



Hydrometallurgical Recovery of Cobalt from Spent Lithium-Ion Battery (LiCoO₂) using Organic Acids

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Abstract

The increasing use of portable electronic devices has led to an increase in lithium-ion battery (LiB) waste, which contains hazardous metals such as cobalt. It is noteworthy that cobalt accounts for up to 30% of the battery weight, significantly higher than its concentration in natural ores (<1%). Conventional recovery methods use strong inorganic acids (e.g., H₂SO₄, HCl), which are effective but produce hazardous wastewater and require complex treatment procedures. In this study, the cobalt extraction process was carried out using a hydrometallurgical method using organic acids, specifically ascorbic acid and citric acid. The extraction process was carried out with variations in organic acid concentrations ranging from 0.5 M to 2.5 M at temperatures of 55°C and 70°C. The extracted liquid was collected and analyzed for cobalt content using an Atomic Absorption Spectrophotometer (AAS). The cobalt leaching process using citric acid successfully dissolved 13.58–13.89 mg/L of cobalt. Meanwhile, from the leaching process with ascorbic acid, dissolved cobalt was obtained in the range of 10.96–12.87 mg/L. Based on the data obtained from the leaching process using organic acids, it can be seen that the best leaching process was obtained with citric acid at a concentration of 0.5 M at a temperature of 55°C, which produced 13,837 mg/L of dissolved cobalt. The extracted cobalt can be used for the synthesis of cobalt acetate, which is a fine chemical. However, not all cobalt dissolved in the leaching process can be precipitated to produce cobalt acetate. Of all the leaching solutions with both organic acids at different concentrations, only the solution from the leaching process with ascorbic acid at a concentration of 0.5 M was successfully precipitated and further synthesized into cobalt acetate with a yield of 17.42 wt%. In addition, FTIR analysis confirmed that the synthesized cobalt acetate was consistent with the reference cobalt(II) acetate tetrahydrate, with the difference in intensity indicating lower hydration in the synthesized sample.

Keywords: ascorbic acid, citric acid, electronic, extraction, leaching.

INTRODUCTION

The fast development of electronic devices has increased the need for lithium-ion batteries (LIBs) as a safe energy storage system with high energy capacity (Xiaodong & Ishchenko, 2024). This situation also contributes to an increase in electronic waste (e-waste) due to the relatively short lifespan of devices, typically 2 years for cell phones and 4–6 years for laptops (Jain et al., 2023). Most of these devices use LIBs, and approximately 80% of lithium-ion batteries currently used in portable electronic devices (Bae & Kim, 2021). It is reported that total global e-waste is estimated to reach 65.3 million tons by 2025, including 464,000 tons of LIB waste (Ye et al., 2024).

As the use of LIBs increases, a major challenge arises in managing used battery waste. This waste contains both valuable and hazardous metals, such as cobalt and lithium (Biswal & Balasubramanian, 2023). If not managed properly, these metals can contaminate soil and water, disrupt ecosystems, and pose health risks through accumulation in the food chain (Mrozik et al., 2021). Therefore, effective management of Li-ion battery waste is crucial to reduce environmental impact and recover the resource value contained within (Zhang et al., 2018).

LIBs consist of several main components, namely the cathode (about 35% of the total weight), anode (15–18%), electrolyte (11–12%), metal casing (25–30%), and plastic materials and other components (Heydarian et al., 2018; Horeh et al., 2016). Among these components, the cathode has the highest economic value because it contains strategic metals such as cobalt. Laptop batteries typically use a lithium cobalt oxide (LCO, LiCoO_2)-based cathode, which contains up to 30% cobalt and about 10% lithium by weight (Heydarian et al., 2018). The cobalt content in this battery is much higher than the cobalt content in natural ores, which is

usually less than 1% (Ma et al., 2022). Therefore, spent battery waste has the potential to become a source of high-value metal reserves while supporting more sustainable resource management.

Cobalt is a first-row transition metal with unique physical properties that make it crucial to technology and science, as well as a strategic element (Brugman et al., 2023). The cathode material in LiB batteries made from Lithium Cobalt Oxide (LCO) is a source of cobalt, which can be extracted and utilized as a precursor to various functional materials. One of the uses of cobalt from LCO waste is to convert it into cobalt(II) acetate, which is widely known as an important precursor in the synthesis of various cobalt-based catalysts and electrocatalysts (Tanwar et al., 2023).

A common method used to extract valuable metals from solid-liquid waste is a hydrometallurgical technique involving acid leaching, followed by extraction and separation of metals such as cobalt from the resulting solution (Parween et al., 2025). Cobalt recovery from battery waste can be carried out through leaching methods using strong inorganic acids, such as H_2SO_4 or HCl , due to their effectiveness in dissolving metals (Jayamuthunagai et al., 2025). However, this method produces hazardous acidic wastewater that can potentially pollute the environment if not properly treated. Furthermore, the use of strong acids requires additional treatment and strict safety procedures, increasing operational complexity and costs (Fan et al., 2025). Therefore, developing alternative, more environmentally friendly leaching methods is crucial. One solution currently being widely explored is leaching using weak organic acids, such as citric acid ($\text{C}_6\text{H}_8\text{O}_7$), which is considered both safer and more effective (Panggabean et al. 2023).

