



University of Belgrade  
Technical Faculty in Bor,  
Mining and Metallurgy  
Institute Bor

**54<sup>th</sup> International  
October Conference  
on Mining and Metallurgy**

# PROCEEDINGS

**Editors:**

**Ljubiša Balanović**

**Dejan Tanikić**



**18-21 October 2023, Bor Lake, Serbia**

**PROCEEDINGS,  
54<sup>th</sup> INTERNATIONAL OCTOBER CONFERENCE  
on Mining and Metallurgy**

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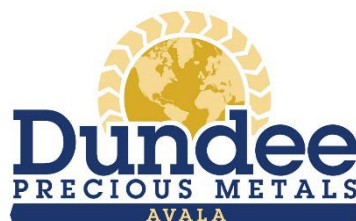


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## PREFACE

On behalf of the Organizing Committee, it is a great honor and pleasure to welcome all esteemed participants of the 54<sup>th</sup> International October Conference on Mining and Metallurgy (IOC 2023), scheduled to take place at the picturesque Bor Lake, Serbia, from October 18<sup>th</sup> to 21<sup>st</sup> 2023.

The collaborative efforts of the University of Belgrade, the Technical Faculty in Bor, and the Mining and Metallurgy Institute Bor have meticulously organized this year's IOC. Our focus remains unwavering on showcasing the latest research findings and advancements in geology, mining, metallurgy, materials science, technology, environmental protection, and other engineering disciplines. Our primary objective is to foster a dynamic environment where academics, researchers, and industry professionals can come together to share their knowledge, experiences, and innovative ideas while exploring opportunities for collaborative research endeavors.

Our conference agenda is rich and diverse, encompassing plenary sessions, engaging invited lectures, technical presentations, enlightening oral and poster sessions, informative technical tours, a diverse exhibition, and memorable social gatherings. At the heart of this event lies our strong commitment to sustainable development within the mining and metallurgy sector. We are dedicated to exploring ecologically conscious methodologies, responsible resource extraction practices, and cutting-edge technologies that reduce the industry's environmental impact and enhance the well-being of local communities.

The conference proceedings comprise 129 papers authored by individuals from universities, research institutes, and industries in 22 countries. We are proud to welcome participants from Bosnia and Herzegovina, Bulgaria, Canada, China, Croatia, Germany, Greece, India, Iran, Kazakhstan, Libya, North Macedonia, Montenegro, Morocco, Romania, Russia, Slovakia, South Africa, Spain, Turkey, United States, and, of course, Serbia.

We are excited to host the 8<sup>th</sup> International Student Conference on Technical Sciences (ISC 2023) as part of IOC 2023. This event offers students from Serbia and the wider region a unique chance to showcase their research and discuss the future of their fields with experts.

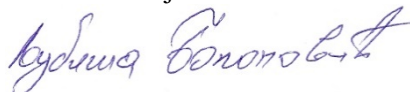
We sincerely thank the Ministry of Science, Technological Development, and Innovation of the Republic of Serbia for their generous financial support. In addition, we express our profound gratitude to all our sponsors, exhibitors, and friends of the Conference for their contributions and unwavering support for playing a pivotal role in ensuring the success of IOC 2023.

We would like to express our heartfelt thanks to all authors, committees, reviewers, speakers, and chairpersons for their invaluable contributions in shaping IOC 2023.

We look forward to welcoming you to the 55<sup>th</sup> International October Conference on Mining and Metallurgy (IOC 2024), which will be held in October 2024.

On behalf of the 54<sup>th</sup> IOC Organizing Committee,

Prof. dr Ljubiša Balanović



## TABLE OF CONTENTS

### Plenary Lectures

**Velimir R. Radmilović** (SERBIA)

*Energy: One of the biggest challenges in 21<sup>st</sup> century* 3-3

**Jing Yu, Mingshui Luo, Junyi Xiang, Yang You, Zhixiong You, Xuewei Ly** (CHINA)

*Efficient extraction of vanadium from vanadium slag* 4-8

### Invited Lectures

**Batrić Pešić** (UNITED STATES)

*The ongoing restructuring of universities to adopt the sophistication offered by internet* 11-19

**Yaima Filiberto, Alberto Montenegro, Eugenio Alvarez** (SPAIN)

*Machine learning applied to improving the scrap recycling and melting process in all types of ferrous alloys and steel* 20-22

**Slobodan Kostić, Qi Fenglai, Savo Pirgić, Nenad Botić, Dobrica Milovanović, Čedomir Sušić, Igor Zlatković** (SERBIA)

*Construction of a new sintering plant 180 m<sup>2</sup> within the HBIS Group Serbia Iron & Steel* 23-26

**Satyananda Patra** (INDIA)

*Acid activation of bentonite: Physico-Chemical characterization and application in goethitic iron ore green pelletization* 27-35

**Ridvan Yamanoglu** (TURKEY)

*Production of metal-based powders by atomization techniques* 36-45

**Yong Du, Rainer Schmid-Fetzer, Jincheng Wang, Shuhong Liu, Jianchuan Wang, Qiang Lu, Yuhui Zhang, Kai Li** (CHINA, GERMANY)

*Computational design of engineering materials: case studies for a cemented carbide and a heat resistant Al alloy* 46-46

### Conference Papers

**Ordinartsev Denis, Nadezhda Pechischeva, Svetlana Estemirova, Andrey Rempel** (RUSSIA)

*Cr(VI) photosorption on composite sorbent of montmorillonite with amorphous TiO<sub>2</sub>* 49-52

