

Article

Efficient Recovery of Lithium and Cobalt from Spent LCO Using Mechanochemical Activation and Ammoniacal Leaching

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Abstract

In this study, we investigate the recovery of Li and Co from spent LiCoO_2 cathodes of spent lithium batteries using a combined approach of mechanochemical activation (MA) and ammoniacal leaching. High-energy ball milling disrupts the layered structure of LiCoO_2 , introduces defects, and increases surface area, which strongly improves subsequent dissolution. Leaching experiments in an ammonia–ammonium sulphate–sulphite medium were optimized by varying the solid-to-liquid ratio, sodium sulfite concentration, and temperature. Under the best conditions (90 °C, 120 min, S/L = 10 g/L, 0.5 M Na_2SO_3), nearly complete recoveries were obtained: 99.5% Li and 96.5% Co. Kinetic modeling based on the shrinking-core model confirmed that dissolution of both metals is controlled by chemical reaction, with activation energies of $45.7 \text{ kJ}\cdot\text{mol}^{-1}$ for Li and $60.7 \text{ kJ}\cdot\text{mol}^{-1}$ for Co. Structural and morphological analyses (XRD, SEM) supported the enhanced reactivity of the activated material. The study demonstrates that MA coupled with optimized ammoniacal leaching provides an efficient process for LiCoO_2 recycling, without using aggressive mineral acids and long treatment times.

Keywords: cathode materials in LIBs; cobalt recycling; kinetic modeling; mechanochemical activation; ammoniacal leaching



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1. Introduction

The rapid growth of lithium-ion battery (LIB) applications has made the recovery of critical metals from spent cells an important matter [1–3]. Lithium (Li) and cobalt (Co) in LiCoO_2 (LCO) cathodes are of particular concern, since their primary production is both energy and carbon intensive and, at the same time, is exposed to supply-chain risks. Recycling helps to ease these pressures: it reduces environmental load, stabilizes availability, and lowers the life-cycle footprint of electrochemical storage systems [4–6].

Various methods for the recycling of spent LIBs—including pyrometallurgy, hydrometallurgy, direct repair, and electrochemical processes—have been comprehensively reviewed in recent studies [7]. Among known approaches, hydrometallurgy remains one of the most practical. It operates under moderate temperatures, allows selective dissolution and precipitation, and can yield target products with relatively modest energy demand. As a rule, leaching is carried out with strong mineral acids such as H_2SO_4 [8–11], HCl [12–14],

or HNO_3 [15,16], often together with reductants (H_2O_2 [17], sulfite [18], organic compounds [19]). These combinations are quite effective; however, they are usually characterized by high reagent consumption, difficulties with effluent treatment, co-dissolution of impurities, corrosion issues, and safety concerns that should not be overlooked.

In search of milder options, researchers have examined organic acids—citric [20,21], oxalic [22], or tartaric [23]. They are biodegradable and less toxic, and they do form complexes with Co^{2+} . Nevertheless, their performance is not straightforward; the reaction rates are often slow, insoluble metal–organic salts may precipitate, and, in practice, the process tends to consume large amounts of reagents. Efficient Co recovery usually requires heating and/or oxidants.

Deep eutectic solvents (DESs) have also been proposed for Li and Co extraction from LIBs [24,25]. They dissolve metals via coordination and acid–base interactions in non-aqueous media. However, DESs are not without drawbacks: the high viscosity and moisture sensitivity, together with generally slow leaching rates, often force the use of auxiliary activation methods such as ultrasonication or mechanical pretreatment.

Ammoniacal leaching represents another route. Aqueous ammonia and ammonium salts dissolve transition metals by forming ammine complexes under mildly alkaline conditions. Such media are well known in hydrometallurgy (e.g., for Cu, Ni, and Co [26]). They are relatively mild, selective for Co(II), and simplify purification. In the LIB context, ammoniacal systems have been tested on several cathodes, including LCO [27–30]. However, their drawback is slower dissolution: LCO often requires hours, mainly due to its compact layered structure, the need to reduce Co^{3+} to Co^{2+} , and passivation phenomena; in addition, graphite and other black-mass components hinder access to reaction sites.

Mechanochemical activation (MA) has therefore been adopted as a pretreatment prior to leaching. High-energy milling reduces particle size, introduces defects, induces partial amorphization, and can trigger in situ redox interactions with carbon or residual metals. These effects substantially increase the leachability of raw material [31–34]. For spent cathodes, MA has been shown to disrupt the layered lattice, enlarge surface area, and thereby speed up dissolution. As demonstrated by Zhang et al., one-step mechanochemical leaching of LCO cathodes enabled metal extraction rates approaching 99.8% for Li and 99.7% for Co under optimized conditions [35]. Dolotko et al. also showed that solvent-free mechanochemical processing yielded up to ~90% recovery for cobalt and ~70% for lithium from pure LCO, even without harsh reagents [36]. Jiang et al. reported high leaching efficiencies for Li and Co (often exceeding 96%) due to the synergistic effect of mechanochemical pretreatment on mixed cathode powders [37]. In a study by Algül and Algül, use of MA followed by citric acid leaching achieved near-complete recovery (94–99%) for both lithium and cobalt depending on the process parameters and optimization [38]. Review studies, such as that of Eom et al., corroborate these results by determining that mechanochemical treatment routinely raises Li and Co extraction yields to 90–99%, regardless of cathode composition [39]. Meng et al. (2019) extend these findings to Ni-rich NCM materials; they achieved metal recovery rates above 90% using combined mechanochemical and thermal processing [40]. Recent studies [41,42] further report that rapid MA can reach or exceed 98–99% for Li and Co under optimized and green processing regimes.

In these cases, ball milling generates strain and defects and improves contact with conductive additives such as carbon black or graphite. The benefits are clear, though the exact extent depends on the cathode chemistry and milling conditions.

Recently, we applied MA to LCO black mass [43]. Optimal milling conditions (800 rpm, 60 min, ball-to-powder ratio (BPR) = 50:1) were identified and led to significantly improved leachability in ammoniacal medium. Structural disordering and defect generation were confirmed as the basis of this effect. Nevertheless, the aforementioned study did not address

the kinetics of dissolution in detail, nor the rate-controlling step, nor did it systematically optimize the ammoniacal chemistry for the activated powders.

To fill these gaps, in this study, we use an ammonia–ammonium sulphate–sulphite system to extract Li and Co from mechanochemically activated LCO, optimize the leaching parameters for the activated material through response surface analysis, and carry out a kinetic study using the shrinking-core model. This allows us to identify the rate-limiting stage and estimate activation energies. The novelty of the study is in combining mechanochemical activation with ammonia-based leaching for LCO cathodes, together with process optimization and kinetic evaluation.