Organic acids such as ascorbic acid also

have the potential to be used as leaching agents. In the LiCoO_2 leaching process, cobalt is in the stable and insoluble oxidation state of Co^{3+} , requiring a reducing agent to increase its solubility (Bouguern et al., 2024). A study shows that environmentally friendly organic reducing agents, such as ascorbic acid, can convert insoluble Co^{3+} in LCO into soluble Co^{2+} , while $\text{C}_6\text{H}_8\text{O}_6$ is oxidized to $\text{C}_6\text{H}_6\text{O}_6$ (Paul et al., 2024). However, the use of both types of leaching agents still faces various challenges. Process efficiency is highly dependent on operational parameters such as the solid-to-liquid ratio, temperature, acid concentration, and reaction time (Demir, 2025).

Another challenge is processing leachate products into economically valuable forms, such as cobalt acetate ($\text{Co}(\text{CH}_3\text{COO})_2$), which can be reused in various industries. Developing effective processes not only reduces environmental impact but also increases the economic value of the extracted metal (Chandra et al., 2022). Cobalt acetate can be used as a precursor for various material syntheses, including the production of cobalt oxides, catalysts, magnetic materials, pigments, and active ingredients in the paint and ink industry (Vodyashkin et al., 2022). Cobalt acetate can be obtained from secondary cobalt present in the cathode of LiCoO_2 batteries. Cobalt in the solid cathode is extracted into Co^{2+} ions through hydrometallurgical principles, producing a stable cobalt solution. The cobalt ions in this solution are then reacted with

acetate to form cobalt acetate crystals of sufficient purity, making this solid compound an important precursor in the synthesis of cobalt-based materials (Ahmad et al., 2017).

This study aims to evaluate the effectiveness of leaching using weak organic acids, such as citric acid and ascorbic acid, for cobalt extraction from LiCoO_2 battery waste. The study was conducted by optimizing the extraction process by considering operational parameters such as solvent concentration, temperature, and leaching time. In addition, this study also focuses on developing a method to produce cobalt acetate as an economically valuable product that can be reused in various industrial applications. The results of this study were expected to increase the efficiency of the metal recycling process, supporting environmentally friendly principles, and helping to manage battery waste sustainably and reduce dependence on primary raw material sources.

METHOD

The research conducted comprises several stages, as illustrated in the flow chart in Figure 1. The materials used in this study include spent LiCoO_2 batteries, sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), acetic acid (CH_3COOH), citric acid ($\text{C}_6\text{H}_8\text{O}_7$), ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$), and standard reference of Cobalt (II) acetate tetrahydrate (AR/ACS CDH) All chemicals used were pure chemical grade ($\geq 99.2\%$).

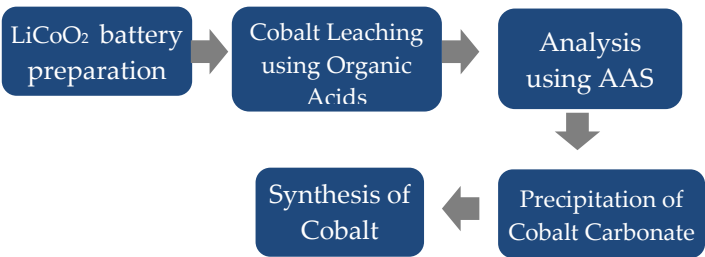


Figure 1. Research Stages of Cobalt Leaching from LCO Batteries and Cobalt Acetate Synthesis

Preparation of LiCoO_2 Batteries

In the initial stage, the battery was discharged before being disassembled for safety reasons. The discharge circuit used

consists of a series of light bulbs connected to the positive and negative poles of the battery. Battery voltage is measured using a digital multimeter in DC Voltage (DCV) mode. If the voltage is higher than 1.5 Volts, the discharge process is repeated with the light bulb circuit until the light dims or goes out, then measure the voltage again. Ensure the voltage is lower than 1.5 V to ensure the battery is safe to disassemble.

The next step is disassembling the battery. Carefully remove the outer layer of the battery using a knife on a stainless steel tray to ensure safety and prevent contamination. Next, cut the positive terminal using pliers to expose the top of the battery. Remove the battery cells from the casing, then separate the cathode foil (aluminium-LiCoO₂) from the anode foil (copper-graphite). The battery and cathode components used in this study are shown in Figure 2.

The cathode material was dried in an oven at 105°C for 24 hours. Then, the LiCoO₂ sheet was cooled at room temperature for 10 minutes, followed by storing it in a desiccator containing silica gel. The mass of the cathode was used to determine the volume of NaOH solution required using a liquid-to-solid ratio of 10:1 (V/wt). For 20 grams of cathode material, a volume of 10% NaOH of 200 mL was used. A total of 20 grams of cathode sheet obtained from ± 7 batteries were soaked in the NaOH solution until all parts were completely submerged. The reaction was run for 5 hours at room temperature until the formation of H₂ gas bubbles stopped, indicating the completion of the aluminum dissolution process. Then, the solution was separated from the cathode solid by slowly removing the remaining liquid. The solid was washed with distilled water several times until the pH of the filtrate was close to neutral, then dried at 60°C for 4 hours. The next stage was calcination, which was carried out by

calcining 50 grams of cathode powder at 500°C for 1 hour using a furnace. The calcined material was then ground with a porcelain mortar and sieved through a 100-mesh screen to obtain a uniform particle size. Storage can be done in a closed container to avoid air and moisture contamination.

