University of Belgrade, Technical Faculty in Bor

29th International Conference Ecological Truth & Environmental Research

EcoTER'22

Proceedings

Editor
Prof. Dr Snežana Šerbula

21-24 June 2022, Hotel Sunce, Sokobanja, Serbia

University of Belgrade, Technical Faculty in Bor

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PREFACE

In today's world, the environment has been endangered by the use of outdated technology, fossil fuels and environmental law violations. Therefore, environmental and many other scientists all over the world have been concerned about finding sustainable technology in resolving these issues. That is why environmental research and ecological truth are at the focus of the 29th International Conference Ecological Truth & Environmental Research 2022 (EcoTER'22), which will be held in Sokobanja, Serbia, 21–24 June 2022. On behalf of the Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the Conference.

We hope to convey the message of the conference, which is that a transformation of attitudes and behavior would bring the necessary changes. This is also an opportunity for the participants who are experts in this field to exchange their experiences, expertise and ideas, and also to consider the possibilities for their collaborative research.

The 29th International Conference Ecological Truth & Environmental Research 2022 is organized by the University of Belgrade, Technical Faculty in Bor, and co-organized by the University of Banja Luka, Faculty of Technology, the University of Montenegro, Faculty of Metallurgy and Technology – Podgorica, the University of Zagreb, Faculty of Metallurgy – Sisak, the University of Pristina, Faculty of Technical Sciences – Kosovska Mitrovica and the Association of Young Researchers, Bor.

These proceedings include 85 papers from the authors coming from the universities, research institutes and industries in 6 countries: Bulgaria, Italia, Albania, Bosnia and Herzegovina, Montenegro and Serbia.

As a part of this year's conference, the 4th Student section – EcoTERS'22 is being held. We appreciate the contribution of the students and their mentors who have also participated in the Conference.

Financial assistance provided by the Ministry of Education, Science and Technological Development of the Republic of Serbia is gratefully acknowledged by the Organizing Committee of the EcoTER'22 conference.

The support of the Platinum donor and their willingness and ability to cooperate have been of great importance for the success of EcoTER'22. The Organizing Committee would like to extend their appreciation and gratitude to the Platinum donor of the Conference for their donation and support.

We appreciate the effort of all the authors who have contributed to these Proceedings. We would also like to express our gratitude to the members of the scientific and organizing committees, reviewers, speakers, chairpersons and all the Conference participants for their support to EcoTER'22. Sincere thanks go to all the people who have contributed to the successful organization of EcoTER'22.

Prof. Snežana Šerbula,

President of the Organizing Committee

TABLE OF CONTENTS

Plenary Lectures

Marija Petrović Mihajlović, M. Antonijević	
PURINES AS GREEN CORROSION INHIBITORS	3
Milan Radovanović, M. Antonijević	
ENVIRONMENTALLY SAFE CORROSION INHIBITORS: AMINO ACIDS	12
Hyusein Yemendzhiev, Y. Mersinkova, R. Koleva, G. Peeva, V. Nenov	
BIO-ELECTROCHEMICAL SYSTEMS – STATE OF THE ART: BIOLOGY, ELECTROCHEMISTRY AND APPLICATION IN WASTEWATER MANAGEMENT	25
Aleksandar Marinković	
GREEN ORGANIC CHEMISTRY: A FRAMEWORK FOR SUSTAINABLE ENVIRONMENTAL PROTECTION	32

Conference Papers

Sanja Mrazovac Kurilić, A. Ćirišan, Z. Podraščanin, Lj. Nikolić Bujanović	
SO_2 POLLUTION IN ŠABAC (2009–2020)	39
Ana Čučulović, J. Stanojković, R. Čučulović, S. Nestorović, N. Radaković	
THE DISTRIBUTION OF THE MASS CONCENTRATIONS OF K, Th AND U IN THE SOILS OF THE TEKIJA REGION, THE NP ĐERDAP	43
Jovana Bošnjaković, M. Bugarčić, M. Milošević, N. Prlainović, R. Salih, P. Batinić, A. Popović, M. Đolić	
APPLICATION OF NANO- MnO_2 MODIFIED LIGNIN – BASED ADSORBENT FOR REMOVAL OF DICHROMATE IONS AND DICLOFENAC FROM WATER	49
Branislava Matić, S. Živković Perišić, D. Jovanović, S. Dejanović, D. Miljuš, L. Kukobat	
MAPPING HEALTH RISKS OF CRITERIA CHEMICAL(S) TO DEMONSTRATE BENEFITS OF RISK ASSESSMENT FOR DECISION-MAKERS	55
Violeta Babić, B. Kanjevac, M. Milenković, S. Stajić, M. Vukin, N. Stavretović, M. Račić	
CHARACTERISTCS OF WINTER TEMPERATURE REGIME IN SPRUCE FOREST ON KOPAONIK	62
Dragana Pavlović, D. Čakmak, V. Perović, M. Matić, M. Marković, M. Mitrović, P. Pavlović	
ENVIRONMENTAL RISK ASSESSMENT OF PTES IN AGRICULTURAL SOILS AFFECTED BY INDUSTRIAL ACTIVITIES IN BELGRADE	68
Olga Kostić, G. Gajić, S. Jarić, Z. Mataruga, D. Sekulić, N. Radulović, M. Mitrović, P. Pavlović	
ANALYSIS OF As AND Pb ACCUMULATION IN GARDEN SOIL AND VEGETABLE CROPS IN THREE BELGRADE MUNICIPALITIES	75