A recent review has summarized electrode-level challenges and material design strategies associated with these advanced cathodes [44]. Among the known cathode materials, LCO remains a widely used and well-characterized system in portable electronics and it is selected here as a representative target for process development.

2. Materials and Methods

2.1. Materials

Consumer-grade cylindrical lithium-ion batteries (LCO type) from used electronics were selected for this study. The service history of each cell was not recorded, but all exhibited common aging features such as capacity reduction and discoloration. Cathode layers were manually separated from aluminum collectors, dried at ambient conditions, and sieved. Disassembled cathode foils after battery opening and drying are presented in Figure 1.



Figure 1. Disassembled cathode foils with LCO coating.

Ethanol rinsing was used to remove residual electrolytes, resulting in a purified LiCoO_2 -based cathodic material for analysis.

The chemical composition of the starting LCO-containing black mass is presented in Table 1.

Table 1. Chemical composition of the spent cathode material.

Li, wt %	Co, wt %	Al, wt %	Cu, wt %
6.8	52.5	0.8	0.4

Minor amounts of Fe, Ni, and Mn were also detected (all below 0.05 wt %) but are not listed in Table 1, as the study focuses on Li and Co leaching from the LCO-based fraction.

The leaching reagents included analytical-grade ammonia solution (25 wt%), ammonium sulphate ((NH₄)₂SO₄), and sodium sulphite (Na₂SO₃), all purchased from commercial suppliers in China. Distilled water was used throughout the experiments.

2.2. Mechanochemical Activation

Mechanochemical activation of the initial LCO sample was performed in a planetary ball mill Pulverisette 7 premium line (Fritsch, Idar-Oberstein, Germany). Then, 10 g of LCO-containing black mass was loaded into a 250 mL zirconia vial together with zirconia balls (10 mm diameter) at a ball-to-powder mass ratio of 50:1. Milling was carried out at 800 rpm for 1 h with 3 min breaks every 20 min to avoid overheating. The activated powders were stored in airtight containers before further use. The process was conducted under ambient air conditions. Particle size distribution (PSD) analysis showed a reduction from 12.6–96.4 µm (P80~68.5 µm) in the pristine sample to 1.5–8.4 µm (P80~7.2 µm) after activation.

2.3. Ammonia Leaching Procedure

Ammonia leaching was conducted in a glass reactor with a working volume of 1 L, equipped with a reflux condenser, mechanical stirrer, and temperature controller. The solution consisted of 4.0 M NH₃·H₂O, 1.5 M (NH₄)₂SO₄, and 0.05–1.0 M Na₂SO₃. The solid-to-liquid ratio (S/L) was varied from 5 to 50 g/L. A fixed leaching solution volume of 1 L was used in all experiments. In a typical test, 10 g of LCO-containing black mass was added to 1 L of the leaching solution. The slurry was agitated at 400 rpm, and the temperature was maintained at 50–90 °C for up to 180 min. At selected intervals, aliquots of 5 mL were recovered and analyzed for Li and Co.

2.4. Analytical Methods

Phase composition of solids (initial and leaching residue) was analyzed using the X-ray diffraction method (XRD, DW-27 Mini, Dandong Dongfang, 128 Dandong, China). Microstructural observations were carried out via scanning electron microscopy (SEM, FEI Quanta 200i). The elemental composition of both solids and liquid leachates was determined via atomic absorption spectrometry (AAS) using a GBC Savant system (GBC Scientific Equipment Pty Ltd., Australia). Before analysis, solid samples were pre-digested via a microwave-assisted treatment in a HCl: H₂O₂ mixture at 80 °C for 2 h to achieve complete dissolution. Each measurement was performed in triplicate, and the relative standard deviation did not exceed ±3%.

2.5. Thermodynamic Modeling

The Eh–pH diagram of the Co–NH₃–H₂O system was constructed to visualize the stability domains of cobalt species under ammonia–ammonium conditions. Calculations were performed at 25 °C using equilibrium constants from the literature: pK_a(NH₄⁺/NH₃) = 9.25 [45] and pK_{sp}(Co(OH)₂) = 14.9 [46]. The potential–pH boundaries were computed from the Nernst equation assuming a total ammonia concentration of 4 M. Water stability limits (H₂/H₂O and O₂/H₂O) were included for reference. The diagram was plotted in Python (Matplotlib, version 3.8.4) based on thermodynamic relations.

3. Results and Discussion

3.1. Ammonia Leaching Behavior

Initial tests were performed on a black-mass-containing LCO, without any prior MA. Leaching was conducted at three temperatures (50, 70, and 90 °C), and extraction of Li and Co vs time is plotted in Figure 2. The solution was prepared using 4.0 M ammonia, 1.5 M ammonium sulphate, and 0.5 M sodium sulfite; 10 g of solid was added per 1 L of leachate (S/L = 10 g/L).

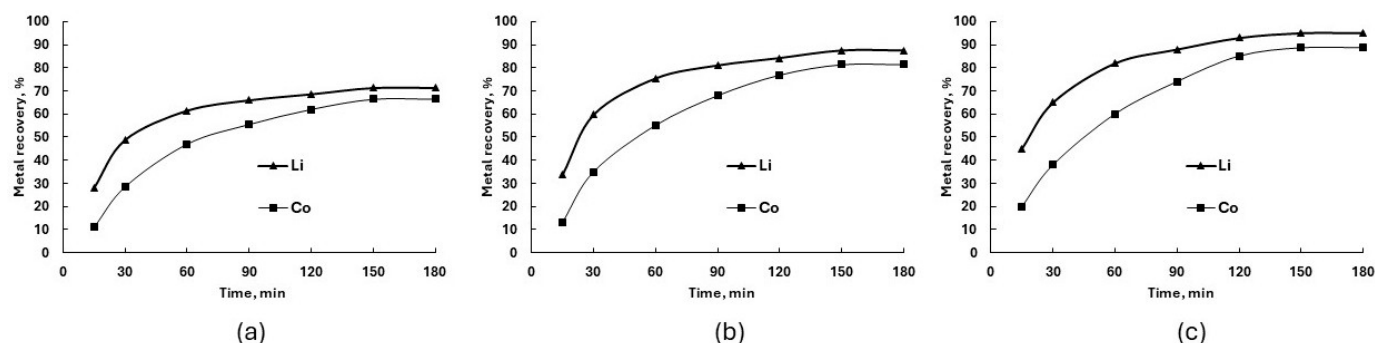


Figure 2. Li and Co extraction from LCO black mass during ammonia leaching over the time (4.0 M $\text{NH}_3 \cdot \text{H}_2\text{O}$ + 1.5 M $(\text{NH}_4)_2\text{SO}_4$ + 0.5 M Na_2SO_3 , S/L = 10 g/L) at 50 (a), 70 (b), and 90 (c) °C.

