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# Optimization of Hydrometallurgical Recovery of Lithium, Aluminum, Iron, and Copper from Lithium-Iron Batteries

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**Abstract:** As lithium-iron batteries play a crucial role in the growth of electric vehicles, their disposal is projected to increase, posing significant environmental and health risks. Recovering the metals that compose these batteries can help mitigate the negative environmental impacts of mining and address raw material shortages. This research employs hydrometallurgy to recover lithium, aluminum, iron, and copper from the electrode mixture of spent lithium-iron batteries. The average metal content found for lithium, aluminum, iron, and copper was approximately 5%, 2%, 18%, and 16%, respectively. Under optimal leaching conditions, the recovery rates for lithium and aluminum reached 100% and 95%, respectively. These metals can be further separated by pH adjustment to produce lithium and aluminum products. The remaining iron and copper in the leaching residue can also be recovered through additional leaching, replacement, and pH adjustment processes to obtain products containing iron and copper.

## Introduction

Battery technology has recently become a focal point of global research due to its essential role in energy storage, electric vehicles (EVs), renewable energy, and other related applications. The lithium-iron phosphate (LFP) battery, a newer rechargeable battery type, is composed of positive and negative electrode materials (Or et al. 2020). The positive electrode is made of LFP, whereas the negative electrode is mainly made of copper and graphite (Raccichini et al. 2019). Lithium-iron (Li-Fe) batteries stand out in the energy storage sectors due to their high energy density, durability, safety, and eco-friendliness (Wang, 2021). They also offer excellent resistance to high temperatures, ensuring reliable performance under extreme conditions (Li et al. 2018; Du et al. 2022).

The surge in Li-Fe batteries demand, driven by the EV market boom, is projected to align with global EV sales reaching 21.5 million by 2030, with a 24% annual growth (International Energy Agency & Birol 2013). This growth is expected to generate five million tons of Li-Fe battery waste by 2030, underscoring the urgency for effective recycling methods to prevent environmental damage and resource loss (Beaudet et al. 2020). If Li-Fe batteries are not properly recycled, heavy metals in the battery waste could contaminate soil and groundwater, posing serious threats to the environment and ecosystems (Zhang et al. 2024). Research identifies three primary recycling methodologies: pyrometallurgy, hydrometallurgy, and direct

recycling (Chen et al. 2019; Baum et al. 2022; Du et al. 2024), each presenting distinct advantages and drawbacks.

Literature identifies hydrometallurgy as the most viable method for recycling Li-Fe batteries due to its lower environmental and energy impacts compared to pyrometallurgy (Wu et al. 2022; Zheng et al. 2023). By employing a recycling process that minimizes harmful emissions, hydrometallurgy addresses both immediate waste management needs and broader environmental sustainability goals (Bodzek & Pohl, 2022; Du et al. 2023), making it a more sustainable recycling solution. This technology, widely established in Asia for waste recycling (Li et al. 2018), utilizes aqueous solutions to dissolve metals, which are then precipitated by adjusting the solution's pH and temperature (Wang & Friedrich, 2015). Studies indicate that pH and temperature critically impact metal precipitation, acting as key factors in determining recovery efficiency in hydrometallurgical processes (Kim et al., 2023; Zhang & Deng, 2024). Specifically, adjusting pH is essential, as the solubility and precipitation of metal ions vary significantly with pH changes (Boonphan et al. 2024; Gawroński et al. 2022). Given its advantages and proven commercial success, this study adopts hydrometallurgy for Li-Fe battery recycling, highlighting its effectiveness in metal extraction and waste treatment through processes such as leaching and purification to recover pure metals or compounds.

Previous studies have focused on the cathode, the positively charged component of lithium-ion batteries,

