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Impact of pH and collector concentration on froth flotation of spent lithium-ion battery materials

Zaera Regitta Prawiranto[1*], Indra Perdana[1], Himawan Tri Bayu Murti Pretus[1]

[1] Department of Chemical Engineering, Universitas Gadjah Mada, Yogyakarta, Indonesia
[*] Corresponding author email: zaeraregittaprawiranto@mail.ugm.ac.id

Abstract

Lithium-ion batteries (LIBs) are widely used in mobile phones and electric vehicles, but their limited lifespan leads to environmental challenges, particularly due to heavy metal waste. Recycling LIBs is essential for reducing pollution and recovering valuable materials from both the anode and cathode. Froth flotation has emerged as a promising technique for separating these materials, though recovery efficiency and optimal conditions remain unclear. Pretreatment is critical for improving recovery, with pyrolysis being effective for removing organic binders like PVDF and CMC, which are commonly used in LIB electrodes. Pyrolysis, performed at 500°C, decomposes binders and recovers valuable cathode materials such as nickel, cobalt, and aluminium. This study investigates the recovery of graphite (anode) and metal oxides (cathode) in froth flotation by varying collector concentrations (850–2500 g/t) and pH levels (4–9). The highest graphite recovery (90.05%) occurred at a collector concentration of 850 g/t, while the metal oxide recovery was 25.5%. Lower collector concentrations resulted in incomplete anode particle coverage, while higher concentrations reduced selectivity. At pH 7, the best recovery was achieved, with graphite recovery of 94.61% and metal oxide recovery of 24.14%. These findings highlight the importance of pyrolysis pretreatment for binder removal and provide insights into optimizing flotation parameters, enhancing the efficiency and sustainability of LIB recycling processes.

Keywords

LIB, Froth flotation, Graphite, Metal oxide, Recovery efficiency

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Introduction

Lithium-ion batteries (LIBs) are widely used in portable electronics, electric vehicles (EVs), and energy storage systems for renewable energy. The growing adoption of EVs is a primary driver of LIB demand, particularly as the transportation sector shifts toward electrification. Additionally, the increasing integration of batteries in electricity grids further accelerates their usage. LIBs are highly valued for their high energy density, which ensures long-lasting performance and efficient energy storage. Their cost-effectiveness and durability further enhance their appeal by reducing the need for frequent replacements. These attributes establish LIBs as vital components in advancing sustainable energy solutions and fostering the future of electric mobility [1][2][3].

Despite their advantages, LIBs face significant challenges, particularly their relatively short lifespan [4]. As batteries degrade over time and lose efficiency, they are ultimately discarded, generating substantial amounts of waste. This waste is particularly concerning because LIBs contain hazardous materials, including heavy metals and toxic chemicals. If not properly disposed of, these materials can leach into the environment, contaminating soil and water and posing serious ecological risks. Improper disposal practices therefore represent a pressing environmental threat that demands sustainable waste management strategies.

Recycling LIBs have emerged as a promising solution to mitigate these environmental challenges. With growing global efforts toward sustainability, recycling has gained momentum as an eco-friendly approach to managing battery waste. As LIBs demand surges, particularly in sectors like electric vehicles and renewable energy storage, the urgency to develop efficient recycling solutions becomes increasingly evident. Recycling not only reduces waste but also recovers critical materials like lithium, cobalt, and nickel. This not only minimizes environmental harm but also conserves finite natural resources, fostering a circular economy. As a result, LIBs recycling aligns with broader goals of sustainable development and reduced ecological impact [5][6].

