

# INSTITUTE OF CLEAN TECHNOLOGIES FOR MINING AND UTILIZATION OF RAW MATERIALS FOR ENERGY USE – A NEW POTENTIAL OF RESEARCH IN OSTRAVA

## INSTITUT ČISTÝCH TECHNOLOGIÍ TĚŽBY A UŽITÍ ENERGETICKÝCH SUROVIN – NOVÝ POTENCIÁL VĚDY A VÝZKUMU V OSTRAVĚ

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### Abstract

A year ago, in Ostrava, one of the major RDI (Research and Development for Innovation) projects was initiated in the Moravian-Silesian Region, called the *Institute of Clean Technologies for Mining and Utilization of Raw Materials for Energy Use*. During the first year, many of the top and often unique research laboratories and workplaces were built with a budget of over CZK200m, such as “Workplace of Electron Microprobe”, „Workplace of Tomographic Methods“, “Workplace of Hydrochemistry and Hydrobiology”, “Workplace of Thermal, Hydraulic and Mechanical (THM) Processes in Rocks”, “Workplace of Water Jet”, “Isotope and GC-TOF Laboratory” and many others. This laid the basis for various research programmes with truly extraordinary extent and impact not only on the Czech industry but also economy of other EU member states.

### Abstrakt

V Ostravě byl před rokem zahájen jeden z významných projektů VaVpI (Věda a výzkum pro inovace) v Moravskoslezském kraji s názvem *Institut čistých technologií těžby a užití energetických surovin*. V průběhu prvního roku byla s rozpočtem více než 200 miliónů korun vybudována řada špičkových a mnohdy i unikátních výzkumných laboratoří a pracovišť, jakými jsou „Pracoviště elektronové mikrosondy“, „Pracoviště tomografických metod“, „Pracoviště hydrochemie a hydrobiologie“, „Pracoviště tepelných, hydraulických a mechanických (THM) procesů v horninách“, „Pracoviště vodního paprsku“, „Izotopová a GC-TOF laboratoř a řada dalších. Tím byl položen základ k řešení výzkumných programů se skutečně mimořádným rozsahem a dopadem nejen na český průmysl, ale i ekonomiku ostatních států EU.

**Key words:** energy resources, clean technologies, RDI.

## 1 INTRODUCTION

A year ago, in Ostrava, one of the major RDI (Research and Development for Innovation) projects was initiated in the Moravian-Silesian Region, called the *Institute of Clean Technologies for Mining and Utilization of Raw Materials for Energy Use* (hereinafter referred to as ICT).

This project is financed by the EU Structural Funds –Research and Development for Innovations Operational Programme with a total budget of nearly CZK 300 million (excluding VAT).

## 2 BUILDING UP ICT WORKPLACES

A bearer of the project and simultaneously its leader is the Faculty of Mining and Geology of the VSB-Technical University of Ostrava whose newly built workplaces represent two-thirds of the whole ICT budget. However, colleagues from other faculties of the VSB-Technical University of Ostrava were invited, namely from the Faculty of Mechanical Engineering, Faculty of Civil Engineering and the Faculty of Safety Engineering to address research programmes.

A project of this importance and scope neither can do, of course, without close cooperation with other leading scientific institutions outside the university. Such key partner of the HGF VSB-TU Ostrava in the Institute of Clean Technologies for Mining and Utilization of Raw Materials for Energy Use is the Institute of Geonics of the Academy of Sciences of the Czech Republic v.v.i. with a high-quality professional base and a number of reputable research teams. The intention is also to address and attract experts from abroad to contribute to the excellence of expected results.

As for its implementation, the project is divided into two stages. The first stage (so-called Start-up) started on 1st January 2011 and shall be finished on 31st December 2014. In this period, most of the costs associated with the construction, start and overall operation of workplaces and laboratories are financed by the EU Structural Funds. Further viability of the entire project will critically depend on the results of this stage. The second project stage shall be the sustainability phase, which will continue in another five years - i.e. until the year 2019. During this period, the project will not be financed by the EU any longer and its activities must be covered by scientific projects and contractual cooperation with industry.

The first essential part of implementing this type of research projects is to build suitable facilities of laboratories and workplaces and to provide them with scientific instruments and equipment. Today, after the first year of ICT operation, we can say that this stage of the ICT project has been successfully completed. More than 380 m<sup>2</sup> of areas of laboratories have been substantially renovated and modernized and more than 60 public contracts for the acquisition of high-end devices (Fig. 1) and systems have been arranged.