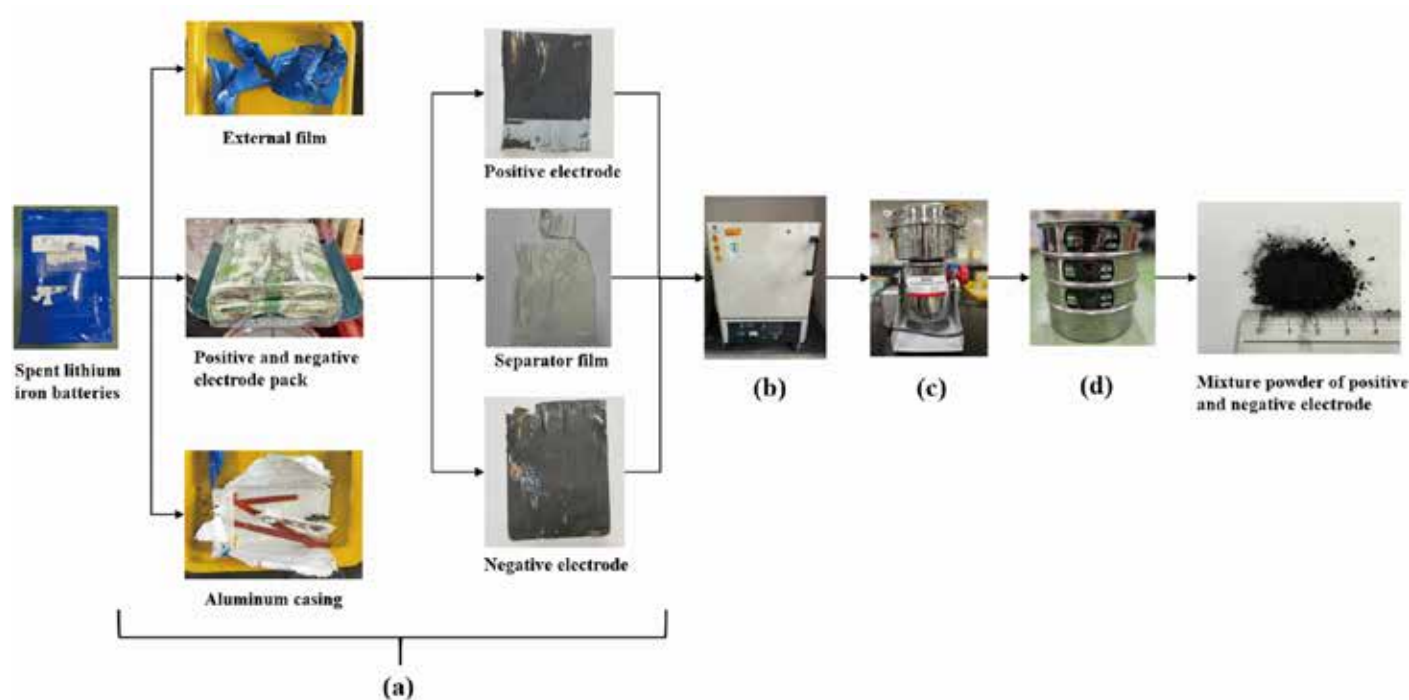


Fig. 1. Pre-treatment of spent Li-Fe batteries. (a) Disassembling, (b) Roasting, (c) Grinding, (d) Sieving.

with limited research on Li-Fe battery recovery (Chen et al. 2019). The anode, the negatively charged component, is often overlooked in recovery process despite containing valuable metals such as copper (Cu). Building on these findings, this study explores the feasibility of recovering metals from both the anode and cathode, focusing on Li, Al, Fe, and Cu. By employing various hydrometallurgical techniques, including pH adjustment, precipitation, crystallization, and replacement, this approach provides an efficient and cost-effective strategy for comprehensive battery recycling.

This study employed hydrometallurgical technology to recycle spent Li-Fe batteries using inorganic acids such as nitric acid ( $\text{HNO}_3$ ), sulfuric acid ( $\text{H}_2\text{SO}_4$ ), hydrochloric acid (HCl), which are widely favored for their high leaching recovery rates. Sodium hydroxide (NaOH) was also used as a leaching reagent, achieving up to 100% leaching recovery for metals like Li and Al. Various operating parameters, including leaching reagents, concentration, solid-liquid (S/L) ratio, temperature, and leaching time, were optimized to facilitate the extraction of Li, Al, Fe, and Cu from the mixture powder of positive and negative electrodes in spent Li-Fe batteries. Additionally, the recovery of these metals from the leaching solution was accomplished by manipulating factors such as pH, precipitation, crystallization, and replacement.

## Experimental

### Sample collection and manual disassembly

This research sourced domestically generated spent Li-Fe batteries from local Li-Fe battery recycling industries. To ensure safety during manual disassembly, the batteries were initially fully discharged, with thorough checks conducted to confirm their complete depletion. After discharge, the disassembly process involved cutting, crushing, and sieving,

