



---

INTERNATIONAL CONFERENCE  
MEĐUNARODNA KONFERENCIJA

---

MEETING POINT OF THE SCIENCE AND PRACTICE IN THE FIELDS OF  
CORROSION, MATERIALS AND ENVIRONMENTAL PROTECTION

---

*STECIŠTE NAUKE I PRAKSE U OBLASTIMA KOROZIJE,  
ZAŠTITE MATERIJALA I ŽIVOTNE SREDINE*

---

# PROCEEDINGS

---

# *KNJIGA RADOVA*

Under the auspices of the  
MINISTRY OF EDUCATION, SCIENCE AND TECHNOLOGICAL  
DEVELOPMENT OF THE REPUBLIC OF SERBIA

*Pod pokroviteljstvom*  
**MINISTARSTVO PROSVETE, NAUKE I TEHNOLOŠKOG RAZVOJA**  
**REPUBLIKE SRBIJE**

September 13-16, 2021 : : Tara Mountain, Serbia

---

CIP - Katalogizacija u publikaciji  
Narodna biblioteka Srbije, Beograd

620.193/.197(082)(0.034.2)

621.793/.795(082)(0.034.2)

667.6(082)(0.034.2)

502/504(082)(0.034.2)

66.017/.018(082)(0.034.2)

**INTERNATIONAL Conference YUCORR (22 ; 2021 ; Tara)**

Meeting point of the science and practice in the fields of corrosion, materials and environmental protection [Elektronski izvor] : proceedings = Stecište nauke i prakse u oblastima korozije, zaštite materijala i životne sredine : knjiga radova / XXII YuCorr International Conference = XXI YuCorr [Jugoslovenska korozija] Međunarodna konferencija, September 13-16, 2021, Tara Mountain, Serbia = [organized by] Serbian Society of Corrosion and Materials Protection ... [et al.] ; [organizatori Udruženje inženjera Srbije za koroziju i zaštitu materijala ... [et al.] ; [editors, urednici Miomir Pavlović, Miroslav Pavlović, Marijana Pantović Pavlović]. - Beograd : Serbian Society of Corrosion and Materials Protection UISKOSAM : Udruženje inženjera Srbije za koroziju i zaštitu materijala UISKOSAM, 2021 (Beograd : Serbian Society of Corrosion and Materials Protection UISKOSAM : Udruženje inženjera Srbije za koroziju i zaštitu materijala UISKOSAM). - 1 elektronski optički disk (CD-ROM) ; 12 cm

Sistemska zahtevi: Nisu navedeni. - Nasl. sa naslovne strane dokumenta. - Tekst na engl. i srp. jeziku.

- Tiraž 200. - Bibliografija uz većinu radova. - Abstracts.

ISBN 978-86-82343-28-8

a) Премази, антикорозиони -- Зборници б) Превлаке, антикорозионе -- Зборници в)

Антикорозиона заштита -- Зборници г) Животна средина -- Заштита -- Зборници д) Наука о материјалима -- Зборници

COBISS.SR-ID 48091145

## **XXII YUCORR – International Conference | Međunarodna konferencija**

### **PUBLISHED AND CD BURNED BY | IZDAVAČ I NAREZIVANJE CD**

SERBIAN SOCIETY OF CORROSION AND MATERIALS PROTECTION (UISKOSAM)

UDRUŽENJE INŽENJERA SRBIJE ZA KORZIJU I ZAŠTITU MATERIJALA (UISKOSAM),

Kneza Miloša 7a/II, 11000 Beograd, Srbija, tel/fax: +381 11 3230 028, [office@sitzam.org.rs](mailto:office@sitzam.org.rs); [www.sitzam.org.rs](http://www.sitzam.org.rs)

**FOR PUBLISHER | ZA IZDAVAČA** Prof. dr MIOMIR PAVLOVIĆ, predsednik UISKOSAM

**SCIENTIFIC COMMITTEE | NAUČNI ODBOR:** Prof. dr M. G. Pavlović, Serbia – President

Prof. dr Đ. Vaštag, Serbia; Dr M. M. Pavlović, Serbia; Prof. dr D. Vuksanović, Montenegro;