Leaching of the untreated LCO showed a clear temperature effect: the higher the temperature, the faster and deeper the extraction of both Li and Co. Li recovery reached 71.3% at 50 °C, 87.5% at 70 °C, and 95.1% at 90 °C. Cobalt followed the same trend, increasing from 66.4% to 88.6% across the same range. In each case, the bulk of the dissolution happened relatively quickly, with the curves flattening after about 120 min. Similar behavior during ammoniacal leaching has been reported for LIB cathodes [47], though that study dealt with NCM ($\text{LiNi}_{0.33}\text{Mn}_{0.33}\text{Co}_{0.33}\text{O}_2$), not LCO as in our research.

The next set of experiments used mechanochemically activated LCO. Milling conditions are described in Section 2 and matched those used in our earlier research [43]. At this stage, the focus shifted to optimization: changing the solid-to-liquid ratio, varying the concentration of sodium sulfite, and later adjusting the leaching duration. To keep the number of variables manageable, leaching time was fixed at 120 min, since earlier tests had shown this interval was generally sufficient to approach the plateau values.

Figure 3 shows the response surface plots illustrating how the S/L ratio and Na_2SO_3 concentration affected lithium and cobalt extraction from MA LCO at 70 °C and 90 °C. The plots were generated from experimentally measured extraction data at a fixed leaching time of 120 min and interpolated using the griddata function in Python to visualize the combined effect of these parameters on Li and Co recovery.

Li extraction consistently increases at lower S/L values across both temperatures, which can be attributed to improved reagent availability and more favorable phase contact in diluted slurries. As the S/L ratio increases, the effective concentrations of free ammonia and ammonium sulphate per unit mass of LiCoO_2 decrease, potentially limiting complexation of Li^+ and slowing the leaching rate. The impact of $[\text{Na}_2\text{SO}_3]$ on Li extraction is modest and non-linear. This behavior reflects the fact that lithium is already present as Li^+ and does not require reduction; thus, sulfite has no direct role in Li extraction and acts mainly as a pH buffer and ionic strength modifier [48,49]. Slight improvements at intermediate sulfite concentrations may stem from the stabilization of pH and enhanced ionic strength.

Co recovery, in contrast, is highly sensitive to $[\text{Na}_2\text{SO}_3]$, especially in the lower concentration range (0.05–0.5 M). This reflects the redox nature of cobalt leaching: Co^{3+} in LiCoO_2 must first be reduced to Co^{2+} in order to form soluble ammine complexes [50].

Increasing sulfite concentration up to ~ 0.5 M leads to a sharp improvement in Co extraction, after which the effect saturates, suggesting that reductive dissolution becomes limited by other factors (e.g., surface availability or ammonia complexation). The temperature rise from 70°C to 90°C further amplifies extraction efficiency for both metals; this confirms that the leaching process is kinetically favored at elevated temperatures.

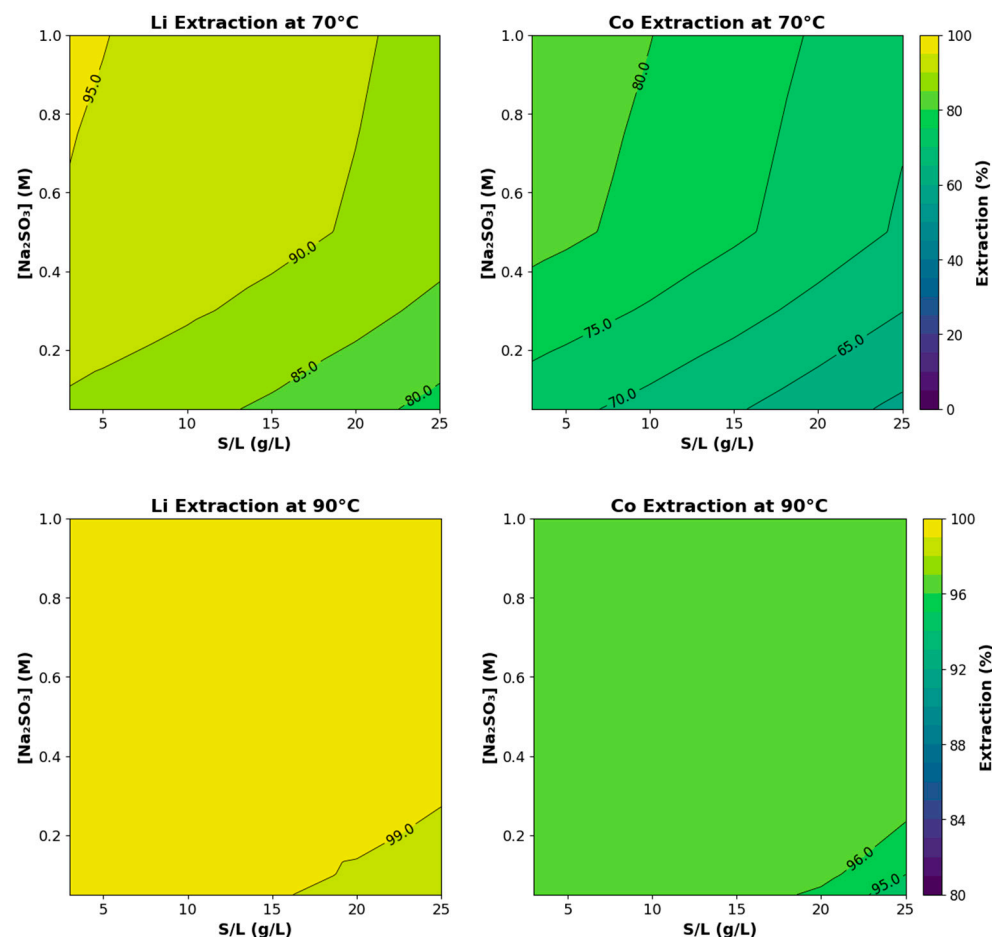


Figure 3. Contour plots showing the effects of solid-to-liquid ratio (S/L) and sodium sulfite concentration on the extraction of lithium and cobalt from mechanochemically activated LiCoO_2 during ammonia-based leaching ($4.0\text{ M NH}_3 \cdot \text{H}_2\text{O} + 1.5\text{ M (NH}_4)_2\text{SO}_4 + \text{Na}_2\text{SO}_3$) for 120 min at 70°C and 90°C .

