

Leaching of Spent Lithium-Ion Battery Black Mass Using Choline Chloride–Based Deep Eutectic Solvents

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Abstract

Deep eutectic solvents (DESs) are becoming a popular area of research as lower-volatility, possibly more sustainable, alternatives to traditional leaching media to recover desired metals in spent lithium-ion battery (LIB) black mass. In this work, choline chloride (ChCl) was initially produced by extraction of the starting feedstock using aqueous solution, then gravity filtration, extended low-temperature evaporation and ultimate dehydration/solidification at 105 °C to form solid ChCl. Three DESs containing ChCl as a hydrogen bond acceptor and urea, ethylene glycol or citric acid as hydrogen bond donors (reline 1:2, ethaline 1:2, and ChCl: citric acid 1:1) were subsequently prepared. These systems demonstrated exhibited different physical characteristics of low-viscosity homogeneity (ethaline) and high viscosity containing visible inclusions (citric acid DES). Experiments on leaching were carried out by putting LIB black mass in touch with each DES at a ratio of black mass: DES mass ratio 1:25 under stirring at a temperature of 80 °C. The color of all mixtures became dark within the period of approximately 2 h, in which there was progressive dissolution, and total visual dissolution occurred after 6 h and addition of water was needed to counter the rise in viscosity and partial solidification in the ethylene glycol and citric acid systems. Overall, the results demonstrate the practical feasibility of ChCl-based DESs for low-temperature black mass dissolution, while highlighting solvent-dependent handling challenges that should be considered in downstream separation and metal recovery workflows.

Keywords: deep eutectic solvents (DESs), choline chloride (ChCl), LIB black mass, viscosity control, leaching.

1. Introduction

The rapid global expansion of electric vehicles (EVs) and other battery-powered technologies is driving unprecedented demand for lithium-ion batteries (LIBs), and therefore for critical metals such as Li, Co, Ni, and Mn [1,2]. As early EV generations begin to reach end-of-life, the growing stream of spent LIBs is becoming both an environmental risk and a strategic secondary resource. Effective recycling pathways are increasingly viewed as essential for reducing supply risks, lowering lifecycle impacts, and supporting a more circular battery economy [2–4].

Current industrial and near-industrial LIB recycling approaches are commonly grouped into pyrometallurgical, hydrometallurgical, and direct (or “short-loop”) recycling routes [3,4]. Pyrometallurgy is operationally robust but energy-intensive and may lead to losses of lithium and other elements into slags or mixed phases, often requiring additional downstream processing [4]. Hydrometallurgy can achieve high recovery and selectivity but typically relies on strong mineral acids, oxidants/reductants, and extensive aqueous separation steps that generate secondary effluents and increase process complexity [3,4]. Direct recycling aims to preserve and regenerate cathode materials with fewer chemical transformations, but it is sensitive to feedstock variability, binder/electrolyte residues, and mixed chemistries – challenges that become more severe as real-world waste streams diversify [4,7].

A central intermediate in many recycling flowsheets is “black mass,” a fine particulate fraction produced after mechanical pretreatment (discharge, dismantling, crushing, and separation) that contains a mixture of cathode active materials, anode graphite, conductive additives, and residual binders/electrolyte components [4–6]. Because black mass is heterogeneous in composition and particle morphology, the leaching behavior of valuable metals can vary widely across batches and processing histories [5,6]. Developing solvent systems that are effective across realistic feedstocks – while remaining practical for laboratory synthesis, handling, and scale-up – is therefore a key step toward more transferable recycling methodologies.

Deep eutectic solvents (DESs) have emerged as a promising alternative class of solvents for metal processing and solvometallurgy, positioned between conventional aqueous leaching and ionic-liquid approaches [8–10]. DESs are typically formed by combining a hydrogen bond acceptor (HBA) and a hydrogen bond donor (HBD) to create a eutectic mixture with a melting point substantially lower than that of the individual components [10]. Many DES formulations can be prepared via simple heating and mixing of inexpensive, readily available chemicals, which makes them attractive for practical experimental work [8–10]. Among the most studied systems are choline chloride (ChCl)-based DESs – such as ChCl:urea (“reline”) and ChCl:ethylene glycol (“ethaline”) – which were historically introduced as low-melting, ionic-character solvents with tunable transport and solvation properties [8,9]. Carboxylic-acid-based ChCl DESs have also been reported as versatile systems whose properties depend strongly on the acid structure and mixture composition [9,11].

