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Hydrometallurgical Recovery of Lithium Metal from Lithium Cobaltate Cathode Active Material of Spent LIBs for the Stationary Energy Storage Systems (SESSs) Applications

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Abstract

The growing use of stationary energy storage systems (SESS) to stabilize renewable energy grids is driving demand for lithium, a critical element in lithium-ion batteries (LIBs). As these batteries reach 'end-of-life', sustainable recovery of lithium has become essential to reduce reliance on primary resources and mitigate environmental risks. Hydrometallurgical recycling offers a promising solution due to its efficiency, scalability, and lower energy requirements compared to pyrometallurgical methods. This study investigates lithium recovery from spent LIBs through as-received mechanically pretreated cathode active materials (CAM), acid leaching, purification, and conversion into reusable compounds. Optimized leaching with $C_6H_8O_7{\cdot}H_2O$-H_2O_2 achieved high lithium extraction while limiting co-dissolution of NMC transition metals. Purification methods, including selective precipitation and solvent extraction, successfully isolated lithium, which was then converted into Lithium Cobaltate ($LiCoO_2$). Recovery rates around 90% highlight the viability of hydrometallurgy for large-scale recycling. Beyond resource conservation, this approach supports circular economy principles by enabling closed-loop recycling, reducing environmental hazards, and strengthening supply chains for SESS deployment. The findings emphasize hydrometallurgical recovery as a cornerstone of sustainable-energy infrastructure, ensuring reliable lithium availability to meet rising demand and advancing global clean-energy initiatives. The recovery of Lithium in this study is straightforward and eco-friendly.

Keywords
Lithium-ion battery (LiB); Citric acid ($C_6H_8O_7{\cdot}H_2O$); Hydrogen peroxide (H_2O_2); $LiCoO_2$, Stationary energy storage systems (SESSs).

1. Introduction
The rapid expansion of stationary energy storage systems, driven by the integration of renewable energy into power grids, has sharply increased global demand for lithium. Lithium-ion batteries, the dominant technology for SESS, are reaching end-of-life in growing volumes, creating both a resource challenge and an environmental burden. Current reliance on primary lithium mining is unsustainable due to finite reserves, high energy consumption, and ecological risks. Without efficient recycling pathways, valuable lithium remains locked in waste streams, while hazardous components pose risks to ecosystems and human health.

Hydrometallurgical recovery offers a promising solution, yet challenges remain in optimizing processes to achieve high lithium selectivity, minimize co-dissolution of transition metals, and produce battery-grade compounds suitable for reuse. The central problem is to develop scalable, cost-effective, and environmentally responsible methods for

extracting lithium from spent LIBs that can reliably supply SESS deployment. Addressing this problem is critical to securing resource availability, reducing environmental impacts, and enabling a circular economy for energy storage materials.

1.1 Objectives

This study aims to advance sustainable lithium recovery from spent lithium-ion batteries (LIBs), addressing the growing demand driven by stationary energy storage systems (SESS) for renewable energy stabilization. With LIBs reaching end-of-life, reliance on primary lithium resources poses environmental and supply chain challenges. Hydrometallurgical recycling is investigated as a viable alternative to pyrometallurgical methods, offering efficiency, scalability, and reduced energy consumption.

The research focuses on mechanically pretreated cathode active materials (CAM), followed by acid leaching, purification, and conversion into reusable compounds. Optimized leaching using citric acid ($C_6H_8O_7 \cdot H_2O$) and hydrogen peroxide achieved high lithium extraction while minimizing co-dissolution of transition metals from NMC cathodes. Subsequent purification through selective precipitation and solvent extraction successfully isolated lithium, which was then converted into Lithium Cobaltate ($LiCoO_2$). Recovery rates of approximately 90% demonstrate the feasibility of hydrometallurgical recycling for large-scale applications.

The most important achievement of this study is the demonstration of a straightforward, eco-friendly recovery process yielding high lithium recovery efficiency, thereby supporting circular economy principles. By enabling closed-loop recycling, this approach reduces environmental hazards, conserves critical resources, and strengthens supply chains essential for clean-energy technologies. Ultimately, the findings position hydrometallurgical recovery as a cornerstone of sustainable energy infrastructure, ensuring reliable lithium availability for future renewable energy deployment.

2. Literature Review

The demand for lithium-ion batteries (LIBs) has increased exponentially due to the rapid growth of the global electric vehicle (EVs) sector (Hasan, Togun et al. 2023). Stationary energy storage systems (SESS), such as those in video cameras, computers, cell phones, and other portable electronics, also utilize lithium-ion batteries (LIBs) for their operations. Due to their updated functionality and lower cost, the use of LIBs, which are made up of crust, electrodes (typically copper foil for negative electrodes and aluminum foil for positive electrodes), electrolyte, and polymer, has increased dramatically in recent years (Contestabile, Panero et al. 2001). According to EV industry estimates, more than 2 million tons of end-of-life power batteries will be produced worldwide by 2030, with over 60% of these batteries composed of metals like lithium, cobalt, nickel, and manganese. Because metallic lithium is explosive, spent lithium primary batteries cannot be disposed of securely unless they are properly removed. Heavy, valuable metals like cobalt and lithium must be recycled in LIBs that use lithium cobalt oxide as the cathodic active material (Anamu, Ayodele et al. 2023).

