RASTVARANJE KATODNOG MATERIJALA IZ LIB U SUMPORNOJ KISELINI U PRISUSTVU AZOTA

DISSOLUTION OF LIBs CATHODE MATERIAL IN SULFURIC ACID IN THE PRESENCE OF NITROGEN

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1. Introduction

- Lithium-ion batteries (LIBs) have a key role in the further development of electric vehicles, energy storage and the renewable sources of energy.
- LIBs are composed of battery cylindrical case, anode, cathode and separator.
- Most commonly used cathode material is $LiCoO_2$.
- According to the European Union, certain components of LIBs, such as Co, P and graphite are classified as „Critical Raw Materials“, because of their economic significance and high supply risk.
- In the extraction process of valuable metals, they are firstly dissolved via leaching process; then the separation, purification and the preparation of the final product follows.

2. Experimental

2.1 Materials and methods

- Cathode material collected from 40 LIBs.
- Chemical composition of examined material was determined using inductively coupled plasma optical emission spectrometry (ICP-OES).
- For the dissolution of cathode material, 65% HNO_3 and 36% HCl (Merck, Darmstadt, Germany) in ratio 1:3, were used.
- For the continuous assessment of Co concentration, UV-VIS spectrophotometry was used (Beckman DU-65).

2.2 Leaching tests

- Leaching experiments were carried out in a reactor with measuring tube (diameter of 1 cm), which was used to determine absorbance.

- The apparatus was placed inside the spectrometer. Spectrometer working conditions, gas flow and temperature were controlled via device based on the microcontroller STM32F103.

- The obtained data was processed and given in a form of Python scripts.

- Leaching efficiencies were calculated using eq 1:

$$\text{Leaching efficiency} = \frac{\text{Metal content in leachate}}{\text{Total amount of metal in cathodic mateirals}} \times 100\% \qquad (1)$$

3. Results and discussion

3.1 Chemical Composition of the Cathode Active Material before leaching

Table 1 Metal contents found in the obtained cathode active material

Metal element	*Li*	*Co*	*Al*
Content [wt. %]	5.98	49.81	0.01

According to the obtained results, it can be concluded that the cathode material is composed mainly of $LiCoO_2$, while the presence of Al can be explained via pretreatment of the batteries.