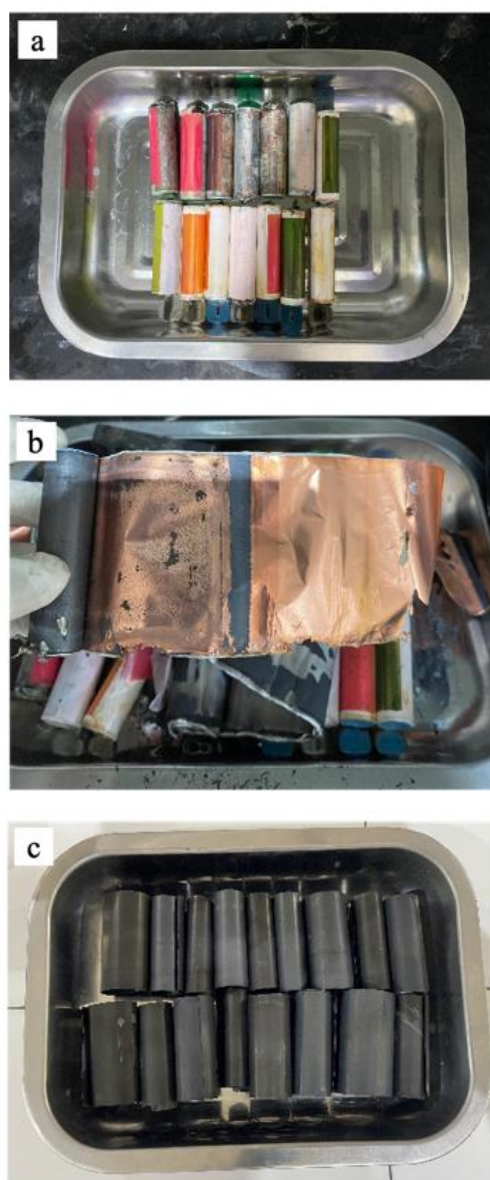


Figure 2. a) Spent Battery, b) Cathode Separation Process, and c) Cathode Component

Cobalt Leaching Process

The leaching process was carried out using weak organic acids, including ascorbic acid and citric acid. The concentration of the

organic acid solution varied from 0.5 M to 2.5 M, with extraction carried out at temperatures of 55°C and 70°C. The LiCoO_2 powder prepared for the leaching process used a solid/liquid ratio of 25 g/L. Therefore, for a 250 mL acid solution, 6.25 grams of LiCoO_2 powder were added. The leaching process was carried out in a glass beaker heated using a hot plate equipped with a stirrer. The stirring speed was set at 300 rpm to maintain a homogeneous mixture during the process. The extraction temperature was maintained at 55°C and 70°C according to the variations carried out. The leaching process was carried out for 30 minutes, then the mixture was cooled. Next, the solids were separated from the filtrate by filtration using filter paper.

Purification of Leaching Process

The next stage was the purification process of the leaching solution. This purification process was carried out by heating the leaching solution to 40°C with stirring at 300 rpm. A 33% NaOH solution was added slowly using a dropper until the pH of the leaching solution reached 6.3 with continuous pH monitoring. The stirring process was maintained for 1 hour at a constant temperature of 40°C to ensure optimal precipitation. After completion, the solution was left for 10 minutes to allow time for precipitation. Next, the solution was filtered with filter paper, and the filtrate was collected in a 250 mL beaker.

Cobalt Precipitation Using Na_2CO_3

After the cobalt-containing leachate was purified, the cobalt precipitation process was carried out by adding a 20% (wt/v) Na_2CO_3 solution. A total of 100 mL of the purified solution was placed in a 250 mL beaker and heated at 40°C with a stirring speed of 300 rpm. The 20% Na_2CO_3 solution was slowly added to

the leachate until the pH of the solution reached 8.2, with continuous pH monitoring. Maintain the stirring process for 45 minutes at a constant temperature of 40°C to ensure optimal precipitation. Then, the mixture was cooled for 10-15 minutes to allow for complete precipitation. The mixture was filtered and the CoCO_3 precipitate was separated from the filtrate. The resulting CoCO_3 precipitate was washed with distilled water until a filtrate with a neutral pH was obtained, then re-filtered. The obtained CoCO_3 precipitate was then dried in an oven at 80°C for 12 hours to obtain dry cobalt carbonate powder.