Lithium-ion batteries (LIBs) account for the majority of batteries used in electric cars; they are also widely used in SESSs due to their high energy and power densities (Armand, Grugeon et al. 2025). High voltage, high energy density, low self-discharge rate, and a broad working temperature range are all benefits of LIBs (Khan, Maddipatla et al. 2026). Lithium and cobalt, which are utilized in the production of cathode materials for LIBs, remain scarce commodities notwithstanding these worries (Fan, Li et al. 2020). The shortage of lithium metal is basically the main justification for the recycling of LIBs (El Mounafia, Aannir et al. 2025). Although lithium is the most common metal used in battery manufacturing, its supplies are limited worldwide. Due to its lack of native lithium resources, South Korea is fully dependent on Lithium imports to produce batteries. However, as the battery industry expands, demand for lithium intensifies, making it more challenging to meet demand solely through imports. Thus, recovering lithium and other vital metals by reutilizing LIBs for battery manufacturing is crucial for maintaining a stable supply of raw materials and avoiding environmental contamination (Jefferson, Rana et al. 2026).

The predominant commercial method, hydrometallurgy, achieves higher recovery of Lithium metal, relative to the pyrometallurgical process, and high alloy recovery rates (>90% for Ni, Co, and Mn) by selectively dissolving electrode-active materials in acidic conditions, followed by solvent extraction, purification, and precipitation (Kallitsis, Korre et al. 2022, Li, Luo et al. 2024). Its benefits include reduced energy intensity and improved product purity. Rezaei, M. et al. report that for Ni, Co, and Mn, standard procedures like the sulfuric acid-reductant system (H_2SO_4/H_2O_2) produce > 90% leaching efficiency at 80 °C (Rezaei, Nekahi et al. 2025). Currently, cobalt, lithium,

and other components are separated or recovered from wasted LIBs using two fundamental groups of recycling processes: chemical and physical (Kilavuz 2025). The recycling technique was primarily used to recover and separate valuable metals. Numerous studies have also examined the re-synthesis of electrode materials or the synthesis of other reactive materials from wasted LIBs. By precipitating cobalt as cobalt oxalate, a novel hydrometallurgical method was put forth to extract crystalline cobalt oxide from wasted LIBs (Van Asselt, Khokhar et al. 2026). To extract important metals from wasted LIBs, a novel combined recovery method that combines chemical precipitation, solvent extraction, and reductive leaching has been suggested (Bao, Wang et al. 2026). Because electrochemical technology uses less energy and requires fewer, safer recycling procedures, it is utilized in the recycling process. Etoile-Rebatt technology was used to recover and restore the layered $LiCoO_2$ phase in a single synthetic step in a new method for recycling LIBs (Rouhi 2026). The recovered $LiCoO_2$ showed a high level of electrochemical activity. According to research, cobalt oxide was recovered by a dehydration process after cobalt ions were removed from waste $LiCoO_2$ using a nitric acid leaching solution and potentiostatically changed into hydroxide on a titanium electrode (Sun, Wang et al. 2025).

As a cathode, lithium cobalt oxide ($LiCoO_2$) is an active substance that is difficult to dissolve in typical leaching agents. In order to recover all of the cobalt or manganese via electrochemical, precipitation, or solvent extraction methods, H_2O_2 is typically added (Shekarian, Rezaee et al. 2025). At 100°C, lithium can be recovered as a carbonate (Li_2CO_3) or with cobalt as Lithium Cobaltate ($LiCoO_2$) (Zhang, Wang et al. 2026). More than 99% of the lithium and cobalt may be recovered by using a strong acid solution as a leachate. Nevertheless, leaching releases Cl_2, SO_3, and NOx, and the acid that is left over after leaching poses a risk to the environment. An eco-friendly method for recovering metal from wasted LIBs has been presented by Saeki (Liu, Li et al. 2025). To create Cobalt and Lithium chlorides mechanochemically, $LiCoO_2$ was co-ground with PVC in a planetary ball mill. Consequently, approximately 100% Li and over 90% Co were recovered. Citric acid is an organic acid and can be dissolved in water easily (Vasconcelos, Gatti et al. 2025). It is frequently utilized in the manufacturing sector as a raw material. The recovery of Pb and PbO from scrap battery paste using citric acid as a reagent in aqueous media was investigated by Patade et al. (Patade, Sharma et al. 2025). Metals can be recovered from sewage sludge using ($C_6H_8O_7$). H_2O, a naturally occurring acid that is biodegradable. Because citric acid breaks down readily in both aerobic and anaerobic environments, treating these waste solutions is simple (Das, Khamaru et al. 2025). For a later leach, the leftover citric acid can be recycled and utilized again.

In this study, we aimed to develop a new hydrometallurgical process that uses an environmentally friendly acid for leaching to recover Lithium from $LiCoO_2$ of cathode active material of spent LIBs, and investigate the reaction conditions using citric acid and hydrogen peroxide ($C_6H_8O_7 \cdot H_2O$ and H_2O_2). A recycling process for spent LIBs has been developed using a combination of chemical precipitation, acid leaching research, and ultrasonic washing. This method can recycle LIBs at an industrial scale and has the benefit of using less energy and requiring fewer recycling stages.

2. Experimental

2.1. Materials, reagents, and characterization.

The research's input materials were provided by AST Recycling, a battery recycler in Johannesburg, South Africa, where old LIBs were pretreated by disassembly, sifting, and grinding before being used in the experiment. Using a LA-960 (HORIBA) Laser Particle Size Analyzer, the mean particles and specific surface area of the cathode active material (CAM), the cathode black mass, were found to be 32.6µm and 4.2m^2/g, respectively. Following the mechanical pre-treatment procedure, the raw material was chemically analyzed using XGT-9000SL X-ray Fluorescence Spectroscopy (XRF) to identify the metal elements present. Additionally, a carbon-sulfur analyzer (EMIA-820V, Horiba, Japan) was used to assess the total carbon content. Table 1 displays the chemical makeup of the spent LIB materials. Li, Ni, Co, and Mn were the primary metals found in the spent LIB materials, according to Table 1's chemical composition (Figure 1, Table 1).

