

**UNIVERSITY OF BELGRADE  
TECHNICAL FACULTY IN BOR  
CHAMBER OF COMMERCE AND  
INDUSTRY OF SERBIA**

**PROCEEDINGS**



**XIII International  
MINERAL PROCESSING and  
RECYCLING CONFERENCE**

**Editors:**

**Grozdanka Bogdanović**

**Milan Trumić**

**Belgrade, Serbia, 8 – 10 May 2019**



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KOMORA  
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# TABLE OF CONTENTS

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<b>PLENARY LECTURES</b>	1
<b>Antonio Gutierrez Merma, Maurício Leonardo Torem, Ronald Rojas Hacha</b> <i>MINERAL BIOFLOTATION: A SHORT REVIEW OF MY RESEARCH GROUP EXPERIENCE</i>	3
<b>Željko Kamberović</b> <i>RECYCLING OF THE CRITICAL RAW MATERIALS FROM WASTE ELECTRONICS</i>	17
<b>SECTION LECTURE</b>	19
<b>Mira Cocić, Mihovil Logar</b> <i>OBTAINING THE APPLICABLE MATERIAL FROM THE FFW AND ZEOLITIC TUFF</i>	21
<b>PAPERS BY SECTIONS</b>	29
<b>Piyush Khatri, Puneet Choudhary, Brahma Deo, Parimal Malakar, Sourav Saran Bose, Gyanranjan Pothal, Partha Chattopadhyaya</b> <i>DETERMINATION OF SURFACE MOISTURE AND PARTICLE SIZE DISTRIBUTION OF COAL USING ON-LINE IMAGE PROCESSING</i>	31
<b>Sonja Milićević, Sanja Martinović, Vladimir Jovanović, Milica Vlahović, Ndue Kanari, Ana Popović, Marija Kojić</b> <i>DEVELOPMENT AND MECHANICAL PROPERTIES OF THE PELLETIZED FLY ASH</i>	38
<b>Maja Pačeškoska, Ružica Manojlović, Jarmila Trpčevska</b> <i>CHARACTERIZATION OF TWO TYPES OF NICKEL ORES AND ANALYSIS OF THE PROSPECTS OF NICKEL CONCENTRATION</i>	45
<b>Farookh Sekh, Vineet Kumar</b> <i>A CASE STUDY OF HEAVY MEDIA CYCLONE EFFICIENCY IMPROVEMENT AT TATA STEEL WEST BOKARO - A NOVEL APPROACH</i>	53
<b>Blagica Cekova, Viktorija Bezhovska, Afrodita Ramos</b> <i>CHARACTERISTICS OF RESIDUE OBTAINED FROM RED OPALITE WITH CHEMICAL ACTIVATION</i>	59
<b>Kucuk Mehmet Emin, Kinnarinen Teemu, Hakkinen Antti</b> <i>CHARACTERIZATION OF MINING AND PULP INDUSTRY SIDE STREAMS: PARTICLE CHARACTERISTICS AND CHEMICAL COMPOSITIONS</i>	64
<b>Nevenka Mijatović, Anja Terzić, Ljiljana Miličić, Dragana Živojinović</b> <i>VALIDATION OF ICP-OES PROCEDURE FOR MAJOR AND TRACE ELEMENTS DETERMINATION IN THE LEACHATES OF FLY ASH AND FLY ASH BASED COMPOSITES</i>	70
<b>Irina Pestriak, Valery V. Morozov, Galina P. Dvoychenkova, Erdenetuya Otchir</b> <i>INVESTIGATION AND DEVELOPMENT OF RECYCLED WATER CONDITIONING BY THE ENRICHMENT OF COPPER-MOLYBDENUM ORES</i>	77
<b>Blagica Cekova, Viktorija Bezhovska, Afrodita Ramos, Filip Jovanovski</b> <i>EXAMINATION OF ADSORPTIONAL ABILITY TO THE NATURAL RAW MATERIAL - PERLITE FROM R. MACEDONIA</i>	85
<b>Marinela Ivanova Panayotova, Vladko Toforov Panayotov</b> <i>SET PLAN AND CRITICAL METALS</i>	90