The Institute of Clean Technologies, as results from its whole name, should help the Czech Republic with finding new ways for obtaining energy resources that would have minimal impact on the environment. Such exploitation must be, on one hand, the most efficient from an economic point of view, while environmentally friendly on the other hand. It should help as far as possible to ensure the maximum possible raw material self-sufficiency of the Czech Republic. The project consists not only in the work on untapped raw materials, but also finding new approaches even to so-called industrial waste. In the Ostrava Region, for example, these are heaps of coal-mining waste. The materials, from which they are formed, should in future be regarded as secondary raw materials with other potential use.

## 2.1 ICT research programme

The project consists of two basic research programmes. The task of the research programme “Poly-phase geological environment” is to gain knowledge about the physical, chemical, isotopic, structural and mechanical properties of environmental components using the most modern devices to significantly increase the level of knowledge and the possibility of its generalization for given geological conditions by means of mathematical modelling. Partial scientific objectives of the research programme then include such research spheres as “Definition of violations of geomaterials based on their internal structure, mode of loading and physical conditions”, “Use of raw materials and wastes to produce modified clays and geopolymers with applications in construction and BAT technologies for the human living environment” and “Identification of gas origin in rock mass and its usefulness for increasing exploitability of deposits”. This information should then be an essential prerequisite and cornerstone for the design of environmentally friendly technologies in exploiting mineral resources.

The research programme “Environmentally friendly technologies” shall solve the problems of utilizing by-products for the introduction of non-waste technology in extracting mineral resources and creating the conditions to minimize safety risks based on the knowledge of causal processes. While minimizing safety risks, the environmentally friendly mining of mineral resources represents under actual economic conditions a complex process that requires addressing the entire spectrum of specific issues starting from the application of new knowledge of the properties and behaviour of the rock environment through the development of new mining technologies and treatment of energy resources to their safety aspects and environmental impacts. The following are then defined as partial research objectives: “Ensuring technology for treatment of mine water in compliance with the Water Framework Directive 2000/60/EC”, “Development of technology for the preparation of mineral precursors and carriers of nanoparticles via high-speed water jet disintegration”, “Technology of mining protective pillars in coal mines with minimum effects on surface deformation and determination of operating parameters of mining techniques” and “Security aspects of environmentally friendly technologies associated with the mining of mineral resources in terms of explosiveness, flammability, spontaneous combustion and air conditioning of mines”.

The results of the research activities, as for the technical as well as personnel aspects, of such top teams should be in practice utilized not only by important companies in the region, such as OKD, a.s.; Diamo, state enterprise; Severočeské doly, a.s.; Litvínovská uhelná a.s.; RWE; MND a.s., but by many other energy mining companies as well.

## 2.2 ICT laboratories and workplaces

The success of implementation of the ICT project is heavily dependent on two factors, both excellently equipped workplaces and individual teams of experts. Over CZK 200 million has been invested into the technical equipment of the laboratories, most of which are now ready for intensive research work. A personal factor, thus the creating of truly productive research teams based on both already mature real scientists (so-called senior researchers) and younger and less experienced junior researchers and “scientific rookies” (doctoral students),

was to the greatest extent covered from own sources of the VSB-TU Ostrava and the Institute of Geonics AS CR, partly then by recruiting highly skilled workers from nonschool workplaces.

From the truly key laboratories we can mention in particular “Workplace of Electron Microprobe”, “Workplace of Tomographic Methods”, “Workplace of Hydrochemistry and Hydrobiology”, “Workplace of Thermal, Hydraulic and Mechanical (THM) Processes in Rocks”, “Workplace of Water Jet”, “Isotope and GC-TOF Laboratory”, “Laboratory of Instrumental Neutron Activation Analysis (NAA)”, “Workplace of X-ray Diffraction”, “Laboratory of Raman Spectroscopy”, “Laboratory of TGA-MS”, “Laboratory of Physical Methods”, “Laboratory of Microcalorimetry and Electrochemistry”, “Laboratory of Thermal Analysis and Rheology of Building Materials”, “Laboratory of Stimulation of Boreholes and Hydrocarbon Deposits” and “Workplace of Safety Analysis”.