Marija Ječmenica Dučić, D. Vasić Anićijević, B. Savić, D. Aćimović, M. Simić, D. Maksin, T. Brdarić	
NEW STRATEGIES FOR DEVELOPMENT OF HIGHLY SELECTIVE MATERIALS FOR CARBON DIOXIDE CAPTURE	81
Miljan Bigović, D. Đurović, I. Nikolić, Lj. Ivanović, B. Bajić	
CHARACTERISTIC, SOURCE AND ECOLOGICAL RISK OF PAHs IN AGRICULTURAL SOILS PLJEVLJA MUNICIPALITY (MONTENEGRO)	85
Jelena Vranković, K. Jovičić, V. Đikanović	
EFFECT OF DIFFERENT ENVIRONMENTAL CONDITIONS ON LIPID PEROXIDATION LEVEL IN *Rutilus rutilus* (ACTINOPTERYGII: CYPRINIDAE)	91
Aurora Bakaj (Çizmja), M. Lika (Çekani)	
EVALUATION OF MICROBIAL ENVIRONMENT ON THE BEACH SAND	96
Dragana Ranđelović, R. Pantović	
IMPACT OF THE MINING ACTIVITIES IN THE GOLIJA NATURE PARK AREAS SITUATED ON THE TERRITORY OF RAŠKA MUNICIPALITY	104
Bojana Tubić, K. Zorić, N. Popović, M. Raković, N. Marinković, M. Paunović	
INDICATIVE ECOLOGICAL STATUS ASSESSMENT OF SELECTED STREAMS ON ROGOZNA MOUNTAIN BASED ON AQUATIC MACROINVERTEBRATES	110
Božica Vasiljević, J. Tomović, A. Atanacković, R. Petrović	
INDICATIVE ECOLOGICAL STATUS ASSESSMENT BASED ON EPILITHIC DIATOMS OF SMALL RIVERS AT ROGOZNA MOUNTAIN	116
Đuro Čokeša, M. Marković, N. Potkonjak, B. Kaluđerović, S. Radmanović, S. Šerbula	
ARSENITE–SOIL HUMIC ACID BINDING BY ISOTHERMAL TITRATION CALORIMETRY: THERMODYNAMICS AND MNIS MODEL	121
Tatjana Anđelković, I. Kostić Kokić, B. Zlatković, D. Anđelković	
Cu(II) ACCUMULATION POTENTIAL OF AQUATIC MACROPHYTE *PISTIA STRATIOTES*	127
Tatjana Anđelković, D. Bogdanović, I. Kostić Kokić, H. Kocić, G. Kocić	
PHTHALATES MIGRATION FROM ABSORABLE SURGICAL SUTURES INTO MODEL SOLUTION	132
Aleksandra Papludis, S. Alagić, S. Milić, I. Zlatanović, M. Filipović, J. Nikolić, V. Stankov Jovanović	
THE CONTENT OF DANGEROUS CONTAMINANTS PAHs IN THE SOIL AND ROOTS OF *HEDERA HELIX* IN SLATINA (BOR'S MUNICIPALITY)	137
Jelena Milosavljević, S. Šerbula, A. Radojević, T. Kalinović, J. Kalinović, B. Spalović	
TOXIC METALS BIOACCUMULATION IN *Plantago lanceolata* FROM ANTHROPOGENICALLY DISRUPTED AREA	142
Mirjana Ocokoljić, Dj. Petrov, N. Galečić, M. Miodrag	
ECOLOGICAL AND AESTHETIC CHARACTERISTICS OF TREES IN "BELGRADE WATERFRONT" PARKING LOTS	149
Mirjana Ocokoljić, Dj. Petrov, A. Tutundzić, D. Skočajić, S. Petrović	
STATE, FUNCTIONS AND PERSPECTIVES OF DENDROFLORA IN THE SCHOOLYARD OF THE MEDICAL SCHOOL IN BELGRADE	154