**Mikhail Korovkin, Ludmila Ananyeva, Andrey Zherlitsyn, Sergey Kondratyev, Olesya Savinova** (RUSSIA)

*Electro-pulse crushing in high-purity quartz production* 53-55

**Žarko Radović, Nebojša Tadić** (MONTENEGRO)

*Analytical simulation of EAF dust enrichment* 56-59



|   |         |
|---|---------|
| <b><u>Nebojša Tadić, Žarko Radović</u> (MONTENEGRO)</b><br><i>Thermal and mechanical relaxation of residual stresses in cold rolled aluminium alloy strips</i>  | 60-63   |
| <b>Dragan Šabaz, Miloš Stojanović, Dejan Petrović</b> (SERBIA)<br><i>Selection of anchor type using AHP method</i>  | 64-67   |
| <b><u>Miloš Stojanović, Veljko Lapčević, Ivica Vojinović</u></b> (SERBIA)<br><i>Blast fragmentation analysis in Jama Bor by using WipFrag software</i>  | 68-71   |
| <b><u>Veljko Lapčević, Toma Jovičić, Slavko Torbica</u></b> (SERBIA)<br><i>Mine ventilation model validation by PQ survey</i>   | 72-75   |
| <b><u>Jelena Đorđević, Jelena Stefanović, Sandra Guševac, Ivan Jelić, Stefan Trujić</u></b> (SERBIA)<br><i>Life cycle analysis (LCA) of asphalt layers containing recycled asphalt pavement</i>   | 76-79   |
| <b><u>Jelena Ivaz, Dejan Petrović, Predrag Stolić, Mladen Radovanović, Dragan Zlatanović, Saša Stojadinović, Pavle Stojković</u></b> (SERBIA)<br><i>Occupational injuries in underground coal mining: statistical analysis of data</i>                            | 80-83   |
| <b><u>Jelena Ivaz, Dejan Petrović, Mladen Radovanović, Dragan Zlatanović, Saša Stojadinović, Pavle Stojković</u></b> (SERBIA)<br><i>Prediction of methane emissions in coalmine - Soko</i>  | 84-87   |
| <b><u>C. Prochaska, E. Kokkinos, D. Merachtsaki, A. Lampou, E. Peleka, K. Simeonidis, G. Vourlias, A. Zouboulis</u></b> (GREECE)<br><i>Recovery of metallic fractions from medical products labelled for single use</i>   | 88-91   |
| <b><u>Nataša Sarap, Marija Janković, Vojislav Stanić, Ivana Jelić, Marija Šljivić-Ivanović</u></b> (SERBIA)<br><i>Analysis of gross alpha and gross beta activity in samples around former uranium mine Gabrovnica</i>  | 92-95   |
| <b><u>Dragan Manasijević, Ljubiša Balanović, Ivana Marković, Uroš Stamenković</u></b> (SERBIA)<br><i>Latent heat of some aluminium based phase change alloys for thermal energy storage</i>   | 96-99   |
| <b><u>Anđelka Stojanović, Ivica Nikolić, Isidora Milošević</u></b> (SERBIA)<br><i>Position of European countries in sustainable resource management</i>   | 100-103 |
| <b><u>Aleksandar Đorđević, Duško Minić, Milena Zečević, Dragan Manasijević</u></b> (SERBIA)<br><i>Mechanical and electrical properties of the ternary Ag-Ge-Sn alloys</i>   | 104-107 |
| <b><u>Milena Zečević, Duško Minić, Aleksandar Đorđević, Dragan Manasijević</u></b> (SERBIA)<br><i>Effect of chemical composition on the corrosion resistance of the ternary Ag-Ge-Sn alloys</i>   | 108-111 |
| <b><u>Tatiana Aleksandrova, Nadezhda Nikolaeva</u></b> (RUSSIA)<br><i>Extraction of low-dimensional structures of nonferrous and noble metals from refractory raw materials</i>   | 112-115 |
| <b><u>Viša Tasić, Tatjana Apostolovski-Trujić, Bojan Radović, Nevena Ristić, Tamara Urošević, Vladan Kamenović, Zvonko Damjanović</u></b> (SERBIA)<br><i>Air quality measurements in the Bor city during the reconstruction of the copper smelter Bor in 2022</i> | 116-119 |

|   |         |
|---|---------|
| <b><u>Slavica Miletić, Biserka Trumić, Suzana Stanković</u> (SERBIA)</b><br><i>Application of control charts in the laboratory for testing the metallic materials</i>   | 120-123 |
| <b><u>Alexey M. Amdur, Sergei A. Fedorov, Andrey A. Forshev, Nikolay V. Grevtsev, Vera V. Yurak</u> (RUSSIA)</b><br><i>Technological aspects of the use of peat as a component of pulverated coal fuel for blast furnaces</i>                                     | 124-127 |
| <b><u>Ljiljana Avramović, Zoran Stevanović, Vanja Trifunović, Radmila Marković, Dragana Božić, Daniela Urošević, Silvana Dimitrijević</u> (SERBIA)</b><br><i>Hydrometallurgical treatment of mining waste from Bor - Serbia in aim of copper recovery</i>         | 128-131 |
| <b><u>Daniel Kržanović, Radmilo Rajković, Ivana Jovanović, Milenko Jovanović, Miomir Mikić</u> (SERBIA)</b><br><i>Determination the final contour of the open pit Veliki Krivelj for the mining capacity 23.1 million tons of ore</i>                             | 132-135 |
| <b><u>Vladan Marinković, Miroslava Maksimović, Milenko Jovanović, Goran Pačkovski</u> (SERBIA)</b><br><i>The use of unmanned aerial vehicles for making the precise 3D topo models and orthophoto images</i>  | 136-140 |
| <b><u>Dejan Tanikić, Anđela Stojić, Jelena Đoković, Miloš Stoljiljković</u> (SERBIA)</b><br><i>Mechanical characteristics of the shape memory alloy Cu-Zn-Al</i>  | 141-144 |
| <b><u>Ljiljana Avramović, Vanja Trifunović, Zoran Stevanović, Radmila Marković, Dragana Božić, Dejan Bugarin, Silvana Dimitrijević</u> (SERBIA)</b><br><i>Copper recovery from RE-flotation tailings by combined process</i>                                      | 145-148 |
| <b><u>Milenko Jovanović, Daniel Kržanović, Radmilo Rajković, Vladan Marinković, Miroslava Maksimović, Miomir Mikić</u> (SERBIA)</b><br><i>Application of hybrid geogrids in mining</i>  | 149-153 |
| <b><u>Stefan Trujić, Miroslava Maksimović, Vladan Marinković, Ljiljana Avramović, Vanja Trifunović, Dragana Božić</u> (SERBIA)</b><br><i>Geological exploration of the technogenic deposit - old flotation tailing pit - Bor with the possibility of leaching</i> | 154-157 |
| <b><u>Zoran Stevanović, Radmila Marković, Ljiljana Avramović, Vojka Gardić, Jelena Petrović, Dragana Božić</u> (SERBIA)</b><br><i>Sustainable and smart mining</i>  | 158-161 |
| <b><u>Snežana Ignjatović, Ivana Vasiljević, Branisav Sretković, Milanka Negovanović</u> (SERBIA)</b><br><i>Using gravity data to define structural correlation affecting the formation of Neogene basins</i>  | 162-165 |
| <b><u>Deniz Eylül Akpınar, Batuhan Turgut, Ugur Gurol, Savas Dilibal</u> (TURKEY)</b><br><i>Characterization of wire arc additively manufactured wear-resistant bimetallic component</i>  | 166-169 |
| <b><u>Mistreanu Sebastian, Ramona Cimpoesu, Dragoș Achiței, Mihai Popa, Daniela Lucia Chicet, Vasile Manole, Ana-Maria Scripcariu, Nicanor Cimpoesu</u> (ROMANIA)</b><br><i>Sandblasting process influence on stainless steel cutting element properties</i>      | 170-174 |

