

## **SUMMARY OF THE MAIN RESEARCH RESULTS AND SCIENTIFIC CONTRIBUTIONS**

**Assoc. Prof. Dr. Eng. Andriana Risk Surleva**

**candidate in a competition for the academic position of "professor" in**

**4.2 Chemical Sciences (Analytical Chemistry),**

**announced in the State Gazette 64 of 05.08.2025**

In this competition I present summarized results of 35 research papers, of which 10 publications are assigned to indicator 4 in journals ranked with Q1-1; Q2-4; Q3-1; SJR-4 for a total of 160 points (Appendix 5d) and 25 publications - to indicator 7 in journals ranked with Q1-1; Q2-4; Q4-2; SJR-17, non-indexed journals - 2 for a total of 289 points (Appendix 5a), 138 citations (Appendix 5b) were noted.

Scientific research is mainly object-oriented with the aim of expanding the analytical information obtained from various methods of chemical analysis, and its application for the development of new technologies or optimization of existing ones. The research is aimed at developing, optimizing and validating/verifying methods for chemical analysis of environmental objects, samples from industrial production and other artifacts of human activity.

The results obtained can be summarized in the following directions:

1. Development of a protocol for chemical analysis and characterization of industrial wastes with the aim of their valorization and reduction of the ecological footprint
2. Development, optimization and validation/verification of methods for chemical analysis of environmental objects with the aim of obtaining up-to-date data for environmental risk assessment
3. Characterization of the analytical behavior of methods for chemical analysis of agricultural soils with the aim of determining plant-available forms of nutritional components
4. Development, optimization and verification/validation of analytical methods and their application for the study of artefacts of human activity

### ***Direction 1: Development of a protocol for chemical analysis and characterization of industrial waste***

The utilization of industrial waste is part of the European Union's circular economy strategy. One of the modern approaches is the development of new geopolymers with a low CO<sub>2</sub> footprint as an alternative to traditional construction materials. Materials rich in aluminum and silicon with appropriate solubility in alkaline media are used to obtain geopolymer materials through appropriate optimization of the technology, based on the study of the chemical composition and some specific characteristics. Tailings materials from various sources in Bulgaria have been characterized: copper extraction waste, fly ash and tailings from coal combustion in Bulgarian thermal power plants. The focus of the research is the development of an algorithm for characterization from the point of view of application of the results for the development and optimization of a technology for utilization of tailings materials and obtaining geopolymer materials. Methods of analysis were modified and verified for specific application to the investigated precursors and the geopolymers obtained from them to ensure comparability of the results.

The results are presented in publications No. 4.3-4.10 and 7.12. These publications have been cited 27 times.

Scientific and applied scientific contributions:

- Materials from three copper mine tailings dumps and two tailings from coal-combustion thermal power plants were characterized as precursors for the production of geopolymers with a low CO<sub>2</sub> footprint;
- Original data on tailing materials were obtained, which can be used both for the production of geopolymer materials and for environmental assessment;
- For the first time, an assessment of the mobility and bioavailability of heavy metals and other potentially toxic components in geopolymer materials obtained from Bulgarian sources was made;
- Geopolymer materials with a low carbon footprint were obtained from precursors: copper mine tailing and fly ash from coal combustion in Bulgaria, suitable for the production of classic and 3D-printed construction materials.

The beginning of interest in geopolymer materials and technologies arose as a result of the collaboration with the Center of Geopolymers and Green Technologies, University of Malaysia Perlis. Geopolymer materials exhibit unique properties and characteristics, including high compressive strength, high temperature stability and low thermal conductivity. An overview of the achievements in the development of refractory materials based on geopolymers, including the analytical methods for their study, is presented in [4.4]. The research continued in collaboration with the team of prof. Petrica Vizureanu, Faculty of Material Science and Engineering, “Gheorghe Assachi” Technical University of Iasi, Romania. The results are part of the project “RecMine – Environmental footprint reduction through eco-friendly technologies of

mine tailings recycling”, BNSF KP 06-DO-02/5 , 2022-2024 EraMin 3, Horizon Europe. The international consortium includes universities from five countries (Romania, Bulgaria, Portugal, Turkey, Spain), the coordinator of the international team is prof. Petrica Vizureanu, and I am the head of the Bulgarian team. The project was completed with a very good grade.