3.2 Leaching of waste cathode material

3.2.1 Effect of acid concentration on leaching

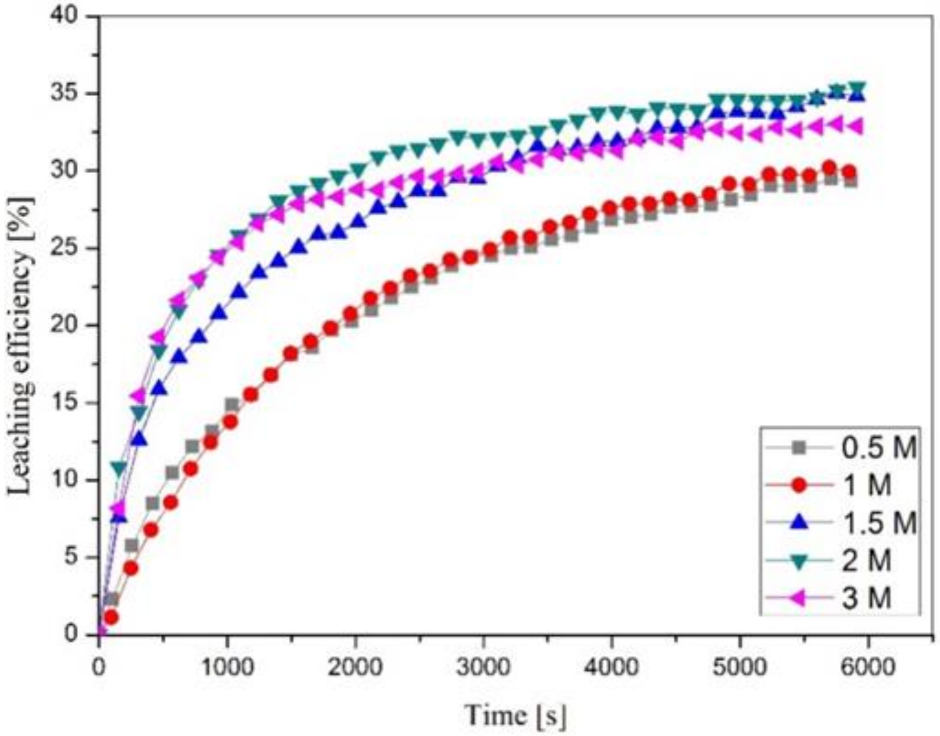

Fig.1 Effect of H_2SO_4 concentration on the leaching of waste $LiCoO_2$

3.2.2 Effect of the solid-to-liquid ratio on leaching

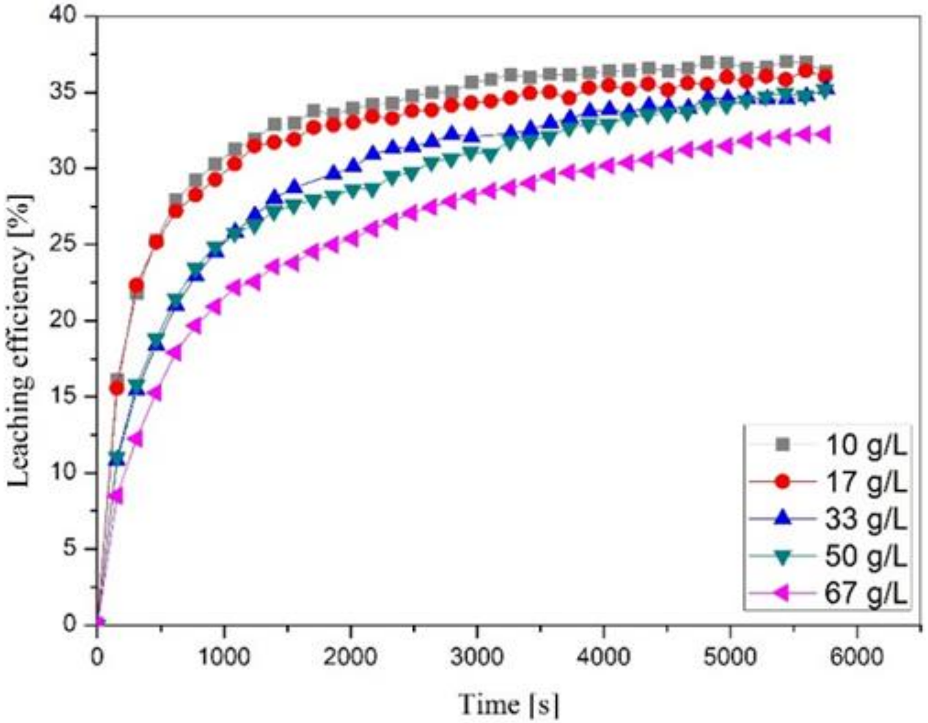

Fig. 2 Effect pulp density on the leaching of waste $LiCoO_2$

3.2.3 Effect of temperature on leaching

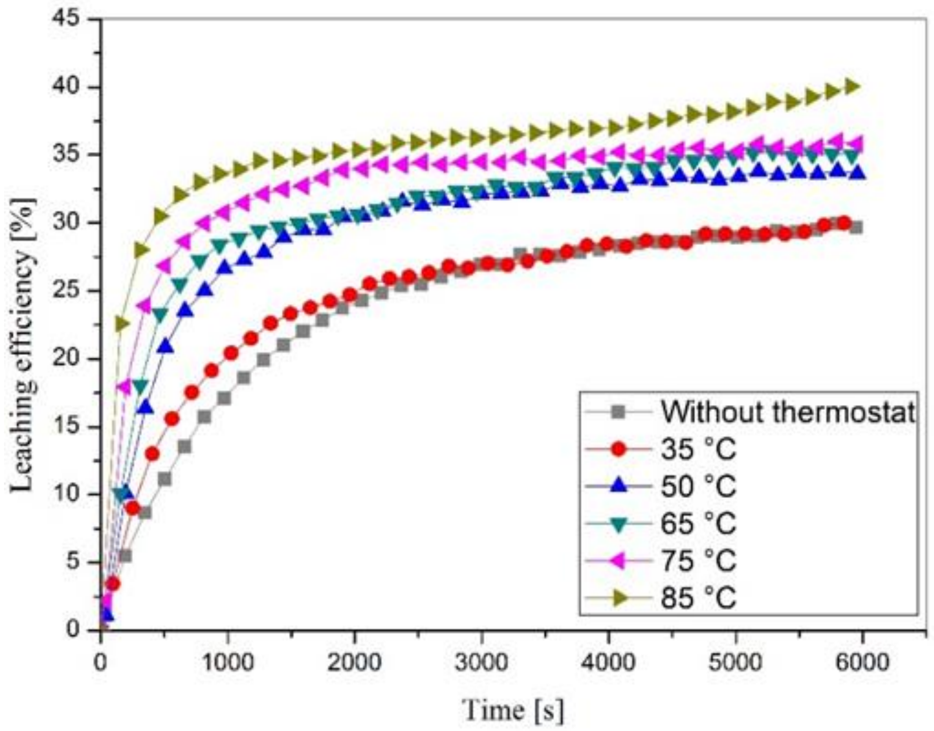

Fig. 3 Effect of leaching temperature on the leaching of waste $LiCoO_2$

4. Conclusion

- Using the H_2SO_4 in the leaching process of the cathode material obtained from the spent LIBs, low leaching efficiency of Co is achieved (around 40%).
- In view of the fact that a strong chemical bond between Co and O_2 in the $LiCoO_2$ can be formed, for a high leaching efficiency usage of an adequate reducing agent is necessary.
- Maximum cobalt leaching efficiency is achieved at the concentration of leachate being 2 M, solid-liquid ratio being 33 g/L, nitrogen flow rate being 2 L/min and at the temperature of 85°C.

THANK YOU FOR YOUR ATTENTION!