|   |         |
|---|---------|
| <b><u>Dorđe Petrović, Katarina Stanković, Latinka Slavković Beškoski, Ksenija Kumrić</u></b><br>(SERBIA)<br><i>Removal of Cu(II) from aqueous solutions using adsorbent based on chitosan hydrogel beads</i>          | 175-178 |
| <b><u>Jovan P. Šetrajčić, Siniša M. Vučenović</u></b> (BOSNIA AND HERZEGOVINA)<br><i>Modified basic properties of electrons in layered nanocrystals with a complex lattice</i>  | 179-182 |
| <b><u>Irena Nikolić, Milena Tadić, Dijana Đurović, Nevena Cupara, Ivana Milašević</u></b><br>(MONTENEGRO)<br><i>Kinetic and thermodynamic aspects of strontium adsorption by steelmaking slag</i>                     | 183-186 |
| <b><u>Miomir Mikić, Milenko Jovanović, Sandra Milutinović, Daniel Kržanović, Radmilo Rajković</u></b> (SERBIA)<br><i>New flotation plant Veliki Krivelj monitoring plan</i>   | 187-190 |
| <b><u>Miomir Mikić, Radmilo Rajković, Daniel Kržanović, Sandra Milutinović</u></b> (SERBIA)<br><i>Recultivation of open pit Veliki Krivelj</i>  | 191-194 |
| <b><u>Farzet Bikić, Khaola Awad, Halim Prčanović, Mirnes Duraković</u></b> (BOSNIA AND HERZEGOVINA)<br><i>Analysis of influenced factors on tropospheric ozone content in the city of Zenica during 2020</i>          | 195-198 |
| <b><u>Sandra Milutinović, Ljubiša Obradović, Daniel Kržanović, Miomir Mikić, Radmilo Rajković</u></b> (SERBIA)<br><i>Flotation tail storage methods</i>   | 199-202 |
| <b><u>Sandra Milutinović, Milena Kostović, Ljubiša Obradović, Srđana Magdalinović, Sanja Petrović</u></b> (SERBIA)<br><i>Methods of transportation and discharge of tails to flotation tailings pond</i>              | 203-206 |
| <b><u>Uğur Gürol, Ceren Çelik, Müesser Göçmen, Mustafa Koçak</u></b> (TURKEY)<br><i>Microstructural and mechanical characterization of armor steel joint welded with sandwich design</i>                              | 207-210 |
| <b><u>Branka Pešovski, Milan Radovanović, Vesna Krstić, Danijela Simonović, Silvana Dimitrijević</u></b> (SERBIA)<br><i>Electrochemical characteristics of the anodized titanium oxide films in sulfuric acid</i>     | 211-215 |
| <b><u>Duško Đukanović, Nemanja Đokić, Zoran Aksentijević, Daniel Radivojević, Branisl Stakić</u></b> (SERBIA)<br><i>Methane as an untapped energy potential of the "Soko" brown coal mine</i>                         | 216-220 |
| <b><u>Žaklina Tasić, Marija Petrović Mihajlović, Ana Simonović, Milan Radovanović, Maja Nujkić, Milan Antonijević</u></b> (SERBIA)<br><i>Electrochemical methods for the determination of tryptophan and caffeine</i> | 221-224 |
| <b><u>Isidora Milošević, Anđelka Stojanović, Sanela Arsić, Ivica Nikolić, Ana Rakić</u></b><br>(SERBIA)<br><i>Circular economy in the era of Industry 5.0</i>   | 225-228 |