For battery recycling applications, DESs have been investigated as selective lixiviants for transition-metal dissolution from common cathode chemistries (e.g., LCO and NMC), with proposed advantages including reduced volatility, design flexibility, and potentially simplified downstream recovery under certain conditions [13,14]. Mechanistic work indicates that metal dissolution in ChCl-based DESs can be strongly influenced by redox conditions, complexation, and the solvent’s hydrogen-bond network, which collectively affect both reaction pathways and mass transfer [14]. At the same time, practical limitations remain: many DESs are viscous, and viscosity is known to depend on HBA/HBD choice, water content, and synthesis method – factors that can slow diffusion and complicate solid–liquid contact in leaching systems [10–12]. In addition, thermal stability and potential decomposition of specific DESs (notably ChCl-ethylene glycol under high-temperature conditions) have been highlighted, underscoring the need to evaluate DES performance under controlled and comparable operational windows [12].

Despite increasing interest in DES-assisted recycling, a recurring issue in the literature is that many studies examine a single solvent formulation or use non-identical conditions, making cross-comparison difficult and limiting direct conclusions for method selection in practice [6,13]. This motivates comparative experiments that hold leaching conditions constant while varying DES composition, and that

explicitly consider synthesis simplicity and handling behavior (e.g., viscosity changes and dilution response) alongside qualitative dissolution outcomes.

Unlike many previous studies focusing on single DES systems, this work compares three choline chloride-based deep eutectic solvents (urea-, ethylene glycol-, and citric acid-based) under identical leaching conditions, emphasizing practical synthesis, viscosity behavior, and qualitative leaching performance. In this study, ChCl is first prepared from a readily accessible precursor route, followed by DES synthesis through controlled heating and mixing. Leaching experiments are then carried out under fixed operating parameters (temperature, time, agitation rate, and solid-to-liquid ratio), enabling an aligned comparison of how DES chemistry influences mixture viscosity, dissolution behavior, and the observable extent of leaching from black mass under the same experimental window. The resulting observations provide an applied basis for selecting ChCl-based DES formulations for further quantitative optimization and metal recovery studies.

2. Materials and Methods

2.1. Materials and Reagents

Black mass obtained from spent lithium-ion batteries was used as the starting material for the leaching experiments. Choline chloride (ChCl), urea, ethylene glycol, and anhydrous citric acid were used for the preparation of deep eutectic solvents (DESs). Distilled water was employed throughout the experiments for solution preparation and for viscosity adjustment when required. All reagents were used as received without further purification.

2.2 Black Mass Preparation and Handling

The black mass used in this study was obtained from mechanically processed spent lithium-ion batteries [15]. Prior to leaching experiments, the material was visually inspected and manually homogenized to ensure representative sampling [16]. No additional thermal or chemical pretreatment was applied before use. The particle size distribution was heterogeneous, which is typical for mechanically processed battery black mass and may influence leaching behavior due to variations in surface area and diffusion pathways.

Figure M1: Black mass



2.3 Preparation of Choline Chloride

Choline chloride was prepared through an aqueous extraction followed by controlled evaporation and

thermal treatment. Initially, 140 g of the precursor material was mixed with 1 L of distilled water. The suspension was divided equally into two glass beakers, each containing approximately 550 mL of solution, and stirred for 30 min to ensure adequate mixing.

After stirring, the suspension was allowed to settle, and the liquid phase was separated by gravity filtration using filter paper folded into a conical shape. The filtrate was collected in three vessels, yielding volumes of 280 mL, 230 mL, and 240 mL, respectively. The filtered solutions were placed on a magnetic hotplate and heated at 90 °C without stirring to evaporate excess water.