Prof. dr D. Čamovska, North Macedonia; Prof. dr M. Antonijević, Serbia; Prof. dr S. Stopić, Germany;

Prof. dr R. Zejnilović, Montenegro; Prof. dr L. Vrsalović, Croatia; Dr N. Nikolić, Serbia;

Dr I. Krastev, Bulgaria; Prof. dr B. Grgur, Serbia; Prof. dr M. Gvozdrenović, Serbia;

Prof. dr S. Hadži Jordanov, North Macedonia; Prof. dr R. Fuchs Godec, Slovenia;

Prof. dr J. Stevanović, Serbia; Dr V. Panić, Serbia; Dr M. Mihailović, Serbia;

Prof. dr V. Marić, Bosnia and Herzegovina; Prof. dr J. Jovičević, Serbia; Prof. dr D. Jevtić, Serbia;

Dr F. Kokalj, Slovenia; Prof. dr A. Kowal, Poland; Prof. dr Prof. dr M. Gligorić, Bosnia and Herzegovina;

Prof. dr M. Tomić, Bosnia and Herzegovina; Prof. Dr B. Arsenović, Bosnia and Herzegovina

**ORGANIZING COMMITTEE | ORGANIZACIONI ODBOR:** Dr Miroslav Pavlović – president

Dr Nebojša Nikolić – vice president; Dr Marija Mihailović – vice president

Prof. dr Miomir Pavlović; Aleksandar Putnik B.Sc.; Dr Vladimir Panić; Jelena Slepčević, B.Sc.;

Dr Vesna Cvetković; Prof. dr Milica Gvozdrenović; Zagorka Bešić, B.Sc.; Gordana Miljević, B.Sc.;

Miomirka Anđić, B.Sc.; Dr Marija Matić; Marijana Pantović Pavlović, M.Sc.; Dr Dragana Pavlović;

Dr Sanja Stevanović; Lela Mladenović – secretary

**EDITORS | UREDNICI:** Prof. dr Miomir Pavlović, Dr Miroslav Pavlović, Marijana Pantović Pavlović, M.Sc.

**SCIENTIFIC AREA | OBLAST:** CORROSION AND MATERIALS PROTECTION | KOROZIJA I ZAŠTITA MATERIJALA

**PAGE LAYOUT | KOMPJUTERSKA OBRADA I SLOG:** Dr Miroslav Pavlović

**CIRCULATION | TIRAŽ:** 200 copies | primeraka

**PUBLICATION YEAR | GODINA IZDANJA:** 2021

**ISBN 978-86-82343-28-8**



Ovaj PDF fajl sadrži elektronsku Knjigu radova prezentovanih u okviru Međunarodne konferencije **XXII YuCorr**. U knjizi su **plavom bojom** obeleženi aktivni linkovi ka pojedinim njenim delovima, iz Sadržaja do naznačenih stranica.

This PDF file contains Proceedings presented on the **XXII YuCorr** International Conference. It can be easily navigated through the book contents by a single click on the appropriate links in Contents (**showed in blue**).

**Autori snose punu odgovornost za sadržaj, originalnost, jezik i gramatičku korektnost sopstvenih radova.**

**Authors bear full responsibility for the content, originality, language and grammatical correctness of their own works.**

**XXII YUCORR IS ORGANIZED BY  
ORGANIZATORI XXII YUCORR-a**



---

**SERBIAN SOCIETY OF CORROSION AND MATERIALS PROTECTION**

*Udruženje Inženjera Srbije za Koroziju i Zaštitu Materijala*



---

**INSTITUTE OF CHEMISTRY, TECHNOLOGY AND METALLURGY,  
UNIVERSITY OF BELGRADE**

*Institut za Hemiju, Tehnologiju i Metalurgiju,  
Univerzitet u Beogradu*



---

**UNION OF ENGINEERS AND TECHNICIANS OF SERBIA, BELGRADE**

*Savez Inženjera i Tehničara Srbije*



---

**ENGINEERING ACADEMY OF SERBIA**

*Inženjerska Akademija Srbije*

---

**XXII YUCORR IS ORGANIZED UNDER THE AUSPICES OF THE  
MINISTRY OF EDUCATION, SCIENCE AND TECHNOLOGICAL  
DEVELOPMENT OF THE REPUBLIC OF SERBIA**