|   |         |
|---|---------|
| <b><u>Almaida Gigović-Gekić, Elvis Agović, Belma Fakić, Hasan Avdušinović</u></b> (BOSNIA AND HERZEGOVINA)<br><i>Effect of delta ferrite on microstructure and hardness welded joints of steel S21800</i>   | 229-232 |
| <b><u>Radmila Marković, Dragana Bozić, Zoran Stevanović, Tatjana Apostolovski Trujić, Vojka Gardić, Ljiljana Avramović, Vesna Marjanović</u></b> (SERBIA)<br><i>Combining neutralization and adsorption methods for metals removal from Saraka stream</i> | 233-236 |
| <b><u>Ana Petrović, Radmila Marković, Emina Požega</u></b> (SERBIA)<br><i>CNTs as potential material for wastewater purification: a review</i>  | 237-240 |
| <b><u>Zdenka Stanojević Šimšić, Ana Kostov, Aleksandra Milosavljević, Slavica Miletić</u></b> (SERBIA)<br><i>Experimental investigations of CuAlNi alloys with 70 at%Cu</i>   | 241-244 |
| <b><u>Ana Kostov, Aleksandra Milosavljević, Zdenka Stanojević Šimšić, Ivan Jovanović</u></b> (SERBIA)<br><i>Determination of melt properties in Cu-Fe alloys</i>  | 245-248 |
| <b><u>Vladimir Nikolić, Milan Trumić</u></b> (SERBIA)<br><i>A simple method of determining of bond work index for finer samples</i>   | 249-252 |
| <b><u>Ivan Jovanović, Novica Staletović</u></b> (SERBIA)<br><i>Management of risk assessment in environmental protection in surface copper mine</i>   | 253-256 |
| <b><u>Jovan P. Šetrajić, Stevo K. Jaćimovski, Siniša M. Vučenović</u></b> (BOSNIA AND HERZEGOVINA)<br><i>Possibility of localized electron states appearance in ultrathin layered crystalline structures</i>  | 257-260 |
| <b><u>Jovica Sokolović, Ivana Ilić, Dragiša Stanujkić, Zoran Štirbanović</u></b> (SERBIA)<br><i>Application of VIKOR method for comparison of the washability of coals</i>  | 261-264 |
| <b><u>Vladimir Jovanović, Dejan Todorović, Branislav Ivošević, Dragan Radulović, Sonja Milićević, Marija Ercegović, Slavica Mihajlović</u></b> (SERBIA)<br><i>The process of obtaining biochar and the development of the products thus obtained</i>      | 265-269 |
| <b><u>Jelena Petrović, Marija Ercegović, Marija Simić, Marija Koprivica, Jelena Dimitrijević, Marija Marković</u></b> (SERBIA)<br><i>Mg/Fe-modified hydrochar with promoted adsorption performances</i>   | 270-273 |
| <b><u>Esra Dokumaci Alkan, Nurdan Ari, Murat Alkan</u></b> (TURKEY)<br><i>A coating application of IN718 via self-propagating high-temperature synthesis method</i>   | 274-277 |
| <b><u>Murat Alkan, Esra Dokumaci Alkan, Dilan Ugurluer, Aslihan Karakanat</u></b> (TURKEY)<br><i>Production of AlCoCrCuXFeNi alloys via self-propagating high-temperature synthesis method</i>  | 278-281 |
| <b><u>Jarmila Trpčevská, Iveta Vasková, Katarína Pauerová, Martina Laubertová, Dušan Oráč</u></b> (SLOVAKIA)<br><i>Zinc volatilization in the primary and the secondary zinc production</i>   | 282-286 |

|  |         |
|--|---------|
| <b><u>Dragan Ignjatović, Lidija Đurđevac Ignjatović, Vanja Đurđevac, Katarina Milivojević, Ivan Jovanović (SERBIA)</u></b><br><i>Application of the numerical method in the definition of a substrate of circular cross section</i>                                    | 287-291 |
| <b><u>Dragan Ignjatović, Lidija Đurđevac Ignjatović, Vanja Đurđevac, Mladen Supić, Dušan Tašić (SERBIA)</u></b><br><i>Influence of the subsoil bearing capacity during formation of high landfills</i>   | 292-296 |
| <b><u>Bojana Živković, Jelisaveta Marjanović, Jelena Đokić, Maja Petrović (SERBIA)</u></b><br><i>Soil and rock properties as a basis for the sanitary landfill settings</i>  | 297-300 |
| <b><u>Milan Gorgievski, Miljan Marković, Nada Štrbac, Vesna Grekulović, Kristina Božinović, Milica Zdravković, Marina Marković (SERBIA)</u></b><br><i>Adsorption kinetics for copper ions adsorption onto onion peels</i>  | 301-304 |
| <b><u>Saba Nourozi, Fatemeh Pourasgharian, Ahmad Khodadadi Darban (IRAN)</u></b><br><i>Recovery of copper from low-grade copper ore using organic acid</i>   | 305-308 |
| <b><u>Maria Krasteva (BULGARIA)</u></b><br><i>Methodology and equipment for researching corrosion cracking processes in steel 3H14L (BDS 3692-78)</i>  | 309-312 |
| <b><u>Jasmina Nešković, Pavle Stjepanović, Nenad Milojković, Dejan Lazić, Klara Konc Janković, Svetlana Polavder, Ivana Jovanović (SERBIA)</u></b><br><i>Testing the Bond work index on limestone from flue gas desulphurization plant in TPP Ugljevik</i>             | 313-317 |
| <b><u>Biljana Zlatičanin, Sandra Kovačević (MONTENEGRO)</u></b><br><i>Impact of titanium addition on microstructure and properties of as-cast Al-Cu15 alloys</i>   | 318-321 |
| <b><u>Biljana Zlatičanin, Sandra Kovačević (MONTENEGRO)</u></b><br><i>Effect of cooling rate on mechanical properties of binary Al-Cu23 alloys</i>   | 322-324 |
| <b><u>Desislav Ivanov, Irena Peytcheva, Marko Holma (BULGARIA)</u></b><br><i>Horizon Europe AGEMERA project - Agile Exploration and Geo-modelling for European Critical Raw Materials: The potential of Assarel porphyry copper deposit for critical raw materials</i> | 325-328 |
| <b><u>Shehret Tilvaldyev, Uzziel Caldiño Herrera, Jose Omar Davalos, Manuel Alejandro Lira Martinez, Marlenne Alejandra Hernandez Lira, Diego Adan Villordo Melendez (CANADA)</u></b><br><i>Problems of anthropogenic pollution of space</i>                           | 329-334 |
| <b><u>Mohammed Derqaoui, Abdelmoughit Abidi, Abdelrani Yaacoubi, Khalid El Amari, Omar Oabi, Abdelaziz Bacaoui (MOROCCO)</u></b><br><i>Apatite flotation from low-grade sedimentary phosphate ore</i>  | 335-338 |
| <b><u>Nadezhda Kazakova, Alexandar Popov, Georgi Chernev (BULGARIA)</u></b><br><i>Influence of the distribution and content of limestone particles on the properties of blended cements</i>  | 339-342 |

