$ and *mer*-$[N(CH_3)_4][Co(picOH)_3]\cdot H_2O$, exhibiting slight deviations from a paramagnetic (Curie-like) behaviour, ascribed to the different coordination spheres of the metallic centres.

Organic-Inorganic Hybrids Lacking Activating Centers. Organic–inorganic hybrids have been prepared with ureapropyltriethoxysilane, methacryloxypropyltrimethoxysilane and acrylic acid modified zirconium(IV) n-propoxide precursors. Planar waveguides have been obtained by spin-coating of the prepared sols on sodalime and silica substrates. Refractive index, thickness, number of propagating modes, and attenuation coefficient have been measured at 543.5, 632.8 and 1550 nm by the prism coupling technique. The synergism between the two hybrid precursors resulted in monomode planar waveguides with low loss in the infrared (0.6–1.1 dB/cm) which also support a number of propagating modes in the visible (losses from 0.4–1.5 dB/cm). Channel waveguides have been obtained by UV photo-patterning, using amplitude or phase masks and propagating modes have been observed at 1550 nm. The zirconium-based nanoparticles and the siliceous nanodomains interact, inducing changes in the hybrids emission features.

New Hybrid Materials. Sol-gel derived amide cross-linked alkylene–siloxane hybrids (di-amidosils) have been prepared as transparent, amorphous and rigid monoliths. The materials are stable up to *ca.* 245 ºC and their siliceous matrices mainly composed of $[-(CH_2)Si(OSi)_3)]$ and $[-(CH_2)Si(OSi)_2(OH)]$ sub-structures. Structural unit lengths of 4.1 - 4.2 Å and average interparticle distances of 12 - 17 Å have been obtained, depending on the number of methylene groups of the alkylene chain. The chains are disordered and adopt gauche conformations. The hybrids are room-temperature white-light emitters, displaying a strong broad emission in the blue/purplish-blue spectral region, ascribed to the convolution of donor–acceptor pair (D–A) recombinations occurring in the NH groups of the amide linkages and in the siliceous nanodomains. The maximum quantum yield is 5.4%.

Sol-gel derived bifunctional bioactive hybrids with interesting photoluminescence properties have been prepared from chitosan and a silane coupling agent in which the covalent bridges, essentially urea, link the chitosan to the poly(siloxane) network. The presence of siloxane nanodomains has been detected by SAXS. The photoluminescence spectra display an additional high-energy band with a lifetime longer than that of the emission of pure chitosan. This band is associated with electron–hole recombinations arising from silicon-related defects at the surface of the siliceous nanodomains. The bioactive behaviour of the materials was also evaluated. Apatite formation depends on the number and arrangement of silanol groups.

Magnetic studies of natural ferritin and iron oxides (ferrihydrite and others) nanoparticles in organic-inorganic hybrids have been conducted. The studies included: effect of partice size and magnetic moment distributions on the magnetic properties; simulation of magnetic properties (Monte-Carlo); influence of magnetic field on the growth and morphology of hybrid materials.

In order to investigate the ability of different hybrid materials to efficiently incorporate lanthanide ions, protecting them from non-radiative channels, thus enhancing their photoluminescence properties, the following organic-inorganic hybrids were synthesised and investigated.

i) Di-ureasils doped with a Eu^{3+} β-deketonate complex. Di-ureasils incorporating $Eu(nta)_3bpy$ (nta=1-(2-naphthyl)-4,4,4-trifluoro-1,3-butanedionate and bpy=2,29-bipyridine, respectively) have been prepared by the acetic acid solvolysis or conventional hydrolysis sol–gel routes. The dimension and condensation degree of the siloxane nanodomains depend on the synthesis route, while the overall emission quantum yield decreases from 15 (conventional hydrolysis) to 6% (solvolysis route). The broad white-light emission typical of the di-ureasil host is not detected, suggesting the activation of energy transfer channels between the hybrid host emitting centres and Eu(III) ions. As the Eu(III) first coordination shell is independent of the synthesis method, the decrease in the emission quantum yield for the di-ureasil prepared by acetic acid solvolysis may be explained by the interaction between the hybrid emitting centres and the nta ligand levels, favouring a larger non-radiative transition probability.

ii) Di-ureasils and di-urethanesils doped with $Nd(CF_3SO_3)_3$. Di-urea and di-urethane cross-linked poly(oxyethylene) (POE)/siloxane hybrids doped with neodymium triflate ($Nd(CF_3SO_3)_3$) have been studied. The goals of this work are to determine which cation coordinating atom of the host matrix (ether or carbonyl oxygens) is active in each material, to analise its influence on the nanostructure of the samples and relation with the photoluminescence properties. The main conclusion is that hydrogen-bonds play a major role in the hybrids nanostructure and properties.

iii) Mono-urethanesils doped with $Eu(CF_3SO_3)_3$. The anionic and cationic local environments in mono-urethanesils doped with europium triflate have been studied. The host matrix of these materials consists of a siliceous backbone bonded through urethane linkages to CH_3-terminated polymer chains containing about seven OCH_2CH_2 units. Samples with $\infty \geq n \geq 5$ ($n=OCH_2CH_2/Eu^{3+}$) have been studied. In terms of ionic association, the level of complexity of these xerogels is very high. The triflate ions are present 'free', weakly coordinated and forming cross-link separated ion pairs. For $20 \geq n \geq 5$, in addition to these species, contact ion pairs also form. Photoluminescence shows the presence of three distinct cation sites (Eu^{3+}/O=C(urethane) cross-links), Eu^{3+}/O-C-C(polyether chains) and weakly coordinated $Eu^{3+}/CF_3SO_3^-$ ionic pairs).

Di-ureasils doped with a wide concentration range of lithium or magnesium triflate, $\infty \geq n > 1$ have been obtained as amorphous monoliths, stable up to *ca.* 340 ºC. Crystalline complexes are detected in samples with $n \leq 10$. Below 90 ºC the hybrids with $n = 20$ exhibit the highest conductivity: 5.8 (28 ºC) and 4.0×10^{-6} $\Omega^{-1}cm^{-1}$ (35 ºC), for the Li- and Mg-hybrids, respectively. The redox stability domain of these hybrids ranges from 4.1 (Li/Li^+) to 3.0 V (Mg/Mg^{2+}). Although FT-IR suggests that the Li^+ and the Mg^{2+} ions are complexed by the POE ether oxygen atoms for $n \leq 10$, this threshold composition is probably at a slightly lower salt content. For n>10 the cations were coordinated to the urea carbonyl oxygens. 'Free' triflate ions and weakly coordinated anions are the main charge carriers. Ion pairs or negatively charged triplets are formed for $n \leq 40$. For $n \leq 5$, positively charged triplets also appear. In the Li-based hybrids multiplets $[Li_3(CF_3SO_3)]^{2+}$ occur at $n = 1$.

C60 Phase Transitions Under High-Pressure. The low-resolution structures of three new high-pressure (>100 kBar) phases of C_{60} have been determined. These phases are characterised by a cuboid cage structure, instead of the 'usual C_{60} spherical

cage, and may be viewed as carbon clathrates, since they do not have a molecular nature. They were previously synthesised during *in-situ* synchrotron diffraction studies of the pressure-temperature phase diagram of C_{60}, at ESRF.

Chemical Modification of Electrodes With Functional Materials. Novel modified electrodes have been prepared and characterized, comprising a mixed layer of ion-exchange polyelectrolytes, poly-L-lysine (PLL) and poly(sodium 4-styrenesulfonate) (PSS) adsorbed onto glassy carbon and further modified with a thin mercury film. The effects of the coating morphology, thickness and the monomeric molar ratio PLL/PSS on the cation exchange ability of the PLL-PSS polyelectrolyte coatings have been evaluated using target species, such as dopamine or lead cation. The semi-permeability of the PLL-PSS coated electrodes based on electrostatic interactions and molecular size, assured an improved anti-fouling ability against several tensioactive species. The analytical usefulness of the PLL-PSS polyelectrolyte coatings on thin mercury film electrodes has been demonstrated *via* square-wave stripping voltammetric measurements of toxic metals (lead, copper and cadmium at the low nanomolar level; accumulation time of 180 s) in estuarine waters containing moderate levels of dissolved organic matter, resulting in a fast and direct methodology requiring no sample pre-treatment.

Another approach was the modification of glassy carbon surfaces with adsorbed single layers of hybrid compounds of heteropolysilicotungstates $[SiW_{11}O_{39}]^{n-}$ and $[SiW_{11}M(H_2O)O_{39}]^{n-}$, M = Fe(III), Co(II), Mn(II), and organic cation tetrabutylammonium. The electrochemistry of the immobilized hybrids has been assessed, namely the redox reversibility and other electrochemistry parameters (formal potentials, number of transferred electrons), effects of the solution pH on the overall behaviour and effects of the loading and electrochemical pre-treatments on the coatings stability.

Some work has been carried out on the chemistry of binary systems like sediments/water, regarding the kinetics of metal sorption processes. These studies are important for understanding the behaviour of electrodes functionalised with mineral phases, such as clays.

Development of Spectroscopic and Electrochemical Techniques. The use of linear scan and square-wave voltammetry in the characterisation of cork stoppers has been successfully used for the first time. In this study 'finger print' type voltammetric scans led to the assignment of specific signals to lignin related phenolics. These signals may be used to follow lignin degradation in the materials. It is possible to distinguish wine model matrices that have been in contact with contaminated cork, confirming that voltammetric data may be used for both monitoring potential cork-wine interactions and as a new, swift, quality control tool in cork stopper and wine-making industries. A procedure for the quantification of the antibiotic trimethoprim (TMP) by adsorptive stripping voltammetry has been developed to the analysis of TMP in complex matrices, such as pharmaceutical suspensions. Mechanistic data on the reduction/adsorption of TMP has been obtained.

Mass spectrometry studies of$[Ru^{II}[9]aneS_3(N\text{-}N)Cl]Cl/\ PF_6$ and $[Ru^{II}[12]aneS_4(N\text{-}N)]Cl_2$ or $(PF_6)_2$ have been performed. Fragmentation patterns of several ruthenium(II) compounds designed to be used for DNA molecular recognition have been studied.

The potential of Surface-Enhanced Raman Scattering in the study of the interaction of metal nanocrystals with molecular

adsorbates continued to be explored, in particular in the investigation of adsorption modes and orientation of molecules on the surfaces, with relevance in heterogeneous catalysis and nanoparticle-assembly studies. Research has been carried out in order to study the potential of this technique in nanobioanalytical processes.

A multiplex phase cycling method has been used to record MQMAS spectra with a very short phase cycling. A straightforward procedure has been developed to easily process the data. Combining this Multiplex approach and the new Soft-Pulse-Adding-Mixing (SPAM) method considerably increases the S/N ratio of the conventional MQMAS experiment. The Multiplex acquisition procedure is much simpler than the echo/ anti-echo method recently proposed.

ELECTROCERAMICS

Microwave Dielectric Materials. Ceramics $(1-x)BaTiO_3-xLa(Mg_{1/2}Ti_{1/2})O_3$ (x = 0, 0.025, 0.05, 0.075 and 0.1) have been dielectrically investigated and the phase diagram of the system determined. Apparent relaxor behaviour sets in when the $BaTiO_3$-type sequence of three phase transitions is reduced to only a diffuse transition. The system $(1-x)LMT\text{-}xLa_{2/3}TiO_3$ (LT) ($0<x<0.52$) has also been subjected to structural and dielectric characterization. Structure transformations occurred as x increased: $P21/n \rightarrow Pnma \rightarrow Imma \rightarrow I2/a \rightarrow R3^{-}c$. Permittivity has been measured as a function of temperature and LT content. The temperature coefficient of the resonant frequency passes the zero value between x=0.49 and 0.52, where the discontinuous $I2/a \rightarrow R3^{-}c$ crossover occurs. These were discussed in terms of the type of the phase transitions (continuous/discontinuous).

Ceramics based on $(1-x)LMT-xATiO_3$ solid solutions (A=Ca, Sr, Ba) have been investigated to study the effect of A-site on the structure and microwave dielectric properties. The structural changes and planar defects have been studied and the changes in microwave properties discussed. The resonant frequency temperature coefficient (Q_f) varies linearly with permittivity for LMT-CT and LMT-ST but, a discontinuity is observed for LMT-BT at $x<0.7$, the composition at which octahedral rotations are no longer present at room temperature. Zero Q_f is achieved at $x<0.5$ in all solid-solution series. The microwave dielectric quality factor (Qf) decreases with increasing x to a minimum at x=0.5 for all solid solution series, followed by an increase at x=0.7, despite the permittivity being higher for x=0.7 than for 0.5. Qf then continued to decrease as x=1 was approached, while permittivity increased.

The evolution of the crystal structure of monoclinic $La(Mg_{1/2}Ti_{1/2})O_3$ (LMT) has been studied upon mixing with Ba_2MgWO_6 (BMW) and $La_{2/3}TiO_3$ (LT), both forming solid solutions. BMW is cubic and the mixing originates one discontinuous phase transition (besides other continuous phase transitions). The influence of this transition in the electrical characteristics at microwave frequencies is under analysis. LT introduces disorder in the A and B-sites of the parent perovskite structure of LMT. The structue and electrical relation found in the LMT-LT solutions lead to the development of a new compound, $La_4Mg_3W_3O_{18}$ (LMW), with a layered superstructure.

Ferroelectric Ceramics. Core-shell structures (1-x) $PbFe_{2/3}W_{1/3}O_3$ – $xPbTiO_3$ (PFW-PT) (x = 0.32, 0.37, 0.45) have been studied. A Ti-rich core and W-rich shell characterise the core-shell structure which occur due to the existence of lead-tungstate liquid phases during the sintering step, forming a Ti-deficient shell after cooling down. The composition of the grains core and shell depends on the solid solution composition and processing conditions, namely cooling ratio after sintering and additional annealing steps. The most significant differences between the compositions of core and shell are observed for the quenched samples. Using relatively fast cooling Ti-rich cores with well defined domain patterns W-rich shells without any pattern are obtained. During slow cooling or post sintering annealing treatments diffusion of W and Ti ions takes place, inwards and outwards of the grain, respectively, which leads to the homogenization of the samples and to core-shell structure disappearance. Core-shell structure in PFW-PT system makes dielectric permittivity vs. temperature peaks wider and shifted to higher T_c, while additional annealing at temperatures below liquid-phase formation sharpen the peaks and slightly shift them to lower T_c. By controlling the cooling ratio after sintering or by performing additional annealing treatments the microstructure and dielectric properties of PFW-PT ceramics may be reproducibly changed.

Ferroelectric Fibers, Single Crystals and Films. $Pb(Zr_xTi_{1-x})O_3$ (PZT) fibers with different macroscopic properties have been obtained using acetic acid and methacrylic acid to modify a PZT precursor. In order to clarify the role of the acid, the molecular structure of the PZT precursors was investigated by GC-MS), FTIR and ^{13}C NMR. When methacrylic acid is used, long gel and ceramic fibers are obtained, because of the strongly coordinating carboxylate groups. Linear chains, like those of methacrylic acid propyl ester and methacrylic acetate, are formed in the PZT precursor sols. In addition after heat treatment the polymer decomposes quickly, so that pure perovskite may be obtained at low temperature. When acetic acid is used short fibers are obtained.

PZT single crystals have been prepared by the method of high temperature flux growth. In order to obtain large single crystals and compositions near the morphotropic phase boundary the thermal cycle and the amount of flux have been considered as variables. The electrical characterisation performed revealed singular permittivity values that need to be further investigated.

High-quality $SrBi_2Nb_2O_9$ single crystals have been grown from a melt using a high-temperature self-flux solution method and Bi_2O_3 added with B_2O_3 as a flux. A suitable thermal profile involving slow cooling rates allowed growing large and translucent SBN crystals exhibiting platelet morphology with typical size ~ 5×5 mm^2 and thickness *ca.* 400 µm. XRD reveals a dominant (001)-orientation of the major face of the platelet crystals and edges oriented parallel to the [110] directions. The dielectric properties have been evaluated along the *ab*-plane and in the *c*-axis direction. The ferro-paraelectric phase transition is observed at T_C = 440 ºC, while the maximum permittivity along the *ab*-plane (3000) is an order of magnitude larger than that along the *c*-axis direction (200). The Curie-Weiss law is obeyed in the *ab*-plane and displays a Curie constant of *ca.* 4.7×10^4 ºC. $SrBi_2Ta_2O_9$ single crystals have also been grown by the same method. The domain structure of $SrBi_2Ta_2O_9$ (SBT) single crystals has been investigated by XRD and piezoelectric force microscopy. Both ferroelectric 180º domains and ferroelastic 90º domains (twins) are revealed at room temperature. Remarkably, the coexisting domains of two types formed a

well-defined 'herringbone' structure with mostly flat 90º walls. Formation of the observed complex domain pattern is attributed to a two-stage process associated with the presence of separate ferroelastic and ferroelectric phase transitions in SBT. The Raman spectra of SBT single crystals in the range 6–1800 cm^{-1} have been studied as a function of temperature, below and above the ferroelastic–ferroelectric phase transition at *ca.* 600 K. The behaviour of the soft ferroelectric mode and of its damping coefficient near the phase transition indicates a strong coupling with an acoustic phonon. The results provided clear evidence for a crossing over from a displacive to an order–disorder component and they are discussed in terms of a complex sequence of the phase transitions in SBT.

The approach of using SBT seeds to improve the synthesis of SBT thin films by a sol-gel procedure has been studied and their effects on the thin film properties evaluated. XRD and SEM of the seeded and unseeded thin films, annealed at different temperatures, showed that the use of SBT seeds lowers the crystallization temperature of the perovskite phase and affects the thin film microstructure inducing the development of well defined and more elongated grains. The dielectric and ferroelectric properties are also improved by seeding. The present technique has the advantage of requiring smoother annealing conditions than the existing methods for producing films with comparable characteristics. A new and environment friendly method for the synthesis of SBT and SBN thin films has been developed. The reactants include bismuth- and strontium- acetates and tantalum ethoxide or niobium ethoxide as cation precursors, and ethanol and urea as solvent and complexing agent respectively. This preparation method is thus less toxic than the methods currently employed for obtaining thin films of the bismuth layered perovskites materials. The features of the films are: SBT - the remanent polarization 5.0 $\mu C/cm^2$ and coercive field 50 kV/cm; SBN - Pr=8.0 $\mu C/cm^2$ and Ec=120 kV/cm.

By using a $Ba_{0.8}Sr_{0.2}TiO_3$ (BST80/20) sol-gel seed layer of optimised thickness, the perovskite phase nucleation and growth of BST80/20 films has been restricted to the bottom interface, resulting in (h00) preferred orientation BST thin films on $Pt/Ti/SiO_2/Si$, with enhanced electric properties. The effect of sol-gel seed layers and their thickness on the structure / microstructure and electric properties of BST sol-gel derived films has been evaluated. 30 nm is the critical thickness for the seed layer. This layer acts as a 'soft template' favouring the heterogeneous nucleation, enhancing the crystallization of the perovskite phase, restricting the nucleation of the crystalline phase to the bottom layer, favouring textured growth through the (h00) direction and decreasing the defect state of the film. 400 nm thick BST films with 30 nm thick seed layer show a maximised (h00) preferred orientation growth with a grain size of 120 nm. The dielectric constant is 300 -830 at 1 kHz and 230 - 600 at 1 MHz, for films without seed layer and with 30 nm thick seed layer, respectively. The tunability of capacitance is increased from *ca.* 17% to 34% at 150kv/cm. The remanant polarization and leakage current of BST films with optimal seed layer are 1.6 $\mu C/cm^2$ with a coercive field of 45 kV/cm and 1×10^{-7} A/cm^2 up to an applied voltage of 167.5 kV/cm, respectively.

PZT thick films with 5 - 20 μm thickness deposited on flexible copper foils by electrophoretic deposition (EPD) show deteriorated properties when compared with PZT thick films deposited on platinum foils. Although the density of the sintered films and the electrical properties are improved by introducing a PbO coating on the films top, the dielectric and ferroelectric

properties of PZT thick films on Cu are still inferior to those of films deposited on Pt. Rutherford backscattering spectrometry, XRD and TEM revealed the formation of a Cu_x-Pb alloy when sintering above 950 °C, accompanied by Ti enrichment of the PZT and the formation of ZrO_2 phases. As the sintering temperature increases the concentration of the metallic phase increases and spreads throughout the film. A new Pb – Cu alloy phase has been identified. The deteriorated electrical properties of PZT thick films on Cu have been correlated with these microstructural features.

Incipient Ferroelectrics. The work on Mn doped ST ceramics continued with dielectric relaxation studies in a broad frequency range (10^2 – 10^{14} Hz). The contribution of individual off-centre Mn^{+2} ions dominates the dielectric spectra at lower concentrations $x < 0.03$ and may be described by an Arrhenius law with activation energy U=52-73 meV and relaxation time $\tau0=1\text{-}7\times10^{-14}$ s, whereas the contribution of the polar clusters, induced by the off-centred ions interacting via crystal lattice, prevails at higher Mn concentrations in the dielectric spectra and can be described by a Vogel-Fulcher relation with U=64-78 meV, $\tau0$ $4\text{-}12\times10^{-14}$ s and freezing temperature 2-10 K. Although for the SMnT ceramics x = 0.03 corresponds to the solid solubility limit it correlates well with the scheme of the dielectric behaviour of moderately doped incipient ferroelectrics, constructed from the analysis of dielectric spectra of $K_{1-x}Li_xTaO_3$ and $Sr_{1-1.5x}Bi_xTiO_3$. The dielectric relaxation is observed also at high frequencies and is detected even in the THz range. Moreover, time-domain THz and IR spectra reveal that the soft mode shifts to higher frequencies and the soft mode phonon contribution to the dielectric response decreases with increasing concentration of Mn^{+2}, being suppressed by the interaction with off-centred ions and polar clusters. The relaxor-type dielectric behaviour observed for SMnT ceramics is attributed to the off-centre hopping of Mn^{+2} ions at Sr sites of highly polarisable $SrTiO_3$ lattice.