Transformation of Cobalt Carbonate (CoCO_3) into Cobalt Acetate ($\text{Co}(\text{CH}_3\text{COO})_2$)

The cobalt acetate synthesis process was carried out by reacting CoCO_3 with acetic acid (CH_3COOH). The volume of acetic acid required was based on a molar ratio of 2:1 to CoCO_3 . The acetic acid solution was used at a ratio of 1:30 with distilled water. The acetic acid solution is put into a 250 ml beaker, then heated to 70°C and stirred at 500 rpm. CoCO_3 powder was added gradually in 3 stages (mass ratio 1:2:3) with a 1-minute interval between stages while continuing to stir. The temperature and stirring were maintained for 30 minutes. The solution is cooled to room temperature, then filtered to separate the insoluble residue. The filtrate containing cobalt (II) acetate was collected in a 250 mL beaker. Evaporation was carried out by heating the beaker in a water bath at 70°C until the solution thickened. The solution was cooled until cobalt (II) acetate crystals formed. Then, the crystals formed were filtered and washed using distilled water. Next, the crystals were dried in an oven at 90°C for 2 hours until a dry cobalt (II) acetate product was obtained.

Cobalt Analysis Using Atomic Absorption

Spectroscopy (AAS)

Atomic Absorption Spectrophotometry (AAS) analysis was performed to determine the cobalt (Co) content dissolved in the leached solution. Measurements were carried out using an AAS instrument (Perkin Elmer PinAAcle 900F), equipped with a Perkin type hollow cathode lamp, with a limit of detection (LOD) of 0.002 ppm. The results, in the form of absorbance values at specific wavelengths, were then compared with a calibration curve to determine the cobalt concentration in the sample. This concentration value was used to assess the effectiveness of the leaching process in dissolving cobalt from LiCoO₂ battery waste. The cobalt concentration in the leaching solution was determined based on the standard curve shown in Figure 3.

$$y = 0.0765x + 0.00007 \quad (1)$$

The linear equation obtained from this curve is shown in Equation (1), where the variable y is the absorbance value obtained from the AAS measurement and the variable x is the dissolved cobalt concentration. The obtained standard curve had a coefficient of determination (R²) of 0.9993, indicating a very strong linear relationship between concentration and absorbance.

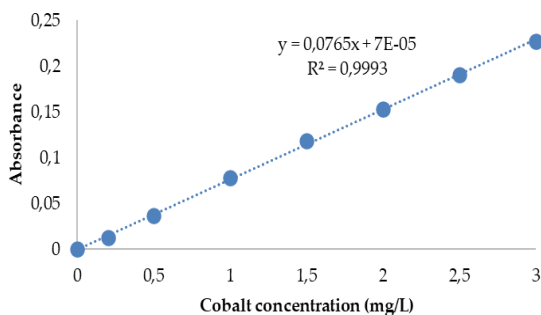


Figure 3. Standard Curve for Quantitative Measurement of Cobalt

RESULTS AND DISCUSSION

The effect of organic acids concentration on cobalt (Co) content

The effect of organic acid concentration

in the leaching process on the dissolved cobalt was observed at concentration variations of 0.5 - 2.5 M at temperatures of 55°C and 70°C. The results of the AAS analysis of cobalt content are shown in Figures 4. Overall, the comparison of leaching processes using ascorbic acid and citric acid showed that citric acid performed better across all observed concentration variations. Meanwhile, the leaching results for each type of organic acid observed showed different trends across these concentration variations

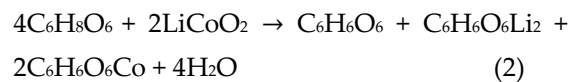
Measurement results showed that using higher concentrations of citric acid in the leaching process resulted in lower cobalt levels. The same trend was observed in the experiments conducted at temperatures of 55°C and 70°C. Theoretically, increasing the citric acid concentration should increase cobalt leaching because the number of H⁺ ions available to dissociate cobalt oxide and the number of citrate ions (Citrate³⁻) as a complexing agent increase (Oke et al., 2025). However, the AAS analysis showed a tendency for leached cobalt to decrease. The decrease in the cobalt levels observed in this study can be explained by the results of research conducted by Hussaini et al., (2024), which stated that dissolved cobalt levels increased until they reached optimum conditions at a citric acid concentration of 1.5 M (1007.2 ppm), then decreased at concentrations of 2.0 M (906.7 ppm) and 2.5 M (591.2 ppm) based on AAS analysis (Hussaini et al., 2024). This phenomenon shows that an increase in citric acid concentration is not always linear with an increase in dissolved cobalt. The decrease in cobalt content with increasing citric acid concentration is due to the characteristic of citric acid as a weak acid that only partially dissociates. At increasing concentrations, the addition of citric acid molecules is not always followed by a proportional increase in H⁺ ions,

so that the leaching effectiveness is limited. In addition, increasing citric acid concentration can cause limitations in ion movement in solution, which inhibits the diffusion of H^+ ions and citrate-cobalt complexes to the particle surface (Hussaini et al., 2024).