|   |         |
|---|---------|
| <b><u>Daniel Ogochukwu Okanigbe, Shade Rouxzeta Van Der Merwe</u> (SOUTH AFRICA)</b><br><i>Rocks of Obafemi Awolowo University and Environ, Nigeria: structural analysis of geological contact</i>  | 343-347 |
| <b><u>Vladan Kašić, Ana Radosavljević Mihajlović, Jovica Stojanović, Slavica Mihajlović, Melina Vukadinović, Nataša Đorđević, Ivana Jelić</u> (SERBIA)</b><br><i>Study of thermally treated zeolitic tuffs of Serbia, deposits "Zlatokop" and "Općište"-Beočin</i>    | 348-352 |
| <b><u>Vesna Grekulović, Aleksandra Mitovski, Milica Zdravković, Nada Štrbac, Milan Gorgievski, Milovan Vuković, Miljan Marković</u> (SERBIA)</b><br><i>Electrochemical behavior of copper in chloride medium in the presence of nettle extract</i>                    | 353-356 |
| <b><u>Marko Pavlović, Marina Dojčinović, Muhamed Harbinja, Atif Hodić, Dragan Radulović, Mirjana Stojanović, Zagorka Aćimović</u> (SERBIA, BOSNIA AND HERZEGOVINA)</b><br><i>Effects of the application of pyrophyllite in the composition of protective coatings</i> | 357-360 |
| <b><u>Tamara Ristić, Nenad Milosavljević, Dobrica Milovanović</u> (SERBIA)</b><br><i>Measures for the processing of iron with a higher incoming phosphorus content at the steel shop</i>  | 361-365 |
| <b><u>Ivana Mikavica, Dragana Randelović, Milena Obradović, Jovica Stojanović, Jelena Mutić</u> (SERBIA)</b><br><i>Microplastic textile fibers in urban soils of Serbia</i>   | 366-369 |
| <b><u>Jianbo Zhao, Xinnan Zhao, Donglai Ma, Yang You, Zhixiong You, Xuewei Lv</u> (CHINA)</b><br><i>Preparation of ferronickel by semi-molten smelting a mixture of two types of laterite ore</i>   | 370-374 |
| <b><u>Mladen Radovanović, Dejan Petrović, Jelena Ivaz, Dragan Zlatanović</u> (SERBIA)</b><br><i>Possibility of copper ores exploitation using in situ leaching method</i>   | 375-378 |
| <b><u>Ivan Jelić, Nikola Lekić, Nikola Stanić, Miomir Mikić</u> (SERBIA)</b><br><i>Selection of an optimal route for relocation of the Čehotina river bed</i>   | 379-382 |
| <b><u>Milica Zdravković, Vesna Grekulović, Bojan Zdravković, Nada Štrbac, Milan Gorgievski, Miljan Marković</u> (SERBIA)</b><br><i>Electrochemical behavior of steel in 0.1 mol/dm<sup>3</sup> HCl in the presence of potato peel juice</i>                           | 383-386 |
| <b><u>Ivana Marković, Dalibor Jović, Uroš Stamenković, Dragan Manasijević, Ljubiša Balanović, Milan Gorgievski</u> (SERBIA)</b><br><i>Microstructure and thermal properties of leaded brass after quenching</i>   | 387-390 |
| <b><u>Mehmet Ali Yildiz</u> (SERBIA)</b><br><i>Hot strip mill walking beam slab reheating project</i>   | 391-394 |
| <b><u>Peter Polyak</u> (SERBIA)</b><br><i>Finishing mill automation upgrade at hot strip mill</i>   | 395-400 |
| <b><u>Branislav Potić, Ana Arifović</u> (SERBIA)</b><br><i>The metallurgical testing results of the boron mineralized material from Valjevo-Mionica basin</i>   | 401-406 |



|   |         |
|---|---------|
| <b>Uroš Stamenković, Ivana Marković, Srba Mladenović, Saša Marjanović, Avram Kovačević, Milijana Mitrović, Filip Basarabić (SERBIA)</b><br><i>The influence of quenching media on different properties of C45 carbon steel</i>  | 407-413 |
| <b>Yang You, Jiabao Guo, Zhixiong You, Xuewei Lv (CHINA)</b><br><i>Investigation of the mixing and granulation behavior of iron ore fines in horizontal high-shear granulator</i>   | 414-417 |
| <b>Jovica Sokolović, Grozdanka Bogdanović, Velizar Stanković, Gracijan Strainović, Ivana Ilić, Milan Gorgievski, Miljan Marković (SERBIA)</b><br><i>Investigation on beneficiation of iron from copper ore of Mauritania Copper Mine (MCM) by magnetic separation</i> | 418-421 |
| <b>Essen Suleimenov, Rustam Sharipov, Galymzhan Maldybayev, Zhibek Orazaliyeva (KAZAKHSTAN)</b><br><i>Investigation of the influence of pulsed electric current on the efficiency of decomposition of aluminate solution</i>  | 422-423 |
| <b>Lovro Liverić, Tamara Holjevac Grgurić, Sunčana Smokvina Hanza, Wojciech Sitek, Vedrana Špada, Marko Kršulja (CROATIA)</b><br><i>Influence of silver content on martensitic transformation of Cu-Al-Ag alloy</i>   | 424-427 |
| <b>Hasan Ali Taner, Vildan Onen (TURKEY)</b><br><i>Evaluation of the efficiency of different collectors in the chalcopyrite flotation</i>   | 428-434 |
| <b>Vesna Conić, Dragana Božić, Miloš Janošević, Ljiljana Avramović, Vanja Trifunović, Dejan Bugarin, Ivana Jovanović (SERBIA)</b><br><i>A pyro-hydrometallurgical process for the recovery of zinc from jarosite waste</i>  | 435-438 |
| <b>Maria Krasteva, Rumen Petkov (BULGARIA)</b><br><i>Research the rate of chemical corrosion of steel 3X14H2 (BDS 3692-78)</i>  | 439-442 |
| <b>Srba Mladenović, Bojan Novaković, Ivana Marković, Uroš Stamenković (SERBIA)</b><br><i>Effect of casting speed and water flow on tensile strength, elongation and microstructure of continuous cast copper wire</i>   | 443-447 |
| <b>Nadira Bušatlić, Ilhan Bušatlić, Dženana Smajić-Terzić (BOSNIA AND HERZEGOVINA)</b><br><i>Dependence of compressive strength of geopolymer based on fly ash and alkaline activator ratio</i>   | 448-451 |
| <b>Gergana Meracheva, Efrosima Zaneva-Dobranova, Nikolay Hristov (BULGARIA)</b><br><i>Hydrocarbon potential of the Lower Paleozoic sediments in NE Bulgaria by geochemistry and well-logging</i>  | 452-455 |
| <b>Dragana Marilović, Grozdanka Bogdanović, Sanja Petrović (SERBIA)</b><br><i>Leaching of flotation tailings with a solution of sulfuric acid and ionic liquid</i>  | 456-459 |
| <b>Ivana Jovanović, Vesna Conić, Dragan Milanović, Daniel Kržanović, Tanja Stanković, Daniela Urošević, Miloš Janošević (SERBIA)</b><br><i>Determination of Bond rod mill work index of a very low-grade copper ore</i>   | 460-463 |