Table 1. Chemical composition of the as-received mechanically pretreated CAM materials used in the study.

Elements	Li	Co	Ni	Mn	Fe	Al	Cu	C
Wt.%	17.07	41.17	26.54	25.97	0.05	0.45	0.015	4.32

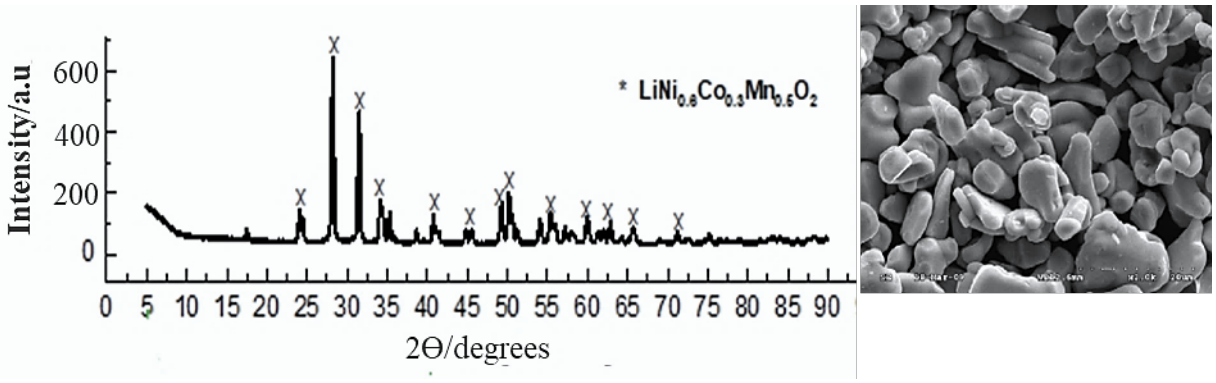

Figure 1. (**a**) X-ray diffraction patterns, and (**b**) Scanning Electron Microscope micrograph of treated LIB materials.

The phase composition of the as-received pretreated spent LIBs (cathode active materials) was determined by XRD characterization using XRD PANalytical X'Pert Empyrean. The microstructural evolution was determined using a scanning electron microscope equipped with energy-dispersive spectroscopy (JEOL JSM 7600 F), as illustrated in Fig. 1. It shows that the main phases of lithium, cobalt, nickel, and manganese are $LiNi_{0.6}Co_{0.3}Mn_{0.6}O_2$, which has a regular spherical morphology.

2.2 Experimental Procedures

For this investigation, the pretreatment plant's as-received cathode black mass—also known as cathode active materials—that had been manually crushed, ground, and sieved from the wasted LIBs was utilized. Fig. 2 depicts the schematic algorithmic pretreatment process that separates the materials in spent LIB, including metals, polymers, electrodes, and crust materials, as well as a cathode and an anode (Figure 2).

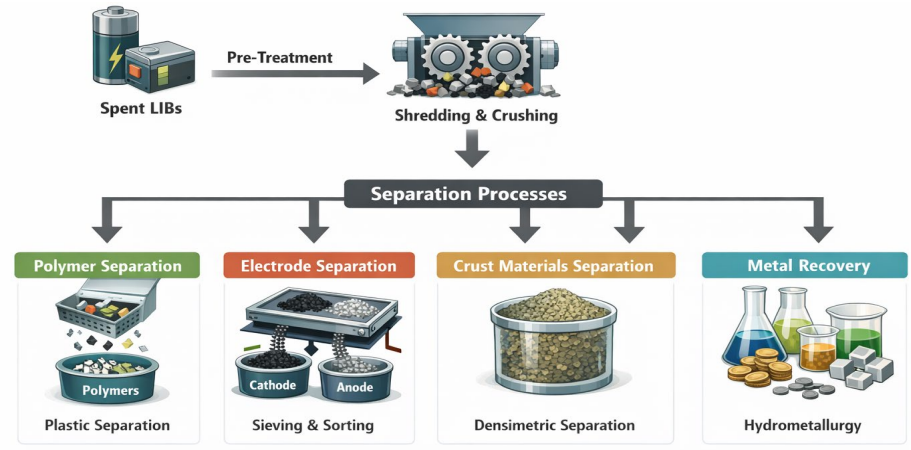

Figure 2. Process diagram for the recovery of cathode active materials from spent LIBs recycling.

In **Figure 3**, the screen underflow materials were processed in an ultrasonic washing container with agitation at an electric power of 90W and an ultrasonic frequency of 45 Hz, while the spent LIBs were placed in the crusher. The procedure was designed to separate carbon powder from the Cu foils and $LiCoO_2$ from the anode material and Al foils. The effects of the aperture's size, temperature, and ultrasonic washing duration were investigated in this step. The screen underflow materials, known as recovered electrodes, were then chosen from the previously described washed materials using a 2.5 mm aperture screen. The recovered electrodes were dissolved in hydrochloric acid after the sifting, and the effects of temperature, time, and pH^+ concentration on leaching efficiency were investigated. Ultimately, the solution's contaminants were eliminated, and Lithium cobalt oxide was recovered using a chemical precipitation process, which also assessed the impact of pH. To facilitate facile leaching, the lithium cobalt oxide was milled for two hours using a planetary ball mill. Greater leaching efficiency and improved dissolving are the results of smaller particle sizes.