Mirjana Ocokoljić, Dj. Petrov	
TRACHYCARPUS FORTUNEI (HOOK.) H. WENDL. IN THE CONDITIONS OF CHANGED TEMPERATE CONTINENTAL CLIMATE IN BELGRADE	159
Marija Perkunić, S. Vukić, Z. Živković, V. Stupar, M. Saulić	
GREEN OASIS EFFECT ON URBAN ENVIRONMENT	164
Anđela Stojić, D. Tanikić	
APPLICATION OF GREEN AREAS AND GREEN ROOFS IN URBAN AREAS	169
Gordana Šekularac, T. Ratknić, M. Aksić, N. Gudžić, M. Vranešević, M. Ratknić	
SOIL EROSION ASSESSMENT USING EPM: A CASE STUDY IN THE CATCHMENT AREA OF THE TURKISH BROOK, WESTERN SERBIA	174
Nataša Knežević, A. Jovanović, R. Salih, Z. Veličković, A. Popović, P. Batinić, A. Marinković, J. Gržetić	
MODIFIED LIGNIN-BASED MICROSPHERES AS A GREEN SORBENT FOR THE REMOVAL OF CHROMIUM IONS	180
Aleksandar Jovanović, N. Knežević, N. Čutović, M. Đolić, N. Prlainović, Z. Veličković, M. Vuksanović	
IMPROVED TECHNOLOGY FOR PRODUCTION OF PE AND PP REGRANULATES	186
Jasmina Dedić, M. Mojsić, D. Lazarević, B. Stojčetović, Ž. Šarkoćević	
A SHORT REVIEW OF TREPČA MINING WASTE IMPACT ON ENVIRONMENT	190
Senad Čergić, H. Husić, V. Aganović	
INFLUENCE OF UNDERGROUND MINING WORKS OF THE OMAZIĆI BROWN COAL MINE BANOVIĆI ON THE SURFACE TERRAIN	195
Snežana Šerbula, J. Milosavljević, T. Kalinović, A. Radojević, J. Kalinović	
ARSENIC IN PARTICULATE MATTER ORIGINATED FROM MINING-METALLURGICAL PROCESSES	202
Nevena Ristić, M. Veličković, D. Voza	
THE ASSOCIATION BETWEEN SHORT-TERM EXPOSURE TO SO_2 AND EMERGENCY ROOM ADMISSIONS IN URBAN AREA. CASE STUDY SERBIA	208
Miljan Marković, M. Gorgievski, N. Štrbac, K. Božinović, V. Grekulović, A. Mitovski, M. Zdravković	
ADSORPTION ISOTHERMS FOR COPPER IONS BIOSORPTION ONTO WALNUT SHELLS	214
Tanja Kalinović, J. Kalinović, S. Šerbula, J. Milosavljević, A. Radojević	
DETECTION OF THE TRAFFIC-RELATED POLLUTION BY THE ROADSIDE SOIL AND PLANT MATERIAL	219
Vladan Nedelkovski, S. Stanković, M. Radovanović, M. Antonijević	
SYNTHESIS AND CHARACTERISATION OF Ti/SnO_2-Sb-TYPE DSA ANODES FOR WASTEWATER TREATMENT	226
Maja Radić, M. Avdagić, B. Markovic, K. Ademović, S. Avdagić, S. Avdagić	
PROTECTION AND PRESERVATION OF NATURAL RESOURCES	233
Mihajlo Stanković	
Annex 2: FAUNA OF MACROCRUSTACEA (INVERTEBRATA-ARTHROPODA) IN PERMANENT AND EPHEMERAL WATERS OF ZASAVICA WETLAND (SPECIAL NATURE RESERVES)	239