|   |         |
|---|---------|
| <b><u>Hasan Ali Taner, Ali Aras, Muhammad Hashim Rasa</u> (TURKEY)</b><br><i>Investigation of the effect of depressant and collector conditioning times on cobalt recovery by flotation</i>   | 464-467 |
| <b><u>Aleksandar Cvetković, Žaklina Tasić, Marija Petrović Mihajlović, Maja Nujkić, Milan Radovanović, Ana Simonović</u> (SERBIA)</b><br><i>Microplastics</i>   | 468-471 |
| <b><u>Sanja Petrović, Srđana Magdalinović, Ljubiša Obradović, Sandra Milutinović, Bojan Drobnjaković, Slađana Krstić</u> (SERBIA)</b><br><i>Tailing management: tailings filtering equipment</i>  | 472-475 |
| <b><u>Jelena Stefanović, Jelena Đorđević, Sandra Guševac</u> (SERBIA)</b><br><i>XRD analysis of corrosion product formed in industrial aggressive environment</i>   | 476-480 |
| <b>Muhamad Ghulam Isaq Khan, Filip Rajković, Miljana Popović, Dejan Prelević, Aleksandar Ćitić, Tamara Radetić</b> (SERBIA)<br><i>Initiation of abnormal grain growth in cold-rolled sheet of AA5182 Al-Mg alloy: role of texture</i>   | 481-484 |
| <b><u>Danijela Voza, Hesam Dehghani, Milica Veličković</u> (SERBIA)</b><br><i>The dissolved oxygen prediction based on the machine learning techniques</i>  | 485-488 |
| <b><u>Hasan Acan, Hasan Ergin</u> (TURKEY)</b><br><i>A novel model for minimizing mine closure costs and the optimum final quarry boundry</i>   | 489-492 |
| <b><u>Ivana Jovanović, Dragan Milanović, Oliver Dimitrijević, Vesna Conić, Igor Svrkota</u> (SERBIA)</b><br><i>Role of wing tank in DMS process. Suspension velocity through the seal leg orifice – case study</i>  | 493-496 |
| <b><u>Dejan Petrović, Jelena Ivaz, Saša Stojadinović, Predrag Stolić, Dragan Zlatanović</u> (SERBIA)</b><br><i>Risk management and mining machines maintenance – a brief review</i>   | 497-500 |
| <b><u>Stefan Đorđievski, Dragana Adamović</u> (SERBIA)</b><br><i>History of surface water pollution by mining and metallurgical activities in Bor, Serbia</i>   | 501-504 |
| <b><u>Olivera Dragutinović, Vaso Manojlović, Đorđe Veljović, Stefan Dikić, Marko Simić</u> (SERBIA)</b><br><i>Investigation of the properties of Co-Cr-W and Co-Cr-Mo alloys coated with hydroxyapatite for use in dental implants</i>  | 505-509 |
| <b><u>Zoran Karastojković, Dragoslav Gusković, Ognjen Ristić, Zorica Kovačević</u> (SERBIA)</b><br><i>About the “relative plasticity” between steel matrix and non-metallic inclusions</i>  | 510-513 |
| <b><u>Aleksandar Jovanović, Mladen Bugarčić, Milena Milošević, Marija Vuksanović, Muna Abdualatif Abdurahman, Miroslav Sokić, Aleksandar Marinković</u> (SERBIA, LIBYA)</b><br><i>Modified hybrid cellulose membrane for Nickel(II) ions removal from industrial wastewater</i> | 514-517 |
| <b><u>Elena Todorova, Nadezhda Kazakova, Georgi Chernev</u> (BULGARIA)</b><br><i>Structural investigation via SEM analysis of silica hybrid materials</i>   | 518-521 |

|   |         |
|---|---------|
| <b><u>Tanja Kalinović, Jelena Kalinović, Jelena Milosavljević, Ana Radojević, Snežana Šerbula (SERBIA)</u></b><br><i>Atmospheric bulk deposition as environmental quality indicator</i>   | 522-526 |
| <b><u>Gordana Marković, Vaso Manojlović, Miroslav Sokić, Jovana Ružić, Dušan Milojkov (SERBIA)</u></b><br><i>Designing biocompatible high entropy alloys using Monte Carlo simulations</i>  | 527-530 |
| <b><u>Tatjana Volkov-Husović, Sanja Martinović, Ana Alil, Milica Vlahović (SERBIA)</u></b><br><i>Application of image analysis for cavitation erosion resistance monitoring of some engineering materials</i>                         | 531-534 |
| <b><u>Milan Nedeljković, Srba Mladenović, Jasmina Petrović, Milijana Mitrović (SERBIA)</u></b><br><i>Changes in the structure and density of copper during the refining smelting process</i>  | 535-538 |
| <b><u>Jasmina Petrović, Srba Mladenović, Ivana Marković, Milan Nedeljković, Milijana Mitrović (SERBIA)</u></b><br><i>Microstructure analysis of EN AW 6061 alloy using a SEM microscope after artificial aging</i>                    | 539-542 |
| <b><u>Milijana Mitrović, Saša Marjanović, Biserka Trumić, Jasmina Petrović, Milan Nedeljković (SERBIA)</u></b><br><i>Effects of cold rolling and annealing processes on the microstructure and properties of micro-alloyed copper</i> | 543-546 |
| <b><u>Makedonka Dimitrova, Jasminka Dimitrova Kapac (NORTH MACEDONIA)</u></b><br><i>Unlocking energy efficiency: financing preferences for SMEs in the Republic of North Macedonia</i>  | 547-555 |
| <b><u>Zoran Štirbanović, Vesna Vojinović, Jovica Sokolović, Maja Trumić (SERBIA)</u></b><br><i>Analysis of the effectiveness of different methods for cutting samples</i>   | 556-559 |
| <b><u>Ivica Nikolić, Isidola Milošević, Anđelka Stojanović (SERBIA)</u></b><br><i>Land turnover increases due to mining: An empirical analysis of Bor, Serbia, 2013-2022.</i>   | 560-563 |
| <b>DONORS</b>   | 565-590 |
| <b>AUTHOR INDEX</b>   | 591-596 |