In order to select suitable precursors for the production of geopolymers, the characteristics of tailings materials from Bulgaria were studied [4.8]. Materials from five tailings facilities were experimentally characterized: “Asarel Medet” LtD, “Elatsite Med” LtD and an inactive mine tailing dump in the Burgas region, as well as fly ash from TPP “Bobov Dol” and mixed tailings material from TPP “Maritsa Iztok”. A combination of preliminary verified analytical methods was applied to obtain detailed information about the composition and characteristics of the materials. The studied samples contain relatively high concentrations of Al and Si (17-21%  $\text{Al}_2\text{O}_3$ ; 68-53%  $\text{SiO}_2$ ), as well as a low concentration of sulfur (0.02-3.5% as  $\text{SO}_3$ ). An exception is the mixed tailings material from TPP “Maritsa Iztok”, in which the content of sulfur-containing components is above the limit for the production of stable geopolymers. The aluminosilicates, as evidenced by XRD, are expected to show high reactivity in alkaline media, but some less reactive phases are also observed. The results indicate that the studied mine tailings and fly ash can be used as precursors for the production of geopolymers after adapting the technology to take into account the specific characteristics of the raw materials.

Copper mine tailings from “Asarel Medet” LtD, Bulgaria [4.3] and fly ash from TPP “Bobov Dol” [4.10] were systematically investigated as precursors for the production of geopolymers. The particle size distribution, chemical and mineralogical composition, as well as the reactivity in alkaline medium, acidity and electrical conductivity of the aqueous suspension were investigated. Sequential extraction was applied to determine the geochemical phase distribution of heavy metals. The studied samples were characterized by high alkalinity of the aqueous extract, which favors the geopolymerization process. The content of water-soluble sulfates (<4%) was favorable for the production of geopolymers, but the Si/Al ratio (about 3) in the mine tailings required mixing with a source of active aluminum to obtain geopolymers for construction purposes. The results obtained showed that a suitable source of reactive aluminosilicates was the fly ash from the TPP “Bobov Dol” [4.10]. The fly ash has been found to have a high content of Si (52%) and Al (21%), a suitable ratio of active Si/Al = 2 for geopolymerization, and a low content of sulfur-containing components.

The alkaline reactivity of aluminosilicate materials is essential for the development of a technology for the production of geopolymers by alkaline activation, but there is no standardized analytical procedure for its evaluation. This required the development of a specific analytical procedure based on ICP-OES measurement of the concentration of Al, Si and Ca in alkaline solution [4.7]. In addition to the determination of reactive silicon and aluminum, the study included calcium, since these components contribute to the development of the geopolymer matrix. The change in the concentrations of

dissolved ions in 6.5 M NaOH was monitored as a function of the contact time between the liquid and solid phases. In contrast to the approach usually described in the literature, which presented alkaline reactivity as the concentration of dissolved ions in the alkaline solution, in this study it was evaluated by the content of the dissolved component, referred to kg of dry sample. This allowed, on the one hand, comparability of data between different laboratories, procedures or raw materials, and on the other hand - it allowed us to determine the relative share of reactive components. The results obtained showed that the reactivity in alkaline medium depended to a greater extent on the reaction time than on the concentration of the NaOH solution. The data made it possible to optimize the amount of alkaline activator and reduce the technological costs for obtaining geopolymers from Bulgarian raw materials.

The characteristics of the aqueous infiltrates are another indicator of the reactivity of the tailings materials in the formation of a geopolymer matrix. The methods for determining the pH and electrical conductivity of the precursors for the obtaining of geopolymers were validated by a certified reference soil material for pH and electrical conductivity CRM498 [7.9]. The value of the Z-index was below 2 and the analytical behavior of the two studied methods was assessed as “satisfactory”. The validated procedures were applied to mine waste and fly ash from Bulgaria, Romania and Portugal. The aqueous suspension of industrial waste showed a pH of 6-12, all studied fly ashes contained a high concentration of components ionized in aqueous solution. The results showed that the studied raw materials have potential as precursors for geopolymers.