Mihajlo Stanković	
OVERVIEW OF GALIS FAUNA (DIPTERA, HOMOPTER AND HYMENOPTER) – ZASAVICA RESERVE	246
Vanja Marković, M. Božanić, O. Arapović, V. Gojšina, A. Tatović, K. Stojanović	
AQUATIC INSECTS DIVERSITY IN THE PART OF NERETVA RIVER DRAINAGE (BOSNIA AND HERZEGOVINA): PRELIMINARY RESULTS	253
Aleksandar Savić, I. Jelić, M. Šljivić-Ivanović, S. Dimović	
RECYCLED CONCRETE AGGREGATE EFFECT ON SELF-COMPACTING CONCRETE AT LOW TEMPERATURES	258
Ivana Bjedov, D. Obratov-Petković, V. Stojanović, M. Nešić, D. Marisavljević	
INVASIVE ALIEN SPECIES IN SERBIA: LEGISLATION, STRATEGY, AND PLANS	264
Jelena Majstorovic, M. Korac, S. Savkovic	
THE EFFECTS OF GEOMECHANICAL RESEARCH "*IN SITU*" ON THE EXPLOITATION CONDITIONS OF THE OPEN PIT BAUXITE MINE "ZAGRAD"	271
Isidora Berežni, T. Marinković, V. Bežanović, M. Živančev, B. Batinić	
ANALYSIS OF HOUSEHOLD'S E-WASTE AWARENESS, AND DISPOSAL BEHAVIOR IN NOVI SAD	276
Maja Radić, M. Avdagić, B. Markovic, K. Ademović, S. Avdagić, S. Avdagić	
ECOLOGICAL ETHICS AND ECOLOGICAL EDUCATION	284
Vanja Marković, K. Zorić, M. Ilić, S. Đuretanović, M. Smederevac-Lalić	
STONE CRAYFISH *AUSTROPOTAMOBIUS TORRENTIUM* (Schrank, 1803) IN SOUTH-EASTERN SERBIA: CHILDREN KNOWLEDGE SURVEY	290
Ana Radojević, J. Milosavljević, T. Kalinović, J. Kalinović, S. Šerbula	
THE IMPACT OF TEXTILE AND CLOTHES PRODUCTION ON THE ENVIRONMENT – PART I: ENVIRONMENTAL ISSUES	295
Ana Radojević, J. Milosavljević, T. Kalinović, J. Kalinović, S. Šerbula	
THE IMPACT OF TEXTILE AND CLOTHES PRODUCTION ON THE ENVIRONMENT – PART II: WHAT CAN WE DO?	301
Giorgia Santini, V. Memoli, L. Santorufo, G. Di Natale, G. Maisto	
EFFECTS OF UN-BIODEGRADABLE AND BIODEGRADABLE PLASTIC SHEETS ON ABIOTIC SOIL PROPERTIES	307
Milica Rajačić, D. Todorović, J. Krneta Nikolić, I. Vukanac, N. Sarap, M. Janković	
RADIONUCLIDE CONTENT IN SAMPLES OF BERRIES	313
Ivana Mikavica, D. Ranđelović, J. Stojanović, J. Mutić	
MICROPLASTIC OCCURRENCE IN URBAN AND SUBURBAN SOILS OF BOR, EASTERN SERBIA	319
Maja Nujkić, V. Stiklić, Ž. Tasić, S. Milić, D. Medić, A. Papludis, I. Đorđević	
BIOSORPTION OF METAL IONS FROM SYNTHETIC SOLUTIONS USING DIFFERENT PARTS OF PLANT MATERIAL – A REVIEW	325
Nataša Đorđević, S. Mihajlović	
ANALYSIS OF CHANGES OF MECHANICALLY ACTIVATED SODIUM CARBONATE SAMPLE	331
Vanja Trifunović, S. Milić, Lj. Avramović, R. Jonović, S. Đorđievski	
ELECTRIC ARC FURNACE DUST – HAZARDOUS INDUSTRIAL WASTE WHOSE TREATMENT IS UNAVOIDABLE	336

Goran Milentijević, T. Marinković, M. Rančić, M. Milošević, I. Đuričković, A. D. Marinković, M. M. Milosavljević	
OPTIMIZATION OF ZINC DIMETHYLDITHIOCARBAMATE SYNTHESIS PROCESS (ZIRAM)	343
Miloš Prokopijević, D. Spasojević, O. Prodanović, N. Pantić, D. Bartolić, K. Radotić, R. Prodanović	
STABILITY OF SOYBEAN PEROXIDASE IMMOBILIZED ONTO HYDROGEL MICRO-BEADS FROM TYRAMINE-PECTIN	350
Nevena Pantić, M. Spasojević, M. Prokopijević, D. Spasojević, A. M. Balaž, R. Prodanović, O. Prodanović	
COVALENT IMMOBILIZATION OF HORSERADISH PEROXIDASE ON NOVEL MACROPOROUS POLY(GMA-CO-EGDMA) FOR PHENOL REMOVAL	354
Dragica Spasojević, M. Prokopijević, O. Prodanović, N. Pantić, M. Stanković, K. Radotić, R. Prodanović	
PREPARATION OF CROSSLINKED TYRAMINE-ALGINATE HYDROGEL USING EDC/NHS WITH SELF-IMMOBILIZED HRP	360
Jelena Mitrović, M. Radović Vučić, N. Velinov, S. Najdanović, M. Kostić, M. Petrović, A. Bojić	
THE ROLE OF HYDROXYL AND SULFATE RADICALS IN THE UV ACTIVATED PERSULFATE DEGRADATION OF TEXTILE DYE RO16	364
Milica Petrović, T. Jovanović, S. Rančev, M. Radović Vučić, J. Mitrović, S. Najdanović, A. Bojić	
ELECTROSYNTHESIZED CERIUM OXIDE CATALYST FOR ATMOSPHERIC PRESSURE PULSATING CORONA PLASMA DEGRADATION OF RB 5	369
Slobodan Najdanović, M. Petrović, M. Kostić, N. Velinov, J. Mitrović, D. Bojić, A. Bojić	
PHOTOCATALYTIC DEGRADATION OF RANITIDINE BY BISMUTH OXO CITRATE	375
Ana Simonović, Ž. Tasić, M. Radovanović, M. Petrović Mihajlović, M. Antonijević	
CAFFEINE AS A GREEN CORROSION INHIBITOR FOR COPPER IN SYNTHETIC BLOOD PLASMA SOLUTION	381
Dragana Medić, S. Milić, S. Alagić, M. Nujkić, A. Papludis, S. Đorđievski, S. Dimitrijević	
RECYCLING GOLD FROM WASTE PRINTED CIRCUIT BOARDS	387
Uroš Stamenković, S. Ivanov, I. Marković	
CHARACTERIZATION OF CARBON AND LOW-ALLOY STEEL AFTER DIFFERENT HEAT TREATMENTS	393
Violeta Babić, M. Milenković, Z. Govedar, S. Stajić, B. Kanjevac	
THE FOREST FIRES IN BULGARIA: THE TRENDS AND THE INFLUENCE OF TELECONNECTIONS	400
Martina Petković, M. Božović, A. Klikovac, D. Knežević	
THE IMPORTANCE OF FIRE PROTECTION ON SHIPS	406
Darko Stojićević, Z. Živković, M. Saulić, T. Sekulić, V. Stupar	
PESTICIDES – IMPACT ON HUMAN HEALTH AND THE ENVIRONMENT	413
Zlata Živković, M. Saulić, D. Stojićević, M. Jevtić Đorović, V. Stupar	
ORGANIC AGRICULTURE: POTENTIAL OF THE FUTURE	419