Various methods have been explored for addressing the environmental challenges posed by LIB waste [7]. Pyrometallurgy, hydrometallurgy, and direct recycling are among the most common approaches. Pyrometallurgy uses a high-temperature metallurgical process to extract valuable metals based on their melting and boiling points. It consists of three stages: pyrolysis, metal reduction, and gas treatment. First, organic materials in LIBs are thermally decomposed, and then metal alloys are formed at around 1500°C with reducing agents. Finally, the gas is cooled at approximately 1000°C to prevent the release of toxic gases [8]. In contrast to the pyrometallurgical process, the hydrometallurgical process recovers metals in the form of alloys. The hydrometallurgical process includes leaching and extraction stages [9]. Various chemical reagents, including acids and alkalis, are used to dissolve metal ions from battery waste. Common leaching agents in hydrometallurgy include inorganic acids like nitric acid (HNO_3), phosphoric acid (H_3PO_4), hydrochloric acid (HCl), and sulfuric acid (H_2SO_4) [10]. However, these acids can release toxic gases such as Cl_2, SO_3, and NO_x during the process. Pyrometallurgical and hydrometallurgical methods recover valuable metals but come with limitations. Pyrometallurgy is energy-intensive, requiring high temperatures that lead to material loss, while hydrometallurgy relies on acids, which generate wastewater and require complex multistep processes [11][12]. In the opposite, direct recycling is a more sustainable and energy-efficient approach. By regenerating cathode materials for reuse in new batteries, direct recycling minimizes emissions and energy consumption. Flotation has emerged as a crucial step in direct recycling, enabling effective separation of materials and enhancing sustainability [13][14]. The flotation method separates components from a mixture based on their hydrophobic or hydrophilic surface characteristics. Gas bubbles are used to selectively attach to the hydrophobic particles, lifting them to the liquid surface, where they form a froth zone for separation. Meanwhile, the hydrophilic particles are removed from the bottom outlet as tailings [15].

Direct recycling begins with a critical feed preparation phase that optimizes flotation separation. This phase includes discharging and dismantling LIBs, as well as removing active materials bound to polymers like PVDF (polyvinylidene fluoride) and CMC (carboxymethyl cellulose) [16]. PVDF is an organic binder that allows the cathode to attach to the surfaces of the separator. PVDF's excellent mechanical, chemical, and electrochemical qualities make it to be among the most significant and widely used binders. Furthermore, PVDF may have an impact on the electrode's mechanical characteristics and cathode active compounds' electrochemical performance [17]. CMC is a commonly used binder for the anode in LIBs, which is a combination of cellulose structure with carboxymethyl groups. CMC has a strong shear-thinning property, allowing for the adjustment of slurry rheology [18]. Discharging typically involves electrolytic processes to neutralize residual charge in batteries, ensuring safety during further processing. After discharging, thermal pretreatment is applied to break down organic binders through pyrolysis. Pyrolysis involves the use of high temperatures in the process. The thermal separation at elevated temperatures breaks the bond between the binder and the positive active material through thermal decomposition [19]. Conducted at 400°C to 600°C, pyrolysis effectively decomposes the binders and recovers valuable cathode materials, such as nickel, cobalt, and aluminum [20].

However, despite progress in recycling methods, the potential of flotation, particularly in conjunction with pretreatment processes like pyrolysis, remains underexplored. Key parameters such as collector concentration and pH, which significantly influence recovery efficiency, have yet to be fully optimized. Addressing this gap offers the opportunity to refine flotation methods, making the recovery of valuable materials from LIBs more efficient and sustainable.

This study aims to evaluate the effects of pyrolysis pretreatment on recovery efficiency, investigate how variations in collector concentration and pH impact the recovery of anode materials, and identify optimal flotation conditions for separating anode and cathode components. These efforts seek to enhance the sustainability and efficiency of LIBs recycling processes.

Method

In this study, spent lithium-ion batteries (LIBs) used for the flotation process were obtained from VIAR Motor Indonesia. These batteries comprised cathode material (NMC: $LiNi_xCo_yMn_zO_2$) and anode material (graphite). To enhance recovery efficiency, the LIBs underwent a series of pretreatment steps prior to the flotation process.

The pretreatment began with discharging the remaining power in the LIBs using an electrolytic discharge method with a 0.1 M Na^+ solution. Following discharging, the LIBs were dismantled using a grinding machine to separate the anode and cathode sheets. To isolate the anode material from the copper sheet, the anode was stirred in reverse osmosis (RO) water at 1600 rpm for 30 minutes. For the cathode material, separation from the aluminium sheet involved stirring in a 0.2 M H_2SO_4 solution at the same speed for 1 hour.

