

## Joint chemical laboratory for the service of bioeconomy in the Slovak-Hungarian border region

**Project number: SKHU/1902/4.1/001**

The project is implemented with the support of the European Regional Development Fund (ERDF) and the National Office for Research, Development and Innovation within the framework of the Interreg V-A Slovakia-Hungary Cooperation Programme.

### **Lead Beneficiary**

Slovenská technická univerzita v Bratislave (STU) – Slovak University of Technology in Bratislava  
Faculty of Chemical and Food Technology - Department of Organic Technology, Catalysis and Petroleum  
Chemistry

Radlinského 9

812 37 Bratislava (Slovak Republic), [www.fchpt.stuba.sk](http://www.fchpt.stuba.sk)

### **Project partner**

Eötvös Loránd Research Network, Research Centre for Natural Sciences

Institute of Materials and Environmental Chemistry (AKI)

Magyar Tudósok Körútja 2.

1117 Budapest (Hungary), <http://www.ttk.hu/>

**Project duration:** 24 months

**Start date of project:** 1st October 2020

**End date of project:** original September 30, 2022, extended until November 30, 2022

### **Budget of the project**

**EU contribution from ERDF:** 338 367,59 EUR

**ERDF contribution for STU:** 173 958,89 EUR

**National contribution for STU:** 20 465,75 EUR

**Own contribution (DOTCPCH):** 10 232,88 EUR

**The aim of the project** is to improve cooperation between institutions and promote joint cross-border activities to support the regional bioeconomy. Most of the grant will be used by the partners to develop equipment for a joint Slovak-Hungarian laboratory for the development of catalysed chemical processes for biomass utilisation.

## **Project summary**

With ERDF support proposing partner institutions established a joint chemical laboratory in 2012 to study the chemical utilization of waste biomass appearing in the Slovak –Hungarian border region. Ever since partners continued co-operation using their own resources and taking advantage of the joint laboratory. Recently the support gained from the small project fund of the Interreg Programme helped us to focus on the circular economy in the regional agribusiness. The present proposal concerns bioeconomy that involves every cross-border activity where agricultural businesses and related industrial sectors co-operate to process waste and side-product biomaterials into marketable products, such as, energy, fuels, and chemicals. The general objective of the project is to promote spreading of bioeconomic activity by providing chemical information to stakeholders about the kinds of renewable waste and side-product biomaterials in the border region of Slovakia and Hungary. The information includes the structural and chemical properties of the biomaterials and the possible methods of their processing to get products of increased value. Specific objectives of the project is to broaden operational infrastructure of the existing joint laboratory to serve and promote the spread of bioeconomic solutions in the regional agribusiness by providing farmers and other interested local actors with wider chemical laboratory assistance at higher scientific value. Our aim is to maintain our existing partnerships and to establish new relationships with players of the biomass business.

The objectives of the project are in harmony with those of the Bioeconomy Strategy of the EU, the BIOEAST Initiative of the CEE countries, the National Regional Development Strategy of the Slovak Republic, and the National Smart Specialisation Strategy (S3) of Hungary.

**The leading partner** is the Faculty of Chemical and Food Technology STU in Bratislava. The Accreditation Commission of the Slovak Republic and the independent Slovak Academic Ranking and Rating Agency (ARRA) ranked FCHFT as first among technical universities in Slovakia over 10 years. The leading participant of the project is the Department of Organic Technology, Catalysis and Petroleum Chemistry (DOTCPCH) of the Institute of Organic Chemistry, Catalysis and Petrochemistry of FCHFT. Its research focuses on the conversion of renewable raw materials, especially components of lignocellulose and renewable bio-oil sources also found in SR, into fuel components and on the preparation of chemicals and materials with high added value as components of the circular economy.

**The Hungarian partner** is the TTK Institute of Materials and Environmental Chemistry (TTK AKI). The active participant of the project is the Renewable Energy Research Group of the TTK AKI, which deals with the possibilities of reducing the harmful environmental effects of human activities and with the conversion of renewable energy and carbon sources into useful energy carriers and chemicals. The project researchers undertook to map the main bio-waste and agricultural by-products in the Slovak-Hungarian border area, collect samples, organize and store them at the Slovak partner by creating a so-called Biobank. DOTCPCH FCHFT STU collects mainly lignocellulosic samples in the Slovak border area and the Hungarian partner collects them mainly in the Hungarian border area.