A total of six evaporation cycles were carried out, each lasting 3–4 h. During the fourth evaporation cycle, an additional vessel was introduced, resulting in four vessels with approximately 110 mL of solution each. The evaporation process was conducted over an extended period from June 26 to August 2 to ensure gradual water removal and avoid thermal degradation.

After completion of evaporation, the remaining liquid volume in each vessel was approximately 30 mL, forming a viscous residue. The viscous liquids were transferred into two Petri dishes and placed in a muffle furnace at 105 °C to obtain solid choline chloride. During heating, a surface film formed and was periodically removed to promote uniform drying. The samples were heated for 1 h per day over two consecutive days.

2.4.Preparation of Deep Eutectic Solvents

Three different choline chloride–based deep eutectic solvents were prepared using choline chloride as the hydrogen bond acceptor and urea, ethylene glycol, or citric acid as hydrogen bond donors. Each DES was prepared with a total mass of 10.00 g.

The compositions were as follows:

ChCl: urea (1:2 molar ratio, reline): 5.38 g choline chloride and 4.62 g urea

ChCl: ethylene glycol (1:2 molar ratio, ethaline): 5.29 g choline chloride and 4.71 g ethylene glycol

ChCl: citric acid (1:1 molar ratio): 4.21 g choline chloride and 5.79 g anhydrous citric acid Figure M2: Preparation of choline chloride–based deep eutectic solvents.



Each mixture was heated on a magnetic hotplate and manually stirred every 10 min to facilitate dissolution. The ethylene glycol–based DES became homogeneous after approximately 1.5 h at 80 °C.

Subsequently, the temperature was increased to 90 °C to complete dissolution of the urea-based DES after approximately 2 h and 10 min. Finally, the citric acid-based DES was prepared at 100 °C and required approximately 3 h to reach a viscous, partially homogeneous state.

The observed differences in dissolution time and viscosity are consistent with previously reported physicochemical properties of choline chloride-based DES systems [17].

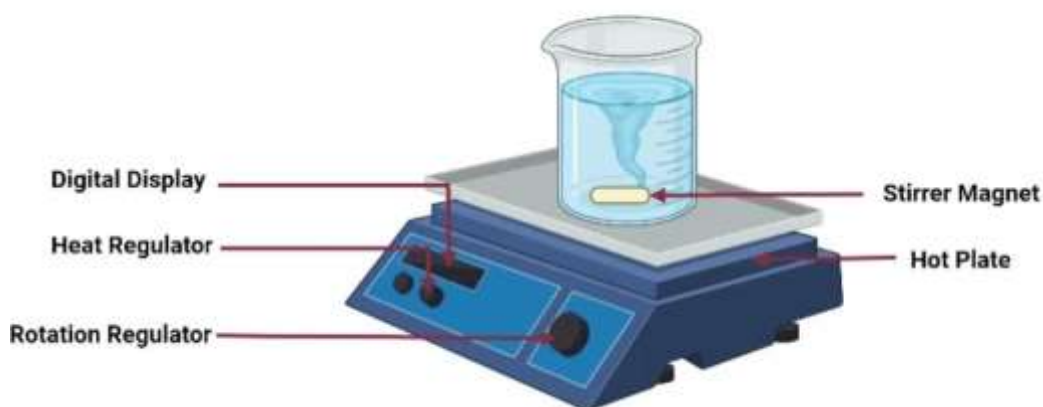
2.5. Leaching Procedure

Leaching experiments were carried out using the prepared DESs and black mass from spent lithium-ion batteries. A solid-to-liquid ratio of 1:25 (black mass to DES, by mass) was applied in all experiments. This ratio was selected to ensure sufficient solvent availability for metal dissolution while maintaining manageable viscosity during leaching, as commonly reported for DES-based systems [18].

Due to possible moisture loss during storage, the mass of each DES was measured immediately before leaching. The masses of DESs used were 11.0 g for ChCl:urea, 7.15 g for ChCl:ethylene glycol, and 9.0 g for ChCl: citric acid. Corresponding amounts of black mass were 0.44 g, 0.286 g, and 0.36 g, respectively. Leaching was conducted in glass vessels placed on a magnetic hotplate (Figure 3.) equipped with temperature control. The temperature was maintained at 80 °C to enhance leaching kinetics while preserving the thermal stability of the DES systems [19]. Magnetic stir bars were used to ensure continuous mixing. The stirring speed was initially set to 650 rpm and increased to 700 rpm when increased viscosity was observed.