***XXII YUCORR JE FINANSIJSKI POMOGLO  
MINISTARSTVO PROSVETE, NAUKE I TEHNOLOŠKOG RAZVOJA  
REPUBLIKE SRBIJE***

**SPONSORS | SPONZORI**

**INTERNATIONAL SOCIETY OF ELECTROCHEMISTRY, Switzerland**

**SAVEZ INŽENJERA I TEHNIČARA SRBIJE, Beograd**

**HELIOS SRBIJA a.d., Gornji Milanovac**

**METAL CINKARA d.o.o., Inđija**

**SURTEC ČAČAK d.o.o., Čačak**

**ALFATERM d.o.o., Čačak**

**INSTITUT ZA PREVENTIVU d.o.o., Novi Sad**

**EKP ELKER a.d., Prijedor, Republika Srpska, B&H**

**EKO ZAŠTITA d.o.o., Bijeljina, Republika Srpska, B&H**

**IPIN d.o.o., Bijeljina Republika Srpska, B&H**

**HEMIPRODUKT d.o.o., Novi Sad**

**INSTITUT ZA OPŠTU I FIZIČKU HEMIJU, Beograd**

**SZR "GALVA", Kragujevac**

**NOVOHEM d.o.o., Šabac**

## Electrochemical investigation of lateritic ore leachates for metal ions extraction

Jonas Mitterecker<sup>1,\*</sup>, Milica Košević<sup>2</sup>, Marija Mihailović<sup>2</sup>, Jasmina Stevanović<sup>2</sup>, Srećko Stopić<sup>1</sup>, Bernd Friedrich<sup>1</sup>, Vladimir Panić<sup>2</sup>

<sup>1</sup> *IME Process Metallurgy and Metal Recycling, RWTH Aachen University, Intzestrasse 3, 52056 Aachen, Germany*

<sup>2</sup> *University of Belgrade - Institute of Chemistry, Technology and Metallurgy – National Institute of the Republic of Serbia, Njegoševa 12, Belgrade, Serbia*

\* [jonas.mitterecker@rwth-aachen.de](mailto:jonas.mitterecker@rwth-aachen.de)

### Abstract

*Electrochemical measurements were employed to study the possibility of the extraction of metal ions from lateritic ore leachates predominantly rich in Fe in order to separate more noble cobalt and nickel metals. Lateritic ore was leached in 1 M H<sub>2</sub>SO<sub>4</sub> solution at 70 °C by standard leaching process. The leachate was afterwards subjected to the pH adjustment that led to the removal of Fe ions by precipitation. Results obtained from cyclic voltammetry and chronamperometry measurements in native leachate and those with adjusted pH showed that optimal pH value for Fe removal was 3.7.*

**Keywords:** *selective separation of cobalt ions; cobalt extraction; electrodeposition; Iron removal*

### Izvod

*Elektrohemijskim merenjima je ispitivana mogućnost izdvajanja kobalta i nikla iz lužine lateritne rude bogate sa Fe. Rastvorne komponente lateritne rude su standardnim procesom luženja, tretiranjem u 1 M rastvoru H<sub>2</sub>SO<sub>4</sub> na povišenoj temperaturi, prevedene u rastvor. U sledećoj fazi prečišćavanja dobijenom rastvoru je podešavana pH vrednost kako bi se joni Fe preveli u talog. Cikličnom voltametrijom i hronoamperometrijom je pokazano da je optimalna vrednost za uklanjanje jona Fe 3,7.*

**Ključne reči:** *selektivno razdvajanje jona kobalta; ekstrakcija kobalta; elektrodepozicija; Uklanjanje gvožđa*

### Introduction

Cobalt and nickel are being considered critical raw materials. Cobalt is essential for manufacturing of Li-ion batteries that find wide usage in all electrical applications, especially in fast growing market of electric vehicles [1]. Nickel demand is expected to grow rapidly in the next decades, mainly due to rising demand for fast-working and high-tech steels, which find broader application in industry [2].