Prior to pyrolysis, the contact angle was measured using the sessile drop method, in which 3 microliters of de-ionized water were placed on the surface of a composite disc. The droplet was then captured with a digital camera and processed and optimized using image analysis software. Pyrolysis was conducted using a muffle furnace (Figure 1a) at a temperature of 500°C for 2 hours. The pyrolysis products were analyzed using Raman spectroscopy, which gives information about the vibrational energy levels of molecules and helps identify their chemical structure.

The flotation tests were performed using a 104 mL flotation cell (Figure 1b) with a magnetic stirrer operating at 1200 rpm at ambient temperature (25°C). In each test, 4.16 g of black mass was weighed and mixed with 102 mL of RO water to form a slurry. The slurry was conditioned for 2 minutes, after which pH modifiers were added, followed by a collector. Stirring continued for 3 additional minutes, and froth containing the floated materials was manually scraped off over another 3 minutes. The resulting concentrates and tailings from the flotation process were filtered, dried, and weighed to determine the recovery efficiency.

The formula used to calculate the recovery is as follows:

1. Recovery

$$R = \frac{C\,.c}{M\,.m}$$

where M is the mass of the sample in the feed = 4.16 g, m is the fraction of the component in the feed, C is the total mass of the concentrate, and c is the fraction of the component in the concentrate.

2. Selectivity

$$S = \frac{cA\,.\;RA}{cK\,.\,RK}$$

where cA is the fraction of the anode in the concentrate, RA is the anode recovery, cK is the fraction of the cathode in the concentrate, and RK is the cathode recovery.

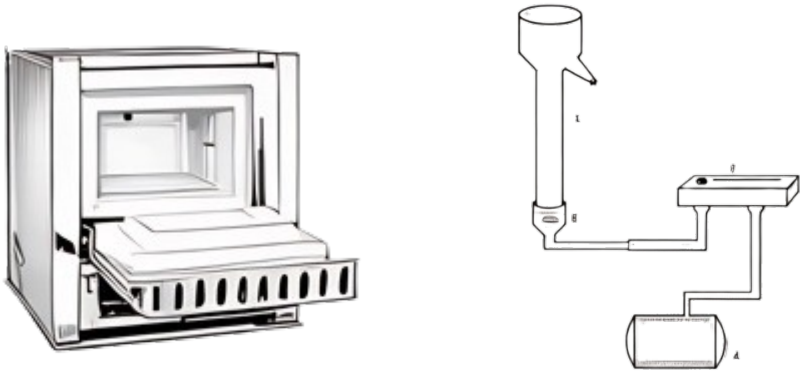

Figure 1. a. Muffle Furnace for Pyrolysis Pretreatment (left) and b. The Flotation Equipment Circuit 1. Flotation Column; 2. Magnetic Stirrer; 3. Flow Meter; 4. Compressor (right)

Results and Discussion

Results

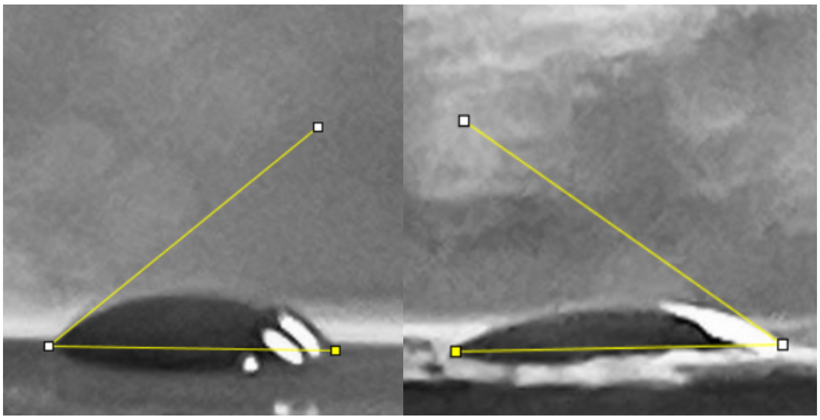

Figure 2. Result of contact angle measurement for cathode (left) and anode (right)

