Mechanochemical Process Enhanced Cobalt and Lithium Recycling from Wasted Lithium-Ion Batteries

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ABSTRACT: Cobalt (Co) and lithium (Li), rare and valuable elements, are mainly used to prepare lithium cobalt oxide (LiCoO2) for applications in lithium-ion batteries (LIBs). Developing an effective method to recover Co and Li from the waste LIBs is of great significance. In the present study, Co and Li were extracted from pure LiCoO2 powders and the extracted cathode materials powders from the waste LIBs after acid dissolution via a mechnochemical reduction process with iron powders. For pure LiCoO2 powders, the effects of Fe to LiCoO2 mass ratio, rotation speed, and mechanochemical reduction time were examined. These parameters influenced positively the extraction of Co, while they showed negligible effects on the leaching of Li. The X-ray diffraction (XRD) and scanning electron microscope (SEM) analyses indicated a promoted extraction of Li arising from the reduction of particle sizes, magnification of specific surface area, and change of the crystal structure of particles. For high-efficiency leaching of Co by the mechnochemical reduction process with iron powders, X-ray photoelectron spectroscopy (XPS) analysis indicated the changes in the valence state of Co. The actual cathode materials disassembled from the wasted LIBs pretreated by this novel mechnochemical reduction process were also explored. The results indicated that the leaching ratios of Li, Co, Mn, and Ni could reach 77.15%, 91.25%, 100%, and 99.9%, respectively. This novel mechnochemical process would be of great importance for the recovery of valuable metals from waste LIBs.

KEYWORDS: Valuable metals, Wasted lithium-ion batteries, Mechanochemical reduction, Hydrometallurgy

INTRODUCTION

Lithium-ion batteries (LIBs) have captured increasing attention for around 16 years, and LIBs outperformed nickel–cadmium and nickel metal–hydride rechargeable batteries, owing to excellent features of small size, high power density, no memory effect, long cycle life, high voltage, low self-discharge, etc.1–4 Therefore, LIBs are widely used in a multitude of portable electronics, especially mobile phones, personal computers, cameras, etc.5 As such, the LIBs are expanding their market share in the area of rechargeable batteries. The quantity and weight of LIBs in 2020 can surpass 25 billion units and 500 thousand tons, respectively.6 With extensive applications of LIBs in portable electronics, it is evident that an ocean of spent LIBs will be produced after the ∼1–3 years of lifetime service of the LIBs.7 If the spent LIBs are simply disposed, a serious environmental concern of soil contamination would be caused by the leaked organic electrolytes as well as toxic metals (Co, Li, Cu, Mn, Ni, etc.) contained in the LIBs.8 Furthermore, the spent LIBs can be used to recover valuable metals (Co, Li, Mn, Ni, etc.) or their compounds.9 For instance, lithium cobalt oxide (LiCoO2), a shared cathode active material for commercial LIBs, accounts for 27.5 wt % in a quintessential lithium secondary rechargeable battery, with a metal content of 5–20 wt % Co and 5–7 wt % Li.10,11 Therefore, the recovery of the valuable metals, especially Co and Li from the spent LIBs, is considered to be highly desirable.

Recently, several studies have been reported for the recycling of Co and Li from the spent LIBs, focusing on pyrometallurgy, hydrometallurgy, or biophydmetallurgy processes.6,12,13 In comparison to the pyrometallurgical technology with the disadvantages of emission of toxic gases and consumption of intensive energy, the biophydmetallurgical approach is more favorable,

