

UNIVERSITY OF BELGRADE
TECHNICAL FACULTY BOR

**52nd International October Conference on
Mining and Metallurgy**



PROCEEDINGS

Edited by

Saša Stojadinović

and

Dejan Petrović

November 29th – 30th 2021

Bor, Serbia

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TABLE OF CONTENTS

Aleksandra Milosavljević

THE COMPLEXITY OF SEM-EDS – WHAT AFFECTS THE QUALITY OF OBTAINED RESULTS? 1

Zoran Karastojković, R Perić, M Srećković

LASER QUENCHING OF CUTTING TOOL STEELS - A REVIEW 5

Slavica Miletić, D Bogdanović, E Požega

IMPACT OF EXTRAORDINARY SECURITY MEASURES TO EMPLOYEES DURING THE PANDEMIC COVID-9 15

Daniela Grigorova, R Paunova

KINETIC STUDY OF SOLID-PHASE REDUCTION OF POLYGRADIENT IRON-CONTAINING MATERIAL 19

Emina Požega, D Simonović, S Marjanović, M Jovanović, L Gomidželović, M Mitrović, Z Stanojević Šimšić

PART I: WHAT MAKES A GOOD THERMOELECTRIC 23

Emina Požega, D Simonović, S Marjanović, M Jovanović, L Gomidželović, M Mitrović, S Miletić

PART II: WHAT MAKES A GOOD THERMOELECTRIC 27

Dragan Manasijević, Lj Balanović, I Marković, M Gorgievski, U Stamenković, K Božinović, D Minić, M Premović

STUDY OF MICROSTRUCTURE AND THERMAL CONDUCTIVITY OF THE Ag–Bi–Sn ALLOYS 31

Vladimir S. Topalović, S Matijašević, S Grujić, J Stojanović, J Nikolić, V Savić, S Zildžović

THE INFLUENCE OF THE PARTICLE SIZE ON CRYSTALLIZATION OF GLASS POWDERS FROM THE SYSTEM $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{GeO}_2-\text{P}_2\text{O}_5$ 35