As shown in Figure 2, the contact angle for the cathode is 40.094°, while for the anode, it is 34.352°. These relatively low contact angles indicate that both the anode and cathode exhibit hydrophilic properties, meaning they are more water-attracting than repelling. This hydrophilic behavior can primarily be attributed to the binders. PVDF effectively binds the cathodes and can change the surface properties of the cathode materials. Meanwhile, CMC may increase the hydrophilicity of the anode materials, complicating their separation from hydrophobic components like graphite, which, due to its polar functional groups, can form strong non-covalent interactions with them [21].

The Raman spectroscopy results presented in Figure 3 reveal the presence of cathode components, including nickel (Ni) and manganese (Mn), in the form of oxides, identifiable at a peak of 548.823 cm^{-1}. This is consistent with findings reported by [22], who also detected Ni and Mn at a peak of 530 cm-1. The anode material, graphite, was detected at peaks 1597.93 cm^{-1} and 1774.66 cm^{-1}, corresponding to the characteristic graphite peaks in the G-mode, which were also shown in studies (e.g., [23]) that found a peak at 1580 cm^{-1}. In addition, if PVDF binders were present, distinct peaks attributed to PVDF, such as -(CH2CF2)-n, would typically appear around 800 and 1400 cm^{-1} [24].

However, these peaks were absent in the results, indicating that after pyrolysis, the PVDF-related peaks, along with those associated with electrolyte components like C=O and O-C=O, were effectively removed. This confirms that the pyrolysis process successfully eliminated both the binders and electrolytes, thereby enhancing the purity of the recovered materials.

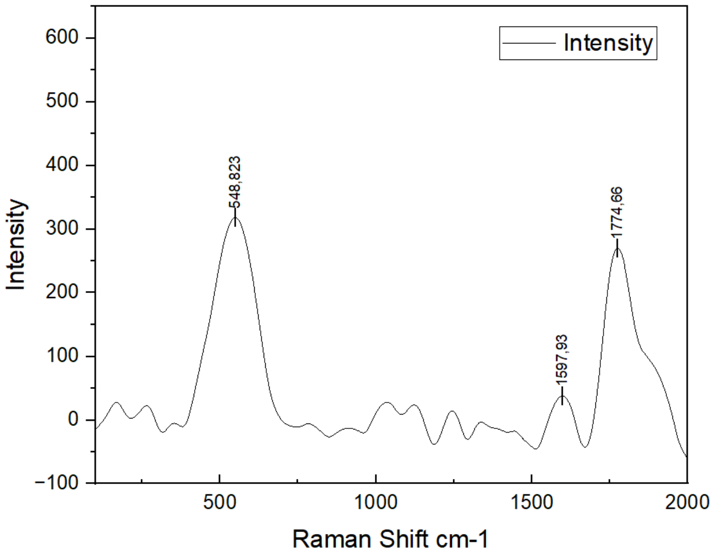

Figure 3. Raman spectra in the region 0–2000 cm^{-1} after pyrolysis treatment

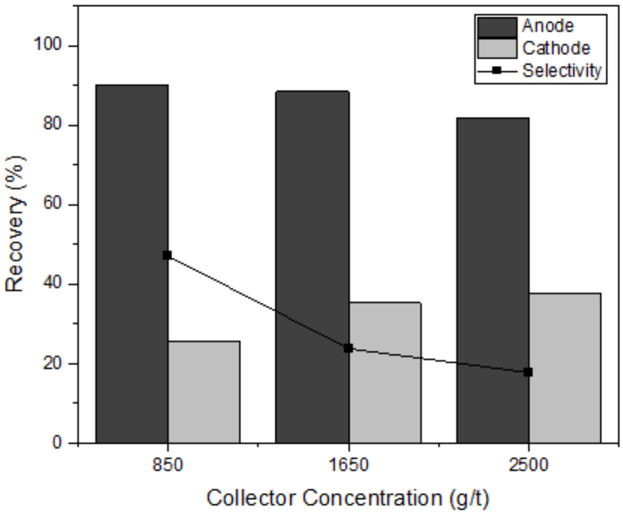

Figure 4. The relationship between collector concentration and recovery from froth flotation