Given the application of the obtained geopolymers as construction materials, it is of interest to study the level of encapsulation of heavy metals in the geopolymer matrix and, accordingly, the ecological footprint of the newly obtained materials. The reason is that for some components of the precursors during alkaline activation, the formation of chemical species with increased solubility can be expected and therefore a potential risk of environmental pollution. Two approaches were applied: (i) study of the content of heavy metals in the residual fraction after prolonged contact with a NaOH solution with a concentration close to the technological one [4.1; 4.10]; (ii) sequential extraction according to the BCR procedure applied to the precursors and geopolymer materials [4.6]. It was found that after contact with an alkaline solution, the major part of the heavy metals remained in the solid phase of the mine tailings and it could be expected that after geopolymerization, they would remain fixed. The possibility of obtaining geopolymers from single precursors - fly ash, allowed us to directly observe the leaching rate of heavy metals in the aqueous media. The results showed a 30-50% increase in the content of heavy metals in immobile (fixed) form after geopolymerisation. A sequential extraction procedure allowed to assess the mobility of heavy metals under different environmental conditions: weakly acidic rain, aerobic and anaerobic conditions [4.6]. A CRM BCR 701 lake sediment was used to verify the sequential extraction procedure. The BCR procedure was modified

by reducing the sample weight, but maintaining the solid-liquid ratio, as well as applying different procedures for separating the supernatant. The concentration of the analytes in the resulting solutions was determined by ICP-OES. From the data obtained from the sequential leaching, contamination indices and encapsulation level of heavy metals were calculated as a basis for assessing the potential ecological footprint of the studied materials. For Cr, Ni, Pb and Zn, a high degree of encapsulation in the geopolymer matrix was observed, but Cu showed increased mobility in the geopolymer samples, which requires further studies.

It should be emphasized that when designing the mixture of precursors (mine tailings and fly ash), in addition to the chemical and physical characteristics of the precursors, it is necessary to take into account the target mechanical and microstructural characteristics of the resulting product. Geopolymer materials containing 25% copper mine tailings from “Asarel Medet” Ltd and 75% fly ash from the TPP “Bobov Dol” were synthesized, with optimal characteristics as building materials [4.3]. A computational algorithm for the material and energy balance of the technology for the production of geopolymers from the indicated precursors was developed [7.12]. The results showed that the energy consumption and carbon dioxide emissions in the industrial production of geopolymers using the developed technology are two times lower compared to construction ceramics.

***Direction 2: Development, optimization and validation/verification of methods for chemical analysis of environmental objects***

In this direction 12 publications are presented: (indicator 4, publications with numbers 4.1 and 4.2) and (indicator 7, 10 publications with numbers 7.5-7.7, 7.19-7.25). Total citations: 48.

The main goal of research in this area is to obtain up-to-date data on the state of environmental objects, which correspond to modern trends in assessing the ecological footprint of human activity. The research includes the use of bioindicators as a means of assessing potential toxicity or contamination of the studied objects. To achieve these goals, various methods of chemical analysis have been combined, appropriately modified and validated, which allow not only to obtain information on the total content of target pollutants, but also to obtain data on their mobility and bioavailability.

The scientific and applied scientific contributions can be summarized as:

- An analytical protocol for assessing the condition of a tailings pond from polymetallic ore mining has been developed, which combines various chemical analysis methods and biological tests.
- New data have been obtained on the composition and current state of the tailings material, soils and waters from the Tarnica-Suceava tailings pond area, Romania.

- New data have been obtained on the double isotope dilution method for the mass spectrometric determination of mercury in aquatic organisms, which are of interest to metrological institutes in the studies of a primary method of analysis.
- An ion-chromatographic method for the determination of standard anions and pollutants in natural waters has been validated.

In cooperation with the Bulgarian Institute of Metrology, analytical methods for assessing the chemical composition of natural waters have been optimized and validated. The state of high-mountain lakes, as well as aquatic organisms, is a sensitive indicator of the level of global, regional and local ecological changes. This requires methods with low detection limits, high reliability and trueness to be applied for the determination of controlled pollutants. The results have been published in 3 articles [7.5, 7.7, 7.25], cited 5 times, one doctoral dissertation has been defended (2022).