Although cobalt levels generally show a decreasing trend, the data show a temporary increase at a concentration of 2 M and then decrease at a concentration of 2.5 M. This increase is possibly related to changes in the equilibrium of cobalt-citrate complex formation in solution, where some of the formed complexes undergo partial dissociation, which contributes to the increase in measured dissolved cobalt. At higher citric acid concentrations (2.5 M), the cobalt-citrate complex tends to be more stable, so that the dissolved cobalt levels decrease again. The formation of complexes between Co^{2+} ions and citrate ligands is strongly influenced by the concentration ratio of citrate ligands, so that the distribution of cobalt in solution can change dynamically according to system conditions (Kublanovsky, 2023). Based on the results of the study, increasing citric acid concentrations in the range of 0.5–2.5 M did not show a significant increase in cobalt levels measured based on AAS analysis. Cobalt levels tended to decrease slowly as citric acid concentration increased, with a transient increase at a concentration of 2 M, possibly related to

changes in the equilibrium of cobalt–citrate complex formation in solution.

The results of the ascorbic acid leaching process that the extracted cobalt concentration increased significantly when the ascorbic acid concentration was increased from 0.5 M to a peak at 1.0 M. Leaching with a 1.0 M ascorbic acid concentration resulted in the highest dissolved cobalt concentration, approximately 12.87 mg/L at 55°C and 12.86 mg/L at 70°C. This phenomenon can be explained by the dual role of ascorbic acid in the leaching process. Ascorbic acid not only functions as a leaching agent (proton source, H^+) that dissolves metal oxides, but also as a strong reducing agent (D. Chen et al., 2020). The fundamental role of ascorbic acid is as a reducing agent, converting cobalt from the insoluble +3 oxidation state (Co^{3+}) to the soluble cobalt ion +2 (Co^{2+}) (Kime & Makgoale, 2016). The redox reactions follow the reaction mechanism as in Equation (2).



Increasing the concentration of ascorbic acid increases the collision rate between reducing molecules and the cathode particle surface (Gerold et al., 2024). A faster reaction rate means more cobalt transfers from the solid to the liquid phase per unit time, which is measured as a higher cobalt concentration.

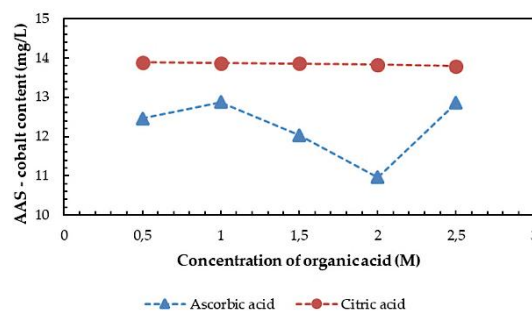


Figure 4. Cobalt Concentration from Leaching using Organic Acid at a Temperature of 55°C

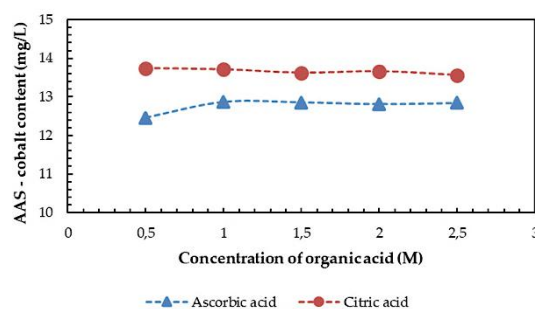


Figure 5. Cobalt Concentration from Leaching using Organic Acid at Temperature of 70°C

A significant phenomenon was observed when the ascorbic acid concentration was increased exceed the optimum point of 1.0 M, where cobalt leaching efficiency actually showed a clear decrease at concentrations of 1.5 M and 2.0 M. This decrease was due to the fact that ascorbic acid is a weak acid that cannot be fully ionized (Berg, 2015). The ability and amount of anion formation from weak electrolytes tend to follow a trend until the optimum condition is reached. The relationship between weak electrolytes and their conductivity is such that as the concentration increases, the conductivity will increase up to a certain level. Further increases in concentration will decrease the conductivity and thus the leaching efficiency (Nurqomariah & Fajaryanto, 2018). However, from this analysis, it can be concluded that the best performance occurred at a concentration of 1.0 M. Further increases in concentration are inefficient and reduce process performance, as the ascorbic acid cannot be fully ionized, thus reducing overall leaching efficiency.

The effect of leaching temperature on cobalt (Co) content

Temperature is one of the main parameters affecting the effectiveness of the cobalt leaching process from lithium-ion battery materials. A comparison of cobalt concentrations at 55°C and 70°C shows differences in leaching capacity at various

ascorbic acid concentrations. The effect of temperature changes on the yield of dissolved cobalt is shown in Figures 6

Figure 6 (a) shows that increasing the temperature from 55°C to 70°C increases the cobalt leaching results. At 70°C, the dissolved cobalt concentration tends to be more stable, ranging from 12–13 mg/L across all ascorbic acid concentrations. Conversely, at 55°C, the dissolved cobalt concentration exhibits greater fluctuations, including a decrease of approximately 10 mg/L at a 2M ascorbic acid concentration. This difference indicates that higher temperatures provide more effective conditions for the cobalt leaching process with ascorbic acid.

