

**Institute of Material and Environmental  
Chemistry**

**Chemical Research Center  
Hungarian Academy of Sciences**

**Annual Report  
2003**

**Budapest  
2004**

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## INTRODUCTION

The Institute of Materials and Environmental Chemistry (IMEC) is a non-profit research institute belonging to the Chemical Research Center of the Hungarian Academy of Sciences. Mission of IMEC is to perform high-level basic and applied research in materials science and engineering and environmental chemistry.

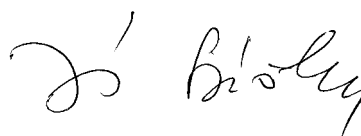
Our research in materials science is aiming at revealing chemical relationships among composition, microstructure, properties and processing of structural and functional materials. The models include advanced metallic and ceramic materials, traditional and new polymers, special surface layers and coatings, and different composite materials.

In environmental chemistry, we strive to disclose basic chemical, technological and engineering phenomena in order to facilitate the development of new processes and technologies of minimum environmental impact. The main research topics are as follows: *(a)* research in environmental analysis and development of new analytical methods for environmental protection, *(b)* research on utilization of renewable sources of energy and *(c)* new methods, processes and technologies of waste processing and utilization.

In the Annual Report, we present details of our activity in the year 2003. We give information about our national and international scientific cooperation, research projects, grants, participation in the university education and research facilities, as well.

I warmly recommend this Report to the Reader's attention.

Budapest, March 2004.



János Szépvölgyi  
Director

# 1 THE ORGANIZATION<sup>1</sup>

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György Horányi, PhD, DSc, scientific adviser  
Tamás Pajkossy, PhD, DSc, scientific adviser  
Sándor Szabó, PhD, DSc, scientific adviser

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<sup>1</sup> As for March 1, 2004.

István Bakos, PhD, senior research fellow  
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## **Secretariat**

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Andrea Kráncz, secretary  
Ágota Mezei-Seres, financial manager  
Gábor Babos, technician  
József Szűcs, technician

## 2 RESEARCH ACTIVITIES IN 2003

### 2.1 Research in materials chemistry

#### 2.1.1 Formation and characterization of nanolayers

*Imre Bertóti, Miklós Mohai, András Tóth, Tamás Ujvári*

During research on the formation of carbon nitride layers in low-pressure plasmas, the optical emission spectra of nitrogen glow-discharge plasma created between graphite electrodes have been studied. In addition to the strong bands of neutral and ionized nitrogen species, bands of CN radicals of high rotational temperatures ranging from 9000K to 13500K were also observed. Most probably, these radicals are playing an important role in the formation of CN<sub>x</sub> layers.

After implantation of aromatic polyimide by Co- and Fe-ions of high fluency, we observed the formation of O- and N-depleted carbonized layers by XPS. In these layers, iron was present in oxidized state, while cobalt in both metallic and oxidized states. High fluency metal ion implantation can be a possible method of preparing metal/polymer nanocomposites with special parameters.

Silicon-containing, carbon-based nanocomposite layers were prepared by plasma assisted CVD using an in-house developed setup. Silicon-containing precursors, such as tetramethylsilane, hexamethyl-disiloxane or hexamethyl-disilazane were introduced into and activated by an electron cyclotron wave resonance plasma beam source. Few hundred nanometre thick layers were deposited onto Si and Al substrates. Chemical characterisation by XPS showed that the Si/C ratio in the deposited layers was always higher than that in the precursor molecules and the Si-content approached 30 at%. Formation of large Si clusters or Si-carbide ones could not be detected. On the other hand, evolution of Ni<sub>3</sub>C clusters were found in the layers of magnetron-deposited Ni-C nanocomposites investigated for comparison.

In a project aiming at the development of human endoprostheses of long service life, samples of UHMWPE were treated by plasma immersion ion implantation. This research, conducted together with Hungarian and foreign partners, resulted in a significant improvement of the nanomechanical and tribological properties of UHMWPE samples, as compared to untreated ones.

#### 2.1.2. Synthesis of advanced materials in thermal plasmas

*Loránd Gál, Zoltán Károly, Ilona Mohai, János Szépvölgyi*

We established the optimum synthesis conditions of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> powders of various morphologies in RF inductively coupled thermal plasmas. Both dense and hollow microspheres were produced. The latter ones are of great importance in gas chromatography. Conditions for producing nanosized powders for ceramic processing were established, as well.

We studied the synthesis of fullerenes from graphite powder in a radiofrequency (RF) thermal plasma reactor. The optimum conditions of fullerene production were determined by optical emission spectroscopy; fullerenes could be produced with higher efficiency in RF plasma reactor than with other synthesis methods. It was verified both theoretically and experimentally that formation of fullerenes of higher molecular mass is favored in RF plasma reactor.

### **2.1.3. Degradation and stability of polymers**

*Edina Epacher, Enikő Földes, Erik Maloshik, Béla Pukánszky*

Correlation was studied among the industrial polymerization conditions of high-density polyethylene (HDPE), the characteristics of the polymer powder produced, the stability of the pellets and the properties of end-product. We developed a method to determine the chain structure of PE powders by DRIFT spectroscopy, and we validated it by <sup>13</sup>C NMR spectroscopy.