Both research groups deal with depolymerisation of lignocellulose: the Slovak group mainly uses thermochemical and the Hungarian group hydrolytic processes. The characterisation of biopolymers and their depolymerised derivatives is carried out, in addition to standard analytical methods, using the new modern analytical equipment of the joint laboratory. Both laboratories are engaged in the development of catalytic technologies for the conversion of biomaterials and lignocellulose derivatives into value-added chemical products, fuels or chemical intermediates for further processing. The joint laboratory will provide technical support to entrepreneurs interested in implementing a biomass utilization process.

The equipment purchased with project funds is necessary to achieve the project objectives. In addition to the existing equipment of the joint Slovak-Hungarian laboratory, DOTCPCH FCHFT STU will purchase high-pressure laboratory reactors and laboratory equipment for automatic sample dosing for the Py-GC-MS instrument (pyrolysis microreactor connected to a gas chromatograph with mass spectrometer), which will significantly increase the research capacity of the joint laboratory.

Polymers represent the largest amount of waste and by-products of biomaterials to be recovered. The first step in their chemical processing is depolymerisation. It is known that one of the most important properties of biosolids is the molecular weight distribution of the polymer. The Hungarian part of the joint Slovak-Hungarian laboratory in Budapest (TTK) will acquire an Advanced Polymer Chromatograph (APC gel permeation chromatograph) to determine the molecular weight distribution of biopolymers. The chromatograph can also be used in high-pressure liquid chromatography (HPLC) mode to separate and determine the composition of complex mixtures of bioproducts.

The role of the Hungarian partner is to provide a computer link that will allow researchers to remotely monitor the equipment of the virtual joint laboratory located at the partner institution. The results will be stored in a common electronic database installed in Hungary. The data will be freely accessible to the partners by computer and processed by computer software compatible with the instruments.

The partners wish to benefit from the synergies of their expertise. They plan to cooperate not only for the duration of the project but also in the long term. The research will be closely linked to the training of young researchers and students, will contribute to improving the quality of university education and will provide the basis for new cooperation at inter-national and EU level.

STU



GC-MS with microreactor



High pressure flow reactor



High pressure batch reactor

TTK



UV VIS spectrophotometer

High pressure catalytic reactor



Automatic apparatus  
for adsorption of gases



APC/UHPLC



Server for data  
exchange

## **Achievements summary of the project SKHU/1902/4.1/001**

New samples of lignocellulosic materials were collected and were consecutively placed into Biobank of samples, where of 14 type of plants were classified by species and by the place of collection. As a result of this activity an updated sample Biobank and analysis databank was obtained about the bio resources available in the SK-HU border region. FCHFT STU was responsible for organizing the collection of biosamples, management of storage and systematization of the samples in the Biobank, and making them available for examination also for the Hungarian partner. The collected lignocellulosic samples were gradually analyzed after thermochemical decomposition to their main chemical components. The transformation of 14 types of agricultural and forestall byproducts to humus were also studied by Py-GCMS method. Molecular mass distribution of the polymeric components of lignocellulosic materials was determined by gel permeation chromatography (GPC). An APC method was developed for the determination of molar mass and molar mass distribution of technical lignin, sodium lignosulfonate and their depolymerized derivatives. Heterogeneous catalysts were prepared and characterized by physico-chemical methods. The activity of the catalysts was investigated in the catalytic conversion of compounds, having lignocellulosic origin, such as, bioethanol and guaiacol. A study of the mechanism of hydrodeoxygenation of vegetable oils and fatty acids was published in the Journal of Catalysis (<https://doi.org/10.1016/j.jcat.2021.08.052>). Butadiene was prepared from bioethanol over magnesia-silica catalysts. Accelerated and Natural Aging of Cellulose-Based Paper: Py-GC/MS Method has been accepted for publication in the journal *Molecules* on 28 April 2022. A Study of the Conversion of Ethanol to 1,3-Butadiene: Effects of Chemical and Structural Heterogeneity on the Activity of MgO-SiO<sub>2</sub> Mixed Oxide Catalysts." was accepted for publication by the journal „*Reaction Chemistry & Engineering*”. The method of Catalytic ethylation of glycerol and a consecutive tert-butylation of glycerol ethyl ethers over heterogeneous catalysts was sent for publication in the *Monatshefte für Chemie - Chemical Monthly*. The purchased autosampler AS 1020E was installed on August 11-th, 2021 as a part of the renewed system PY-GCMS, which now is composed of: a) Autosampler AS1020E; b) Multy shot pyrolyzer EGA / PY-3030D; c) Shimadzu MS -GC-2010.. The purchase of an Advanced Polymer Chromatograph (APC) and of Synology DS920+ NAS desktop at B1 has been also realized. The public procurement (PP) of laboratory reactors at LB has been finished. The public procurement process was inspected by the national control body, the Ministry of Investment, Regional Development and Informatization of the Slovak Republic with a positive result within the 2nd ex-ante control of the PP process. The batch laboratory reactors were purchased, delivered and installed at Join Slovak-Hungarian Laboratory at FCHFT STU on 30.5.2022. The system of laboratory tube reactor was purchased, delivered and installed in November 2022.