Main source of these metals are lateritic ores [3]. These ores are in oxidic state, containing high amounts of iron, which is being undesired for the extraction processes of cobalt and nickel. Due to low content of cobalt and nickel in the ores, a diligent process route is detrimental. The development of new technologies, including advanced precipitation methods, make the processing of such ores economically and technically feasible.

In general, iron precipitation takes place at lower pH, at which cobalt and nickel are stable in the solution. Iron removal is usually performed with addition of bases in order to adjust the pH of the pregnant leach solution. Precipitation has proved to be a challenge due to overlapping behavior of iron, nickel and cobalt. Deeper investigation of this ternary system was the motivation for this paper. A new approach using electrochemical methods for extraction experiments is partially presented.

## Experimental

Leaching of lateritic ore was performed in a standard glass cell, with stirring unit and temperature control. The flask was placed inside of a temperature controlled heating unit (SAF Wärmotechnik GmbH, Weinheimer Str. 2A, 69509 Mörlenbach, Germany). Temperature was kept constant at 70 °C for 2 h. The lateritic ore was leached with 1M H<sub>2</sub>SO<sub>4</sub> at a stirring speed of 300 rpm. Liquid to solid ratio of the leaching was 5:1. The ore solid residue was filtered off using a centrifuge, and clear solution was then used for further electrochemical investigations.

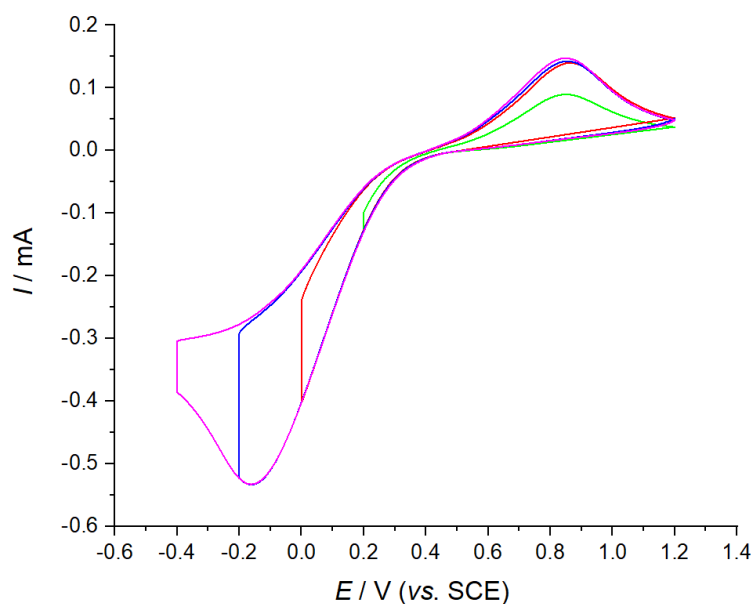
Electrochemical measurements were performed in a standard three electrode system using potentiostat/galvanostat, model SP-200 (Bio-Logic SAS, France). Glassy carbon electrode served as working electrode, Pt wire as counter electrode and saturated calomel electrode (SCE) as a reference electrode. All measurements were conducted in the as prepared filtered solution (pH 0.5) and in the NaOH-treated filtered solution (pH 3.7). Addition of NaOH was performed under constant stirring at 500 rpm in a separated beaker. Cyclic voltammetry advanced (CVA) was performed with a sweep rate of 10 mV s<sup>-1</sup>. Chronoamperometry (CA) was employed for the electrodeposition of ions from the solution on the GC electrode. An experimental scheme can be found in **Table 5**.