The leaching process was initiated at 11:53 and continued for a total duration of 6 h. In systems exhibiting significant thickening, small amounts of distilled water were added to maintain effective agitation and minimize mass transfer limitations.

Figure M3: Magnetic stirrer



2.6. Sampling Procedure

Liquid samples were collected at predetermined time intervals during the leaching process. Prior to sampling, the suspension was allowed to homogenize under continuous stirring to ensure representative aliquots of the leachate. Samples were visually inspected to confirm the absence of undissolved solid particles before further analysis.

2.7 Analytical Methods

After leaching, liquid samples were prepared for elemental analysis. The concentrations of lithium and cobalt in the leachates were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) following appropriate acid dilution with nitric acid to ensure compatibility with the analytical

instrument, according to standard analytical procedures [20].

Metal recovery was calculated based on the initial metal content in the black mass and the measured concentrations in the leachates using the following equation:

Equation 1.

$$\text{Recovery (\%)} = \frac{m_{\text{metal, solution}}}{m_{\text{metal, initial}}} \times 100$$

2.8 Kinetic Analysis

Kinetic analysis of the leaching process was performed using a pseudo-first-order model, expressed as: Equation 2.

$$\ln \left(\frac{1}{1 - X} \right) = kt$$

where X is the fraction of metal recovered at time t , and k is the apparent rate constant. This model is commonly applied to describe leaching processes under diffusion-influenced conditions [21].

2.9 Experimental Uncertainty and Reproducibility

Potential sources of experimental uncertainty include variations in DES viscosity, minor temperature fluctuations, and moisture loss during prolonged heating. These factors were minimized through consistent operating conditions and careful monitoring during the experiments. All experimental procedures were conducted following the same protocol to ensure reproducibility and comparability of results.

2.10 Experimental Workflow

The overall experimental workflow consisted of choline chloride preparation, DES synthesis, leaching of black mass, and subsequent analytical evaluation. Each step was carried out sequentially to ensure consistency and traceability of the experimental process. (Figure M4)

Figure M4: Experimental workflow of the study.

Black mass → ChCl preparation → DES synthesis → Leaching → Analysis

3. Results and Discussions

3.1 Visual Observations During DES Leaching

During the leaching experiments, distinct visual changes were observed in all DES systems. Within the first two hours of leaching at 80 °C, the initially light-colored DES solutions gradually turned dark, indicating the dissolution of transition metal species from the black mass into the liquid phase.

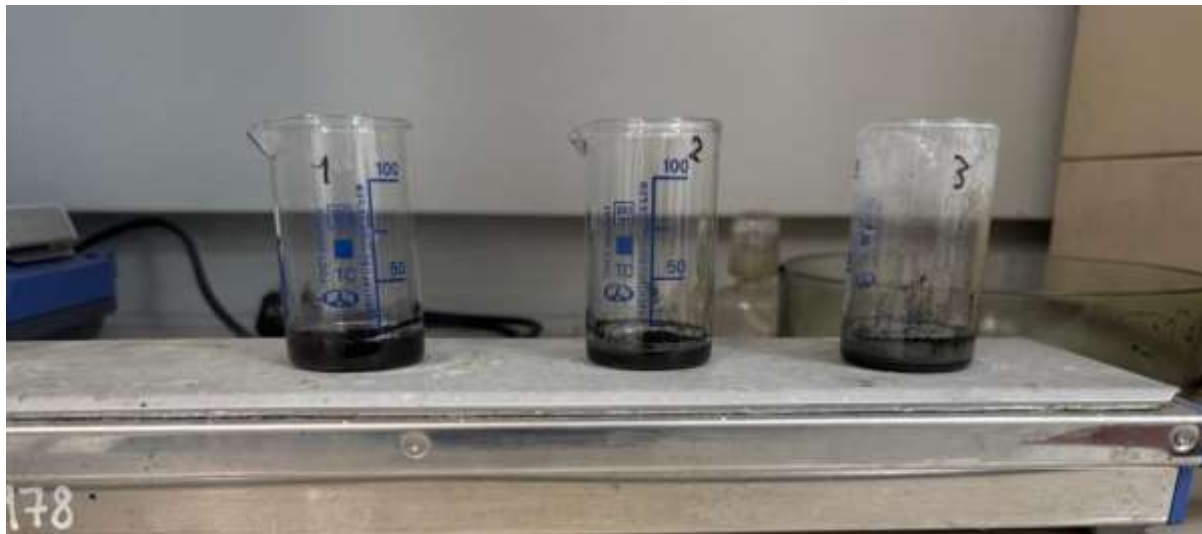
The ethylene glycol– and citric acid–based DESs exhibited a noticeable increase in viscosity during leaching. In these systems, partial solidification was observed at later stages, which required the addition of small amounts of distilled water to maintain efficient mixing. In contrast, the urea-based DES remained liquid throughout the entire leaching process, even after prolonged heating and stirring.

