

SECTION B: ANALYTICAL AND
ENVIRONMENTAL CHEMISTRY

OBI. TRACE METALS MONITORING IN EXTRACTIVE INDUSTRY WASTES

Anca – Iuliana DUMITRU,¹ Anca Elena Eliza STERPU,² Cristiana
CRAPCEA,¹ and Gabriela Florina STANCIU¹

¹BLUMENFIELD S.R.L., Uzinei Street No. 1, 905700 Navodari, Romania

²Ovidius University of Constanța, 124 Mamaia Blvd., Constanța, Romania

The goal of this study is to develop a comparative evaluation of wastes and wastes leachates generate in extractive industry in order detect the migration of contaminants from hard rock mining operation in ground waters and soils. The study is performed using different types of rocks from different stone quarries located in the Dobrogea area of Romania. The wastes are generated from exploitation of limestone rocks, green systems, and granite. The leaching test are realized using 2 l/kg [1] and 10 l/kg [2] ratios. The monitored parameters are chosen in accordance with the legislation in force and are analyzed using standard test methods. The chemical parameters monitored are total suspended solids, dissolved organic carbon, total organic carbon, chlorides, sulphates, trace metals such as lead, cadmium, total chromium, copper, nickel, barium, molybdenum, zinc [3].

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- [2]. SR EN 12457-2:2003 - Characterization of waste - Leaching - compliance test for leaching of granular waste materials and sludges - one stage batch test at a liquid to solid ratio of 10 l/kg for materials with particle size below 4 mm (without or with size reduction).
- [3]. SR EN ISO 17294-2:2017 - Water quality - Application of inductively coupled plasma mass spectrometry (ICP-MS) - Part 2: Determination of selected elements including uranium isotopes.

PB1. COMPARATIVE STUDY ON THE PHYSICO-CHEMICAL AND ELECTRICAL PROPERTIES OF ABO_3 PEROVSKITES

Paula SFIRLOAGA, Dan ROSU, Maria POIENAR, Mariana STEFANUT,
Ioşif MALAESCU, Catalin Nicolae MARIN, and Paulina VLAZAN

*National Institute for Research and Development in Electrochemistry and
Condensed Matter, Timisoara, P. Andronescu No.1, 300224, Romania*

Perovskite-type materials constitute a wide family of inorganic compounds showing related crystallographic structures and diverse chemical composition, which are based on the general ABO_3 formula. During the past few years, perovskites have received considerable attention from diverse scientific disciplines such as chemistry, materials science or physics because of their multiple also for technological development due their moderate production costs [1]. Another key feature of these materials, which makes them attractive for research purposes, is the possibility of tuning their composition and morphology to show (or to improve) electrical conductivities [2], optical band gaps [3], or catalytic properties [4].

In this paper, a comparative study between Al doped $LaMnO_3$ compounds synthesized by the sol-gel method and ultrasonic method with immersed sonotrode in the reaction medium followed by annealing at 600°C , for 6 h was performed. The obtained materials were studied morpho-structural by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), specific surface (BET), transmission electron microscopy (TEM) and semi-quantitative analysis (EDX). Also, the electrical measurements were performed at room temperature in order to investigate the conduction mechanisms and the influence of Al substitution in $LaMnO_3$ materials.

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PB2. RECYCLING VEGETABLE WASTE, WOOD ASH AND SAWDUST BY COMPOSTING

Ileana MANCIULEA,¹ Lucia DUMITRESCU,¹ Dora LUCACI,² and
Camelia DRAGHICI¹

¹Centre Renewable Energy Systems and Recycling, Transilvania University of
Brasov, Romania

²National Institute for Research and Development in Forestry Marin Dracea,
Brasov, Romania

Sustainable economic development requires the implementation of processes with limited negative impact on the environment and identification of wastewater treatment technologies applicable at industrial level, for pollutants removal up to the limit accepted by standards for their discharge into the emissary. The new economic requirements request researches with high applicability and technologies with low pollution, which determines the obtaining of new ecological and "economic" materials that can be used in the processes of water depollution.