Collectively, the observed trends suggest that Li leaching is governed mainly by physical parameters such as slurry dilution (S/L), which facilitates solid–liquid interaction and promotes Li^+ release. Unlike Co, Li does not rely on redox reactions or strong complexation, which explains its relatively weak dependence on sodium sulfite concentration. Co leaching is strongly influenced by the availability of reducing agents, especially at lower Na_2SO_3 concentrations.

Minor side reactions may occur during ammoniacal leaching, including partial oxidation of sulfite to sulfate and transient formation of Co-NH_3 complexes with variable stability, which can slightly influence the solution redox state. However, no significant Li loss or secondary precipitation was observed. The presence of Fe, Ni, Al, and Cu in the initial black mass did not noticeably affect leaching performance: Al and Cu remained largely undissolved under mildly alkaline conditions, confirming the selectivity of the ammonia–ammonium sulfate system toward Co(II) over base-metal impurities. Only minor Fe and Cu traces ($<0.1\text{ wt\%}$) were detected in residues, indicating negligible co-dissolution and minimal interference with overall metal recovery.

It should be noted that the applied leaching medium contained concentrated ammonia (4 M NH_3), which ensures rapid dissolution but requires proper gas scrubbing and ventilation when scaled up. Industrial implementation should include closed-loop recovery and neutralization systems to minimize emissions and reagent losses. Alternatively, a stepwise leaching with 2–3 M NH_3 in recirculating setups could reduce safety risks and operational costs while preserving selectivity.

To further rationalize the dissolution behavior of cobalt in the ammonia–ammonium–sulfite medium, the Eh–pH diagram of the Co– NH_3 – H_2O system was constructed (Figure 4).

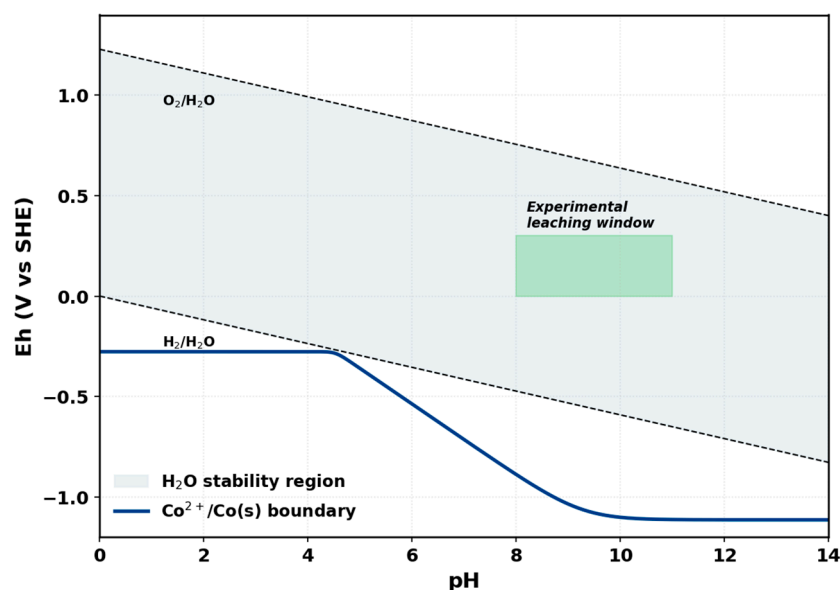


Figure 4. Simplified Eh–pH diagram of the Co– NH_3 – H_2O system (25 °C, 4 M NH_3).

Under the experimental conditions ($\text{pH} \approx 9$ – 10 , $E_h \approx 0$ – 0.3 V), Co is thermodynamically stable in the divalent soluble state, where coordination with ammonia predominates over hydrolysis or precipitation. Co^{3+} oxides are unstable and readily reduced to Co^{2+} by sulfite, whereas metallic cobalt and $\text{Co}(\text{OH})_2$ are outside this potential–pH window. Li, in contrast, does not form coordination complexes with ammonia and remains as a simple hydrated ion Li^+ .

Figure 5 presents contour plots illustrating the extraction efficiencies of lithium and cobalt from mechanochemically activated LiCoO_2 as functions of leaching time and temperature, under fixed conditions of $\text{S/L} = 10$ g/L and $[\text{Na}_2\text{SO}_3] = 0.5$ M.

The recovery of both Li and Co increased with increasing temperature, but the degree and rate of recovery differed for the two metals.

Li recovery reached >95% by 90 min at 70 °C and reached a plateau (99%) 120 min after the start of leaching. Li, being a monovalent metal and already present in the soluble oxidation state (Li^+), dissolves relatively easily, requiring no redox conversion. With increasing temperature, leaching kinetics accelerated due to the increased mobility of ions and solvent molecules, leading to virtually complete recovery of this metal in a relatively short time, especially at temperatures of 70 °C and above.

Co recovery was somewhat slower. High recoveries were achieved only at elevated temperatures (90 °C) and longer leaching times (≥ 120 min). This phenomenon is caused by the need for the chemical reduction of Co^{3+} to Co^{2+} before dissolution [46]. The strong temperature dependence suggests a chemically controlled mechanism involving a redox transformation facilitated by sulfite ions. Co is initially present as Co^{3+} in the LiCoO_2 lattice, which must undergo chemical reduction to Co^{2+} to become soluble in an ammo-

nial medium. This redox step is kinetically slower and more temperature dependent. The observed delay in achieving high Co recovery (>90%) at lower temperatures and shorter times reflects the energy barrier associated with the need to reduce cobalt before dissolution. Based on response surface analysis and time and temperature optimization, leaching conditions were established that ensure virtually complete metal extraction from mechanochemically activated LiCoO_2 : 90 °C, 120 min, 10 g/L, and 0.5 M Na_2SO_3 in an ammonia–ammonium sulphate system. Under these conditions, the extraction efficiency of Li and Co reached 99.5% and 96.5%, respectively. The initial pH of the leaching solution was ~9.7 and decreased to ~9.2 after 120 min of leaching.