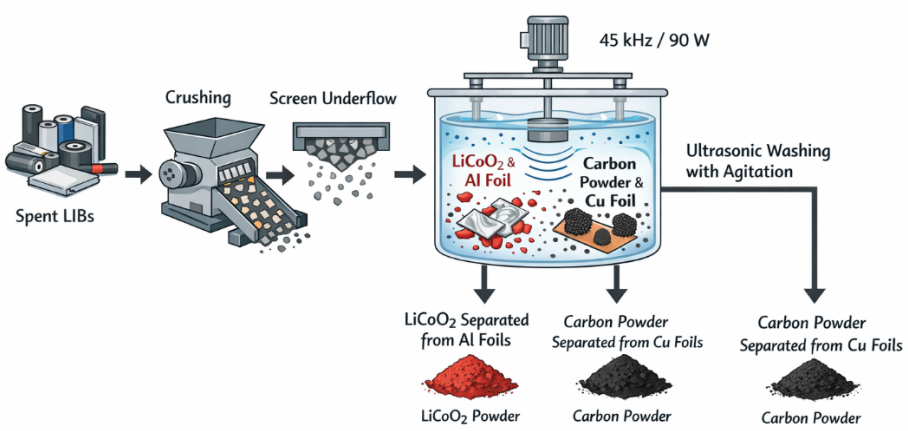

Figure 3. Pretreatment process for spent lithium-ion batteries (LIBs).

2.3. Metal leaching of $LiCoO_2$

The reactor, a plastic container, was filled with a predetermined quantity of waste $LiCoO_2$ powder and H_2O_2 solution. The reactor chamber was agitated to ensure uniformity. The material was ground up and sent for multi-element ICP-OES and XRF investigations using a temperature control facility. The reactor was equipped with a thermometer for precise temperature readings at regular intervals, a vapor condenser to minimize water loss through evaporation, and a magnetic stirrer. An electric mantle with temperature control provided the heating, and the reflux condenser prevented vapor loss at high temperatures. After adding a predetermined volume and strength of citric acid, the reactor was left to establish thermal equilibrium. The reactor was filled with a predetermined amount of waste $LiCoO_2$ powder and H_2O_2 solution, and stirred by the magnetic stirrer. Leaching tests were conducted under a variety of settings, including varying the concentration of citric acid, the S/L ratio, the temperature, and the H_2O_2 concentration, in order to determine the ideal parameters. Samples were collected at prearranged intervals throughout the leaching experiment. A pink solution and a black residue were the results of filtering and water washing. New samples of leached solids were treated with the leftover acid solution. To investigate their leaching behavior toward cobalt and lithium in the cathodic material, other organic acids, such as acetic acid and malic acid, were selected as candidates. For safe operation, safety glasses, gloves, and gas masks were used at every stage of the experiment (Figure 4).

Figure 4. The leaching apparatus: (A) Leaching kinetics set-up, including the system's *temperature, time, and pH control*; (B) *An experimental set-up of a typical duration monitor of the leaching conditions.*

3. Results and discussion

3.1. Characterization of powdered lithium cobalt oxide ($LiCoO_2$) in wasted LIBs

The hydrometallurgical method previously mentioned was used to calcine the cathode black mass of the spent LIBs, which was delivered 'as received'. The metal content of the samples from the active components is displayed in **Table 1**. The active ingredients in this recycling process are metal values, which are derived from the batteries' interior, including cobalt, lithium, manganese, and nickel. High concentrations of Ni and Mn were found in the cathode

according to XRF analysis; this is because doping and surface modification were employed to boost the LIBs' capacity, which precipitates these metals. As shown in Table 1, 17.07% of Li and 41.17% Co were found in the active materials to be high according to environmental standards, so LIBs need to be recycled. Li easily dissolves into the liquid solution during leaching, enabling high-purity separation, whereas other metals frequently stay in the solid phase or are sorted out afterwards.

An X-ray diffractometer was used to analyze the amount of $LiCoO_2$ in the cathode following dissolution and calcination. The results of earlier research on Li recovery from cathode chemicals were confirmed by XRD analysis, which clearly recognized the crystalline $LiCoO_2$ phase with some residues of carbon phase (**Fig. 4**). The cathodic active materials' XRD patterns are displayed in Fig. 4 before calcination and Fig. 4 following calcination at 350 °C. According to X-ray diffraction results, the spent LIBs' cathode composition was determined to be $LiCoO_2$ and C. XRD data showed that the cathodic material contained $LiCoO_2$ and a small trace of Co_3O_4, which is thought to have originated from a transformation of the active material ($LiCoO_2$)(Freitas and Garcia 2007), present in the calcined dust; however, calcination at 350 ◦C burned off the carbon and binder.

Scanning electron micrographs (SEM) of $LiCoO_2$ powders from commercial $LiCoO_2$ powder and the cathode of spent LIBs are displayed in Fig. 4. When compared to commercial $LiCoO_2$ powder, $LiCoO_2$ powders that have lost their effectiveness have uneven morphologies and appear to contain larger agglomerates. The cycles of charging and discharging produced a homogeneity in particle size. In contrast, the commercial powder is made up of smoother, more rounded particles. Therefore, unless they are recovered and reused, $LiCoO_2$ powders derived from used LIBs cannot be employed directly as an active material in the cathode (Figure 5).