The optimal parameters of the analysis of water samples for the simultaneous determination of eight inorganic anions:  $F^-$ ,  $BrO_3^-$ ,  $Cl^-$ ,  $NO_2^-$ ,  $Br^-$ ,  $NO_3^-$ ,  $PO_4^{3-}$  and  $SO_4^{2-}$  by ion chromatography with chemical suppression and conductometric detection [7.5, 7.7] were established. An uncertainty budget was developed and the main contributing factors for each of the studied ions in different concentration ranges were estimated. The results showed that the uncertainty of the calibration function and the repeatability of measurement have the largest contribution to the uncertainty of measurement. Of the studied anions, those with higher concentrations ( $Cl^-$ ,  $NO_3^-$  and  $SO_4^{2-}$ ) are determined with an uncertainty of 1% to 6%, while the anions with relatively lower concentrations ( $F^-$ ,  $BrO_3^-$ ,  $NO_2^-$ ,  $Br^-$  and  $PO_4^{3-}$ ) - with about 10%. The applicability of the method in a real working environment has been demonstrated and the results obtained are the basis of an internal methodology and validation protocol of the Department of Chemical Measurements and Ionizing Radiation, Bulgarian Institute of Metrology. An initial study of six high-mountain lakes in Pirin, Bulgaria [7.25] was carried out, determining the elemental composition by ICP-MS and the anion content by ion chromatography. The results showed that the concentrations of the studied analytes are within the levels considered safe for aquatic organisms. It should be noted that for an adequate assessment, additional components should be determined, as well as the oxidation-reduction processes, changes in active acidity and the presence of ionized components should be monitored.

In collaboration with the Laboratoire national de métrologie et d'essais, Paris, France, a comparative study of methods for the determination of mercury and its forms in samples of marine organisms as a bioindicator of water pollution has been carried out. The analytical characteristics of mass spectrometric techniques for the quantitative determination of mercury in samples of aquatic organisms have been studied: ICP-SFMS, ICP-QMS, GC-ICP-SFMS and elemental mercury analyzer, with the aim of their validation, including the procedures for sample preparation and selection of optimal operating parameters, calibration approaches and uncertainty assessment

[7.6]. New data on the double isotope dilution method in the mass spectrometric determination of mercury in aquatic organisms have been obtained, which are of interest to metrological institutes in the studies of a primary method of analysis.

The long-term operation of a polymetallic ore mine in the Tarnica-Suceava region, Romania, has resulted in a huge amount of waste materials collected in tailing ponds and basins. The mine operated till 2006 and the tailings pond was remediated. A number of studies have shown a potential negative effect on the environment, but the impact of tailings on the surrounding ecosystem is far from being fully assessed. This has provoked research in this area. The research was carried out in collaboration with the team of prof. Gabi Drochioiu, Biochemistry and Toxicology group, Faculty of Chemistry, “Alexandru Ioan Cuza” University of Iasi, Romania. They are aimed both at development of approaches for characterization of tailings materials with a focus on assessing the bioavailability of potentially toxic pollutants for humans and the environment, as well as study of appropriate treatment procedures for decontamination of soils in order to reduce the bioavailability of heavy metals.

To assess the ecological footprint and potential environmental impact of the remediated mine tailings pond, a protocol was developed that combines different analytical approaches for determination of chemical composition, as well as germination tests, in an attempt to assess the ability of the soil to support plant growth and the bioavailability and uptake rate of heavy metals [4.1, 7.19, 7.21]. The germination tests used wheat (*Triticum aestivum*) as a bioindicator to assess the bioavailability of heavy metals in the tailings and drainage waters [4.1, 4.2, 7.19, 7.22, 7.23]. In the tailings pond area and around the drainage water basins, plants, adapted to unusually high concentrations of elements, including heavy metals, have been found [4.1]. This fact has provoked research on the impact of various pollutants on plants [4.2, 7.19]. For this purpose, methods for chemical analysis of tailings, soils, waters and plants have been modified and verified [7.20, 7.21]. The studies also included a preliminary assessment of some approaches to reduce the toxicity of soils and tailings [7.23, 7.22, 4.2]. The assessment was based on the bioavailability of potentially toxic elements by quantification of heavy metals and some other components in different parts of the bioindicator.