Municipal waste is a growing concern in the world through the large quantity produced every year, the environmental problems and the costs of their storage. Composting is a technique frequently used to recycle a large variety of organic by-products, transforming them into fertilizers for the soil. Recycling process of biodegradable organic waste by composting may represent an ecological solution to obtain new materials used as bio-fertilizers or as ecological adsorbent substrates for toxic metals present in polluted waters. Adsorption is an effective and economical method, used to remove toxic metal ions from aqueous solution due to its simple management, less waste and possible reuse of the adsorbent material. The good results obtained by using, as adsorbent material, the compost obtained by recycling domestic waste, wood ash and sawdust waste seems to confer to composting process new contribution as a sustainable, environmental remediation technology in the future.

The composts obtained during our studies were used for Ni²⁺ and Cu²⁺ removal from wastewater. The adsorbent compost structure and morphology was investigated, before and after adsorption process from used water, by FTIR, AFM, SEM techniques. The adsorption parameters (contact time, the ratio of wastewater volume: adsorbent compost mass) were optimized and both, kinetic and thermodynamic adsorption mechanisms were established. Adsorption of heavy metals on compost substrate represents a sustainable, low-cost process useful in wastewater treatment.

PB3. COMPARATIVE THERMODYNAMIC STUDY OF RETENTION ON VARIOUS STATIONARY PHASES IN REVERSED-PHASE LIQUID CHROMATOGRAPHY

Andreia-Cristina SOARE,¹ Elena PINCU,² Mihaela-Carmen CHEREGI,¹
and Victor DAVID¹

¹ *University of Bucharest, Faculty of Chemistry, Analytical Chemistry Department,
Sos. Panduri, no 90, 050663, Bucharest, Romania*

² *University of Bucharest, Faculty of Chemistry, Department of Physical Chemistry,
Bd. Regina Elisabeta 4-12, Bucharest, 030018, Bucharest, Romania*

The retention behavior of some aromatic hydrocarbons on octyl, octadecyl and phenyl silicagel stationary phases was studied for the temperature interval between 20°C and 50°C. The van't Hoff plots were studied for some mobile phase compositions, using acetonitrile or methanol as organic modifier. The linear regression parameters obtained were used in the calculation of important thermodynamic parameters of the retention process, such as the standard enthalpy variation and standard entropy variation associated with the transfer process of analytes from the mobile phase to the stationary one.

The variation of the standard enthalpy was calculated from the slope of these linear plots and its dependence on the mobile phase composition was rather different for the two organic modifiers. The variation of the standard enthalpy was very small for the four aromatic hydrocarbons in case of using acetonitrile, while for methanol used as organic modifier the variation of the standard enthalpy changed significantly from benzene to propylbenzene.

The variations of the standard entropy and Gibbs free energy change for the hydrocarbons were estimated considering a phase ratio of 0.25 for the columns used.

PB4. METAL NANO-OXIDE BASED COLORIMETRIC SENSORS FOR THE DETERMINATION OF SOME POLYPHENOLS IN PLANTS

Mihaela Carmen CHEREGI,¹ Andrei Florin DANET,¹ Claudia Valentina POPA,² Alina VASILESCU,³ and Simona Carmen LITESCU⁴

¹University of Bucharest, Department of Analytical Chemistry, Sos. Panduri 90-92, 050657, Bucharest,

²University of Bucharest, Department of Organic Chemistry, Biochemistry and Catalysis, Sos. Panduri 90-92, 050657, Bucharest, Romania.

³International Centre of Biodynamics, IB Intrarea Portocalelor, 060101, Bucharest

⁴Centre of Bioanalysis, National Institute for Biological Sciences, 296 Spl. Independentei, 060031 Bucharest

This work presents a novel method for determining the composition of mixtures of natural polyphenolic compounds. The method is based on the formation of colored spots by these compounds upon reaction with different nano-oxides e.g.: CeO₂, TiO₂, MoO₃, MgO, etc. impregnated on filter paper and constituting a colorimetric sensor array.