Tatjana Anđelković, K. Kitanović, I. Kostić Kokić, B. Zlatković, D. Bogdanović	
CHANGES IN NITRATE AND NITRITE CONTENT IN FOR LETTUCE, CHARD AND SPINACH AFTER FREEZING	425
Ivana Perović, S. Brković, N. Zdolšek, G. Tasić, M. Seović, S. Mitrović, J. Ciganović	
CAN LASER MODIFICATION OF COATED ELECTRODES IMPROVE THE HYDROGEN EVOLUTION REACTION IN ALKALINE ELECTROLYSERS?	430
Nikola Zdolšek, S. Brković, I. Perović, M. Ćurčić, S. Dimović, M. Vujković	
NEW GENERATION OF ELECTROCHEMICAL SUPERCAPACITORS	436
Nebojša Potkonjak, Đ. Čokeša, M. Marković	
THE HYDROGEN PRODUCTION ON Ni ELECTRODE CO-DEPOSITED WITH Co+V_2O_5: THE ELECTROCALYTIC SYNERGETIC EFFECT	441
Tatjana Ratknić, G. Šekularac, M. Ratknić, Z. Poduška, M. Aksić	
EFFECTS OF CLIMATE CHARACTERISTICS ON THE DIAMETER INCREMENT OF CEDAR IN THE CITY OF BELGRADE (SERBIA)	446
Natalija Čutović, M. Vuksanović, M. Milošević, M. Bugarčić, J. Bošnjaković, J. Gržetić, A. Marinković	
RECYCLED POLY(ETHYLENE TEREPHTHALATE) BASED-PLASTICIZER FOR PVC REGRANULATES PRODUCTION	452
Robert Vigi, G. Štrbac, D. Štrbac, M. Novaković	
SYNTHESIS OF NEW Ag DOPED CHALCOGENIDE GLASS FOR APPLICATION IN ENERGY CONVERSION AND MEMORY STORAGE	459
Tatjana Miljojčić, I. Jelić, M. Šljivić-Ivanović, S. Dimović, U. Ramadani	
SUSTAINABLE UTILIZATION OF CATHODE-RAY TUBE WASTE GLASS IN CEMENTITIOUS MATERIALS – A REVIEW	465
Dejan Riznić, A. Fedajev, A. Jevtić	
ECOTURISM AS A FORM OF GREEN ECONOMY	471
Vesna Đikanović, J. Vranković, K. Jovičić	
DIET OF TWO CYPRINID SPECIES, WHITE BREAM (BLICCA BJOERKNA) AND COMMON ROACH (RUTILUS RUTILUS) IN THE DANUBE RIVER, BELGRADE	477
Aleksandra Radić, D. Voza, Đ. Nikolić, M. Vuković	
EVNIRONMENTAL PERFORMANCE CLASSIFICATION OF BALKAN COUNTRIES BASED ON TOPSIS-SORT METHODOLOGY	483
Marijana Pantić, T. Maričić, S. Milijić	
CITIZEN PARTICIPATION OFFERS LESSONS TO CLIMATE CHANGE MANAGEMENT	489
Tanja Brdarić, D. Aćimović, B. Savić, N. Abazović, M. Čomor, M. Ječmenica Dučić, D. Maksin	
INVESTIGATION OF ZrO_2 AND ZrO_2/TiO_2 ELECTRODES BY CYCLIC VOLTAMMETRY	495
Branislava Savić, D. Aćimović, T. Brdarić, M. Ognjanović, D. Vasić Anićijević, M. Ječmenica Dučić, M. Simić	
TESTING THE ELECTROCHEMICAL BEHAVIOR OF BPA ON GC, WO_3 AND MWCNT ELECTRODES	499
Marijola Božović, M. Petković, S. Marković, B. Stojčetović	
DISASTER RISK MANAGEMENT FRAMEWORK	504
Author Index	510

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ANALYSIS OF CHANGES OF MECHANICALLY ACTIVATED SODIUM CARBONATE SAMPLE

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Abstract

For mechanical activation of sodium carbonate, a vibro mill at a frequency of 3000 oscillations per minute was used for 28 minutes. After activation, samples were placed for 31 days in the air at room conditions. In aim to monitor the kinetics of the transformation process of sodium carbonate to bicarbonate, Fourier-transform infrared (FTIR) spectroscopy analysis was used. As a function of relaxation time characteristic groups were observed: CO_3^{2-}, HCO_3^- and OH^-. The obtained results provided kinetics parameters for the transformation of carbonate into bicarbonate as a result of chemisorption of moisture and carbon dioxide from the atmosphere.

