



Determination of metallic impurities in the copper deposit obtained by electroextraction from solution resulted in the recycling of Waste Printed Circuit Board



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Introduction

Waste Electrical and Electronic Equipment (WEEE) generation has become a global concern due to the continuously increasing quantities disposed (49.8 million tonnes in 2018) (Adeola 2018) principally (~40%) in landfills (Cucchiella 2015). This phenomenon is attributed to the increased demand in the electronic sector of innovative electronic devices coupled with their decreased lifespan. Up to 60 elements could be leached and contaminate the groundwaters (Bloodworth 2014). Despite the fact that Printed Circuit Boards (PCBs) represent only a small weight percent of the total mass of WEEEs, they contain both heavy metals (e.g. Pb, Hg, Sn) and valuable metals (e.g. Cu, Au, Ag, Pt). Copper, the most abundant metal in PCBs, is present in up to 10 times higher concentrations than in primary ores (Isildar 2018).

Therefore, waste PCBs has become a valuable Cu source and as a consequence should be recovered. Suitable analytical methods are needed to analyze the metallic impurities in the copper deposit obtained by electroextraction.

Aim

The aim of this study was the validation of the Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method for the determination of metallic impurities in copper deposits obtained by electroextraction from Waste Printed Circuit Boards, more specifically desktop PC motherboards.

Experimental strategy

Based on previous research conducted by our group (Dorneanu 2017) it was proposed the validation of ICP-OES method that is capable to determine at least 0.1% metallic impurity in the presence of 80 – 100% copper.

Nine synthetic solutions were prepared with a composition similar to that resulted from the deposit solubilization in aqua regia. A 10-fold subsequent dilution was performed and the resulted solutions were analyzed by ICP-OES. The accuracy, precision and method figures of merit of the method were evaluated.

Metal concentration (mg L⁻¹) in synthetic solutions

Metals	S1	S2	S3	S4	S5	S6	S7	S8	S9
Cu	80	80	80	90	90	90	100	100	100
Pb	0.500	1.00	1.50	0.500	1.00	1.50	0.500	1.00	1.50
Sn	0.300	0.500	0.800	0.300	0.500	0.800	0.300	0.500	0.800
Zn	0.300	0.500	1.00	0.300	0.500	1.00	0.300	0.500	1.00
Ni	0.200	0.400	0.600	0.200	0.400	0.600	0.200	0.400	0.600
Fe	0.100	0.200	0.300	0.100	0.200	0.300	0.100	0.200	0.300

References

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Acknowledgments. This work was supported by a grant of the Romanian National Authority for Scientific Research and Innovation, CCCDI-UEFISCDI, project number PN-III-P1-1.2-PCCDI-2017-0652 / 84PCCDI/2018, within PNCDI III.

Operating conditions for the ICP-OES instrument

SPECTRO CIROS^{CCD} spectrometer (Spectro, Kleve, Germany)

Characteristic	Operating conditions
Plasma power	1500 W; 27.12 MHz radiofrequency
Argon flow rate	Outer gas: 12 L min ⁻¹ Nebulizer gas: 1 L min ⁻¹ Auxiliary gas: 0.6 L min ⁻¹
Sample uptake type	Cross-flow nebulizer
Sample flow rate	2 mL min ⁻¹
Signal processing	Peak height
Integration time	48 s
Background correction	Linear two point model

Results

Element	Wavelength (nm)	Calibration range (mg L ⁻¹)	Coefficient of determination (R ²)	Limit of detection (mg L ⁻¹)	Limit of quantification (mg L ⁻¹)
Cu	324.754	0 - 10	0.9999	0.019	0.057
Pb	220.351	0 - 1	0.9994	0.034	0.102
Sn	189.991	0 - 1	0.9992	0.010	0.030
Zn	213.856	0 - 1	0.9992	0.025	0.075
Ni	341.476	0 - 1	0.9999	0.018	0.054
Fe	259.940	0 - 1	0.9993	0.010	0.030

Element	R _{min} ± C.I. (%)	R _{max} ± C.I. (%)	R _{average} ± C.I. (%)	RSD (min. - max., %)
Cu	98 ± 4	103 ± 5	100 ± 5	1.1 - 2.6
Pb	96 ± 8	108 ± 8	102 ± 8	1.5 - 3.9
Sn	100 ± 9	105 ± 6	102 ± 6	1.3 - 3.7
Zn	95 ± 9	108 ± 9	101 ± 6	1.6 - 3.7
Ni	94 ± 7	109 ± 9	101 ± 7	1.5 - 3.8
Fe	100 ± 8	109 ± 9	104 ± 7	1.8 - 3.8

R - recovery degree;

C.I. - confidence interval for 95 % confidence level and n=3 measurements;

RSD - relative standard deviation.

Conclusions

- LOQs were at least 3 times lower than the metal concentration in the synthetic solutions;
- ICP-OES method is able of determining Pb, Sn, Zn, Ni and Fe impurities below 0.1% in copper matrix at a precision in the range of 1.1 – 3.9% and recovery in the range of 94 – 109%.