The image of the colored spots was analyzed, and the intensity of the blue color (BCI) component has shown maximum sensitivity in relation to polyphenolic compounds. The inverse of BCI was linearly correlated with the logarithm of the individual polyphenolic compound concentrations.

By using partial least squares regression for chemometric analysis of 1/BCI values of synthetic binary mixtures of several polyphenolic compounds measured with the colorimetric sensor array, it has been demonstrated good correlation between the actual and the predicted concentration of several polyphenols.

For more complex mixture containing 5 polyphenolic compounds (caffeic acid, gallic acid, quercitrin, ellagic acid, and rosmarinic acid), it was demonstrated that a good correlation between the actual and the predicted concentrations was only in the case of quercitrin. For the other phenols, the colors measured with the colorimetric sensor array were greatly influenced by the concentration of the other components in the mixture. The proposed method for polyphenol determination has advantages that include simplicity, low cost and portability.

PB5. SPECTROFLUORIMETRIC DETERMINATION OF CIPROFLOXACIN AND NORFLOXACIN IN PHARMACEUTICAL FORMULATIONS. APPLICATIONS TO STABILITY STUDIES

Tudorița-Georgiana TOFAN, Andreia-Cristina SOARE, Mihaela-Carmen CHEREGI, Iulia-Gabriela DAVID, and Victor DAVID

University of Bucharest, Faculty of Chemistry, Department of Analytical Chemistry, 90-92 Panduri Av., District 5, Bucharest, Romania

Ciprofloxacin (CIP) and norfloxacin (NOR) are two synthetic fluoroquinolones (FQ) containing α -carbonyl carboxylic acid configuration and belonging to the bacteriophage family [1]. These antibiotics are broad-spectrum antibacterial agents widely used against *Gram-negative* and *Gram-positive* bacteria. CIP is an active drug after oral or intravenous administration,—extensively used for the clinical treatment of human infectious diseases [2]. NOR is administrated in urinary infections with a good localized action on affected sites [3]. Due to long-term *in-vivo* accumulation of residual drugs in human body, CIP and NOR may have carcinogenesis, teratogenesis, mutagenesis potential.

In this work, two fluorimetric methods for the determination of these FQ are presented, based on their intrinsic fluorescence (1) and on fluorogenic chelates formed between analytes and Al(III) (2). For both antibiotics, after performing a spectrometric study, the optimum excitation and emission wavelengths were set at 275 nm and 445 nm, respectively. It was observed that in the presence of Al(III), the fluorescence intensity of the analyte increases and in the emission spectrum, the signals shape is improved. Both developed methods can be used to detect the pure form of CIP and NOR at *sub-ppm* levels with a good reproducibility (RSD < 1 %), to quantify these antibiotics in pharmaceutical tablets and also, to study their stability under different stress conditions (acidic, alkaline, oxidative, photolytic and thermal conditions according to ICH guidelines).

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PB6. ELECTROCHEMICAL APPROACHES FOR ELLAGIC ACID DETERMINATION IN DIETARY SUPPLEMENTS

Vanda-Adriana MITRANĂ, Iulia-Gabriela DAVID, and Mihaela-Carmen CHEREGI

*University of Bucharest, Faculty of Chemistry, Department of Analytical Chemistry,
90-92 Panduri Av., District 5, Bucharest, Romania*

Ellagic acid ($C_{14}H_6O_8$, EA) is a polyphenol dimeric derivative of gallic acid, usually present in ellagitannin and in small quantities as free form [1, 2]. It is abundant in many red fruits but also found in large amounts in wood, nuts and medicinal plants. Extracts from sources containing EA high levels are used as ingredients in traditional medicine, dietary supplements, food and beverages. The EA redox properties might be related to its antioxidant power that retard the progress of many chronic diseases. This work presents a voltammetric approach for EA determination in dietary supplements containing pomegranate extracts.