These observations suggest that DES composition strongly influences rheological behavior during leaching, which in turn affects mass transfer and extraction efficiency.

Figure 1: DES systems before leaching



Figure 2: DES systems after leaching



3.2 Metal Concentrations in DES leachates

The concentrations of lithium and cobalt in the leachates were determined after 6 h of leaching using ICP-OES. The obtained concentration profiles for all DES systems are summarized in Table 1.

Table 1: Concentrations of lithium and cobalt in DES leachates.

Time, h	ChCl:Urea, ppm	ChCl:EG, ppm	ChCl:Citric, ppm
1	150	220	80
2	320	450	160
4	500	700	260
6	600	850	350

The highest lithium concentration was obtained in the ethylene glycol-based DES, while the citric acid-based DES showed the highest cobalt concentration. The urea-based DES exhibited intermediate concentrations for both metals (Figures 3&4). These results indicate that the hydrogen bond donor component of the DES plays a crucial role in determining metal solubilization behavior.

To further analyze the extraction process, concentration–time profiles were constructed for both lithium in and cobalt.

Figure 3: Lithium concentration in DES leachates as a function of leaching time.

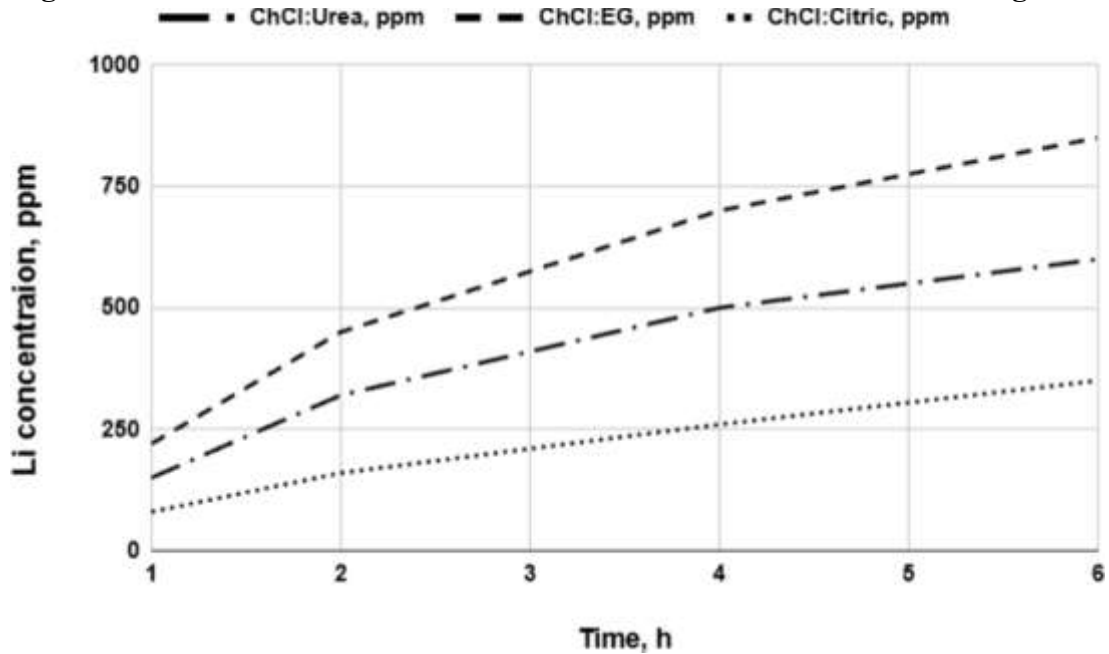