Raman spectroscopy and *in situ* electron diffraction (ED) using a cold-stage TEM microscope have been used in the study of $SrTiO_3$:Mn ceramics to ascertain the different positions of Mn ions at A- and B-sites in ABO_3 perovskite lattice. It has been shown that: (i) additional Raman lines of $Sr_{1-x}Mn_xTiO_3$ and $SrTi_{1-y}Mn_yO_3$ ceramics suggest a formation of additional structural bonds with their own dynamics, confirming the incorporation of Mn into the Sr- and Ti- sites of the perovskite lattice, respectively; (ii) Raman R-modes and superlattice reflections in ED patterns of $Sr_{0.975}Mn_{0.025}TiO_3$ are observable at *ca.* 150 K, a temperature at least 40 K higher than that of undoped ST; (iii) improper ferroelastic phase transition temperature T_a in $SrTi_{0.95}Mn_{0.05}O_3$ is ≤80 K, at least 30 K lower than that of undoped ST; (iv) Mn incorporation at the Sr site increases T_a by means of decreasing the tolerance factor *t*, whereas the Mn incorporation at the Ti site raises *t* and decreases T_a. This work clarified the occupancy of Mn ions in the ST lattice reflected in the different vibrational modes and lattice distortions of $Sr_{1-x}Mn_xTiO_3$ and $SrTi_{1-y}Mn_yO_3$ ceramic samples. Due to the strong decrease of the structural phase transition in $SrTi_{1-y}Mn_yO_3$ this system is considered as promising material to be used as a substrate for the SQUID applications.

$Sr_{1-x}Mg_xTiO_3$, films prepared by sol-gel, with $0.10\leq x\leq0.30$ reveal homogeneous monophasic microstructures, for all compositions annealed at 750 °C. $MgTiO_3$ is observed for x=0.30 annealed at 800 – 900 °C. RBS and TEM analysis confirms the presence of Mg homogeneously distributed in SMT films with $x\leq0.30$ and annealed at 750 and 900 °C. TEM clearly reveals Mg-rich second phases for SMT films with $x\leq0.15$ annealed at 900 ºC. The solid solubility limit of Mg in ST films

annealed at 750 ºC is x≥0.30, decreasing for higher annealing temperatures (800-900 °C) to about x=0.10. The solid solubility limit of Mg in ST is higher in thin films prepared by sol gel than in Mg doped ST ceramics.

Piezoelectric Characterization. A novel technique for the measurements of electric field-induced displacements in ferroelectric materials has been presented. The method relies on a high sensitivity of the fiber-optic probe Fotonic Sensor that measures the displacement of a specially designed cantilever beam having electrical and mechanical contacts with the deforming sample. In this way, the major disadvantages of the standard Fotonic Sensor technique is avoided. The method provides relatively high sensitivity down to 4 Å, high stability 7% over 8 h, and sufficiently broad frequency range. The capabilities of the proposed measurement setup have been validated by the strain measurements on bulk PZT ceramics and thin films.

Multiferroic Ceramics and Thin Films. Multiferroic LSMO-LuMO ceramics have been sintered by solid-state reaction. The solid solubility limit is only 2% and separate grains of LSMO and LuMO form, showing ferroelectric and ferromagnetic properties. Magnetic susceptibitlity has been measured for all sintered compositions and results show that Vegard law is valid for composites.

Nanoscale Properties of Ferroelectrics. The effect of piezoelectric nonlinearity and piezoelectric hysteresis have been studied. The dependence of local piezoelectric deformation on the driving voltage has been investigated in polycrystalline PZT and Pb_{1-x} La_xTiO_3 (PLT) films by SFM. Almost linear piezoelectric behavior is observed in PLT films, while significant nonlinearity and instabilities of the piezoelectric response is found in PZT layers. These measurements have been compared with macroscopic nonlinearity studied by laser interferometry. A model of electric-field induced depinning of domain walls has been used to explain strong instabilities and nonlinear effects in PZT thin films. These polarization instabilities and nonlinearity, which are observed for areas with seemingly uniform piezoelectric contrast, indicate new capabilities of SFM to investigate ferroelectric films. The ac field dependence of piezoelectric response reveals the presence of hidden domain walls 'trapped' under the ferroelectric surface. The observation of piezoelectric instabilities may provide additional information on the characteristics of domain-wall pinning in ferroelectrics, because the corresponding critical voltages depend on the interaction of domain boundaries with lattice defects. The lateral configuration of hidden domain walls and other polarization inhomogeneities may also be revealed in this way by scanning the ferroelectric surface with different ac voltages. As compared to other techniques, such as laser-induced modulation, SFM provides much better lateral resolution.
Ferroelectric $Pb(Zr_xTi_{1-x})O_3$ thin films of different compositions (x=0.2-0.6) derived from the sol-gel diol route have been studied by PFM. A high contrast between opposite polarization states exists in PZT20/80 and PZT30/70 films due to their high (111) texture. Rhombohedral grains in PZT52/48 and PZT60/40 films exhibit much lower contrast due to the large number of domains with polarization vector directed at a small angle relative to the plane of the films. The variation of local piezoresponse signal measured by hysteresis loops versus Zr/Ti ratio has been explained based on the variation of the

dielectric constant and polarization of these films. Thus, it has been possible to explain the relatively high local piezoelectric activity observed in PZT30/70 films. The tilt of the measured hysteresis loop is roughly proportional to the value of spontaneous polarization P_s and inversely proportional to the dielectric permittivity of the film ε. The local self-polarization of PZT films changes from negative (domains terminated at the bottom electrode) to positive (oriented to the free surface of the films) with increasing Zr/Ti ratio. The drift of the oxygen vacancies caused by the thermal stress explains this behaviour. The difference between macroscopic and microscopic ferroelectric and piezoelectric parameters of sol-gel derived PZT films has been discussed.

Local piezoelectric hysteresis measurements has been performed by the piezoelectric force microscopy in PZT thin films within the single grain. A number of novel phenomena are observed with increasing dc bias voltage, including the jump of the ferroelectric domain wall to the grain boundary, the 'fingerlike' instability of domain wall, and local phase transition into ferroelectric phase.

The studies on ferroelectric relaxors were continued in order to obtain new information on the nature of the polarization state in relaxors. PLZT 9.75/65/35 ceramics have been investigated by piezoresponse force microscopy. Complex nanodomain structures exist inside the individual grains (at least near the surface) and confirm that the local symmetry of PLZT is different from the cubic macroscopic one. Random network of these domains is attributed to La-induced disorder. The stable ferroelectric state could be locally induced by the application of moderate voltages through the PFM tip. The local piezoelectric response of epitaxial PMN thin films has been studied as a function of applied dc voltage, time and pulse duration, using a piezoresponse force microscopy. Without a dc bias, no piezoelectric activity is observed on the epitaxially smooth surface of the films. Applying dc voltage pulses of sufficiently large magnitude results in the appearance of ferroelectric-like order and hysteresis. The relaxation of the induced polarization is two-stage process with the kinetics governed by the Kohlrausch-Williams-Watt-type dependence.

Bipolar cycling has been shown to strongly modify the domain patterns in PZT ceramics. A large number of ferroelastic domains accommodate high mechanical stresses arising during multiple polarization switching. Fatigue is stronger in ferroelectric grains close to the electrodes, where the concentration of defects, which pin the domain walls, is high. In polycrystalline materials, the immobilization of domains in grains adjacent to electrodes is strong enough to block polarization switching in the entire sample. The self-polarization observed in fatigued grains is switched locally and no completely frozen domains are observed (as opposite to thin film case). High-temperature annealing restores the initial domain pattern resulting in the recovery of switchable polarization. Hysteresis loops of the piezoelectric coefficient have been measured on virgin and fatigued PZT ceramics. Four parameters have been directly extracted: internal bias field, offset piezoelectric coefficient, coercive field, and remnant piezoelectric coefficient. The reduction in remanent d33 displays the decreasing switchable polarization with fatigue cycling. Coercive field and d33 offsets are linearly related. After thermal annealing, both offsets disappear, while coercive field increases and remanent d33 decreases with annealing. The microscopic entities responsible for the offsets were less stable than those for reduced switching.

A novel technique for the measuring electric field-induced displacements in ferroelectric materials has been presented. The method relies on a high sensitivity of the fiber-optic probe Fotonic Sensor that measures the displacement of a specially designed cantilever beam having electrical and mechanical contacts with the deforming sample. In this way, the major disadvantages of the standard Fotonic Sensor technique are avoided. The method provides relatively high sensitivity down to 4 Å, high stability 7% over 8 h, and sufficiently broad frequency range. The capabilities of the proposed measurement setup have been validated by the strain measurements on bulk PZT ceramics and thin films.

Structure, orientational features, and twinning of epitaxial superconductive YBa_2Cu_3Ox (YBCO) thin films and$YBCO/CeO_2$ heterostructures on (110) $NdGaO_3$ (NGO) and tilted-axes $NdGaO_3$ substrates with an inclination of normal of the substrate from the [110] axis have been investigated by XRD and AFM. The results showed large morphological influence of tilted axes substrates as revealed by AFM. $Bi_2Sr_2CaCu_2O_{8+x}$ (2212) single crystals grown by SC, TSSG and TSFZ methods have been studied. The typical concentrations of dislocations in the Bi-2212 crystals are estimated from the width of peaks. These crystals exhibit misorientation anisotropy around different crystallographic axes, which is a consequence of incommensurate modulation in this compound, shown by AFM.

MAGNETOSTRUCTURAL MODULATION OF STRONGLY CORRELATED ELECTRIC MATERIALS

Colossal Magnetoresistive Materials. A main topic of study has been the complex interplay of lattice structure, oxygen vacancy, defects and doping on the properties of CMR manganites, leading to phase segregation at different length scales: charge or orbital ordered; insulator *vs*. metallic. The prospect of application to magnetic cooling using the magnetocaloric effect has been investigated.

As far as preparation of bulk and thin film samples of: a) La-(Ca,Sr)MnO_3 and rare-earth (Er,Eu) doped b) Pr-$CaMnO_3$ system and derived with vacancies in A and B site, the following studies were performed:

i) Structural (X-ray diffraction) for phase purity, lattice parameters and their temperature dependence near structural phase transitions

ii) Magnetic measurements as a function of temperature and magnetic field. The Landau theory of phase transitions was used to provide a systematic understanding of the magnetostructural coupling. The mean field approaches to the study of magnetic interactions were also applied. Magnetocaloric properties were studied, namely the effect of RE substitution on the cooling power for near-room-temperature applications. A study of magnetic entropy in competing phase systems (Ferromagnetic and charge-order) was also performed.

iii) Electrical properties (electrical resistivity and magnetoresistance) were studied in mixed phase regions. Non-linear effects and exchange bias were detected.

iv) Hyperfine local probe using implanted radioactive isotopes at ISOLDE-CERN, with Perturbed Angular Correlation Spectroscopy and Emission Channeling was used to provide local and element selective information on doping mechanisms. The following topics were studied: lattice site and electronic characterisation of the doping elements; disorder and quenched random field effects at the Mn site, in the vicinity of the charge or orbital ordered/ferromagnetic phase instability; polaron

dynamics and percolative effects in lightly doped ferromagnetic insulator manganites; effect of charge and orbital ordering on hyperfine measurements.

Complex systems with long-range interactions or distributed characteristic parameters have been described using Tsallis non-extensive statistics.

***High Temperature Superconductors*.** Magnetic studies of oxide superconductor materials prepared by LFZ have been performed: phase studies, critical currents and their relation with structural and phase characteristics in BSCCO fibers. Studies of new MgB_2-type superconductors: preparation and HIP processing. Magnetic and electrical properties studies: superconducting fraction and critical currents. Hyperfine studies of Hg-HighTc superconductors. Role of oxygen defects in fluorinated compounds.

ADVANCED MOLECULAR AND SUPRAMOLECULAR MATERIALS

Hydrogen Bonds. The main objective of this work is the study of the hydrogen bonds and their role in the formation of supramolecular structures, from simple dimers to large molecular aggregates. The importance of hydrogen bonds in the molecular association has been assessed for a group of carbonyl containing systems, using both theoretical (*ab initio* calculations) and experimental technique, including vibrational spectroscopy, NMR, Inelastic Neutron Scattering and XRD.

***Cyclodextrins*.** The diimine ligand *N,N*'-bis(ferrocenylmethylene)ethylenediamine (FcNN) has been treated with $MoO_2Cl_2(THF)_2$ and $Mo(CO)_6$, to obtain the polynuclear complexes $FcNNMoO_2Cl_2$ (1) and $FcNNMo(CO)_4$ (2). These complexes have been encapsulated in heptakis-2,3,6-*tris*-O-methyl-beta-cyclodextrin (TRIMEB) and characterised. Results suggest that both adducts of TRIMEB with 1 and 2 have an inclusion geometry in which each ferrocenyl sub-unit penetrates deeply into the cyclodextrin cavity in axial mode, with a 2:1 host:guest stoichiometry. Complex 1 and its TRIMEB inclusion compound catalyse, with high selectivity, the liquid phase epoxidation of cyclooctene, with tert-butyl hydroperoxide (TBHP) as the oxidant. Complex 2 has electrochemical activity due to oxidation of the $Mo(CO)_4$ fragment (E_{pa} = 0.56 V) and the two ferrocenyl moieties (E_{pa} = 0.75 V and 0.85 V) that is dramatically modified by TRIMEB inclusion, with the disappearance of the $Mo(CO)_4$ oxidation.

Aiming at preparing pseudo-polyrotaxanes comprising cyclodextrins and ferrocenyl(dimethyl)silane polymers, small oligomers [dimer (3) and trimer (4)] have been encapsulated in β– and γ–cyclodextrins for a comparative study. For the β–CD·dimer, channel-packed structures form, whereas trimer (4) is too large to be threaded in β-CD. The resulting inclusion compound is, thus, less stable and amorphous. γ–CD is the cyclodextrin of choice for encapsulation of these guests, as it formed channel structures with both 3 and 4.

Cyclodextrins have been used as drug delivery and protective agents for Cp_2MoCl_2, Cp = cyclopentadienyl (5), using as hosts TRIMEB and (2-hydroxypropil)-beta-cyclodextrin (HPBCD), a modified cyclodextrin with low toxicity. Structural characterisation shows the incorporation of Cp_2MoCl_2 rather than hydrolysis products, such as $[Cp_2Mo(H_2O)X]^+$ (X = Cl, OH) or $[Cp_2Mo(H_2O)_2]^{2+}$. Results also indicate a weak host-guest interaction in the TRIMEB adduct, while encapsulation in HPβCD features a stronger interaction, involving penetration of one or both Cp ligands in the HPBCD cavity. The cytotoxicity experiments carried out on human adenocarcinoma cells indicate that cyclodextrin encapsulation enhances the antitumour activity of Cp_2MoCl_2. TRIMEB·Cp_2MoCl_2 is the most effective antiproliferative and cytotoxic agent, featuring a 60%, hardly reversible, viability decrease in adenocarcinoma and low-toxicity towards healthy cells.

Oxomolybdenum Catalysts. Dioxomolybdenum(VI) complexes $[MoO_2X_2L]$ (X = Cl, Br) have been prepared with nitrogen-ligands such as 4,4'-dialkyl-2,2'-bipyridine, $Ph_2C{=}NCH_2CH_2N{=}CPh_2$ and $PhCH{=}NCH_2CH_2N{=}CHPh$, chiral 1,4-diazabutadienes R*-N=CPh-CPh=N-R*, and chiral (1*R*,2*R*)-N,N'-dibenzylidenecyclohexane-1,2-diamine. The complexes bearing the bipyridine and ethylenediimine ligands were applied as homogeneous catalysts in the epoxidation of cyclooctene using *tert*-butyl hydroperoxide (TBHP) as the oxidant. The desired epoxide, epoxycyclooctane is the only product, and the activity of dibromo complexes is much lower that of the dichloro complexes. For the complexes with bipyridine ligands, a kinetic model has been built up for a homogeneous batch reactor, based on a simplified mechanism involving three steps: reversible coordination of TBHP to the starting Mo^{VI} complex giving a Mo^{VI} alkylperoxide; irreversible oxidation of cyclooctene to cyclooctene oxide by the species formed in the previous step, with formation of the starting complex and *tert*-butanol; reversible coordination of *tert*-butanol to the starting complex. The first mechanism step has been studied by following the kinetics of the reaction of the starting complexes with TBHP using UV/Vis spectroscopy. The results indicate an associative mechanism in which a seven-coordinate intermediate is formed. The dioxomolybdenum(VI) complexes bearing the chiral ligands have been evaluated as catalysts for the asymmetric epoxidation of *cis*- and *trans*–β-methylstyrene by TBHP. The reactions proceeded with high retention of olefin configuration and high selectivity to the epoxide, but significant enantiomeric excess has been obtained only for *cis*-β–methylstyrene. $CpMo(CO)_3Cl$ has been examined as a precatalyst for the oxidation of olefins by TBHP. Oxidative decarbonylation leads to the formation of the active dioxomolybdenum(VI) catalyst $CpMoO_2Cl$. The catalytic recyclability and the effect of temperature have been examined, and a reaction mechanism proposed. The complexes $(\eta^5\text{-}C_5R_5)Mo(CO)_3X$ (X = Me, Cl; R = H, Me) have also been studied in systems containing room-temperature ionic liquids (RTILs). The catalytic performance for cyclooctene epoxidation strongly depends on the water content of the system, catalyst solubility in the RTIL, and reaction behaviour of the RTIL. Organotin-oxomolybdate coordination polymers $[(R_3Sn)_2MoO_4]\cdot nH_2O$ (R = Me, *n*Bu, Cy, Ph, Bz) have been tested as catalysts for the epoxidation of cyclooctene at 35 ºC. The best system has been obtained using the tri-*n*-butyltin derivative as catalyst, aqueous H_2O_2 as oxidant and dichloromethane as solvent.

Novel Transition Metal Complexes. In order to study the carcinogenicity and mutagenicity mechanisms of chromium, some Cr^{V} compounds have been synthesised and characterised, particularly with peptides (Gly-Ala and Gly-Asp) and carbohydrate-type acids (gluconic, glucuronic and galacturonic acids).

The formation of supramolecular aggregates of 4-phenyl-benzaldehyde in the liquid phase via weak C–H…O hydrogen bonding interactions has investigated by XRD and molecular dynamics. Structural studies on Ru(II) complexes incorporating thiacrown ligands ([12]ane-S4, [14]ane-S_4, [16]ane-S_4), and 2,2'-bipyridine (bpy) or pyridine have been carried out using a variety of techniques. These complexes are fluxional, involving lone-pair inversion of coordinated macrocyclic sulphur donor atoms rather then a cleavage of the bpy Ru-N bond. The energetics and dynamics of invertomer formation and conversion for macrocyclic coordination complexes has been investigated. These studies reveal that the steric constraints of assembling each sulphur macrocycle and bpy ligand around the octahedral Ru(II) center lead to close intramolecular contacts, which are largely dependent on the orientation of the electron lone pairs of equatorial sulphur donor atoms and correlate with the comparative stability of the different invertomeric forms. Thus, the conformational preferences of the three macrocyles in [Ru([n]ane-S_4)(bpy)]$^{2+}$ complexes are determined by steric rather than electronic effects.

Macrocycles. Novel macrocylic architectures have been synthesised for the selective uptake of a variety anionic substrates including phthalates (polluants). The ligands are also able to encapsulate larger heavy atoms, such as Cd, Pb, Hg, and UO_2 and lanthanides, or form homodinuclear complexes with small metal centres (Co, Ni or Cu) which can be used as sensors of the organic substrates forming for cascade complexes. Aza-macrocycles having large cavities, several binding sites and incorporating rigid structural units have been designed *ab-initio* using molecular dynamics and molecular mechanics methods. The coordination behaviour of the prepared macrocycles towards a wide range of transition metal ions and organic substrates, in solution, has been evaluated. The structures of metal complexes and supramolecular associations have been determined by XRD. Among the synthetic receptors prepared is the hexaprotonated macrocycle $(H_6Me_2[30]pbz_2N_6)^{6+}$, which has been used for the molecular recognition of aliphatic and aromatic carboxylate substrates. The receptor-substrate binding behaviour of $(H_6Me_2[30]pbz_2N_6)^{6+}$ with aliphatic [$^{-}O_2C(CH_2)_nCO_2^{-}$, n = 0 to 4] and aromatic (benzoate, phthalate, isophthalate, and terephthalate) substrates has been evaluated by potentiometry and ^{1}H NMR spectroscopy. The constants and the values found for the aliphatic substrates are much lower than the aromatic ones. Molecular dynamics simulations in water have also been used to investigate the nature of the binding association between the receptor and the three aromatic dicarboxylate anions (phthalate, isophthalate, and terephthalate). The $(H_6Me_2[30]pbz_2N_6)^{6+}$ receptor encapsulates the terephthalate anion, forming an inclusion supermolecule stabilised by multiple N-H···O hydrogen bonding and π-π interactions. The molecular recognition between the receptor and the other two aromatic anions, phthalate and isophthalate, also occurs *via* N-H···O hydrogen bonds, but outside of the macrocyclic cavity. The enthalpy and the entropy associated with formation of complexes have been calculated and showed that the binding association between the receptor and these anions is favourable. The binding association calculated theoretical agrees well with experimental values.