The polymers are usually stabilized before processing. The stabilizing mechanism and the effectiveness of different phosphorous antioxidants were studied and it was concluded that the general reaction mechanism given in the literature is oversimplified, and not valid. The chemical nature i.e., type of groups connected to the phosphorous atom strongly affected the efficiency and reaction mechanisms of stabilizers.

HDPE is used for producing pipes in largest quantities. The fate of stabilizers in water pipes is still unknown, though the question is important from the points of view of health and environmental protection. The hydrolytic stability of some common phenolic antioxidants was determined, and found to be strongly dependent on the chemical structure of the stabilizer. The stability of the antioxidant used the most frequently in water pipes is low the practical consequences of which are unpredictable.

### **2.1.4. Heterogeneous polymer systems**

*Ágnes Ábrányi, Erika Bódi-Fekete, Livia Dányádi, János Gulyás, János Móczó, András Pozsgay, Tünde Pozsgay, Béla Pukánszky, Béla Pukánszky Jr., László Százdí*

Research of the heterogeneous polymer systems involves traditionally the study of polymer blends, multicomponent polymer systems and composites filled with fibers. Lately general correlation was established among the miscibility of the polymers, the structure formed during blending and the properties of the blend.

In 2003, we started to the PVC-based blends, in cooperation with BorsodChem Co. The main objective of the research was to determine the interaction between PVC, chlorinated PVC and chlorinated polyethylene, as well as its effects on the mutual miscibility, structure and properties of blends. The conclusions of particular research study of polymer blends were summarized in a book chapter.

We found relationships among the chemical reactions during electrochemical surface treatment of carbon fibers, the number of reactive groups formed, and the interfacial adhesion in the polymer/carbon fiber composites. The interfacial reactions and adhesion were studied in epoxy and polycarbonate matrices. The strength of adhesion depended on the number of reactive groups on the fiber surface in epoxy matrix, while in polycarbonate on the limited number of functional end-groups of the polymer. The strength of interfacial interaction was modified by treatment with coupling agents. Correlation was established among the chemical reactions, the structure of the interphase and the interfacial shear stress.

Relationship between structure and properties of the composites filled with particles was also investigated. We concluded that aggregation of the particles depends on the characteristics and the amount of them, as well as on the processing conditions. The aggregation leads to some deterioration of the properties of the composite. Effects of the inter-phase in particulate filled composites on the properties and the deformation mechanism were studied by stress analysis. The interphase increased the yield stress and the modulus of the composite.

Study of polymer layered silicate nanocomposites prepared by delamination yielded in important new results. We showed that the surface treatment of the layered silicate and the degree of delamination determine the properties of nanocomposites.

### **2.1.5. *Biologically degradable polymers***

*Szilvia Klébert, Béla Pukánszky*

Research aiming at the preparation of biologically degradable polymers and the use of natural raw materials was conducted in two directions. Modification of cellulose acetate with different aliphatic polyesters [polycaprolactone and poly(lactic acid)] was investigated. Effects of the conditions of modification on the structure and properties of the product were determined. The temperature and concentration of catalyst yielding maximum grafting were established for caprolactone. The monomer and the grafted caprolactone affected the properties of cellulose acetate in different ways: the former one resulted in internal, while the second one in external plasticizing effect. Effectiveness of the two processes was completely different.

Acidic and enzymatic decomposition of cellulose was studied for producing biologically degradable polymer by reacting the decomposition product with aliphatic polyester. The acidic decomposition yielded in more uniform products than the enzymatic decomposition. PP/natural fiber composites can be used for preparation of common articles and acoustic devices.

### **2.1.6. *Synthesis of well-defined polymer architectures***

*Gábor Erdődi, Tamás Fónagy, Béla Iván, Orsolya Kovács, István Szanka, Márta Szesztay*

Our studies aimed at the preparation of well-defined polymer structures in terms of functionality, composition, molecular mass, monomer sequence order and topology by new polymerization techniques. The novel macromolecular systems were subjected to complex investigations from the viewpoint of material science. On the basis of this strategy, several linear and branched polymers with different functionalities have been synthesized and characterized.

In cooperation with the Polymer Institute Dresden, poly(propylene-*g*-styrene) graft copolymers with well-defined structure and molecular weights were synthesized by metallocene catalyzed copolymerization of propylene and polystyrene macromonomers obtained by quasiliving radical polymerization. For the first time, on basis of particular graft copolymers, the effect of graft microstructure on the efficiency of compatibilization was systematically investigated by scanning electron microscopy in polypropylene/polystyrene blends. Surprisingly, in contrast to the expected tendency, graft copolymers with shorter polystyrene side chains produced better compatibility. We explained these striking new results by considering not only the composition and length of the side chains, but also the effect of branching frequency of the well-defined poly(propylene-*g*-styrene) graft copolymers on the compatibilization efficiency.