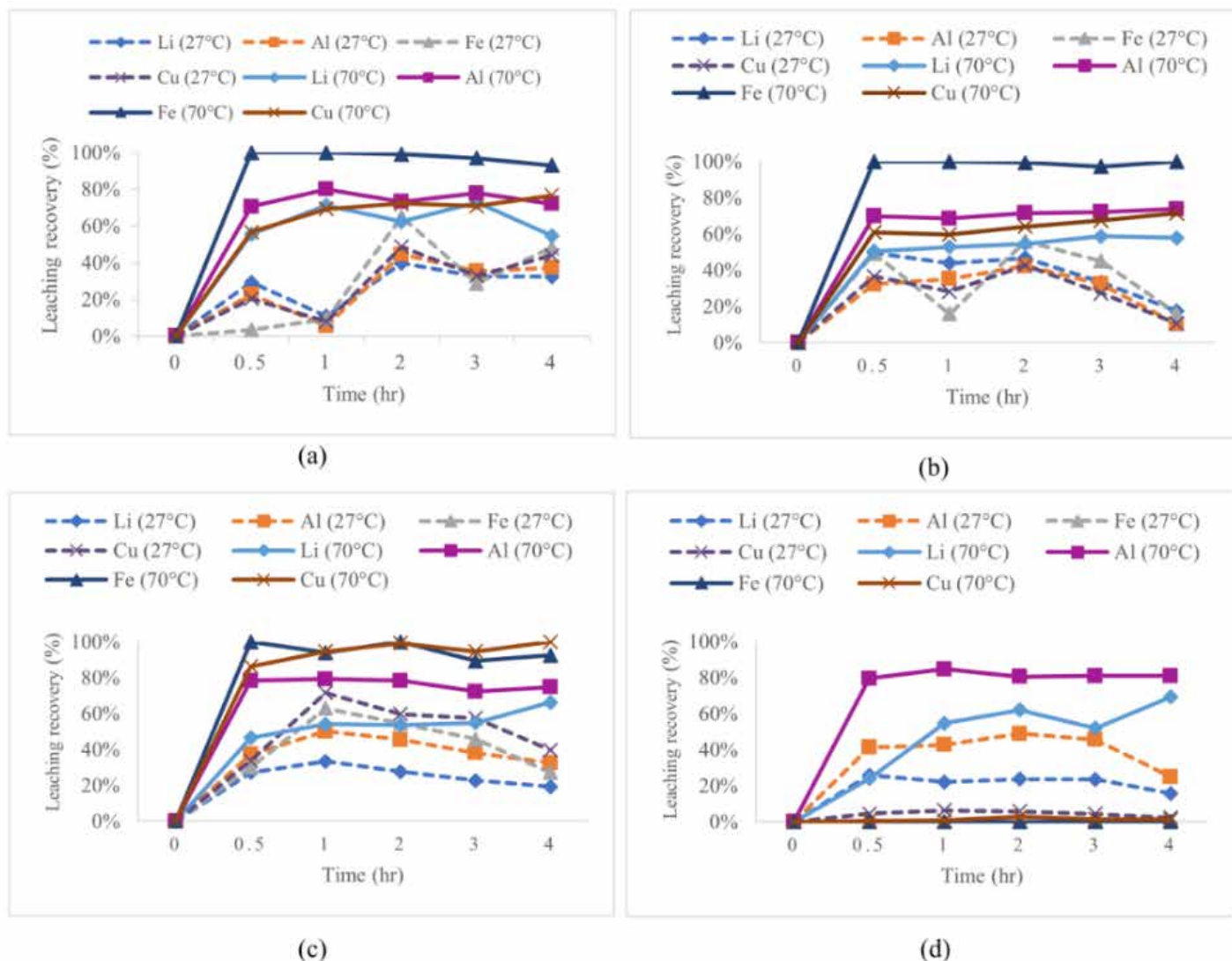
which separated the cells into metal casings and a mixture of electrode materials.

### Pre-treatment and Composition analysis

To remove organic substances, this study subjected the mixtures of positive and negative electrodes from spent Li-Fe batteries to a roasting process at  $800^\circ\text{C}$  for 3 hours. After roasting, the samples were ground to achieve a particle size smaller than 100 mesh (0.149 mm), optimizing the recovery of metals in subsequent processes. The roasted mixtures underwent thorough analysis to determine their moisture, ash, combustible content, specific gravity, and full metal content. For moisture content, this study employed the indirect measurement method (NIEA R203.02C) designated for waste management by the National Environmental Research Academy in the Republic of China (Ministry of the Environment 2009). Analysis of ash and combustible material followed the Academy's waste analysis guidelines (Ministry of the Environment 2003). Full metal content was quantified using the Aqua Regia Digestion Method (NIEA S321.65B) for heavy metals in soil, as recommended by the same institution (Ministry of the Environment 2018). Specific gravity measurements were conducted with a Weil pycnometer to determine the density of the electrode mixtures. This study provided critical insights into the composition and metal content of positive and negative electrode materials in Li-Fe batteries, facilitating advancements in recycling and recovery techniques.

### Leaching

The experiments, conducted in batches, assessed the leaching recovery process across various operational parameters: reagents concentration (1N ~ 10N  $\text{HNO}_3$ , 1N ~ 18N  $\text{H}_2\text{SO}_4$ , 1N ~ 6N HCl, and 1N ~ 18.33N NaOH), leaching times (0.5 ~ 4 hours), S/L (1g ~ 5g/50mL), and temperatures ( $27^\circ\text{C}$  and  $70^\circ\text{C}$ ). Thorough mixing was achieved with a magnetic stirrer.



**Fig. 2.** Effect of temperature on the leaching recovery of Li, Al, Fe, and Cu under 1g/50ml with (a) 1N HNO<sub>3</sub>, (b) 1N H<sub>2</sub>SO<sub>4</sub>, (c) 1N HCl, (d) 1N NaOH.