REACTIVE CERAMIC COMPONENTS FOR PROCESS CONTROL

Materials For High-Temperature Electrochemical Applications. The composition of apatite-type silicate electrolytes ($La_{10-x}Si_{6-y}M_yO_{27-d}$, M=Fe, Al) has been modified to optimise the ionic conductivity and understand their stability and limitations. $La_2Mo_2O_9$-based electrolytes have been re-examined in what concerns the transport properties, reducibility and phase change on cooling. A numerical analysis has been proposed to re-examine the kinetics of phase transformations near equilibrium occurring in similar cases, and modified compositions $La_{1.7}Bi_{0.3}Mo_2O_9$, $La_2Mo_{1.7}W_{0.3}O_9$ and $La_2Mo_{1.95}V_{0.05}O_9$ have been used to avoid the phase transformation. Layered potassium antimonates of different structure types ($K_{0.59}M_{1-y}Sb_yO_2$ with M=Mg,Ni,Co and $K_{0.86}Co_{0.62}Sb_{0.38}O_2$) have been investigated as potassium ion conductors.

Several materials, including a new hyperstoichiometric material ($YBaCo_4O_{7+d}$) with excepcional oxygen storage ability, have been studied and assessed as potential oxygen electrodes in contact with lanthanum gallate electrolyters. Attempts have been made to tune mixed conducting properties and oxygen storage ability of other ceria-based materials (*e.g.* $Ce_{1-x-y}Pr_xGd_yO_{2-d}$). Different composite materials have been tested as fuel electrodes, such as Ni- and Cu-containing cermets, fluorite-type YSZ, CGO and TbZrO4, zircon-type $Ce_{0.8}Ca_{0.2}VO_4$, pyrochlore $Gd_{2-x}Ca_xTi_2O_{7-d}$, and perovskite $La_{1-x}Sr_xAl_{1-y-z}Fe_yMg_zO_{3-d}$. The role of mixed conductors on electrode kinetics is related to behaviour and stability under fuel conditions.

Mixed Conducting Materials For Oxygen Separation or Partial Oxidation of Hydrocarbons. Several mixed conductors have been assessed in terms of their applicability in partial oxidation of methane to attain selective conversion to H_2 and CO. These studies have been performed with mixed conducting membrane reactors, under steady-state conditions, or in fixed bed reactors with a mixed conducting ceramic catalyst. Selective conversion to CO and H_2 depends on oxygen deficiency of $La_{0.3}Sr_{0.7}Fe_{0.8}M_{0.2}O_{3-d}$ with M=Ga,Al and $SrFe_{0.7}Al_{0.3}O_{3-d}$. Redox stable additives enhance the stability and lower the thermal expansion of other mixed conductors. Though materials with high permeability (e.g. $La_{0.3}Sr_{0.7}Co_{0.8}Ga_{0.2}O_{3-d}$) yield high methane conversion, their selectivity for CO is often poor. Due to its selectivity, $SrFe_{0.7}Al_{0.3}O_{3-d}$ has been selected for improvements and up-scaling. Alumina excess acts as sintering additive to assist processing of tubular reactors. Its effects on transport properties and stability have been studied. $SrFe_{1-y-z}Al_yCr_zO_{3-d}$ and $Sr_{1-x}Ca_xFe_{1-y}Al_yO_{3-d}$ have been studied to obtain a compromise between chemical and phase stability, thermal and chemical expansion, transport and catalytic properties.

The transport numbers and mobilities of charge carriers of other mixed conductors ($La_{1-x}Sr_xM_{1-y-z}A_yMg_zO_{3-d}$ with M=Fe,Ni,Co and A=Al,Ti,Ga, $La_{1.9}M_{0.1}O_{4+d}$, M=Co,Cu) have been studied by experimental methods and simulation using a statistical thermodynamic approach. The p(O-2)-T-d diagrams have been interpreted based on a non-ideal defect chemistry model, which includes repulsion between similar charged species, other interactions between dissimilar species, and their relation to

electronic and ionic conductivities. Simulations clarified the role of isovalent redox-stable species on transport properties and provided guidelines for optimisation.

Catalysts For Selective Oxidation of Hydrocarbons. . $SrFe_{0.7}Al_{0.3}O_{3-d}$ shows the highest selectivity for partial oxidation. Selectivity depends mainly on surface state of ions and point defects in the ceramic mixed conductor. This includes ordering of oxygen vacancies and co-existence of perovskite- and brownmillerite-like domains, without reduction to metallic Fe or Fe^{2+}. Correlations have been found between the catalytic behaviour of $La_{0.3}Sr_{0.7}Fe_{0.8}M_{0.2}O_{3-d}$ with M=Ga,Al and M-O bonding energy, oxygen desorption, oxygen ionic transport, thermal expansion, and incipient transformations (*e.g.* perovskite to brownmillerite). Other catalytic studies have been performed with 2-phase composites, and materials with different structures (*e.g.* $CeVO_{4+d}$). Porous layers of $SrFe_{0.7}Al_{0.3}O_{3-d}$ were synthesised by a cellulose precursor method to enhance the catalytic performance.

Materials For Other Electrochemical Technologies.

Cellulose precursor and polymer gel templating methods have been used to prepare macroporous nanocrystalline materials (*e.g.* $La_{0.65}Sr_{0.3}CoO_{3-d}$, $NiCo_2O_4$) for potential application as electrodes in room-temperature electrochemical applications, including macroporous oxygen electrodes for alkaline aqueous solutions.

Microstructural Effects. Ceria-based electrolytes with sub-micrometric grain sizes and enhanced grain boundary behaviour have been prepared with sintering additives to lower the firing temperatures. Composite ceramic membranes, based on selected combinations of ionic conductors (CGO or LSGM) and electronic/mixed conductors (LSCF, $La_2Ni_{0.8}Cu_{0.2}O_4$) have been processed and characterised aiming at identifying the key features in the design and optimisation of mixed conductors. Though materials interaction may require improved processing to retain phase integrity, some cases also show unique features for the design of new composite materials. Mechanical activation is being used to modify the characteristics of ceramic powders and the resulting ceramics with different microstructural features, including sub-micrometric grain sizes and different domain textures. $CaTi_{0.8}Fe_{0.2}O_{3-d}$ has been found to be an interesting model material for studying the relations between transport properties and unusual microstructural features.

CERAMIC COMPOSITES AND ULTRA-HARD COATINGS FOR MECHANICAL APPLICATIONS

Diamong Coatings. CVD diamond and DLC coatings have been studied for tribological and biomedical applications. Si_3N_4 excels as a substrate for diamond coatings due to its low thermal expansion coefficient mismatch and good adherence. The effect of diamond grain size and film thickness on the tribological behaviour of CVD diamond coatings has been studied by reciprocating sliding ball-on-flat wear tests. Extremely low friction has been attained after a short running-in regime. The larger grain sized and thicker coatings present smaller compressive residual stresses and delayed delamination. New mechanical seals based on CVD diamond coated silicon nitride (Si_3N_4) rings have been proposed. Seals are designed to

prevent fluid leakage from pressurized chambers in fluid transport and to avoid lubricant outflow in moving parts. Diamond tribosystems guarantee sealing with low stationary friction coefficient for very long running times and without measurable wear.

CVD diamond has been studied as cutting tool material in machining of metal with extreme hardness of the workpiece. Silicon nitride round inserts having different edge geometry (sharp, honed and chamfered edges) have been produced by pressureless sintering to hold CVD diamond. Turning of hardmetal containing has been conducted in a numerically controlled lath. CVD diamond has also been studied to replace electroplated diamond in dentistry, thus eliminating metal contamination from the binder matrix and extending the lifetime. Si_3N_4 substrates were sintered, disk shaped, laser patterned and diamond coated by microwave plasma CVD. A performance comparison in grinding materials used in dentistry allowed better understanding of the effects of surface roughness attained by laser patterning, diamond grain size and shape.

Microwave plasma-assisted CVD has been used to grow nanocrystalline diamond (NCD) for mechanical seals, biomaterials or cutting tools for hard machining operations. The performance is due to its small grain size and surface smoothness. Optimal deposition conditions allowed production of continuous NCD films with very low roughness. Pulsed microwave discharges enabled the deposition of good-quality NCD films on ball-shaped Si_3N_4 ceramics.

Other Hard and Ultra-Hard Materials. Optimised processing yielded fully dense and nearly defect free Fe40Al/TiC composites, with improved strength and hardness. New representations have been developed to allow the selection of sealing systems and as a measure of the seal ring thickness reduction.

Colloidal Processing. Colloidal processing has been studied with emphasis on modification of oxide powders from hydrophilic to hydrophobic for low-pressure injection moulding. The method is based on chemisorption of a long-chain carboxylic acid or its salts. Surface treatment allow rheology control of alumina suspensions and increase in green density.

The rheological properties of aqueous concentrated AlN suspensions have been investigated in the presence of a sintering aid, deffloculant, binder and plasticizers, in order to screen experimental conditions for tape casting. Added binder and plasticizers do not change the shear thinning behaviour, but the binder increases viscosity and allows higher flexibility for tape casting. Dispersion of multicomponent reaction sialon precursor in non-aqueous media has been optimised with solids loading up to 60 vol%.

Other Processing Methods. Anatase nanoparticles have been prepared by hydrothermally treating amorphous titania particles peptized with different amounts of tetraethylammonium hydroxide, allowing changes in particle morphology. Doping with Fe(III) led to in situ formation of anatase nanorods. Dispersed Co_3O_4 single crystals have been prepared hydrothermally.

Rod-like α-SiAlON and β-SiAlON crystals and whiskers have been obtained by combustion synthesis. The effects of experimental parameters on phase composition, phase transformation and microstructure of Yb α-SiAlON ceramics prepared

by pressureless sintering have been studied. Low-temperature *in situ* toughened Yb α-SiAlON ceramics have been densified by spark plasma sintering (SPS), with combustion synthesised seed crystals.

Calcium phosphate powders (HAP-based and β-TCP) were prepared in the form of single phase or multiple-phase compositions, including nano-sized powders. Powders were used to produce scaffolds with tailored-pore size distributions. A new model formulation of a biocompatible glass on the system SiO_2-Al_2O_3-B_2O_3-MgO-CaO-Na_2O-F has been developed and tested *in vitro*. Interfacial interactions between the molten glass and solid metallic and ceramic substrates used in biomedicine have been studied.

Other activities have been devoted to recycling/reuse of industrial wastes and/or sub-products in ceramic or cement-based materials, including sludges from anodizing or galvanizing, paper-pulp, quarries, water and wastewater treatment. In addition to incorporation in common or technical ceramic products, new matrices have been studied, such as mortars, cement and lightweight aggregates. The activities involved rheology studies, development of new functional formulations and rehabilitation, often in straight cooperation with industrial partners. Selected fully-recycled formulations have been processed by extrusion. Stress–deformation curves, obtained by compression, are related with ceramic formulations and moisture contents. Deformation and rupture stress studies have been used as guidelines for plastic behaviour and to design extrudable formulations.

Corrosion Protection Methods. Corrosion protection studies have been performed for metallic products, with emphasis on degradation of coatings and mechanisms of corrosion and inhibition. Hybrid organosilane and sol-gel derived composite films have been studied on aluminium alloys and galvanized steel. Stable Si-O-Me chemical bonds afford good adhesion, avoid delamination and prevent contact between the metal and corrosive environments. Hybrid composite sol-gel films with oxide nanoparticles give an even higher corrosion protection. Lanthanides have been introduced to obtain additional active corrosion protection effect. Ce(III) has been found to be very effective when added as oxide nanoparticles. Environmentally-friendly compounds have been tested as potential candidates for corrosion inhibitors, to replace chromate, including 1,12-bis(1,2,4-triazolyl)dodecane for carbon steels in acidic environment, phosphates for zinc galvanised steel and triazole derivatives for aluminum. Thin silica-based films have been deposited on organic coatings by plasma polymerization, to improve mechanical properties and change the hydrophobicity of polymer surfaces. Different conditions and precursors have been used to tailor the surface without loss of protection.

AREA 3 – Chemistry and Technology of Polymeric and Lignocellulosic Materials and Biomaterials

MACROMOLECULAR MATERIALS AND LIGNOCELLULOSICS

Lignocellulosics. The silica-cellulose hybrid (SCH) materials obtained from bleached *E. globulus* bleached pulp (BP) work have been characterised on dimensional stability (expressed as relative weight, ΔW%, and volume, ΔV%, increment of material after soaking in water), thermal conductivity, mechanical strength and ignition resistance. The incorporation of silica into BP changes radically its hydrophilicity (ΔW% decreased from 235% in BP to 50-100% in hybrids and ΔV% from 60% to 5-20% respectively). CSHs show an improved bending strength (σ), similar to middle/high density fibreboards (3-5 MPa). Thermal conductivity (λ) measured in the range 40-180 ºC is slightly higher in hybrid materials (0.11-0.15 W/m.K) when compared to BP (0.08 W/m.K), but comparable to values reported for commercial insulating polymer materials. Hybrid materials show three times lower heat release rate during ignition at around 800ºC when compared to bleached kraft pulp and one thousand times lower when compared to commercial insulating polymers, *i.e.* hybrids are much less flammable materials. The acoustic tests of low density CSHs are in progress. The experience gained in preparation of silica-cellulose hybrid (SCH) materials allowed the extension of the same basic synthesis principles for the papermaking technologies. The results of this confidential research are under patent pending.

New cellulose fibre hybrids with $CaCO_3$, ZnS or ZnO nanoparticles have been synthesised and characterised. New synthesis routes of TiO_2/cellulose hybrids have been investigated, aiming to decrease fibre damage associated to $TiOSO_4/H^+$ reaction system, previously investigated. The papermaking properties of the selected hybrid materials have been evaluated. The opacity of sheets prepared with TiO_2/cellulose hybrids is highly dependent on the particle diameter. TiO_2/cellulose hybrids have been tested as reinforcing materials in PLA (polylactic acid) based composites. New SiO_2/cellulose hybrid materials have been prepared either by *in situ* synthesis of SiO_2 nanoparticles or by adsorption on fibre surfaces of previously synthesised SiO_2 nanoparticles, in the presence of polyelectrolytes. The synthesis and characterization of new cellulose/Au hybrid materials has been initiated.

The studies on controlled heterogeneous modification of cellulose fibres, including the esterification with fatty acids as acyl chlorides, have been pursued. The surface modification of cellulose fibres by reaction with bi-functional reagents such as di-anhydrides or di-isocyanates has been attempted. Studies on the chemical modification of fibres with fluorinated structures (esterification with trifluoracetic anhydride or pentafluorobenzoil chloride) have been initiated. The grafting polyethylene oxide derived structure on fibre surface, aiming to improve papermaking properties of fibres or, alternatively, render them partially thermoplastic, allowing the preparation of fibre-based composites, has been initiated.

The work on polyoxometalate (POM) catalysis in oxygen delignification using a combinatory (alteration treatment of unbleached kraft pulp) approach with laccase in a multi-stage system for the oxygen bleaching of pulp has been continued. A series of Mn-substituted polyoxotungstates, $[SiW_{11}Mn^{III}(H_2O)O_{39}]^{5-}$ and $[PW_{11}Mn^{III}(H_2O)O_{39}]^{4-}$, and $[SiW_{11}V^{V}O_{40}]^{5-}$ were applied as catalysts for the oxygen delignification of unbleached eucalypt kraft pulp with laccase of *Trametes versicolor*. Unlike to modest results obtained in the Laccase-Mediator System (LMS) at 45-60ºC (lignin oxidation and catalyst re-oxidation occurred at the same stage), a sustainable delignification with removal of about 50% of residual lignin is achieved with $[SiW_{11}Mn^{III}(H_2O)O_{39}]^{5-}$ and $[SiW_{11}V^{V}O_{40}]^{5-}$ when the kraft pulp treatment is carried out with polyoxometalate at 110 ºC (lignin oxidation stage) and with laccase at 45 ºC (catalyst re-oxidation stage) in separate stages. The use of $[PW_{11}Mn^{III}(H_2O)O_{39}]^{4-}$ in this multi-stage process is limited by the low re-oxidation rate with laccase. The best selectivity on the pulp delignification is found with polyoxoanion $[SiW_{11}Mn^{III}(H_2O)O_{39}]^{5-}$, whereas $[SiW_{11}V^{V}O_{40}]^{5-}$ is the most effective in the oxidative delignification. The influence of process factors on the POMs re-oxidation, such as the amount of laccase, oxygen pressure and temperature is discussed. POMs are stable after redox turnovers during the pulp delignification.

A series of transition-metal substituted polyoxotungstates (TMSP) have been tested as catalysts for the oxygen delignification of eucalypt kraft pulp, including Mn-substituted polyoxotungstates of Keggin-type, α-$[SiW_{(12-n)}Mn^{III}{}_n(H_2O)_nO_{(40-n)}]^{(4+n)-}$ (n=1(1) or n=2 (2)), sandwich-type α-B-$[(PW_9O_{34})_2Mn^{II}{}_{(4-n)}Mn^{III}{}_n(H_2O)_2]^{(10-n)-}$ (n = 1 (3) or n = 3 (4)), and the transition metal mono-substituted Keggin-type anions α-$[XW_{11}M(H_2O)O_{39}]^{n-}$ [M = Co^{III}, X = Si (5) and X= B (6); M = Ru^{IV}, X = Si (7), and X=P (8)]. All TMSP show relevant activity, particularly high selectivity of the residual lignin oxidation during oxygen delignification catalysed by 1-4, though the final kappa number of delignified pulps varies significantly. This is due to the removal of hexeneuronic acid residues at low pH diminishing the corresponding pulp kappa number counterpart. A comparison of the kappa number decrease and the delignification selectivity in conventional oxygen-alkaline bleaching process and TMSP-catalysed oxygen delignification shows significant advantages for the latter process. The best results, concerning the delignification degree and selectivity of delignification, are obtained with TMSP α-$[SiW_{11}Mn^{III}(H_2O)O_{39}]^{5-}$ (1), α-B-$[(PW_9O_{34})_2Mn^{II}Mn^{III}{}_3(H_2O)_2]^{7-}$ (4) and α-$[SiW_{11}Co^{III}(H_2O)O_{39}]^{5-}$ (5). The application of catalysts 6-8 is limited by their structural instability under the delignification conditions.

The effect of 4-*O*-methylglucuronoxylan (GX) on the hornification of bleached kraft and acid sulphite *Eucalyptus globulus* chemical pulps has been investigated. Almost straight-line dependence of kraft pulp hornification on the GX content is explained by the diminishing of fibrils aggregation and better accessibility of amorphous cellulose regions to water in GX enriched pulps. The higher hornification of sulphite than kraft pulp is assigned to lower GX content in the former and to the unfavourable rearrangement of cellulose molecules in crystalline and amorphous regions during acid sulphite pulping.

The contribution of different components in *E. globulus* kraft pulp to its kappa number (KN) and brightness have been investigated. Industrial unbleached *E. globulus* kraft pulp of KN 11.8 have been washed according to common mill practice (until pH 6.5-7.0 of washing filtrate) and used for multi-stage fractionation. This analytic scheme allowed evaluating the contribution to KN of each kraft pulp component, *i.e.* difficult to leave black liquor components (7%), lipophilic and

hydrophilic extractives (5%), HexA (26%) and residual lignin (62%). The residual lignin inserts the major contribution to the pulp dark colour followed by extractives of polyphenolic origin (mainly ellagic acid and non-hydrolysable tannins). Among the analysed pulp components no clear major contributor to the unbleached pulp brightness reversion has been found.

The technique of selective ^{13}C-enrichment of specific carbons has been applied for the preparation of synthetic lignin-carbohydrate complex (LCC) by feeding ^{13}C-enriched coniferin and syringin into the newly formed soft xylem isolated from plantation *Eucalyptus globulus* wood. LCC was analysed in fed xylem by ^{13}C CP/MAS NMR and, after xylem ball milling and extraction with DMSO, by a set of 1D, 2D and 3D liquid-state NMR techniques. This allowed the reliable identification of lignin structures and the detection of their linkages with polysaccharides. It is suggested that γ– and α-esters formed between β–*O*-4 structures and uronosyl moieties of heteroxylan and/or pectin compounds are predominant in LCC followed by benzyl ether type β–*O*-4 structures possessing the linkage between benzylic carbon and the C-6 atom of hexose residues.

The studies on ESI-MS application for the structural characterization of lignin have been continued. Dehydrogenation polymers have been synthesised from coniferin and fractionated by GPC. Several fractions were analysed by ESI-MS/MS and the oligomer structures have been inferred based on MS/MS spectra.