We developed a completely new method for the synthesis of hyperbranched polystyrenes. Linear polystyrenes with chlorine terminus were synthesized by quasiliving atom transfer radical polymerization. In the presence of Lewis acid, this chain end can react with the pendant phenyl ring of another polystyrene molecule by Friedel-Crafts alkylation. The repetition of self-alkylation process yields hyperbranched polystyrene. NMR spectroscopy and investigations on the solvent properties confirmed the structure of these new macromolecules.

### **2.1.7. Carbocationic polymerization**

*Péter Werner Groh, Béla Iván, Árpád Máthé, Zsuzsanna Nagy, Viktória Pálfi, Narmandakh Mijid, Kálmán Tóth*

We have carried out detailed investigations on the effect of chelating compounds on quasiliving carbocationic polymerization of isobutylene. Comparison of our newest results with our earlier experimental findings surprisingly shows that only special catalyst systems containing some kind of nitrogen-bearing chelating compounds lead to the formation of polyisobutylenes with ultra narrow molecular weight distribution (MWD). In addition, we have discovered a new catalyst system leading to polyisobutylenes with MWDs narrower than the theoretical values. We have proposed a kinetic model for this unique polymerization mechanism not reported previously according to our knowledge.

### **2.1.8. Amphiphilic co-networks**

*Attila Domján, Gábor Erdődi, Márton Haraszti, Béla Iván, Péter Mezey, Sándor L. Szabó*

Amphiphilic conetworks are multicomponent polymer systems consisting of covalently bonded hydrophilic and hydrophobic polymer chains. Owing to the nature of their strong chemical bonding, the two immiscible polymers form a characteristic nanostructure which differs significantly from that of other multicomponent polymer systems.

We have developed a new synthesis method of amphiphilic polymer conetworks. This procedure is based on coupling of functional end-groups of the hydrophilic and hydrophobic polymers under appropriate conditions. Hydroxyl-telechelic three-arm star polyisobutylene was synthesized by quasiliving carbocationic polymerization. Hence, it was possible to synthesize poly(ethylene oxide)-polyisobutylene conetworks of different compositions by using diisocyanate as coupling agent. The effect of experimental parameters on network formation was studied in details. Based on these results the optimum synthesis conditions were determined.

One special property of particular conetworks is that the crosslinking points are situated in the central trifunctional core of the polyisobutylene star polymers. Our studies showed that the swelling characteristics and the degree of crystallinity of poly(ethylene oxide) in the conetworks strongly depend on the composition of new amphiphilic conetworks. It was also found that the ratio of crystalline poly(ethylene oxide) is significantly reduced in poly(ethylene oxide)-polyisobutylene conetworks. The melting point of poly(ethylene oxide) was found to be lower in the conetworks than in the homopolymer.

Novel structural information was obtained on nanophased amphiphilic conetworks composed of hydrophilic and hydrophobic polymer chains using spin-diffusion solid state NMR and small angle X-ray scattering techniques. These results clearly proved the nanophase separation of the hydrophilic and hydrophobic polymer components with 5-15 nm average domain sizes. Surprisingly, the interphase between phase boundaries was extremely small. The two phases are practically distinct from each other. Based on their peculiar constitution and properties, these new polymer conetworks are likely to become promising candidates for the production of novel nanostructured hybrid composites.

### **2.1.9. Research in electrochemistry, electrosorption, electrocatalysis and corrosion**

*Béla Lengyel, Tamás Pajkossy, Éva Dániel-Fekete, Gabriella Lendvay-Győrök, Gábor Mészáros, György Horányi, Sándor Szabó, István Bakos*

We interpreted the peak observed on the double layer capacity vs. electrode potential curve of Pt(111) single crystal as being due to the flip-over of the surface water layer. The impedance spectra of Pt(111) electrodes were analyzed and various adsorption and diffusion related processes were identified.

The  $\text{Fe}^{2+}/\text{Fe}^{3+}$  redox system was studied by the combined impedance- and noise analysis, which had been developed in the former years in our institute to determine the kinetic parameters of reversible electrode reactions. We found that the symmetry factor of the electrode transfer in particular system, within an uncertainty limit of 1%, equals to 0.5, just as predicted theoretically.

We have shown that - in contrast to previous views - in acidic solutions the perchlorate ions can be reduced by metallic iron and cobalt to a significant extent. These results may be important when designing and performing environmental protection actions for diminishing perchlorate pollution.

We proved that processes comprising equilibrium between  $\text{H}^+$  and  $\text{H}_2$  are involving catalysis. According to experience, corrosion protection of zinc or iron is diminished at elevated temperatures. As we have demonstrated, in the presence of oxygen this is due to the increased rate of the cathodic process, since ZnO is a good catalyst of the  $\text{O}_2$  ionization.

### **2.1.10. Synthesis and characterization of metal complexes**

*Judit Fodor, László Kótai, Klára Szentmihályi, Péter Vinkler*

Ionic permanganate complexes were synthesized and studied by TG-MS, TG-gas-titrimetry and XRD. Thermal decomposition of zinc- and copper permanganates containing ammonium ligands involves intramolecular redox reaction with formation of ammonia and  $\text{MeMn}_2\text{O}_4$  type compounds. In these processes partial oxidation of ammonia and formation of small amounts of NO and  $\text{NO}_2$  were detected. Redox reactions were experienced during the thermal decomposition of cadmium salts and hexaammine nickel(II)-permanganate, while the redox reaction of diammin silver(II) salt yielded silver.