Each trial used deionized water to maintain accuracy and reproducibility. To evaluate the leaching recovery of metals, this study used the following equation (1):

$$\text{Leaching recovery (\%)} = \frac{(W_l)}{(W_i)} \times 100\% \quad (1)$$

Where,  $W_l$  is the weight of the leached metal, and  $W_i$  is the initial weight of the metal in the sample. The leaching solution was analyzed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) to measure the concentrations of Li, Al, Cu, and Fe, enabling the evaluation of leaching recovery.

### Purification

This study utilized methods such as pH adjustment, replacement, and crystallization to recover Li, Al, Fe, and Cu from the leaching solution of the mixture powder from positive and negative electrodes. The recovery efficiency of these methods was evaluated using ICP-MS. To assess the recovery of target metals from the mixture powder, the precipitation and replacement recoveries were calculated using the Eq. (2) after determining the initial weight of the target metal in the

leaching solution ( $W_s$ ) and the final weight of the target metal in the solution after the recovery process ( $W_f$ ).

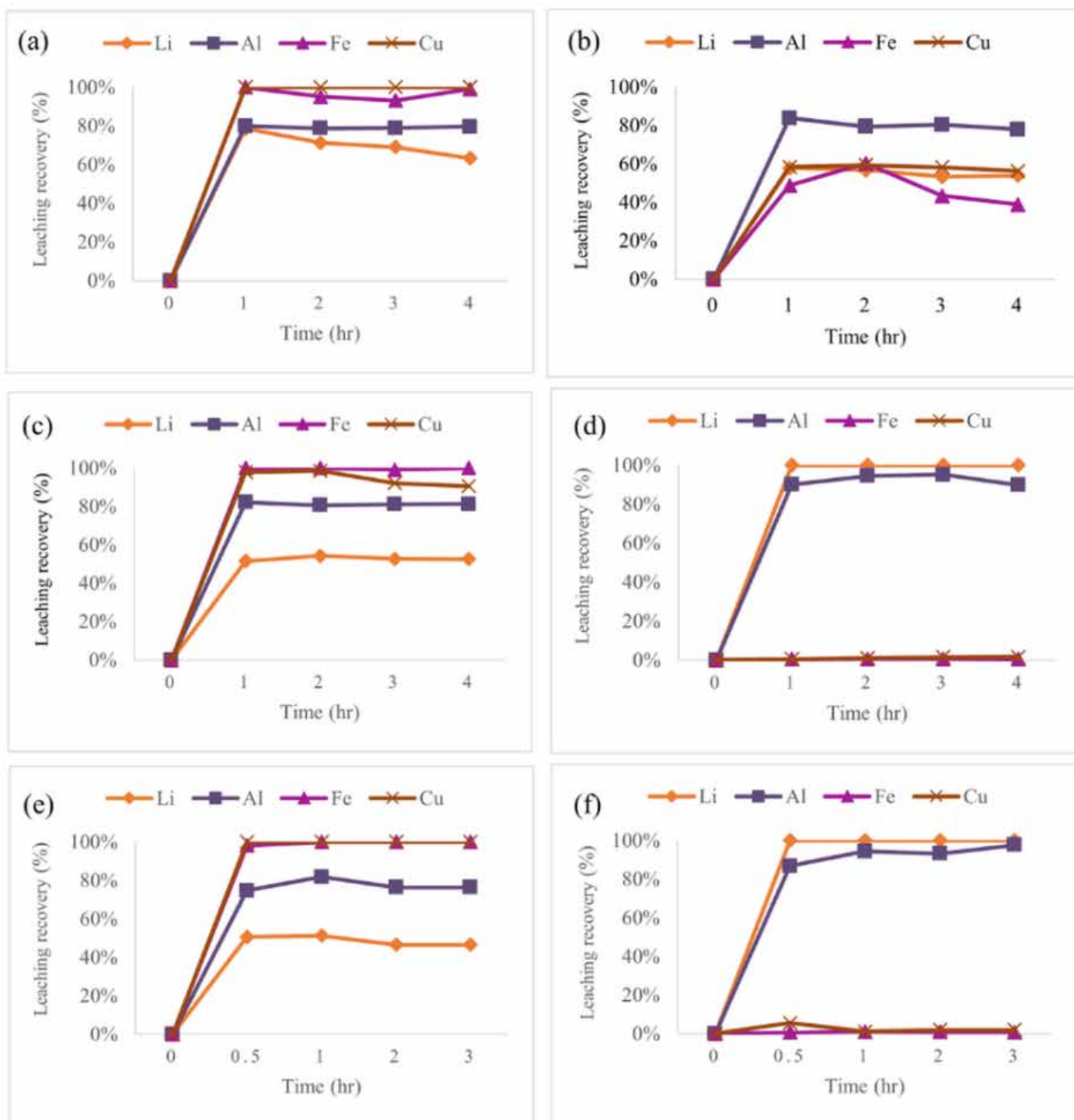
$$\text{Precipitation (Replacement) recovery (\%)} = \left( \frac{(W_s - W_f)}{W_s} \right) \times 100\% \quad (2)$$

For pH adjustment, the pH values of the leaching solution were systematically altered (1, 3, 5, 7, 9, and 11) using H<sub>2</sub>SO<sub>4</sub> and NaOH. For replacement, an iron plate was introduced into the leaching solution for varying durations (1-4 hours) to facilitate metal-ion replacement. Additionally, this study optimized the conditions for the crystallization process to enhance the separation and purification of targeted metals.