Xylo-oligosaccharides (XOS) obtained by partial acid hydrolysis of *E. globulus* xylan have been successful separated by LEX/SEC in different neutral and acidic fractions and identified by ESI-MS. Acetylated neutral XOS (Xyl_nAc_m) and acidic XOS (Xyl_nAc_mMeGlcA, and Xyl_nAc_mMeGlcAHex) have been studied by ESI-MS/MS. Isomers with acetylated and non-acetylated terminal xylose have been identified by the observation of ions due to loss of non-reducing XylAc and Xyl, respectively. Diacetylated XOS show fragments due to loss of two CH_3CO_2H, and loss of $XylAc_2$ indicating the presence of diacetylated terminal xyloses. Several possible location of acetyl groups are identified. In AXOS, the presence of the acetyl groups at the Xyl substituted with MeGlcA is confirmed by the observation of the ion $[Xyl_{res}AcMeGlcA+Na]^+$ at m/z 387. Tandem mass spectrometry is a powerful tool for the characterisation of acetylation patterns present in heteroxylans, since XOS occur in complex mixtures are very difficult to isolate.

Aiming at improving the sulphite eucalypt pulp brightness from 88-89% to 91-92% ISO the conditions of the conventional EP(O)-EP bleaching have been optimised. This yielded a final pulp brightness of *ca.* 90%. Several hydrogen peroxide catalysts/activators (TAED, CyA and PAG) and peracetic acid have been used in the second EP stage without significant improvement of the pulp brightness. A complementary reduction stage with sodium dithionite carried out after the optimised EP(O)-EP bleaching is effective in reaching *ca.* 91% ISO brightness of the final pulp. The laboratory scale developments have been expanded to the industrial scale confirming the practical feasibility of the main bleaching modifications suggested. The post-treatment of bleached pulp with ozone, chlorine dioxide and sodium hypochlorite do not allow increasing the final pulp brightness to 92% ISO. Hence, the pre-bleaching oxygen delignification stage has been proposed and the bleaching conditions in this stage optimised. The proposed O(P)- EP(O)-EP sequence allows the production of sulphite bleached pulp of *ca.* 92% ISO without decreasing the pulp strength properties when compared to conventional bleached pulp of 88% brightness.

A project on the improvement of yield and mechanical strength of acid sulfite pulp has been completed. The wood-to-pulping liquor ratio has been varied in order to estimate the effect of changes made in pulping on the pulp yield, mechanical strength properties and the pulp bleachability. The effect of chips pre-steaming and of the slow impregnation on the sulphite pulping of eucalypt wood has been studied. The increase of wood-to-pulping liquor ratio (L/W) from 3 to 5 favours the pulp yield at the same final pulp kappa number though without significant improvement of the pulps strength. This is due to the higher pH of the pulping liquor and better xylans retention. The bleachability of pulp produced with L/W 4 in conventional EP(O)-EP sequence is similar to that pulped at L/W 3 and is better than the pulp obtained at L/W 4. Thus, L/W 4 is optimal and the pulping conditions for this point have been optimised. The pre-steaming and the slow impregnation of wood chips before the pulping do not have a significant advantage over conventional high-pressure impregnation in terms of pulp yield and quality. The distribution of chips dimension is an extremely important factor influencing the strength properties of sulphite eucalypt pulp.

The investigation of different approaches or process modifications to improve the retention of polysaccharides during *Eucalyptus globulus* kraft pulping has been pursued, particularly the study of the effect of the splitting of alkali charge along the different phases of pulping. The characterization of the polysaccharides dissolved along the kraft pulping was also pursued. Particular attention was devoted to the glucans fraction dissolved in early stages of cooking. The studies aiming to demonstrate the re-precipitation of xylans at the end of the kraft cooking were pursued.

The impact of bleaching parameters (particularly in D0/E1 and D2 stages) as well as the substitution of the final D2 stage by a hydrogen peroxide stage (P) on the brightness stability of ECF bleached *Eucalyptus globulus* kraft pulp, was investigated. The contribution of low molecular weight compounds (previously existing in wood or formed during pulping and bleaching operations) to the brightness stability of bleached pulps, was preliminarily assessed.

The characterization of *Eucalyptus globulus* clones by Py-GC-MS technique, aiming at relating their composition with pulping performance, has been concluded.

The structures of the natural hybrid of *Paulownia elongata* and *Paulownia fortunei* have been studied. *Paulownia* produce versatile, dimensionally stable, consistently knot-free, marketed primarily for specialty solid wood products, oriented strand board, veneer and also for pulp to produce fine papers. It has been suggested that the main chemical composition of *Paulownia* and *Eucalyptus globulus* woods are similar, though the extractives composition is rather different. The chemical composition of the dichloromethane extract of a natural hybrid *Paulownia elongata/Paulownia fortunei* wood has been studied. The major components of the extract are sesamin, β-sitosterol, and paulownin, followed by a series of fatty acids.

The influence of galactomannans molecular weight on the gelation of whey protein/polysaccharide mixtures at neutral pH has been investigated. The differences in the molecular weight of a polysaccharide have a significant influence on the gel microstructure. Homogeneous mixtures and phase separated systems, with dispersed droplet and bicontinuous morphologies, have been observed by changing the polysaccharide/protein ratio and/or molecular weight. At 11% whey-protein, below the gelation threshold of the protein alone, the presence of the non-gelling polysaccharide induces gelation. At higher protein

concentration, the main effect of the polysaccharide is a re-enforcement of the gel. However, at the higher molecular weight and concentration of the non-gelling polymer, the protein network starts to loose elastic perfection, probably related in a complex way with the formation of bi-continuous structures with lower connectivity.

The decolourisation and detoxification of kraft effluent by *Trametes versicolor* has been studied. The effluent colour is primarily determined by lignin and its derivates, which are discharged mainly from pulping, bleaching and chemical recovery processes in the kraft pulp mill. White-rot fungi produce non-specific extracellular oxidative enzymes to initiate the degradation of lignin and polyphenolics. *Trametes versicolor* is one of the white-rot basidiomycetes that produce ligninolytic enzymes such as, laccase and manganese peroxidase. This work aims at studying the capacity of white-rot fungi *Trametes versicolor* to reduce the chemical oxygen demand and decolourise the effluent of the paper industry Portucel. The enzymatic activity of laccase and manganese peroxidase is higher than that obtained without any effluent. The maximum decolourisation, of 60%, is attained at the tenth day, and a reduction of the chemical oxygen demand > 60% is attained at the end of fermentation. This fungus exhibits a very good capacity to develop in toxic environments once its cell growth is enhanced in the presence of Portucel effluent and decolourisation and detoxification are attained.

A study on the new applications for cork industry by products has been started. Industrial cork, cork powder, 'black condensates' and 'cooking' water samples have been fractionated and characterised. Particular attention is devoted to low molecular weight compounds and suberin depolymerization products.

Other Polymer Systems and Materials. Progress has been made in the preparation of polymer-based nanocomposites following the grafting from strategy. Q-dots@THPO-Cl/PBA have been prepared by ATRP in miniemulsion. The synthesis of thioesters was developed and Q-dots@THPO-thioester/PS have been prepared by RAFT in miniemulsion.

A plasma surface treatment method to modify polymeric surfaces in order to tune adhesion of yeast cells has been implemented. Different surface characterisation methods of SiO_2 nanoparticles and of a new bioemulsifier produced by Yarrowia lypolitica have been studied. The zeta potential method, titration of double bonds and contact angles measurements, by different techniques, have been used.

Work continiued on the preparation and characterisation of SiO_2/exopolysaccharide nanocomposites. The controlled synthesis of SiO_2 particles has been optimised. The modification of SiO_2 surfaces with PGMA has also been studied. Xhantan gum static and dynamic rheological studies under different conditions (pH, temperature and ionic strength) have been carried out to assess the role of the fillers size, morphology and surface characteristics.

A study on the preparation and mechanical properties of DWCNTs/Poly(meth)acrylate(s) nanocomposites using the grafting from approach via ATRP has been started.

The predictive, analytical (non-simulative) cooperative segmental theory of molecular dynamics (CSTMD) has been applied to the description of (amorphous and semi-crystalline) polymer non-linear creep. The installation of the new Laboratory of Thermal Analyses started, including power compensation differential scanning calorimetry (DSC), simultaneous calorimetry

and thermogravimetry (DSC/TG) and dynamic mechanical thermal analysis (DMTA), in combination with other associated facilities (at the IST, Lisbon) for thermo-stimulated polarization / depolarisation (TSDC), dielectrical thermal analysis (DETA) and other melt and solid rheometry techniques.

BIOMEDICAL AND BIOMIMETIC MATERIALS

Biological, Structural and Identification FTIR, NMR and Other Studies. On the mechanisms of chromium toxicity, some new Cr(V) compounds, mainly with biologically relevant ligands, such as quinic acid and small peptides, were synthesised and characterised. A few of the new obtained compounds have been tested in in vivo with mice in order to contribute to the evaluation of the role and to follow the routes of the chromium intermediates in the so-called "chromium reduction toxicity mechanisms". The application of a wide range of citology and histology techniques to different organs has demonstrated several lesions induced by those compounds.

^{13}C CPMAS NMR studies of novel biomedical blended chitosan/soya membranes have been initiated to probe the dynamics of the chitosan amino groups and attempt to detect the type of interactions present between the components of the soya-chitosan membranes.

The stability of the Ni, Zn and Cd forms of Desulforedoxin (small model protein for bioinorganic metal centers) and a number of mutants has been determined. The Zn forms are stable while the stability of the Ni forms is, surprisingly, very sensitive to the N-terminal sequence. The structure of the Zn form of the Q14A mutant has been determined.

The 3D structure of the novel Heme Binding Protein, p22HBP, has been determined by NMR. Chemical shift mapping has localised the tetrapyrrole binding site and fluorescence quenching determined the dissociation constants (ca. nM) of the protein with hemin and protoporphyrin-IX.

Solid-state NMR and FTIR, have been used to characterise hydrogels based on vinylacrylate grafted dextrin. Peak identification of the vinyl and dextrin moieties has been carried out with the aim of quantifying the degree of grafting, polymerization and hydrolysis present in the hydrogel samples.

Solid-state NMR has been used to evaluate liver graft quality and compatibility as well as metabolism of donors and recipients. High resolution NMR of biofluids (bile, amniotic fluid and plasma) has been used to characterise Glygogen Storage Disease (GSD) in children, diabetes in selected patient groups and attempt early diagnostic of several disorders found in foetuses and during pregnancy. High field NMR (800 MHz) spectra of human bile and amniotic fluid have been recorded. In addition, work on new biomaterials for use as implants, contrast agents and/or drug carriers has been initiated.

Characterisation of beers by NMR and construction of a NMR/chemometrics model for the control of the process and materials from UNICER have been carried out.

The structure of polysaccharide gels has been studied and characterised by solid-state NMR. The derivatisation of cellulose and dextrin in order to promote the formation of hydrogels has been studied by NMR and FTIR. Models for quantitation of cross-linking degree with basis on spectroscopic properties are under investigation. Solid maltose has been used as a model for the use of an NMR method to detect and characterise weak CH…O hydrogen bonds in solids.

A variation of the DOSY methods in liquids has been developed to allow the recording of COSY 2D data as a function of diffusivity information. This adds to a family of DOSY-based experiments which significantly aid the assignment of complex mixtures.

Glass and Ceramic-Based Biomaterials. Research on SiO_2-CaO-P_2O_5-MgO, SiO_2-P_2O_5-CaO-MgO-K_2O and SiO_2-Na_2O-MgO has been carried out with the aim of studying the relationship between structure and surface reactivity, and understanding the mechanisms of adhesion of apatite layers to glasses and to glass-ceramics. A project on mineralisation studies in Ti-containing glasses has been started. Mineralisation in Si-based glasses is highly dependent on the formation of Si-OH groups at their surface when immersed in simulated plasma (SP). Ti-OH groups also form at the surface of Ti-containing materials. The behaviour of Si and Ti-containing glasses has been compared in SP solutions.

Organic-inorganic Si-based materials have been prepared by the sol-gel process using different silicon precursors and Ti and Zr as network modifiers. The chemical structure and the morphology of the hybrid gels and the resulting materials have been studied. Some of the obtained nanocomposites may be used as carriers for cell immobilisation.

Other New Biomaterials. Trametes versicolor has been used for the production of laccase and biopolymers. Five well-defined culture media have been studied to select the medium that maximizes production of EPS by T. versicolor. Biomass, reducing sugars and EPS concentrations along with the rheological behaviour of the broth, has been followed during 9 days fermentations. The yeast malt extract medium yields the highest production of EPS. Fermentation conditions with YM medium have been further investigated to optimise EPS production by T. versicolor. pH and, in particular, the initial glucose concentration, are the most important factors in EPS production and cell growth.

Concerning the laccase production the best inducers and operational strategies to maximise laccase production have been studied. In the cell growth medium, various glucose concentrations have been compared for improving laccase production. A significant stimulation of enzyme production under carbon limitation has been obtained. Copper, 2,5-xylidine, and a phenolic mixture have also been used as laccase inducers. The inducers have a cooperative effect on laccase production. Mixtures of inducers produce higher laccase activities, reaching 5500Udm^{-3}. Further productivity enhancement is obtained using the inducers and the carbon limitation strategy. It is shown that low laccase concentrations are obtained by a primary metabolism of T versicolor, and that phenolic compounds and carbon limitation induce a secondary metabolism, providing higher laccase concentrations. A mathematical model for laccase production based on a direct experimental measure of biomass, along with

substrate consumption and enzymatic activity over time has been proposed for non-homogeneous fermentations of T versicolor.

We have studied the enhanced aeration under high pressure and the baric stress on the cells the effect of pressure (0.1–1.5 MPa) and oxygen concentration on the growth and viability of Saccharomyces cerevisiae. A model taking into account cell viability has been developed and used to describe the measured data. The model reveals the opposing effects of oxygen availability and baric and oxidative stresses and successfully describes both the traditional biomass–product–substrate (X–P–S) evolution and the ratio of viable cells with time.

PROCESS DEVELOPMENT AND OPTIMISATION

Phase Behaviour and Transport Properties Relevant in Environmental Protection, Chemical Processing and New Materials Production. New high pressure data and further understanding on the crystallisation of waxes in live oils has been obtained The wax appearance temperature and the amount and composition of wax precipitation have been determined as a function of pressure in a synthetic complex system tetracosane (multiparaffinic commercial wax) and two binary systems (tetradecane/hexadecane and tetradecane/pentadecane). Measurements have been performed on a high pressure microscopy apparatus for the solid–liquid phase boundary measurement up to 100 MPa.

Experimental results have been compared with the values calculated by a predictive model. The influence of pressure on wax formation is as important as the influence of temperature and the model used provides an excellent description of the melting behaviour over a wide pressure range.

It has been shown that the model developped by us helds up to 1.0 GPa. (solid + liquid) equilibria (SLE) of {*n*-alkanes (*n*-tridecane, *n*-hexadecane, *n*-octadecane) + *n*-hexane} at very high pressures up to about 1.0 GPa was investigated in the temperature range 293 - 363K using a thermostated apparatus for the measurements of transition pressures from the liquid to the solid state in two component isothermal solutions. The polynomial based on the general solubility equation at atmospheric pressure has been satisfactorily used in the description of the pressure-temperature-composition relation of the high pressure (solid + liquid) equilibria. The main aim of this work is to predict the SLE of the mixtures using only pure components data in a wide pressure range, far above the pressure range in which cubic equations of state are normally applied. The fluid phase is described by the corrected SRK-EOS using van der Waals one fluid mixing rules.

A new model for wax formation in live oils has been proposed allowing the simultaneous prediction of fluid–fluid and solid–fluid equilibrium of light gases/heavy hydrocarbons systems under pressure. This new approach simplifies the predictive approach previously proposed making it more useful and accessible to users at the petroleum industry. The main modification with respect to the complex linear combination of Huron–Vial and Michelsen (LCVM) mixing rule that was previously used for the attractive term “a” of the equation of state (EOS). The LCVM mixing rule has been replaced by the classical quadratic van der Walls one-fluid mixing rule. The deviation that using this mixing rule introduces into the gamma–phi approach is cancelled by a new parameter ξ, introduced in the calculation of the solid-phase activity coefficient. This procedure has been

tested for a large range of multicomponent systems with the parameter ξ fitted to match the wax appearance temperature (WAT) at atmospheric pressure. From the ξ values obtained, a correlation relating the parameter ξ to the carbon number in the heavy fraction has been developed, allowing a prediction of wax formation in petroleum fluids. It is shown that this approach may be be applied to the prediction of wax formation at high pressure for live oils.

A new solid phase model has also been proposed. Aiming at identify the shortcomings of the available models for the description of the solid phase non-ideality, new solid liquid equilibrium data for multicomponent hydrocarbon systems have been measured. The Predictive UNIQUAC and Wilson models for the description of the solid phase non-ideality, previously proposed, are compared against this new data. The weaknesses displayed by these models on the predictive description of the measured phase behavior help to identify their limitations. A new UNIQUAC based model has been proposed, which overcomes the limitations of the models identified in this work.

Using previous knowledge on the phase behavior and thermophysical properties of paraffins aquired with our work in petroleum fluids, we atempted to use them as phase-change materials, in particular, composites of legnocellulosic materials with paraffins for thermal insulation and energy storage.

A correlation based on the corresponding states theory has been proposed for the description of the thermal conductivity of liquid paraffins, with the aime of developing a simple and accurate model to estimate the liquid thermal conductivity of heavy *n*-alkanes suitable for the design of efficient PCMs. Corresponding states theory has been selected, based on previous improvements for equilibrium and transport properties of pure and mixed heavy *n*-alkanes, using a second-order perturbation model on the Pitzer acentric factor. Results for the *n*-alkane series show that this new model predicts thermal conductivities in a broad temperature and pressure range with a deviation of 3%, whereas common deviations using a linear perturbation model are close to 16%.

Some commercial paraffinic waxes have been characterised and a number of composites prepared and characterised mechanically and physically. Some of these materials have also been tested for their insulation and energy storage capacities. A patent has been produced to protect some of the composites studied.

The thermal conductivity of three mixtures of nitrogen and cyclopentane have been measured using a transient hot-wire method, at 315.14 - 395.89 K and pressures up to 0.53 MPa. These mixtures are important in the evaluation of the evolution of the thermal insulation capacity of closed cell polyurethane foams considering their aging during the respective applications total life time. The increase of the nitrogen fraction in cyclopentane-nitrogen mixtures leads to higher thermal conductivities than has been predicted by ECST using only pure-component data.

The influence of the molecular architecture on the phase equilibria behavior of PEG oligomers and their mixtures based on predictions from a molecular-based equation of state, soft-SAFT EoS, is discussed. The same equation is used to describe the EG oligomer mixtures with several compounds, including carbon dioxide, benzene, methane and n-hexane. Soft-SAFT is used as a predictive tool to systematically study the influence of the chain length, polarity and hydrogen bonding formation.

An enzymatic method to measure the glucose content in solution has been adapted for measurements of oxygen content in oil in water emulsions at 310 K with an accuracy of about 1%. To study the effect of the perfluorocarbon and surfactant in the oxygen solubility, two perfluorocarbons, n-perfluorohexane and perfluorodecalin, in combination with three nonionic surfactants, Lecithin, Span 20 and Pluronic F-68 have been used. The oxygen solubility is independent of the surfactant used but depends on the perfluorocarbon employed in the emulsions studied.

The ageing mechanisms of perfluorocarbon emulsions have been investigated by image analysis. Oil-in-water emulsions of two perfluorocarbons, n-perfluorohexane and perfluorodecalin, were prepared with three emulsifiers, Lecithin, Span 20 and Pluronic F-68. The effect of the temperature and the replacement of water by an aqueous phase consisting of a microbial culture medium were also studied. The stability of perfluorocarbon in water emulsions depends on all the parameters investigated and that two ageing mechanisms, coalescence and molecular diffusion, may take place.

The solubilities of oxygen in 1Br-perfluoro-n-octane, 1H-perfluoro-n-octane, and 1H, 8H-perfluoro-n-octane have been measured at 288 - 313 K and pressures close to atmospheric. The experimental data allow the calculation of derived properties as enthalpy and entropy of solution, helping to elucidate the solvation of oxygen in these fluorinated compounds. The study of the solubility of oxygen in linear fluorinated molecules presenting different degrees of substitution on the terminal CF_3 groups indicates the existence of strong interactions between the oxygen and those groups.

Vapor pressures have been measured in at 288 – 333 K with an apparatus based on the static method. Liquid densities have been measured with a vibrating tube densimeter at 288 -313 K. Experimental data was fitted to appropriate equations and correlated with the soft-SAFT equation of state. The boiling points, enthalpies of vaporization and thermal expansion coefficients, derived from vapor pressure and density data, are also reported.

The solubility of the hexafluorobenzene in water and aqueous salt solutions have been measured, in the range 280 to 340 K at atmospheric pressure, using a liquid-liquid extraction method. The method has been validated by comparing the measured-solubility of ethylbenzene in water with the literature data. The estimated uncertainty in the experimental data is less than 6%. The effect of the salt concentration and the cation/anion effect on the solubility have also been established. Thermodynamic functions have been obtained from the temperature dependence of the solubility data.

Studies involving the vapor–liquid interfacial tensions of some selected associating and non-associating pure components (water, ethanol, n-butane, n-pentane, n-hexane, n-heptane) using the gradient theory have been combined with the Cubic-Plus-Association equation of state (CPA EOS) are presented and discussed. The good description of equilibrium properties such as vapor pressure and liquid and vapor phase densities is shown in the full range of the vapor–liquid saturation line.