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and involves higher efficiency, lower costs, and quite a few industrial requirements. However, the treatment period of bihydrometallurgical process is too long, and the required microbes are arduous to incubate effectively. By contrast, for the recovery of metals from spent LIBs, the broad hydrometallurgical processes, involving higher metal recovery rates with good purity, low energy consumption, and minimal gas emission, are considered to be more superior, and mainly consist of acid leaching, chemical precipitation, chemical displacement reaction, solvent extraction, hydrothermal reaction, crystallization and electrochemical accumulation, etc. Generally, the cathode active materials, separated by a series of pretreatment steps, such as mechanical separation, and thermal processes, are leached by an acidic solution in order to transfer the target metals into the solution, and followed by the treatment of the acid leachate and final wastes to recover the metals of interest by means of chemical precipitation, chemical displacement, solvent extraction, electrochemical accumulation, etc. As such, acid leaching is a key step for the recovery of valuable metals. The leaching of LiCoO$_2$ from spent LIBs is usually carried out by using inorganic acids, such as sulfuric, hydrochloric, and nitric acids as leachants, among which higher extractions of Co and Li were obtained with the hydrochloric and nitric acids as the leaching media. Moreover, for the leaching process of LiCoO$_2$ in the hydrochloric acid media, special antisepticizing equipment needs to be installed to treat chlorine (Cl$_2$) generating from HCl oxidation, leading to much greater recycling cost or serious environmental problems if this kind of equipment is not available. Furthermore, the hydrogen peroxide is generally used as a reductant during acid leaching, for the purpose that additional H$_2$O$_2$ easily reduces Co(III) to Co(II) present in the spent LIBs, which are more favorably solubilized than the unreduced moieties; otherwise, more concentrated acids are necessary to obtain comparative dissolutions. For instance, Lee et al. indicated that the leaching efficiency was increased by 45% for Co and 10% for Li in the nitric acid leaching process with the addition of H$_2$O$_2$ as a reducing agent, compared with the nitric acid leaching process without reducing agents. Moreover, whether H$_2$O$_2$ is contained in the nitric acid leaching media or not, a temperature higher than 75 °C is needed. However, the addition of H$_2$O$_2$ or high temperature might be regarded as promoting or activating processes for nitric acid extraction, and might result in a higher average costs and operational environmental risk. Therefore, an economic and safe recovery process of Co and Li from waste LIBs is highly desirable.

The mechanochemical approach has attracted more attention for potential applications in wide ranges based on the triggered physicochemical changes, including phase transformations, structural defects, strain, amorpholization, and even direct reaction under normal temperature and pressure. This approach can also make technically feasible the environmentally friendly recycling of metals from some specific wastes, such as cathode ray tube funnel glass, tin-doped indium oxide containing waste, and waste fluorescent lamps, significantly improving the recycling efficiency. As mentioned before, with the traditional hydrometallurgical acid leaching of spent LIBs, little extraction of valuable metals can be obtained at room temperature without the addition of other enhanced solvents. Herein, a mechanochemical process to pretreat the spent LIBs might be supposed to be a promoting or activating method for nitric acid extraction of valuable metals Co and Li.

Therefore, the aim of the present study is to examine the feasibility of a mechanochemical process to enhance the acid leaching of Co and Li at room temperature from the spent LIBs. Metallic iron powders served as grinding aids. Pure lithium cobalt oxide (LiCoO$_2$) powders were selected to study the influences of mechanochemical parameters on the Co and Li extraction, such as the Fe/LiCoO$_2$ powder mass ratio, rotational speed, and mechanochemical milling time. The physicochemical changes established by X-ray diffraction (XRD), scanning electron microscopy (SEM), and X-ray photoelectron spectroscopy (XPS) gave insights into the mechanism of the mechanochemical method. Ultimately, the actual scrap sample separated and concentrated from the cathode materials in the waste LIBs was examined to verify the behavior of this novel process.

**EXPERIMENTAL SECTION**

**Materials.** Lithium cobalt oxide (LiCoO$_2$, 99.8% metals basis) powder reagent was purchased from Aladdin Industrial Co. Ltd., Shanghai, China. Iron powder (Fe, ≥98% in purity), and nitric acid (HNO$_3$, AR) were obtained from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China. Ultrapure water (18.2 MΩ cm) was used for all of the experiments. The spent LIBs were used in mobile phones which were collected from Samsung Electronics Co. Ltd. (model AB463446BC and capacity 800 mAh).

**Experimental Procedure and Analysis.** A planetary ball mill (DECO-PBM-V-0.4L, DECO, China) was used for the experiments. Stainless steel pots with a volume of 100 mL and balls with a diameter of 10.0 mm were used as grinding bodies for the ball milling experiments. Prior to each experiment, certain aliquots were transferred to the reactor vessel to obtain the desired amounts. The rotation speed of the planetary disk was set as a certain value, and the rotation direction changed automatically every 30 min. After the milling, all of the samples were collected for characterization as well as acid leaching. To avoid the interference of other substances and exactly elucidate the mechanochemical mechanism, pure LiCoO$_2$ powders were first selected to study the influences of mechanochemical parameters on the Co and Li extraction and physicochemical changes established by XRD, SEM, and XPS. Ultimately, the leaching of Co and Li in the waste cathode materials from spent LIBs after mechanochemical process was also examined. Prior to mechanochemical treatment, the spent LIBs were pretreated by discharging, dismantling, and separating of cathode materials, which were followed by heat treatment to realize the separation of the cathode materials and the aluminum foil, and

![Figure 1](Image)
removal of the polyvinylidene fluoride (PVDF) binder and acetylene black conductive agent. The detailed pretreatment was presented in the Supporting Information (SI).

