This is an electronic reprint of the original article.
This reprint may differ from the original in pagination and typographic detail.

Elomaa, H.; Halli, P.; Sirviö, T.; Yliniemi, K.; Lundström, M.

A future application of pulse plating–silver recovery from hydrometallurgical bottom ash leachant

Published in:
Transactions of the Institute of Metal Finishing

DOI:
[10.1080/00202967.2018.1507320](https://doi.org/10.1080/00202967.2018.1507320)

Published: 03/09/2018

Document Version
Peer reviewed version

Please cite the original version:
Elomaa, H., Halli, P., Sirviö, T., Yliniemi, K., & Lundström, M. (2018). A future application of pulse plating–silver recovery from hydrometallurgical bottom ash leachant. *Transactions of the Institute of Metal Finishing*, 96(5), 253-257. <https://doi.org/10.1080/00202967.2018.1507320>

A future application of pulse plating – silver recovery from hydrometallurgical bottom ash leachant

Heini Elomaa*, Petteri Halli*, Tuomas Sirviö*, Kirsi Yliniemi*,[†], Mari Lundström*¹

*Department of Chemical and Metallurgical Engineering, Aalto University School of Chemical Engineering, P.O. Box 16300, FI-00076 AALTO, Finland

[†]Department of Chemistry and Materials Science, Aalto University School of Chemical Engineering, P.O. Box 16100, FI-00076 AALTO, Finland

Abstract

In the current study, electrodeposition-redox replacement was applied for hydrometallurgical solution with the main elements of Ca (13.8 g/L), Al (4.7 g/L), Cu (2.5 g/L), Zn (1.2 g/L), Fe (1.2 g/L), S (1 g/L), Mg (0.8 g/L), P (0.5 g/L) and Ag (3.5 ppm). The solution originated from the leaching experiment of incinerator plant bottom ash, which was dissolved into 2 M HCl media at $T = 30\text{ }^{\circ}\text{C}$. The resulting deposit on electrode surface was analysed with SEM-EDS and the observed Ag/(Cu+Zn) ratio (0.3) indicated remarkable enrichment of silver on the surface, when compared to the ratio of these elements (Ag/(Cu+Zn)) in the solution ($6.8 \cdot 10^{-5}$). The enrichment of Ag vs. (Cu+Zn) could be demonstrated to increase ca. 4500 fold compared to ratio of the elements in solution.

Keywords: bottom ash, leaching, silver recovery, electrodeposition-redox replacement, circular economy

1 Introduction

In order to improve circular economy of metals, it is critical also to consider materials with minor concentrations of valuable metals as secondary raw materials. As a result, the concept of potential raw materials for metal recovery can be expanded also to industrial solid and liquid wastes [1-2]. Municipal solid waste incineration (MSWI) bottom ashes are an excellent example of such a secondary raw material of future from which minor valuable metals could be recovered more efficiently. The traditional treatment of bottom ashes includes separation processes based on crushing, sieving and magnetic methods to separate metal rich fractions. Solidification/stabilization and thermal methods can also be conducted to produce safely disposable or usable product fractions. Bottom ashes can be applied in loose construction aggregates, substitute materials in construction, raw-material in cement production and feedstock for ceramic material production [3]. Leaching of bottom ashes and their utilization in previously mentioned applications have earlier been investigated [3-7]. However, the produced liquid streams from bottom ash leaching may contain major amounts of impurities and only minor concentration of valuable metals, which causes challenges for feasible metal recovery of the valuable elements.

¹ Corresponding author: mari.lundstrom@aalto.fi

The principle of electrodeposition or electroplating of surfaces as well as metal recovery by electrowinning of metals from hydrometallurgical process solutions are theoretically very similar – metals present in the solution are electrically reduced on the selected surface material. The deposition can be done either by constant current (or potential) or providing short current (or potential) pulses. In hydrometallurgical terms, the first option results in electrowinning and it is applied in industrial scale e.g. for copper, nickel or zinc deposition on the permanent or seed plate cathode. Hydrometallurgical process streams differ typically from synthetic plating baths, as they contain a huge variety of base metal ions, impurities and also precious metal ions [8-21]. Typically, valuable metals such as Ag are present at ppb-ppm level, making their selective recovery challenging.