Heteropoly acids (HPA) with the Keggin structure-type are active catalysts for the homogeneous liquid phase dehydration of xylose to furfural. The catalytic results depend on the reaction temperature, type of solvent and HPA composition. Kinetic studies reveal that the rate of xylose consumption exhibits a first order dependence with respect to the initial xylose concentration and a non-linear dependence on initial HPA concentration. The most promising systems studied are tungsten-containing HPAs, which are comparable with H_2SO_4 in terms of furfural yield. Further studies have been carried out in heterogeneous phase to facilitate product separation, catalyst recovery and recycling. A series of inorganic composites

comprising $H_3PW_{12}O_{40}$ supported on mesoporous silicas have been prepared and characterised, exhibiting higher activity than non-supported $H_3PW_{12}O_{40}$, comparable to H_2SO_4 in terms of furfural yield. The catalytic performance depends on the interplay of several factors, such as the preparation method, type of support, PW loading, and reaction conditions.

On the hydrodynamic behaviour of process-relevant liquid-liquid dispersions, a new non-invasive ultra-sound technique has been developed to accurately measure dispersed phase hold-up fractions. Extensive experimental validation data has already been obtained and used in current theoretical simulations of the dispersions' behaviour.

SECTION 3

ACTIVITY PLAN 2006

Follows the Activity Plan for 2006 for the same areas and 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

INORGANIC MULTIFUNCTIONAL MATERIALS AND ORGANIC-INORGANIC HYBRIDS

New Microporous Materials. Work on microporous silicates containing lanthanide (La) centres will continue. This work will encompass new stoichiometric La materials and transition-metal heteropolyhedra silicates doped with La^{3+}. In this context, a particularly exciting possibility is the preparation of chiral framework phosphors. The materials will be prepared as powders and also in the form of films and membranes.

The synthesis of aluminophosphates, IST-1 and IST-2 will be compreensively studied. These are the first examples of a dual templating role of water and methylamine in generating microporous structures. The structure of microporous aluminophosphate AlPO-40 will be revisited using 2D and 3D solid-state J-coupling mediated NMR experiments. We will also report on aluminum methylphosphonate materials. The characterization of Y zeolites dealuminated by solid-state reaction with ammonium hexafluorosilicate will be finished.

Recently, considerable effort was devoted to systematise all the structural (unit cell dimensions and symmetry, coordination sequences, framework density, accessible volumes, etc) and energetic information calculated for the uninodal and binodal hypothetical frameworks derived from tiling theory. The main goal for 2006 is to report all the information for trinodal structures (*i.e.*, structures containing three crystallographically independent tetrahedral sites) derived from simple tilings. This is a challenging task because of the considerable number of structures derived from computational methods (>1000).

Mesoporous Materials. Work will continue on the functionalisation of ordered mesoporous silicas with multidentate ligands and their application as supports for the heterogenisation of metal complexes with interesting catalytic or photophysical properties. Oxomolybdenum(VI) complexes and CH_3ReO_3 will be immobilised in the MCM-41 functionalised with a pyrazolylpyridine ligand. Model complexes containing the ligand ethyl[3-(2-pyridyl)-1-pyrazolyl]acetate will also be prepared. The supported materials and the model complexes will be tested as catalysts in the liquid-phase epoxidation of olefins. Where appropriate, the stability of the supported materials will be studied by recycling the solid catalysts several times.

A comparative study of Al-MCM-41 materials prepared at room temperature with different aluminium sources and by some hydrothermal methods will be reported.

Layered Materials. Work will continue on the intercalation of metallo-organic complexes with interesting photofunctional or catalytic properties into layered double hydroxides (LDHs). For example, oxomolybdenum and oxotungsten complexes of 3,4-dihydroxybenzoic acid, D-(-)quinic acid and benzilic acid, with the general formula $[MO_2L_2]^{n-}$, will be incorporated into Mg-Al and Zn-Al LDHs by ion-exchange reactions with precursor materials in nitrate or chloride form. Other complexes to be

studied include anionic tetrakis(β–diketone) lanthanide derivatives and ruthenium cyanocomplexes $[Ru(CN)_x(L)]^{m-}$. All materials will be characterized by a battery of physical techniques, including powder X-ray diffraction, nitrogen adsorption, thermal analysis (TGA, DSC), MAS NMR, FTIR, Raman and photoluminescence spectroscopies. Selected materials will be tested as catalysts for the oxidation of organic compounds, including asymmetric oxidations in the case of chiral guests. The results will direct further synthetic work, with the aim of producing truly heterogeneous catalysts that can be recycled without loss of activity or selectivity.

The work on photoluminescent layered AV-22 silicates will be terminated. Particular attention will be given to the thermal decomposition into tunnel structures. The photoluminescence properties of AV-22 doped with Ce^{3+} will also be evaluated.

Nanostructured Materials. The development of novel biomarkers based on several types of quantum dots will be continued and processes to associate the nanocomposites to magnetic nanoparticles are starting. New nanocomposites based on magnetic nanoparticles and biopolymers will be prepared and investigated as MRI agents.

The synthesis of semiconducting nanophases at metal oxide surfaces (*e.g.* TiO_2) will be investigated using our single-source method. Photocatalytic experiments for the degradation of organic substrates will be performed, to evaluate the use of materials based on such powders as alternative catalysts.

Polyoxometalates. Organic-inorganic hybrid materials in which polyoxometalates (POMs) are used as the inorganic components, will be prepared. The synthesis of new organic-inorganic hybrid coordination compounds containing lanthanides, POMs and an organic ligand will be investigated. The effect of the organic ligand and POMs on the luminescent properties will be investigated. 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 and polyoxometalate-anion-pillared layered double hydroxides.

New synthetic procedures (including hydrothermal and mechanochemical methods) will be used, in order to prepare compounds with good nonlinear optical properties. Compounds to be used in the development of modified electrodes functionalised with heteropolytungstates will be studied. Silica supported transition metal-substituted POM will be prepared and evaluated as oxidative heterogeneous catalysts. POM will be the Keggin and sandwich anions, so far used in catalytic studies in solution.

NovelLluminescent Systems. Novel molecular or supramolecular lanthanide materials and systems supported in a nanosized material will be prepared. Research will be continued on the synthesis of photoactive lanthanide complexes with aromatic ambidentate ligands, aiming at obtaining multidimensional coordination compounds. The possibility of using ligands to sensitise the lanthanide emission will be investigated. The incorporation of lanthanide compounds into nanosized SiO_2 and other substrates will be explored.

New full-colour phosphors based on a dense systems isostructural with $Na_2Ysi_3O_9$ will be reported (terminating work in progress).

Crystal Engineering of Organic-Inorganic Hybrids. The hydrothermal synthesis of novel crystalline hybrid frameworks will continue. N-(phosphonomethyl)iminodiacetic acid (H_4pmida) will be employed as the basic primary building unit in the construction, along with V^{4+} centres, of the centrosymmetric $[V_2O_2(pmida)_2]^{4-}$ building block. This Secondary Building Unit (SBU) will be self-assembled with transition metal centres and appropriate organic spacers, such as pyrazine, to construct novel multi-dimensional frameworks.

Previous studies with $[V_2O_2(pmida)_2]^{4-}$ and transition metal cations showed that these units effectively impose distances which prevent magnetic interactions between the centres. This structural feature will be employed to spatially separate lanthanide centres in hybrid multi-dimensional frameworks, thus leading to luminescent materials.

H_4pmida will also be self-assembled with Fe^{2+}, Cu^{2+} and non-paramagnetic centres such as Ge^{4+}. A novel SBU containing Ge^{4+} is expected to be isolated, which will ultimately allow its detailed study by solid-state NMR.

Organic-Inorganic Hybrids Lacking Activating Centers. The emission red-shift as the excitation wavelength increases (common to amorphous semiconductors, *e.g.* amorphous hydrogenated silicon, a-Si:H) will be studied. Some recombination models previously reported for a-Si:H will be applied to organic/inorganic hybrids allowing the estimation of the energy gap and the density of localized states within the gap. A new model of thermal relaxation within localized states based on the extended multiple-trapping framework will be derived. To understand better the physical mechanisms of white-light emission in organic/inorganic hybrids lacking activating centers, two new model compounds will be synthesized and characterised. The photoluminescence features, physical nature of their mechanisms, and the interactions between the hybrid-host emitting centres will be investigated.

New Hybrid Materials. With the goal of improving the luminescence and chemical stability under UV irradiation, lanthanide complexes will be incorporated into organic-inorganic hybrids, such as di-ureasils. The hybrid host contribution to the decrease of the non-radiative paths accessible to the lanthanide ions will be studied. The contribution of the hybrid host emitting centers to the energy transfer mechanisms (host-to-complex ligands and host-to-lanthanide ions) will also be studied. The chemical stability of the lanthanide complexes under UV exposure will be investigated in the UVA, UVB and UVC regions. The photoluminescence features in steady state and time resolved modes of the exposed samples will be measured and compared with those of non-exposed hybrids. Preliminary results concerning the incorporation of $[Ln(btfa)_3(4,4'\text{-bpy})(EtOH)]$ (Ln = Eu, Gd; 4,4'-bpy = 4,4'-bipyridine; btfa = 4,4,4-trifluoro-l-phenyl- 1,3-butanedione) complexes into di-ureasil hybrids show a quantum-yield enhancement (50±5%), relatively to the Eu^{3+} complex, suggesting an effective hybrid host-metal ion interaction and an active energy transfer channel between the hybrid host and the Eu^{3+} complex. The Eu^{3+}-based di-ureasils seem to be photostable under UV-A (360 nm) excitation for several hours, whereas under UV-B (315 nm)

excitation photodegradation occurs in the same time interval, demonstrating the potential of these hybrid materials to be used in UV-A photostable light emitting devices and UV-B dosimeters.

Integrated Optical Devices. Development of organic-inorganic di-ureasils modified by zirconium n-propoxide (ZPO) stabilized with metacrilic acid to produce cost effective integrated optics (IO) devices. The utilization of the proposed organic/inorganic hybrids in the development of such IO functionalities will be studied through the architectural structure of the waveguides, in order to obtain the required functionalities. Two key components for the implementation of these functionalities are the narrow-band optical filter, used as de-multiplexers, to access the desirable wavelengths in a Dense wavelength division multiplexing (DWDM) (a fiber-optic transmission technique that employs light wavelengths to transmit data parallel-by-bit or serial-by-character) system and the low-finesse optical cavity for the realization of optical clock recovery. The feasibility of using organic/inorganic hybrids in substrates for the production of IO architectures will be evaluated based on waveguide numerical simulation methods (beam propagation method and/or frequency domain time domain). The relevant properties for optical, electrical or thermal actuation, nonlinear refractive index, thermo-optic and electro-optic coefficients, will be compared with those typical of subtract materials presently used, such as silica, silicon, lithium niobate, polymers, glass or III-IV semiconductors. The final devices will be implemented in an optical network test bed, for final performance evaluation.

C60 Phase Transitions Under High-Pressure. The search for new C60 phases above 100 kBar and the characterisation of their corresponding pressure-temperature phase diagrams will be carried out. In this sense, new *in situ* diffraction experiments at the ESRF will be scheduled. The study by computational methods of the new high-pressure phases of C60 (carbon clathrates) will begin.

Development of Spectroscopic Techniques. 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 SERS as a tool for trace detection. 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.

Mass spectrometry studies with Ru(II) polinuclear systems complexes. Identification and fragmentation patterns of new synthesised binuclear Ruthenium(II)-polypyridylic complexes will be continued using advanced methods, mainly ESI-MS. The interaction with tetramer duplexes will also be tested.

ELECTROCERAMICS

Microwave Ceramic Dielectrics. The structure and dielectric behavior of (1-x)La($Mg_{1/2}Ti_{1/2}$)O_3 – xBa ($Mg_{1/2}W_{1/2}$)O_3 and (1-x)$CaTiO_3$ – x$LaAlO_3$ microwave ceramics, namely the sequence of structure transformations and the microwave dielectric characteristics of the ceramics will be evaluated as a function of their composition. Lead-free ceramics based on sodium-

bismuth titanate, (1-x)($Na_{1/2}Bi_{1/2}$)TiO_3 - x$La(Mg_{1/2}Ti_{1/2})O_3$ [(1-x)NBT-xLMT] ($0 \leq x \leq 1$), will be prepared by the conventional mixed oxide method and Pechini route, and their crystal structure and dielectric properties studied. The compositional evolution of structure and dielectric characteristics of the ceramics obtained will be discussed in respect to size, charge and polarizability of the cations involved. Regarding these ceramics, measurements of dielectric characteristics at radio and microwave frequencies will be performed for the LMT based compositions with different substitutions. For the assessment of the major contributing mechanisms we shall continue to use far infrared spectroscopy. The study of the effect of sintering atmosphere will help to understand the role of certain defects in the dielectric properties.

Some of the previous work, namely on the $Ba_{2-2x}Sr_{2x}MgWO_6$ solid solutions, will be concluded. In the case of the LMW compound, its electrical characterisation will be performed. The crystal analysis already completed in this material, with very interesting ordering of La (A-site) vacancies along one of the crystallographic axis due to the ordering of the B-site cations, leads to the development of other related materials, where the B-site ordering with different chemical features induces a different superstructure. The crystal and electrical characterisation of these compounds will be performed.

Ferroelectric Ceramics. The microstructural design of PZT ceramics in order to obtain dense materials with large grain size will be pursued, namely with the use of the already obtained single crystals as seeds for grain growth. The theoretical estimation of the spontaneous polarization in randomly oriented and textured BLP ceramics by considering two configurations of domain wall switching (DWS), 180º DWS and both 180º and 90º DWSs will be attempted. The results will be applied to SBT ceramics with different degrees of texture for comparing the predicted polarization values with those measured from hysteresis loops.

Ferroelectric Fibres, Single Crystals and Films. The PZT single crystals obtained by the method of high temperature flux growth will be further characterized in terms of structure, composition and electrical properties. Scanning Force Microscopy will be used to study the local properties under an applied electrical field. The investigation of the SBT and SBN single-crystals domain structure will be carried out by XRD and piezoelectric force microscopy.

In order to understand the role of the seeds in the improvement of SBT thin-film properties, the effects of SBT seeds with respect to the diffusion of SBT thin films components into the bottom Pt electrode will be studied. Rutherford Backscattering and Particle Induced X-Ray Emission (PIXE) will be used to access the Bi, Ta, Sr, O (film components) profiles as well as that of bottom electrode and/or buffer layer elements (Pt and Ti) in seeded and unseeded SBT films. The current work involving a doping approach for improving the ferroelectric properties of the SBT and SBN thin films will be continued. The effects of dopants on the ferroelectric properties of the films (remanent polarization, coercive field, leakage current and fatigue endurance) and the temperature dependence of dielectric behaviour will be investigated. High temperature piezoelectrics studies will be initiated and concentrated on developing new piezoelectric materials capable of working at high temperatures.

Previous work on PZT thick films on Cu foils showed that for films processed above 900 ºC the foil oxidation is not the critical aspect contributing to the deterioration of the final electrical properties. The formation of a CuxPb alloy and the consequent partitioning of lead and Ti play a dominant role in the degradation of the final properties of these films. Thus, it is important to process PZT on Cu at low temperatures. We will attempt to decrease the sintering temperature of these films including studying the role of the powder particle size and different sintering aids. Preparation and characterisation of thick films of $BaNd_2Ti_5O_{14}$-based high Q dielectrics by electrophoretic deposition for microwave communications will be initiated.

Incipient Ferroelectrics. The work will continue along the following lines: (i) effect of the sintering atmosphere on the dielectric response of Mn doped ST ceramics; (ii) structural and dielectric characterisation of Cr doped ST ceramics; (iii) electrical studies on La, Mg and Mn doped ST ceramics with special emphasis on the evaluation of pyroelectric currents and its relation with the dielectric spectroscopy at rf frequencies; (iv) sintering and dielectric studies on the effect of non stoichiometry on the incipient ferroelectric behaviour of ST and (v) studies on the stress effects on the crystallization, texture and dielectric response of doped and undoped ST thin films.

Nanoscale Properties of Ferroelectrics. The study of the local properties of ferroelectric thin films, single crystals and ceramics will be continued. It is planned to investigate in detail the local properties under varying electric field. The formation of the nanoscale domains with a size approaching that of the SFM tip will be attempted The investigations will be extended to lead-free materials. Multilayer actuators will be investigated at the nanoscale, aiming of improving their electromechanical performance. Nanolithography programme will be developed to write polarization patterns on the surface of ferroelectrics.

Studies on ferroelectric relaxors including new materials (Pb-free relaxors) will be continued, in order to understand the nature of ferroelectric disorder at the nanoscale. Pure PMN and PLZT that should freeze at low temperatures will be investigated using a new AFM setup. A novel measurement technique (local hysteresis mapping) will be introduced.

This work will continue with the studies at the nanoscale level of the ferroelectric fatigue behaviour of PZT thin films prepared by sol-gel on Si platinized substrates. Properties will be studied, using SPM tip in contact mode, acting as a mobile top electrode local ferroelectric. The nanodomain imaging will be performed in ferroelectric films with different seeds types and contents. The results will be correlated with the macroscopic measurements, in order to understand the mechanisms behind ferroelectric fatigue of Pt/PZT/Pt capacitors and the role of seeds on this behaviour.

***Multifunctional Ceramic Films and Composites*.** LSMO-LuMO, $BiFeO_3$-$BaTiO_3$ and novel manganite compositions will be investigated. Novel composite thick film materials ($BiFeO_3$-PZT) will be fabricated.

Bioferroeelctricity and Polarization-Induced Self-Assembly. This new direction will be devoted to the understanding of ferroelectricity in biological object-like cell membranes and polarization patterning in them.

Nanoscale Materials. A new line of work will be initiate comprising the preparation and characterisation of nano functional materials: (a) Mesoporous functional materials. Development of new organic-inorganic mesoporous hybrid materials by self-assembly. Study of the nanodisperse porosity. Preparation of mesoporous hybrids containing at least one Si-C bond as thin films for low dielectric constant insulators. Preparation of perovskite based mesoporous fims and determination of their electrical properties. The films will be characterised and their dielectric properties evaluated. (b) Nanostructured functional materials. Development of multifunctional perovskite type nanostructures by hydrothermal methods. Study of the relation size – structure – properties.

MAGNETOSTRUCTURAL MODULATION OF STRONGLY CORRELATED ELECTRIC MATERIALS

Colossal Magnetoresistive Materials. Work will start on thin film preparation with the new RF sputering deposition system for oxide thin films, structural (X-ray), magnetic and electrical properties; Studies will be carried out using implanted radioactive isotopes at ISOLDE-CERN. Other studies include: magnetocaloric materials and magnetostructural effects; CMR materials as spin polarizing electrodes for polymer LEDs; electronic phase segregated systems: competition and interface effects; multiferroic manganite materials: $REMnO_3$ (RE=Er,Lu hexagonal ferroelectric) and composites $LaSrMnO_3$-$REMnO_3$ and $BaTiO_3$-$LaBaMnO_3$ manganites.

Theoretical Approaches to Magnetic Materials Using Generalized Thermodynamics. Non-extensive statistics will be applied to manganites and other complex magnetic systems. Magneto-elastic coupling effects. Monte-Carlo simulations of magnetic systems.

High Temperature Superconductors. Physical characterisation studies of oxide superconductor materials prepared by LFZ. Studies in new MgB_2-type superconductors: preparation and processing. Microstructure. Magnetic and electrical properties studies.

ADVANCED MOLECULAR AND SUPRAMOLECULAR MATERIALS

Hydrogen Bonds. The role of hydrogen bonds in the structure and properties of molecular and supramolecular materials will be accessed for a range of organic, inorganic and hybrid systems, using theoretical (*ab initio* calculations) and experimental techniques (vibrational spectroscopy, NMR, inelastic Neutron Scattering and XRD). Particular attention will be devoted to the importance of this kind of interaction in materials of the so-called ‘class I’ (in which the molecular unities are bound through non-covalent interactions). Properties related with the hydration of crystalline samples (pseudo-polymorphism) and energy transfer in hybrid luminescent materials will be addressed during this year.

Cyclodextrins Studies on cyclodextrins (CDs) as second sphere ligands for metal complexes will continue. $CpM(CO)_nCl$ (M = Mo, Fe; n = 2, 3) will be encapsulated in native and permethylated beta-CD (β-CD and TRIMEB, respectively). The resulting inclusion compounds will be characterised by a range of techniques, particularly powder XRD. An effort will be made to refine the structure of the complexes using these data. The metal complexes also act as precursors for catalysts, by loss of the metal-bound CO groups. Thus, the catalytic properties of the metal complexes and their CD adducts will be investigated and related to the CO-releasing ability.

Inclusion compounds comprising europium(III) diketonates will be prepared. In particular, $Eu(NTA)_3{\cdot}2H_2O$ (NTA = 1-(2-naphthoyl)-3,3,3-trifluoroacetonate) will be encapsulated in octakis(2,3,6-tris-O-methyl)-gamma-cyclodextrin (TRIMEG). The results will be compared with those obtained for the corresponding native gamma-CD adduct. CD encapsulation may increase the efficiency of the Eu(III) sensitisation. Water molecules in $Eu(NTA)_3{\cdot}2H_2O$ can be replaced by ligands, giving 7-, 8- or even 9-coordinate complexes and contributing to the reduction of non-radiative relaxation of the Eu(III). The adduct $Eu(NTA)_3{\cdot}$bipy will be prepared by reaction with 2,2'-bipyridine and included in β-CD. Photoluminescence spectroscopy of the free and encapsulated complex will allow a comparative study of the complexes luminescent properties. The enhancement of the Eu^{3+} sensitised process as a result of encapsulation of the $Eu(NTA)_3{\cdot}$bipy molecules will be investigated.