Vesna Marjanović, R Marković, V Krstić

TECHNOLOGIES FOR PHYSICAL TREATMENT OF WATER CONTAINING SELENIUM: A REVIEW 39

Vesna Marjanović, R Marković, V Krstić

TECHNOLOGIES FOR BIOLOGICAL TREATMENT OF WATER CONTAINING SELENIUM: A REVIEW 43

Milenko Jovanović, M Mikić, M Maksimović, D Kržanović, R Rajković, E Požega

USAGE SPECIFICS OF GEOGRIDS 47

Srećko Manasijević, Z Zovko Brodarac, N Dolić, M Djurdjević, R Radiša	
INTERMETALLIC BONDING BETWEEN A RING CARRIER AND AN ALUMINUM PISTON ALLOY	51
Snežana Šarboh	
PATENTED INVENTIONS OF LJUBOMIR KLERIĆ	55
Miomir Mikic, M Jovanović, R Rajković, D Kržanović, E Požega	
DEGRADED AREA OF VELIKI KRIVELJ QUARRY RECULTIVATION	59
Dragana Adamović, D Ishiyama, H Kawaraya, O Yasumasa	
EFFECTS OF TAILINGS ON GROUNDWATER ALONG BOR AND BELA RIVERS IN THE BOR MINING AREA, EASTERN SERBIA	63
Ana Kostov, Z Stanojević Šimšić, A Milosavljević,	
CHARACTERIZATION OF ALLOYS CuAlAu0.5	67
Marija Milenković, V Jovanović, J Paunković, V Krstić	
MULTICRITERIA ANALYSIS OF THE LEVEL OF SUSTAINABLE DEVELOPMENT OF THE TOPLICA DISTRICT USING THE ELECTRE METHOD	71
Daniel Kržanović, R Rajković, D Stevanović, M Mikić, M Jovanović, S Petrović	
LONG-TERM PLANNING OF MINING THE LEAD AND ZINC ORE DEPOSIT IN THE BRSKOVO ORE FIELD, THE REPUBLIC OF MONTENEGRO	75
Radmilo Rajković, D Kržanović, M Mikić, M Jovanović	
CALCULATION OF SAFETY DISTANCE FOR THE OPERATION OF MINING EQUIPMENT IN THE WORKING ENVIRONMENT WITH WEAKENED CHARACTERISTICS AT THE OPEN PIT "NORTH MINING DISTRICT" OF THE COPPER MINE MAJDANPEK	79
Zdenka Stanojević Šimšić, A Kostov, A Milosavljević, E Požega	
HARDENSS, MICROHARDNESS AND ELECTROCONDUCTIVITY OF ALLOYS WITH VARIABLE Cu CONTENT IN Cu-Al-Ag SYSTEM	83
Miodrag Banješević	
STRATIGRAPHY AND AGE OF ROCK UNITS AND MINERALIZATION IN THE TIMOK MAGMATIC COMPLEX AND THE BOR METALLOGENIC ZONE – A REVIEW	87
Milan Radivojević, Z Stević, M Tanasković	
DUALPHASED FOURWAYINTERSECTION REGULATED BY TRAFFIC LIGHTS WITH FIXED AND ADAPTIVE MOD OF OPERATION	93
Filip Gramić, N Rančić, S Filipović, J Đorđević	
USE OF COPPER TAILING AND COPPER SLAG IN 3D PRINTED CONCRETE PROCESSES	97
Filip Gramić, N Rančić, S Filipović, J Đorđević,	
POSSIBILITY OF USING MINING WASTE IN THE PRODUCTION OF BRICK PRODUCTS	101

Stepan O. Vidysh

GOLD-SILVER ALLOYS ANODIC DISSOLUTION RESEARCH IN HYDROCHLORIC ACID ELECTROLYTES **105**

Milan Gorgievski, M Marković, D Božić, Vr Stanković, N Štrbac, V Grekulović, M Zdravković

ADSORPTION ISOTHERMS FOR COPPER IONS ADSORPTION ONTO WALNUT SHELLS **109**

Miljan Marković, M Gorgievski, N Štrbac, V Grekulović, A Mitovski, K Božinović, M Zdravković

pH AND CONDUCTIVITY CHANGE DURING THE RINSING AND ADSORPTION OF COPPER IONS ONTO WALNUT SHELLS **113**

Vesna Grekulović, A Mitovski, M Rajčić Vujasinović, N Štrbac, M Zdravković, M Gorgievski, M Marković

ELECTROCHEMICAL BEHAVIOR OF COPPER IN CHLORIDE MEDIUM IN THE PRESENCE OF WALNUT SHELL MACERATE **117**

Marija Šljivić-Ivanović, S Dimović, I Jelić,

EXPERIMENTAL DESIGN APPROACH IN RADIONUCLIDE SORPTION **121**

Ivana Jelić, A Savić, M Šljivić-Ivanović, S Dimović

INFLUENCE OF SILICA FUME ON SCC CONCRETE PROPERTIES **125**

Milan Radovanović, A Simonović, M Petrović Mihajlović, Ž Tasić, V Nedelkovski, M Antonijević

L-LYSINE AS CORROSION INHIBITOR OF STAINLESS STEEL IN RINGER'S SOLUTION **129**

Dragana Marilović, M Trumić, M Trumić, Lj Andrić

THE INFLUENCE OF CALCIUM IONS ON DEINKING FLOTATION RECOVERY UNDER DIFFERENT CONDITIONS **133**

Dragana Medić, S Milić, S Alagić, M Nujkić, S Đorđević, A Papludis

OPTIMIZATION OF CATHODIC MATERIAL LEACHING PROCESS IN ACID-SULPHATE SOLUTION **137**

Milijana Mitrović, D Gusković, S Marjanović, B Trumić, E Požega, U Stamenković, J Petrović