## Results and Discussions

### Manual disassembly

This study successfully disassembled Li-Fe batteries manually, separating their components into external film, aluminum casing, positive electrode, negative electrode, separator film, and electrolytes. The disassembly process and corresponding components are shown in Fig 1. Analysis determined the



**Fig. 3.** The leaching recovery of Li, Al, Fe, and Cu under 70°C and 1g/50ml with (a) 7N HNO<sub>3</sub>, (b) 18N H<sub>2</sub>SO<sub>4</sub>, (c) 6N HCl, (d) 13.75N NaOH, (e) 10N HNO<sub>3</sub>, (f) 18.33N NaOH.

following average weight percentages: external film (1.19%), aluminum casing (12.50%), positive electrode (47.71%), negative electrode (30.66%), separator film (2.14%), and electrolytes (5.80%). Significantly, the combined weight of the positive and negative electrodes constituted 78.37% of the Li-Fe batteries, highlighting their critical role in the recycling process, particularly for metal recovery. In contrast, the lower percentages of the external film and separator film indicate their smaller contributions to the Li-Fe battery's overall composition, however, their recycling remains essential to ensure comprehensive waste management.

### **Composition of the mixture powder of positive and negative electrode**

The mixture powder of positive and negative electrodes was roasted and sieved to a particle size of less than 100 mesh (<0.149 mm) for further analysis to quantify metal content and other components. ICP-MS results indicated average metal contents of Li (5%), Al (2%), Fe (18%), and Cu (16%), highlighting a notable presence of Fe and Cu in the mixture powder. Additionally, the mixture powder contained 0.17% moisture, a significant high ash content of 98.91%, and 1.00% combustible material. The density of these samples was measured at 2.63g/cm<sup>3</sup>.



### Leaching

The leaching process aimed to optimize conditions for the complete extraction of metals, including Li, Al, Fe, and Cu, from the mixture powder of positive and negative electrodes. To enhance leaching recovery, batch tests were conducted under varying conditions, including leaching reagents, temperature, and solid-to-liquid ratio (S/L), while maintaining a constant stirring speed of 150rpm.

#### Effect of temperature on leaching

The results of different temperatures on the leaching recovery of Li, Al, Fe, and Cu under a solid-to-liquid ratio of 1g/50ml and 1N leaching reagents are shown in Fig 2. The results indicate that, at the same reagent concentration, increasing the temperature from 27°C to 70°C significantly improved the leaching recovery of Li, Al, Fe and Cu. Notably, the leaching recovery of Fe reached 100% at 70°C. However, the leaching recoveries for Li, Al, and Cu still showed room for improvement, suggesting that further research is needed to optimize the reagent concentration at 70°C to enhance the leaching recovery of these metals.

#### Effect of concentration on leaching reagents

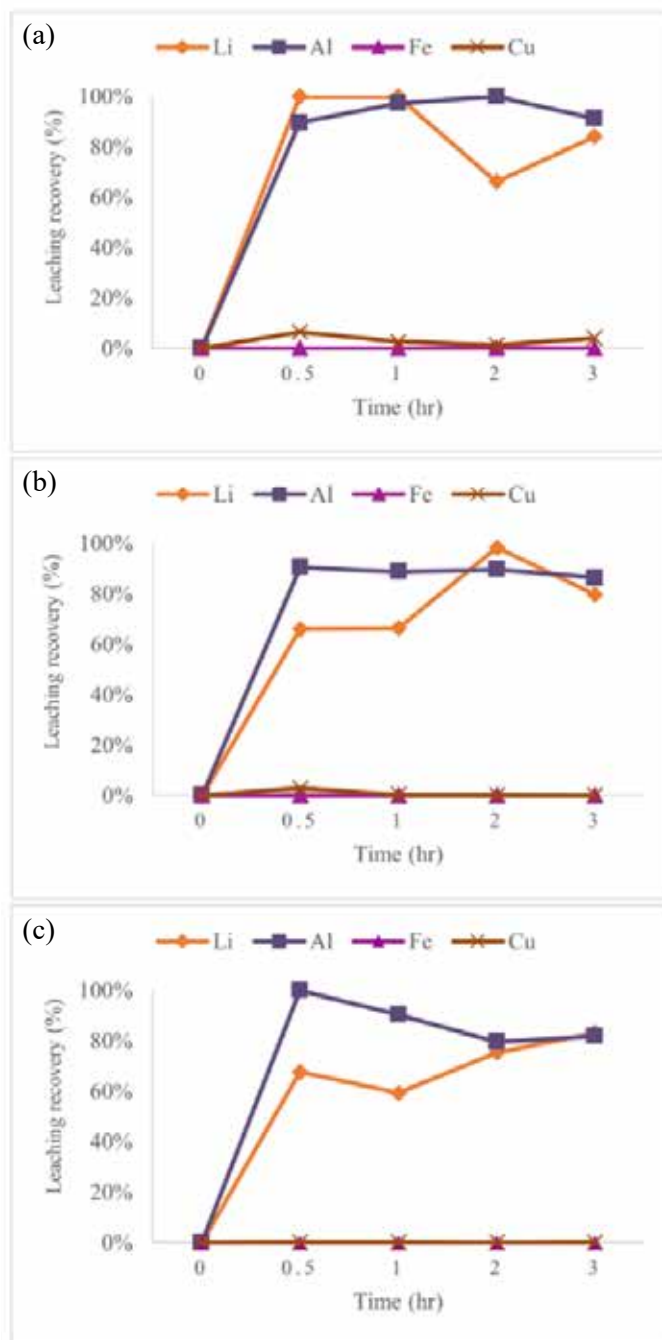
The effect of different leaching reagents and concentrations under 70°C and S/L of 1g/50ml is shown in Fig 3. The results demonstrated that increasing the concentrations of HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, HCl, and NaOH significantly improved leaching recovery compared to the baseline 1N solution. Notably, at 7N HNO<sub>3</sub>, the leaching recovery of Fe and Cu reached 100%, while Li and Al achieved approximately 80% recovery. Furthermore, the results showed that 13.75N NaOH enabled 100% recovery of Li and approximately 95% recovery of Al but was ineffective for Fe and Cu, highlighting its potential for selective metal separation.