Oxomolybdenum Catalysts. A series of ionic dioxomolybdenum(VI) complexes of general formula $[MoO_2Cl(L)]Y$ (Y = Cl, BF_4^-) containing tridentate nitrogen ligands (L) such as 1,4,7-triazacyclononane (tacn), 1,1,1-tris(methylaminomethyl)ethane (Me_3-tame) and N,N',N''-tribenzyl-1,1,1-tris(methylaminomethyl)ethane (Bn_3Me_3-tame) will be prepared and examined as catalysts for the epoxidation of cyclooctene, cyclododecene, 1-octene, *trans*-2-octene, (*R*)-(+)-limonene and α–pinene, using *tert*-butyl hydroperoxide (TBHP) as the oxidant. Tricarbonyl complexes $LM(CO)_3$ (M = Mo, Cr) bearing tridentate ligands (L) will also be prepared and examined as catalyst precursors for epoxidation. 4-coordinate (tetrahedral) and 6-coordinate (octahedral) dioxomolybdenum(VI) complexes such as $MoO_2(OSiPh_3)_2$ and $MoO_2(OSiPh_3)_2$bipy (bipy = 2,2'-bipyridine) will be prepared and used in the catalytic epoxidation of olefins.

Metal Complexes of Bio-Inorganic Interest. Designed compounds displaying DNA-specific binding characteristics and reduced toxicity (for DNA molecular recognition), with transition metal and polypyridyl and/or thioether will continue to be synthesised and characterised.

With the objective of studying the mechanisms of of chromium Cr^V complexed with peptides, carbohydrates and carbohydrate-type acids, which have proved to be important intermediates in the intracellular-Cr(VI) reduction, will continue to be explored, mainly in what concerns their solution behaviour in physiological conditions.

Novel Transition Metal Complexes. The electronic properties of bimetallic ruthenium complexes incorporating tetrathia-macrocycles will be investigated by DFT methods. The binding association constants between proteins and porphyrin ligands will be carried through the molecular mechanics free energy calculations using explicit or implicit water solvent models.

Macrocycles. Particular attention will be devoted to the molecular design of novel macrocyclic receptors for the selective binding of metal ions and/or organic substrates. The anionic binding between the receptors and the organics substrates will be evaluated in solution via ^{1}H NMR and potentiometry with determination of binding constants. Free-energy calculation and analysis of the intermolecular binding interactions involved in the molecular recognition process will be performed. The results of these molecular modelling studies will be used in the design of new more efficient receptors, in terms of selectivity and binding ability for organic substrates. The novel macrocyclic architectures containing chiral centres will be anchored on silica gel supports to afford chiral stationary phases for HPLC. Theoretical studies will be carried out in order to understand the enantioselective resolution of analytes.

Molecular modelling studies on biscalix[4]diquinone ionophores will be carried out, in order to understand the reported selectivity of these ligands towards metal ions in solution.

REACTIVE CERAMIC COMPONENTS FOR PROCESS CONTROL

Materials For High-Temperature Electrochemical Applications. Activities will include studies of solid electrolytes, electrodes and other related materials for fuel cells, vapour electrolysers and electrochemical pumps. The mechanisms of ionic conduction on oxide materials are being examined with an emphasis on differences between fluorite-type materials, apatites and protonic conductors. Microstructural effects will be evaluated, including two phase materials, the role of grain boundaries and different approaches to minimize their blocking effects. For silicate-based apatites the degradation and limitations of operation under fuel conditions will be studied, and the use protecting layers will be attempted.

The transport properties of cerate-based materials are being re-examined, taking into account the co-existence of protonic and oxygen ion conducting and the onset of minor electronic contributions. New materials are being considered as potential electrodes for solid electrolyte cells, with emphasis on novel concepts of electrodes with high oxygen storage ability ($YBa(Co,Fe)_4O_7$, $Ce(Pr,Zr)O_{2-d}$), and design of alternative electrode materials with two co-existing redox pairs ($Sr_{1-x}Ce_xMn_{1-y}Al_yO_{3-d}$). The role of mixed conduction on anode materials, including nanostructured components, will be addressed. Glass-ceramic seals are being developed to adjust operation temperatures and to optimise inertness.

Mixed Conducting Materials and Catalysts For Oxygen Separation or Partial Oxidation of Hydrocarbons.

Previous work on perovskite materials based on $LaFeO_3$, $LaCoO_3$, $SrFeO_{3-d}$, $LaAlO_3$, $LaGaO_3$, with different acceptors or mixed valency additives in A and B sites, and K_2NiF_4-type nickelates, will continue. The work is designed to improve the overall performance in terms of transport properties, redox behaviour, chemical and thermomechanical stability, phase transformations, and electrocatalytic performance in electrochemical applications. These studies will rely on a wide range of experimental techniques and also on atomistic simulations designed to attain a better understanding of observed limitations and to establish criteria to introduce further improvements. Other studies will be based on perovskite and related materials with A:B off-stoichiometry (e.g $La_{1-x}Ni_{0.5}Ti_{0.5}O_{3-d}$), addition of donor species (e.g. $La_{1-x-y}Ce_xSr_yFeO_{3-d}$, $La_{1-x}Sr_xFe_{1-y}Nb_yO_{3-d}$), and materials with co-existence of two different mixed valency species (e.g $CeNbO_{4+d}$) to identify less common features.

Microstructural Effects. In addition to composites containing one ionic conductor ($La_{1-x}Sr_xGa_{1-y}Mg_yO_{3-d}$ or $Ce_{0.8}Gd_{0.2}O_{2-d}$) and one electronic or mixed conductor (e.g. $La_2Ni_{0.8}Cu_{0.2}O_{4+d}$, $La_{1-x}Sr_xFe_{1-y8}Co_yO_{3-d}$ or $La_{1-x}Sr_xMnO_{3-\delta}$), new composites (e.g. $La_{0.5}Sr_{0.5}FeO_3$–$SrAl_2O_4$, $La_{0.5}Sr_{0.5}FeO_3$-$Sr_{1-x}Fe(Al)O_3$, and $SrFeO_{3-d}$-$SrAl_2O_4$) are being designed to suppress common limitations of mixed conductors. The studies encompass materials with improved thermomechanical behaviour (thermal shock, toughness, hardness) and stability under reducing conditions, without affecting the permeability and electrocatalytic activity. Different powder preparation (*e.g.* glycine-nirate and spray pyrolysis) and ceramic processing methods (*e.g.* isostatic

pressing, extrusion) will be used to modify the microstructural characteristics of these composite membranes. The research will identify the key features for the design and optimisation of materials performance. Materials interaction, from modest compositional changes (addition of sintering additives) to formation of new phases with major changes in composition, will be studied to identify optimised processing conditions for phase preservation, and for the development of entirely new concepts of composites, including compositional gradients at grain size level.

Materials For Other Electrochemical Technologies. Several ceramic materials with relatively high electrical conductivity are being studied as potential electrodes for aqueous electrochemical applications. The emphasis will be on rare-earth cobaltite perovskites (*e.g.* $Ln_{1-y}A_yCoO_{3-d}$ with Ln= Pr, Nd, Sm and A= Sr, Ca, Ba, $La_{1-x-y}Sr_xBi_yCoO_{3-d}$, $SrCo_{1-y}M_yO_{3-d}$ with M=Fe, Ni, Ti, Cu) and nickelates. The work will include a re-examination of stability as a function of pH and potential, and the electrocatalytic activity for oxygen evolution. We will attempt to establish correlations between the electrode performance and the transport properties and defect chemistry of those ceramic electrodes, including the oxygen stoichiometry, and A:B cation off-stoichiometry. A possible application of these electrodes is for the electrochemical reduction of metals, including reduction of metal oxides in strongly alkaline conditions and reduction in alkaline baths containing salts and a variety of complexing agents such as sodium pyrophosphate, sodium tartrate and Na2-EDTA. The efficiency of these processes will be studied and we will attempt to optimise the texture of metallic deposits and to obtain nanostructured deposits.

CERAMIC COMPOSITES AND ULTRA-HARD COATINGS FOR MECHANICAL APPLICATIONS

Diamong Coatings. Nanocrystalline diamond (NCD) films are being deposited on silicon nitride ceramics and developed to enhance their tribological behaviour. NCD films open new opportunities for selected tribological applications such as unlubricated and boundary lubricated tribosystems. These features are needed for machine elements (seals, valves, bearings) and metalworking processes (wire-drawing, extrusion, deep-drawing) requiring low friction and high wear resistance. Smoothness, high hardness and a self-lubricant effect resulting from the presence of sp^2 carbon in the tribocontact will be crucial for a superior behaviour. Very smooth NCD tools may excel PCD and CVD diamond tools in machining of abrasive materials like hardmetal, MMC's and CFRC's. The absence of large columnar grains may be an advantage.

Prospective biomedical applications (*e.g.* joint implants) will be envisaged for NCD coated Si_3N_4, combining bioactive Si_3N_4-bioglass composites with an ultra-hard NCD film. Biotribological systems will be characterised using ball-on-flat wear testing with physiological fluids.

CVD is also being used to obtain diamond coating (*ca.* 10 μm) on Si_3N_4 tools. The grain size of the direct coatings varies in the range of nano- (<100nm) to poly-crystalline (2-10 μm) diamond, affecting the wear mechanisms. Cutting materials are tested in turning of hard metal and investigation will continue on graphite and ceramic materials machining. Turning tests will be assessed by real-time acquisition and subsequent evaluation techniques, tip wear measurement, SEM and AFM, analysis of

the film residual stresses by Raman spectroscopy, evolution of the cutting edge roughness and evaluation of surface finishing quality by profilometry.

Self-mated CVD diamond tribological systems present potential for applications requiring low friction and high wear resistance. Reciprocating sliding ball-on-flat wear tests will be conducted in air, at room temperature, with or without lubrication, at variable normal load, involving CVD diamond coated Si_3N_4 parts, ranging from nano- to microcrystalline coatings. Friction and wear results together with structure and surface characterisation will lead to the measurement of tribological coefficients, friction and wear mechanisms.

Other Hard and Ultra-Hard Materials. New hardmetal grades with sub- to nano-metric grain size with superior erosive wear resistance are being developed. A current goal is to extend the lifetime under severe conditions, as expected for equipments in oil and natural gas extraction, chemical and pharmaceutical industries. New hardmetal compositions will be developed from sub-micrometric (0.7 0.8 micron), ultrafine (0.1-0.6 micron) and nanopowders (< 100nm) with reduced amounts of binder phase (16 wt%), to attain suitable mechanical properties (hardness, fracture toughness) and improved wear resistance. These characteristics will be extensively evaluated by suitable testing. Component prototypes will be produced for in-service tests in industrial conditions.

Processing Methods. Work on processing of advanced ceramic and glass-ceramic materials by colloidal shaping will be pursued. Aqueous media will be preferentially used in these studies and non-aqueous media will be only be used for multicomponent systems incompatible with water. New direct shaping methodologies are under development, which will enable the consolidation of large and homogeneous ceramic parts. Such systems are also expected to perform well in rapid manufacturing (ink-jet printing). Recycled industrial wastes and by-products will be used to prepare new added-value materials, including the improvement of existing products or development of products with new functionalities.

New glass and glass-ceramic compositions will be developed for different applications (structural, optical, biomedicine, electronics). Macroporous ceramic bodies will be tailored with porous microstructures for bone-ingrowth in biomedical applications. Mechanical and *in vitro* and *in vivo* characterization will be carried out.

Corrosion Protection Methods Work on corrosion protection will be based on passive protection and active self-healing. Research will focus on environmental-friendly active protection systems based on the intelligent release systems incorporated in polymer coatings. Polyelectrolyte shells and meso-/nano-porous particles impregnated with corrosion inhibitors will be used to produce controllable nano-containers. Nanoscaled reservoirs may help in achieving uniform distribution and controllable release of inhibiting species. This approach will allow the introduction of the different inhibitors in the polymer matrixes without negative effects on the stability of the coating and without the deactivation of inhibitor. Polyelectrolyte shells may allow the controllable release of the inhibitor triggered by corrosion-induced change of pH.

AREA 3 – Chemistry and Technology of Polymeric and Lignocellulosic Materials and Biomaterials

MACROMOLECULAR MATERIALS AND LIGNOCELLULOSICS

Lignocellulosics. The study of the paper surface modification using the sol-gel process will continue and concentrate mainly on the optimization of sol-gel formulations, development of application methods for the deposition of these formulations on the paper surface and paper surface analyses (image analyses, contact angles, surface energy, etc.). The printability tests will be carried out on selected coated papers.

The work on polyoxometalate (POM) catalysis in oxygen delignification, using laccase as biocatalyst for the POMs re-oxidation, will continue. Special attention will be paid to the possibility of continuous pulp delignification with POM and POM re-oxidation by laccase in a separate stage without direct contact with pulp.

The work on ESI-MS application for the structural characterization of lignin will continue an the study on specific structural features of hardwood hemicelluloses employing ESI-MS/MS and MALDI-TOF/TOF will be started.

The study of the chemical composition of macromolecular components of the natural hybrid of *Paulownia elongata* and *Paulownia fortunei* will be pursued. The evaluation of papermaking potential of this new fast growing wood will be completed, as well as the study on decolourisation and detoxification of kraft effluent by *Trametes versicolor*.

Two projects on the use of by-products from the acid sulphite pulping of *E. globulus* wood will begin: on the production of acetic acid and furfural from condensate of pulping liquor evaporation, and on the analysis of industrial circuits and utilization of lignosulphonates for binders and polymers.

The study of the conditions and factors determining the precipitation of xylans on the surface of fibres at the last stages of the kraft pulping will be pursued. Also, the effect the addition of anthraquinone in the overall polysaccharides retention during kraft pulping will be investigated and optimised.

Within the scope of the new cellulose/nanoparticles hybrid materials, studies will be aimed at understanding the nature of the interaction between nanoparticles and cellulose and the effect of the nature and morphology of nanoparticles on the properties of the hybrids and derived composite materials.

The thorough characterisation of cork industry by-products will be pursued. Suberin samples will be isolated from cork and the corresponding hydroxyacids used in the preparation of new polyesters.

Studies on the heterogeneous chemical modification of cellulose fibres will continue.

Two studies will be started: on the use of chitosan as source of coatings and blends with other natural polymers (particularly cellulose and starch); and on hyperbranched polymers based on the reversible Diels-Alder coupling of furans with maleimides.

Applied research on novel adhesives based on renewable resources and on the exploitation of olive stones as a source of novel polymeric and composite materials will be developped.

Other Polymer Systems and Materials. Work on the preparation of polymer-based nanocomposites via living polymerisation mechanisms (RAFT and ATRP) in miniemulsion will proceed. Attempts will be made to prepare block copolymers and to functionalise end groups for use as bio labels. The photoluminescent properties of these materials will be investigated.
Regarding the polysaccharide nanocomposites project, other polysaccharides will be tested, namely chitosan. The preparation of thin films from such composites will receive much attention.

Work will be initiated on the preparation of CNTs/polymer materials and the study of their mechanical performance, and on the preparation of CNTs/conducting polymers.
Methodologies for plasma surface modification of PP and PET fibres will be investigated in order to coat them with natural polysaccharides, namely chitosan for the development textiles for biomedical applications.
The influence of polymer ageing on the properties of cements will be investigated
The new Laboratory of Thermal Analyses will be used in an integrated way to probe various materials structures within very wide ranges of temperature and time scales (from $< 10^{-4}$ to $> 10^{5}$ Hz), in support of on-going theoretical developments on materials dynamics.

BIOMEDICAL AND BIOMIMETIC MATERIALS

Biological, Structural and Identification FTIR, NMR and Other Studies. New Cr(V) compounds began to be tested in *in vivo* with mice in order to contribute to the evaluation of the role of chromium intermediates in the so-called 'chromium reduction toxicity mechanisms'. The application of a wide range of citology and histology techniques to different organs has demonstrated several lesions induced by those compounds. Toxic effects of transition metal compounds, derived from industry, are known to interfere with male fertility. 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 with occupational aims.
NMR relaxation studies and HRMAS based 2D experiments of novel biomedical blended chitosan/soya membranes will be continued to identify the protein-polysaccharide interactions that stabilise the membranes.
The solution structures of the Zn and ^{113}Cd forms of a number of Desulforedoxin (small model protein for inorganic metal centers) mutants will be carried out to probe the interactions that stabilise inorganic metal centres in proteins. The determination of the structure of the Ni and Zn/Ni forms of Desulforedoxin will be initiated.
The 3D structure of the tetrapyrrole bound form of the novel Heme Binding Protein p22HBP will be determined by NMR. The interaction of p22HBP with functional porphyrins will be studied using NMR and capillary electrophoresis in order to assess p22HBP as a heme biosensor.

The characterisation of biofluids and biological tissues (e.g. liver, heart, breast) by NMR will be pursued, involving the construction of compositional database of those systems and determining the profiles characteristic of healthy subjects and the occurrence of several disorders under study. The study of biomaterials will be carried out with emphasis on the use of NMR and MR Imaging, aiming at characterising new biomaterials based on magnetic nanoparticles or biopolymer/inorganic scaffolds and measuring their *ex-vivo* and *in-vivo* performance.

Work on the applications of the NMR methods for the control of the brewing process and the development of new products will continue. Databases of metabolic profiles will be constructed for different types of yeasts and different growth conditions.

Solid-state NMR will be used to determine the degree of polymerisation and hydrolysis present in hydrogels based on vinylacrylate grafted dextrin (highly biocompatible gels). The degree of polymerisation of these gels is directly linked with the porosity of the gel and the hydrolysis kinetics will determine the release characteristics. In addition, a new project on the structure of cork and the role of depolymerising enzymes on the biopolymeric nanostructure will be initiated.

Glass and Ceramic-Based Biomaterials. Structural studies and evaluation of surface reactivity will proceed with Si-based systems SiO_2-CaO-P_2O_5-MgO and SiO_2-Na_2O-MgO and Ti-based system TiO_2-CaO-P_2O_5. Si-based glasses suggesting *in vitro* bioactive behaviour will be added to different polymeric matrices (PMMA, PE, SEVA, PHB) to obtain novel composites for biomedical applications. The new formulations will be studied in terms of chemical and mechanical behaviour. Synthesis of hybrid organic-inorganic materials will also proceed with the aim of producing materials with improved mechanical properties, namely elastic modulus, and enhanced surface area for cell immobilisation applications.

Work will also be directed to (i) refine the process of crystallization and ageing of calcium phosphates in SBF solutions with CO_2 / HCO_3^- buffer, (ii) study the influence of several trace metals in the crystallization and ageing of hydroxyapatite phases, (iii) refine the process of analyses of solid calcium phosphates by infrared spectroscopy, and (iv) apply speciation models to solutions from plants, soils and sediments from Aljustrel, Braçal and Penedono mines.

Other New Biomaterials. We will continue to carry our studies concerning cultures in multiphase reactors. Measurements of kLa's will be carried to evaluate the enhanced aeration obtained in the presence of a second organic phase of oil or prefluorocarbon compounds. Enhanced production of activity of enzimes in the presence of perfluorocarbons will be studied for tirosinase.

Given the interest on extractive fermentations using ionic liquids, studies on the toxicity of these fluids towards different families of microorganisms will also be carried.

The production of bioemulsifierss using *Yarrowia lipolytica* and the characterization of both the emulsifier and the cell surface will continue, and studies of bioremediation will be carried out using *Yarrowia lipolytica*.

Phase Behaviour and Transport Properties Relevant in Environmental Protection, Chemical Processing and New Materials Production. Composites of paraffins, vegetal waxes, fatty acids and others with legnocellulosic compounds will be developped and their mechanical and thermal properties characterised. The phase behaviour of fatty acids and vegetal waxes will be addressed with the purpose of developing models for the design of new phase change materials. The phase diagrams will be measured by DSC and their crystal habit by XRD measurements. Their thermal conductivities will be measured using a modified hot wire method.

The new class of ionic liquid compounds is expected to originate a number of environmentally-friendly replacements for various organic solvents currently used in synthesis and separations. We have been dealing with measurements of their thermophysical properties and phase equilibria. We will be focusing on their thermal conductivities aiming at their use as fluids for heat transfer, surface and interfacial tensions, and liquid-liquid equilibrium for liquid-liquid extraction. Gas solubilities on ionic liquids help assessing the potential of some of them to be used for gas separation and preparation of liquid membranes. Studies of their impact on the environment and biota are usually based on the octanol-water partition coefficient. For gaining insight on this subject Pow measurements will be carried.

Modelling of the measured phase equilibria will be attempted using the COSMO-RS approach. The thermophysical properties will be addressed using QSPR methods.

As far as phase changes of petroleum-based materials are concerned, we will work on the development of a deposition loop to study wax deposition from real crudes. We will also study the crystal habit of the waxes extracted from Brazilian crudes and the nature of these waxes.

Concerning our ongoing project on hydrocarbon dispersion on water measurements on the PAHs water solubility and the influence of the salt concentration on it will continue to be studied. The interfacial tensions between water and hydrocarbons will be measured and modelled using the gradient theory coupled to the CPA equation of state. The production and analysis of biodiesels will start.