OBTAINING MULTILAYER COPPER STRIPS BY ARB (ACCUMULATIVE ROLL BONDING) ROLLING PROCESS **141**

Nataša Đorđević, S Mihajlović, N Obradović, A Peleš, S Filipović

THE INFLUENCE OF HIGH COMPACTION PRESSURE ON CORDIERITE-BASED CERAMICS **145**

Nataša Đorđević, S Mihajlović, M Sokić, B Marković

SEM AND X-RAY ANALYSES OF SINTERED MgO / Bi₂O₃ BINARY SYSTEM **149**

Ivana Ilić, J Sokolović, M Trumić, Z Stirbanović

**COMPARATIVE RESULTS OF COPPER FLOTATION FROM SLAG BEFORE AND AFTER THE
PROCESS OF MAGNETIC CONCENTRATION** **153**

Daniela Grigorova

FERROSILICON OBTAINING USING IRON-SILICATE –FAYALITE **157**

Slavica Mihajlović, M Jovanović, N Đorđević, A Patarić, M Vlahović, V Kašić

THE CLAY PRELIMINARY TESTING FROM MUNICIPALITY AREA OF REKOVAC **161**

Milan Milosavljević, M Premović, D Minić, Dn Mansijević, Ar Đorđević, M Kolarević

**EXPERIMENTAL AND THERMODYNAMIC STUDY OF ISOTHERMAL SECTIONS AT 600 °C
AND 400 °C OF TERNARY Bi-Cu-Ge SYSTEM** **165**

Aleksandar Đorđević, D Minić, M Premović, D Mansijević, M Milosavljević, V Ristić

STUDY OF TEMPERATURE PHASE TRANSFORMATION OF THE TERNARY Bi-Cu-Ge SYSTEM **169**

Aleksandar Savić, I Jelić, M Šljivić-Ivanović, S Dimović, N Pudar, A Pfandler

**RECYCLED COARSE AGGREGATE AND FLY ASH EFFECT ON COMPRESSIVE STRENGTH OF
SELF-COMPACTING CONCRETE** **173**

Vladan Kašić, D Životić, V Simić, A Radosavljević-Mihajlović, J Stojanović, S Mihajlović, M
Vukadinović

FORECAST RESOURCES OF ZEOLITHIC TUFFS OF SERBIA **177**

Vladan Kašić, A Radosavljević-Mihajlović, S Radosavljević, J Stojanović, S Mihajlović, M
Vukadinović

**GEOLOGICAL AND MINERAL CHARACTERISTICS OF ZEOLITHIC TUFF TOPONICA DEPOSITS
NEAR KOSOVSKA KAMENICA** **181**

Konstantin Petkov, V Stefanova, P Iliev

**METHOD FOR UTILIZATION OF THE SULFURIC ACID OBTAINED DURING AUTOCLAVE
DISSOLUTION OF PYRITE CONCENTRATE** **185**

Stefan Đorđević, D Ishiyama, Y Ogawa, Z Stevanović, O Osenyeng, D Adamović, V
Trifunović

**MONITORING OF pH VALUE AND CONCENTRATION OF COPPER IN
RIVERS DOWNSTREAM FROM BOR MINE IN PERIOD 2015-2021** **189**

Viša Tasić, M Cocić, B Radović, T Apostolovski-Trujić

**CHEMICAL COMPOSITION OF PARTICULATE MATTER IN THE INDOOR AIR AT THE
TECHNICAL FACULTY IN BOR (SERBIA)** **193**

Snežana Ignjatović, I Vasiljević, M Negovanović

DEFINING STRUCTURAL CORRELATION USING OF TOTAL HORIZONTAL GRADIENT **197**

Velizar Stanković, M Janošević

**INCREASING THE CAPACITY OF THE COPPER SMELTING COMPANY IN THE COMPANY
"SERBIA ZIJIN COPPER" - CHALLENGES AND CONSEQUENCES TO THE ENVIRONMENT** 201