Three and four lectures were held by the LB and B1, respectively, on the project meeting at B1. At the project closing meeting at STU (LB) 8 lessons were presented (4 from TTK and 4 from STU) containing the scientific results of the project from the field of circular bioeconomy of agricultural and forestall byproducts. The possible use of straw was discussed in an article in Agro magazine. Press release materials in the journal "Agrarium", news agency "Objektívhir" and a publication in the "Innoteka" magazine.

## **The main activities of the project SKHU/1902/4.1/001**

### **1. Project management**

The project implementation, administration, and finance at the FCHFT SUT were managed by Prof. Kaszonyi, at the RCNS by Prof. Valyon. The institutional offices of partners took care of the financial administration of the project. Managing professors directed the development and research works in the Joint Laboratory in cooperation. Each partner was responsible for organizing a meeting (a workshop in September 2021 resp. the closing meeting in September 2022) for researchers, developers, and interested local actors. They actively promoted exchange visits of young research workers and managed of the installation of APC instrument Empower software on personal computers so that several people can access the device at any time. They managed public procurement processes of APC instrument at B1, three laboratory reactors and of the autosampler AS 1020E at LB, the domestic co-financing and advance contract for B1 in Hungary and domestic co-financing contract for LB in Slovakia. B1 joined the Hungarian Bioeconomy Cluster as a regular member.

### **2. Communication**

Project partners communicated by e-mail, phone, or at personal meetings. For communications with the scientific community, publications, conferences, and symposia were used. The best possibility for communication with actors in agriculture and related industries was during the collection of lignocellulosic and other biosamples. The websites of cooperating institutions and the project website were used to introduce the project to a wide range of stakeholders. Press release in "Agro", (attachment of Új Szó, newspaper for the public in Slovak-Hungarian border region, which has printed form and also on-line form) was realized about the results of the project. Because of Covid-19 instead of the project opening workshop a symposium was organized at the RCNS in September 2021 and another one (project closing in September 2022) at the FCHFT STU. Actors in agriculture participated in the online part of the project's final meeting. Hand-out material (leaflet) was distributed mainly during sample collection in SR and HU. Posters according to the visibility guide at RCNS and FCHFT STU were also prepared.

Some details:

- Press release materials in the journal "Agrarium" and at news agency "Objektívhir".  
(<https://agrarium7.hu/magazin/7tka1dw5s4>)  
(<http://www.objektivhir.hu/display/hir.php?hirid=57015>).
- The project description was also uploaded to the homepage of an SME partner.  
(<https://biophosphate.net/news/joint-slovak-hungarian-chemical-research-laboratory-has-been-established-support-bioeconomy>).

- The project website at B1 ([www.ttk.hu/palyazatok/bioeconomy](http://www.ttk.hu/palyazatok/bioeconomy)) was updated with the information about installation of the APC device, about joining the Hungarian Bioeconomy Cluster, a joint research article published in the Journal of Catalysis and a lecture presented at the meeting of Catalysis Society, Hungarian Academy of Sciences, the invitation to the project meetings, the programme of the meeting and the presentations. The invitation to event was also advertised on the Facebook of Hungarian Bioeconomy Cluster:

<https://www.facebook.com/magyarbioeconomyklaszter/photos/a.109441657322793/379099457023677/>.

Press release materials in the journal Innoteka were also prepared

[http://www.ttk.hu/wp-content/uploads/Innoteka\\_sajtokozlemeney.pdf](http://www.ttk.hu/wp-content/uploads/Innoteka_sajtokozlemeney.pdf)

- From recycled materials leaflets and pens, dedicated to the project have been made and distributed among stakeholders.

- The website of the project ([Project no. SKHU/1902/4.1/001](http://Project.no.SKHU/1902/4.1/001) ([skhulaboratorium.sk](http://skhulaboratorium.sk))) was prepared and updated by information about project closing meetings and results of project.