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## MODIFIED HYBRID CELLULOSE MEMBRANE FOR NICKEL(II) IONS REMOVAL FROM INDUSTRIAL WASTEWATER

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### Abstract

Nickel pollution of water induces several problems for the environment. The purpose of this paper was to investigate the adsorption of Ni<sup>2+</sup> ions on fabricated biomembranes. The proposed adsorbent was prepared from epoxy and amino-functionalized waste cellulose fibers, able to participate in cross-linking with amino acid lysine - wCell/Mn-Fe\_LDH. The prepared material underwent preliminary structural characterization by Fourier-transform infrared spectroscopy. In a batch system, the influence of pH, contact time, temperature, and initial concentration on adsorption efficiency was investigated. The effectiveness of the membrane was demonstrated by acceptable adsorption capacities of 40.49 mg g<sup>-1</sup> obtained for Ni<sup>2+</sup> at 45°C. The kinetic study, using the Weber-Morris model, indicates intraparticle diffusion as the rate limiting step. Adsorption mechanism physisorption was proposed based on thermodynamic behaviors. The outcomes demonstrated that environmentally friendly sustainable technology has been successfully developed.

**Keywords:** water treatment, wCell/Mn-Fe\_LDH, adsorption, environmental engineering.

### 1. INTRODUCTION

The total environment, including the air, water, and soil, is widely distributed with a transition element nickel. Nickel might originate from both anthropogenic activity and natural sources. Industries, the use of liquid and solid fuels, as well as municipal and industrial waste, may all contribute to nickel contamination of the environment. Consequently, exposure to nickel can have several adverse effects on people, including allergies, kidney and heart problems [1]. Although it can exist in some oxidative states, the +2 oxidation state (Ni<sup>2+</sup>) is the most prevalent [1]. Water pollution by nickel under the special attention of researchers.

Treatment of wastewaters can be performed by utilizing different processes like photocatalysis [2], adsorption [3], electrochemical oxidation [4] and so on. Among them, adsorption poses the most viable and economical way to solve problems in this area. Various materials are employed during different probes, but hybrid (combination of organic/inorganic) materials show the best results. Due to their numerous uses in catalysis, energy conversion and storage, medicine delivery, environmental cleanup, and carbon dioxide preservation, layered double hydroxides (LDH) are a diverse class of materials [5]. On the other hand, one of nature's most plentiful and eco-friendly polymers is cellulose [6]. Crude cellulose exhibits a poor level of adsorption ability. Because of

this, it has been common practice to chemically or physically modify cellulose to increase its capacity to bind heavy metals [7]. A combination of LDH and cellulose derivates can be the solution for before mentioned issues.

The main objective of the present study was to develop a novel wCell/Mn-Fe\_LDH adsorbent with high affinity toward metal ions (+2 oxidation state). Determination of the ability to remove Ni(II) as a model pollutant by varying process conditions, during adsorption tests, was performed.

## 2. EXPERIMENTAL

### 2.1 Synthesis of biobased membrane

All acquired chemicals for synthesis were p. a. grade without needing further purification (all obtained from Merck). Preparation of membranes was done according to Perendija et al. [1] with deposition of Mn-Fe\_LDH [8] onto the surface of wCells.

### 2.2 Structural characterization

Preliminary structural properties (the presence of organic and inorganic bond in materials) were obtained by Fourier-transform infrared spectroscopy (FTIR). FTIR spectra were collected with a Nicolet™ iS™ 10 FT-IR Spectrometer (Thermo Fisher SCIENTIFIC) with Smart iTR™ Attenuated Total Reflectance (ATR) Sampling accessories. The spectra were recorded in the range 4000-500 cm<sup>-1</sup>, in 20 scans mode, and at a resolution of 4 cm<sup>-1</sup>.

### 2.3 Adsorption experiments

Adsorption tests were carried out by adding 1, 2, 3, 4, 5, 7.5, and 10 mg of adsorbent to an 8 cm<sup>3</sup> vial of standard Ni<sup>2+</sup> solutions with  $C_i = 7.55 \text{ mg dm}^{-3}$ . Adsorptions were conducted at 25, 35, and 45 °C and pH 7. Atomic absorption spectrometry (AAS) was used to determine the amounts of Ni<sup>2+</sup> ions (Perkin Elmer AAnalyst 300). Similar to the equilibrium study, the kinetic study was carried out by altering the contact time: 1, 5, 15, 30, 45, 60, 75, and 90 minutes at 25, 35, and 45 °C.

Using Eq. (1), the adsorption capacity was determined:

$$q = \frac{(C_i - C_f)}{m} V \quad (1)$$

where  $q$  is the adsorption capacity in mg g<sup>-1</sup>,  $C_i$  and  $C_f$  are the initial and final concentrations of ions in the solution in mg dm<sup>-3</sup>,  $V$  is the volume of solution in dm<sup>-3</sup>, and  $m$  is the mass of the adsorbent in g.

## 3. RESULTS AND DISCUSSION

### 3.1 Structural characterization of prepared material

Preliminary structural characterization of fabricated material is presented in Figure 1. ATR-FTIR scans of precursors and finally obtained membrane are depicted together in Figure 1.