This study introduces a variation of pulse plating into hydrometallurgical solutions, more specifically electrodeposition followed by redox replacement (EDRR). In the presented method the redox replacement time is typically several minutes long period without applied current or potential. This differs from traditional pulse plating where t_{off} time is typically short and intended for surface activation while the long redox replacement allows diffusion and enrichment of more noble metal on the cathode surface by oxidising the already deposited less noble metals, due to difference in reduction potentials of these metals. The results demonstrate the possibility of recovering Ag from pregnant leach solution (PLS) originating from bottom ash leaching by EDRR method. Our recent research shows that changing the potential between the pre-defined deposition potential and open circuit potential (i.e. EDRR) provides a promising route to effectively recover very low concentrations (even <1 ppm) of Ag from concentrated Zn solutions [22]. The EDRR method has been previously investigated also to deposit different metals such as, Au, Pt, Pb, and Cu on surfaces from pure synthetic solutions with optimised concentrations [23-34] and only very limited studies have been performed on industrially relevant solutions [2, 22, 35] – and actually, there are no reports demonstrating improved Ag recovery by the EDRR method from municipal bottom ash leaching solutions.

2 Materials and Methods

2.1 Material

The bottom ash from municipal solid waste incineration was provided by a Finnish incineration company. The material used in the leaching experiments was a non-ferrous concentrate of aged bottom ash, which had undergone mechanical treatments, producing a fraction with particle size <0.1 mm. This fraction had aluminum (51200 mg/kg), copper (21400 mg/kg), lead (2250 mg/kg) and zinc (10900 mg/kg) and in addition relatively high silver (39.8 mg/kg) concentration. Additionally, it contained lower amounts of iron (23000 mg/kg) compared to the other bottom ash fractions. The raw material analysis was conducted by inductively coupled plasma optical emission spectrometry, ICP-OES (Thermo Fisher Scientific iCAP 6500 duo) after total dissolution. Fire-assay analysis was used for noble metal analysis using atomic absorption spectroscopy, AAS (PerkinElmer). The results are presented in Table 1.

Table 1. Chemical analysis of bottom ash fraction (<0.1 mm) used in leaching experiments.

Analyze method	Fire assay FAAS	Analysis by ICP-OES			
Elements	(mg/kg)	Elements	(mg/kg)	Elements	(mg/kg)
Au	0.67	Al	51200	Mo	20.4
Ag	39.8	As	12.5	Na	2430
Pd	0.15	B	239	Ni	171
Pt	0.076	Ba	154	P	4830
		Be	0.59	Pb	2250

Ca	147000	Rb	7
Cd	17.7	S	23400
Co	26.3	Sb	94
Cr	172	Sc	1.35
Cu	21400	Sr	326
Fe	23000	Ti	1740
K	1670	V	32.4
La	9.2	Y	6012
Mg	8360	Zn	10900
Mn	1830	Zr	20

2.2 Leaching

The bottom ash fraction investigated was divided using a rotating sample divider (Microscal L/MSR) to produce homogenous samples for leaching experiments. The applied solid to liquid ratio was 1:10 in all leaching experiments. Experimental set-up included a multiple magnetic stirrers (IKA RT10). Temperature was kept constant at 30 °C in all experiments. 300 mL Erlenmeyer flasks were used as reactors with agitation of 300 RPM and air purging was supplied to the flasks. Figure 1 presents a schematic picture of the set-up. The pH (Mettler Toledo multiparameter and Mettler Toledo InLab Expert Pro-ISM probe) and redox potential (Mettler Toledo InLab redox meter Ag/AgCl) were measured at the beginning of the test, after the leaching and after the filtration. 2 M hydrochloric acid (37 %, EMSURE® ACS, ISO, Millipore Sigma) was employed in the leaching tests. The total leaching time was 24 h and solution samples for analysis were collected after leaching. The samples were filtrated via syringe filter (Whatman filter unit 0.2 µm) and strengthen with nitric acid.

Figure 1. Experimental test-up: 1. Multi magnetic stirrer with heat plates, 2. magnetic stirrers and 3. air supply.

2.3 EDRR set-up

A three electrode cell was used for the EDRR measurements: 99.5% purity platinum (Kultakeskus Oy, Finland) was used as working electrode (WE) and counter electrode (CE) and saturated calomel (SCE, B521, SI Analytics) as reference electrode (RE). IviumStat.XRe 24-bit (NL) was employed for the electrochemical measurements and SEM-EDS (Scanning Electron Microscope, Leo 1450 VP, Zeiss, Germany – Energy Dispersion Spectroscopy, INCA-software, Oxford Instruments, UK) was used for chemical analysis of the electrodes after EDRR experiment. In prior to EDRR experiments, cyclic

voltammetry (CV) measurements in the synthetic solution was conducted in order to determine the deposition and stripping peaks characteristics for the investigated solution, containing Cu and Ag (0.0 V $\rightarrow$ 0.45 V $\rightarrow$ -0.3 V $\rightarrow$ 0.0 V vs. SCE). The scan rate was 50 mV/s.