<b>Aleksandar Pačevski, Janez Zavašnik, Aleš Šoster, Andreja Šestan, Aleksandar Luković, Ivana Jelić, Aleksandar Kremenović, Alena Zdravković, Suzana Erić, Danica Bajuk-Bogdanović</b> <i>MICRO - TO NANOSCALE TEXTURES OF ORE MINERALS: METHODS OF STUDY AND SIGNIFICANCE</i>	98
<b>Ivana Jelić, Janez Zavašnik, Predrag Vulić, Aleksandar Pačevski</b> <i>MICRO - TO NANOSCALE TEXTURE OF GOLD-BEARING ARSENOPYRITE FROM THE GOKČANICA LOCALITY, SERBIA</i>	101
<b>Vladimir Nikolić, Milan Trumić, Maja S. Trumić</b> <i>INSTRUMENTAL METHODS FOR CHARACTERIZATION OF ZEOLITE</i>	104
<b>Valery V. Morozov, Ganbaatar Zorigt, Delgerbat Lodoy, Y. P. Morozov</b> <i>IMPROVEMENT OF OPTICAL METHODS ANALYSIS OF ORE GRADE AT OPTIMIZATION OF MINERAL PROCESSING PROCESSES</i>	111
<b>Dragan S. Radulović, Ljubiša Andrić, Milan Petrov, Darko Božović, Marko Pavlović</b> <i>OBTAINING FILLERS BASED ON LIMESTONE FROM DEPOSIT "BRIJEG"-ULCINJ FOR APPLICATIONS IN VARIOUS INDUSTRIES</i>	119
<b>Katarina Balanović, Milan Trumić, Maja Trumić</b> <i>EFFICIENCY OF ZEOLITE GRINDING AND ITS POTENTIAL APPLICATION</i>	127
<b>Veljko Savić, Vladimir Topalović, Srdjan Matijašević, Jelena Nikolić, Snežana Zildžović, Sonja Smiljanić, Snežana Grujić</b> <i>PRODUCTION OF GLASS- CERAMICS FROM COAL FLY ASH AND LIMESTONE</i>	135
<b>Ilhan Bušatlić, Nadira Bušatlić</b> <i>THE POSSIBILITY OF USING THE FLY ASH FROM THERMAL POWER PLANT "STANARI" DOBOJ IN THE DEVELOPMENT OF GEOPOLYMERS</i>	141
<b>Dragana Medić, Snežana Milić, Boban Spalović, Ivan Đorđević</b> <i>IDENTIFYING CHEMICAL COMPOSITION OF CATHODE MATERIALS IN LITHIUM-ION BATTERIES</i>	148
<b>Zoran Štirbanović, Predrag Mitrović, Zoran Stević, Jovica Sokolović, Zoran Milkić</b> <i>APPLICATION OF WASTE GLASS IN PRODUCTION OF INSULATORS</i>	154
<b>Buket Kabacaoglu, Birgul Benli</b> <i>SILVER NANOPARTICLE SYNTHESIS AND USES IN SEPIOLITE-ALGINATE NANOCOMPOSITES FOR PROTECTIVE COATINGS</i>	161
<b>Tomas Vrbický, Jiri Botula, Richard Prikryl</b> <i>PROCESSING EXPERIMENTS OF FELDSPAR RAW MATERIAL USING COMBINED MAGNETIC AND GRAVITY CONCENTRATING SEPARATIONS</i>	168
<b>Yrii Chugunov, Vladyslav Ivanchenko</b> <i>ECO-TECHNOLOGY FOR COMPLEX PROCESSING OF ORES AND INDUSTRIAL WASTES</i>	172
<b>Symbat Dyussenova, Bagdaulet Kenzhaliyev, Rinat Abdulvaliyev, Sergey Gladyshev</b> <i>PROCESSING OF CHROMITE PLANT TAILINGS</i>	178
<b>O. V. Yurasova, T. A. Kharlamova, A. A. Gasanov, A. F. Alaferdov</b> <i>OPTIMIZATION OF THE PROCESS OF ELECTROCHEMICAL OXIDATION OF CERIUM IN THE PROCESSING OF RARE METALS RAW MATERIALS</i>	183
<b>Valentine A. Chanturiya, Igor Zh. Bunin, Maria V. Ryazantseva</b> <i>THE ALTERATIONS OF THE CALCIFEROUS MINERALS SURFACE PROPERTIES AND FLOATABILITY</i>	190
<b>Erdenezul Jargalsaikhan, Valery Morozov</b> <i>OPTIMIZATION OF FLOTATION GRINDING PROCESSES USING MODEL- BASED CRITERIA</i>	197