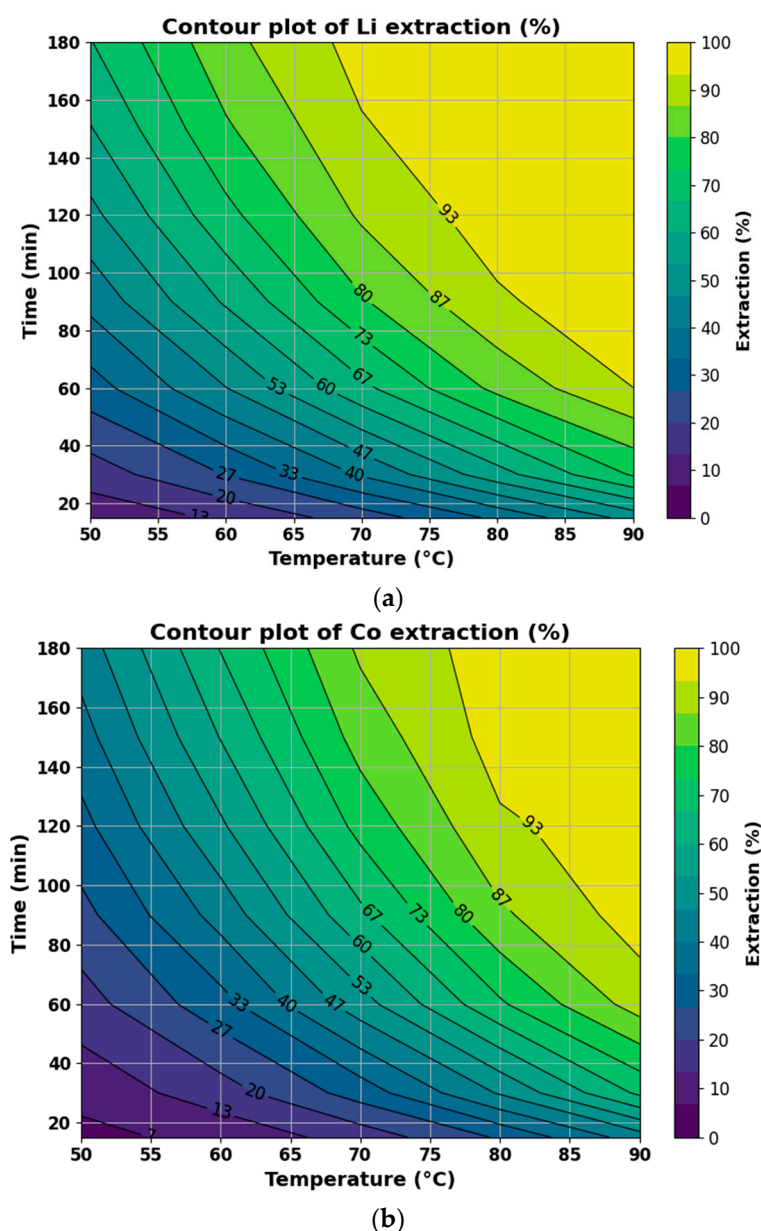


Figure 5. Contour plots of Li and Co extraction from mechanochemically activated LiCoO_2 as a function of leaching time and temperature at fixed $S/L = 10$ g/L and $[\text{Na}_2\text{SO}_3] = 0.5$ M: (a) Li extraction (%); (b) Co extraction (%).

3.2. Leaching Kinetics

The determination of kinetic parameters is essential for understanding the rate-limiting steps of leaching processes and for enabling reliable scale-up and process optimization. The shrinking core model (SCM) is widely used to describe the kinetics of solid–liquid leaching

systems, especially in hydrometallurgical processes involving the dissolution of metal oxides [51,52]. It allows for the identification of the rate-controlling step by correlating extraction data with time using physically meaningful expressions. In the present study, SCM-based analysis was applied to better understand the leaching behavior of Li and Co from mechanochemically activated LiCoO_2 in ammonia-based media. Preliminary leaching results (Figure 3) suggested a gradual approach to equilibrium and temperature-dependent extraction, indicating that either surface reaction or product-layer diffusion could be the rate-determining step.

To discriminate between these two possibilities, we plotted the experimental leaching data at 90 °C using two SCM kinetic models, the chemical-reaction-controlled (Equation (1)) and the diffusion-controlled (Equation (2)) models:

$$1 - (1 - X)^{1/3} = k\tau \quad (1)$$

$$1 - \frac{2}{3}X - (1 - X)^{2/3} = k\tau \quad (2)$$

where X is the fractional extraction and k is the apparent rate constant.

The resulting plots are shown in Figure 6.

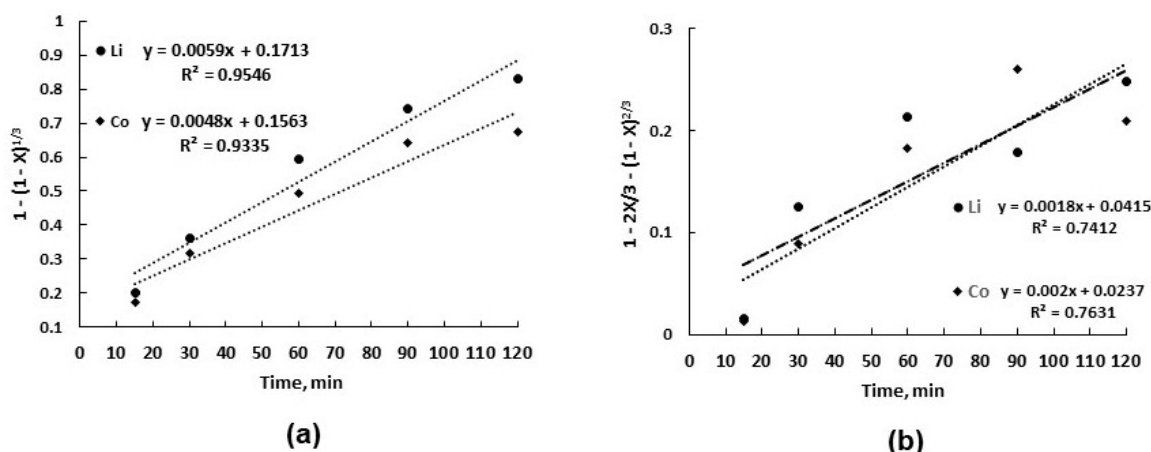


Figure 6. Linearized SCM plots for Li and Co extraction from the mechanochemically activated LCO cathode during ammonia leaching at 90 °C under chemical (a) and diffusion (b) control.

Based on the data presented in Figure 4, the leaching kinetics of both Li and Co are better described by the chemical-reaction-controlled SCM. This is evidenced by the higher correlation coefficients ($R^2 = 0.9546$ for Li and 0.9335 for Co) observed in plot (a), compared to lower R^2 values in the diffusion-controlled model shown in plot (b) ($R^2 = 0.7412$ for Li and 0.7631 for Co).

Figure 7 shows a plot of $1 - (1 - X_{Me})^{1/3}$ vs leaching time for Li (a) and Co (b) recovery from LCO cathode during ammonia leaching at $S/L = 10$ g/L.