In the EDRR experiments, deposition potential (E_1) investigated was -0.3 V vs. SCE and the deposition time t_1 was 5, 10 or 15 s. Cut-off potential (E_2) was 0.3 V vs. SCE, and cut-off time was $t_2 = 1000$ s, i.e. the next ED step started when the cut-off potential E_2 or cut-off time t_2 was reached, whichever occurred first. The amount of EDRR cycles, n , was kept constant at 10.

3 Results

3.1 Leaching of bottom ash

The pH of the 2 M HCl solution was measured before leaching (pH = 0.5-0.6), after leaching (pH = 0.45-0.5) and after filtration (pH = 0.3-0.35). Similarly, the redox potential was measured at the same points, and it increased by 100 mV from 350 mV vs. Ag/AgCl (before leaching) to 450 mV (after leaching) while filtration did not affect the potential any further (450 mV after filtration). The achieved extractions of metals to 2 M HCl leaching are shown in Figure 2. In the current study, the metals of interest were Ag, Al, Cu, Pb and Zn (Figure 2 A). The corresponding amounts in PLS for these metals were Ag = 3.5 ppm, Pb = 0.2 g/L, Zn = 1.2 g/L, Cu = 2.5 g/L, and Al = 4.7 g/L. Figure 2B presents the extraction of other elements analysed in solution. A remarkable decrease in bottom ash mass was observed during the leaching tests - 20.34 g of bottom ash was exposed into leaching whereas the leach residue (solid) mass in the end was only 6.37 g, corresponding to 69% leaching.

Figure 2. The extractions of metals after bottom ash leaching by 2 M hydrochloric acid. Focus metals (Ag, Al, Cu, Pb and Zn) shown in A and the other metals in B.

3.2 Recovery of Ag by Electrodeposition-redox replacement (EDRR)

Prior to applying the EDRR method for the real PLS solution for metal recovery, a synthetic solution was prepared having Ag, Zn and Cu in corresponding amounts (10 ppm, 1.2 g/L and 2.5 g/L, Table 2). The aim of EDRR is to enrich Ag on the electrode surface from PLS containing a high concentration of Cu^{2+} ions and a low concentration of silver ions. In the electrodeposition (ED) step mainly Cu is deposited on the electrode surface at constant cathodic potential E_1 for a defined time t_1 thus the deposit potential is selected from the CV according to Cu deposition potential. In the redox replacement (RR) step, the Cu layer was spontaneously replaced with the more noble silver metal ions, and the cut-off potential was selected to be before Ag stripping, thus creating a silver rich layer on the electrode surface. The ED and RR steps were repeated for multiple cycles.

Table 2. The composition of PLS and synthetic solution used in EDRR measurements.

Element	in real PLS (g/L)	in synthetic solution (g/L)
Ag	3.5 ppm	10 ppm
Al	4.7	-
Cu	2.5	2.5
Pb	0.2	-
Zn	1.2	1.2

Figure 3 presents the obtained CV of synthetic solution, from which can be detected the oxidation/reduction peaks of Cu^+ and Cu^{2+} , these can be possibly also cuprous complexes $[\text{CuCl}_2]^-$, $[\text{CuCl}_3]^{2-}$, $[\text{Cu}_2\text{Cl}_4]^{2-}$, $[\text{Cu}_3\text{Cl}_6]^{3-}$ and cupric complexes such as $[\text{CuCl}]^+$, $[\text{CuCl}_2]^0$, $[\text{CuCl}_3]^-$, $[\text{CuCl}_4]^{2-}$ [36]. The deposited Cu metal can be then replaced by Ag, due to its higher reduction potential. According to CVs, the parameters for EDRR experiments were determined. More information about the methodology for the optimization of the parameters for EDRR measurements is presented in the earlier publication [23]. The employed EDRR parameters to demonstrate Ag recovery were $E_1 = -0.3$ V and $E_2 = +0.3$ V vs. SCE, $t_1 = 10$ s, $t_2 = 1000$ s and $n = 10$. The effect of deposition time (5-15s) was investigated in the current study.