<b>Bijay Shankar Tiwari, Amit Ranjan, Ashwani Kumar, Bhalla Srinivas Rao</b> <i>ENHANCEMENT OF FLOTATION KINETICS THROUGH APPLICATION OF MODIFIER</i>	203
<b>Jelena Čarapić, Vladan Milošević, Branislav Ivošević, Dejan Todorović,</b> <b>Zoran Bartulović, Vladimir Jovanović, Sonja Milićević</b> <i>INVESTIGATION OF THE FLOTATION PARAMETERS FOR THE ORE FROM THE</i> <i>"CEROVO-C2" DEPOSIT – CEMENTATION ZONE</i>	208
<b>Jovica Sokolović, Rodoljub Stanojlović, Ljubiša Andrić, Zoran Štirbanović,</b> <b>Nikola Ćirić</b> <i>THE EFFECT OF DIFFERENT COLLECTORS ON THE FLOTATION RESULTS IN THE</i> <i>COPPER MINE MAJDANPEK</i>	213
<b>Vineet Kumar, B. V. Sudhir Kumar</b> <i>A CASE STUDY OF EMULSIFYING THE COLLECTOR IN COAL FLOTATION TO IMPROVE</i> <i>THE SEPARATION EFFICIENCY OF FLOTATION CELL</i>	219
<b>Degodya Elena Yurevna, Shavakuleva Olga Petrovna</b> <i>INFLUENCE OF REAGENTS ON PHYSICAL AND MECHANICAL PROPERTIES OF</i> <i>MINERALS</i>	224
<b>Parveen Kumar Dhall, Himanshu Sarangi, Ranjan Kumar, Parag Mukherjee,</b> <b>Vineet Kumar</b> <i>A CASE STUDY OF FLOWABILITY IMPROVEMENT USING SUPER ABSORBENT</i> <i>POLYMER AT TATA STEEL NOAMUNMDI NDCMP</i>	227
<b>Ilker Acar, Ozkan Acisli</b> <i>EFFECT OF SODIUM OLEATE ON HYDROPHOBICITY OF CALCITE</i>	234
<b>Taissa Felisberto Rosado, Ronald Rojas Hacha, Mauricio Leonardo Torem,</b> <b>Antonio Gutierrez Merma</b> <i>UTILIZATION OF HYDROGEN BUBBLES IN ELECTROFLOTATION OF FINES OF IRON</i> <i>ORE TAILINGS USING A BIOSURFACTANT</i>	239
<b>Antonio Gutiérrez Merma, Mauricio Leonardo Torem, Vinicius de Jesus</b> <b>Towesend, Caroline D. Grossi</b> <i>BIOSORPTIVE FLOTATION OF NICKEL AND COBALT BY RHODOCOCCUS</i> <i>ERYTHROPOLIS</i>	246
<b>Dragana Marilović, Maja Trumić, Milan Trumić, Ljubiša Andrić</b> <i>THE INFLUENCE OF pH VALUE ON DEINKING FLOTATION</i>	253
<b>Andreza Rafaela Morais Pereira, Ronald Rojas Hacha, Antonio Gutierrez</b> <b>Merma, Mauricio Leonardo Torem</b> <i>RECOVERY OF HEMATITE FROM IRON ORE TAILING USING A BIOSURFACTANT</i> <i>FROM THE RHODOCOCCUS OPACUS STRAIN AS A COLLECTOR</i>	258
<b>H. A. Oliveira, A. Azevedo, J. Rubio</b> <i>INNOVATIVE FLOCCULATION PROCESS FOR TREATING DISPERSED IRON</i> <i>TAILINGS BEARING WATERS</i>	266
<b>Marcelo Carneiro Camarate, Maurício Leonardo Torem, Antonio Gutierrez</b> <b>Merma, Ronald Rojas Hacha</b> <i>SELECTIVE BIOFLOCCULATION OF HEMATITE USING THE YEAST CANDIDA</i> <i>STELLATA</i>	268
<b>Arun Misra, Debaprasad Chakraborty, Bhargav Dhavala</b> <i>EVOLUTION OF COAL PROCESSING PRACTICES AT TATA STEEL</i>	275
<b>Victor Atrushkevich</b> <i>UNDERGROUND COAL ENRICHMENT</i>	281
<b>Aleksander K. Nikolaev, Sergey Yu. Avksentyev, Julia G. Matveeva</b> <i>DEVELOPMENT AND OPERATION OF HYDROTRANSPORT SYSTEMS UNDER SEVERE</i> <i>ENVIRONMENTAL CONDITIONS</i>	287

<b>Birgul Benli, Atacan Adem</b> <i>BOX-BEHNKEN EXPERIMENTAL DESIGN FOR MICROWAVE ENERGY ROASTING OF REFRACTORY GOLD FLOTATION CONCENTRATE</i>	294
<b>Pavel V. Aleksandrov, A. S. Medvedev, V. A. Imideev</b> <i>NICKEL-COPPER CONCENTRATES PROCESSING BY LOW-TEMPERATURE ROASTING WITH SODIUM CHLORIDE</i>	300
<b>Naguman P. Nigmatullayevich, Sherembayeva R. Tyulyukhanovna, Omarova N. Kakibayevna, Rakhimbekova A. Berikovna</b> <i>STUDYING THE PROCESS OF SULFURIC ACID TREATMENT OF OXIDIZED COPPER</i>	308
<b>Svetlana Bratkova, Anatolii Angelov, Elena Zheleva, Ekaterina Todorova, Stefan Stamenov, Emanuil Kozhuharov, Peter Delov, Elisaveta Valova, Zhivko Vasilev</b> <i>MULTI-DISCIPLINARY APPROACH FOR THE REHABILITATION OF HISTORICALLY DISTURBED LANDS</i>	314
<b>Aliya Mambetzhanova, Lyudmila Bolotova, Vladimir Luganov, Kulzira Mamyrbayeva, Tatyana Chepushtanova, Gulnar Gusseinova</b> <i>INVESTIGATION OF COPPER EXTRACTION FROM PREGNANT SOLUTIONS USING C-100 AND S-930/4880 PUROLITE ION EXCHANGE RESIN</i>	321
<b>Eugenia Panturu, Antoneta Filcenco-Olteanu, Aura Daniela Radu, Marius Zlagnean</b> <i>URANIUM PURIFICATION INCREASING USING ULTRASOUNDS</i>	325
<b>Cyril Bourget, Jean-Yves Dumousseau, Keith Cramer</b> <i>PRACTICAL ASPECTS OF OPERATING COPPER SOLVENT EXTRACTION PLANTS</i>	332
<b>Madali Naimanbayev, Nina Lochhova, Zhazira Baltabekova, Yerzhan Kuldeyev</b> <i>RARE-EARTH METALS EXTRACTION OUT OF SULPHATE SOLUTION BY SORPTION</i>	338
<b>Luana Caroline da Silveira Nascimento, Maurício Leonardo Torem, Ellen Cristine Giese, Luiz Carlos Bertolino</b> <i>FUNDAMENTALS OF NEODYMIUM SORPTION IN PLYGORSKITE</i>	343
<b>Farookh Sekh, Vineet Kumar</b> <i>STUDY OF THE EFFECT OF NEW GENERATION DEWATERING AID CHEMICAL TO IMPROVED DEWATERING KINETICS AND REDUCTION IN COAL CAKE MOISTURE OF FINES COAL</i>	349
<b>Mihal Đuriš, Tatjana Kaluđerović Radoičić, Zorana Arsenijević</b> <i>MATERIAL HOLD-UP AND RESIDENCE TIME IN FLUIDIZED BED OF INERT PARTICLES SLURRY DRYER</i>	357
<b>Andrey Goryachev, Evgenia Krasavtseva, Dmitry Makarov</b> <i>AMMONIUM COMPOUNDS IN THE TECHNOLOGIES OF COMPLEX PROCESSING OF LOW-GRADE COPPER-NICKEL RAW MATERIALS IN THE ARCTIC CONDITIONS</i>	365
<b>Nikolay Simonski, Georgy Bozhilov, Stoyko Peev, Stilian Minkin, Elisaveta Valova</b> <i>DUNDEE PRECIOUS METALS CHELOPECH - INNOVATIVE SOLUTIONS FOR EFFICIENT PRODUCTION IN HARMONY WITH NATURE</i>	371
<b>Lyubomir Ilchev, Nadezhda Davcheva-Ilcheva</b> <i>SUSTAINABLE ORE RESOURCE BASE AND CIRCULAR ECONOMY</i>	379
<b>Antoneta Filcenco-Olteanu, Marius Zlagnean, Eugenia Panturu, Aura Daniela Radu, Nicolae Tomus</b> <i>THE OXIDATIVE PROCESSES AND MIGRATION OF ELEMENTS IN HISTORICAL TAILINGS</i>	386
<b>Vladko Toforov Panayotov, R. Imhof, M. Panayotova</b> <i>A POSSIBILITY FOR PURIFICATION OF INDUSTRIAL EFFLUENTS FROM ARSENIC IN HIGH CONCENTRATIONS</i>	394