Metal complexes of polygalacturonate (PG) and malate were prepared and studied by FTIR, Raman spectroscopy, DSC, TG and XPS. The free radical properties of complexes were investigated by chemiluminometry and LDL-oxidation measurements, respectively. Iron(II)-polygalacturonate formed only under nitrogen and by lyophilization. On 50-60°C and by drying in air,  $\text{Fe(II):Fe(II):PG}=1:1:3$  complex formed, independently of reaction conditions. Investigation of the free radical properties of metal complexes showed that both systems were suitable for determining the antioxidant properties of complexes.

### **2.1.11. Analysis of medicinal plants and studies on their efficiency**

*Krisztina Lado, Gabriella Taba, Klára Szentmihályi, Péter Vinkler*

The organic agents and metal content of several medicinal plants were analyzed by UV spectrometry, GC, GC-MS, HPLC and ICP. Metal ion content and bioactive agents in some medicinal plants originated from abroad differ significantly from that of Hungarian ones. High concentration of Al, As, Ba, Cr, Cu, Fe, Mn, Ni, Ti and caffeine was measured in some Chi-

nese plants. The consumption of teas from these plants may be dangerous, especially in case of metabolic disorders, when the toxic and heavy metals accumulate in liver and bile.

## **2.2 Research in environmental chemistry**

### ***2.2.1. Analysis of organic materials in atmospheric aerosols***

*Marianne Blazsó, Zsuzsanna Czégény*

The organic carbon content of fine aerosols plays an important, yet undetermined role in the atmospheric processes. The sources of the organic material in tropospheric aerosols and the processes taking place in the atmosphere have been studied in cooperation with the Air Chemistry Group of the Hungarian Academy of Sciences (University of Veszprém, Hungary).

We successfully applied the method of thermally assisted hydrolysis and methylation coupled to GC/MS for the chemical analysis of the organic carbon content of aerosols. This method, which is widely spread for the analysis of synthetic or natural polyethers and polyesters in the last years, has been adapted for the analysis of the organic material in the size-resolved fractions of atmospheric aerosols. The constituents of particular, hardly soluble samples of 10-100 µg mass can not be identified by any other methods nowadays. Our results revealed that the organic compounds originating from the burning of forests and pastures partly combined to macromolecules in the aerosols.

### ***2.2.2. Application of liquid chromatography in the environmental protection***

*Eszter Forgács-Tóth, Annamária Jakab, Erzsébet Gere-Pásztí*

We developed new, high-performance liquid chromatography supports. Their retention characteristics were determined, and they were used for pharmaceutical and environmental analyses, as well.

The relationship between the molecular structure and the chromatographic properties was studied for different models. Special attention was devoted to environmental applications.

A novel method was developed for the characterization of plant oils based on their triacylglycerol profile. Based on HPLC/APCI-MS and MALDI-TOFMS analyses the triglycerol profile was evaluated by linear discriminant analysis. Out of the 73 samples 68 were correctly classified indicating a correct classification of nine different types of oils (almond, avocado, grape seed, linseed, mustard seed, olive, pumpkin seed, sesame seed and soybean)

### ***2.2.3. Development of material and energy effective technologies***

*György Mink, László Horváth*

A prototype of a low cost solar hot water system (SHWS) consisting of a flat plate collector made of solar grade plastic and atmospheric hot water tank has been developed, tested and analyzed in co-operation with the Polytechnic of Dunaújváros. The capital cost of particular unit is much lower than that of an SHWS of similar capacity. According to total life cycle cost calculation, the unit offers about 30-40 % cost reduction in 20 year lifetime.

The applicability of batch type extractive distillation using entrainers for the separation of azeotropes of minimum and maximum boiling point, respectively, has been studied in co-

operation with the Department of Chemical Engineering of the Budapest University of Technology and Economics. Efficiency and feasibility of the process have been analyzed by model calculations, and conclusions of these calculations were verified by pilot scale experiments.

#### **2.2.4. Studies on the utilization of biomass materials by thermal methods**

*Gábor Várhegyi, Emma Jakab, Erika Mészáros, Ferenc Till*

The products of a Hungarian experimental plantation for energy crops were investigated. A group of 12 thermogravimetric experiments, representing two grinding levels, three plant genera and four different heating programs were evaluated simultaneously by the method of least squares employing a reaction kinetic model of independent pseudocomponents. All evaluated experiments were well described by the same set of kinetic parameters; only the parameters describing the peak area of the partial processes differed. A technique was developed for the appropriate handling of the non-random errors in the simultaneous evaluation of experiment series.