Figure 3. The obtained cyclic voltammetry (0.0 V $\rightarrow$ 0.45 V $\rightarrow$ -0.3 V $\rightarrow$ 0.0 V vs. SCE) of a solution containing 10 ppm Ag, 1.2 g/L Zn, 2.5 g/L Cu and 2 M HCl as base. The scanning rate was 50 mV/s.

Figure 4 presents an example of the EDRR data in 2 M HCl originating from bottom ash leaching. As it can be seen, the time to reach cut-off potential is increased with increasing number of cycles: this is due to the accumulation of Cu on the electrode surface, and hence a longer time to replace it with Ag is needed.

Figure 4. An example of the obtained EDRR data during measurements. The employed EDRR parameters were $E_1 = -0.3$ V and $E_2 = +0.3$ V vs. SCE, $t_1 = 10$ s, $t_2 = 1000$ s and $n = 10$.

3.3 Enrichment of Ag on surface

The SEM analysis was conducted as point and area analysis and presented as average of these in Table 3. The composition of the deposit on the surface consisted mainly of Cl, Cu, Zn and Ag. In addition, the SEM-EDS showed also minor amounts of Al, Ca, O and Si. Traces of Cd and Mg were also observed mostly in area analysis. As can be seen, the increased deposition time (t_1) also results in higher Ag wt-% on the surface.

Table 3. The analyzed amounts of Cl, Cu, Zn and Ag (SEM-EDS analysis) on the cathode surfaces after EDRR measurements. The employed EDRR parameters were $E_1 = -0.3$ V and $E_2 = +0.3$ V vs. SCE, $t_1 = 5, 10$ or 15 s, $t_2 = 1000$ s and $n = 10$.

Deposition time	Cl wt- %	Cu wt- %	Zn wt-%	Ag wt- %
5 s	7.03	0.81	0.45	0.17
10 s	3.10	0.28	0.32	0.19
15 s	5.32	0.52	0.30	0.21

The enrichment factors were calculated based on the values in Table 3 in order to evaluate the silver enrichment on Pt surface. The observed Ag/(Cu+Zn) ratio (0.3) indicated remarkable enrichment of silver on the surface, when compared to the ratio of these elements (Ag/(Cu+Zn)) in the solution ($6.8 \cdot 10^{-5}$). Ag vs. base metal (Cu+Zn) ratio can be increased to ca. 4500 fold on the cathode surface compared to the original leaching solution. The highest enrichment of silver compared to Cu and Zn was achieved with deposition time of 10 s (Figure 5). The deposition time (10 s) was shown to provide the most favourable level for Cu deposition, replaced by Ag in the studied parameter range. Due to fact that the redox replacement reaction takes place only at the deposit/solution interface, it is favourable to create a thin layer of sacrificial metal with a certain level of defects to gain a higher Ag enrichment. The thickness of the deposited Cu layer increases with a higher deposition time (15 s), affecting the purity of deposited surface. [22]

Figure 5. The calculated enrichment factors with studied electrodeposition times ($t_1 = 5, 10, 15$ s).

Kowalska et al. (2015) [37] present the results of silver recovery from complex matrix electrolyte after chloride leaching of copper flotation concentrate using potential-controlled electrolysis. Their solution included 50.7 mg/L Ag and 3650 mg/L Cu. Kowalska et al. [37] suggest pulse potential-controlled electrolysis for recovery of silver from complex matrix rather than continuous electrolysis. This in order to avoid co-deposition of Cu as with shorter deposit times pure Ag surfaces can be formed. They reported that the best results of pure silver recovery were obtained at $E = -0.15$ V vs. Ag/AgCl for deposition time of 1800s -7200s. Chatelut et al. [38] investigated the electrowinning of silver from photographic fixing solutions using zirconium cathode. They reported that silver could be recovered from the solution with an applied potential lower than 0.9 V between the cathode and a carbon graphite anode. 98% of total silver was recovered leaving the final Ag concentration in the bath to 20 mg/l. Sathaiyan et al. [39] investigated the recovery of silver from waste oxide button cells. The silver was separated from this material as silver chloride. Silver chloride was used to prepare silver thiosulfate solution for electrolysis, having 12.5 g/L of Ag. The purity of obtained product was 99.8% silver. Potential control between -0.4 and -0.6 V (SCE) is required for efficient electrodeposition.

