Effects of Leaching Parameters on Cobalt and Lithium Recovery from Spent Li-ion Battery Electrode

Siriya Chansakun^{*} Preecha Termsuksawad and Choochat Nitipanyawong

Division of Materials Technology, School of Energy, Environment and Materials, King Mongkut's University of Technology Thonburi, 126 Pracha Uthit Rd., Bang Mod, Thung Kru, Bangkok 10140, Thailand *Corresponding author, E-mail: siriya_jack@hotmail.com

Abstract

A hydrometallurgical process is proposed to recover cobalt and lithium from spent Li-ion batteries. The first stage to recover these metals is electrode leaching. Effects of leaching parameters: sulfuric acid (H_2SO_4) concentrations, temperature, amount of hydrogen peroxide (H2O2) and solid to liquid ratio (S/L ratio) on leaching percentage were investigated. In this study, the electrodes of Li-ion batteries were dissolved with a mixture of H_2SO_4 and H_2O_2 at various temperatures. Amounts of lithium and cobalt ions were investigated by ICP in order to calculate leaching percentage of each ion. The ratio of leaching percentage for each metal to the amount of the leaching agent (LP/LA) was determined. The results indicated that leaching of electrode with 2M H₂SO₄ at leaching temperature of 30 °C with S/L ratio of 1:10 g/ml., the leaching percentages for both cobalt and lithium were approximately 60 wt.% and increased to about 78 and 80 wt%, respectively when leaching temperature is 60 °C. An increase of H_2O_2 had little effect on the increase of leaching percentage for both elements at these conditions. However, leaching percentage for cobalt and lithium increased to about 91 and 96 wt% when 3M H2SO4 was applied. A reduction of S/L ratio also increased leaching percentage when leaching temperature is 30 °C but this effect was not obvious at 60 °C or in 3M H₂SO₄. The maximum LP/LA ratios for cobalt and lithium were obtained from the conditions: S/L 1:10 g/ml, 3M of H₂SO₄, 15 vol % of H₂O₂ at 60 °C for 30 min. The amount of cobalt and lithium obtained from these conditions were 52.08, 5.85 and 4.98 g/l, respectively. The leaching percentages for cobalt and lithium increased with the increases of H₂SO₄ concentration, temperature and amount of H2O2 but with the decrease of S/L ratio. The optimum conditions were S/L 1:10 g/ml, 3M of H₂SO₄, 15 vol% of H₂O₂ at 60 °C for 30 min., determined by LP/LA ratio.

Keywords: hydrometallurgical process, Li-ion batteries, leaching, cobalt, lithium, recovery