A linear relationship was observed in all cases, which confirms the applicability of the SCM with chemical reaction as the rate-controlling step. The rate constants (k) increased with temperature: for Li, from 0.0009 min^{-1} at 50 °C to 0.0059 min^{-1} at 90 °C; and for Co, from 0.0004 min^{-1} to 0.0048 min^{-1} . The high R^2 values (≥ 0.93) for all three studied temperatures support the kinetic model's validity and enable estimation of E_a values.

Arrhenius plots for Li and Co recovery from mechanochemically activated LCO cathode during ammonia leaching were constructed using the determined rate constants (Figure 8).

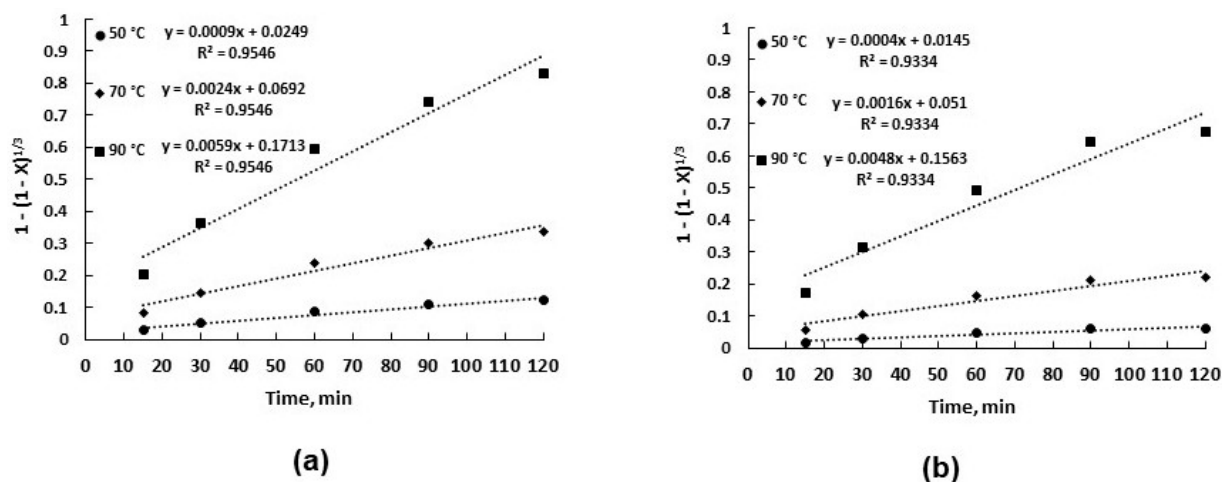


Figure 7. A plot of $1 - (1 - X_{Me})^{1/3}$ vs leaching duration for Li (a) and Co (b) recovery from mechanochemically activated LCO cathode during ammonia leaching (S/L = 10 g/L).

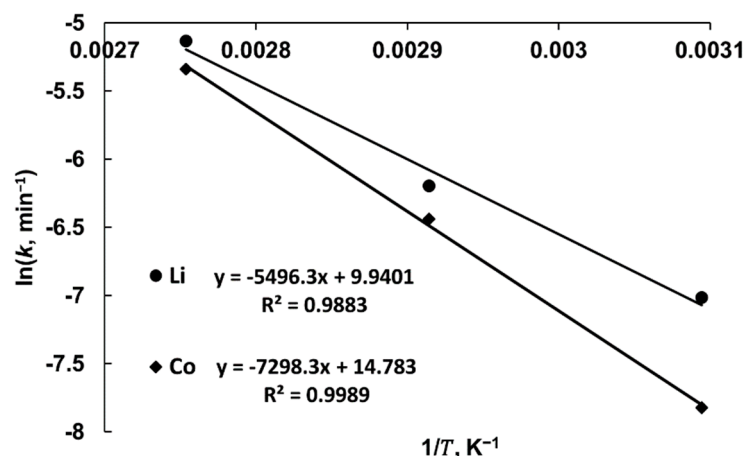


Figure 8. Arrhenius plot for Li and Co recovery from mechanochemically activated LCO cathode during ammonia leaching (S/L = 10 g/L).

The activation energies for Li and Co recovery from mechanochemically activated LiCoO_2 were determined as 45.7 and 60.7 kJ/mol, respectively. These values are in an agreement with the lower range of literature reports for similar systems. For instance, a recent study by Su et al. reported activation energies of 47.7 kJ/mol for cobalt during ammonia leaching of spent LIB cathodes [53], while additional studies have documented values of 91.7 kJ/mol for Co and 143–157 kJ/mol for Li in ammoniacal solutions, depending on the cathode material and process conditions [54,55].

These values fit the range characteristic of processes, where the chemical reaction is the rate-limiting stage [56,57]. The relatively high E_a for Co shows the important role of the $\text{Co}^{3+} \rightarrow \text{Co}^{2+}$ reduction step, which makes possible its dissolution in ammoniacal media and has been demonstrated in earlier work on Co recovery from layered oxides. Li shows a somewhat lower E_a value, due to faster kinetics and weaker temperature dependence. The latter is consistent with the simple monovalent dissolution mechanism of Li without redox transformations. Comparable E_a values for Li recovery into solution have been reported in ammonia-based and organic acid systems, generally in the 35–50 kJ/mol range.

3.3. Material Characterization

Figure 9 shows the diffraction patterns of the cathode material (LCO) at various stages of processing.