Next, this study increased the nitric acid concentration to 10N and found no improvement in the leaching recovery of Li, indicating that no further improvement occurred beyond the concentration threshold. Likewise, increasing sodium hydroxide to 18.33N slightly improved the leaching recovery of Al but also increased the leaching recovery of Cu, indicating a delicate balance required to optimize leaching concentrations for the recovery of target metals. Ultimately, this study selected 13.75N NaOH as the optimal leaching reagent and concentration to achieve the best leaching recovery for metal separation.

This study critically examined the intricate relationship between leaching reagent concentrations and leaching recovery. The findings suggest that while certain concentrations significantly improve recovery for specific metals, achieving a universal solution for 100% leaching recovery across the target metals remains a significant challenge.

#### Effect of solid-to-liquid ratio on NaOH leaching

The results of different S/L ratios under 13.75N NaOH and 70°C are shown in Fig 4. This study observed that increasing the S/L ratio (2g/50mL, 3g/50mL and 5g/50mL) reduced the leaching recovery of Li and Al compared to S/L ratio of 1g/50mL (see Fig. 3 (d)). Meanwhile, the leaching recovery of Cu and Fe remained consistently low. Therefore, an S/L ratio of 1g/50mL was determined to be the most effective for separating Li and Al from Fe and Cu at this stage, resulting in

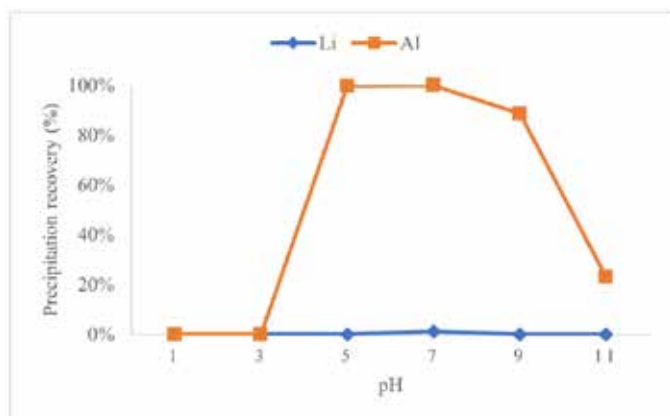


**Fig. 4.** Effect of S/L of (a) 2g/50ml, (b) 3g/50ml, (c) 5g/50ml on the leaching recovery of Li, Al, Fe, and Cu under 13.75N NaOH and 70°C.

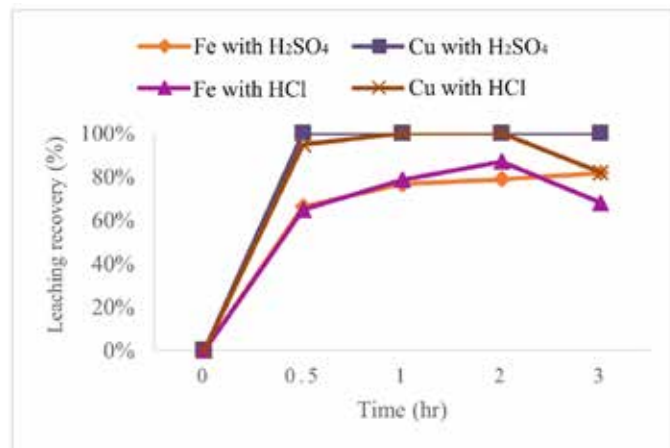
a leaching solution containing Li and Al and a leaching residue containing Cu and Fe.