As can be seen from these studies the used solutions for Ag electrowinning are from 4 to 14 times richer in silver compared to the PLS solution used in this study. For example, in the study of Chatelut et al. [38] the final concentration after electrowinning is higher than initial concentration used in this study. The study demonstrates that by the presented EDRR method it is possible to enrich even minor concentrations of silver, which are not recoverable by conventional methods, selectively from complex hydrometallurgical bottom ash leaching solution. Kowalska et al. [37] discussed the problems with co-deposition of Cu, thus creating impure product, which needs further treatment. The benefit of the presented EDRR method in this study is that with the optimized deposit time copper can be dominantly replaced by valuable silver, thus creating pure surfaces. In the study of Sathaiyan et al. [39] high cathodic potentials are needed for the deposition of silver. Advantage of EDRR method is that

only short times of deposition are required and the replacement of silver occurs in open cell, based on the redox potential difference between metals.

4 Conclusions

In this study, the EDRR method was applied for silver recovery from hydrometallurgical solution originating from bottom ash leaching by hydrochloric acid. The deposits achieved on electrode surface were analysed with SEM-EDS. The highest enrichment ratio for silver was achieved with 10 s deposit time. Ag/(Cu+Zn) ratio (0.3) indicated remarkable enrichment of silver on the surface, when compared to the ratio of these elements (Ag/(Cu+Zn)) in the solution ($6.8 \cdot 10^{-5}$). Thus, with EDRR method Ag vs. base metal (Cu+Zn) ratio could be increased to ca. 4500 fold on the cathode surface compared to the original leaching solution.

EDRR method provides a suitable route to recover selectively silver from hydrometallurgical bottom ash leaching solution. The method allows minor metal concentrations found in secondary raw materials, such as bottom ashes, to be recovered directly from PLS improving the circular economy of metals.

5 Acknowledgements

Academy of Finland (NoWASTE - Project No: 297962), Finnish Innovation Agency (CMEco –Project No: 7405/31/2016), and Finnish Steel and Metal Producers (METSEK-project) are acknowledged for financial support of this work. The authors specifically thank Jan Österbacka and Anne Kulmala (Fortum Waste Solutions) and Jukka Marmo and Sari Lukkari (Geological Survey of Finland, GTK) for providing the raw material and conducting the mechanical treatment for the raw material used in the leaching tests. The research also made use of the Academy of Finland supported “RawMatTERS Finland Infrastructure” (RAMI) based at Aalto University.

References

- [1] U.S. Mohanty, L. Rintala, P. Halli, P. Taskinen, M. Lundström: *Metals*, 2018, **8**(1), 40.
- [2] K. Yliniemi, Z. Wang, I. Korolev, P. Hannula, P. Halli, M. Lundström: *ECS Transactions*, 2018, **85**(4), 59-67.
- [3] C.H. Lam, A.W. Ip, J.P. Barford, G. McKay: *Sustainability*, 2010, **2**(7), 1943-1968.
- [4] I. Agcasulu and A. Akcil: *Miner. Process. Extr. Metall. Rev.*, 2017, **38**(3), 199-206.
- [5] R. Bunge: ‘Recovery of metals from waste incinerator bottom ash’, Verband der Betreiber Schweizerischer Abfallverwertungsanlagen (VBASA), Hochschule für Technik Rapperswil: Rapperswil, Bern, Switzerland, 2016.
- [6] S. Feng, X. Wang, G. Wei, P. Peng, Y. Yang, Z. Cao: *Chemosphere*, 2007, **67**(6), 1133-1137.
- [7] H. Zhang, P.J. He, L.M. Shao, X.J. Li: *J. Mater. Cycles Waste Manage.*, 2008, **10**(1), 7-13.
- [8] H. Fukubayashi: ‘The effect of Impurities and Additives on the Electrowinning of Zinc’, Ph.D. Thesis, Missouri University of Science and Technology, USA, 1972.
- [9] D.R. Fosnacht and T.J. O’Keefe: *Metall. Trans. B*, 1983, **14**(4), 645-655.
- [10] A.R. Ault and E.J. Frazer: *J. Appl. Electrochem.*, 1988, **18**(4), 583-589.
- [11] M. Maja and P. Spinelli: *J. Electrochem. Soc.* 1971, **118**(9), 1538–1540.
- [12] S. Ju, Y. Zhang, Y. Zhang, P. Xue, Y. Wang: *J. Hazard. Mater.* 2011, **192**(2), 554–558.
- [13] P. Kangas, P. Koukkari, B.P. Wilson, M. Lundström, J. Rastas, P. Saikkonen, J. Leppinen, V. Hintikka: *Proc. of Conference in Minerals Engineering*, Luleå, Sweden, Feb. 2017.