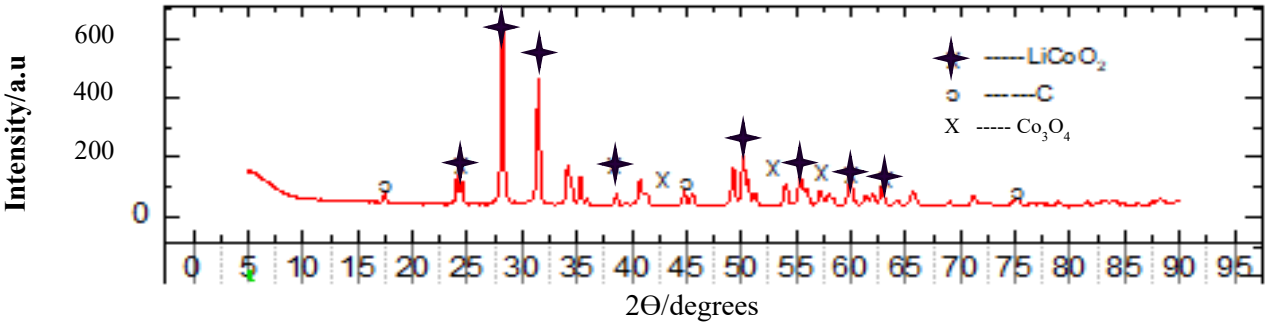

Figure 5. XRD patterns of the commercial $LiCoO_2$ sample during a calcination process at 350 °C for 5h.

3.2 Leaching of $LiCoO_2$ waste

Figure 6 depicts the potential reaction products of citric acid, a typical organic acid. One $C_6H_8O_7$ molecule has three carboxyl groups, and it is theoretically possible to make three mol. H+ when one mol. of citric acid dissociates in distilled water. Actually, some of the H+ is not released into the solution. The expression below is the dissociation process of citric acid:

$$H_3C_6H_8O_7 = H_2\,C_6H_8O_7^{-} + H^{+}, \qquad K_{\alpha1} = 7.4 \times 10^{-4} \quad \text{---------- (eq.1)}$$

$$H_2C_6H_8O_7^{-} = H\,C_6H_8O_7^{2-} + H^{+}, \qquad K_{\alpha2} = 1.7 \times 10^{-5} \quad \text{---------- (eq.2)}$$

$$HC_6H_8O_7^{2-} = C_6H_8O_7^{3-} + H^{+}, \qquad K_{\alpha3} = 4.0 \times 10^{-7} \quad \text{---------- (eq.3)}$$

Using citric acid as a leachate, waste $LiCoO_2$ can be leached in three stages. The leaching reaction between a $C_6H_8O_7 \cdot (H_2O)$ solution and waste $LiCoO_2$ can be shown as follows:

$$6H_3C_6H_8O_7(aq) + 2LiCoO_2(s) + H_2O_2(aq) = 2Li^{+}(aq) + 6H_2C_6H_8O_7^{-}(aq) + 2Co^{2+}(aq) + 4H_2O + O_2(g) \quad \text{---------- (eq.4)}$$

$$6H_2C_6H_8O_7(aq) + 2LiCoO_2(s) + H_2O_2(aq) = 2Li^{+}(aq) + 2Co^{2+}(aq) + 6HC_6H_8O_7^{2-}(aq) + 4H_2O + O_2(g) \quad \text{---------- (eq.5)}$$

$$6HC_6H_8O_7{}^{2-}(aq) + 2LiCoO2(s) + H_2O_2(aq) = 2Li^+(aq) + 2Co^{2+}(aq) + 6C_6H_8O_7{}^{3-}(aq) + 4H_2O + O_2(g) \quad \text{(eq.6)}$$

A study to increase the leaching efficiency of cobalt by adding the reducing H_2O_2 solution during the acid leaching of waste $LiCoO_2$ is extended for recommendation in this study because it can be predicted from Eqs. (4) to (6) that the addition of a reductant can facilitate the forward reaction and for the recovery of cobalt, with evidence of the reactant reduction (Swain, Jeong et al. 2007). However, the main focus of this work and its associated processes is the recovery of lithium active material. Cobalt and lithium were leached as $Co(C_6H_8O_7)_2$, $Li(C_6H_8O_7)$, Co^{2+}, and Li^+, respectively, during the leaching reaction. The majority of the $LiCoO_2$ was dissolved during the leaching reaction, according to XRD patterns. Because Co_3O_4 does not entirely dissolve in citric acid, the residues were C and Co_3O_4, which cannot be leached.

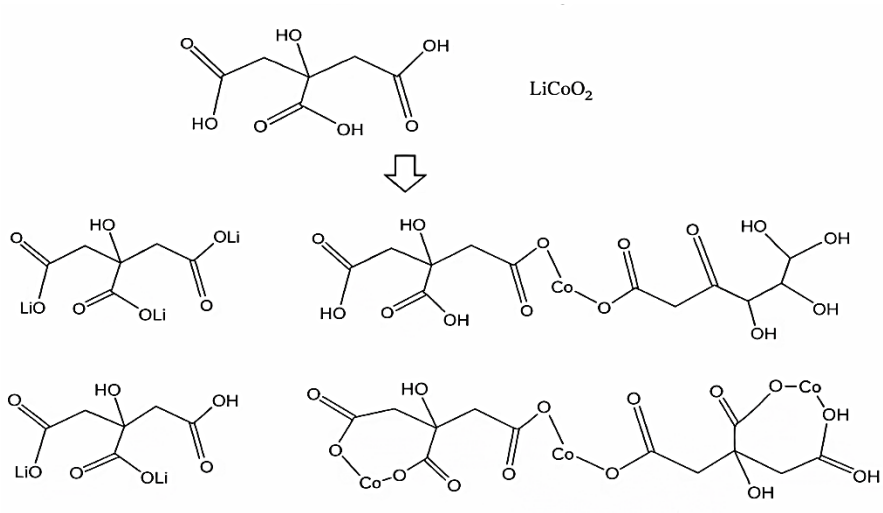

Figure 6. Citric Acid-Induced Surface Reactions of $LiCoO_2$

The hydrometallurgical phases of $LiCoO_2$ active material for lithium recovery are depicted in the diagram, which progresses from citric acid leaching to the creation of cobalt complexes and lithium citrate. It illustrates the separation of lithium for eventual recovery as metallic lithium or lithium salts by graphically mapping the conversion of the cathode material into soluble species.