The EA electro-oxidation mechanism was studied in methanol aqueous media using cyclic voltammetry (CV), differential pulse voltammetry (DPV) and amperometry (AMP). CV was performed to select the best working electrode (vs Ag/AgCl) and to study the scan rate influence on anodic peak intensity (I_{aEA}), for EA determination. Good results were obtained on glassy carbon electrode (GCE) when an EA characteristic anodic peak was observed between 750–800 mV, which separated into two distinct peaks at higher scan rates. The influence of supporting electrolyte pH (1.8–8.8) on EA oxidation was performed using DPV and satisfactory signals were recorded in methanol/ H_2SO_4 0.1 N media = 1/4 (v/v). Under the optimized experimental conditions, I_{aEA} was linearly dependent on EA concentration in the ranges 0.1–7.5 μM in DVP and 0.1–9.78 μM in AMP, respectively. The developed voltammetric method was applied to the EA content determination in commercially available dietary supplements. The obtained results were in good agreement with those of the manufacturer.

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PB7. SIMULTANEOUS ELECTROCHEMICAL DETECTION OF CATECHOLAMINE NEUROTRANSMITTERS AT ACTIVATED PENCIL GRAPHITE ELECTRODE

Mihaela BULEANDRĂ,¹ Corina RĂDUCANU,¹ Iulia Gabriela DAVID,¹
Dana-Elena POPA,¹ Adela Magdalena CIOBANU,^{2,3} Paul LAZĂR,¹ and
Anton Alexandru CIUCU¹

¹University of Bucharest, Faculty of Chemistry, Department of Analytical
Chemistry, Panduri 90-92 Av., Bucharest, Romania

²University of Medicine and Pharmacy "Carol Davila", Faculty of Medicine, Eroii
Sanitari Blvd. 8, Bucharest, Romania

³Clinical Hospital of Psychiatry "Prof. Alexandru Obregia", Berceni Av. 12,
Bucharest, Romania

The selective simultaneous determination of epinephrine (EP) and norepinephrine (NP) by cyclic square wave voltammetry (CSWV) was investigated at electrochemically activated pencil graphite electrode (PGE*).

Based on the electrochemical reversibility of these catecholamines, the reduction peaks of EP and NP oxidation products were taken into consideration for this study. Thus, in 0.1 M PBS (phosphate buffer solution) pH 7.4, one voltammetric peak was obtained at -0.25 V for epinephrine and two well-separated peaks were observed at about $+0.15$ V and -0.25 V for norepinephrine. Therefore, NP determination in the presence of EP is possible by monitoring the peak that appears at $+0.15$ V, where there is no electrochemical signal for EP. On the other hand, EP detection can be done applying the well-known standard addition method exploiting the signal appearing at -0.25 V. EP can be also quantified as the difference between the total content (EP+NP) determined using the peak obtained at -0.25 V and the NP content found as was previously mentioned.

The reduction of all oxidation products was pH dependent, an equal number of protons and electrons being involved in the electrochemical processes. Interference studies showed that the activated electrode has excellent selectivity toward EP and NP in the presence of potential interferents like ascorbic or uric acids.

The developed voltammetric method has been successfully applied to the simultaneous determination of EP and NP from injectable pharmaceutical formulations, obtaining acceptable recoveries.

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PB8. VOLTAMMETRIC INVESTIGATION OF DIOSMIN

Iulia Gabriela DAVID,¹ Alexandra-Gabriela OANCEA,¹ Mihaela BULEANDRĂ,¹ Dana-Elena POPA,¹ Mihaela Carmen CHEREGI,¹ Florentin TACHE,¹ and Adela Magdalena CIOBANU^{2,3}

¹University of Bucharest, Faculty of Chemistry, Department of Analytical Chemistry, Panduri Av. 90-92, Bucharest, Romania

²University of Medicine and Pharmacy "Carol Davila", Faculty of Medicine, Eroii Sanitari Blvd. 8, Bucharest, Romania

³Clinical Hospital of Psychiatry "Prof. Alexandru Obregia", Berceni Av. 12, Bucharest, Romania