#### Optimal leaching condition

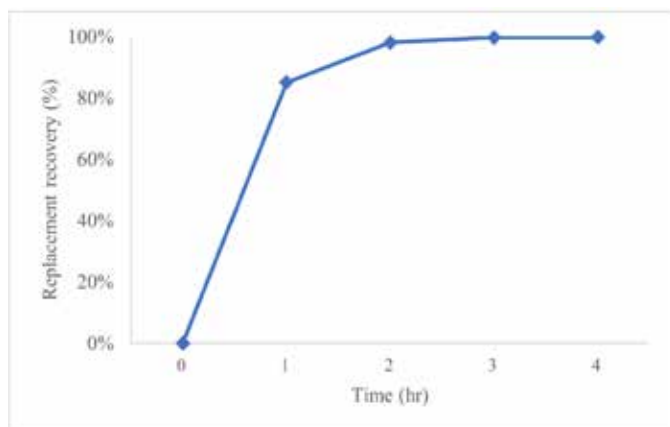
Based on the results of the above leaching processes, the optimal conditions for leaching the mixture powder of positive and negative electrodes were identified as 13.75N NaOH, 70°C, an S/L ratio of 1g/50ml, and a duration of 2 hours. Under these conditions, 100% of Li, 95.14% of Al, and minimal amounts of Fe and Cu (1.38% and 0.44%, respectively), were leached, effectively separating Li and Al from Fe and Cu. This process produced an optimal leaching solution containing Li and Al, while the leaching residue, containing Fe and Cu, was



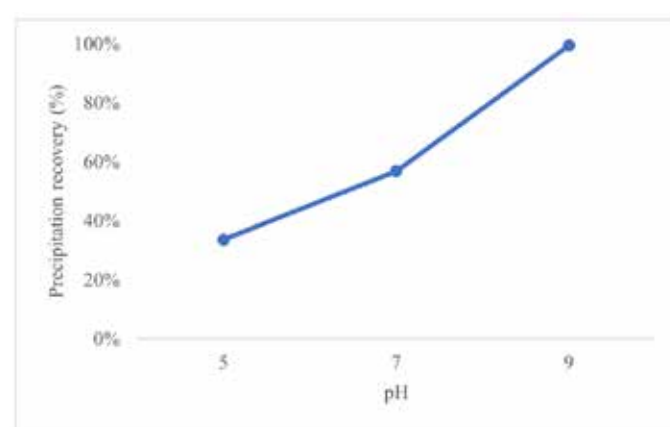
**Fig. 5.** Effect of pH value on the precipitation recovery of Li and Al for optimal leaching solution.



**Fig. 6.** The leaching recovery of Fe and Cu for optimal leaching residue under 70°C, 1g/50ml, 1N H<sub>2</sub>SO<sub>4</sub> and 1N HCl.



**Fig. 7.** Replacement recovery of Cu for H<sub>2</sub>SO<sub>4</sub> leaching solution with iron plate.



**Fig. 8.** Effect of pH value on the precipitation recovery of Fe for Fe filtrate.

filtered out. Furthermore, the residue containing Fe and Cu can potentially undergo subsequent leaching steps to obtain a separate leaching solution for Fe and Cu.

#### **Recovery of Li and Al from optimal leaching solution**

The optimal leaching solutions in this study were collected after the leaching process under the optimal conditions. These solutions then underwent pH adjustment and crystallization methods to facilitate the purification, separation, and recovery of Li and Al. The focus of this study was on using H<sub>2</sub>SO<sub>4</sub> to adjust the pH of the optimal leaching solutions, aiming to identify the best conditions for precipitating Al. The results, depicted in Fig 5, show that adjusting the pH of the optimal leaching solutions to 5 with H<sub>2</sub>SO<sub>4</sub> resulted in no precipitation of Li, while 99.69% of Al precipitated. However, at a pH of 7, the precipitation recovery for Al reached 100%, but 1.12% of Li also precipitates. Based on these findings, the study selected a pH value of 5, adjusted with H<sub>2</sub>SO<sub>4</sub>, as the optimal condition for effective precipitating Al from Li.

The Li filtrate obtained after adjusting the pH to 5 was further subjected to crystallization in this study. The temperature of the Li filtrate was set to 80 °C, and the operating time was set to 5 hours. These conditions were optimized to ensure the complete transformation of the Li filtrate into solid crystals. As

a result, a Li crystallization product was successfully obtained through this process.