3.2.1. The impact of time and temperature on leaching

Using 1.5M citric acid, the impact of temperature and duration on lithium leaching efficiency was investigated. The H_2O_2 concentration was 1 vol.%, and the S: L was kept at 50 g L^{-1} throughout the leaching process. The findings are displayed in **Figure 7**, which shows that the recovery of lithium is closely correlated with the overall rise in temperature. Put otherwise, as the temperature rose, so did the metal leaching efficiency.

Figure 7 shows that temperature and time had a substantial impact on the metal leaching efficiency.
The leaching efficiency of the metals was significantly improved by raising the temperature, and this is because citric acid undergoes an endothermic reaction during the dissociation process, and as the temperature rises, more H^+ is available in the solutions. The data unequivocally demonstrate that lithium dissolves more quickly at higher temperatures, with efficiency plateauing after around 60 minutes. Lower temperatures (40–60 °C) result in slower kinetics and lower total recovery, whereas recovery approaches ~90% at 95 °C. The pH meter connected to the leaching kinetics confirmed that when the leaching efficiency for the Lithium and Cobalt yield increased, it also did for the leaching velocity of $LiCoO_2$. As a result of this, the citric acid begins to gradually evaporate from the solution as the temperature reaches 90 °C. The substantial correlation between thermal input and reaction time in hydrometallurgical leaching optimization is highlighted by this picture.

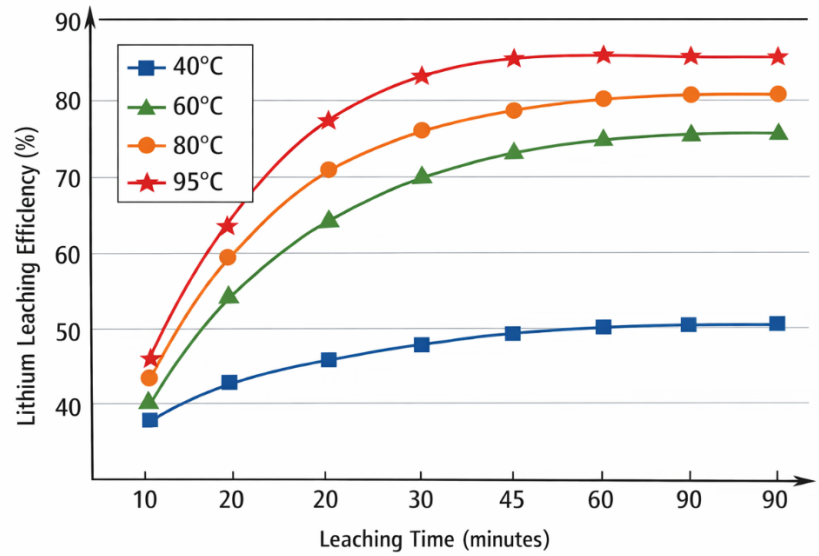

Figure 7. Leaching kinetics (temperature and time) influence the lithium recovery efficiency from $LiCoO_2$ leaching under the specified conditions (1.5M citric acid, H_2O_2 = 1 vol.%, S: L = 50 g/L, agitation speed = 350 rpm).

3.2.2. Dissolution of metals at solid/liquid ratios

Under experimental circumstances of 90 °C, 1.5M $C_6H_8O_7$, 40 min reaction time, and 1.0 vol.% H_2O_2, the impact of solid/liquid ratio (S: L) on the leaching efficiency for lithium was investigated from 50 to 100 g L^{-1}. A 95% of the lithium was leached at 50 g L^{-1}, which is thought to be appropriate for leaching the waste $LiCoO_2$. **Figure 8** shows that the leaching efficiencies of lithium improved as the S: L ratio dropped. Because the citric acid was insufficient to leach the $LiCoO_2$ powder, the leaching efficiency of Li was observed to be at 70% at an S: L of 50 g L^{-1}, and it stayed constant until the end of the leaching. For the remainder of the reaction, a plateau was observed. The ideal conditions for the leaching of lithium from waste $LiCoO_2$ were thus identified, taking into account the reduced chemical consumption and comparatively higher leaching efficiency.

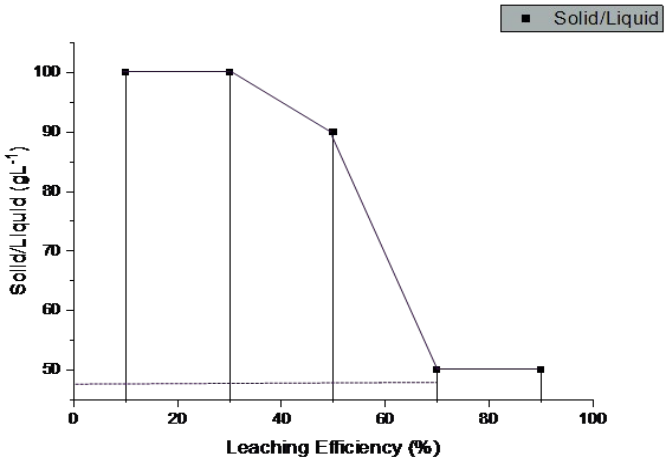

Figure 8. Impact of the solid/liquid ratio on the leaching of waste $LiCoO_2$ using 1.5M citric acid at 90 °C for 40 minutes (agitation speed = 350 rpm and H_2O_2 = 1 vol.%).