The Workplace of Electron Microprobe is equipped with the FEI Quanta 650 FEG cutting-edge scanning electron microscope (FEI, USA) provided with WDX, EDX and EBSD analysers (Fig. 1). It is a system fitted with analytical tools for detailed chemical, crystallographic and morphological characteristics of various materials. The microscope alone allows working in an extremely large range of zoom (6x to 2 000 000x) even at very low and variable vacuum (up to 4000 Pa in the ESEM mode) and without surface finish of samples by plating. The microscope as one of these analytical tools is equipped with energy- and wave-dispersive and cathodoluminescence analyser including a spectrometer and an apparatus for diffraction of backscattered electrons [1]. An integral accessory is a cooling table for the analysis of biological samples and an apparatus for ion polishing and cutting.



**Fig. 1** Laboratory of Electron Microprobe

The Workplace of Tomographic Methods can boast of the first system of this type and performance in our country. It consists of two X-ray tomography systems – XTH 450 2D/3D and XTH 225 ST (NIKON Metrology NV, Japan). The first one has a maximum acceleration voltage and power output of X-ray source 450kV/640W with a focal spot size of 80µm/300µm (for 200W/600W). The maximum weight, diameter and height of scanned objects can reach up to 100 kg/approx. 0.6 m/0.8 m with a theoretically radiable thickness of analysed materials of 395 kg/m<sup>2</sup>. The performance of the other device is slightly smaller, but it can analyze samples with a weight of up to 50 kg.

The Isotope (Fig. 2) and GC-TOF Laboratory is equipped with systems for determining isotope ratios and the study of products of pyrolytic cleavage. The new and unique sector Delta V Advantage Spectrometer (IRMS) (Thermo Fisher Scientific, Inc., USA) in conjunction with the Trace GC Ultra Gas Chromatograph (Thermo Fisher Scientific, Inc., USA) is characterized by excellent parameters for the measurement of isotope ratios <sup>13</sup>C/<sup>12</sup>C, <sup>15</sup>N/<sup>14</sup>N, <sup>18</sup>O/<sup>16</sup>O, <sup>34</sup>S/<sup>32</sup>S. The H/D ratio measurement in a dynamic mode excels in high

sensitivity, monolithic analyser with fixed settings of all components of ion optics [2, 3]. The pyrolytic system Trace GC Ultra Gas Chromatograph (Thermo Fisher, USA) with the PyroProbe 5000 device (CDS Analytical, USA) and with TOF Mass Spectrometer (Time-Of-Flight) (ALMSCO International, United Kingdom) will allow both monitoring the behaviour of chemical compounds in a selected temperature regime, and their targeted degradation before the very analysis.



**Fig. 2** Isotope Laboratory

The Workplace of Thermal, Hydraulic and Mechanical (THM) Processes in Rocks boasts of a large complex of instrumentation and software, such as MTS Servohydraulic Test System and 656.06 Triaxial Cell for tests of strength and deformation properties of rocks, CSM Instruments Micro-Hardness Tester (USA), SETSYS TG-DTA/DSC 24 Thermal Analyser with a mass spectrometer (Setaram Instrumentation, Canada), NICOLET 6700 FT-IR Spectrometer with the NICOLET NXR FT-Raman module (Thermo Scientific, USA), NICOLET iN10 FT-IR Microscope (Thermo Scientific, USA), NIKON Eclipse 80i (Nikon, Japan) and OLYMPUS BX 50 (Olympus, Japan) Fluorescence Microscopes, OLYMPUS LEXT OLS 3100 Confocal Laser Scanning Microscope (Olympus, Japan) including the image processing and analysis software – NIS Elements and MATLAB Image Processing Toolbox.

The Workplace of Water Jet involves the ABB IRB 6640-180//2.55 Master Robot (Fig. 3) for handling of a water jet cutting head with high pressure pumps – Hammelmann GDP 253 (maximum working pressure of 160 MPa, maximum flow rate of 67 l/min) and PTV75-60 with two pressure multipliers (a working pressure of 40 to 415 MPa, maximum flow rate of 7.8 l/min at 415 MPa). This machine is completed with a system for visualization and measurement of flow velocity fields (2x PIV camera, Imager Pro X 2M CCD with a double pulse laser system with optics to create a light sheet NL 135-15 PIV and HighSpeedStar 3G CMOS high-speed camera). Part of the workplace is a computing system for flow modelling equipped with the ANSYS CFD software.