Theoretically, increasing the temperature will increase the kinetic energy of the molecules, thereby accelerating the rate of chemical reactions, including redox reactions and the complexation process between cobalt and ascorbic acid (Xu et al., 2021). Higher temperatures are known to accelerate the diffusion of metal ions from the solid to the solution phase and increase the ability of the solvent to break metal bonds from its matrix, so that at a temperature of 70°C, cobalt dissolves more easily and its concentration is more stable. These results are supported by research conducted by Li et al., in which cobalt leaching from LiCoO₂ (LCO) cathode material using ascorbic acid achieved the highest yield of 94.8% at a temperature of 70°C (Kime &

Makgoale, 2016).

At lower temperatures, the leaching process becomes less efficient. The reaction runs more slowly, and important redox intermediates may become unstable. Temperature strongly affects how fast metals dissolve and also changes the leaching mechanism. At higher temperatures ($>45^{\circ}\text{C}$), cobalt dissolution is mainly controlled by diffusion through a product layer. At lower temperatures, surface reactions dominate, which leads to slower kinetics. This explains why low temperatures limit ion release due to slower diffusion rates (Golmohammadzadeh et al., 2017). This behavior matches the curve, where the dissolved Co concentration decreases at several points at 55°C . A temperature of 70°C provides better conditions for cobalt leaching with ascorbic acid because it speeds up reactions, enhances the reduction process, and improves cobalt stability in solution.

The leaching results using citric acid (0.5–2.5 M) at 55°C and 70°C are shown in Figure 6 (b). The results indicate that cobalt dissolution at 70°C is slightly lower than at 55°C . The average cobalt concentration at 55°C is 13.85 mg/L, while at 70°C it is 13.66 mg/L, with a small difference of 1.4%. This suggests that increasing the temperature from 55°C to 70°C does not significantly increase cobalt dissolution, and a slight decreasing trend

begins to appear.

Based on the Arrhenius equation, increasing temperature generally increases the reaction rate because molecules more easily reach activation energy. However, within this narrow temperature range, the effect is not very significant because the process begins to be limited by surface diffusion kinetics (Wongnaree et al., 2024). In addition, cobalt loss due to co-precipitation becomes more significant at temperatures above 70°C (Stein et al., 2018). This indicates that 70°C is a transition point, where cobalt levels start to decrease slightly, although the effect is still small.

Synthesis of cobalt acetate

After the leaching process dissolved cobalt from the cathode, the next step was to recover it by precipitation. First, the dissolved cobalt was converted into cobalt carbonate, and then further processed into cobalt acetate. This compound is commonly used as a precursor and serves as a catalyst in organic synthesis, especially in industrial oxidation reactions (Asundi et al., 2023). The yield of cobalt acetate obtained from this synthesis step is shown in Table 1.

When using ascorbic acid at a concentration of 0.5 M, the yield of cobalt acetate was 17.42%. This shows that the precipitation process occurred successfully.

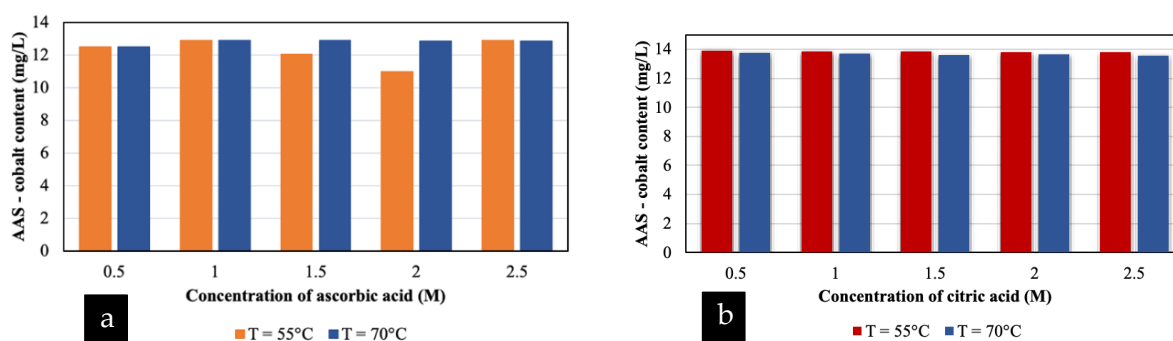
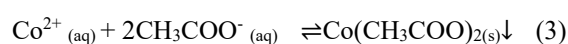


Figure 6. Cobalt concentration from leaching using (a) ascorbic acid, (b) citric acid with a concentration of 0.5 M – 2.5 M

Table 1. Yield of Cobalt Acetate Obtained from the Synthesis Process

Type of organic acid	Concentration	Yield of cobalt acetate from cathode precursor (wt%)	Yield of cobalt acetate from cobalt carbonate (wt%)
Ascorbic acid	0.5	17.42	56.6
	1.5	0	0
	2.5	0	0
Citric acid	0.5	0	0
	1.5	0	0
	2.5	0	0

In this reaction, the acetate ion (CH_3COO^-) acts as a ligand, meaning it donates an electron pair to the cobalt ion (Co^{2+}). At low concentrations, this interaction forms a neutral compound, cobalt acetate ($\text{Co}(\text{CH}_3\text{COO})_2$), which then precipitates, as shown in Equation (3) (Gutiérrez-Martín et al., 2022).