Vladimir Jovanović, D Todorović, B Ivošević, D Radulović, S Milićević, D Nišić

**CHARACTERIZATION OF PELLET SAMPLES OBTAINED BY PELETIZATION OF LIMESTONE
AND SEAWEED** 205

Vanja Trifunović, L Avramović, R Jonović, S Milić, S Đorđievski, M Jonović

**HYDROMETALLURGICAL TREATMENT OF ELECTRIC ARC FURNACE DUST IN AIM OF ZINC
SEPARATION** 209

Jovana Bošnjaković, N Knežević, N Čutović, M Bugarčić, A Jovanović, Z Veličković, S
Manasijević

**EVALUATION OF ADSORPTION PERFORMANCE OF PHOSPHATES REMOVAL USING CELL-
MG HYBRID ADSORBENT** 213

Dragan Radulović, Lj Andrić, D Božović, V Jovanović, B Ivošević, D Todorović,

**POSSIBILITY OF USING LIMESTONE FROM "PJEŠIVAČKI DO"-DANILOVGRAD DEPOSIT AS
FILLER IN VARIOUS INDUSTRY BRANCHES** 217

Predrag Stolić, J Ivaz, D Petrović, Zoran Stević

**ADVANTAGES OF MINING ENGINEERING CURRICULUM REALIZATION USING
SOLUTIONS BASED ON FREE SOFTWARE** 221

Slađana Krstić, E Požega, S Petrović, S Magdalinović, D Urošević, S Miletić, Z Stojanović
Šimšić

**QUALITY INVESTIGATION OF SAND FOR THE PRODUCTION OF AGGREGATES ON
VINOGRADI LOCALITY (DELIBLATSKA PEŠČARA)** 225

Saša Marjanović, D Gusković, M Mitrović, E Požega, B Trumić, U Stamenković

**INFLUENCE OF COLD ROLLING AND ANNEALING ON HARDNESS OF BIMETALLIC STRIP
Cu– Al** 229

OPTIMIZATION OF CATHODIC MATERIAL LEACHING PROCESS IN ACID-SULPHATE SOLUTION

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Abstract

Lithium cobalt oxide is the first commercialized electrode material with the greatest potential for recycling. In this paper, the influence of different process parameters (leaching agent concentrations, solid phase concentrations and temperatures) on the degree of cobalt leaching was investigated. It was found that the maximum degree of cobalt leaching (about 100%) is achieved at the following process parameters: sulfuric acid concentration was 3 mol/dm³, solid phase concentration was 33 g/dm³, volume flow of sulfur dioxide was 2 dm³/min, temperature was 85°C and leaching process duration was 60 minutes.

Keywords: LiCoO₂, leaching, sulfur dioxide.

1. INTRODUCTION

Sony commercialized the production of lithium-ion batteries in 1991, and their use in many electronic devices and electric vehicles has grown since then [1]. Improper disposal of waste batteries enables the release of cobalt into the environment, which presence in water systems can cause intestinal disorders, hearing loss, ischemia of the heart muscle, etc. [2]. Battery recycling technology protects the environment, preserves natural metal resources, and achieve significant economic benefits [1]. The methods that can be applied in the process of battery recycling are: hydrometallurgical, pyrometallurgical and biometallurgical [3,4]. Although the pyrometallurgical process is characterized by high efficiency, its application leads to high energy consumption, material loss and emissions of gaseous pollutants [5]. Compared to the traditional pyrometallurgical process, the hydrometallurgical process requires less energy consumption, emits less harmful gases and provides a high degree of metal valorization [6]. In order to reduce costs and increase process efficiency, biometallurgical processes have been developed in recent years. However, these processes take a very long time due to the difficulties caused by the growth of bacteria [7]. For these reasons, most of the research is focused on the hydrometallurgical process for recycling of valuable metals from lithium-ion batteries.