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<b>Ivan Milojković, Zorana Naunović, Novak Pušara, Sreten Beatović</b> <i>SOLID WASTE MANAGEMENT IN THE GACKO MINE AND THERMAL POWER PLANT</i>	402
<b>Viktorija Bezhovska, Blagica Cekova, Filip Jovanovski</b> <i>APPLICATION OF FLY ASH IN THE CONSTRUCTION INDUSTRY</i>	410
<b>Agapi Vasileiadou, S. Zoras, A. Dimoudi, A. Iordanidis, V. Evagelopoulos</b> <i>THE ENERGETIC POTENTIAL OF DIFFERENT BIO-WASTE SAMPLES COLLECTED FROM AN INTEGRATED WASTE MANAGEMENT PLANT</i>	417
<b>Marko Pavlović, Marina Dojčinović, Ljubiša Andrić, Dragan Radulović, Milan Petrov</b> <i>DETERMINATION OF THE CAVITATION RESISTANCE OF GLASS-CERAMIC SAMPLES BASED ON RAW BASALT AND INDUSTRIAL WASTE RAW MATERIALS FOR USE IN METALLURGY</i>	423
<b>Yelena Bochevskaya, Z. Karshigina, A. Sharipova, Z. Abisheva, E. Sargelova</b> <i>PHOSPHORIC SLAG - A TECHNOGENIC SOURCE OF RECEIVING "WHITE SOOT" AND CONCENTRATE OF RARE-EARTH METALS</i>	430
<b>Aleksandra Stanojković-Sebić, Zoran Dinić, Jelena Maksimović, Aleksandar Stanojković, Radmila Pivić</b> <i>APPLIANCE OF METALLURGICAL SLAG AS A BY-PRODUCT OF THE STEEL INDUSTRY IN AMELIORATION OF ACID SOILS AND RESPONSES OF PARSLEY</i>	437
<b>Lyudmila Agapova, S. Kilibayeva, A. Zagorodnyaya, A. Sharipova, B. Kenzhaliyev, Zh. Yakhiyayeva</b> <i>RECYCLING OF RHENIUM, NICKEL AND COBALT FROM THE WASTE OF HEAT-RESISTANT ALLOYS</i>	445
<b>Roland Szabo, Mucsi Gabor</b> <i>EFFECT OF <math>SiO_2/Al_2O_3</math> MOLAR RATIO ON STRUCTURE AND MECHANICAL PROPERTIES OF FLY ASH BASED GEOPOLYMER</i>	452
<b>Himanshu Tanvar, Nikhil Dhawan</b> <i>EXTRACTION OF RARE EARTH VALUES FROM DISCARDED CFL-S</i>	459
<b>Zoran Stević, Silvana Dimitrijević, Aleksandra Ivanović, Milan Jovanovic Stevan Dimitrijević</b> <i>RECOVERY OF COBALT FROM DIAMOND CORE DRILLING CROWNS</i>	468
<b>V. A. Luganov, T. A. Chepushtanova, G. D. Guseynova, K. K. Mamyrbayeva, O. S. Baigenzhenov, E. S. Merkibayev</b> <i>DEVELOPMENT OF TECHNOLOGY FOR GOLD-ARSENIC-COAL CONCENTRATES PROCESSING</i>	475
<b>Birgul Benli, Meltem Yildiz</b> <i>THERMODYNAMIC APPROACHES OF THE REMOVAL OF CYANIDE FROM WATER SOLUTIONS BY HALLOYSITE CLAYS</i>	482
<b>Markus Wilke, Laura Carbone, Marija Bakrac</b> <i>GEOSYNTHETICS IN SLUDGE DEWATERING AND SLUDGE LAGOON COVERS</i>	489
<b>Jelena Mičić, Una Marčeta, Bogdana Vujić, Višnja Mihajlović</b> <i>IDENTIFICATION OF FINAL DISPOSAL WAYS OF WASTE CLOTHES IN ZRENJANIN MUNICIPALITY</i>	497
<b>Jovanka Milićević, Milica Alimpić</b> <i>RAISING ENVIRONMENTAL AWARENESS AS AN IMPORTANT FACTOR FOR IMPROVING PACKAGING WASTE MANAGEMENT</i>	504
<b>Jelena Drobac-Petrović, Predrag Maksić, Vesna Alivojvodić, Marina Stamenović</b> <i>SUSTAINABLE GRAPHIC DESIGN: POSSIBILITIES OF PACKAGING</i>	509