With regard to its mission in the area of research of explosion risk analysis, the Workplace of Safety Analyses is equipped with a device (Hartmann Tube) for determining the minimum ignition energy (OZM Research, CR), CLASIC 5506 SZ ovens for self-heating (Classic CZ, CR) for determining conditions of the self-heating of solids and GX 51 Inverted Metallographic Microscope (Olympus, Japan) for the identification of changes in materials during combustion and explosion with an automatic analysis of records, and highly prized FLACS advanced modelling software (GexCon AS, Norway) [4,5].

The Laboratory of thermogravimetric and mass analysis (TGA-MS) boasts of a unique system, which is the first of its kind in Europe and the twelfth in the world, of a high-pressure thermal analyser with the TGA-MS-HP50U Mass Spectrometer (TA Instruments, USA). The mass spectrometer with a range of 1-300 amu at the output analyzes gaseous products leaving the reactor.



**Fig. 3** Robot for Water Jet Cutting

By its concept, the Workplace of X-ray Diffraction is focused on qualitative and quantitative analysis of mineral-phase composition of samples of various kinds. Using the Bruker Advance D8 Powder Diffractometer (Bruker, USA) (Fig. 4) equipped with the LynxEye linear semiconductor detector and energy dispersive SOL-XE Detector, it is possible to analyse the composition and to identify almost any material (rock samples, waste, chemicals, pharmaceutical ingredients, etc.).

The Laboratory of Raman Spectroscopy is equipped with the multifunctional 3D Scanning Confocal Microscope with the Nanofinder S Spectrometer (Tokyo Instruments, Japan). The system uses simultaneously the 2D optical microscopy with high resolution, 3D laser, 3D luminescent and 3D Raman microscopy, which enables a multi-functional micro-structural three-dimensional analysis with a high spatial and spectral resolution.

The instrumentation of the Laboratory of Microcalorimetry and Electrochemistry consists of a high-end system to measure the Zeta-potential, dependence of the Zeta-potential on the environment stability and determination of isoelectric points of colloidal suspensions Zetasizer NANO ZS (Malvern Instruments Ltd., United Kingdom), ED-Z electro-membrane separation unit (MEGA, CR) using electro-dialyse units fitted with replaceable polymer membranes and the TAM III adiabatic simultaneous micro-calorimeter of a new generation offering maximum sensitivity, flexibility of measurement and performance (TA Instruments, USA).

The Laboratory of thermal analysis and rheology of building materials is equipped with the AR G2 Rheometer (rotational viscometer), TAM Air eight-channel isothermal micro-calorimeter (TA Instruments, USA) with the SDT Q600 high thermal module (TA Instruments, USA), allowing simultaneous measurement of mass changes and heat flow up to 1500°C. An essential part of the laboratory is the C-40/1000-S Climatic Chamber used to study the properties of building materials.



**Fig. 4** X-ray Diffractometer

### 3 CONCLUSIONS

Within one year, 15 key workplaces of the Institute of Clean Technologies for Mining and Utilization of Raw Materials for Energy Use were built. These are formed not only by research devices and systems at the global level, but also by highly erudite and experienced personnel having all the prerequisites to achieve and meet individual objectives of the two research programmes.

#### LIST OF ABBREVIATIONS

|        |  |
|--------|--|
| BAT    | - Best Available Technologies                    |
| CFD    | - Computational Fluid Dynamics                   |
| EBSD   | - Electron Backscatter Diffraction               |
| EDX    | - Energy-Dispersive X-ray spectroscopy           |
| FT-IR  | - Fourier Transform – Infrared                   |
| GC-TOF | - Gas Chromatography with Time-off-Flight        |
| H/D    | - Hydrogen/Deuterium                             |
| IRMS   | - Isotopic Ratio Mass Spectrometry               |
| NAA    | - Neutron-Activation Analysis                    |
| RTG    | - X-ray  |
| TGA-MS | - Thermogravimetric Analysis – Mass Spectrometry |
| THM    | - Thermal, hydraulic and mechanical              |
| RDI    | - Research and Development for Innovations       |
| WDX    | - Wavelength Dispersive X-ray Spectroscopy       |