บทคัดย่อ

งานวิจัยนี้ประยุกต์ใช้กระบวนการไฮโครเมทัลเลอจีเพื่อแยกสกัดโลหะโกบอลต์และลิเธียมจากแบตเตอรี่ลิเธียมไอออนที่ใช้แล้ว โดยศึกษา กระบวนการซึมษะละลายด้วยสารละลายกรคซัลฟิวริกกับไฮโครเจนเปอร์ออกไซด์ ซึ่งเป็นขั้นตอนแรกในการแยกสกัดโลหะด้วยแนวทางนี้ ดังนั้น เพื่อศึกษาผลของกวามเข้มข้นของกรดชัลฟิวริก อุณหภูมิ ร้อขละ โดยปริมาตรของไฮโครเจนเปอร์ออกไซด์ และอัตราส่วนของแข็งต่อสารละลาย ต่อ ร้อขละการละลาขของโคบอลต์ และ ลิเธียม การทคลองเริ่มจากการซึมชะละลาขอิเลกโทรคด้วยสารละลายที่เครียมจากกรคซัลฟีวริก และไฮโครเจน เปอร์ออกไซค์โดยใช้อัตราส่วนของปริมาณอิเลกโทรคต่อสารละลายที่ปริมาณต่างๆที่อุณหภูมิต่างๆ ปริมาณของไอออนลิเธียมและโคบอลต์จาก สารละลาขวิเคราะห์โดขใช้ ICP เพื่อหาร้อขละการซึมชะละลาขของโลหะแต่ละประเภท และหาอัตราส่วนการซึมชะละลาขของแต่ละธาตุต่อปริมาณ สารละลายที่ใช้ (LP/LA) ผลการทดลองพบว่าร้อยละการซึมชะละลายของโคบอลด์ และ ลิเธียม ที่สภาวะความเข้มข้นของกรดชัลฟิวริค 2 โมลาร์ อุณหภูมิ 30 องศาเซลเซียส และอัตราส่วนของอิเลก โทรคต่อสารละลาย 1:10 กรัมต่อมิลลิลิตรมีค่าประมาณร้อยละ 60 โคยน้ำหนัก และเพิ่มขึ้นถึง ประมาณร้อยละ 78 และ 80 โดยน้ำหนักตามลำคับเมื่ออุณหภูมิเพิ่มขึ้นเป็น 60 องศาเซลเซียส ปริมาณของไฮโคนเจนเปอร์ออกไซด์มีผลเพิ่มร้อยละ การซึมชะละลายเล็กน้อย การเพิ่มความเข้มข้นของกรดซัลฟิวริคเป็น 3 โมลาร์สามารถเพิ่มร้อยละการซึมชะละลายของโคบอลต์และลิเธียมเป็นร้อยละ 91 และ 96 โดยน้ำหนักตามลำดับ อัตราส่วนปริมาฉอิเลค โทรคต่อปริมาตรสารละลายที่ลคลงช่วยเพิ่มร้อยละการซึมชะละลายของทั้งโคบอลต์และลิ เชียมที่อุณหภูมิ 30 องศาเซลเซียส แต่ไม่เห็นผลเมื่ออุณหภูมิเพิ่มขึ้นเป็น 60 องศาเซลเซียสหรือเมื่อเพิ่มความเข้มข้นของกรคซัลฟิวริคเพิ่มเป็น 3 โมลาร์ และเมื่อพิจารณาอัตราส่วน LP/LA พบว่าสภาวะการซึมชะละลายที่อัตราส่วนของแข็งต่อของเหลวที่ 1:10 กรัมต่อมิลลิลิตร ความเข้มข้นของกรด ซัลฟีวริก 3 โมลาร์ ปริมาณไฮโครเจนเปอร์ออกไซค์ที่ร้อขละ 15 โดยปริมาตร และอุณหภูมิที่ 60 องศาเซลเซียสเป็นเวลา 30 นาที ให้ก่าอัตราส่วน LP/LA ที่มากที่สุด โดยที่ปริมาณ โคบอลต์ ลิเชียมและอลูมิเนียมที่ละลายในสารละลายที่สภาวะนี้มีค่า 52.08, 5.85 และ 4.98 กรัมต่อลิตร ตามลำดับ ผลการทดลองพบว่าร้อยละการซึมชะละลายของโคบอลต์ และ ลิเซียม เพิ่มขึ้นเมื่อความเข้มข้นของกรคซัลฟีวริก อณหภมิ ร้อยละ โดยปริมาตรของ ้ไฮโครเจนเปอร์ออกไซด์เพิ่มขึ้น หรืออัตราส่วนของแข็งต่อสารละลาขลคลง โดยสภาวะที่เหมาะสมที่สุดสำหรับการซึมชะละลายคือ อัตราส่วน

ของแข็งต่อของเหลวที่ 1:10 กรัมต่อมิลลิลิตร ความเข้มข้นของกรคซัลฟีวริก 3 โมลาร์ ปริมาณไฮโครเจนเปอร์ออกไซค์ที่ร้อยละ 15 โคยปริมาตร และ อุณหภูมิที่ 60 องศาเซลเซียสเป็นเวลา 30 นาที โคยพิจารณาจากอัตราส่วน LP/LA

้ คำสำคัญ: กระบวนการแยกสกัคโลหะ ลิเธียมใอออนแบตเตอรี่ ซึมชะละลาย โคบอลต์ ลิเธียม การกู้คืน