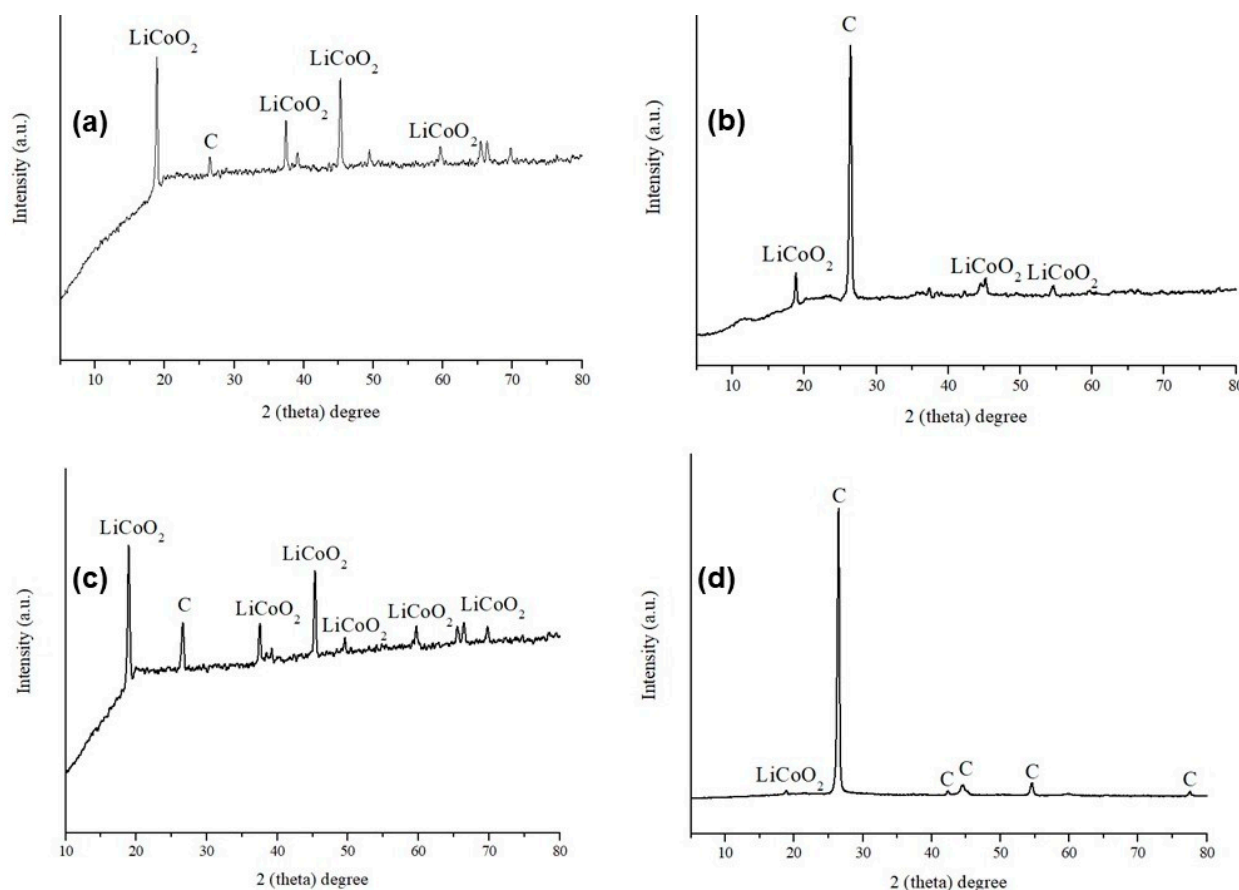


Figure 9. XRD patterns of the initial material at different treatment stages: (a) pristine LCO, (b) mechanically activated LCO, (c) LCO after ammonia leaching (90 °C, 120 min, 10 g/L), (d) mechanically activated LCO after ammonia leaching.

The initial sample (Figure 9a) is characterized by intense and narrow peaks corresponding to the LiCoO₂ phase. Weak carbon signals are associated with the conductive additive and the current collector. After mechanochemical activation (Figure 9b), the LiCoO₂ peaks significantly broaden and lose intensity, while the contribution of the carbon signal increases sharply. This indicates the destruction of the ordered layered structure and an increase in the degree of amorphization due to mechanical action. Ammonia leaching of the initial oxide (Figure 9c) is accompanied by a decrease in the intensity of LiCoO₂ reflections and the appearance of a more pronounced diffuse background, which reflects partial dissolution and disorganization of the crystal lattice; at the same time, the part of the initial phase is still preserved. In the case of mechanically activated material after ammonia leaching (Figure 9d), the LiCoO₂ reflections almost completely disappear, and only the carbon peaks remain, which indicates a deeper degree of destruction of the active phase. This confirms that mechanochemical activation significantly accelerates and facilitates the leaching process.

Figure 10 shows SEM images of LiCoO₂ cathode material at various stages of processing.

In the initial state of LCO-containing black mass (Figure 10a), the particles are large and have clearly defined edges, characteristic of a well-crystallized material. After mechanochemical activation (Figure 10b), particle destruction is observed, with the formation of rough surfaces and a fine dispersed fraction. Ammonia leaching of the starting material (Figure 10c) leads to partial dissolution of the surface with the formation of pores and cavities while maintaining the overall morphology of the grain. The most significant changes occur with a combination of mechanical activation and leaching (Figure 10d); the

particles lose their original shape, become loose, and represent agglomerates of small porous fragments. The enhanced dissolution after MA can be attributed mainly to defect generation, increased surface area, and partial lattice distortion rather than phase transformation. This interpretation aligns with previous mechanochemical studies, where amorphization and microstrain formation were shown to accelerate subsequent leaching kinetics.

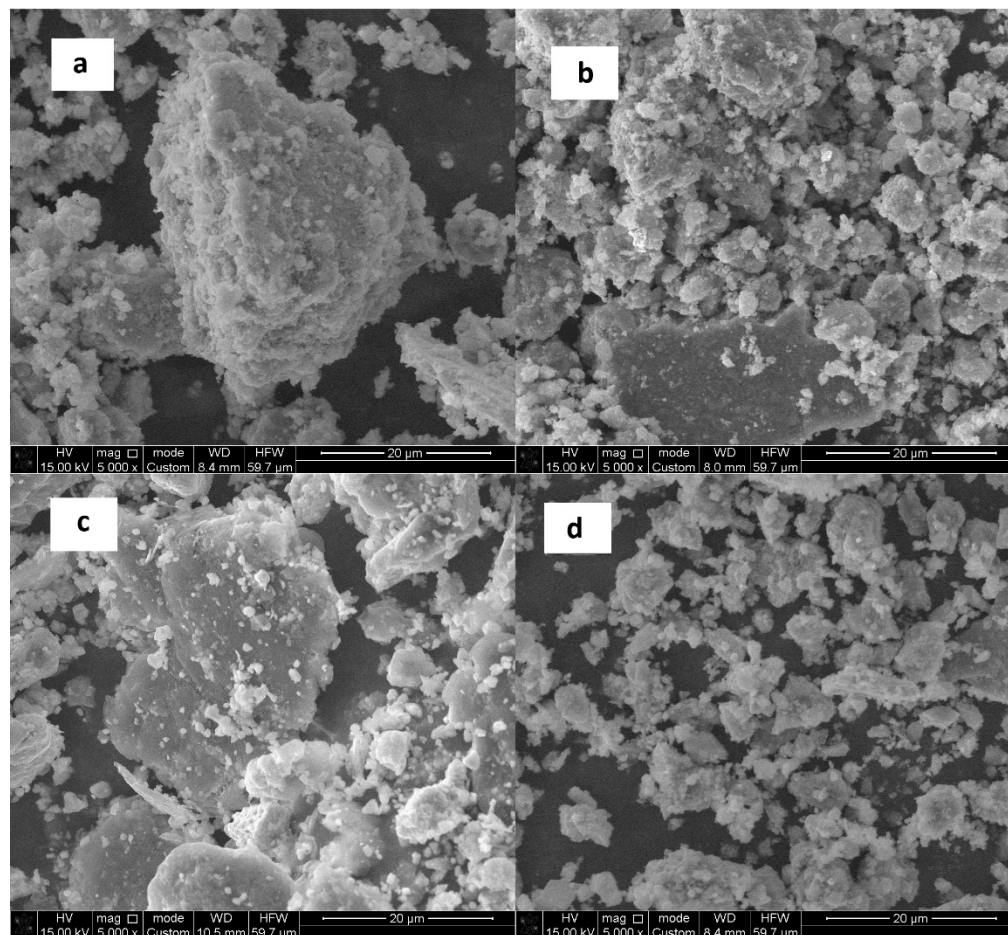


Figure 10. SEM images of the initial material at different treatment stages: (a) pristine LCO, (b) mechanically activated LCO, (c) LCO after ammonia leaching (90 °C, 120 min, 10 g/L), (d) mechanically activated LCO after ammonia leaching.