The use of bioindicators (wheat seeds as well as endemic species for the area) required validation/verification of chemical analysis methods. Publication 7.20 presents the results of the validation of a protocol for plant tissue analysis for the quantitative determination of heavy metals, based on acid digestion and ICP-OES quantification of heavy metals. The sample preparation procedure and ICP-OES measurement were validated by analysis of a certified reference material. In addition, the standard addition method was applied before sample pretreatment to assess the accuracy of the method in the concentration range of heavy metals found in plants from the contaminated area. The recovery was between 97 and 105%. The precision of the complete protocol, which includes germination tests, sample digestion and ICP-OES measurements, ranges between (RSD) 4.7% for Mn and 11% for As.

The results of the verification of the ICP-OES method for the determination of heavy metals in soils and mine tailings after acid digestion are presented in [7.21]. The “ERA Metals in soil” certified reference material and the standard addition method were used to assess the influence of the matrix. Wavelengths subjected to the least possible spectral interference at the concentrations of the analytes and matrix components in the studied samples were identified. The sensitivity, bias and precision of the method were determined. The achieved method detection limits were: As 0.80; Cu 0.29; Pb 0.29; Zn 0.30; Cd 0.11; Cr 0.09; Ni 0.02, mg/kg.

The combined protocol of chemical and biological tests [4.1] was applied to assess the state of the area around the mine tailings pond through chemical analysis of tailings material, waters, soils and plants. The total content and exchangeable forms of heavy metals, active acidity and the content of carbon and nitrogen were determined. A modified sequential extraction method was used to determine the geochemical phase distribution of heavy metals. As a result of the studies, it was established that the most abundant heavy metals in the studied samples were Cu, Zn and Pb. Elevated concentrations of As were also found. The results of the sequential extraction showed that up to 51% of the copper was retained by amorphous and crystalline iron oxides. Higher lead content was observed in the amorphous iron oxide fraction. During the dry season the concentration of heavy metals in the river water, into which the drainage waters from the tailings pond are discharged, varied from 0.13 mg/L Fe to 4.2 mg/L Zn and was below the maximum permissible concentration in drinking water. The high content of iron-containing components possibly favored the process of water purification. A possible explanation was the formation of amorphous iron hydroxide, which absorbed high concentrations of other components. It was accumulated in the river sediments, in which high content of Fe and heavy metals was observed. The toxicity of the soil and the bioavailability of heavy metals in the tailings pond and surrounding soils were investigated using a bioindicator and germination tests. It was found that the germinated plants on the studied soils accumulated increased concentrations of heavy metals. The results of model studies of the influence of various chemical components of the tailings on the bioindicator showed that arsenic significantly reduced the germination of wheat seeds, but phosphate ions showed an antagonistic effect, which partly explained the vitality of some plants in the area [7.19].

Within the framework of these studies, the possibilities for treating the tailings and surrounding soils aiming at reducing the bioavailability of heavy metals were also evaluated. The results showed that soil treatment with solutions of sodium hydroxide [7.19] and magnesium nitrate [4.1] reduced the toxic effect on the bioindicator: increased germination and development of wheat sprouts were observed, as well as lower concentrations of heavy metals in the different parts of the bioindicator. It is known that complexon III or sodium citrate are effective for removing heavy metals from various matrices by complexation, however, in the studied samples, no stimulation of sprout growth and the expected reduced soil toxicity were observed



[7.19]. It has been shown that despite the effectiveness of *Saccharomyces cerevisiae* (brewer's yeast) in binding heavy metals, a preliminary step of washing with water or alkaline solutions was necessary to reduce bioavailability of toxic components and increase the effect of bioremediation [7.23, 7.22]. A detailed study of the influence of aqueous extracts from the copper main tailing on the bioindicator as a model of the drainage waters and their effect on plant species from the surrounding environment has also been conducted [4.2]. A strong negative effect on the germination and development of wheat sprouts due to the high concentrations of Cu, Pb and Fe, determined by ICP-OES and AAS was observed.