The ratio of wood and bark in the young shoots is significantly different from that of an older wood. That is why great emphasis has been put on the comparative study of the decomposition characteristics of wood and bark by thermogravimetry - mass spectrometry (TG-MS). Considerable differences have been found in these investigations: more volatiles were produced from wood than from bark; the temperature range and the rate of decomposition also differ. One of the main differences between the chemical composition of wood and bark is the amount of inorganic ions: the mineral matter content is significantly higher in the bark than in the wood. We have pre-treated the samples with a hot water washing procedure in order to eliminate a significant part of these inorganic components and investigate their effects on the thermal decomposition of the samples. The results imply that the thermal behavior of wood and bark are still considerably different after the elimination of some of the inorganic components. However, the macromolecular components decompose at similar temperatures in wood and bark. Since we had a large number of samples and TG-MS data, we have employed a chemometric tool, principal component analysis (PCA) to help the evaluation of the results and the comparison of the samples.

Another work was carried out in an international cooperation with the University of Naples "Federico II" and SINTEF Energy Research, Norway. The variations in chemical composition and the effects of sample origin and pre-treatments were studied. A wood containing high amounts of extractives (chestnut, *Castanea sativa*) was examined. For comparison, a wood with more usual properties from the same plant family (beech) was employed. The similarities and differences of 18 samples were evaluated by 10 empirical characteristics of the TG/DTG curves and by a kinetic modeling. The reliability of the TG/DTG characteristics and the reaction kinetic parameters were also assessed.

#### **2.2.5. Studies on the recycling of plastics and polymer mixtures by pyrolysis**

*Marianne Blazsó, Zsuzsanna Czégény, Emma Jakab*

The interaction of different thermal decomposition reactions was studied in polymer mixtures and in plastics wastes. The effect of a polymer or a plastic additive on the thermal decomposition, pyrolysis product distribution and char formation of another polymer component of the mixture was investigated in details. Our research focused on chemical processes being important in the pyrolytic recycling of plastic wastes. Moreover, reaction conditions promoting or hindering the evolution of polluting decomposition products were established. Analytical pyrolysis experiments were performed online, between 300–900°C by pyrolysis-GC/MS. Evolution of volatiles was followed by pyrolysis-MS or by thermogravimetry/MS.

Thermal decomposition of high impact polystyrene which was flame retarded with brominated additives and antimony trioxide synergist has been studied by thermoanalytical methods. The flame retardants did not influence significantly the primary decomposition reactions of the polymer. However, a two-step reaction took place in the presence of antimony trioxide.

Modification of thermal decomposition reactions have been studied in halogen-containing flame retardants, and in flame retardant-containing wood or plastic wastes due to the presence of zeolite molecular sieve or carbon black in the reaction mixture. The significant changes of product distribution indicated that the alkaline zeolite of large pores held back the accumulation of halogenated aromatic hydrocarbons of larger size (i.e. chlorinated and brominated bisphenol A) in the pyrolysis oil. However, these sieves could not hinder the formation of chloro- and bromophenols. Substantial change in the distribution of thermal decomposition products of ABS (acrylonitrile-butadiene-styrene copolymer, a typical plastic component of electronic and electric waste) was observed in the presence of carbon black. It was interpreted and proved as the involvement of additional reaction routes.

### **2.2.6. *Environment friendly degradation and modification of PVC***

*Béla Iván, Tibor Szakács*

Systematic investigations have been carried out on the chain degradation and fragmentation of PVC under thermo-oxidative conditions. In dilute PVC solutions and in dioctyl-phthalate, the most common plasticizer of PVC, severe chain scission occurs on thermo-oxidative treatment. This phenomenon occurs in the presence of thermal stabilizers too, so these industrial stabilizers are unable to prevent the oxidative chain fragmentation. These new results can be utilized as starting points for oxidative fragmentation of PVC, or for developing new recycling methods under mild oxidative conditions.

### **2.2.7. *Processing of hazardous wastes in thermal plasmas***

*Loránd Gál, Katalin Főglein, Zoltán Károly, Ilona Mohai, János Szépvölgyi*

Decomposition of  $\text{CHCl}_3$  and  $\text{CFCl}_3$  was investigated in a radiofrequency (RF) thermal plasma reactor in neutral and slightly oxidizing conditions, respectively. In neutral conditions highly disperse soot, and chlorinated- and/or fluorinated benzenes were formed as main products. Several polycyclic and aromatic compounds were identified by GC-MS in both the exhaust gas and the toluene extract of the soot.

Extraction of the metal content of red mud, a typical waste from alumina production was studied by different pyrometallurgical processes. We could extract approximately 75% of the iron content by a smelting technology in arc plasma. The molten iron was contaminated by carbon in a few percent, while by silicon, magnesium, titan and aluminum in a smaller degree. Practically all niobium dissolved into the iron melt.

Separation of the metallic components of red mud by roast chlorination was also investigated. It was found that iron and titan could be extracted completely by chlorination with phosgene at 850°C, while aluminum and sodium were extracted in 68% and 82%, respectively.

### ***2.2.8. Purification of soil and groundwater contaminated by chlorobenzenes***

*György Mink, László Horváth*

The possibilities to mitigate harm in the area of Hidas and Garé (Hungary) where the non-proper deposition of about 16 000 tons of polychlorobenzene residues resulted in serious contamination of soil and groundwater have been studied. Pilot scale technologies have been developed and optimized both technical and economic terms. The contaminated soil could be purified by reactive thermal desorption combined with catalytic oxidation of the effluent. For the purification of contaminated water, various technologies (stripping of water combined with the catalytic oxidation of exiting chlorobenzenes, mineralization of chlorobenzenes both by UV oxidation in lamp reactor and in solar photocatalytic reactor) have been developed on pilot scale. Feasibility analysis of technological variants revealed that a reasonable combination of the three methods as above would be the best choice of industrial implementation.