3.4. Comparative Discussion

In Table 2, previously reported studies on ammoniacal leaching of mechanochemically activated LiCoO_2 cathodes are summarized alongside the present research.

Li et al. showed that ball milling followed by leaching in an $\text{NH}_3\text{--Na}_2\text{SO}_3\text{--NH}_4\text{Cl}$ medium cut down the dissolution time and almost completely dissolved cobalt (98.2%), but lithium recovery stayed below 90% [48]. In [49], Zhang et al. pushed the approach further by adding in situ chlorination during milling. This step allowed them to reach 100% Li recovery and nearly full Co dissolution, which likely led to the formation of soluble LiCl and CoCl_2 phases, but it came at a price: the process required acidic conditions with ammonium chloride, which are less selective and raise corrosion issues. Mussapyrova et al. took another route, co-milling with metallic additives such as Al and carbon. Lithium recovery was high (94.6%), but cobalt reached only ~84%, and the leaching dragged on for about six hours [42]. Against this backdrop, our results stand out. By pairing high-energy milling with an ammonia–ammonium sulphate–sulphite system, we achieved both high cobalt (96.5%) and lithium (99.5%) recoveries at 90 °C in just two hours. The method

delivers efficiency and selectivity together, without relying on aggressive acids or on unreasonably long leaching times.

Table 2. Reported studies on mechanochemically activated LCO cathodes subjected to ammoniacal leaching.

MA Conditions	Leaching Conditions	Co Recovery (%)	Li Recovery (%)	Refs.
High-energy ball milling (wet, zirconia media), 5 h	NH ₃ -Na ₂ SO ₃ -NH ₄ Cl solution, pH $\approx$ 10, 80 °C, $\sim$ 4 h (after MA; vs. 48 h without MA)	98.22	89.86	[58]
Ball milling with NH ₄ Cl, 500 rpm, 120 min (in-situ chlorination)	1 M H ₂ SO ₄ + 0.03 M NH ₄ Cl, 80 °C, 120 min, L/S $\approx$ 10 mL·g ⁻¹	99.22	100	[59]
Planetary ball milling, 800 rpm, 60 min, BPR $\approx$ 50:1, co-milled with 5 wt% Al + 2.5 wt% C	(NH ₄) ₂ CO ₃ -NH ₃ solution: 3.0 M NH ₃ ·H ₂ O + 1.0 M (NH ₄) ₂ CO ₃ , 60 °C, 6 h, L/S = 25 mL·g ⁻¹	83.7	94.6	[43]
Planetary ball milling, 800 rpm, 60 min, BPR = 50:1	4.0 M NH ₃ ·H ₂ O + 1.5 M (NH ₄) ₂ SO ₄ + 0.5 M Na ₂ SO ₃ , S/L = 10 g/L, 90 °C, 120 min	96.5	99.5	This work

A related study by Liu et al. reported ammoniacal leaching of mixed LIB cathodes using an (NH₄)₂CO₃-NH₃-(NH₄)₂SO₃ system, with Co and Li recoveries of 84.6% and 90.3%, respectively [60]. In that study, cobalt and nickel were extracted as ammine complexes, while manganese partially precipitated in the presence of excess sulphite. Compared to Liu's system, our approach uses a simpler reagent composition (NH₃, (NH₄)₂SO₄, Na₂SO₃), targets a specific LCO feedstock, and achieves higher recoveries (96.5% Co, 99.5% Li) in shorter times (2 h vs. 4–6 h). These results were made possible by the use of mechanochemical activation, which enhances leachability by introducing structural defects and increasing surface area, in combination with thermodynamic optimization of the ammoniacal system.

Despite its high efficiency, the mechanochemical route has several practical limitations. Planetary ball mills, while effective for activating LCO at the laboratory scale, exhibit high specific energy consumption (often >1 kWh kg⁻¹ of powder) and limited batch capacity, which constrains industrial scalability. Continuous or attritor-type mills could mitigate these issues but require effective cooling and wear-resistant media to prevent Co or Zr contamination to minimize maintenance frequency. The heat generated during high-energy milling may also promote unwanted surface oxidation or partial decomposition of Li-Co-O phases, and it emphasizes the need for energy management and temperature control. Nevertheless, mechanochemical activation remains valuable at the pilot scale for enhancing reactivity and shortening leaching time in ammonia media.

In addition, compared with conventional acid leaching, the ammonia–ammonium sulfate–sulfite system uses milder reagents and generates negligible acidic effluents, which lowers the overall environmental footprint of the process despite the higher energy input required for mechanical activation.

4. Conclusions

The results of this study show that the combination of mechanochemical activation and ammoniacal leaching provides an efficient approach for recovering lithium and cobalt from spent LCO cathodes. The main difficulty in recycling LCO lies in its compact layered structure and the slow kinetics of cobalt dissolution in mild media. This problem was effec-

tively resolved through mechanochemical activation, which disrupted the lattice, increased surface area, and generated structural defects that accelerated metal dissolution. Optimization of the ammonia–ammonium sulfate–sulfite system showed that moderate sulfite addition enhances Co reduction and leaching efficiency, while Li extraction is primarily governed by solid–liquid interactions. Kinetic analysis using the shrinking-core model indicated that the dissolution of both metals proceeds under surface-reaction control, with activation energies of 45.7 kJ mol^{-1} for Li and 60.7 kJ mol^{-1} for Co. Compared with acid leaching, the proposed ammonia system avoids corrosive reagents and minimizes acidic effluents, thus reducing the overall environmental impact. At the same time, several practical aspects were identified. Planetary ball milling, while effective for laboratory activation, involves relatively high energy consumption and limited throughput. These constraints can be mitigated by employing continuous or attritor-type mills and by integrating ammonia recovery into closed-loop operations.

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