All the batch leaching experiments were performed in 100 mL of HNO₃ solution with the concentration of 1 mol/L in a conical flask with a 200 mL capacity immersed in a constant temperature bath at 25 ± 2 °C under magnetic stirring. Briefly, a 0.3 g portion of a ground sample was added into 1 mol/L HNO₃ solution. After immersion for 2 h, the samples were centrifuged. A 1 mL leaching sample was diluted in a 50 mL colorimetric tube with ultrapure water. After that, the sample solutions were detected with an inductively coupled plasma-atomic emission spectrometry (ICP-AES) instrument (iCAP-6300, THERMO). Briefly, the solution was diluted 50 times with ultrapure water before testing. The detection limits of Co and Li were 0.005 and 0.002 μg/mL, respectively.

The metal leaching efficiency was calculated according to eq 1.

\[ R = \frac{C_0 \times V_0}{m \times w\%} \times 100\% \]  

Here, the following abbreviations apply: \( R \) is the metal leaching efficiency; \( C_0 \) is the mass concentration of metal ions in the leachate; \( V_0 \) is the volume of leachate; \( m \) is the mass of samples; and \( w\% \) is the metal mass fraction.

**Characterization of the Samples.** Besides the ICP determinations above, the chemical composition changes of the solid samples during the mechanochemical process were also characterized. XRD was conducted using an X-ray diffractometer (D8 ADVANCE, BRUKER-AXS Corporation) with Cu Kα radiation at a scan speed of 8° (2θ) per minute ranging 2θ = 10–80°. The SEM observations were carried out on a JSM 6400, JEOL, from Japan. XPS was conducted using X-ray photoelectron spectrometry (ESCALAB 250Xi, ThermoVG Scientific), and operated via a monochromatic Al Kα X-ray source (1486.6 eV) at 150 W.

Figure 2. Influence of Fe/LiCoO₂ mass ratio on Co and Li leaching. Conditions: grinding time = 250 min, rotation speed = 650 rpm.

Figure 3. Influence of rotation speed on Co and Li leaching (a) and XRD pattern change (b). Conditions: grinding time = 250 min, Fe/LiCoO₂ mass ratio = 1:1.

Figure 4. Influence of mechanochemical reduction time on Co and Li leaching (a) and XRD pattern change (b). Conditions: rotation speed = 650 rpm, Fe/LiCoO₂ mass ratio = 1:1.
RESULTS AND DISCUSSION

Evaluation of the Performance of the Mechanochemical Process. To evaluate the performance of the leaching efficiency of the samples, the powders pretreated by the mechanochemical process were tested. The results show that less than 23% of Co and 39% of Li can only be released from pure LiCoO₂ powders after leaching for 2 h (Figure 1). However, even with leaching for 4 h, only less than 31% of Co and 55% of Li can be extracted (Figure S1), indicating that direct acid leaching is not an effective approach for the extraction of Co and Li. In a comparison with direct leaching of the raw sample, the Li extraction of both the activated sample (pure LiCoO₂ pretreatment by milling) and the mechanochemical sample (LiCoO₂ and Fe with the mass ratio at 1:1 pretreatment by milling) was increased significantly after leaching for 2 h. However, the dissolution of Co from pure LiCoO₂ powder after being ground for 250 min cannot be increased significantly by acid leaching. By comparison, the leaching efficiency of Co was increased to around 80% as the reductive metallic iron was added during the mechanochemical pretreatment process, showing a good performance for extraction of valuable metals.

Influence of Fe/LiCoO₂ Mass Ratio. The Fe/LiCoO₂ mass ratio was potentially critical for the extraction of Co and Li, since the leaching efficiency changed when the reductive metallic iron was added. Figure 2 shows that the leaching efficiency of Li stays almost the same for both the activated samples and the mechanochemical reduction samples of different mass ratios. However, the extraction of Co was increased from 23% to 80% with the addition of Fe, and the maximum dissolution rate of Co (80%) was obtained when the Fe/LiCoO₂ mass ratio was above 1:1. When the Fe/LiCoO₂ mass ratios were above 1:1, the leaching of Co was almost constant. Therefore, Fe/LiCoO₂ mass ratio of 1:1 was chosen to explore the subsequent research.