3.2.3. Impact of hydrogen peroxide and citric acid concentration on leaching

Figure 9 illustrates the dependence of citric acid concentration on lithium leaching and the impact of H_2O_2 concentration on Li leaching. A temperature of 90 °C, a concentration of 1 vol.% H_2O_2, and a leaching period of 40 minutes were used to vary the concentration of $C_6H_8O_7$ from 0.13M to 2.25M at an S: L of 50 g L^{-1}. Almost all of the $LiCoO_2$ dissolved at a citric acid concentration of 1.5 M. As the $C_6H_8O_7$ concentration rose from 0.3M to 2.25, the

leaching efficiency for lithium increased significantly. Using the same parameters as previously mentioned, the graphical permutation confirmed that lithium recovery increased with leaching under the same conditions, with a higher H_2O_2 dosage. We found that the hydrometallurgical route for recovering Li from $LiCoO_2$ in spent LIBs using hydrogen peroxide and citric acid to mobilize metals from the cathodic active material greatly enhanced the leaching efficiency of Li. The optimal conditions for the leaching of lithium from waste $LiCoO_2$ were identified based on the aforementioned investigation and by taking into account the lower energy consumption, lower chemical consumption, and comparatively superior leaching efficiency.

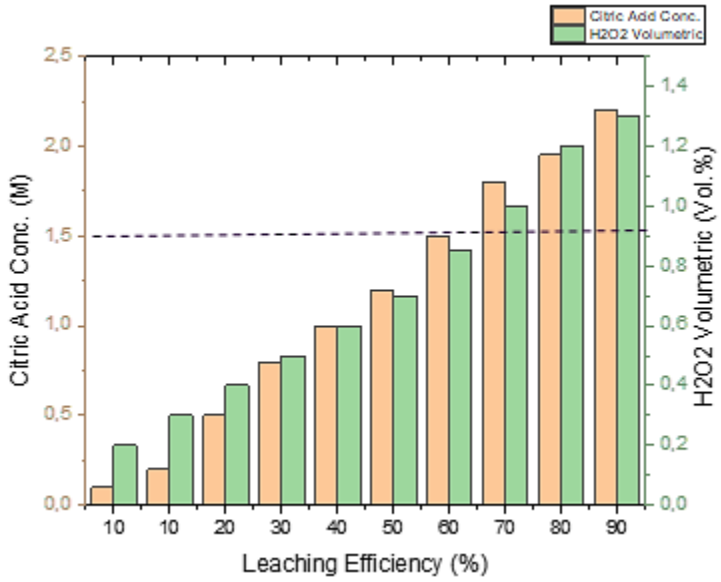

Figure 9. Graphical representation of the impact of citric acid and H_2O_2 concentrations on lithium leaching

4. Conclusion

The study demonstrates that citric acid ($C_6H_8O_7$) combined with hydrogen peroxide (H_2O_2) under optimized conditions is highly effective for leaching lithium from $LiCoO_2$ cathode material. The process is environmentally benign, avoiding strong mineral acids and minimizing secondary pollution.

The key highlights of this study are summarized as follows, viz.

- Citric acid acts as a chelating agent, forming soluble complexes with lithium and cobalt ions.
- Hydrogen peroxide enhances cobalt dissolution by reducing Co^{3+} to Co^{2+}, which is more soluble.
- Lithium recovery is often the final step, requiring high-purity separation from residual ions.
- Process optimization (e.g., pH control, reagent dosage) is critical to avoid co-precipitation losses.
- Environmental impact: Hydrometallurgy is more sustainable than pyrometallurgy, with lower CO_2 emissions and better metal selectivity.
- These conditions balance acid strength, oxidative potential, and thermal activation, making the process viable for sustainable lithium recovery.

5. Recommendation

Consequent upon the process adopted in the study, and the concentration effects on the leaching reductants, viz, the Citric Acid Concentration: 0.13 M to 2.25 M, H_2O_2 Concentration: Constant at 1 vol.%; Solid-to-Liquid Ratio: 50 g/L; Temperature: 90 °C, and the leaching time: 40 minutes, an optimal leaching is hypothesized to occur at ≥2.0 M citric acid and ≥1.2 vol.% H_2O_2 concentrations, achieving >95% efficiency.

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Biographies

Dr. Ogunmefun, Olakunle Anthony, is an industrial mechanical engineer with over two decades of experience in industry and over eight years in academia. He transitioned fully into academia in 2018, earning his MEng in Mechanical Engineering and later a Ph.D. in Metallurgical Engineering from the University of Johannesburg, South Africa. Currently, he is a Postdoctoral Research Fellow in the Department of Chemical, Metallurgical, and Materials Engineering, where he pursues advanced interdisciplinary research at the intersection of engineering, biomedical science, and materials, and Sustainable and Clean Energy. His expertise lies in nanoengineering, advanced materials, and material characterization, particularly through 3D printing and powder metallurgy. This specialization drives his pioneering work in developing optimized materials for biomedical applications, such as surgical tools and implants, designed to improve patient outcomes. Beyond biomedical research, Dr. Ogunmefun is deeply engaged in geo-metallurgical projects that address the pressing challenge of decarbonizing energy-intensive metallurgical processes—critical for producing metals essential to clean energy technologies like solar, wind, batteries, and electric vehicles.
The most important achievement of his career is his contribution to innovative advanced materials for biomedical applications, leveraging state-of-the-art techniques to enhance surgical efficiency and patient well-being. His work exemplifies the integration of engineering with broader societal goals, aligning with the UN's Sustainable Development Goal 7 (clean energy). With several well-cited publications on advanced materials and high-entropy alloy (HEA) development, Dr. Ogunmefun stands out as a researcher committed to both scientific advancement and global sustainability.