**Table 5.** Parameters of electrochemical measurements

Technique	Parameters
CVA	According to Open circuit potential; -1200 mV to 1200 mV; Cathodic holding time 2 minutes
CA_1	-680 mV, 1h; 1000 rpm
CA_2	-900 mV, 1h; 1000 rpm

## Results and discussion

Cyclic voltammograms of different cathodic cycling limits of as prepared leachate (pH 0.5) are shown in Figure 3. The anodic branch is recorded upon hold at the cathodic limit for 2 min.



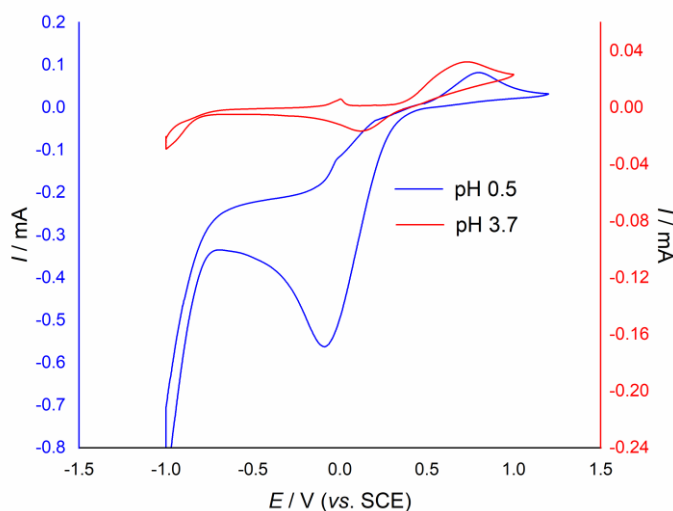
**Figure 3.** Cyclic voltammograms of as prepared filtered solution. N<sub>2</sub> saturated,  $\nu = 50 \text{ mV s}^{-1}$ . Reversible Fe<sup>3+</sup>/Fe<sup>2+</sup> redox transition dominates the response indicating the excess of 3+ state by higher currents of the reduction peak. Going more cathodically results in shifting potential of



$\text{Fe}^{3+}/\text{Fe}^{2+}$  reduction peak to the more negative values. Simultaneously, anodic peak potential that appeared at around 0.85 V is shifted to slightly negative potentials too.

Influence of the pH on the Fe ions content in the solution can be observed by comparing cyclic voltammograms of as prepared leachate (pH 0.5) and filtered solution treated with NaOH to adjust pH to 3.7 (**Figure 4**). Namely, in contrast to the CV of as prepared solution that was comprised of the well pronounced  $\text{Fe}^{3+}/\text{Fe}^{2+}$  redox couple, the CV of the NaOH treated solution was almost featureless.

The onset of a side reaction of hydrogen evolution (HER) is found at pH 0.5 at around  $-0.8$  V in the CV. Consequently, the shift of the cathodic cycling limit in the region of HER causes the appearance of the two weak anodic shoulders at around 0.0 and 0.2 V, which were not seen in CVs with more positive cathodic limits (Fig. 1).

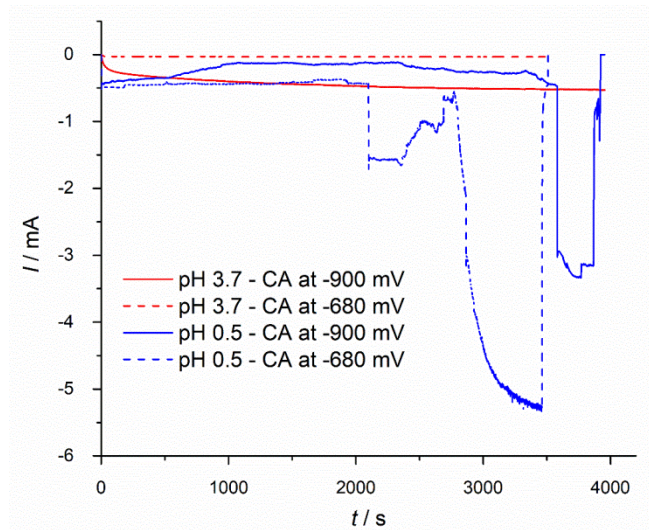


**Figure 4.** Cyclic voltammograms of as prepared filtered solution (pH 0.5) and filtered solution treated with NaOH (pH 3.7),  $\nu = 10 \text{ mV s}^{-1}$ .

