INCDCP ICECHIM PRESENTATION

The mission of INCDCP-ICECHIM is to perform, at a high level of performance, research and development activities in the field of chemistry and petrochemistry, contributing to the economic competitiveness and efficiency and supporting the economic and societal sustainable development.

INCDCP- ICECHIM strategic objectives are:

(*i*) knowledge creation and contribution to the knowledge development, by providing top scientific results, competitive at international level, targeting the increase of the contribution of INCDCP-ICECHIM to the development of global knowledge stock, to an increased international scientific visibility and transfer of results in the economy and society;

(ii) contribution to an increased competitiveness of the Romanian economy through innovation

having impact upon the economic operators and the transfer of knowledge into economic sector;

(iii) Increase social quality through the development of solutions, generating direct benefits for the society, in particular through training of new researchers and contribution to a framework which enhance the quality of life (by promoting sustainable chemistry and analytical environmental services).



The Research & Development activity of INCDCP-ICECHIM was focused in the last years on three interrelated directions:

- Development of innovative technologies for bio-resources valorization

- Development of new nanomaterials and biomaterials for a wide range of applications

- Sustainable management of the resources and environment

Starting from these three main directions and from the expertise of our researchers, the interdisciplinary teams of ICECHIM approached research topics such as: bioproducts and biofuels preparation; biotechnology and bioanalysis; synthesis of nanostructured polymers, bionanocomposites and multifunctional materials; waste remediation; nanomedicine; green energy (Fig. 1).

Fig. 1. The diagram of the main R&D direction of ICECHIM

These topics correspond with the thematic priorities of the European and National R&D Programs, including those related to the transectorial domain of bio-economy.

INCDCP-ICECHIM has applied for numerous R&D projects in all national and international calls where it was eligible and a good rate of success was recorded. INCDCP-ICECHIM was/is *partner in several FP6/FP7 projects*: FP6 no. ACC-SSA-2-2004-017494 "Enhancement the quality participation at FP6 projects in the polymer nanomaterials field – ENPONA" (2005 – 2008) (750,000 Euro);FP6 FOOD-CT-2006-036296 "Development of high throughput approaches to optimize the nutritional value of crops and crop-based foods (DEVELONUTRI)" (2007 – 2011) (210,000 Euro); FP7 213436/2008 "Nanostructured Toughened Hybrid Nanocomposites for High Performance Applications (NANOTOUGH)" (2008 – 2011) (603,794 Euro); FP7 –NMP3-LA-2008-213277 "High Aspect Ratio Carbon based Nanocomposites (HARCANA)" (2008 -2012) (550,000 Euro);FP7 – NMP-2012-2-GA 314744. "The Electric Vehicle revOLUTION enabled by advanced materials highly hybridized into lightweight components for easy integration and dismantling providing a reduced life cycle cost logic (EVOLUTION)". *Other European Projects* on which the Institute was involved are: LIFE 10 ENV/RO/734 "Carbon dioxide mitigation from greenhouse gases in algal photosynthetic systems" (2011 – 2014) (1,038,286 Euro); BS ERANET 7-045/2011 "Innovative materials for waste

water purification systems to be installed in tourist and other small polluting objectives on the Black Sea coast (IMAWATCO BS ERANET)" (2011 – 2013) (80,000 Euro); CROSS BORDER project Romania – Bulgaria "Danube WATER integrated management. WATER" (2011 – 2013) (178,832 Euro)



Fig. 2. Percentage of the projects value cofinanced by industrial partners

Regarding the national projects, one of our priorities of INCDCP-ICECHIM was and is the collaboration with the industry by developing technologies, products and services adapted to its requirements. In Fig. 2 it can be observed that 28 % from the total value of national projects are co-financed by partners from industry.

INCDCP-ICECHIM used the recently introduced finacing instrument of innovouchers (PN2 P5 Innovation) to transfer results of R&D activity to economy. In 2012 the Institute successfully implemented 15 innovouchers.

Five representative projects, which are representatives for the coherent, structured research activity, leading to a fulfillment of the three strategic objectives of INCDP-ICECHIM will presented below.