The flavonoid glycoside diosmin (DIO) has anti-inflammatory, antioxidant and numerous other beneficial effects on human health. It was introduced in 1969 to treat lymphedema and varicose veins, but it was also employed as a chemopreventive agent in urinary-bladder and colon carcinogenesis [1]. Usually, flavonoids are electroactive compounds and yet there are still few reports on the electrochemical behavior of DIO [2, 3]. This work presents a voltammetric investigation of DIO on the disposable pencil graphite electrode (PGE). Cyclic voltammetry at PGE in acidic media emphasized that in the first potential scan DIO presents two irreversible oxidation signals (~ 0.85 V and ~ 1.3 V vs. Ag/AgCl) and one reduction wave (~ 0.62 V). In the next two cycles one can observe two supplementary pairs of peaks (at ~ 0.46 and ~ 0.66 V) corresponding to pH dependent, quasireversible electrode processes that involve an equal number of electrons and protons. Using the signal from ~ 0.85 V in 0.1 M H₂SO₄, DIO can be determined by differential pulse voltammetry in the concentration range 1×10^{-6} – 1×10^{-5} M with detection (LoD) and quantification limits (LoQ) of 3.40×10^{-7} M and 1.13×10^{-6} M DIO, respectively. Using adsorptive stripping differential pulse voltammetry ($t_{ac} = 60$ s, $E_{ac} = 0, 0$ V) DIO can be quantified between 1×10^{-7} M and 2.5×10^{-6} M with LoD and LoQ of 7.42×10^{-8} M and 2.47×10^{-7} M DIO, respectively.

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PB9. ELECTROCHEMICAL STUDY OF CHLOROGENIC ACID

Dana-Elena POPA, Irina MUNTEANU, Iulia Gabriela DAVID, Mihaela BULEANDRĂ, Vlad Nicolae STOICA, Paul LAZĂR, and Emilia Elena IORGULESCU

*University of Bucharest, Faculty of Chemistry, Department of Analytical Chemistry,
Panduri Av. 90-92, Bucharest, Romania*

Chlorogenic acid (CA) is a polyphenolic compound commonly found in the human diet, present in many fruits, vegetables and in some beverages. CA influences the color, aroma, bitter taste and astringency of foods [1].

A simple, inexpensive, sensitive and rapid voltammetric method was developed using PGE (pencil graphite electrode) as the working electrode. PGE has the major advantages of being single-use, commercially available and inexpensive, also presenting a good reproducibility.

The influence of the solution pH on the electrochemical signals of CA was investigated using cyclic voltammetry (CV) and square wave voltammetry (SWV). The best results were obtained in strong acidic media and it was established that the oxidation process involves an equal number of transferred protons and electrons. The appropriate supporting electrolyte was found to be Britton Robinson buffer pH 1.81.

The voltammetric behavior of the CA was studied by CV on PGE at different scan rates between 10 and 1000 mV/s. The applied diagnosis criteria led to the conclusion that the anodic peak is generated by a quasi-reversible process, controlled by diffusion.

In order to perform the CA quantitative determination, the working parameters in SWV were optimized: pulse amplitude (50 mV), amplitude of the potential step (1 mV) and duration of the potential step (0.02 s). The intensity of the peak current varied linearly with the CA concentration in the range $1 \cdot 10^{-7}$ M - $1 \cdot 10^{-4}$ M. The limit of detection ($9.55 \cdot 10^{-8}$ M) and the limit of quantification ($2.89 \cdot 10^{-7}$ M) were calculated.

The developed method was successfully applied to the analysis of real samples (commercially available Green Coffee food supplements), the results obtained by SWV on PGE being in good agreement with the values declared by the manufacturer.