The flotation experiment results with varying collector concentrations of 850, 1650, and 2500 g/ton revealed the following: At a collector concentration of 850 g/t, the anode recovery reached 90.05%, with a cathode recovery of 25.5% and a selectivity value of 47.0330. At a concentration of 1650 g/t, the anode recovery was 88.39%, the cathode recovery was 35.22%, and the selectivity decreased to 23.7486. Finally, at 2500 g/t, the anode recovery was 81.75%, with a cathode recovery of 37.75%, and the selectivity value further decreased to 17.6856. These results show a clear trend: as the collector concentration increases, the selectivity decreases, which suggests that a higher selectivity is associated with a more efficient separation process. The increase in collector concentration leads to greater adsorption of particles onto the collector, making both the anode and cathode materials more hydrophobic. This is reflected in the higher combined recovery values, but the lower selectivity indicates that undesirable materials are also being collected, which negatively impacts the separation quality.

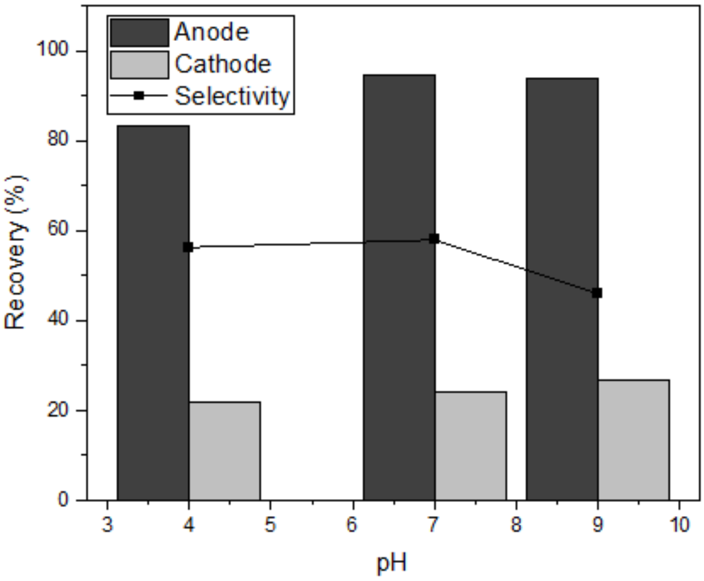

Figure 5. The relationship between pH and recovery from froth flotation

Figure 5 shows the results where variations in pH were applied during the flotation process under acidic, alkaline, and neutral conditions corresponding to pH values of 4, 9, and 7. At pH 4, the anode recovery was 83.54%, and the cathode recovery was 21.63%, with a selectivity of 56.2290. When the neutral pH was alkaline (9), the anode recovery increased to 94.61%, and the cathode recovery was 24.24%, yielding a selectivity of 57.9270. At neutral pH (7), the anode recovery was 93.77%, and the cathode recovery was 26.86%, with a selectivity of 45.9695.

Discussion

The results obtained in this study offer several important insights into the flotation process, specifically concerning the hydrophilic nature of cathode and anode materials, the impact of pyrolysis on binder and electrolyte removal, and the role of collector concentration and pH on recovery and selectivity.

The contact angle measurements indicate that both the anode and cathode exhibit hydrophilic properties. These results align with previous studies highlighting the hydrophilic nature of PVDF-bound electrodes. PVDF (polyvinylidene fluoride) is a polymer binder commonly used in LIBs. PVDF's adhesive and chemical stability properties are crucial for securing active materials to the electrodes, but they also contribute to the materials' reduced flotation potential, making them less likely to float in water [25]. PVDF significantly affects graphite, which exhibits hydrophobic properties by nature with contact angles as high as 80° [13]. The difference highlights the challenges of recovering materials such as anodes and cathodes through flotation processes, where hydrophobicity typically plays a key role in enhancing flotation efficiency.