Keywords: sodium carbonate, mechanical activation, mechanochemical transformation

INTRODUCTION

In the world, the activation of materials is widely used as a procedure for bringing energy to the system so that it begins to react in a controlled manner. This allows the system to skip over the "energy barrier" on the reaction path [1,2]. The method of mechanical activation, which, in various technical and technological ways, brings mechanical energy to solid materials thus changing and regulating many physico-chemical characteristics related to increasing the system's reactivity, is becoming more and more topical [3,4]. With the development of this procedure, many research fields related to obtaining new materials and monitoring the kinetics of the solid phase reactions have been opened and significant savings compared to classical methods of material activation achieved [5].

MATERIALS AND METHODS

The sodium carbonate used in this study was anhydrous and in p.a. quality. According to the MERCK index 11.8541, the chemical composition of sodium carbonate is: 99.5% Na_2CO_3, 0.002% Cl^-, 0.005% SiO_2, 0.0005% Pb, 0.0005% Fe, 0.01% Al, 0.0005% Mg, 0.01% K, 0.005% SO_4^{2-}, 0.005% Ca, and 0.5% loss on drying at 180°C; specific mass 2500 kg/m^3; decomposition temperature 851°C. Na_2CO_3 has a monoclinic crystal lattice (a=8.907, b=5.239, c=6.043); the bond energy values are: Na–O (364 kJ/mol), C–O (1076.4 kJ/mol), C=O (532.2 kJ/mol).

Sodium carbonate was mechanically activated in the high-energy mill KHD Humboldt Wedag AG. Samples in the amount of 50 g each were activated for 28 minutes (activation

time) at a frequency of 3000 oscillations per minute thus obtaining four series for further investigation. Relaxation time was 31 days.

The effects of mechanical activation were monitored by FTIR spectroscopy analysis. FTIR spectroscopy is a very important non-destructive method, given the high sensitivity and precision of frequencies, excellent resolution, quick recording, and possibility to repeat recording as well as different ways to interpret the obtained data. Changes were observed on the characteristic groups: CO_3^{2-}, HCO_3^- and OH^-. FFT infrared spectra of the samples were recorded on a spectrometer Bomem-Hartman & Braun Michelson MB-100, in the wave numbers range of 4000–300 cm^{-1} and at a resolution of 2 cm^{-1}. Samples were prepared in the form of a suspension using "Nujol". Changes in the peaks' areas, as well as the widths at the half-heights, were observed for the given characteristic groups: CO_3^{2-}, HCO_3^{2-} and OH^-. The results are presented as a function of relaxation time for all four series, i.e. for different activation periods.

RESULTS AND DISCUSSION

FTIR spectrogram of the initial, non-activated sodium carbonate sample is presented in Figure 1.