Influence of Rotation Speed. Figure 3a represents the leaching rates of the Co and Li dissolved from the powders ground at different rotation speeds. In the milling process conducted by the planetary ball mill, different rotation speeds provided different energies, which significantly affected the reaction rate of the mechanochemical reduction. The extraction of Co increased with an increase in the rotation speed, and around 80% Co was obtained at rotation speeds over 450 rpm. However, the Li extraction was not increased significantly, and this might be related to the crystal structure change. Figure 3b demonstrates the dramatic change of the crystal structure of the ground sample powders. The results show that the XRD pattern intensity decreased and the full width at half-maximum (fwhm) of all peaks was also broadened with the increase of rotation speed, and ultimately the peaks of LiCoO₂ disappeared.

Influence of Mechanochemical Reduction Time. Figure 4a shows the mechanochemical reduction time ranging from 0 to 250 min. The leaching rate of Co and Li increased more greatly with prolonged mechanochemical reduction time. The extraction of Co and Li was increased from 23% to 80% and from 39% to 63%, respectively, when the reaction time was extended from 0 to 100 min, and the recovery ratio of Co and Li was stable at a high level when the reaction time was over 100 min. Furthermore, the XRD pattern intensity decreased with an increase in the grinding time, and the peaks of LiCoO₂ disappeared after the material was ground for 100 min (Figure 4b), indicating that the higher leaching of Li and Co might arise from the amorphous LiCoO₂ crystal.

Characterization Analysis and Mechanism Discussion. To elucidate the reaction mechanism in detail, physicochemical changes were also investigated. As shown in Figures 3b and 4b, for the samples pretreated by mechanochemical process with the addition of Fe, the XRD pattern intensity decreased, and the fwhm of all peaks was also broadened with the increases in
Table 1. Properties of raw LiCoO2 powders and mechanically activated powders.

<table>
<thead>
<tr>
<th>Property</th>
<th>Raw LiCoO2</th>
<th>Activated with Fe</th>
<th>Activated without Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle size (nm)</td>
<td>20 ± 3</td>
<td>15 ± 2</td>
<td>22 ± 3</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>4.34</td>
<td>4.37</td>
<td>4.35</td>
</tr>
<tr>
<td>Specific surface area (m²/g)</td>
<td>10 ± 2</td>
<td>15 ± 3</td>
<td>12 ± 4</td>
</tr>
</tbody>
</table>

Figure 6. XPS core level spectra of Co, O, and Fe in different samples.

The larger binding energy peak is broader in the mechanochemically reduced sample by acid leaching. Two resolved peaks can be observed in each spectrum of the O 1s in Figure 6b. The BE of the O 1s spectrum in activated samples is at 529.45 eV, which can also be in good agreement with the values of 529.0 and 529.6 eV reported for LiCoO2.32,33 Meanwhile, the BE of the O 1s spectrum in activated samples (Figure 6b) is at 529.45 eV, which can also be in good agreement with the values of 529.0 and 529.6 eV reported for LiCoO2.32,33

In order to gain more evidence for the enhanced leaching mechanism of Co during the mechanochemical reduction process with reductive metallic iron, XPS was performed to determine the valence states of metals in the examined samples. Calibration was conducted using the C 1s peak of 284.8 eV. The photoelectron spectra of O 1s, Co 2p, and Fe 2p in LiCoO2 powders were analyzed by XPS (Figure 6). The binding energy (BE) of Co 2p1/2 in activated samples is at 779.86 eV (Figure 6a), very consistent with that of 779.9 eV reported for LiCoO2.32,33

The Fe 2p spectra for the mechanochemical reduction samples show a satellite structure. Herein, the intense satellite structures have been about 5−6 eV above the Co 2p1/2 and the Co 2p3/2 transitions, in the case of high-spin Co2+ (S = 3/2) compounds.34 However, the satellites in activated samples were very weak, and located about 10 eV above the core level lines, in the case of diamagnetic Co3+ (S = 0).35 The Co 2p3/2−Co 2p1/2 separation could increase with more unpaired electrons.36 Also, the separation was 15.0 eV for diamagnetic Co3+, 15.4 eV for low-spin Co2+, and 16.0 eV for high-spin Co2+ ions.35 In this work, the Co 2p spectrum of the mechanochemically reduced samples in Figure 6a shows satellite structure about 6 eV above the main Co 2p transition, and the Co 2p3/2−Co 2p1/2 separation was found to be 15.6 eV. The Co 2p spectrum of activated samples shows a satellite structure about 10 eV above the main Co 2p transition, and the Co 2p3/2−Co 2p1/2 separation is found to be at 15.1 eV. These results indicate that the valence state of Co was not changed after the activated process without Fe, i.e., Co(III), while the oxidation state of Co was changed from Co(III) to Co(II) after a mechanochemical reduction process with Fe. This might be responsible for the difficult extraction of Co from the activated sample but easy extraction from the mechanochemical reduction sample by acid leaching.

