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Identification of metals from residual solutions obtained in the recovery process of active paste from the cathode of spent Li-ion batteries

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Introduction

Li-ion batteries are in the spotlight for manufacturers of phones, tablets, laptops, camcorders, and electric vehicles. Starting in 1991, Sony launched the first Li-ion batteries, which have since been continuously diversified ($LiCoO_2$ / LCO, $LiMn_2O_4$/LMO, $LiNiMnCoO_2$ or NMC, $LiFePO_4$ / LFP, $LiNiCoAlO_2$ / NCA and $Li_4Ti_5O_{12}$ / LTO). For the recovery of the active paste from the aluminium cathode, the ultrasonic method was chosen in citric, acetic, or lactic acid medium.

Cobalt is a rare and precious metal, and is a relatively expensive material compared with the other constituents of lithium ion batteries

The present paper presents the results obtained by identifying the metals from the residual solutions resulting from the process of ultrasonic recovery in acid medium of the cathodic paste (with Co, Ni content) from used Li-ion batteries.

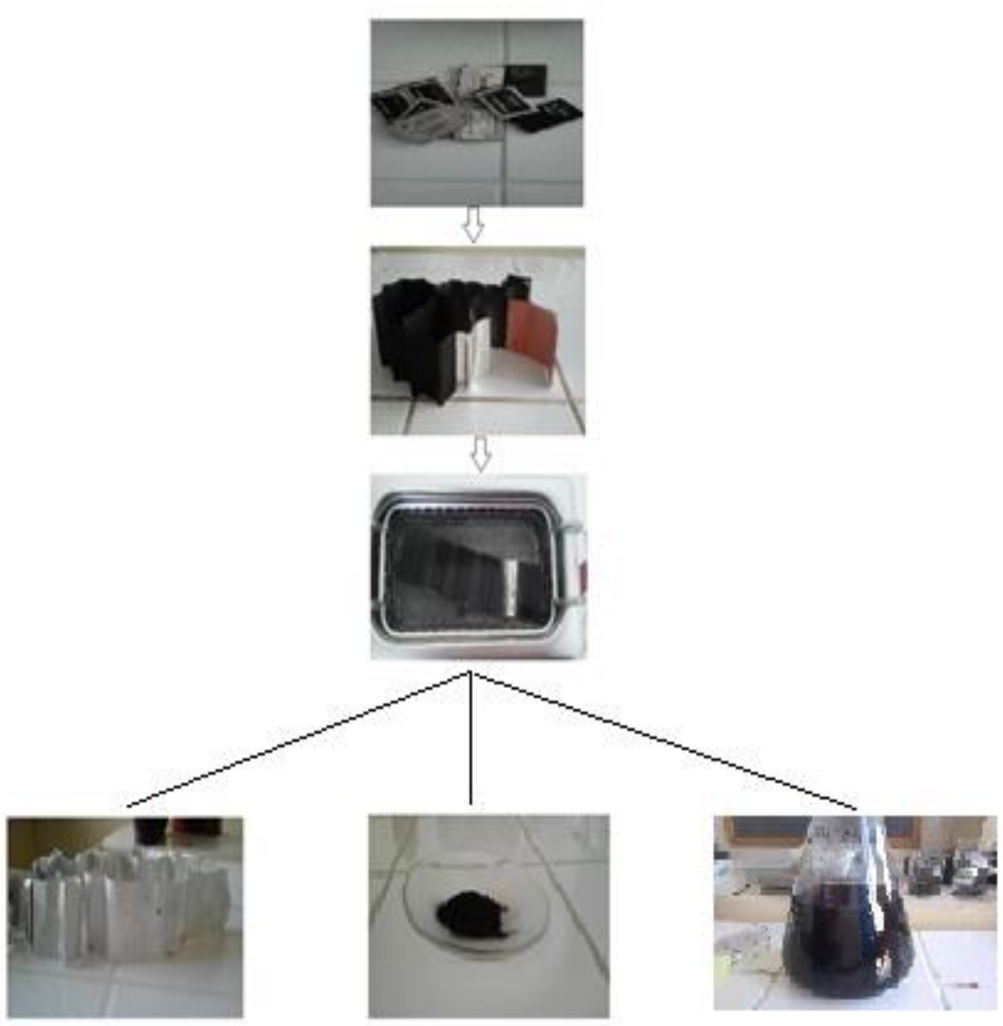

Fig.1 The flow sheet of the cathode material separation process.

The spent LIBs were discharged completely in a salt solution for 1h and dismantled manually to separate the cathode materials coated on the aluminum foil. The Al-foil was cut into small pieces initially (keep intact **for final** experiments) and immersed in acidic solution (lactic acid) and subjected for ultrasonic cleaning.

The ultrasonic cleaning machine (Emmi12-HC) used has the following technical specifications: housing – stainless steel, cleaning frequency = 45 kHz; cleaning time = 1- 60 min; volume = 1.2 l; heating temperature = 20- 80 ºC; bath dimension 200x100x65 mm; ultrasonic power= 50/75/100W.

Once all the active cathode material was detached from aluminum foil, it was filtered and washed with alcohol, and filtered again.

The results obtained by identifying the metals from the residual solutions resulting from the process of ultrasonic recovery in lactic acid medium of the cathodic paste from used Li-ion batteries. The instruments used for this purpose were Waters Ion Chromatographs (with conductivity detector) and the specific column for cations.

Table 1. The results of the analysis of the residual solution, after the recovery of the active paste

Nr. crt	Element analyzed	Unit of measurement	Detection limit	Quantification limit	Result (lactic acid – $C_3H_6O_3$)
ICP-OES					
1.	Lithium	µg/g	0.015	0.050	42.72
2.	Manganese	µg/g	0.003	0.010	1.308
3.	Cobalt	µg/g	0.750	2.500	323.440
4.	Nickel	µg/g	0.003	0.010	0.252

Conclusion

The optimal parameters for the ultrasonic process and obtaining a high separation efficiency of the active paste were:

✓ Concentration of lactic acid solution: 1.5 M;
✓ Temperature of the ultrasonic bath: 50^0C;
✓ The power of ultrasonic bath: 80 W; Time range between 1.5 to 2.5 minutes.
✓After the optimal parameters have been established, we tested the process using an entire cathode foil.
✓The result was a maximum separation efficiency of η = 88.08 %.

The concentrations of useful metals identified in the residual solution (especially the cobalt concentration) lead to the conclusion that the recovery process must be continued for the full recovery of the useful metals.

References

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