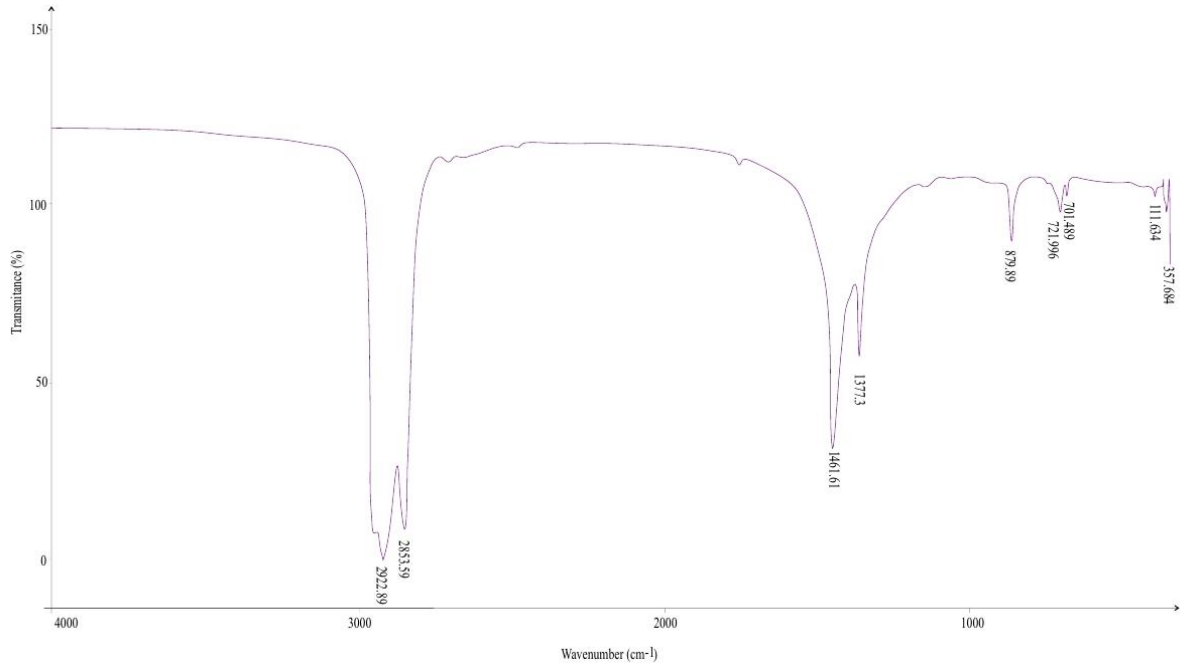

Figure 1 *FTIR spectrogram of non-activated* Na_2CO_3

Table 1 shows the wavelengths of the characteristic peaks monitored by FTIR spectroscopy in this study.

Table 1 *Wavelengths of characteristic groups of sodium carbonate, sodium bicarbonate and intermediate compound*

Na_2CO_3	$NaHCO_3$	CO_3^{2-}	HCO_3^-	OH^-	$CO_2 + H_2O$
1775 cm^{-1}	1800 cm^{-1}	1461.61 cm^{-1}	1800–1980 cm^{-1}	350–3600 cm^{-1}	2300–2400 cm^{-1}
1420 cm^{-1}	1000 cm^{-1}	1090 cm^{-1}	1000 cm^{-1}	1645 cm^{-1}	
878 cm^{-1}		874 cm^{-1}		590–720 cm^{-1}	
702 cm^{-1}		725 cm^{-1}			

Figure 2 shows comparative FTIR spectrograms of all four series of sodium carbonate samples for different relaxation periods at room conditions.

The change in the characteristic peaks as a function of relaxation time is clearly noticeable in the diagrams in Figure 1. The initial curves (0 days of relaxation) for all four activation times, at a wavelength of 3500–3600 cm^{-1}, do not have clearly defined peaks, and with the increasing relaxation time. The appearance of the OH group in the activated sodium carbonate sample, as well as changes in the peak area of the OH group, can be explained by the fact that the Na_2CO_3 sample, due to the energy obtained by mechanical activation and disturbed crystal structure had increased adsorption and chemisorption affinity to moisture from the atmosphere during relaxation. The valence OH groups bind to the crystal lattice of activated Na_2CO_3, and the diagrams show that the peak characterizing the OH group increases with the increasing relaxation time. A similar change is observed at a wavelength of 1645 cm^{-1}, and the database revealed that these changes are also related to the OH group.

At a wavelength of 1420 cm^{-1}, which characterizes Na_2CO_3, that is CO_3^{2-} ion, a tendency of peak decrease with the increasing relaxation time can be seen. Similar changes are observed at a wavelength of 873 cm^{-1}. This is a consequence of the conversion of carbonate to bicarbonate ion due to the absorption of moisture and carbon dioxide from the air. Parallelly with this change, a peak characteristic for the bicarbonate ion at a wavelength of 1800 cm^{-1} appeared. This peak is more pronounced with the increasing relaxation time.

At wavelengths from 2300 to 2400 cm^{-1}, a peak that principally disappears gradually with the progress of relaxation time can be observed. These wavelengths are typical for adsorbed moisture and carbon dioxide. During relaxation, chemisorption of moisture and carbon dioxide occurs and the conversion of carbonate to bicarbonate, which explains the decrease and disappearance of this peak on the observed curves.

At wavelengths from 700 to 800 cm^{-1}, with emphasis on 725 cm^{-1}, a peak, while both the intensity and the surface area are decreasing with the increasing relaxation time, can be observed. The analysis determined that it is a carbonate ion, whose amount decreases as a function of the relaxation time due to the process of chemisorption of moisture and carbon dioxide from the air. Parallelly with this result, the peak characteristic for the HCO_3^- group increases, which is a consequence of the transformation of carbonate into bicarbonate.

In the discussion that follows, based on the obtained FTIR spectrograms of activated sodium carbonate, areas of peaks characteristic for ion groups CO_3^{2-}, HCO_3^-, and OH^-, as well as peaks widths at half-height during relaxation time were estimated and graphically presented.