<b>Predrag Maksić, Jelena Drobac Petrović, Vesna Alivojvodić, Marina Stamenović</b> <i>UPCYCLING DESIGN PROTOCOL: FUNCTIONAL INTERIOR DESIGN USING WASTE MATERIALS</i>	516
<b>Vladimir Pavićević, Darko Radosavljević, Ana Popović, Marina Stamenović, Vesna Alivojvodić, Aleksandra Božić</b> <i>INSTITUTIONAL CRITERIA FOR INFRASTRUCTURE PROJECTS - CONDITION FOR SUSTAINABLE DEVELOPMENT</i>	523
<b>Dragana S. Božić, Nobuyuki Masuda, Radmila Marković, Masahiko Bessho, Zoran Stevanović</b> <i>HOW IS THE PROBLEM OF ACID MINE DRAINAGE OF THE CLOSED MATSUO MINE SOLVED IN JAPAN</i>	530
<b>Grozdana Bogdanović, Žaklina Tasić</b> <i>REMOVAL OF COPPER IONS FROM WASTEWATER USING NATURAL ZEOLITE</i>	535
<b>Ecehan Ayygul Gonul, Alican Mert, Birgul Benli</b> <i>COLORING PERFORMANCE OF IRON OXIDE NANOPIGMENTS PREPARED FROM A SYNTHETIC ACID MINE DRAINAGE</i>	541
<b>Jovica Sokolović, Branislav Stakić, Suzana Stanković, Vojka Gardić, Miloš Kirov</b> <i>REMOVAL OF OIL FROM WASTEWATER BY ANTHRACITE COAL</i>	548
<b>Dragana S. Božić, Milan Gorgievski, Velizar Stanković, Nada Štrbac, Vesna Grekulović, Miljan Marković</b> <i>ADSORPTION ISOTHERMS FOR DESCRIBING THE MECHANISM OF COPPER IONS BIOSORPTION ONTO OAT STRAW</i>	555
<b>Ivana Smičiklas, Marija Egerić, Mihajlo Jović, Marija Šljivić-Ivanović, Ana Mraković</b> <i>ZINC AND STRONTIUM REMOVAL EFFICIENCY BY THERMALY MODIFIED SEASHELL WASTE</i>	561
<b>Tatjana Šoštarić, Marija Petrović, Zorica Lopičić, Jelena Petrović, Marija Kojić, Marija Koprivica, Katarina Pantović Spajić</b> <i>APRICOT SHELLS AS BIOSORBENT FOR CU(II) IONS: DETERMINATION OF OPTIMAL ALKALINE TREATMENT CONDITIONS</i>	568
<b>Ana Popović, Sonja Milićević, Vladan Milošević, Branislav Ivošević, Jelena Čarapić, Dragan Povrenović</b> <i>HOMOGENEOUS FENTON PROCES FOR MINERALIZATION OF METHYLENE BLUE</i>	575
<b>Aleksandra Cvetković, Miroslava Marić, Darko Milošević</b> <i>POTENTIALS OF BIOMASS USE IN SERBIA</i>	580
<b>Miomir Mikić, Milenko Jovanović, Srđana Magdalinović, Daniela Urošević</b> <i>ENVIRONMENTAL RISKS OF MINING ACTIVITIES</i>	588
<b>Miomir Mikić, Radmilo Rajković, Milenko Jovanović, Branislav Rajković</b> <i>ENVIRONMENTAL IMPACT OF MINING WASTE DISPOSAL</i>	594
<b>AUTHORS INDEX</b>	599



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### APRICOT SHELLS AS BIOSORBENT FOR CU(II) IONS: DETERMINATION OF OPTIMAL ALKALINE TREATMENT CONDITIONS

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**ABSTRACT** – The apricot stones (KK) were investigated as biosorbent of copper ions from aqueous solution. The rigidity of lignocellulosic compact molecular arrangement, induce the necessity of its modification. The aim of this paper was to establish optimal parameters of KK modification in order to improve low-cost biosorbent with improved biosorption characteristics. The modification parameters were: initial NaOH concentration, contact time and biomass/base solution ratio. After sets of experiments, the optimal modification parameters for copper removal were found to be: initial concentration of modification agent 1.0 mol/L NaOH, solid/liquid ratio 1:20 and 180 minute of contact time.