The study of the permeation properties of biodegradable polymers will be addressed. Experimentally, a permeation cell and a quartz crystal microbalance will be used to obtain permeability and solubility. Special attention will be devoted to the permeation of aromas and flavors. Intelligent packaging, based on poly(lactic acid) (PLA), will be developed aiming at its application in the food packaging industry.

Phase equilibria of PLA-industrially relevant solvents systems will be measured. From the theoretical point of view, models for correlating and predicting phase behavior polymer-solvent systems will be studied. In particular, the soft-SAFT model will be developed and used to model associating polymer-solvent systems.

Surfactant systems will also be the focus of our research. Emulsions are important systems in drug delivery. Drug delivery using PLA is also currently being addressed. The interfacial properties of surfactant-polymer systems, with special attention to ionic liquids and PLA, will also be studied.

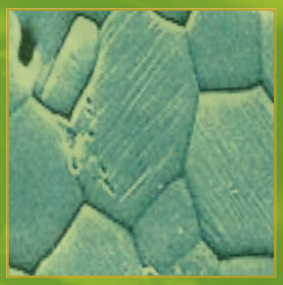
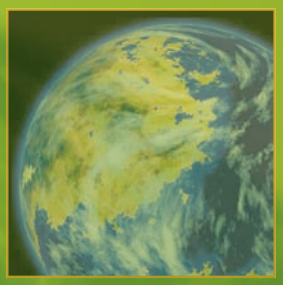
The use of solid-acid catalysts for the transformation of carbohydrates into furfural may be a promising alternative to the use of mineral acids. Niobium-containing materials such as hydrated niobium oxide and niobium phosphate exhibit unique activity, selectivity and stability for many different catalytic reactions. In the last few years, there has been interest in the synthesis of porous niobium silicates, as the presence of niobium in molecular sieves has the potential of generating catalysts with size/shape selective properties and acidic or redox characteristics. In the future, we will focus on the use of novel microporous materials, such as AM-11, and mesoporous Nb-MCM-41 niobium silicates as catalysts. The synthesis of AM-11 in the NH_4-form will be attempted for the first time. These catalytic results will be compared to those obtained with conventional zeolites.

The recently developed ultra-sound dispersed phase hold-up measurement technique will continue to be explored to gather data that will be used in the modelling and simulation of dispersions' hydrodynamic behaviour including drop breakage and coalescence.

SECTION 4

SCIENTIFIC PRODUCTION

Until 31st December 2005

COMPLEXOS DE RUTÉNIO COM LIGANDOS POLITIOÉTERES E/OU POLIPIRI-DÍLICOS: SÍNTESE, CARACTERIZAÇÃO E INTERACÇÃO COM O ADN

MADUREIRA JPL
SUPERVISOR(S): SANTOS TM AND FÉLIX VMS
UNIVERSITY OF AVEIRO, PORTUGAL
2005

COMPOSTOS DE INCLUSÃO EM CICLODEXTRINA-BETA A HUMIDADES RELATIVAS DEFINIDAS

CUNHA-SILVA L
SUPERVISOR(S): TEIXEIRA-DIAS JJC
UNIVERSITY OF AVEIRO, PORTUGAL
2005

CRISTALIZAÇÃO DE BIOMATERIAIS VITROCERÂMICOS E MINERALIZAÇÃO EM MEIO FISIOLÓGICO SIMULADO

QUEIROZ CMGA
SUPERVISOR(S): FRADE J AND FERNANDES MHV
UNIVERSITY OF AVEIRO, PORTUGAL
2005

DESENVOLVIMENTO E CARACTERIZAÇÃO DE MATERIAIS CERÂMICOS A PARTIR CINZAS DE CARVÃO MINERAL

KNIESS CT
SUPERVISOR(S): KUNHEM NC; RIELLA HG AND SEGADÃES AM
UNIVERSITY OF AVEIRO, PORTUGAL
2005

DESENVOLVIMENTO E CARACTERIZAÇÃO DE PIGMENTOS CERÂMICOS OBTIDOS A PARTIR DE LAMAS GALVÂNICAS

GOMES V
SUPERVISOR(S): LABRINCHA JA
UNIVERSITY OF AVEIRO, PORTUGAL
2005

ELÉCTRODOS DE FILME FINO DE MERCÚRIO PARA ANÁLISE DE METAIS VESTIGIAIS – ESTUDOS DE OPTIMIZAÇÃO, CARACTERIZAÇÃO E MODIFICAÇÃO QUÍMICA COM POLI-4-(ESTIRENOSULFONATO) DE SÓDIO

MONTERROSO SCC
SUPERVISOR(S): CARAPUÇA HMCS
UNIVERSITY OF AVEIRO, PORTUGAL
2005

INCLUSÃO EM MOLÉCULAS ANFIFÍLICAS EM CICLODEXTRINA-BETA, EM SOLUÇÕES AQUOSAS DE ELECTRÓLITOS FORTES

LIMA S
SUPERVISOR(S): TEIXEIRA-DIAS JJC
UNIVERSITY OF AVEIRO, PORTUGAL
2005

INFLUÊNCIA DA ESTRUTURA QUÍMICA DOS COMPONENTES DA MADEIRA NO SEU DESEMPENHO NOS PROCESSOS DE PRODUÇÃO DE PASTAS CELULÓSICAS. ESTUDO COMPARATIVO ENTRE EUCALYPTUS GLOBULUS E OUTRAS FOLHOSAS

PINTO PC
SUPERVISOR(S): PASCOAL NETO C AND EVTUGUIN DV
UNIVERSITY OF AVEIRO, PORTUGAL
2005

INFLUÊNCIA DAS PENTOSANAS NAS PROPRIEDADES FUNCIONAIS DO GLÚTEN E DO AMIDO DE TRIGO

SANTOS DM
SUPERVISOR(S): LOPES DA SILVA J AND GIL AM
UNIVERSITY OF AVEIRO, PORTUGAL
2005

NOVOS COMPOSTOS COM POLIOXOMETALATOS: SÍNTESE, CARACTERIZAÇÃO E APLICAÇÃO EM CATÁLISE OXIDATIVA

VIEIRA ICMSS
SUPERVISOR(S): CAVALEIRO AMV AND NEVES MGPMS
UNIVERSITY OF AVEIRO, PORTUGAL
2005

NOVOS SISTEMAS LUMINESCENTES CONTENDO LANTANÍDEOS: DE COMPOSTOS DE COORDENAÇÃO A NANOMATERIAIS

SOARES-SANTOS PC
SUPERVISOR(S): NOGUEIRA HIS AND TRINDADE T
UNIVERSITY OF AVEIRO, PORTUGAL
2005

PREPARATION AND PROPERTIES OF FERROELECTRIC SINGLE CRYSTALS AND CERAMICS OF BISMUTH LAYERED PEROVSKITES

AMORIN H
SUPERVISOR(S): COSTA MEV AND KHOLKIN AL
UNIVERSITY OF AVEIRO, PORTUGAL
2005

SOLUBILIDADE DE GASES EM SUBSTITUTOS DO SANGUE

DIAS A
SUPERVISOR(S): MARRUCHO IM AND COUTINHO JAP
UNIVERSITY OF AVEIRO, PORTUGAL
2005

STRONTIUM TITANATE BASED CERAMICS FOR TUNABLE DEVICE APPLICATIONS

TKACH O
SUPERVISOR(S): VILARINHO PM AND KHOLKIN AL
UNIVERSITY OF AVEIRO, PORTUGAL
2005

COMPOSIÇÃO DA MADEIRA DE CLONES DE EUCALYPTUS GLOBULUS E SUA INFLUÊNCIA NA APTIDÃO AO COZIMENTO KRAFT

XAVIER AFA
PASCOAL NETO C AND SILVESTRE AJD
UNIVERSITY OF AVEIRO, PORTUGAL
2005

COMPÓSITOS POLÍMERO/FOSFATO DE CÁLCIO PARA APLICAÇÕES BIOMÉDICAS

JÚNIOR JVA
COSTA MEV AND ALMEIDA MML
UNIVERSITY OF AVEIRO, PORTUGAL
2005

EFEITO DO TEOR E ESTRUTURA DE XILANA DE PASTAS BRANCAS DE E. GLOBULUS NA SUA TENDENCIA PARA A HORNIFICAÇÃO

REBUZZI DOS SANTOS F
EVTYUGIN DV
UNIVERSITY OF AVEIRO, PORTUGAL
2005

ESTUDO DA INFUÊNCIA DOS COMPONENTES DA PASTA KRAFT DE E. GLOBULUS SOBRE O ÍNDICE KAPPA E BRANCURA

DIAS DO NASCIMENTO V
EVTYUGIN DV AND SILVESTRE AJD
UNIVERSITY OF AVEIRO, PORTUGAL
2005

ESTUDO DE ARGAMASSAS DE REABILITAÇÃO DE EDIFÍCIOS ANTIGOS

MARQUES SM
LABRINCHA JA AND FERREIRA VM
UNIVERSITY OF AVEIRO, PORTUGAL
2005

ESTUDO DE MANGANITES MODIFICADAS COM IÕES DE TERRA RARA

AMARAL JCS
AMARAL V
UNIVERSITY OF AVEIRO, PORTUGAL
2005

ESTUDO DO DESEMPENHO TRIBOLÓGICO DE VEDANTES MECÂNICOS RECOBERTOS POR DIAMANTE CVD

TOMÉ MM
SILVA RF AND CARRAPICHANO JM
UNIVERSITY OF AVEIRO, PORTUGAL
2005

FERRAMENTAS DE CORTE DE DIAMANTE CVD PARA TORNEAMENTO A SECO DE LIGAS AL-SI

CASTRO GC
SILVA RF AND SACRAMENTO JG
UNIVERSITY OF AVEIRO, PORTUGAL
2005

LIGAÇÕES INTER- E INTRA-MOLECULARES: PERSPECTIVAS DE ENSINO / APREN-DIZAGEM NPOS DIFERENTES GRAUS DE ENSINO

OSÓRIO MMJP
SANTOS TM AND ROCHA GMO
UNIVERSITY OF AVEIRO, PORTUGAL
2005

MOLDES PARA FUNDIÇÃO EM AREIA: SUBSTITUIÇÃO DO LIGANTE POR CIMENTO DE FOSFATO DE MAGNÉSIO

MORELLI GL
MORELLI MR AND SEGADÃES AM
FEDERAL UNIVERSITY OF SÃO CARLOS, BRASIL
2005

POLIOXOTUNGSTATOS: APLICAÇÕES NA OXIDAÇÃO CATALÍTICA DE COMPOSTOS ORGÂNICOS COM H_2O_2

SOUSA ACEMG
CAVALEIRO AMV AND NEVES MGPMS
UNIVERSITY OF AVEIRO, PORTUGAL
2005

PREPARAÇÃO DE VIDROS E VITRO-CERÂMICOS DO SISTEMA SIO_2-AL_2O_3-B_2O_3-CAO-MGO-LI_2O-$(K,NA)_2O$-F PARA POTENCIAIS APLICAÇÕES EM MEDICINA DENTÁRIA

PINA S
FERREIRA JMF
UNIVERSITY OF AVEIRO, PORTUGAL
2005

REMOÇÃO DE CONTAMINANTES EM MEIO AQUOSO POR LEITOS DE ARGILA EXPANDIDA

ALBUQUERQUE C
LABRINCHA JA
UNIVERSITY OF AVEIRO, PORTUGAL
2005

REOLOGIA DE ARGAMASSAS

PAIVA MH
LABRINCHA JA
UNIVERSITY OF AVEIRO, PORTUGAL
2005

SÍNTESE E CARACTERIZAÇÃO DE MATERIAIS HÍBRIDOS DO TIPO ORGÂNICO/INORGANICO COM BASE EM FIBRAS DE CELULOSE

SEQUEIRA SM
EVTYUGIN DV AND PORTUGAL BRANCO MI
UNIVERSITY OF AVEIRO, PORTUGAL
2005

VALORIZAÇÃO DE RESÍDUOS DA FILEIRA FLORESTAL PARA A PRODUÇÃO DE AGREGADOS LEVES

PINTO S
LABRINCHA JA AND FERREIRA VM
UNIVERSITY OF AVEIRO, PORTUGAL
2005

VALORIZAÇÃO DE RESÍDUOS NA PRODUÇÃO DE ESMALTES CERÂMICOS

GASSMAN T
LABRINCHA JA AND FERREIRA VM
UNIVERSITY OF AVEIRO, PORTUGAL
2005

AN INVESTIGATION OF WEAK CH CENTER DOT CENTER DOT CENTER DOT O HYDROGEN BONDS IN MALTOSE ANOMERS BY A COMBINATION OF CALCULATION AND EXPERIMENTAL SOLID-STATE NMR SPECTROSCOPY

YATES JR; PHAM TN; PICKARD CJ; MAURI F; AMADO AM; GIL AM AND BROWN SP
2005, J AM CHEM SOC 127 (29), 10216-10220

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SILVA NJO; CARLOS LD AND AMARAL VS
2005, PHYS REV LETT 94 (3), Art. No. 039707

HYBRID MESOPOROUS MCM-41 TYPE MATERIAL CONTAINING 1,4-DIAZOBUTADIENE CHELATE LIGAND IN THE WALLS

FERREIRA P; NUNES CD; VAZ PD; BION N; BRANDÃO P AND ROCHA J
2005, PROG SOLID STATE CH 33 (2-4), 106-113

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$[CO(H_2O)_6]\{[CO(C_4H_4N_2)(H_2O)_2][V_2O_2(PMIDA)_2]\}\cdot 2H_2O$ [H_4PMIDA IS N-(PHOSPHONOMETHYL)IMINODIACETIC ACID]: THE FIRST TWO-DIMENSIONAL HYBRID FRAMEWORK CONTAINING $[V_2O_2(PMIDA)_2]^{4-}$ BUILDING BLOCKS
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12º ENCONTRO DA SOCIEDADE PORTUGUESA DE MATERIAIS
UNIVERSITY OF AVEIRO, PORTUGAL
2005

13TH INTERNATIONAL SYMPOSIUM ON WOOD, FIBRE AND PULPING CHEMISTRY (ISWFPC)
AUCKLAND, NEW ZEALAND
16-19 MAY 2005

14ª CONFERÊNCIA NACIONAL DE FÍSICA, SOCIEDADE PORTUGUESA DE FÍSICA
PORTO, PORTUGAL
01- 03 DEC 2005

2ND ULCOS SEMINAR – ULTRA LOW CO2 STEELMAKING
UNIVERSITY OF AVEIRO, PORTUGAL
21-22 SEPT 2005

4º ENCONTRO DA DIVISÃO DE ENSINO E DIVULGAÇÃO DA QUÍMICA - SOCIEDADE PORTUGUESA DE QUÍMICA
PAVILHÃO DO CONHECIMENTO E FIL – LISBOA, PORTUGAL
27-28 OCT 2005

6º ENCONTRO DE QUÍMICA INORGÂNICA - SOCIEDADE PORTUGUESA DE QUÍMICA
FUNCHAL, MADEIRA, PORTUGAL
31 MAR - 02 APR 2005

ADVANCES IN PHYSICAL SCIENCES: MEETING IN HONOUR OF PROFESSOR A. L. LEITE VIDEIRA
UNIVERSITY OF AVEIRO, PORTUGAL
05 - 07 SEPT 2005

CIAT- CO2 NAS INDÚSTRIAS DE ALTA TEMPERATURA
UNIVERSITY OF AVEIRO, PORTUGAL
16 DEC 2005

ELECTROCERÁMICA - VII REUNION NACIONAL
TERUEL, ESPANHA
JUN - JUL 2005

EUROBIC8
UNIVERSITY OF AVEIRO, PORTUGAL
02 - 06 JUL 2006

EUROCORR-2005
LISBOA, PORTUGAL
04 - 07 SEPT 2005

4TH CHINA INTERNATIONAL CONFERENCE ON HIGH-PERFORMANCE CERAMICS - CICC-4
CHENGDU, CHINA
23-26 OCT 2005

II JORNADAS CICECO
UNIVERSITY OF AVEIRO
07 - 08 JAN 2005

III INTERNATIONAL MATERIALS SYMPOSIUM: MATERIAIS2005
UNIVERSITY OF AVEIRO, PORTUGAL
21-23 MAR 2005

INTERNATIONAL CONFERENCE ON POROUS CERAMIC MATERIALS
BRUGGE, BELGIUM
20-21 OCT 2005

INTERNATIONAL SYMPOSIUM MICRO- AND NANOSCALE DOMAIN STRUCTURING
EKATERINBURG; RUSSIA
15-19 NOV 2005

IX ECERS CONFERENCE
PORTOROZ, SLOVENIA
20-24 JUN 2005

MATERIAIS 2005, XII PORTUGUESE MATERIALS SOCIETY MEETING SPM, III INTERNATIONAL MATERIALS SYMPOSIUM
UNIVERSITY OF AVEIRO, PORTUGAL
20-23 MAR 2005

MICROSCOPY CONFERENCE 2005, MC 2005
DAVOS, SUÍÇA
28 AGO - 02 SEPT 2005

SCHOOL ON MATERIALS AND CULTURAL HERITAGE (IN MATERIALS 2005)
UNIVERSITY OF AVEIRO, PORTUGAL
20 MAR 2005

SIMPÓSIO: FERTILIDADE MASCULINA: PROBLEMAS E PERSPECTIVAS
UNIVERSITY OF AVEIRO, PORTUGAL
29 JUN 2005

SSI 15 - INTERNATIONAL CONFERENCE ON SOLID STATE IONICS
BADEN, GERMANY
JUN 2005

SYMPOSIUM E-MATERIALS IN ELECTROMAGNETIC FIELDS-9TH EUROPEAN CERAMIC SOCIETY CONFERENCE
PORTOROZ, SLOVENIA
19-23 JUN 2005

WORKSHOP – POLIURETANOS: ASPECTOS FUNDAMENTAIS E PROCESSOS DE FABRICO
UNIVERSITY OF AVEIRO, PORTUGAL
30 SEPT 2005

WORKSHOP ON MATERIALS AND TECHNOLOGIES ASSOCIATED TO MICRO/NANO – FABRICATION OF STRUCTURAL COMPONENTS
UNIVERSITY OF AVEIRO, PORTUGAL
23 MAR 2005

XIX ENCONTRO DA ASSOCIAÇÃO DE TÉCNICOS DE CELULOSE E PAPEL DE PORTUGAL (TECNICELPA)
TOMAR, PORTUGAL
20-23 APR 2005

1º CURSO DE TRABALHOS PRÁTICOS DO 12º ANO
UNIVERSITY OF AVEIRO, PORTUGAL
12 NOV 2005

2º CURSO DE TRABALHOS PRÁTICOS DO 12º ANO
UNIVERSITY OF AVEIRO, PORTUGAL
28 NOV 2005

ANALYTICAL TOOLS WITH APPLICATIONS FOR WOOD AND PULPING CHEMISTRY (COST E41 ACTION)
2005

CO2 NAS INDÚSTRIAS DE ALTAS TEMPERATURAS – CIAT
UNIVERSITY OF AVEIRO, PORTUGAL
16 DEC 2005

JOINT MASTER PROGRAMME IN MATERIALS SCIENCE AND ENGINEERING
2004

COST 525 – GRAIN BOUNDARY ENGINEERING, SUMMER SCHOOL
GÉNOVA, ITÁLIA
MAY 2005

CURSO AVANÇADO: FLOW CYTOMETRIC AND MICROSCOPY ANALYSES OF REPRODUCTIVE CELLS
UNIVERSITY OF AVEIRO, PORTUGAL
30 SEPT 2005

MATERIAS 2005
UNIVERSITY OF AVEIRO, PORTUGAL
20-23 MAR 2005

THE SCIENCE & TECHNOLOGY WEEK
2005

ACTIVIDADE LABORATORIAL (AL 1.5): COMO DETERMINAR A CONCENTRAÇÃO DE UMA SOLUÇÃO CORADA PELA INTENSIDADE DA SUA COR? (TRABALHOS PRÁTICOS DO 12º ANO")

PHYSICOCHEMISTRY OF LIGNIN
ARKHANGELSK TECHNICAL UNIVERSITY,
03 - 06 JUL 2005

POLIURETANOS: ASPECTOS FUNDAMENTAIS E PROCESSOS DE FABRICO
UNIVERSITY OF AVEIRO, PORTUGAL
30 SEPT 2005

SESSÕES DE DEBATE SOBRE TECNOLOGIA CERÂMICA: A CONFORMAÇÃO NA TECNOLOGIA CERÂMICA: PERSPECTIVAS DE DESENVOLVIMENTO
UNIVERSITY OF AVEIRO, PORTUGAL
16-19 MAY 2005

SESSÕES DE DEBATE SOBRE TECNOLOGIA CERÂMICA: A SECAGEM E A COZEDURA NA TECNOLOGIA CERÂMICA: PERSPECTIVAS DE DESENVOLVIMENTO
UNIVERSITY OF AVEIRO, PORTUGAL
16-19 MAY 2005