### ***2.2.9. Utilization of industrial wastes***

*László Kótai, Judit Fodor, Klára Szentmihályi*

Biodiesel has been prepared from baking oil by esterification with calcium oxide catalyst. Methods were developed and tested for the characterization of products.

A new technology has been developed for the separation of zinc and iron from galvanic sludge. The zinc content of the sludge was transformed into  $ZnSO_4$  by thermal treatment with  $FeSO_4 \cdot 7H_2O$ . The zinc salt could be removed by aqueous washing, and the iron oxides could be utilized in the ferrous metallurgy.

By saturating waste ion exchanger resin with iron followed by thermal treatment, a special catalyst was prepared which could be applied for the efficient and selective acylation of alcohols and amines by carboxylic acids as acylating agents.

### 3. PARTICIPATION IN NATIONAL RESEARCH PROJECTS

#### Hungarian Scientific Research Fund (OTKA)

- XPS study of the stability of supramolecular Langmuir-Blodgett systems (T25789)
- EIS (Electrochemical Impedance Spectroscopy) method extended to medium amplitude perturbation and its application to corrosion research (T29727)
- Quasiliving, radical polymerizations (T29711)
- Studies of interfacial phenomena in heterogeneous polymers (T29719)
- Research on the biocompatibility of amphiphilic conetworks (F29728)
- Studies on relaxation processes at metal/electrolyte interfaces (T30150)
- Preparation of solid catalysts by structural and surface modification, and their application in transformation of organic compounds (T30156)
- Investigation of layer structures containing  $C_3N_4$  és  $CN_x$  (T30424)
- Structure – property relationships in heterogeneous polymers (T30579)
- Investigation of mechanical properties of modified surface layers (T30833)
- Role of classical and modern methods in the contemporary research of electrocatalysis and electrosorption (T31703)
- Studies of rhenium, iridium and rhodium adsorption on other metal surfaces, and acceleration of corrosion by rhenium (T31846)
- Synthesis of new, asterisk polymers and studies on their solutions (T33107)
- Interaction of thermal decomposition reactions in polymer mixtures (T33111)
- Beta-polypropylene and its binary systems (T34230)
- Study on factors determining the extent and mechanism of polymer degradation and investigation as well as certain aspects of stabilization (T37687)
- Corrosion properties of copper-aluminum bimetallic systems (T37693)
- Effect of additives on thermal decomposition reactions in waste utilization (T37704)
- Basic research by thermal analysis for the optimum utilization of biomass fuels (T37705)
- Studies on the swelling dynamics of new, amphiphilic conetworks (F31901)
- Studies on electrosorption processes (T42452)
- Preparation and study of polymer composites with particles of controlled size and nano particles: adhesion, modification and deformation mechanisms (T43517)
- Formation of organic aerosol in cloud processes (T43578)
- Development of new MINP models for mass transfer networks (F35085)

## **Other Hungarian research grants**

- Development of human joint prostheses for long service life (NRDP 1/013/2001)
- Research on complex prevention methods for improving the hygienic state in Hungary: in vitro and in vivo investigations on natural antioxidants (NRDP 1/016/2001)
- Design of drugs and diagnostics based on validated target molecules: investigation of natural organic substances and their metal complexes with free radical scavenger activity (NRDP 1/047/2001. Coordinator: IC CRC HAS)
- Development of marketable products of low environmental impact from wastes of aluminum industry (NRDP 3/035/2001. Coordinator: University of Veszprém)
- Production and application of products made of composites being biologically degradable and/or based on natural raw materials (NRDP 3A/0036/2002. Coordinator: University of Debrecen)
- Polyamide-6 nanocomposite (Ministry of Education ALK-00151/2001)
- Importance of non-nutritive alimentary factors in the therapy of liver and inflammatory bowel diseases: experimental and human studies (Ministry of Welfare ETT 250/2000)
- Role of natural antioxidants on metabolism of metals and on redox homeostasis in liver and inflammatory bowel diseases, intestinal tumors and porphyria cutanea tarda (Ministry of Welfare ETT 02/2000)
- Measuring system for the comprehensive research of hybrid separation processes (Ministry of Education, 02335/2000)
- Purification of soil and groundwater contaminated by chlorobenzenes (Ministry of Education 00919/2002)
- Studies on the synergic and antagonic effects of environmental pollutants on the fertility of soils (Ministry of Environmental Protection KO441232001)
- Adsorption and desorption of surfactants on Hungarian soils (Ministry of Environmental Protection, KAC KO441242001)
- New supporting material for biological destruction of mosquitoes by helicopters (Ministry of Education, 00708/2003)
- Grant for research facilities (Ministry of Education, MU-55/02)