In this paper, leaching process of cathodic material was optimized in acid sulphate solution with the use of sulfur dioxide, as a reducing agent.

2. EXPERIMENTAL

Seventeen lithium-ion batteries with cathodes made of LiCoO₂ were collected and used in the experiments. After the application of pretreatment techniques, the cathode material was obtained and the chemical composition is presented in Table 1. Cobalt and lithium, as basic components of cathode material, were detected by various analytical methods. The presence of aluminum in the cathode material is probably a consequence of pretreatment on spent lithium-ion cells [8,9]. The

total cobalt content was determined using the inductively coupled plasma optical emission spectroscopy (ICP-OES), while the concentration of Co^{2+} in the initial sample of cathode leaching material was determined by UV-VIS spectrophotometric method. The values of the obtained cobalt concentrations, determined with UV-VIS method, indicated that cobalt in solution was present in the stable ionic form of Co^{2+} , which is confirmed by other authors [10,11].

Table 1 -Chemical composition of the initial cathode material for leaching

Element	Mass content (wt.%)	Analytic method
Co	49.81	ICP-OES
Co (Co^{2+})	49.00	UV-VIS
Li	5.98	ICP-OES
Al	0.01	ICP-OES

3. RESULTS

3.1 Optimization of the leaching process

In order to optimize the leaching process of the cathode material in an acid sulphate medium, the influence on the leaching process of various process parameters (leaching agent concentrations, solid phase concentrations and temperature) was examined.

3.1.1 Influence of sulfuric acid concentration on the degree of cobalt leaching

In order to determine the optimal concentration of H_2SO_4 in the process of leaching of the cathode material, in a sulfur dioxide atmosphere, a series of experiments were performed with the different concentrations of H_2SO_4 (0.5 mol/dm³ - 3 mol/dm³). The experiments were performed at a temperature of 35°C, with a solid phase concentration 33 g/dm³, and with a volume flow of 2 dm³/min SO_2 . Experiment time was 100 min.

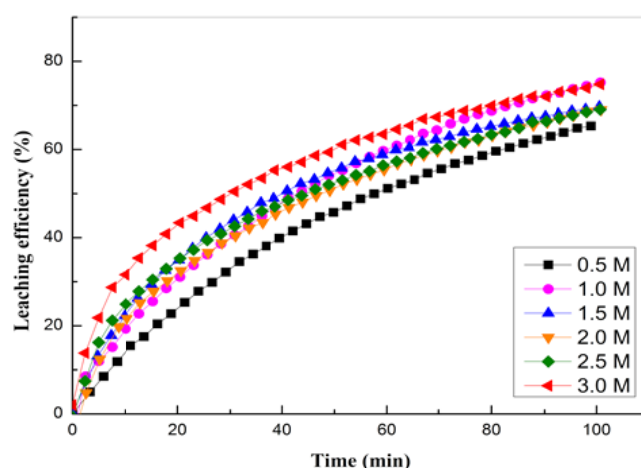


Figure 1 - Influence of H_2SO_4 concentration on the degree of cobalt leaching

The results of the research, shown in Figure 1, indicate that with an increase in the H_2SO_4 concentration 0.5 mol/dm³ - 3 mol/dm³, the degree of cobalt leaching increases from 65.3% to 73.9%. Considering the obtained results, the H_2SO_4 concentration of 3 mol/dm³ can be taken as optimal value and was used in further experimental studies.