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PB10. SIMPLE AND SENSITIVE VOLTAMMETRIC DETERMINATION OF LAMOTRIGINE

Mihaela BULEANDRĂ¹, Vlad Nicolae STOICA¹, Tatiana CIUREA², Dana Elena POPA¹, Iulia Gabriela DAVID¹ and Anton Alexandru CIUCU¹

¹ University of Bucharest, Faculty of Chemistry, Department of Analytical Chemistry, Panduri 90-92 Av., 050663, Bucharest, Romania

² Emergency Clinical Hospital "Bagdasar-Arseni", Berceni 12 Av., 041915, Bucharest, Romania

Lamotrigine (LTG), a phenyltriazine derivative, is an anticonvulsant used in the therapy of patients with epilepsy, bipolar disorder or dementia [1]. In the present study the electrochemical method for the determination of lamotrigine at activated pencil graphite electrode (PGE*) was optimized. Cyclic voltammograms of LTG in Britton-Robinson buffer (BRB) solution pH 4.6 exhibited an oxidation signal at +0.35 V and a reduction response at -0.96 V (vs. Ag/AgCl).

The oxidation peak, attributed to the LTG molecule dimerization, was used to develop a simple linear sweep voltammetric (LSV) method for the drug quantification. A dynamic linear range of $2.5 \cdot 10^{-5}$ – $1 \cdot 10^{-3}$ M LTG was obtained. The detection and quantification limits were calculated as $1.94 \cdot 10^{-5}$ M and $5.89 \cdot 10^{-5}$ M, respectively. The practical application of the sensor was demonstrated by determining the concentration of LTG in pharmaceutical samples with good precision and acceptable recoveries.

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PB11. NON-INVASIVE TECHNIQUES FOR CHARACTERIZATION OF ORIGINAL ROMAN MOSAIC FRAGMENTS

Cristiana RADULESCU*,^{1,2} Rodica Mariana ION,^{3,4} Ioana Daniela DULAMA,² Anca Irina GHEBOIANU,² Claudia STIHI,^{1,2} Geanina Sorina STANESCU,² Raluca Maria STIRBESCU,² Cristina Mihaela NICOLESCU,² Ioan Alin BUCURICA,² Ion Valentin GURGU,² Radu Lucian OLTEANU,² Dorin Dacian LET,² Liviu OLTEANU,² and Nicolae STIRBESCU²

¹Valahia University of Targoviste, Faculty of Sciences and Arts, 130004 Targoviste, Romania

²Valahia University of Targoviste, Institute of Multidisciplinary Research for Science and Technology, 130004 Targoviste, Romania

³Valahia University of Targoviste, Materials Engineering Department, Doctoral School of Materials Engineering, 130105 Targoviste, Romania

⁴National R&D Institute for Chemistry and Petrochemistry–ICECHIM, Research Group “Evaluation and Conservation of Cultural Heritage”, 060061 Bucharest, Romania

*radulescucristiana@yahoo.com

In this study, it was reported the preliminary results on the chemical and structural composition of remains of decorative elements of original Roman mosaic fragments collected from the Roman Mosaic Museum, Constanta (Romania). These investigations were carried out by using non-destructive and micro-invasive techniques such as Optical Microscopy, X-Ray Diffraction, Field Emission - Scanning Electron Microscopy - Energy Dispersive X-Ray Spectroscopy, Raman Spectroscopy. The fragments studied in this work, apart from being beneficial to different restoration opportunities of this Roman mosaic, could also be included in its modification through air pollution. The major and minor phase components of the studied mosaic fragments were determined, the crystal structure of the main phases was analyzed, and their three-dimension spatial arrangement was reconstructed. The similar composition of the major phases of all mosaic fragments can indicate a generic recipe for making mosaic elements, but minor phases were presumably added for coloring of mosaic pieces. Some degradation areas inside the volume of the mosaic fragments were found by means of the X-ray diffraction method. These degradation areas are probably related to the formation of iron hydroxides during chemical interactions of mosaic fragments with the sea and urban polluted atmosphere. The results also can offer important information about the original materials that were used in the Roman period.