## 4. PARTICIPATION IN INTERNATIONAL RESEARCH PROJECTS

### Research Projects of the European Community

- New surface modified flame-retarded polymeric systems to improve safety in transportation and other areas (FLAMERET; G5RD-CT-1999-00120).
- Novel surface engineered counter-face systems for prostheses application (NSE PRO; G5ST-CT-2002-50247).
- Novel arc plasma process for the decomposition of hazardous wastes and for the simultaneous production of valuable building materials (WASTILE; GRDI-2000-25035)
- Waste management and recycling of WEEE-process. Integrated thermochemical treatment of halogen-containing materials (GIRD-CT-2002-03014)

### Other research cooperation

- Research in plasma chemistry (RAS Institute for General and Inorganic Chemistry, Moscow, Russia)
- Determination of the potential of zero charge of Pt(111) electrodes (MÖB-DAAD program; cooperation with the Department of Electrochemistry, Ulm University, Germany)
- Synthesis and reactions of permanganate salts (MTA-INSA 3/2001-2003; cooperation with Jodhpur University, India)
- Study on preparation and properties of organic adsorbents and catalyst supports (MTA-INSA 8/2001-2003; cooperation with the Indian Institute of Technology, Hyderabad, India)
- Development of sisal fiber reinforced composites as new engineering materials (Hungarian – South African cooperation DAK-2/2001)
- Development of new analytical methods for studying the environmental impact of hazardous materials and drugs (cooperation with the Department of Drug Chemistry, Medical University of Gdansk, Poland)
- Studies on the biological and environmental effects of anionic surfactants (cooperation with the Institute of Polymer Chemistry, Bratislava, Slovakia)
- Electrolytic modifiers of the background in capillary electrophoresis (cooperation with the Institute of Physiology, Prague, Czech Republic)
- Development and application of new methods for stabilizing coloring agents in red wines (cooperation with the National Institute of Agrochemistry, Lisbon, Portugal)

## 5. PARTICIPATION IN THE UNIVERSITY EDUCATION

In 2003, coworkers of IMEC delivered the following graduate and post-graduate lectures:

### **Budapest University of Technology and Economics**

- Advanced methods for surface modification and characterization. Introduction to materials science (lecture courses by Imre Bertóti)
- Electronics and instrumentation. Electronics and measurement techniques (lecture courses by Tamás Pajkossy)
- Environmental chemistry (lecture course by János Szépvölgyi)
- Laboratory course in electronics (by Gábor Mészáros)
- Laboratory course in biochemistry (by Krisztina Lado)
- Laboratory course in plasma chemistry (by Ilona Mohai, Zoltán Károly)

The Department of Applied Chemistry and Physics of IMEC and the Department of Plastics and Rubber Technology of BUTE form a unit. The Group of Polymer Physical Chemistry is located at the University. The whole unit participates in education.

- Plastics. Processing of plastics. Polymer physics. Polymer blends and composites (lecture courses by Béla Pukánszky)
- Applications of plastics (lecture course by Erika Fekete)
- Plastics and environmental protection (lecture course by Enikő Földes)
- Laboratory courses in application of plastics and technology of plastics (Tünde Pozsgay, János Móczó, Szilvia Klébert, Erika Fekete, Enikő Földes)

### **Eötvös Loránd University, Budapest**

- Characterization of macromolecular materials by complex analytical techniques. Thermal degradation producing new materials, and eliminating wastes. (special courses by Marianne Blazsó)
- Electrocatalysis (PhD course by György Horányi)
- Polymer chemistry and technology. Fundamental macromolecular chemistry. Designed synthesis of polymers. Physical, organic and analytical principles of molecular engineering of macromolecular systems (lecture courses by Béla Iván)
- Laboratory courses in chemical technology (Gábor Erdődi, Sándor Szabó, Béla Iván, Tamás Fónagy, Péter Groh Werner)
- Laboratory course in physical chemistry (Gábor Mészáros)

### **Miskolc University**

- Advanced ceramic materials (PhD course by János Szépvölgyi)

### **Polytechnic Institute of Dunaújváros**

- Renewable sources of energy (lecture course by György Mink)

### **Semmelweis University, Budapest**

- The role of metals and metal complexes in free radical processes (PhD course by Klára Szentmihályi)

### **Veszprém University**

- Advanced ceramic materials (lecture course by János Szépvölgyi)

## 6. INDUSTRIAL R&D PROJECTS

- **AKZO-NOBEL Coatings Ltd.**  
Characterization of AKZO paint systems
- **BASF AG**  
Participation in solving different R&D problems
- **Béres Pharmaceutical Co.**  
Production of effective bone strengthening medicine
- **BorsodChem**  
Studies on PVC-based polymer blends
- **Clariant Huningue SA (Switzerland)**  
Development of new stabilizers
- **Du Pont Co.**  
Participation in solving different R&D problems
- **DUNAFERR Rt.**  
Development of monitoring systems for environmental protection  
Processing of metallurgical wastes in thermal plasmas
- **General Electric Hungary Co.**  
R&D on various technological problems, electrical characterisation of gas-discharge lamps, software development
- **General Electric Co. (USA)**  
Participation in solving different R&D problems
- **IN VITRO R&D Ltd.**  
Production of an effective substance for the therapy of anaemia
- **Kalle Nalo Hungaria**  
Investigation of multilayered polymer films
- **Middle-Tisza Agricultural Co.**  
Preparation of biofuels from waste vegetable oils
- **MAGYAR LAKK Ltd.**  
Characterization of industrial and commercial paints
- **Hungarian Combustion Technique Ltd. (MATÚZ)**  
Thermal investigations of solid fuels
- **NABI North American Bus Industries Ltd. Co.**  
Comparison of coatings developed from different paints of low solvent content
- **TVK Co.**  
Development of polyethylene and polypropylene products