#### **Recovery of Fe and Cu from optimal leaching residue**

Due to the presence of Fe and Cu in the leaching residue, this study used 1N H<sub>2</sub>SO<sub>4</sub> and 1N HCl as leaching reagents to extract Fe and Cu from optimal leaching residue. The conditions were as follows: temperature of 70°C, S/L ratio of 0.5g/25ml, stirring speed of 150rpm, and leaching times of 0.5, 1, 2, 3 and 4 hours. The leaching results for optimal leaching residue are shown in Fig 6. The results showed that after leaching for 1 hour under the conditions of 1N H<sub>2</sub>SO<sub>4</sub> and 1N HCl, the leaching recovery of Cu reached 100%, while the leaching recovery of Fe exceeded 75%. These results showed that both 1N H<sub>2</sub>SO<sub>4</sub> and 1N HCl were effective for leaching Fe and Cu from the optimal leaching residue. Based on these findings, this study finally selected 1N H<sub>2</sub>SO<sub>4</sub> as the best leaching reagent for the extraction of Cu and Fe from the optimal leaching residue.

The leaching solution containing Fe and Cu in this research was collected after the 1N H<sub>2</sub>SO<sub>4</sub> leaching phase. These solutions then underwent replacement and pH adjustment techniques to facilitate the purification, segregation, and recovery of Fe and Cu from the leaching solutions. In this study, an iron plate was

used to replace Cu from the leaching solution containing Fe and Cu over a period of 1 to 4 hours, aiming to determine the optimal conditions for separating of Cu from Fe. The results, as shown in Fig 7, indicate that using an iron plate to replace Cu in the 1N H<sub>2</sub>SO<sub>4</sub> leaching solution for 4 hours allowed for the replacement of 99.77% of the Cu, effectively separating Cu from this leaching solution. This method demonstrated the potential of using replacement reactions for metal recovery, particularly for the efficient separation and recovery of Cu from the leaching solution containing Fe and Cu. After the Cu replacement, a Fe filtrate was obtained for further treatment. NaOH was employed to adjust the pH of the Fe filtrates to 5, 7, and 9, aiming to identify the optimal precipitation recovery for Fe from the Fe filtrates. The results, as illustrated in Fig 8, indicate that adjusting the pH to 9 with NaOH facilitated the precipitation of 99.41% of Fe in the Fe filtrate. The use of these methods has proven effective in improving the recovery of valuable metals from spent Li-Fe batteries, thereby promoting the sustainable recycling and utilization of these resources, including Li, Al, Cu and Fe.

## Conclusions

The focus of this study is to use hydrometallurgy to identify the optimal leaching conditions and effectively separate and recover Li, Al, Fe, and Cu from Li-Fe batteries. The main conclusions drawn are as follows:

- The specific components of Li-Fe batteries include the external film, aluminum casing, positive electrode, negative electrode, separator film, and electrolyte, with the average weight percentages as follows: external film (1.19%), aluminum casing (12.50%), positive electrode (47.71%), negative electrode (30.66%), separator film (2.14%), and electrolyte (5.80%).
- In the pre-treatment phase, the Li-Fe batteries were first manually disassembled to remove the aluminum casing and external film, leaving the positive and negative electrode pack. The disassembled components were then roasted at 800°C for 3 hours, after which they were ground and sieved to a size smaller than 100mesh (0.149mm), resulting in a mixture powder of the positive and negative electrodes.
- An analysis of the mixture powder of the positive and negative electrodes showed the following composition: moisture content of 0.17%, ash content of 98.91%, and combustible material content of 1.00%. The specific gravity of the mixture was found to be 2.63 g/cm<sup>3</sup>. The metal content in the mixture powder included 5% lithium (Li), 2% aluminum (Al), 18% iron (Fe), and 16% copper (Cu).
- The optimal conditions for leaching Li and Al from the mixture powder of positive and negative electrodes were determined to be a 13.75N NaOH, 70°C, 1g/50ml and 2 hours. Under these conditions, complete leaching recovery for Li (100%) and significant leaching of Al (95%) were achieved, while the leaching recovery for Fe and Cu was negligible (0% to 1%), effectively separating Li and Al from Fe and Cu.
- Adjusting the pH of the optimal leaching solution to 5 with H<sub>2</sub>SO<sub>4</sub> effectively recovered 99.69% of Al, leaving a filtrate enriched in Li. Li was effectively separated and recovered by crystallizing the Li filtrate at 80°C for 5 hours.
- The optimal leaching residue under 1N H<sub>2</sub>SO<sub>4</sub> at 70°C with 1g/50ml ratio for 3 hours, facilitated complete leaching of Cu (100%) and substantial leaching of Fe (over 80%). Using an iron plate as a replacement in the 1N H<sub>2</sub>SO<sub>4</sub> leaching solution containing Fe and Cu for 4 hours resulted in the replacement of 99.77% of the Cu, effectively separating it. After this separation, adjusting the pH of the remaining Fe filtrate to 9 allowed for the effective precipitation and separation of 99.41% of the Fe.
- Based on the optimal recycling process developed in this study, the overall recovery rate of Cu from the negative electrode (anode) material was calculated to be 98.40%. For the positive electrode (cathode) material, the overall metal recovery rates were determined to be 100% for Li, 94.85% for Al, and 79.53% for Fe.

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