The yields shown in Table 1 were calculated based on the initial mass of the cathode precursor and the mass of cobalt carbonate, respectively. However, interpretation of this value must take into account the context of the raw material, i.e., the polymetallic cathode material, containing cobalt as a minor component along with nickel (Ni), manganese (Mn), aluminum (Al), and lithium (Li). The actual cobalt content in the black mass is only around 24.9% by weight, so the yield to total cathode mass is inherently lower and cannot be used as a sole indicator for evaluating cobalt conversion efficiency (Wang & Zhang, 2022).

The leaching process with an ascorbic acid concentration of 0.5 M at 70 °C, followed by precipitation, successfully produced 0.7693 grams of cobalt carbonate. The visual appearance of the products at different stages is shown in Figure 7.

At very high ascorbic acid concentrations (1.5 M and 2.5 M), the synthesis did not produce any solid product (0% yield).

This is mainly caused by two dominant effects: strong chelation and high ionic strength in the solution. Ascorbic acid plays a dual role. It acts as a reducing agent that dissolves cobalt from the cathode, and also as a ligand that binds with Co^{2+} ions to form a stable complex. When the ligand is present in excess, the reaction equilibrium shifts toward the formation of a dissolved cobalt–ascorbate complex, following Le Châtelier’s principle. In this condition, Co^{2+} ions prefer to remain bound to the ligand rather than forming a solid compound (Doble et al., 2025).

The high stability of this complex prevents the next step, which is the precipitation of cobalt carbonate (CoCO_3). In simple terms, cobalt ions become “trapped” by ascorbic acid and are no longer available to react with carbonate ions (CO_3^{2-}). This creates competition between the chelating agent (ascorbic acid) and the precipitating agent (carbonate ions). Chelating agents are known to keep metal ions in solution by reducing the number of free ions needed for precipitation (Hassan et al., 2020).

In addition, high concentrations of ascorbic acid increase the ionic strength of the solution. This reduces the activity coefficient of ions, meaning the ions become less reactive (Bulavin, 2017). As a result, the interaction between Co^{2+} and CO_3^{2-} becomes weaker, making precipitation even more difficult.

The combination of strong complex formation and reduced ion activity keeps cobalt

in the dissolved phase and prevents solid formation. A similar result was observed when using citric acid. In this case, cobalt carbonate could not be formed and the synthesis of cobalt acetate also failed. This is likely due to the high stability of the cobalt–citrate complex in water, which keeps cobalt in solution even after pH adjustment (Nikitenko et al., 2023).

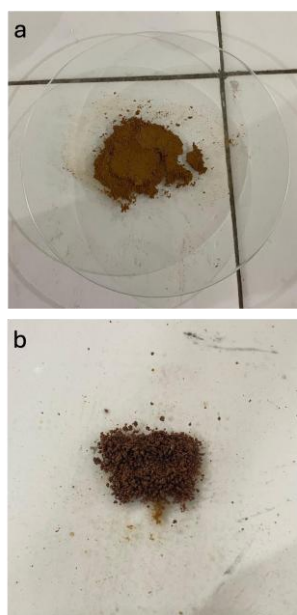


Figure 7. (a) Cobalt Carbonat Precipitate, (b) The Synthesized Cobalt Acetate

In Figure 7, the cobalt carbonate precipitate appears as a fine brownish powder. This powdery form indicates that the precipitation process occurred effectively,

where dissolved Co^{2+} ions reacted with carbonate ions (CO_3^{2-}) to form solid CoCO_3 . The relatively uniform and dry texture suggests that the precipitate was properly filtered and dried, which is important for obtaining a stable intermediate before further synthesis. In Figure 7(b), the synthesized cobalt acetate is shown after conversion from cobalt carbonate. The material appears darker and more compact, forming small aggregated particles rather than a loose powder. This change in color and texture indicates a successful chemical transformation from cobalt carbonate to cobalt acetate. The darker brown appearance is typical of cobalt-based compounds after coordination with organic ligands such as acetate. The more clustered morphology also suggests differences in crystal structure and bonding compared to the initial carbonate form.

The visual differences between Figure 7(a) and Figure 7(b) clearly demonstrate the progression of the synthesis process, from the formation of cobalt carbonate as an intermediate to the final cobalt acetate product. Although visual observation alone cannot fully confirm the chemical identity, these changes in color, texture, and morphology provide initial evidence of successful conversion.

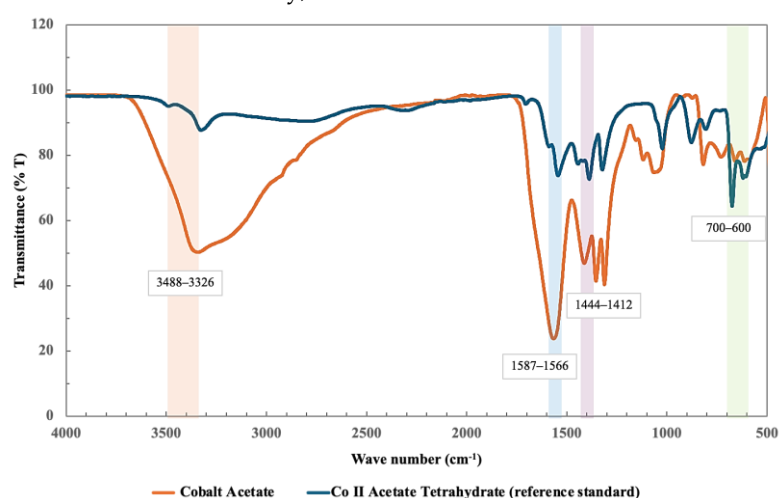


Figure 8. The FTIR Spectrum of the Synthesised Cobalt Acetate Compared to the Reference Cobalt (II) Acetate Tetrahydrate (AR/ACS CDH)