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## RESUMÉ

V Ostravě byl před rokem zahájen jeden z významných projektů VaVpI (Věda a výzkum pro inovace) v Moravskoslezském kraji s názvem Institut čistých technologií těžby a užití energetických surovin. Klíčovým partnerem Hornicko-geologické fakulty VŠB-TU Ostrava je v ICT ostravský Ústav geoniky Akademie věd České republiky se svou skutečně kvalitní odbornou základnou a řadou uznávaných vědeckých týmů. Na realizaci výzkumných programů se však budou podílet i výzkumní pracovníci z dalších fakult VŠB-TUO, konkrétně z Fakulty strojní, Fakulty stavební a Fakulty bezpečnostního inženýrství. Záměrem je rovněž oslovit i a přitáhnout i odborníky ze zahraničí, kteří by mohli přispět ke špičkové kvalitě očekávaných výsledků.

Realizačně je projekt členěn do dvou bloků. V prvním (do roku 2014) je většina nákladů spojená s vybudováním, rozjezdem a celkovým provozem pracovišť a laboratoří hrazena ze Strukturálních fondů EU. Druhou časovou etapu projektu tvoří tzv. fáze udržitelnosti, která potrvá po dobu dalších pěti let – tedy až do roku 2019. Během tohoto období již projekt ICT nebude financován z prostředků EU a na svoji činnost si musí zajistit finanční prostředky komerčně poskytovanými službami.

První podstatnou částí realizace bylo vybudovat vhodné prostory laboratoří a pracovišť a vybavit je vědeckými přístroji a technikou. Dnes, po roce velice intenzivní a někdy vyčerpávající práce, lze konstatovat, že tato etapa projektu ICT byla úspěšně naplněna. Bylo zrekonstruováno a podstatně zmodernizováno více než 380m<sup>2</sup> ploch laboratoří a proběhlo více než 60 veřejných zakázek na pořízení špičkových (a v řadě případů i unikátních) přístrojů (obrázek 2) a systémů, z nichž dokonce některé byly instalovány jako vůbec první v Evropě.

Institut čistých technologií, jak už ostatně vyplývá i z jeho celého názvu, by měl České republice pomoci s hledáním nových způsobů získávání energetických surovin a zdrojů, které by měly minimální dopady na životní prostředí. Úkolem projektu je nejen práce na dosud nevytěžených surovinách, ale i nalézání nových přístupů a to i k takzvaným průmyslovým odpadům

Projekt je tvořen dvěma základními výzkumnými programy. Zadáním výzkumného programu „Vícefázové horninové prostředí“ je získat poznatky o fyzikálních, chemických, izotopových, strukturních a mechanických vlastnostech složek prostředí pomocí moderní instrumentální techniky, která zásadním způsobem zvyšuje úroveň poznání a možnosti jejich zobecnění pro dané geologické podmínky pomocí matematického modelování. Výzkumný program „Environmentálně šetrné technologie“ se bude zabývat problematikou využití vedlejších produktů pro zavedení bezodpadových technologií při těžbě nerostných surovin a vytvoření podmínek pro minimalizaci bezpečnostních rizik na základě poznání příčinných procesů.

Úspěch realizace celého projektu ICT je silně závislý na dvou faktorech – unikátních a špičkově vybavených pracovištích a skvěle kvalifikovaných odborných týmech. Do technického vybavení laboratoří bylo investováno přes více než 200 miliónů korun a většina z nich je již nyní připravena k náročné vědecké práci. Bylo vybudováno 15 klíčových laboratoří a pracovišť: „Pracoviště elektronové mikrosondy“, „Pracoviště tomografických metod (RTG-CT)“, „Pracoviště hydrochemie a hydrobiologie“, „Pracoviště tepelných, hydraulických a mechanických (THM) procesů v horninách“, „Pracoviště vodního paprsku“, „Izotopová a GC-TOF laboratoř“ (obrázek 3), „Laboratoř instrumentální neutronové aktivační analýzy (INA)“, „Pracoviště RTG difrakce“, „Laboratoř Ramanovské spektroskopie“, „Laboratoř TGA-MS“, „Laboratoř fyzikálních metod“, „Laboratoř mikrokcalorimetrie a elektrochemie“, „Laboratoř termické analýzy a reologie stavebních hmot“, „Laboratoř stimulace vrtů a ložisek uhlovodíků“ a „Pracoviště bezpečnostních analýz“.