Dr. M.L. Teffo is the Director of the Institute for Nano-Engineering Research at Tshwane University of Technology (TUT), Pretoria, South Africa, and serves as a Senior Lecturer in the Department of Chemical, Metallurgical, and Materials Engineering. Her expertise lies in powder metallurgy and advanced manufacturing, with a strong focus on process optimization and materials characterization for advanced alloy development. Her research integrates microstructural engineering with applications across structural, energy, and biomedical fields, reflecting her passion for interdisciplinary innovation. She is particularly dedicated to bridging metallurgical engineering with biotechnology and medical applications, advancing materials that support both industrial performance and human health. The most important achievement of Dr. Teffo's career is her leadership in nano-engineering research and advanced alloy development, which has positioned her as a key contributor to sustainable and high-performance materials science. By combining powder metallurgy with cutting-edge characterization techniques, she has advanced the design of alloys tailored for diverse applications, from energy systems to biomedical devices. Beyond her research, Dr. Teffo is committed to academic leadership and mentorship. Through teaching and supervision, she equips emerging scientists and engineers with the analytical, experimental, and design skills necessary for impactful innovation. Her career embodies technical excellence, interdisciplinary vision, and a dedication to shaping the next generation of researchers.

Dr. M. Lucey Mavhungu is the Head of Department and Senior Lecturer in the Department of Chemical, Metallurgical, and Materials Engineering at Tshwane University of Technology (TUT), Pretoria, South Africa. She is a chemical engineer whose research focuses on wastewater treatment using nanomaterials, biodegradable polymers, and environmental remediation. Her academic leadership is complemented by an active research profile, with numerous publications in indexed journals that contribute to advancing sustainable engineering solutions. She is deeply committed to interdisciplinary innovation, particularly in developing environmentally friendly materials and processes that address pressing global challenges. The most important achievement of Dr. Mavhungu's career is her pioneering research in wastewater treatment and environmental remediation using nanomaterials and biodegradable polymers, which directly supports sustainability and clean technology initiatives. This work not only advances scientific knowledge but also provides practical solutions for improving water quality and reducing environmental impact. Equally dedicated to education, she plays a vital role in training and mentoring the next generation of scientists and engineers. Through teaching, supervision, and departmental leadership, she equips students with the analytical, experimental, and design skills necessary for impactful research and innovation. Her career reflects both technical excellence and a strong commitment to sustainability and capacity building in engineering education.

Prof. Emmanuel Sadiku is an Emeritus Professor of Polymer Technology at Tshwane University of Technology, Pretoria, South Africa. He earned his Ph.D. in Polymer Science from the University of Strathclyde, Glasgow, in 1986.

Over his distinguished career, he has held academic and research positions across Nigeria, Italy, Sweden, Germany, and South Africa, working under renowned mentors such as Prof. Giovanni Alfonso, Prof. Ulf Gedde, Prof. Gert Strobl, and Prof. Ron Sanderson. His expertise spans polymer physics, rheology, composites/nanocomposites, hydrogels, and biomedical and energy applications. Sadiku's most significant achievement lies in his prolific research output and global impact on polymer science. He has authored/co-authored over 700 peer-reviewed articles, more than 140 book chapters, and co-edited several volumes with leading publishers, including Elsevier, Springer, and the Royal Society of Chemistry. His innovative contributions are further evidenced by seven granted patents (with several more pending across multiple jurisdictions), covering advanced polymer technologies for biomedical and energy applications. Through decades of international collaboration and mentorship, Prof. Sadiku has established himself as a leading figure in polymer technology, advancing both theoretical understanding and practical applications. His extraordinary publication record and patent portfolio stand as his most important achievements, cementing his legacy as a pioneer in polymer science and a global contributor to materials innovation.

Dr. Sindile Francisca Mkatshwa is a registered geologist with SACNASP and a lecturer at the University of Johannesburg (Doornfontein campus). She brings extensive professional experience from hard-rock underground mining and exploration geology, where she served as a superintendent geologist. She earned her Ph.D. in Geology at the University of Johannesburg under the supervision of Professors Stephanus Viljoen, Albertus Smith, and Bradley Guy. Her doctoral research focused on geo-metallurgy, specifically uranium and gold-bearing ore characterization, aimed at improving recovery factors in mines on Johannesburg's West Rand. Her academic and research interests extend to process mineralogy and economic geology, with a strong emphasis on interdisciplinary approaches that bridge geological mining and metallurgical engineering. This integration reflects her commitment to advancing resource recovery efficiency and sustainable mining practices. The most important achievement in Dr. Mkatshwa's career is her specialized contribution to geo-metallurgy through uranium and gold ore characterization, which directly enhances recovery processes in mining operations. This work not only advances scientific understanding but also has significant practical implications for the mining industry. Equally dedicated to education, she is passionate about training the next generation of geologists and scientists, equipping them with analytical, experimental, and design skills essential for impactful research and innovation. Her career embodies both technical excellence and a commitment to knowledge transfer, positioning her as a vital contributor to the future of geology and metallurgical sciences.