1. Introduction

A lithium-ion battery is comparatively smaller and lighter but provides much more energy per unit volume than those of the nickel-cadmium (NiCd) or the nickel-metal hydride (NiMH) batteries. In addition, it has no memory effect. Lithium-ion battery is extensively used as electrochemical power sources in modern electronic equipment due to its favorable characteristics such as high energy density, high cell voltage, long storage life, low self-discharge rate and wide working temperature range. For lithium-ion battery, anode and cathode are made of graphite and powdered metal oxide such as LiCoO₂ or LiMnO₂, respectively. The cathode of lithium-ion battery generally consists of heavy metals such as cobalt, manganese, iron etc., in the proportional range of 70-75 wt%. Other elements are 7-10 wt% lithium and 12-15 wt% aluminum, varying slightly depending on manufacturers as reported by Ferreira, Prados, Majuste, & Mansur (2008). The revenue of lithium-ion transportation batteries for global market was expected to increase from 20 billion \$US in 2013 to about 53 billion \$US in 2020 (Franco, 2015). In addition, Li-ion battery consumption has increase with the growth of smartphone market. Therefore, it is expected that a massive amount of spent lithium-ion batteries will be disposed; consequently, causing a serious environment problem. The increasing trend of the waste flux generated a rapid decrease in primary resources and strict environmental regulations stimulate the need for improved waste recycling. Two basic types of recycling processes, including physical and chemical processes have been applied to recover heavy metals, lithium and other constituents from spent lithium-ion batteries. Physical processes include mechanical processes such as crushing and physical separation such as magnetic separation. The outputs from physical processes are mostly scrap and other residuals, which cannot be used directly. They need other chemical treatments to recover metals from the scraps. Chemical processes refer to chemical reaction; for example, acid leaching, solvent extraction, chemical precipitation and electrochemical process (Ferreira et al., 2008). To recover metal from the scrap, leaching is one important process because the process can recover metal in compound form from the scrap obtained from physical separation for further treatments. In this work, the various leaching process parameters were studied to obtain the maximum recovered metal. The leaching of Li-ion batteries had been conducted using H₂SO₄ as leaching agent. The leaching powers of H_2SO_4 to leach lithium are higher than that for cobalt (Swain et al., 2007). The leaching reaction of LiCoO₂ by H₂SO₄ solution can be represented as follows:

$$4LiCoO_{2(s)} + 6H_2SO_{4(aq)} \rightarrow 4CoSO_{4(aq)} + 2Li_2SO_{4(aq)} + 6H_2O_{(g)} + O_{2(g)} \dots (1)$$

However, leaching percentage for cobalt by H_2SO_4 was around 30 wt% (Swain et al., 2007). An addition of a reductant, such as H_2O_2 , is introduced because it can reduce Co(III) to Co(II) and thus increases leaching percentage of cobalt. When it decomposes, it forms water and releases oxygen, which makes it an attractive environmental friendly product. It is therefore a clean oxidant (Ferreira et al., 2008). The sulfuric leaching reaction of LiCoO₂ in the presence of H_2O_2 solution can be expressed as Swain et al., (2007).

$$2\text{LiCoO}_{2(s)} + 3\text{H}_2\text{SO}_{4(aq)} + \text{H}_2\text{O}_{2(aq)} \rightarrow 2\text{CoSO}_{4(aq)} + \text{Li}_2\text{SO}_{4(aq)} + 4\text{H}_2\text{O}_{(g)} + \text{O}_{2(g)} \dots (2)$$

The effect of H_2O_2 is possibly related to some change in the leaching kinetics of $LiCoO_2$ including the solubility of CoO_3 and the reduction of CO^{3+} in the solid to CO^{2+} in aqueous phase. From the reactions (1) and (2), cobalt and lithium in $LiCoO_2$ were leached to $CoSO_4$ and Li_2SO_4 , respectively. There are studies reported that leaching percentages of cobalt and lithium increased with the increases of concentrations of H_2SO_4 and H_2O_2 , temperature and S/L ratio (Ferreira et al., 2008; Swain et al., 2007; Shin et al., 2005; Xua et al., 2008). However, the applied temperature in these studies was 70 °C or above, which is comparatively high and there was massive loss of leaching solution due to evaporation. In addition, analysis of leaching percentage related to amount of used resources such as leaching agent was not considered. In this study, effects of concentration of H_2SO_4 , amount of H_2O_2 , temperature and S/L ratio on leaching percentage at lower temperature were investigated. Furthermore, the leaching percentages of each metal relative to amount of used leaching agent were analyzed.