In these studies, the application of *Triticum aestivum* L. (wheat), germination tests and chemical analysis of heavy metal accumulation in the bioindicator allowed a more adequate assessment of the bioavailability and potential toxic effect of pollutants. This approach, combined with the determination of the chemical composition, the distribution of heavy metals in the geochemical fractions, is a tool for assessing both the state and potential environmental risks of mine tailings, as well as for a primary assessment of the effectiveness of treatment procedures.

### ***Direction 3: Characterization of the analytical behavior of methods for chemical analysis of agricultural soils and determination of plant-available forms of soil nutrients***

The research in this direction has been carried out in collaboration with the company “Sembodja” Ltd. The aim of this research is to study and select methods that allow the determination of plant-available forms of K, P and S, which are agronomically calibrated for Bulgarian agricultural soils and crops. The results obtained are applied in the routine work of the company’s “Laboratory for soil testing”.

In this direction, I present 4 publications (No. 7.8-7.11). These publications have received a total of: 9 citations. Publication 7.11 was accepted for publication 09.2025 in Plant, Soil and Environment (Q2). One doctoral dissertation has been defended (2023).

Scientific and applied scientific contributions:

- A comparative study of the effectiveness and uncertainty of the result of the determination of plant-available K in Bulgarian agricultural soils after extraction by 5 methods followed by AES quantification of the analyte [7.8, 7.9] was conducted. Data for a similar study for Bulgarian soils were not found in the literature.
- The experimental conditions were optimized and the analytical characteristics of the spectrophotometric determination of phosphate ions by the molybdenum blue method in soil extract in the presence of acetate-lactate buffer reagent were studied. It was found that the inhomogeneity of the sample

and the repeatability of the extraction procedure were the factors that have a major contribution to the uncertainty [7.10].

- For the first time, a correlation study of the acetate-lactate extraction method and Mehlich 1 for the determination of plant-available K and P in Bulgarian agricultural soils was done [7.8]. The results for both nutrients showed a coincidence within the uncertainty. A mathematical model for data transfer between the studied methods was derived. In 76% and 60% of the cases, the calculated nutrient status of the soil in terms of P and K stock, respectively, coincided with the experimentally determined ones. It should be noted that the conversion of results from one method to another should be considered with particular care, since it is influenced by the type and condition of the soil.
- Nowadays, the wider distribution of sulfur-deficient regions requires optimization of soil testing procedures to increase their accessibility to laboratories. A modified version of the turbidimetric method for the determination of water-soluble sulfates in soil after leaching with  $\text{CaCl}_2$  reagent was proposed [7.11]. The method showed: MLOD 5.0 mg/kg; precision  $\text{RSD} < 3\%$ ; recovery  $(103 \pm 18) \%$ , expanded uncertainty 2.3 mg/kg  $\text{SO}_4^{2-}\text{-S}$  ( $K=2$ , norm.). The proposed test protocol was lower cost, fast and accessible, with characteristics suitable for the assessment of water-soluble sulfate in arable soils. The results showed that 74% of the 546 soil samples were sulfur deficient ( $\text{SO}_4^{2-}\text{-S}$  content was less than 10 mg/kg and the availability index  $< 6.0$ ). The accessibility of laboratory analysis to a wider group of farmers could contribute to effective fertilization programs, as innovative fertilizer blending technologies are based on an adequate assessment of sulfur bioavailability in arable soils.

***Direction 4: Development, optimization and verification/validation of analytical methods and their application for the study of artefacts of human activity***

***a) optimization and validation of SEM/EDS method for identification of gun shot residue***

The research is in collaboration with the Forensic Agency of Kosovo and is defended in a doctoral dissertation (2023). The results are presented in 2 publications (No. 7.1 and 7.2) in journals with a rank of Q2 and have been cited a total of 12 times.

The influence of operating parameters on SEM/EDS method as well as validation of SEM/EDS for identification of elemental composition of inorganic gunshot residue (GSR) from incidents in the Republic of Kosovo was done for the first time [7.1]. Based on the results obtained, a database for the elemental composition of GSR-specific particles has been developed. The database allowed for the identification of newly appearing weapons on the regional market. The validated method is applied in the routine activities of the laboratory at the Forensic Agency of Kosovo.