FTIR analysis was carried out by comparing the synthesized cobalt acetate with a commercial Cobalt(II) acetate tetrahydrate reference, as shown in Figure 8. Overall, both spectra display very similar characteristic absorption bands, indicating that the synthesized product has the same main functional groups as the reference and confirming the successful formation of cobalt acetate. The broad band observed in the region 3488–3326 cm^{-1} corresponds to O–H stretching vibrations, which indicate the presence of water molecules. This band appears much stronger in the reference sample, showing that it contains more crystal water, while the synthesized product likely has lower hydration or more weakly bound water.

The most important evidence of cobalt acetate formation is the presence of two strong peaks in the regions 1566–1587 cm^{-1} and 1412–1444 cm^{-1} . These peaks correspond to the asymmetric (ν_{as}) and symmetric (ν_{s}) stretching vibrations of the carboxylate group (COO^-). The presence of these two bands confirms that acetate ions are coordinated to cobalt. The difference between these two peaks ($\Delta\nu$) also suggests a bidentate coordination mode, for metal–acetate complexes.

Additional peaks further support this structure. Bands in the range 1354–1311 cm^{-1} are assigned to CH_3 bending, while peaks around 1154–1065 cm^{-1} correspond to C–O stretching vibrations, both of which confirm the presence of acetate groups. In the lower wavenumber region, peaks at 728–612 cm^{-1} and 481–441 cm^{-1} are attributed to Co–O stretching and vibration, providing direct evidence of bonding between cobalt ions and oxygen atoms from the acetate ligand.

Although the overall peak positions are very similar between the synthesized sample and the reference, slight differences in intensity are observed, especially in the O–H region.

These differences are mainly related to variations in water content and do not indicate major structural changes. Therefore, the FTIR results clearly demonstrate that the synthesized material is cobalt acetate with a structure comparable to the commercial standard, with only minor differences in hydration level.

CONCLUSION

This study examined the effectiveness of the cobalt leaching process from LiCoO_2 battery cathode waste using weak organic acids, namely ascorbic acid and citric acid. The analysis revealed that the concentration of ascorbic acid significantly affected the cobalt extraction yield from LiCoO_2 . The leaching process using ascorbic acid dissolved approximately 12 mg/L of cobalt. Increasing the ascorbic acid concentration from 0.5 M to 1.0 M increased the dissolved cobalt concentration due to the increased effectiveness of the reduction of Co^{3+} to Co^{2+} . The optimum concentration was found at 1.0 M, while using higher concentrations decreased leaching efficiency due to the limited ionization of ascorbic acid as a weak acid. Meanwhile, the leaching process using citric acid successfully dissolved cobalt, with dissolved cobalt concentrations obtained based on AAS analysis in the range of 13.58–13.89 mg/L. Overall, the leaching process with citric acid produces higher dissolved cobalt compared to the leaching process using ascorbic acid. Temperature significantly influences the cobalt leaching process using organic acids. Increasing the temperature to 70°C results in a higher and more stable concentration of dissolved cobalt compared to 55°C. This is because higher temperatures increase the reaction rate, ion diffusion, and cobalt stability in solution. Therefore, 70°C is a more favorable condition for cobalt leaching. However, in the citric acid leaching process, leaching at 50°C

and 70°C produced relatively similar dissolved cobalt concentrations. Temperatures higher than 70°C are necessary to determine the more significant effect of temperature. Based on the data obtained from the leaching process using organic acids, it can be seen that the best leaching process was obtained with citric acid at a concentration of 0.5 M at a temperature of 55°C, which produced dissolved cobalt of 13,837 mg/L. The next stage carried out in this research was to precipitate the dissolved cobalt ions into cobalt carbonate compounds and then convert them to cobalt acetate. The result showed that leaching with 0.5 M ascorbic acid yielded 17.42% in the cobalt acetate synthesis stage. Synthesis efficiency is highly dependent on the ascorbic acid concentration. At high concentrations (1.5 M and 2.5 M), synthesis failed (0% yield) due to the dominant chelation effect and the increased ionic strength of the solution, which stabilized the Co^{2+} ions in the dissolved phase, inhibiting the formation of the final product. Similarly, synthesis from the leached solution using citric acid did not produce the expected cobalt acetate. This is due to the high stability of the cobalt-citrate complex in the liquid phase. FTIR results showed a strong agreement between the synthesized cobalt acetate and the reference cobalt(II) acetate tetrahydrate, which confirmed the successful formation and structural consistency of the target compound.

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