PNCDI II – P2 CAPACITIES project PM/CP 1/2008 "Development of the Bioresources Department by R&D infrastructure modernizing – BIORES" (2008 – 2011). It was developed and modernized the existing infrastructure of the Bioresources Department in order to establish a modern department with four laboratories: extraction, biofuels, bioproducts, tests and measurements. Major equipments were purchased and commisioned: Photobioreactor (180 L volume), Continuous Flow Tubular Reactor, Fermenter, Supercritical Fluid Extraction System, GS-MS/MS Triple Quad, LC-MS/TOF. Infrastructure created by project PM/CP 1/2008 BIORES allows the conception, development, execution and conclusion of projects related to sustainable use of renewable natural resources for the production of "green chemicals" and further development of bio-economy - fig. 3.



Fig. 3. "Green chemicals" resulted from R&D projects related to the infrastructure developed through the Capacity project PM/CP 1/2008 BIORES.

One example of such project is PN2 P4 22-085 "Mitigation of greenhouse gas emissions by means of useful output microalgal systems". This project is representative for production of results which are relevant for society (green field development of algal practical photosynthesis is one of the solutions for "green chemistry" / biorefinery activity which is not competing with food and feed use of agricultural production). The results of this project are:

1. Laboratory technology for cultivation, harvesting and extraction of useful components from algae *Scenedesmus opoliensis 141* grown in photobioreactors;

2. Laboratory technology for *Haematococcus pluvialis* growth with the new elements mean additional growth with vitamin B12 BBM, modification of lighting conditions (increased irradiance to 630 mmol \cdot m2 \cdot s-1, under continuous light) associated with increasing environmental salinity growth (4 g / 1 NaCl) leads to improved growth performance of astaxanthin in Haematococcus biomass of 10 times (4-5,7 mg/l).

3. Mathematical modelling of growth and cultivation of algae in the laboratory stage to determine the amount of biomass results and the amount of oil extracted from the algal biomass. By implementing the model for algal growing, according to SuperPro Designer software, simulation was made in accordance with the technological process proposed. After implementing the material balance it follows that for a full cycle of 1 month for growth *Scenedesmus* algae, are obtained 48 g dry biomass by introducing 300 g inoculum, through processing (solvent extraction and separation by distillation), we obtain 18 g algal oil.

4. Development of a lab-scale technology for growing algae, algal biomass harvesting, extraction of lipids fraction and characterization of fatty acid spectrum of algal oil was achieved in pilot-plant experiments. The proportion of over 87% fatty acids C16, C18 and C18: 1 from lipids extracted from algal biomass is convenient conversion into biodiesel (by transesterification), renewable diesel (by hydrolysis of fats, fatty acids separation and decarboxylation and hydrogenation mixture of hydrocarbons derived) and aviation fuels - by hydroprocessing fatty acids to produce synthetic paraffinic kerosene.

The above presented results were included in 2 patents application and 3 scientific articles. The 2 patents application are related to the first Romanian patent application to algal strains with high efficiency in CO2 mitigation by practical photosynthesis. Project PN2 P4 22-085 is representative for INCDCP – ICECHIM capacity to produce results relevant to society. Project results exploitation and further developments will be achieved through a technological demonstration project co-funded by Life+ Programme, LIFE 10ENV/RO/000734 "Carbon dioxide mitigation from greenhouse gases in algal photosynthetic systems". Budget of the project is: total amount - 1,099,548 EUR; EU Co-funding: 45.05%; Duration - Start: 22/09/2011; End - 31/08/2014.

Another infrastructure development porject was the project entitled 'BIOTECHNOLOGY AND BIOANALYSIS LABORATORY (B&B LAB)', Project 49 / CP / I / 2007 entitled 'BIOTECHNOLOGY AND BIOANALYSIS LABORATORY (B&B LAB)', value 1.468.303 lei. which aimed the modernization of the Biotechnology Lab facilities from INCDCP-ICECHIM and endowment it with modern equipment research and development, competitive at the European level. Modernizations made have been considered both continuing the R&D activities in the biotechnology in which the laboratory was highly experienced, and also increasing of the R&D in the bioanalysis field.