The persistence of GSR particles identified by SEM/EDS on a specific surface of the hand of a shooter using firearms from criminal cases in the Republic of Kosovo was assessed as a function of sampling time, number of shots fired and caliber of the weapon used [7.2]. The data obtained are the basis for revising the working protocol for GSR sampling in the Republic of Kosovo.

*b) improving analytical control in industrial production*

The research is in collaboration with the team of prof. Fatos Rexhepi, University of Mitrovica, Kosovo. Publications 7.3 and 7.4, cited 6 times.

Acrylamide is a neurotoxin that occurs in thermally processed foods, especially in potato chips, bread and coffee, and is subject to regulatory control. A method for the quantitative analysis of acrylamide in potato chips samples was developed using FTIR-ATR spectroscopic techniques in combination with multivariable calibration [7.3]. The calibration and validation of the method was performed using LC/MS-MS. It was demonstrated that the FTIR-ATR spectrometry technique, combined with an appropriate PLSR model, could be applied for monitoring acrylamide content in commercial products, with the main advantages being lower cost, simple, fast and with minimal sample preparation. The proposed methodology is in line with the concepts of green analytical chemistry. The main limitation is the strong matrix effect, which requires calibration for each matrix. The method is applied in the factory's laboratory of a company in Kosovo.

The oxidation process induced by thermal treatment of vegetable edible oils was investigated using a combination of titrimetric methods, UV-Vis and FTIR spectrometric techniques [7.4]. A hypothesis for the mechanism of the oxidation process was derived based on IR spectral data and it was demonstrated that FTIR spectrometry was a suitable tool for monitoring the processes and assessing the degree of lipid oxidation in the earliest phases of thermal degradation of the studied sunflower, corn and olive oils. The obtained data showed that corn oil was the most thermally stable oil among the studied ones.

*c) improving technologies for recycling and waste valorization*

This research is in collaboration with a team of prof. Subaer, National University of Makassar, Indonesia and was aimed at utilizing biowaste from fruits widely distributed in the region with resulting materials effective under sunlight without additional energy sources. The obtained results are presented in two publications No. 7.17 and 7.18 in journals with SJR, cited 25 times.

Silver nanoparticles (AgNPs) were synthesized with the bioreductant aqueous extract of papaya fruit (*Carica papaya*) [7.17]. The obtained nanoparticles showed selective response to Hg(II) ions and are a potential chemical photometric sensor.

A green chemistry method was used for the first time to synthesize copper nanoparticles (Cu-NPs) using CuSO<sub>4</sub> as a precursor and red dragon fruit (*Hylocereus*

costaricensis) peel extract as a bioreductant [7.18]. The obtained Cu-NPs showed efficiency as photocatalysts.

During the harvesting of poplars in the region of Western Kazakhstan, a large amount of unused waste is generated, which can be valorized by developing technologies for obtaining biologically active substances [7.16]. The qualitative composition of extracts from different parts of poplar, as well as from the wood waste, was determined. The results showed that the extract from poplar processing waste contained high concentrations of flavonoids, tannins, saponins, amino acids and polysaccharides. The optimal conditions for extraction were established.

In collaboration with the team of prof. prof. Vitalij Savinkin, “Manash Kozijbaev” University of Petropavlovsk, Kazakhstan [7.14, 7.15], the influence of the phase composition of the material on the recycled components subjected to thermal and mechanical stress was studied. The results are presented in 2 papers with 9 citations.

Recycling of steam turbine blades by laser-plasma treatment is a rational approach to improving the characteristics and reducing the costs of the process. The phase composition of the recovered material determined the durability of the recycled parts. A detailed study of the phase structure of a turbine blade recovered by laser-plasma deposition of  $\text{Al}_2\text{O}_3$  was conducted [7.15]. The design, technological parameters and parameters that control the process of forming an optimal phase structure of a material with improved mechanical properties and increased durability were studied [7.14]. The optimal technological parameters of the laser plasma recovery process were established empirically, which allowed the development of a technological approach for optimizing the phase composition of the recycled material in accordance with the composition of the base material.