3.1.2 Influence of solid phase concentration on the degree of cobalt leaching

The influence of solid phase concentration (in the range of 10 g/dm³ to 67 g/dm³) on the degree of cobalt leaching was examined under the following conditions: H_2SO_4 concentration was 3

mol/dm³, volume flow sulfur dioxide was 2 dm³/min, temperature was 35°C, and time of leaching was 100 min. The obtained results, shown in Figure 2, indicate that with the increase of the solid phase concentration, the degree of cobalt leaching decreases. The highest degree of cobalt leaching was recorded at a solid phase concentration of 10 g/dm³. From the industrial point of view, in the process of material leaching, it is desirable to use a higher solid-liquid phase ratio, regardless of the achieved lower degree of metal leaching [12,13]. For this reason, a solid phase concentration of 33 g/dm³ was taken as the optimal concentration and used in further experimental studies.

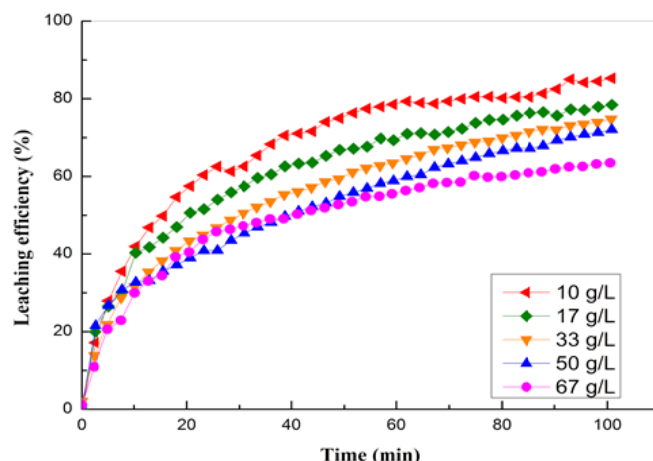


Figure 2 -Influence of solid phase concentration on the degree of cobalt leaching

3.1.3 Influence of temperature on the degree of cobalt leaching

In order to examine the kinetic laws of the process, the influence of the temperature of the cathode material on the degree of cobalt leaching was examined under the following conditions: acid concentration was 3 mol/dm³, volume flow SO₂ was 2 dm³/min and solid phase concentration was 33 g/dm³. The results of the research, shown in Figure 3, indicate that temperature has a positive effect on the degree of cobalt leaching. It was concluded that with an increase of the leaching temperature 35°C to 85°C, the degree of cobalt leaching increases from 74.8% to 99.4%.

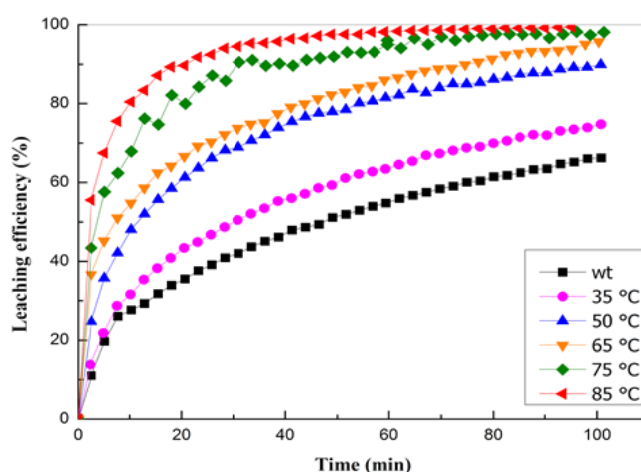


Figure 3 -Influence of temperature on the degree of cobalt leaching

4. CONCLUSION

Based on the results of leaching of the cathode material in a sulfur dioxide atmosphere, it can be concluded that the temperature and duration of the process have the greatest influence on the

leaching process. Also, the shape of the kinetic curves of leaching indicated that the concentration of sulfuric acid and the concentration of the solid phase, slightly influence the leaching of cobalt from the cathode material of spent lithium-ion batteries. The maximum degree of cobalt leaching (about 100%) was achieved at a sulfuric acid concentration of 3 mol/dm³, a solid phase concentration of 33 g/dm³, a volume flow of sulfur dioxide of 2 dm³/min, temperature of 85°C and leaching process duration of 60 minutes.

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