2. Objectives

In this study, the electrodes of Li-ion batteries were dissolved with a mixture of sulfuric acid and hydrogen peroxide. Effects of leaching parameters: H_2SO_4 concentrations, temperature, amount of H_2O_2 and solid to liquid ratio (S/L ratio) on leaching percentage were studied.

3. Materials and methods

3.1. Materials

The cathode used in this study, analyzed by wavelength-dispersive X-ray fluorescence (WDS-XRF) spectrometry, consisted of 96.6 wt% of cobalt. However, due to WDS-XRF limitation, the amount of lithium could not be identified. 98 %(v/v) of sulfuric acid (H₂SO₄) and 30 %(v/v) of hydrogen peroxide (H₂O₂) were used as leaching chemical reagent.

3.2. Characterization of Li-ion battery cathode

The quantitative analysis to identify the cathode composition and phase were WDS-XRF spectrometer (Bruker AXS, S4 Pioneer) and X-ray diffractometer (Bruker, D8 Advance), respectively. The metal content in solution was evaluated by inductively coupled plasma mass spectrometer (ICP-MS, NexION300Q ICP-MS Spectrometer) after dissolving samples in aqua reagent.

3.3. Leaching procedure

Since lithium content cannot be analyzed by WDS-XRF, the exact composition cannot be estimated. Therefore, ICP was employed to investigate the composition of the cathode after it was completely dissolved by leaching agent in very severe condition and the composition analyzed from this leaching solution was used as a reference. The solution was prepared by mixing 5 ml of 30 % (v/v) H_2O_2 and 10 ml of 98 % (v/v) H_2SO_4 in 35 ml of DI water. To determine the condition to acquire the highest metal content, the leaching process was carried out by dissolving 1 g of battery sample with 50 ml of the prepared solution at 75 and 85 °C for 150 and 250 min. The effects of leaching temperature, H_2SO_4 concentration, solid to liquid ratio (S/L) and amount of H_2O_2 on leaching percentage was investigated. The applied temperature, H_2SO_4 concentration, S/L ratio and amount of H_2O_2 were shown in Table 1. These conditions were determined after reviewing literatures (Ferreira et al., 2008; Swain et al., 2007) and conducting a preliminary experiment.

$H_2SO_4(M)$	Temp (°C)	S/L ratio (g/ml)	$H_2O_2(\% (v/v))$
2	30	1:10, 1:15	5, 10, 15, 20
2	60	1:10, 1:15	5, 10, 15, 20
3	60	1:10, 1:15	15, 20

 Table 1 Experimental conditions.

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The metal content in leaching solution was measured by inductively coupled plasma mass spectrometry (ICP-MS, NexION300Q ICP-MS Spectrometer) after dissolving samples in aqua reagent.

In the leaching reaction, cobalt and lithium were leached as $CoSO_4$ and Li_2SO_4 , respectively and the leaching percentage was determined by:

$$Leaching percentage = \frac{amount of metal content in specific leaching condition (mg)}{amount of metal content in reference solution (mg)} x 100 \qquad \dots \dots (3)$$

From the equation 3, leaching percentage of cobalt and lithium were determined relative to their contents in reference solution prepared from extreme conditions. At these conditions, leaching solution was a mixture of 5 ml of 30 % (v/v) H_2O_2 and 10 ml of 98 % (v/v) H_2SO_4 in 35 ml of DI water and applied S/L ratio, leaching temperature and leaching time of 1:50 g/ml, 85°C and 150 min, respectively. Using only leaching percentage to determine the appropriate conditions is not quite suitable because the resource consumption, such as amount of leaching agent, is not considered. Thus, the ratio of leaching percentage to the amount of the consumed leaching agent (LP/LA) was also proposed to identify the appropriate leaching conditions, the conditions to obtain the maximum LP/LA ratio. The LP/LA ratio was determined by:

 $LP/LA \text{ ratio} = \frac{\text{leaching percentage for metal (\%)}}{\text{amount of H2SO4 and H2O2 in the condition (ml)}}$ (4)

4. Results and Discussion

4.1 Characterization of Li-ion battery cathode.

The composition of the cathodes, determined by WDS-XRF, was shown in Table 2. The cathode comprised of cobalt as a main element and following by phosphorus, which is constituted in the electrolyte, LiPF₆. However, lithium was not found by WDS-XRF due to limitation of this technique. The crystalline LiCoO₂ and graphite in the battery was clearly indicated by the X-ray diffraction (XRD), as shown in the Figure 1 This result indicated the existence of lithium in the cathode.

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Table 2 Composition of a Li-ion battery cathode in this study analyzed by XRF technique.

 Composition	Element											
(wt%)	Со	Р	Al	Mg	Cu	S	Au	Zn	Ni	Fe	Si	Sum
Cathode	96.62	1.11	0.34	0.20	0.76	0.25	0.19	0.15	0.15	0.22	-	100
Al foil Substrates	0.0087	-	48.99	-	0.0412	-	-	0.0286	-	0.3407	0.1259	100



Figure 1 XRD patterns of the studied cathode.

4.2 Composition of reference leaching solution

Figure 2 showed the amount of cobalt and lithium in leaching solution after the cathodes were leached at various temperatures and times. The results indicated that an increase of temperatures increased the solubility of cobalt and lithium but leaching time did not affect the leached amount of these elements at this temperature range. An increase of temperature increases energy of elements in the system; thus increases leaching rate. However, the maximum amount of leached element for all elements may be obtained from leaching condition at leaching time of 150 min. As a result, an increase of leaching time from 150 to 250 min did not increase amount of leached elements. The maximum amount of cobalt and lithium obtained from these conditions in 10 ml of solution were 600.5, 64.7 and 109.9 mg respectively. These data were acquired using the conditions of leaching 1 g of cathode at leaching temperature of 85 °C for 150 min. Although the further increase of temperature could be used to obtain higher amount of leached cobalt and lithium, the temperature above 85 °C was not applied because vapor pressure of the acid mixture was too high.



Figure 2 The leached amount of cobalt and lithium from spent lithium battery at various conditions: 75-150 for the temperature at 75°C and time of 150 min, 75-250 for the temperature at 75°C and time of 250 min, 85-150 for the temperature at 85°C and time of 150 min.

4.3 Leaching

Effects of H₂O₂, temperature and S/L ratio on leaching percentage for cobalt and lithium using 2M H₂SO₄ solution were shown in Figure 3. The figure showed that leaching percentage for cobalt and lithium increased with an increase of H_2O_2 . Role of H_2O_2 is to accelerate leaching kinetics as by Ferreira et al., (2008). From this study, without H_2O_2 , LiCoO₂ in leaching sulfuric solution seems to form both solid Co_3O_4 and aqueous $CoSO_4$. Thus, amount of Co(II) in the solution is limited by the Co_3O_4 formation and extra H_2SO_4 is required. With H_2O_2 addition, it helps reducing Co(III) in Co_3O_4 to Co(II) in the form of aqueous CoSO₄; therefore, amount of Co^{2+} increases. The study also mentioned that H_2O_2 helps reducing Co(III) in the form of LiCoO₂ to form aqueous CoSO₄. As a result, amount of lithium ion in leaching solution increases. Interaction effect of temperature and S/L ratio on leaching percentage for cobalt and lithium was found. From Figure 3, leaching percentage for cobalt and lithium significantly increased with an increase of temperature when the applied S/L ratio was 1:10 but slightly increased at S/L ratio of 1:15. An increase of temperature increases activation energy of the reactant as well as molecular collision frequency. As a result, leaching rate increased. At S/L ratio of 1:15, an increase of temperature had less effect on leaching percentage due to that the leachability of LiCoO₂ at S/L ratio of 1:15 at 30 °C almost reached its limit. Thus, an increase of temperature from 30 to 60 °C slightly increased leaching percentage at this S/L ratio. Although Figure 3 showed that leaching percentage for cobalt and lithium were high, approximately 80 wt. %; there is still gap to increase the leaching percentages. To further increase leaching percentage for cobalt and lithium, an increase of H_2SO_4 concentration was proposed. The other conditions for further experiments were selected by considering the result from Figure 3. Temperature of 60 °C and the amount of H2O2 of 15 and 20 vol % were chosen because the higher leaching percentages were obtained in these conditions as shown in Figure 3. Figure 4 demonstrated the leaching percentage for cobalt and lithium using 3M H₂SO₄ solution at temperature of 60 °C and amount of H₂O₂ of 15 and 20 vol%. As shown in the figure, the leaching percentage for cobalt and lithium increased to 90 and 93 wt% at S/L ratio of 1:10 and to 93 and 97 wt % at S/L ratio of 1:15, respectively. This increase is about 15 percent compared with those using 2M H_2SO_4 solution. However, amount of H_2O_2 has an insignificant effect on leaching percentage for cobalt and lithium. This phenomenon may be due to limitation of leachability of 3M H₂SO₄. It should be noted that leaching percentage for cobalt and lithium in 3M H₂SO₄ were very high and almost reached 100 wt%.