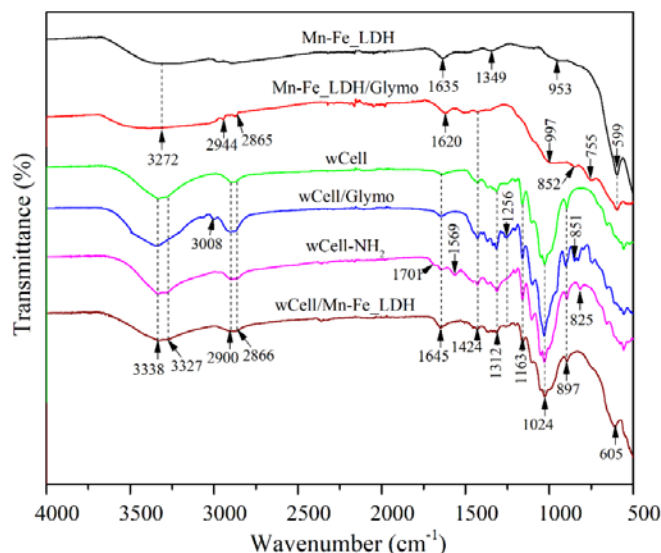


Figure 1 - FTIR spectra of membrane constituents and wCell/Mn-Fe\_LDH membrane

The wCell/Mn-Fe\_LDH membrane's FTIR spectrum shows the existence of all constituent functionalities that were included during cross-linking and Mn-Fe\_LDH deposition. A peak at 605  $\text{cm}^{-1}$  confirms that Mn-Fe\_LDH successfully covered the surface.

### 3.2 Adsorption results

Removal of  $\text{Ni}^{2+}$  ions from water solution was described by the Langmuir model. Results in Table 2 for  $\text{Ni}^{2+}$  demonstrate that  $q_m$  decreases as temperature rises. The strong connections between the functional groups on the adsorbent surface and the adsorbate were shown by the high values of the Langmuir constant ( $K_L$ ), which measures adsorption affinity.

Table 1 - The results of non-linear fitting using Langmuir isotherm for  $\text{Ni}^{2+}$  adsorption onto wCell/Mn-Fe\_LDH membrane

| Langmuir model   | $T$ ( $^{\circ}\text{C}$ ) | $q_m$ ( $\text{mg g}^{-1}$ ) | $K_L(\text{dm}^3 \text{mg}^{-1})$ | $K_L (\text{dm}^3 \text{mol}^{-1})$ | $R^2$ |
|------------------|----------------------------|------------------------------|-----------------------------------|-------------------------------------|-------|
| $\text{Ni}^{2+}$ | 25                         | 42.68±1.45                   | 6.289±0.097                       | 369120.2±5693.2                     | 0.999 |
|                  | 35                         | 41.68±1.54                   | 5.538±0.10                        | 325017.5±5927.5                     | 0.998 |
|                  | 45                         | 40.49±1.64                   | 4.775±0.11                        | 280273.9±6339.2                     | 0.999 |

Thermodynamic parameters are shown in Table 2.

Table 2 - Calculated thermodynamic parameters for  $\text{Ni}^{2+}$  adsorption onto wCell/Mn-Fe\_LDH membrane

| $T$ (K) | $\Delta G^{\ominus}$ ( $\text{kJ mol}^{-1}$ ) |        |        | $\Delta H^{\ominus}$ ( $\text{kJ mol}^{-1}$ ) | $\Delta S^{\ominus}$ ( $\text{J mol}^{-1} \text{K}^{-1}$ ) | $R^2$ |
|---------|---|--------|--------|---|--|-------|
|         | 298   | 308    | 318    |   |  |       |
|         | -41.73  | -42.81 | -43.80 | -10.85  | 103.63   | 0.996 |

At all temperatures, negative Gibbs free energy ( $\Delta G^{\ominus}$ ) values demonstrate the spontaneity of adsorption onto wCell/Mn-Fe\_LDH (Table 2). The exothermic nature of the whole process is mirrored by lower energy  $\text{Ni}^{2+}$  electrostatic interactions with surface functions, on the other hand. The increasing randomness of the entire system at the established equilibrium state and the high survivability of the examined processes are both indicated by positive entropy change ( $\Delta S^{\ominus}$ ).



Table 3 - PSO model parameters and activation energy ( $E_a$ ) for the adsorption of  $\text{Ni}^{2+}$  ions onto wCell/Mn-Fe\_LDH membrane at 25, 35, and 45 °C

| <i>Ion</i>       | <i>T</i> (°C) | $q_e$ (mg g <sup>-1</sup> ) | $k_2$ (g (mg min) <sup>-1</sup> ) | $R^2$ | $E_a$ (kJ mol <sup>-1</sup> ) |
|------------------|---------------|-----------------------------|-----------------------------------|-------|-------------------------------|
| $\text{Ni}^{2+}$ | 25            | 52.22±1.34                  | 0.00190±0.00052                   | 0.997 | 21.26                         |
|                  | 35            | 50.33±1.22                  | 0.00246±0.00048                   | 0.998 |                               |
|                  | 45            | 48.59±0.99                  | 0.00325±0.00018                   | 0.999 |                               |

The diffusion-controlled processes i.e., ion exchange/complexation are characteristic for the values of  $E_a$  between 8 and 40 kJ mol<sup>-1</sup> [9] and according to the results from Table 3, the diffusion is a rate-controlling step.

#### 4. CONCLUSION

FTIR analysis of obtained composite membrane wCell/Mn-Fe\_LDH showed the presence of functionalities that enable very high adsorption affinity towards cation species. Correlating the equilibrium adsorption results with Langmuir adsorption isotherm model give fine fitting results, and these experiments also showed that adsorption process is exothermic and spontaneous. Kinetics of adsorption process showed that process is in higher order of reaction (third order or higher) which is expected for such kind of materials (composites with complex chemical structure). Performing continuous measurements of adsorption capacity as a function of contact time on different temperatures provided the data needed to determine activation energy of the adsorption process. Obtained value of only 21.26 kJ mol<sup>-1</sup> indicate that adsorption process is very fast due to very low activation energy barrier. In current study we utilized this membrane as an adsorbent of nickel (II) ions and the results we obtained showed that this composite membrane may be perspective choice for cation pollutants diminution.

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