The Raman spectroscopy results confirmed the presence of key components in the cathode, including nickel (Ni) and manganese (Mn) oxides, at peaks consistent with those identified in previous research [22]. The absence of binder related peaks after pyrolysis indicates that the binder was effectively removed, resulting in higher purity of the recovered materials. This study shows progress in achieving better results compared to those that did not undergo pyrolysis pretreatment, as seen in the study J. Yu et al (2017), which reported anode and cathode recovery rates of 10.91% and 13.68%, respectively, and in the study H. Shin et al (2020) , which reported a cathode recovery of 33.95%[26][27]. This is a critical observation, as the pyrolysis process not only aids in binder removal but also contributes to enhancing the efficiency of material recovery by improving the purity of the substances being floated.

The flotation results reveal a clear relationship between collector concentration, recovery, and selectivity. As the collector concentration increased, the recovery of both anode and cathode materials improved. This finding is similar to the study of S.Husin et al (2024); they found out that the collector also tends to form bonds with both anode and cathode particles. The primary flotation product is formed at a collector concentration of 150 g/t, with the froth phase containing 99.3% anode mass. However, at collector concentrations of 300 g/t and 400 g/t, the anode mass in the froth phase decreases due to the higher amount of cathode binding. However, this improvement came at the cost of selectivity. Higher collector concentrations promoted greater adsorption of particles, making both materials more hydrophobic. While this enhanced overall recovery, it also led to the inclusion of unwanted materials, reducing the separation efficiency. These findings align with previous studies of S.Husin. et al (2024) indicating that a balance between collector concentration and selectivity is crucial for achieving optimal flotation outcomes.

The pH variation results demonstrated that pH significantly influences the flotation process. At acidic conditions, the dissociation of H+ ions happens. They will react with metal oxides in the cathode to form salts, reducing the recovery of gangue minerals. Additionally, the oxidation of graphite at acidic pH further lowered the recovery of valuable minerals. In contrast, alkaline conditions neutralize H+ ions, reducing salt formation and promoting interactions between the collector and hydroxide ions, which enhances the flotation of gangue minerals [28]. At alkaline conditions, the recovery was slightly lower for both the anode and cathode (93.77% and 26.86%, respectively), with selectivity falling to 45.9695. These results are consistent with previous studies (e.g., [3][28]) that demonstrate the benefits of alkaline pH in flotation processes. The optimal results were obtained under neutral pH conditions. This suggests that while neutral pH provides a stable environment for flotation. This study, unlike the previously mentioned literatures, offers new insights particularly in the context of the trade-offs between recovery and selectivity, and highlights the complex dynamics between pH, collector concentration, and flotation efficiency.

Conclusion

This study considers various factors, including the hydrophilic properties of electrodes, pyrolysis treatment, collector concentration, and pH, that influence the flotation process in lithium-ion battery recycling. The results of this study highlight that the optimal flotation conditions for separating anode from cathode were achieved at a collector concentration of 850 g/t and a neutral pH of 7. Under these conditions, a high recovery of 94.61% for anode and 24.14% for cathode was obtained, with minimal changes in surface properties. This combination of collector concentration and neutral pH ensures effective recovery. Additionally, the pyrolysis treatment effectively eliminates the binder from both the anode and cathode materials, facilitating better separation during flotation. The findings confirm existing theories about the impact of hydrophobicity and pH on flotation efficiency while also offering new insights into the pyrolysis process and its role in material recovery. Future research should focus on optimizing flotation conditions, particularly by fine-tuning collector concentration and pH, and further exploring the interaction between different electrode materials to enhance both recovery and selectivity in lithium-ion battery recycling.

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Nomenclature

C_i = Total mass of the concentrate
c_i = Fraction component in the concentrate
CMC = Carboxymethyl Cellulose
LIB = Lithium-ion Battery
m_i = Fraction of the component in the concentrate
M_i = Mass of the sample in the feed
PVDF = Polyvinylidene Fluoride
R_i = Recovery
S = Selectivity

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