The specific objectives of the project consisted of the total rebuilding of the Microbiology Lab according to the Good Practices in Laboratory (GPL) rules by equipping it with all the necessary installations and facilities (air treatment, conditioning, Biohazard II microbiological hoods, class C clean room), corresponding subdivision of the lab spaces in order to assure the correct flows of personnel, materials and samples, etc. Also, it were modernized the Lab of active principles separation, the Lab of Optical Bioanalysis and the Biosensors Lab. The most important equipments bought were: System for microbial taxonomic identification (BIOLOG Inc., SUA), Bioreactor KLF 2000 with insitu sterilization system (Bioengineering, Switzerland), Amperometric detector model Epsilon 5 (BioAnalytical Systems Inc., SUA), HPLC system with DAD and FL detectors Surveyor Plus (Thermo Scientific, USA).

The project results and effects were: (*i*) Improving the quality and efficiency of the RD activities performed in the B&B LAB by offering research and high-level scientific expertise to SMEs

and other beneficiaries. For example, three Innovation Vouchers were accomplished for SMEs (121CI/14.08.2012; 84CI/29.06.2012; 100CI/05.07.2012) which led to 3 EPO patent application and 3 OSIM patent applications. (ii) Creation of local / regional / national partnerships with universities, R&D institutes, SMEs and participation with joint proposals to national calls for research projects. In frame of the projects PNCDI II 61-045, 71-079 and 32-115 the modernized infrastructure of the Microbiology lab has facilitated the development of new biotechnological approaches for fungal infectious deseas biocontrol at plant, of new methods for biodegradability evaluation and of innovative biodegradation technologies of the plastic waste. In the Biosensors Lab during the project PNCDI II 61-030 were developed flow immunoassy systems and an SPR immunosensor for mycotoxins determination. The new infrastructure of B&B LAB was a good basis for preparation of numerous joint proposals with SMEs in frame of the 2011 and 2013 national calls for partnership projects; (iii) A high level laboratory for the young researchers, undergraduated and postgraduated students. Four postgraduated students were employed in the B&B LAB, where thei have prepared / are preparing their Ph.D. Thesis. Also, stages for practical training of students were hosted according to the agreements with national (UPB, UB, USAMV Bucuresti) and international (France, UK) universities. In B&B LAB were ellaborated 3 licence theses, 5 disertation theses and 1 Ph.D. thesis; (iv) An increased international visibility of the researchers and of the research results of B&B LAB by publication in ISI journals, works presentation in frame of prestigeous international conference, invitation of the research to act as EU evaluator experts and journal reviewers; (v) Invitation of the B&B LAB to join to international R&D partnerships for European calls (ERANET, EUREKA, Leonardo da Vinci). In november 2013 will start a Leonardo da Vinci project dedicated to biosciences. value 1.468.303 lei

The project "New innovative methods of fotochemotherapy with nanostructured photosensitizers– from synthesis to clinical trial" supported in conception, development, execution and conclusion by the modernized infrastructure of INCDCP – ICECHIM deals with the implementation procedure of therapy in the pathology of human dermatological area, through the establishment of a network of research institutions, universities in different regions of the country and from hospital units, owning scientific and practical experience necessary. From this project resulted 1 patent, 23 ISI publications; 4 Ph.D. thesis, 79 conference papers, 1 COST Action COST D39.

The High Aspect Ratio Carbon-based Nanocomposites" (acronym HARCANA) project received funding from the European Community's 7th Framework Programme for Research and Technological Development under the Grant Agreement number NMP3-LA-2008-213277. The project was developed during 48 months (July 2008 to June 2012). The total budget amount was 7.4 Millions Euro with a European Community financial contribution of 5.4 Millions Euro (or 73 % of the planned total budget).

The consortium of the project consisted of 7 academic (including INCDCP-ICECHIM) and 5 industrial partners from 5 countries under the coordination of Helmholtz-Zentrum Geesthacht. The project was spanning a bridge between research and development of the nanocomposites on one side to the demonstration of the developed results on the other side.

This project under industrial leadership was therefore aimed at mastering, at the nanometric and mesoscale level, the spatial organization of carbon-based nanoparticles (CNP) with various surface functionalities, sizes and shapes having large aspect ratios in bulk, foamed and thin film (membranes) polymers by using industrially viable processes. More precisely, the aim of this proposal consisted in generating polymerbased nanocomposites with a percolating nanoparticle structure that reinforced the material and imparted it with improved electrical and thermal conductivity at a minimum of nanoparticle loading.