The results show that modified apricot shells doubled the binding affinity toward copper ions, and could be used as an efficient low-cost biosorbent, promoting more sustainable production and to stop waste disposal at landfill sites.

**Key words:** apricot shells, biosorption, Cu(II) ions, modification.

#### INTRODUCTION

The waste utilization of fruit processing industries has become one of the greatest challenges in world nowadays. Apricots have a significant role in Serbia's fruit production, but average annual production of 30,063 t (Statistical Office of the Republic of Serbia 2018) generates approximately 1,879 t apricot stone waste. Although apricot shells can be used for active carbon production and seeds can be used in food or pharmaceutical industry, most of the apricot stone waste ends up in landfill sites. In order to minimize this type of waste, apricot shells could be used as a biosorbent for removal of different types of pollutants. Because of all mentioned and faced with local environment pollution problems apricot shells was chosen for sorption experiments in the present study. This lignocellulosic biomass fulfils the criteria of unconventional sorbent: it is abundant, cheap and it is waste.

Since preliminary experiments showed that native apricot shells have low affinity

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toward copper ions [1], need for modification of this material has been emerged. In order to get better biosorption performances alkaline modification has been applied. It is well known that this type of treatment has a significant impact on swelling of cellulose fibres causing the hydrogen bond breakage. Due to realising of OH groups cellulose becomes more reactive. Also this type of treatment removes superficial impurities waxes and fats and consequently functional groups become more accessible [2].

In order to get biosorbent with improved biosorption characteristics, raw apricot shells were object of alkaline treatment. Focus of this study was to investigate the optimal parameters of modification, which have effect on chemical, structural and morphological changes of this lignocellulosic material, in order to get biosorbent with improved sorption characteristics..

## **MATERIALS AND METHODS**

### **Untreated biomass (KK)**

Apricot stones were obtained from juice Factory from the Rasina district of Serbia. They were manually separated from kernels and only shells were used for further experiments. Shells were grinded in mill (KHD Humbolt Wedag AG, Germany) and sieved at particle size less than 0.3 mm.

### **Metal solution preparation**

Initial copper solution with concentration of 1g/L were prepared by dissolving precise amount of  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  in deionized water.

### **Adsorption experiments**

The removal of copper ions was investigated with differently obtained biosorbents. Batch experiments were carried out when 0.2 g of each obtained sorbent was put in 100 ml glass flask, left on orbital shaker at constant rate (250 rpm/min) for 120 min. The pH value of metal solution was 5.0. After sorption experiment suspension were filtered and residual metal concentration in filtrate were determined by usage of atomic adsorption spectrophotometer. The biosorption capacity was calculated by using the following equation:

$$q_e = \frac{V (C_i - C_e)}{m} \quad (1)$$

Where  $q$  is the biosorption capacity - amount of adsorbed metal ions (mg/g);  $C_i$  is initial metal concentration while  $C_e$  is equilibrium metal concentration (mg/L);  $V$  is volume (L) and  $m$  is mass sample (g).

### **Determination of optimal modification parameters**

In order to design biosorbent with best adsorption performance, the effects of three modification parameters on the rate of adsorption process were observed by varying initial concentration of modified agent (NaOH), ratio between KK and NaOH solution and contact time between modified agent and KK.

### **Effect of initial NaOH concentration**

The first set of experiments was conducted with different initial NaOH concentration: 0.25; 0.5, 1.0; 2.0; 3.0 and 4.0 mol/L. Contact time between base and KK was 120 minute and ratio between them was 1:20. Each obtained biosorbents were rinsed with distilled water until neutral reaction. After drying we tested each of this individual sorbents through set of sorption experiments previously described in section 2.3.

### **Effect of m/V ratio**

Next set of experiments was conducted with different ratio between KK and base: 1:20; 1:40; 1:60; 1:80 and 1:100. Contact time was 120 minute and initial NaOH concentration was 1 mol/L as it was chosen from first set of experiments.

### **Effect of contact time**

The final set of experiments was conducted by varying the contact time between KK and base: 30, 60, 90, 120, 180, 240 and 300 minute. The ratio between solid and liquid phase of 1:20 were applied and initial NaOH concentration was 1 mol/L due to previous set of experiments.

## **RESULTS AND DISCUSSION**

In this section, results of investigation of optimal modification parameters have been presented with the focus if chemical treatment onto raw biomass was sufficient to produce the desired effect (improved biosorption characteristic). According to literature, effects of initial base concentration, contact time and temperature have major effects on results of alkaline treatment [2]. In this paper effect of temperature was not the object of study and all experiments were done on room temperature. Ratio between liquid and solid phase were examined, instead.