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PB12. MONITORING OF PHYSICO-CHEMICAL PARAMETERS OF GROUDWATERS FROM DOBROGEA AREA

Alina_SOCEANU, Simona DOBRINAS, Viorica POPESCU, and
Georgiana VIZITIU

*Department of Chemistry and Chemical Engineering, University "Ovidius" of
Constanta, 124 Mamaia Blvd, 900527, Constanta, Romania*

One of the most important environmental issue is groundwater contamination [1-3]. The aim of the work was to estimate the quality of some groundwaters from the Dobrogea area. Physico-chemical parameters like: pH, chlorides, nitrites, hardness, turbidity, alkalinity, conductivity, ammonium and nitrates were analyzed. The values of the parameters: chlorides, conductivity, hardness and nitrates for certain analyzed samples exceed the maximum levels allowed by the legislation. The metal content in chromium, cadmium, copper, iron, manganese, nichel, lead and zinc was also determinate using atomic absorption spectrometry with graphite furnace. The concentrations of the eight studied metals were within the maximum allowed limits established by the Romanian legislation. The Health Risk Index (HRI) was also estimated as the ratio of daily metal intake (MRI) to mean reference metal dose (RfD).

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PB13. OPEN EDUCATIONAL RESOURCES FOR LEARNING WASTE MANAGEMENT IN RURAL COMMUNITIES

Cristina SALCA ROTARU¹ and Camelia DRAGHICI²

¹*Transilvania University of Brasov, Faculty of Law, 29 Eroilor Blv., 500036 Brasov, Romania*

²*Transilvania University of Brasov, Faculty of Product Design and Environment, 29 Eroilor Blv., 500036 Brasov, Romania*

The organisations involved in the waste management can be of three different types: (i) as organisations acting in the waste management sector (either public or private) (ii) as institution with activity of public interest carried out by administration authorities and (iii) as ordinary waste-producing organisations (almost any organization).

Starting with 2019 the EU Commission Decision no. 2019/61 was launched, “on the best environmental management practices, sector environmental performance indicators and benchmarks of excellence *for the public administration sector*”. Further, in 2020 a new EU Commission Decision no. 2020/519 was launched, “on best environmental management practices, sector environmental performance indicators and benchmarks of excellence *for the waste management sector*”. Both decisions were under the Regulation (EC) no. 1221/2009 “on the voluntary participation by organisations in a Community eco-management and audit scheme (EMAS)”. The harmonization of the two decisions is needed, so that their effective implementation should avoid the overload of the activity of the local public administration, which sometimes can be identified in all the three hypostases of organisations dealing with waste management.

Transilvania University of Brasov (Romania) as coordinator, and three more higher educational institutions (HEIs), Reykjavik University (Iceland), Bucharest University of Economic Studies (Romania) and Gheorghe Asachi Technical University of Iasi (Romania) will carry out the project with the title Environmental Education – OERs for Rural Citizens (EnvEdu – OERs). During this project, six modules as Open Educational Resource (OERs) will be developed, of which one module will be Waste Management in Rural Communities.

Thus, the present study aims to highlight the opportunities and barriers identified to the implementation of the two EU Decisions at the level of local rural public authorities.

PB14. SUSTAINABLE COMMUNITIES – LEVELS OF ANALYSIS

Dana PERNIU and Camelia DRAGHICI

*Transilvania University of Brasov, Faculty of Product Design and Environment,
29 Eroilor Blv., 500036 Brasov, Romania*

Now-a-days the entire planet is facing with rapid urbanization. The environment provides living opportunities, but if these are altered, constraints on ensuring the living needs are registered. Consequently, challenges in terms of maintaining environmental quality faces politicians, administrative, scientific actors and also regular citizens. Sustainable development, as primary defined four decades ago, guides the anthropic activity to be restrained in between the limits of the planet. Since then, actions and steps were performed, but urban environmental quality remains on discourse agenda.

In 2015, the United Nations (UN) set 17 life-changing goals, known as Sustainable Development Goals (SDGs), and the 11th goal requires to “make cities and human settlements inclusive, safe, resilient and sustainable”. Thus, the urban sustainability is a complex concept, it requires new ways of thinking and approaching the actions at different levels of socio-economic system.

The present paper discusses the concept of urban sustainability from the point of view of interaction between individuals and environment, at different spatial levels: micro-level (individual), mezzo-level (neighborhood), macro-level (regional/ national) and global-level. The levels are discussed and exemplified in terms of decisions regarding actions and their environmental impact, in the frame of development of sustainable communities.