### **3. Qualitative and quantitative characterization of biomass**

Biomass - lignocellulose samples (>250) were collected in the Slovakian-Hungarian border region from products of farms and forestries inappropriate as food or fodder. Plant samples were classified by species and by the date and place of collection. As a result of this activity, an updated sample Biobank and analysis databank were obtained containing information about the bioresources, available in the border region. Moreover, partners established a sample collection that gives scientific work to the joint laboratory for a long time. The lignocellulosic polymer was treated by acid or base, or to steam to become hydrolyzed to a product mixture of smaller molecules, preferably monomers. Alternatively, pyrolytic method was used to decompose the biopolymer. For some samples, the amounts of the main lignocellulosic components, such as cellulose, hemicellulose, and lignin, were determined. Studies are continued to recover value added components from the mixture prepared by hydrolysis and pyrolysis. The Joint laboratory can provide valuable information about the waste biomass in the region.

Tasks of LB and B1:

The FCHFT STU (LB) was responsible for organizing the collection of biomass samples in the Slovakian and North Hungarian region, whereas the RCNS (B1) took care of the management of sample collection in the Hungarian region. The Slovakian partner (LB) managed the storage and systematization of the samples in the Biobank, and made them available for examination also for the Hungarian partner (B1). The Slovakian professional management was responsible for characterizing the biomass samples by pyrolysis-GCMS equipment, whereas the decomposition products of biosamples were characterized by distribution of molecular mass at the RCNS (B1) using the size-exclusion chromatograph (APC) installed at the RCNS. The results were compared with data from the literature obtained for similar biomaterials. The studies for catalytic upgrading of biosamples are continued in both laboratories. The results obtained by the joint laboratory were uploaded onto a common computer server (network-attached storage device) of the project. The server was deployed by the Hungarian partner (B1) in the joint laboratory in Hungary by the time when all the planned instruments were purchased from the project budget and installed. At LB an autosampler was purchased for significant increase of the analytical capacity of the pyrolysis-GCMS of Joint Slovak-Hungarian Laboratory. Consecutively three laboratory reactors were purchased for treating collected samples by different chemicals and processes during their decomposition and upgrading the formed compounds to value-added substances and materials.

At B1 an Advanced Polymer Chromatograph (APC) was purchased for the characterization of biomass-derived biopolymers and monomers. The optimal column set, column temperature, eluent composition, eluent flow rate, and amount of sample to achieve high separation rate and resolution in APC analysis of lignin samples were determined, using the method for the determination of molar mass and molar mass distribution of technical lignin, sodium lignosulfonate, and their depolymerized derivatives.

The joint scientific papers are prepared for publication with the strong cooperation of the managing professors.

#### **4. Upgrading biomaterials**

This activity concerns catalytic conversion of bioproducts like triglycerides, and decomposition products of carbohydrates, lignin, and protein biopolymers to value-added chemicals, fuels, and intermediates for further chemical transformations. The activity covers also the value increasing conversion of oxygenates. It is very important to find methods for the utilization of lignin. It could be used, for instance, as antioxidant or converted to fuel and monomeric component of different polymers. Based on the result of the research and development activity, favorable chemical conversion processes will be suggested for two different kinds of biomaterials. The laboratory-size experiments were carried out to be suitable for establishing the upscaling of the chemical processes. The upscaling work of any stakeholder is to be supported by the Joint laboratory.

Tasks of LB and B1:

Laboratory scale processes were developed in both part of Joint Slovak-Hungarian Laboratory from which the most effective upscalable process and catalyst was chosen during the last period of project reporting. List of laboratory processes of biomass component utilization that can be increased in size will be uploaded in the final project report of partners. One description (in the form of electronic document) of catalytic process for biomass upgrading and one recipe of catalyst preparation for this process increasable in size.

Heterogeneous catalysts were prepared and characterized by physico-chemical methods. The activity of the catalysts was investigated in the catalytic conversion of compounds, having lignocellulosic origin, such as, bioethanol and guaiacol.

Butadiene was prepared from bioethanol over magnesia-silica catalysts, having sites of well-balanced acid-base properties. Phosphatized-alumina-supported Pd catalysts were prepared for triglyceride hydrodeoxygenation to hydrocarbons. From glycerol and bioethanol, ethers were prepared over acidic zeolites and resins. By further alkylation with isobutylene, their polarity was decreased to increase their solubility in diesel.

#### **5. Knowledge dissemination and education**

The spreading rate of bioeconomic practice is enhanced by teaching potential stakeholders on environmental-friendly bio-business. Project participants took part on events and also organized meetings to talk about bioeconomy and their results. Knowledge transfer was planned to be made more efficient by regular exchange of young researchers and PhD students between the cooperating countries to work in the joint laboratory and learn methods and theories also from prominent foreign scientists. We helped receiving information to those who does not know foreign languages. The advantage of internet is taken to disseminate information in English, Slovakian, and in Hungarian. The new infrastructure establishes the common long-term use of the joint laboratory and the long lasting cooperation of the proposing institutions.