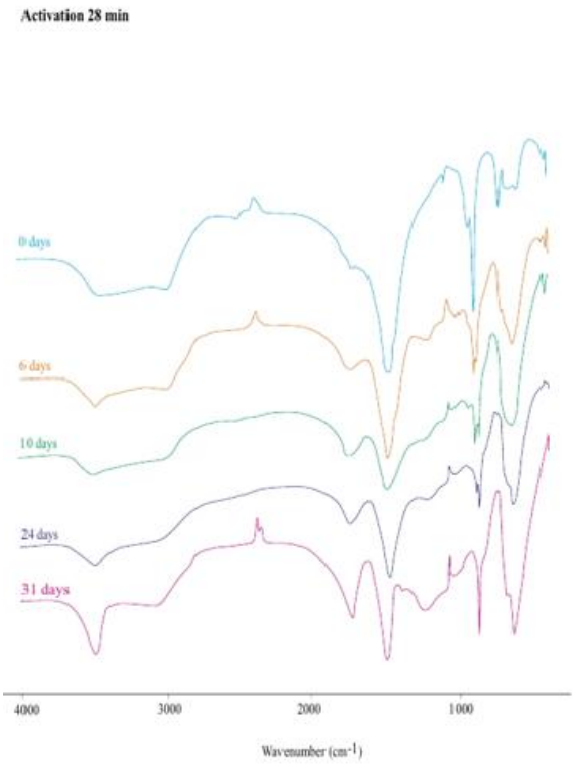

Figure 2 *FTIR spectrograms of Na_2CO_3 samples activated for 28 min during relaxation time (0, 1, 6, 10, 24, and 31 days) at room conditions*

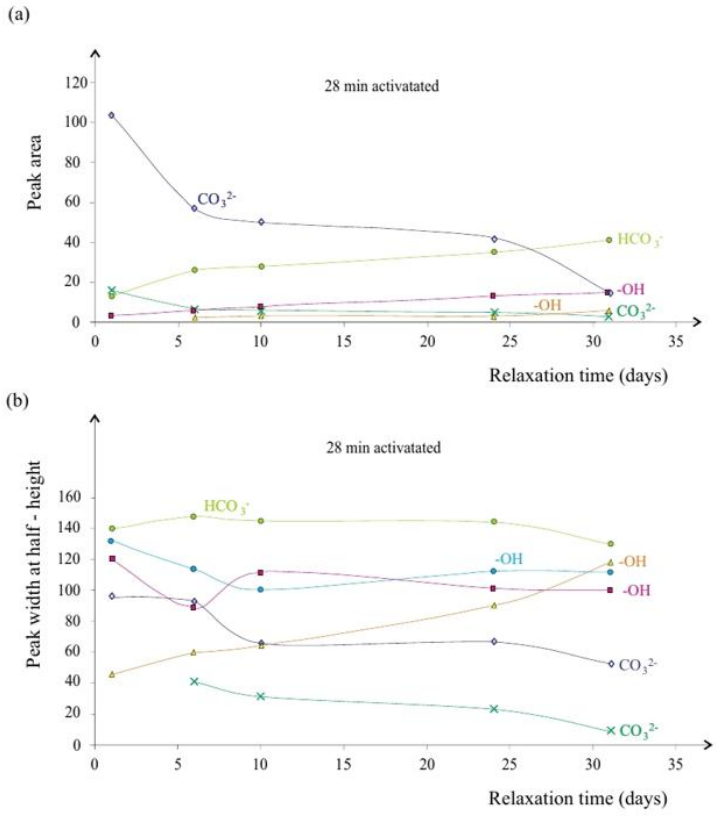

Figure 3 *a) Areas of peaks characteristic for ion groups; b) Widths at peak half-height, for Na_2CO_3 sample activated for 28 min as a function of relaxation time*

The peak area characteristic for the CO_3^{2-} ion (Figure 3a) decreases with the relaxation time progress, whereby this drop is sharp during ten days, especially on the first day of the sample aging. The phenomenon can be explained by a significantly enhanced energy of just activated sodium carbonate due to the energy delivered to the system by mechanical activation as well as by the weakened bonds in the crystal. This caused the sudden adsorption of moisture and carbon dioxide from the air as well as the formation of a bicarbonate ion, which is reflected in the increased width of the peak characteristic for the HCO_3^- ion. A similar trend of corresponding width changes at the half-heights of the characteristic peaks can be seen in the diagram shown in Figure 3b. The reduced width value at the half-height of the peak characteristic for the CO_3^{2-} ion is considerable. The most significant changes for all ions occur in the first 10 days of relaxation time.

CONCLUSION

The results of this study showed a gradient of changes in the analyzed samples during relaxation depending on the activation time. The changes in terms of reducing the number of carbonate ions in all four series of samples in favor of the formation of HCO_3^{2-} ions during the relaxation period are clearly noticeable. The key moment in the transformation process is the weakening of the Na–O chemical bond, where during the relaxation time the Na^+ ion was replaced by the H^+ ion, thus forming the OH group. The formation of sodium bicarbonate began by bonding the valence OH group to the activated crystal lattice of sodium carbonate. This transformation was taking place after the 25th day of the relaxation period owing to the existence of a sufficient amount of the OH group which directed the reaction towards the bicarbonate formation.

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