The conditions to obtain the highest leaching percentage for cobalt and lithium, approximately 94 and 97 wt.%, respectively, from the waste LiCoO₂ were at 3M H₂SO₄, temperature of 60 °C, S/L ratio of 1:15 mg/l and 20 vol% of H₂O₂. However, these conditions may not be the suitable conditions because resource utilization was not considered. Therefore, the ratio of leaching percentage to the amount of spent leaching agent, LP/LA ratio, was proposed to identify the suitable conditions.





Figure 3 The effect of volume percentage H_2O_2 on leaching percentage for cobalt and lithium with 2M H_2SO_4 solution for 30 min:

- A) Temperature at 30 °C, solid/liquid ratio of 1:10 mg/ml
- B) Temperature at 30 °C, solid/liquid ratio of 1:15 mg/ml
- C) Temperature at 60 °C, solid/liquid ratio of 1:10 mg/ml
- D) Temperature at 60 °C, solid/liquid ratio of 1:15 mg/ml



Figure 4 The effect of volume percentage H_2O_2 on leaching percentage for cobalt and lithium with 3M H_2SO_4 solution at 60°C for 30 min:

- A) solid/liquid ratio 1:10 mg/l
- B) solid/liquid ratio 1:15 mg/l

Figure 5 demonstrated the LP/LA ratio at various conditions. From the figure, the maximum LP/LA ratio was obtained at the conditions of $3M H_2SO_4$, S/L ratio of 1:10, 15 vol % H_2O_2 and temperature at 60 °C. At these conditions, leaching percentage for cobalt and lithium were approximately 87, 91 and 45 wt %, respectively. At these conditions, the leaching percentages were lower than the maximum leaching percentage of only six percent, but LP/LA ratio was higher than those at the condition obtaining the

maximum condition from 3.8 to 5.8 or about 52 percent. Therefore, the conditions in which the maximum LP/LA ratio obtained, 3M H_2SO_4 , S/L ratio of 15 vol % H_2O_2 and temperature of 60 °C, was considered the optimum conditions for leaching.



Figure 5 The LP/LA ratio for (a) cobalt and (b) lithium at conditions of 3M H₂SO₄ solution at 60 °C for 30 min.

5. Conclusion

In this study, metal recovery from Li-ion battery cathode by leaching was studied. It was found that leaching percentage for cobalt and lithium increased with increases of H_2SO_4 concentration, H_2O_2 , temperature but a decrease of S/L ratio. Interaction effect of temperature and S/L ratio on leaching percentage was found. This effect indicated the limitation of leaching power by H_2SO_4 and H_2O_2 . The conditions to obtain the maximum leaching percentage for cobalt and lithium were 3M H_2SO_4 , temperature of 60 °C, S/L ratio of 1:15 g/ml, and 20 vol % H_2O_2 . However, considering resource utilization through LP/LA ratio, the suitable conditions were 3M H_2SO_4 , 15 vol % H_2O_2 , S/L ratio of 1:10 and temperature at 60 °C.

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