The Fe 2p spectra for the mechanochemical reduction sample are shown in Figure 6c. The BEs of Fe 2p1/2 and Fe 2p3/2 were measured as 724 and 710.76 eV. According to the handbook of X-ray photoelectron spectroscopy,37 the peaks of Fe(III) in Fe2O3 are located at 724.3 eV for Fe 2p1/2, and 710.7 eV for Fe 2p3/2 respectively. That is to say that Fe2O3 appeared after the mechanochemical reduction process with Fe. Two resolved peaks can be observed in each spectrum of the O 1s in Figure 6b. The larger binding energy peak is broader in the mechanochemical reduction samples than that on the activated samples, which is caused by the formation of Fe2O3 and the break of LiCoO2. Therefore, the change of valence state in Co was proposed for the mechanochemical reduction process (eq 2).
3Co(III) + Fe(0) → 3Co(II) + Fe(III)  (2)

On the basis of these prior observations, the destroyed crystal structure, amorphous state, and much rougher surfaces of LiCoO₂ were the reasons that leaching efficiency of Li was improved. Also, the high leaching efficiency of Co was improved by the valence state change of Co in the LiCoO₂ in the mechanochemical reduction process with iron powders.

Cobalt and Lithium Extraction from the Wasted LIBs.

To verify the effectiveness of the novel mechanochemical reduction process for the pretreatment of valuable metals leaching from spent LIBs in acidic solutions, the actual cathode materials disassembled from waste LIBs were also examined, and the experimental conditions were the same. The metal contents of the cathode materials are shown in Table S1. In a comparison with pure materials, the extractions of Li, Co, Mn, and Ni were all obviously increased after 2 h leaching in a 1 mol/L HNO₃ solution at room temperature, and these observations were in good agreement with the aforementioned results. It is worth mentioning that the extraction of Co, Mn, and Ni was significantly higher than that of activated samples. The yields of Co increased from 20.43% to 91.25%, and the yields of Mn and Ni also increased from 33.19% and 38.67% to 100% and 99.9%, respectively, between actual material and mechanochemical reduction samples (Figure 7a). In addition, over 99% of Fe can be leached out by this mechanochemical reduction process in acid solutions. Figure 7b shows the XRD patterns of different samples. The crystal structure changed dramatically, and the XRD pattern intensity was decreased; the peaks nearly disappeared in both the activated and mechanochemical reduction samples.

In a comparison with many other studies, the leaching efficiency is relatively higher. For example, the leaching efficiency of Co is 70% under the conditions of 2 mol/L H₂SO₄ + 5% H₂O₂ with a leaching time of 1 h at 75 °C,38 and 90% of Co under the conditions of 1.5 mol/L DL-malic acid (C₄H₅O₆) + 2.0% H₂O₂ with a leaching time of 40 min at 90 °C,14 and also 45% of Co by bioleaching using Aspergillus niger.39 In a comparison with these present conditions of 1 mol/L HNO₃ with leaching time of 2 h at room temperature, the aforementioned conditions in the previous studies38,14,39 need higher acid concentrations and higher temperature, which might result in more environmental risk and operational difficulties. Therefore, the results of cathode materials disassembled from waste LIBs further illustrate that the mechanochemical reduction process with Fe is an effective pretreatment approach.

CONCLUSIONS

An enhanced leaching for valuable metals in dilute acid solution from wasted LIBs via a mechanochemical reduction process with Fe is examined. The mass ratios of Fe/LiCoO₂, rotation speed, and mechanochemical reduction time have positive influences on the leaching of Co, while they have almost no effects on the extraction of Li. On the basis of the characterization analysis conducted by XRD, SEM, and XPS, the mechanochemical mechanism was proposed. The reduction of particle sizes, increase of specific surface area, and changes of the crystal structure of particles by mechanical activation enhance the leaching of Li. However, for high-efficiency extraction of Co, besides for the aforementioned reasons for Li, the mechanochemical reduction by Fe to change the valence state of Co played a dominant role. Ultimately, over 77% of Li, 91% of Co, 100% of Mn, and 99% of Ni can be extracted from actual wasted LIBs pretreated by this novel mechanochemical reduction process in dilute acid solutions. The present study presents a promising technology for cyclic regeneration of valuable metals from waste LIBs.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acssuschemeng.6b02337.

Pretreatment of the waste cathode materials, metal contents, influence of direct leaching time, and XRD patterns of activated sample (PDF)

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Notes

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