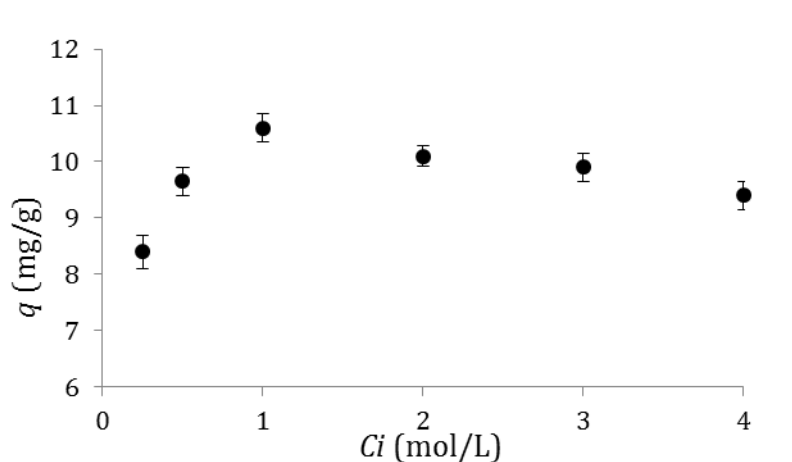
### **Effect of initial concentration of modified agent - NaOH**

Native apricot shells (KK) has been modified with different initial concentration of NaOH solution and the results are presented at Figure 1.

As concentration of NaOH rise from 0.25 to 1 mol/L biosorption capacity has risen from 8.4 to 10.6 mg/g, too. However, with further increase of NaOH concentration biosorption capacity started to decrease. The reason for this could be that severe concentration of alkaline effects lignocellulosic structure causing loss of functional groups due to hydrolyses of hemicellulose and depolymerisation of lignin [1]. Similar results were obtained when modified poplar sawdust were used for removal of Cu(II) ions in biosorption experiments by Šćiban [2].

Furthermore, each modified sample (KKM) was tested if it was washed properly: the idea was to test if any deposition of hydroxi groups into the pores of the biosorbent, had occurred. This would lead to sorption false results. In accordance with this the final pH value of solution after biosorption experiments was measured and results are presented in Table1.





**Figure 1.** Effect of initial concentration of NaOH onto sorption capacity:  $m/V = 1:20$ , contact time was 120 min and concentration of Cu(II) solution was 60 mg/L

**Table 1.** Change of pH value after Cu(II) ions adsorption by native biomass modified at different NaOH concentration: initial pH value was 5.00

NaOH concentration (mol/L)	0.25	0.50	1.0	2.0	3.0	4.0
$pH_f$	4.25	4.27	4.28	4.31	4.30	4.37

As can be seen from Table 1 it is evident that there wasn't any noticeable change in pH value of residual solution after biosorbent experiments. This means that the material was washed thoroughly during alkaline treatment and most importantly it means that the increase of biosorption capacity is due to higher porosity and functional groups accessibility of modified biosorbent KKM [1]. Therefore, initial concentration of 1 mol/L of modified agent NaOH was chosen for further experiments.

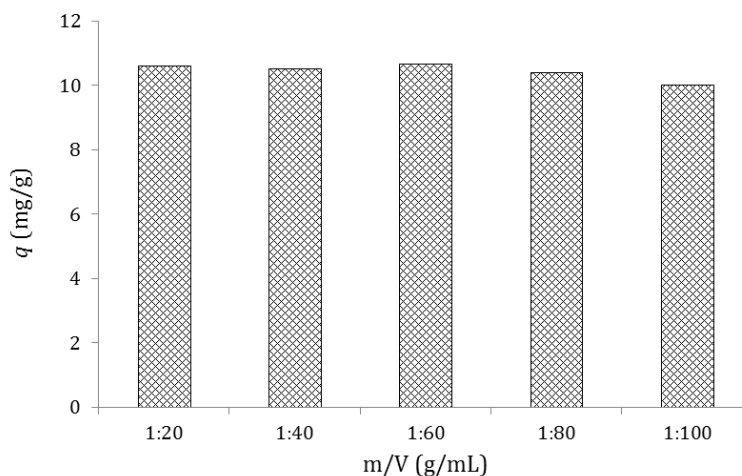
#### Effect of ratio between KK and base during the modification process onto biosorption capacity

In order to exam if the ratio between solid (KK) and liquid (NaOH) phase have impact on biosorption capacity, the sorption experiments were conducted by varying the  $m/V$  ratio from 1:20 to 1:100. The results are presented at Figure 2.

Figure 2 show that the solid/liquid ratio doesn't have any effect on biosorption capacity of obtained material. In order to get economical and eco-friendly biosorbent  $m/V$  ratio of 1:20 was chosen for alkaline treatment. Table 2 shows that after each set of biosorption experiments finale pH value stays unchangeable.