High aspect ratio carbon-based nanoparticles (nanotubes (CNT), nanofibres (CNF), and nanosheets or exfoliated graphite (CNS)) were introduced into bulk polymers, into polymeric foams and into membranes in order to improve and modify the properties of these families of materials, allowing them to reach new markets.

In order to control the CNP organization within the polymer matrix, a large set of techniques were used. They range from synthetic approaches ("grafting from", "grafting to", "grafting through", emulsion polymerization) to (reactive) melt or solution blending processes, and to preparation in supercritical CO_2 . Finally were generated new classes of engineering materials for various applications

like EMI shielding, antistatic packaging materials and membranes, as well as biocompatible materials with brain cells that could be used to prepare scaffolds for tissue engineering in the future.

Project FP7 213436/2008 "Nanostructured Toughened Hybrid Nanocomposites for High Performance Applications (NANOTOUGH)" (2008 – 2011) was a Large Scale Integrating Collaborative Project, with the following partners: Aalborg University AAU Denmark – Coordinator, INCDCP-ICECHIM Romania, University of Hamburg UHAM Germany, Technical University of Denmark DTU Denmark, University of Genova UGDIE Italy, Association pour la Recherche et le Développement des Méthodes et Processus Industriels ARMINESCEMEF France, Centro Ricerche Fiat CRF Italy, The Danish Technological Institute DTI Denmark, Laviosa Chimica Mineraria LCM Italy, FPK S.A. FPK Spain, Aviospace AVS Italy.

The aim of this project was to obtain new nanocomposite materials based on polypropylene (PP) and nanolayered silicates, characterized by a good toughness - stiffness balance which would replace materials currently used in the manufacture of automotive parts and in other high performance applications. The development of new materials was motivated by the growing demand of the industry for functional materials, cheaper and lighter, with tailored properties for applications and which are not currently available on the market.

A main scientific objective of the project was the deeper understanding of interfacial properties in polyolefin nanocomposites in order to solve the current problems that hinder the commercialization of these materials. Medium-term objective was to obtain materials with superior properties for specific applications and for new products with affordable price.

In this project, ICECHIM was responsible of work package no. 1 (WP1 Leader). The main task of ICECHIM was to obtain PP nanocomposites using new compatibility agents developed on the basis of functionalized or non-functionalized block copolymers and new dispersing agents based on PET waste, in order to allow uniform dispersion of nanofiller in PP matrix, strong interaction between the components and outstanding improvement of PP properties.

The main results of INCDCP-ICECHIM activity were functionalized and non-functionalized block copolymers and oligoterephthalates (obtained by PET waste valorisation), developed and evalauted for their efficiency as compatibilizer agents in nanocomposites based on PP. Both types of compatibilizers are subject to patent applications filed with the Romanian Patent Office. Several milestones: the degree of interaction between the components was evaluated, masterbatches were prepared being established the ratio between the components, their mixing order and the technological processing parameters.

As a result of INCDCP-ICECHIM's work, new high performance materials have been developed, characterized by an increased toughness (15 times higher as compared to the reference), an improvement of creep resistance and of the resistance at high temperature, increased resistance to repeated loading cycles and better barrier properties. Moreover these new developed materials showed good results when tested by end users partners of the project. Two industrial samples of masterbatch (~ 300 kg) were made by ICECHIM and sent to the end user (FPK) to manufacture auto parts and for material and parts characterization. The new materials were tested in combination with glass fibre to achieve car bumpers leading to weight reduction of the part by 30% in comparison with current solutions. The materials developed by ICECHIM were also validated for spare wheel well, whose behaviour to impact improved by 50%. One advantage of these materials is the easy recycling at end of life of the parts since they do not contain metal inserts.

Following the work of ICECHIM new materials with superior performance for automotive industry, aerospace, packaging and tires have been developed. All these results determined the selection of this project by the EC as a "Success Story". Also, ICECHIM was asked to participate as a partner in a new FP7 project, on-going since November 2012, The Electric Vehicle revOLUTION enabled by advanced materials highly hybridized into lightweight components for easy integration and dismantling providing a reduced life cycle cost logic (EVOLUTION), GA 314744.