"RELATIVIDADE À VELOCIDADE DO CARACOL"
AMARAL V
UNIVERSITY OF MINHO, PORTUGAL
2005

"RELATIVIDADE À VELOCIDADE DO CARACOL"
AMARAL V
UNIVERSITY OF AVEIRO, PORTUGAL
2005

"POLYMERS FROM RENEWABLE RESOURCES"
GANDINI A
UNIVERSITY OF AVEIRO, PORTUGAL
JUN - JUL 2005

"BIOMASS AS A SOURCE OF NEW PLASTICS: A GREEN ALTERNATIVE TO PETROLEUM"
GANDINI A
SEMANA DA CIÊNCIA E DA TECNOLOGIA, UNIVERSITY OF AVEIRO, PORTUGAL
OCT 2005

A NON DESTRUCTIVE METHOD TO ASSESS DELAMINATION OF CERAMIC
TILESTARI G; ROMAGNOLI M; FERREIRA JMF
IX CONFERENCE & EXHIBITION OF THE EUROPEAN CERAMIC SOCIETY, PORTOROZ, SLOVENIA
19 - 23 JUN 2005

CIÊNCIA E ARTE' SEMANA CULTURAL 2005
COUTINHO JAP
ESCOLA SECUNDÁRIA DE ARCOZELO, PORTUGAL
MAY 2005

CO-EDITOR OF SPECIAL ISSUE ON NANOSCALE FERROELECTRIC IN IEEE TRANSACTIONS ON ULTRASONICS, FERROELECTRICS AND FREQUENCY CONTROL
KHOLKIN A L
2005

COLLABORATION WITH TWO LOCAL INDUSTRIES: FLEXIPOL AND WEBER CIMENFIX, VIA INDUSTRIAL INTERNSHIPS WITH THE PURPOSE OF ESTABLISHING FUTURE RESEARCH COLLABORATIONS

COLLOIDAL PROCESSING – THE BEST APPROACH FOR SELF-REINFORCING AND PRESSURELESS SINTERING NITRIDE-BASED CERAMICS
FERREIRA JMF; OLHERO SMH; XIN X; LIU G AND CHEN K
EUROPEAN CONGRESS ON ADVANCED MATERIALS AND PROCESSES, PRAGUE, CZECH REPUBLIC
5-8 SEP 2005

COMO VICE-PRESIDENTE DA SOCIEDADE PORTUGUESA DE FÍSICA COLABOREI NA PREPARAÇÃO E ORGANIZAÇÃO DE DIVERSAS ACTIVIDADES PARA COMEMORAÇÃO DO ANO INTERNACIONAL DA FÍSICA, 2005

COTEC COURSE – TECHNOLOGY COMMERCIALIZATION
WU A
UNIVERSITY OF PORTO, PORTUGAL
FEB-JUN 2005

"DE COMPOSTOS DE COORDENAÇÃO AOS NANOMATERIAIS"
TRINDADE T
UNIVERSITY OF COIMBRA, PORTUGAL
18 MAY 2005

DESENVOLVIMENTO DE ACTIVIDADES LABORATORIAIS PARA A 1ª UNIDADE DO PROGRAMA DA DISCIPLINA DE QUÍMICA DO 12º ANO E DEMONSTRAÇÕES DESSAS ACTIVIDADES LABORATORIAIS DEDICADAS A PROFESSORES E ALUNOS
FRADE J
NOV 2005

DOS 0ºC AOS -195ºC EM 10 SEGUNDOS – DEMONSTRAÇÃO EXPERIMENTAL PARA ALUNOS
RIBEIRO-CLARO, P
SEMANA DA CIÊNCIA E DA TECNOLOGIA, UNIVERSITY OF AVEIRO, PORTUGAL
23 NOV 2005

EDITOR ASSOCIADO DA REVISTA INTERNACIONAL "TRANSACTIONS ON ULTRASONICS, FERROELECTRICS AND FREQUENCY CONTRO DE IEEE"
KHOLKIN A L
USA
2005

FROM MOLECULAR COMPOUNDS TO NANOSTRUCTURED MATERIALS (LECTURE)
TRINDADE T
UIMP, PONTEVEDRA, GALIZA- SPAIN
05 - 09 SEPT 2005

IMPLEMENTAÇÃO DA PLATAFORMA QUIMIMATER PARA COLABORAÇÕES COM ESCOLAS NO ÂMBITO DA DISCIPLINA DE QUÍMICA DO 12º ANO
FRADE J
2005

INFLUENCE OF LITHIUM OXIDE AS AUXILIARY FLUX ON THE PROPERTIES OF TRIAXIAL PORCELAIN BODIES
TULYAGANOV D; FERNANDES H; AGATHOPOULOS S; FERREIRA JMF
IX CONFERENCE & EXHIBITION OF THE EUROPEAN CERAMIC SOCIETY, PORTOROZ, SLOVENIA
19-23 JUNE 2005

MEMBER OF THE EDITORIAL BOARD OF THE JOURNAL OF SOLID STATE
KHARTON V
2005

MEMBRO DA COMISSÃO FERROELÉCTRICA DA SOCIEDADE IEEE
KHOLKIN AL
USA
2005

NANOTECNOLOGIA: PROGRAMA RADIOFÓNICO NA TSF
TRINDADE T AND ROCHA J
26 DEC 2005

OLIMPÍADAS DE QUÍMICA - FINAL
RIBEIRO-CLARO, P
UNIVERSITY OF AVEIRO, PORTUGAL
04 MAI 2005

OLIMPÍADAS DE QUÍMICA – SEMIFINAL
RIBEIRO-CLARO, P
UNIVERSITY OF AVEIRO, PORTUGAL
06 MAR 2005

OLIMPÍADAS DE QUÍMICA JÚNIOR
RIBEIRO-CLARO, P
UNIVERSITY OF AVEIRO, PORTUGAL
23 APR 2005

ORGANIZADOR E MEMBRO DO CONCELHO CONSULTIVO DE CONFERÊNCIA EUROPEIA ECAPD
KHOLKIN AL
2005

ORGANIZADOR E MEMBRO DO CONCELHO CONSULTIVO DE CONFERÊNCIA INTERNACIONAL ISDS
KHOLKIN AL
2005

PALESTRA COM FORMATO DE DEMONSTRAÇÕES EXPERIMENTAIS SOBRE O TEMA “À VOLTA DE UMA BEBIDA TIPO COLA”
EM CONJUNTO COM J A L COSTA
SEMANA DA CIÊNCIA E TECNOLOGIA, UNIVERSITY OF AVEIRO, PORTUGAL
23 NOV 2005

PALESTRA DO TIPO “CONVERSAS COM OS ALUNOS” SOBRE O TEMA “ONDE ESTÁ A QUÍMICA NO AMBIENTE?”
SEMANA DA CIÊNCIA E TECNOLOGIA, UNIVERSITY OF AVEIRO, PORTUGAL
23 NOV 2005

PARTICIPAÇÃO NA SEMANA DA PRÁTICA PEDAGÓGICA
SANTOS T

PARTICIPAÇÃO NAS OLIMPÍADAS NACIONAIS DA QUÍMICA
SANTOS T

PROCESSING OF ADVANCED CERAMICS BY ENVIRONMENTALLY FRIENDLY TECHNIQUES
FERREIRA JMF
INSTITUTO DE INGINERÍA, UNAM, MÉXICO
02 DEC 2005

PROCESSING OF TRADITIONAL CERAMICS FROM RAW MATERIALS TO FINAL PRODUCTS
FERREIRA JMF
INSTITUTO DE INGINERÍA, UNAM, MÉXICO
30 NOV 2005

PROGRAMA “EUREKA – ESPECIAL DE NATAL”
RIBEIRO-CLARO P
RÁDIO TSF, PORTUGAL
18 DEC 2005

ROGRAMA “QUIMIMATER” – PLATAFORMA DE COLABORAÇÃO PARA O ENSINO DA QUIMICA DE MATERIAIS NO 12º ANO DO ENSINO SECUNDÁRIO
SILVA R F
2005

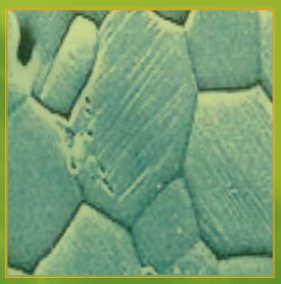
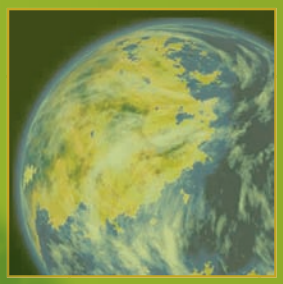
RECYCLING OF INDUSTRIAL RESIDUES IN TRADITIONAL CERAMIC FORMULATIONS – THE STRIDE FOR A CLEAN AND FRIENDLY ENVIRONMENT
FERREIRA JMF; TORRES P AND MANJATE RS
IX CONFERENCE & EXHIBITION OF THE EUROPEAN CERAMIC SOCIETY, PORTOROZ, SLOVENIA
19-23 JUNE 2005

SEMANA DA CIÊNCIA E TECNOLOGIA DA UA - 'ENSINAR A ENSINAR MATERIAIS'
VILARINHO P M
UNIVERSITY OF AVEIRO, PORTUGAL
21-27 NOV 2005

SUITABILITY OF TRADITIONAL CERAMIC FORMULATIONS TO INCORPORATE SEVERAL INDUSTRIAL WASTES – CASE STUDIES
FERREIRA JMF
INSTITUTO DE INGINERÍA, UNAM, MÉXICO
01 DEC 2005

SECTION 5

PROJECTS


Projects Terminated

During 2005

ACER -ADVANCED CERAMIC MATERIALS: SYNTHESIS AND STRUCTURE
FP5 HPMT/CT/2000/00206

AROMA PERMEABILITY IN PACKING MATERIALS
POCTI/EQU/43356/2001

BIO-REACTOR BASED VALCULAR BONE TISSUE ENGINEERING - TISSUE REACTOR
G5RD-CT-2000-00282

CARACTERIZAÇÃO DE ARGAMASSAS CALCÁRIAS POR RESSONÂNCIA MAGNÉTICA NUCLEAR E CARACTERIZAÇÃO DE BETÕES REFRACTÁRIOS POR MICROSCOPIA ELECTRÓNICA DE VARRIMENTO, AMBIENTAL
ACÇÕES INTEGRADAS

CELLULOSE: SURFACE PROPERTIES, INTERACTION WITH BINDING DOMAINS AND ENZYMATIC MODIFICATION
POCTI/44368/2002

COMPOSITE SCAFFOLDS FOR BONE TISSUE ENGINEERING
CTS 2002/05

COST 525 ACTION – ADVANCED ELECTRONIC CERAMIC GRAIN BOUNDARY ENGINEERING
COST PROGRAMS

COST 528 ACTION - CHEMICAL SOLUTION DEPOSITION OF THIN FILMS
COST PROGRAMS

DESENVOLVIMENTO DE FILMES FERROELÉCTRICOS RELAXORES E TECNOLOGIAS PARA DISPOSITIVOS PIEZOELÉCTRICOS
ACORDOS DE COOPERAÇÃO

DEVELOPMENT OF ON LINE CONSUMER EDUCATION TOOLS FOR ADULTS (DOLCETA)
COMISSÃO EUROPEIA, DIRECTÓRIO GERAL PARA A PROTECÇÃO DA SAÚDE E DO CONSUMIDOR

EFFECTS OF MAGNESIUM ON THE STRUCTURE AND SURFACE REACTIVITY OF SILICA-BASED GLASSES FOR BIOMEDICAL APPLICATIONS
POCTI/CTM/46251/2002

ESPECTROSCOPIA DE RMN DE ALTA PRESSÃO DE POLÍMEROS E BIOPOLÍMEROS EM EMULSÕES EM CO_2 / HIGH - PRESSURE NMR SPECTROSCOPY OF POLYMERS AND BIOPOLYMERS IN CO2 EMULSIONS
POCTI/QUI/4231/2001

ESTUDO DA TOXICIDADE DE COMPOSTOS RELEVANTES EM AMBIENTES INDUSTRIAIS
PEDIDOS DE APOIO PARA INVESTIGAÇÃO AO IDICT

IMMOBILIZED SURFACE RARE EARTH ARYLOXIDES. ACTIVATION OF SMALL MOLECULES (CO_2, CH_4, C_2H_6)
POCTI/QUI/42919/2001

MELHORIA DA BRANCURA DAS PASTAS DE EUCALIPTO OBTIDAS PELO MÉTODO DO SULFITO ÁCIDO COM BASE DE MAGNÉSIO
CAIMA – INDÚSTRIA DE CELULOSE

MELHORIA DO RENDIMENTO E DAS CARACTERÍSTICAS FÍSICO-MECÂNICAS DAS PASTAS DE EUCALIPTO OBTIDAS PELO MÉTODO DO SULFITO ÁCIDO COM BASE DE MAGNÉSIO
CAIMA – INDÚSTRIA DE CELULOSE SA

MIXED CONDUCTING MEMBRANES FOR PARTIAL OXIDATION OF NATURAL GAS TO SYNTHESIS
NATO SfP 978002

NANOENGINEERING OF MAGNETIC AND LUMINESCENT PARTICLES FOR CELL SEPARATION AND BIOLABELING TECHNIQUES
Nanoenge.../CTS15

NMR STRUCTURAL STUDIES OF THE ACTIVE CENTER OF 5 - AMINOLEVULINATE SYNTHASE, THE FIRST ENZYME OF THE HEME BIOSYNTHETHIC PATHWAY
(POCTI/BME/39184/2001

NOVOS MATERIAIS COM BASE EM FIBRAS DE CELULOSE
PRAI-CENTRO

SIMULATED PLASMA FOR ASSESSMENT OF THE IN VITRO BIOACTIVITY OF MATERIALS
POCTI/QUI/47051/2002

SIMULATION OF CYCLIC SEPARATION PROCESSES USING PARALLEL COMPUTING
POCTI/EQU/46055/2002

STUDIES OF COLOSSAL MAGNETORESISTIVE OXIDES WITH RADIOACTIVE ISOTOPES (IR)
CERN/FNU/49509/2002

TAILORED OXOMOLYBDENUM CATALYSTS
POCTI/QUI/37990/2001
TAILORED THIN PLASMA POLYMERS FOR SURFACE ENGINEERING OF COIL COATED STEEL
ECSC Contract 7210-PR-383 CECA

TÉCNICAS, FERRAMENTAS E PARADIGMAS INOVADORAS DE IMAGIOLOGIA PARA DIAGNÓSTICO
CTS/25

THEMATIC NETWORK ON SOLID OXIDE FUEL CELL TECHNOLOGY (SOFCNET)
ENG2-CT2002-20652 SOFCNET

TRADITIONAL AND NOVEL DIAGNOSIS TECHNIQUES IN THE BIOMONITORING OF THE MALE FERTILITY
CTS/22/2002

VEDACERAM - ANÉIS VEDANTES COMPÓSITOS DE MATRIZ CERÂMICA (NITRETO DE SILICIO)
POCTI/2002/6443

Projects in Progress

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

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

APPLICATION OF NMR METHODS FOR THE CHARACTERIZATION AND QUALITY CONTROL OF BEER

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

BIOLEARN - LEARNING FROM NATURE HOW TO DESIGN BIOMIMETIC ROUTES FOR PRODUCING CALCIUM-PHOSPHATE COATINGS ON POLYMERI
POCTI/CTM/38803/2001

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

CATALYTIC CONVERSION OF CARBOHYDRATES INTO FURAN DERIVATIVES
POCI/QUI/56112/2004

CHARACTERIZATION OF HETEROPOLYSACCHARIDES USING MASS SPECTROMETRY
POCI/QUI/59337/2004

CHEMICAL BATH DEPOSITION OF BISMUTH BASED PIGMENTS
POCTI/QUI/46199/2002

COMBUSTION SYNTHESIS OF ONE DIMENSION ELONGATED α-SIALON CRYSTAL TO BE USED AS REINFORCING AGENTS FOR CMC PROCESSED BY COLOSSAL
POCTI/CTM/39419/2001

CONTROLLING THE LENGTH SCALE THROUGH "CHEMIE DOUCE": FROM INORGANIC FUNCTIONAL MATERIALS TO ORGANIC-INORGANIC HYBRIDS
POCTI/CTM/46780/2002

CONTROLO DE QUALIDADE NA ÁREA DA TECNOLOGIA DE FUNDIÇÃO
PROTOCOLO DE COLABORAÇÃO ENTRE FUNFRAP – FUNDIÇÃO PORTUGUESA E A UNIVERSIDADE DE.AVEIRO

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

COST 539 ACTION – ELECTROCERAMICS FROM NANOPOWDERS PRODUCED BY INNOVATIVE METHODS-ELENA
COST PROGRAMS

DEPOSIÇÃO DE CERAS PARAFÍNICAS EM OLEODUTOS
GRICES – CAPES

DESENVOLVIMENTO TECNOLÓGICO APLICADO A MATÉRIAS-PRIMAS, PROCESSOS E PRODUTOS CERÂMICOS
ACORDOS DE COOPERAÇÃO

DEVELOPMENT AND CHARACTERIZATION OF CERAMIC COMPOSITE MATERIALS FOR THERMOMECHANICAL APPLICATIONS
REEQ - PROGRAMA NACIONAL DE RE-EQUIPAMENTO CIENTÍFICO

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

DEVELOPMENT OF A FIXED SITE CARRIER CERAMIC ULTRAMICROPOROUS MEMBRANE REACTOR FOR THE SELECTIVE SEPARATION AND OXIDATION OF CARBON MONOXIDE TO CARBON DIOXIDE
POCI/EQU/59345/2004

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

DIELECTRIC PROPERTIES AND LATTICES DYNAMICS OF GRANULAR MATERIALS
POCTI/CTM/45284/2002

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

ECOFUELS - CONVERSÃO ELECTROQUÍMICA DE COMBUSTÍVEIS
REEQ - PROGRAMA NACIONAL DE RE-EQUIPAMENTO CIENTÍFICO

ENERGY TRANSFER FROM ELECTROLUMINESCENT POLYMERS TO PHOSPHORESCENT DOPANTS
POCTI/CTM/40063/2001

EQUILIBRIUM AND INTERFACIAL PROPERTIES OF WATER + OIL SYSTEMS
POCI/58239/2004

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

FAME - NETWORK OF EXCELLENCE: FUNCTIONALISED ADVANCED MATERIALS AND ENGINEERING OF HYBRIDS AND CERAMICS
FP6 2004 NMP3/CT (NOE 500159)

FORMULAÇÃO DE UMA PASTA DE GRÉS VERMELHO CERALFA

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

IMMOBILIZED PHOTOSENSITIZER AS NEW MATERIALS IN WATER TREATMENT
POCI/CTM/58183/2004

IMPROVED MATERIALS PROCESSING THROUGH TAILORING THE SURFACE CHARACTERISTICS OF NANO AND MICRON-SIZED POWDERS
ACORDOS DE COOPERAÇÃO

IMPROVING THE YELD OF *EUCALYPTUS GLOBULUS* KRAFT PULP PRODUCTION: STRATEGIES, MECHANISMS AND IMPACT ON PULP ECONOMY
POCTI/EQU/46124/2002

INDUSTRIAL ECOLOGY: USUING SLUDGE FROM WASTEWATER TREATMENT PLANT OF SURFACE TREATMENT AS RAW MATERIAL FOR THE PRODUCTION OF REFRACTORIES
POCTI/CTA/42448/2001

LABORATÓRIO DE BIOMATERIAIS E DISPOSITIVOS BIOMÉDICOS
REEQ - PROGRAMA NACIONAL DE RE-EQUIPAMENTO CIENTÍFICO

LABORATÓRIO DE MICROSCOPIA ELECTRÓNICA ANALÍTICA DE RESOLUÇÃO ATÓMICA - GRANDES EQUIPAMENTOS DE USO COMUM
REEQ - PROGRAMA NACIONAL DE RE-EQUIPAMENTO CIENTÍFICO

LUMINESCENT LANTHANIDE SILICATE SYNTHESIS USING LANTHANIDE COMPLEXES AS STRUCTURE DIRECTING AGENTS
POCI/CTM/58863/2004

MATERIALS SCIENCE AND FUNDAMENTAL RESEARCH ON NANO-STRATIFIED RARE EARTH BASED COMPOUNDS FOR MAGNETIC REFRIGERATION AND SENSOR APPLICATIONS - UNUSUAL MARTENSITIC TRANSITIONS DRIVEN BY THE ELECTRONIC FLUID
POCI/CTM/61284/2004

MELHORAR O LABORATÓRIO CENTRAL DE DIFRACÇÃO DE RAIOS-X DA UNIVERSIDADE DE AVEIRO
REEQ -PROGRAMA NACIONAL DE RE-EQUIPAMENTO CIENTÍFICO

MNAA - NETWORKED CENTRE OF EXCELLENCE IN MATERIALS FOR THE ECONOMIC DEVELOPMENT OF THE ATLANTIC AREA
INTERREG III

MOLECULAR HYBRIDS OF POLYOXOMETALATES AND DIPOLAR ORGANIC MOLECULES: SYNTHESIS, CHARACTERIZATION AND DETERM
POCTI/CTM/37713/2001

MOULD PROTEOME REGULATION BY SUBSTRATE BIOAVAILABILITY: DEPOLYMERISING ENZYMES LIKELY TO ALTER BIOPOLYMER NAN
POCTI/QUI/56229/2004

MULTIFUNCTIONAL ANIONIC CLAYS
POCI/CTM/58507/2004

NANOCRYSTALLINE DIAMOND (NCD) FILMS ON SILICON NITRIDE CERAMICS FOR TRIBOLOGICAL APPLICATIONS
POCI/CTM/59449/2004

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