**Table 2.** Change of pH value after each sets of biosorption experiments:  $pH_i = 5.00$

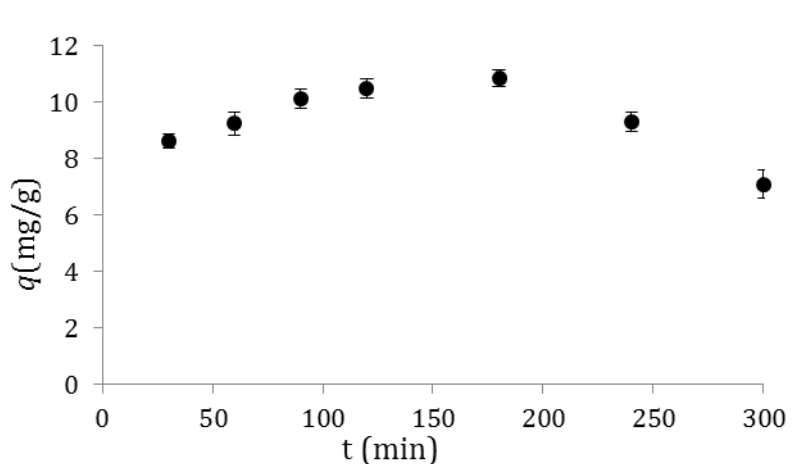
$m/V$	1:20	1:40	1:60	1:80	1:100
$pH_f$	4.28	4.25	4.32	4.25	4.24



**Figure 2.** Effect of ratio among KK and NaOH on biosorption capacity towards Cu(II) ions: initial concentration of NaOH was 1mol/L; contact time:120 min, and metal concentration was 60 mg/L

#### Effect of contact time between KK and NaOH onto biosorption capacity

The effect of treatment time on biosorption capacity of KKM toward Cu(II) ions was investigated in order to determine the necessary time for best results. The obtained results are presented on Figure 3.



**Figure 3.** Effect of alkaline treatment time on biosorption capacity of KKM: concentration of NaOH was 1mol/L; m/V= 1:20, concentration of Cu(II) solution was 60 mg/L

Prolonging the contact time between KK and alkaline solution from 30 to 180 minute, biosorption capacity increases from 8.6 to 10.9 mg/g, respectively. However, afterwards biosorption capacity decreases for almost 40 %. If the contact time is longer than 180 minute, the lignocellulosic biomass become more disturbed which

causes the biomass to be chemically altered and not suitable for copper ions bonding: the number of active sites becomes lower. As in previous experiments final pH values of solution were measured and the results are presented in Table 3.

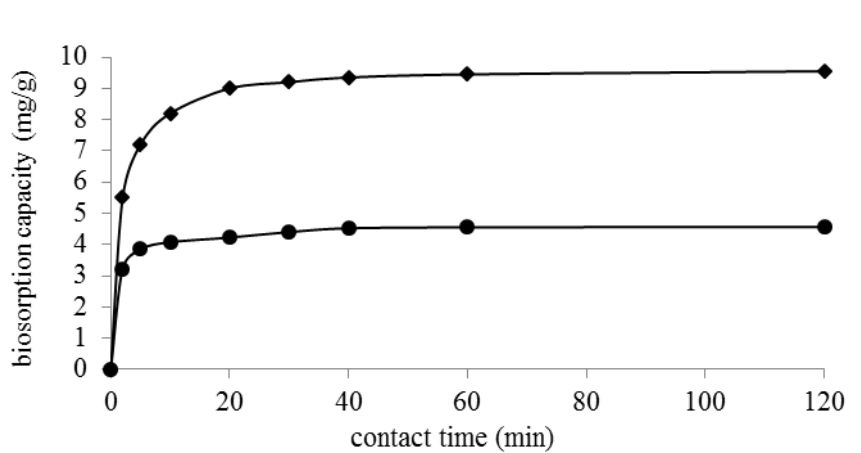
**Table 3.** Change of pH value after biosorption experiments where contact time between KK and NaOH was varied:  $pH_i = 5.00$

Contact time (minute)	30	60	90	120	180	240	300
$pH_f$	4.31	4.24	4.22	4.20	4.25	4.16	3.98

As can be seen from Table 3 final pH value of residual solution after biosorption experiments doesn't varied till 180 minute, while after this point it starts to decrease slightly: after 300 minute it is  $pH < 4.0$ . Contact time of 180 minute was chosen as time on which modification will be occurred.

Considering all previous experiments the chosen optimal parameters of KK modification are: initial concentration of modified agent is 1.0 mol/L NaOH, solid/liquid ratio is 1:20 and 180 minute of contact time among KK and NaOH. After alkaline treatment, treated biomass was washed thoroughly in distilled water, filtered and left in oven for 24 h at 50 °C. Obtained material was label as KKM.

The effect of alkaline treatment on native apricot shells biomass onto biosorption capacity toward Cu(II) ions is presented at Figure 4. It is evident that alkaline treatment doubled the value of biosorption capacity due to physical and chemical changes of this lignocellulosic biomass as it was later profoundly presented [1].



**Figure 4.** Difference in biosorption capacity between KK and KKM

## CONCLUSION

The presented study demonstrates that alkaline modification has significant effect on biosorption performance of rigid native lignocellulosic biomass. Throughout set of experiments the optimal parameters of modification were establish: initial concentration of modified agent is 1.0 mol/L NaOH, solid/liquid ratio is 1:20 and 180 minute of contact time. Acquired biosorbent KKM achieve to double the value of biosorption capacity of native biomass. As apricot shells are

widely available in the Republic of Serbia as an agricultural waste, biosorbent made of this type of biomass has twofold significance: economical solution for wastewater treatment and preventing the waste from going to landfill.

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