- [14]P. Koukkari, P. Kangas, M. Nystrom, M. Lundstrom, B.P. Wilson, J. Rastas, P. Saikkonen: Proc. of ALTA2017, Perth, Australia, May 20–27, 2017, ALTA Metallurgical Services.
- [15]L. Muresan, G. Maurin, L. Oniciu, D. Gaga: Hydrometallurgy, 1996, **43**(1–3), 345–354.
- [16]L. Mureşan, G. Maurin, L. Oniciu, S. Avram: Hydrometallurgy, 1996, **40**(3), 335-342.
- [17]E. Küzeci, R. Kammel S.K. Gogia: J. Appl. Electrochem., 1994, **24**, 730-736.
- [18]J. Lu, Q.-H. Yang, Z. Zhang: Transactions of Nonferrous Metals Society of China, 2010, **20**, 97 -101.
- [19]N.H.J. Freire, D. Majuste, M.A. Angora, V.S.T. Ciminelli: Hydrometallurgy, 2017, **169**, 112-123.
- [20]L. Muresan, S. Varvara, G. Maurin, S. Dorneanu: Hydrometallurgy, 2000, **54**(2-3), 161-169.
- [21]B. McKevitt, D. Dreisinger: Hydrometallurgy, 2009, **98**(1-2), 122-127.
- [22]P. Halli, H. Elomaa, B. P. Wilson, K. Yliniemi, M. Lundström: ACS Sustainable Chem. Eng. 2017, **5**(11), 10996-11004.
- [23]M. Fayette, Y. Liu, D. Bertrand, J. Nutariya, N. Vasiljevic, N. Dimitrov: Langmuir, 2011, **27**(9), 5650 – 5658.
- [24]D. Gokcen, S.-E. Bae, S.R. Brankovic: J. Electrochem. Soc., 2010, **157**(11), D582-D587.
- [25]D. Qu, C-W J. Lee, K. Uosaki: Bull. Korean Chem. Soc., 2009, **30**(12), 2875-2876.
- [26]T.S. Mkwizu, M.K. Mathe, I. Cukrowski: Langmuir, 2009, **26**(1), 570-580.
- [27]M.F. Mrozek, Y. Xie, M.J. Weaver: Analytical Chemistry, 2001, **73**(24), 5953- 5960.
- [28]Y.G. Kim, J. Y. Kim, D. Vairavapandian, J. L. Stickney: J. Phys. Chem. B, 2006, **110**(36), 17998-18006.
- [29]A. Sarkar and A. Manthiram: J. Phys. Chem. C, 2010, **114**(10), 4725- 4732.
- [30]D.A. Wragg, K. Yliniemi, T.M. Watson, D.A. Worsley: ECS Transaction, 2013, **53**(24), 11-17.
- [31]K. Yliniemi, D. Wragg, B.P. Wilson, H.N. McMurray, D.A. Worsley, P. Schmuki, K. Kontturi: Electrochimica Acta, 2013, **88**, 278-286.
- [32]E. Herraro, L.J. Buller, H.D. Abruña: Chemical Reviews, 2001, **101**(7), 1897-1930.
- [33]L.T. Viyannalage, R. Vasilic, N. Dimitrov: J. Phys. Chem. C, 2007, **111**(10), 4036 – 4041.
- [34]S.R. Brankovic, J.X. Wang, R.R. Adžić: Surface Science, 2001, **474**(1-3), L173-L179.
- [35] I. Korolev, P. Altinkaya, P. Halli, P.M. Hannula, K. Yliniemi, M. Lundström: J. Clean Prod., 2018, **186**, 840-850.
- [36] M. Lundström, J. Liipo, P. Taskinen, J. Aromaa: Int. J. Miner. Process., 2016, **102**, 13-18.
- [37]S. Kowalska, A. Lukomska, P. Los, T. Chmielewski, B. Wozniak: Int. J. Electrochem. Sci, 2015, **10**, 1186-1198.
- [38]M. Chatelut, E. Gobert, O. Vittori: Hydrometallurgy, 1999, **54**(2-3), 79-90.
- [39]N. Sathaiyan, V. Nandakumar, P. Ramachandran: J. Power Sources, 2006, **161**(2), 1463-1468.