Potentiostatic currents obtained during electrodeposition at 1000 rpm from as prepared leachate were considerably higher in comparison to those obtained in solution treated with NaOH (**Figure 5**). This is due to higher ions concentration in the as prepared leachate containing Fe ions. HER is not expected to considerably take place at the potentials positive to  $-900$  mV in the treated solution, as indicated by Fig. 2. It follows that HER currents contribute also to the overall currents registered during CA in as prepared leachate.

NaOH treated solution-related curves were of fairly stable currents during electrodeposition time, suggesting only metal ion deposition. On the other hand, curves obtained during electrodeposition from as prepared leachate consisted of two zones (deposition at  $-680$  mV) and even three zones (deposition  $-900$  mV). Both of the curves at pH 0.5 show sudden increase in currents upon certain deposition times (ca. 2900 and 3500 s at  $-680$  and  $-900$  V, respectively). This indicates the onset of HER on the metal deposits on GC. At the deposition times before sudden increase the chronoamperograms are different, with even lower currents at more cathodic potential of  $-900$  mV. Since codeposition of Fe is not expected at the potentials positive to ca.  $-700$  mV, it follows that Fe deposition affects not only the electrochemical features of the metal-extracting process, but also the deposit composition and its activity for HER.





**Figure 5.** Chronoamperograms registered during electrodeposition from as prepared leachate (blue curves) and NaOH treated solution (red curves) at two different potentials: -900 mV (line) and -680 mV (dash line), using SCE as a reference electrode.  $N_2$  saturated, 1000 rpm.

## Conclusion

Results of the CV and CA measurements showed that pH of 3.7 was optimal for removal of Fe ions from the pregnant leach solution of the lateritic ore. In comparison to CV at pH 0.5 that comprised completely of well developed  $Fe^{3+}/Fe^{2+}$  redox transition, the shape of CV at pH 3.7 indicate considerable decrease in  $Fe^{3+}/Fe^{2+}$  transition currents, suggesting that most of the Fe ions were removed from the solution. Consequently, the deposition currents of metals are registered prior the onset of hydrogen evolution being pushed cathodically due to increase in pH. The dissolution currents of these components are registered in the anodic counterpart of CV as a small peak, which position corresponds to indicative shoulder in CV recorded at pH 0.5. Presented data indicate that performed electrochemical procedure is useful for the treatment of multicomponent leachates to extract and separate low contents of Co and Ni from Fe-rich ores.

## Acknowledgements

This paper is the result of the Project “Controlled Acidic Eh-Potential Selective Leaching of Non-ferrous Metals” – CAPTAIN- No.6463002 Funded by Science Fund of the Republic of Serbia. Partner on this project is IME Process Metallurgy and Metal Recycling, RWTH Aachen University.

## References

1. Deetman, S.; Pauliuk, S.; van Vuuren, D. P.; van der Voet, E.; Tukker, A.: Scenarios for demand growth of metals in electricity generation technologies, cars, and electronic appliances, *Environmental science & technology* 52 (2018), no. 8, pp 4950–4959
2. [Davis, J. R. (ed.): *Nickel, cobalt, and their alloys*, 1. ed., Materials Park, Ohio: ASM International, 2000. (ASM specialty handbook)
3. Slack, J. F.; Kimball, B. E.; Shedd, K. B.; Schulz, K. J.; DeYoung, J. H.; Seal, R. R.; Bradley, D. C.: *Critical Mineral Resources of the United States—Economic and Environmental Geology and Prospects for Future Supply*, 2017