## 7. RESEARCH FACILITIES

### Department of Materials Chemistry

- X-ray photoelectron spectrometers (Kratos XSAM 800, VG Escascope)
- Fast atom beam treatment facility
- RF glow discharge treatment facility
- Nanotribology tester (Nanotest 600)
- ICP-OES spectrometer (Thermo Jarell Ash Atomscan 25)
- High temperature reactors for gas-solid reactions
- RF induction plasma systems (Linn, Tekna)
- Spectrometer with CCD-3000 detector (Jobin-Yvon TRIAX 550)
- Electrochemical measuring techniques (potentiostatic and galvanostatic, stationary and transient voltametric facilities, electrode impedance spectroscopy, noise spectroscopy, harmonic analysis)
- Determination of corrosion rate in high-resistance systems
- Rapid corrosion resistance tests
- Methods for the evaluation of lifetime and paint-technological properties of coatings
- Polarography-voltametry

### Department of Polymer Chemistry and Materials Science

- Gel permeation chromatograph (Waters 510)
- Laboratory ozonizer (Yanko Industry Ozone Services)
- Tester of PVC degradation (Donaulab)

### Department of Applied Polymer Chemistry and Physics

- Laboratory rolling mill (Schwabentan)
- Single-screw extruder (Haake Rheomex S 3/4")
- Twin-screw compounder (Brabender DSK 42/7)
- Internal mixer (Brabender, 50 ml)
- Laboratory press (Fontijne SRA 100)
- Injection molding machines (Battenfeld BSKM 30/50, BA 200 CD)
- High-speed fluid mixer (Thyssen Henschel FM/A10)
- Vacuum thermoforming machine (VFP 0505 1SL)
- Thermal analyzers (Perkin Elmer DSC2, DSC7, TGA6, Mettler DSC30, TMA40, TG50)
- Thermomechanical analyzer (Polymer Labs, DMTA II)
- Fourier transform infrared spectrophotometer (Mattson Galaxy 3000)
- UV-VIS spectrophotometer (Hewlett Packard 8452A)
- Rheometry (Göttfert 2002 capillary viscometer, Göttfert MPS-D MFI tester, Brabender Rheotron rotational viscometer, Rheolab rheometer, Physica UDS 200 Universal dynamic spectrometer)
- Mechanical testing system (Zwick 1445, Fritz Heckert FPZ 10, Instron 5566)
- Impact testers (Ceast Charpy 6546, Ceast Resil 5.5, Zwick, Izod, Charpy)
- Optical instruments (Hot Stage Mettler FP 82 HT, Polaroid DMC1 digital camera, Hunterlab ColourQuest 45/0)
- High pressure liquid chromatograph (Knauer HPLC 64)
- Gas chromatograph (Perkin Elmer XLGC)
- Microtome (Reichert-Jung, Polycut)
- Contact angle goniometer (Rame-Hart 100-00-(115)-S Automated Goniometer)

- Gas permeation analyzers (Brugger GDPC, Systech 8000 oxygen permeation analyzer)

### **Department of Environmental Chemistry**

- Thermobalance-mass spectrometer system (Hiden Hall 300 PCI, Perkin-Elmer TGS-2)
- High pressure thermobalance (Hiden Hall IGA, high temperature furnace)
- Reactive thermobalance (Mettler)
- Analytical pyrolysers (CDS Pyroprobe 2000)
- Gas chromatograph-mass spectrometer (Agilent 6890 GC / 5973 MSD)
- Gas chromatograph-mass spectrometer (HP 5985B)
- Gas chromatograph (HP 5880A)
- Laser-diffraction particle size analyzer (Malvern 2600C)
- Fourier transform infrared spectrometer (Perkin Elmer 1700)
- Volumetric adsorption system for studying adsorption and chemisorption
- Differential scanning calorimeters (Setaram DSC 111, Perkin Elmer DSC 2)
- Solar still simulator
- Two-column GC with automatic dosing system (Perkin-Elmer Autosystem XL)
- Semi-preparative HPLC equipment (Waters LC-Module 1)
- HPLC/MS facility (Shimadzu LCMS 2010)
- HPLC equipment (Waters 9110)
- HPLC system (Merck Hitachi)
- Pump for column preparation (Shandon)
- Equipment for evaluating thin-layer chromatograms (Shimadzu)
- UV-VIS-NIR spectrophotometer (Jasco)

### **Laboratory of Environmental Protection**

- UV-VIS spectrophotometer (Unicam)
- ICP-OES spectrometer (Jobin-Yvon Ultrace 138)

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