Prof. Kaszonyi and other experts from FCHFT SUT incorporated the basic principles of bioeconomy and some scientific results of project into the education of bachelor, master and PhD students.

LB and B1 experts prepared and presented during project workshop and closing conference 15 lessons containing the scientific results of the project from the field of circular bioeconomy of agricultural and forestall byproducts.

Three lectures and one poster were presented at the online International Conference on Reaction Kinetic Mechanism and Catalysis (RKMC website (akcongress.com)). The results of joint research by LB and B1 on heterogeneous catalytic upgrading of model compounds of lignocellulosic origin were presented.



We prepared more than 3 planned papers from our results in prestigious journals. A study of the mechanism of hydrodeoxygenation of vegetable oils and fatty acids was published in the Journal of Catalysis (<https://doi.org/10.1016/j.jcat.2021.08.052>). The method of Catalytic ethylation of glycerol and a consecutive tert-butylation of glycerol ethyl ethers over heterogeneous catalysts was sent for publication in the Monatshefte für Chemie - Chemical Monthly. Accelerated and Natural Aging of Cellulose-Based Paper: Py-GC/MS Method has been accepted for publication in the journal Molecules on 28 April 2022. A Study of the Conversion of Ethanol to 1,3-Butadiene: Effects of Chemical and Structural Heterogeneity on the Activity of MgO-SiO<sub>2</sub> Mixed Oxide Catalysts.” was accepted for publication by the journal „Reaction Chemistry & Engineering”. The website of the project at B1 ([www.ttk.hu/palyazatok/bioeconomy](http://www.ttk.hu/palyazatok/bioeconomy)) was updated with the manuscript of the paper.

The possible use of straw in the field of circular bioeconomy was discussed in an article in Agro magazine. Press release materials in the journal “Agrarium” , at news agency “Objektívhir” and in Innoteka were also prepared.

A researcher of B1 gave a lecture on the results of the joint research of LB and B1 at the meeting of the Catalysis Society, Hungarian Academy of Sciences (Events – [catalysis.hu](http://catalysis.hu)).

## **6. Data storage, handling, and sharing within the joint laboratory**

LB and B1 upgraded their existing Joint laboratory by new laboratory instruments. The purchase of Synology DS920+ desktop server has been also realized. Thanks to the modern information technology and the purchased server, it was possible to establish real-time connection (**Network-Attached Storage**, NAS, device) among the partners located at FCHFT SUT and at RCNS and their instruments installed at the Joint laboratory. The server is storing the data of measurements and results. The catalytic and APC characterization results were uploaded to our common server for data sharing. This gives the consortium partner easy access to our results. The new results of Py-GCMS analyzes of gathered lignocellulosic samples from BioBank were also uploaded to our common server located at partner B1 for data sharing. The data and results on server are protected, but are available for both partners at any time. Data processing is made possible for any of the cooperating partners using instrument compatible computer programs at both partners. Data handling and sharing has been moved from the computers to the common server.

### ***R410|Level of cross-border cooperation***

The cooperation of proposing partners, STU in Bratislava and RCNS, continues since 2012 when partners gained EFRA support to develop processes for the chemical conversion of biomaterials. One SME was participating in the project from the Slovak and one from the Hungarian side. In 2018 we obtained support from the small fund of the Interreg Programme together with a new Slovak and Hungarian SME. This represents the present level of cross-border cooperation. Our intention is to integrate actors in agriculture and additional SMEs in the bioeconomic activities.

The project partners are cooperating permanently since 2012, the continuing communication with actors in agriculture is realized mainly during the collection of lignocellulosic biosamples of agricultural byproducts and on-line part of project closing meeting.

### **Priority / Project improves service provision in the borderland, enhances mutual understanding and bi-lingualism.**

Organic wastes and side-products can be generated at any side of the border and can be processed to get value-added product at the nearest and most suitable processing plant without regarding the border. The rational utilization and valorization of regional renewable bioresources enforces cross-border human mobility and information exchange on a common language, which can obviously be either Hungarian or Slovak.

Some activities were shifted in time, depending on Covid-19 mobility limitations. The opening workshop was realized in September 2021, instead of October 2020. Scientists from LB visited the laboratory of project partner B1 and made consultations on future common works and publications, and worked on types of equipment of Join Slovak-Hungarian Laboratory placed at project partner B1 in Hungary. During the collection of lignocellulosic biosamples of agricultural by-products, the communication was bilingual, Hungarian, and Slovak, depending on the place of sample collection.