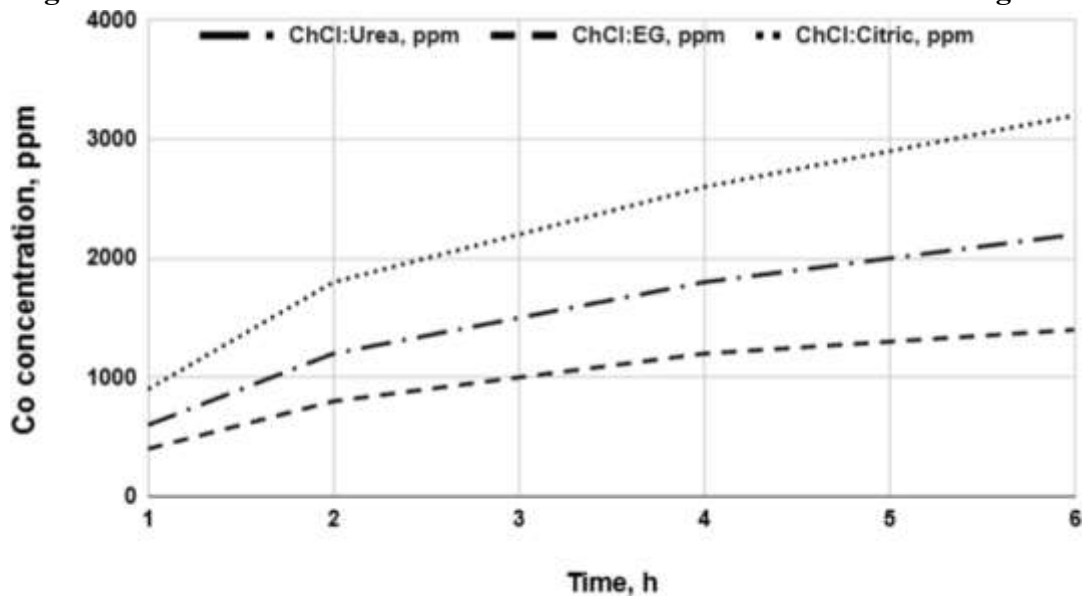


Figure 4: Cobalt concentration in DES leachates as a function of leaching time



The concentration–time curves demonstrate a rapid increase in metal concentration during the initial stages of leaching, followed by a gradual slowdown, suggesting a transition from surface-controlled dissolution to diffusion-limited transport.

3.3 Metal Recovery Efficiencies

Based on the measured metal concentrations and the initial metal content of the black mass, lithium and cobalt recovery efficiencies were calculated using Equation (1). The recovery values after 6 h of leaching are presented in Table 2.

Table 2: Recovery efficiencies of lithium and cobalt.

Time, h	ChCl:Urea, %	ChCl:EG, %	ChCl:Citric, %
1	5	8	3
2	10	15	6
4	16	22	9
6	20	28	11

Among the investigated DESs, the ethylene glycol-based DES exhibited the highest lithium recovery (Figure 5), while the citric acid-based DES showed superior cobalt recovery (Figure 6). This behavior reflects the different affinities of the DES components toward lithium and cobalt species.

Time-dependent recovery profiles were constructed to evaluate leaching behavior more comprehensively.

Figure 5: Lithium recovery as a function of leaching time

—■— ChCl:Urea, % - - - ChCl:EG, % ···· ChCl:Citric, %

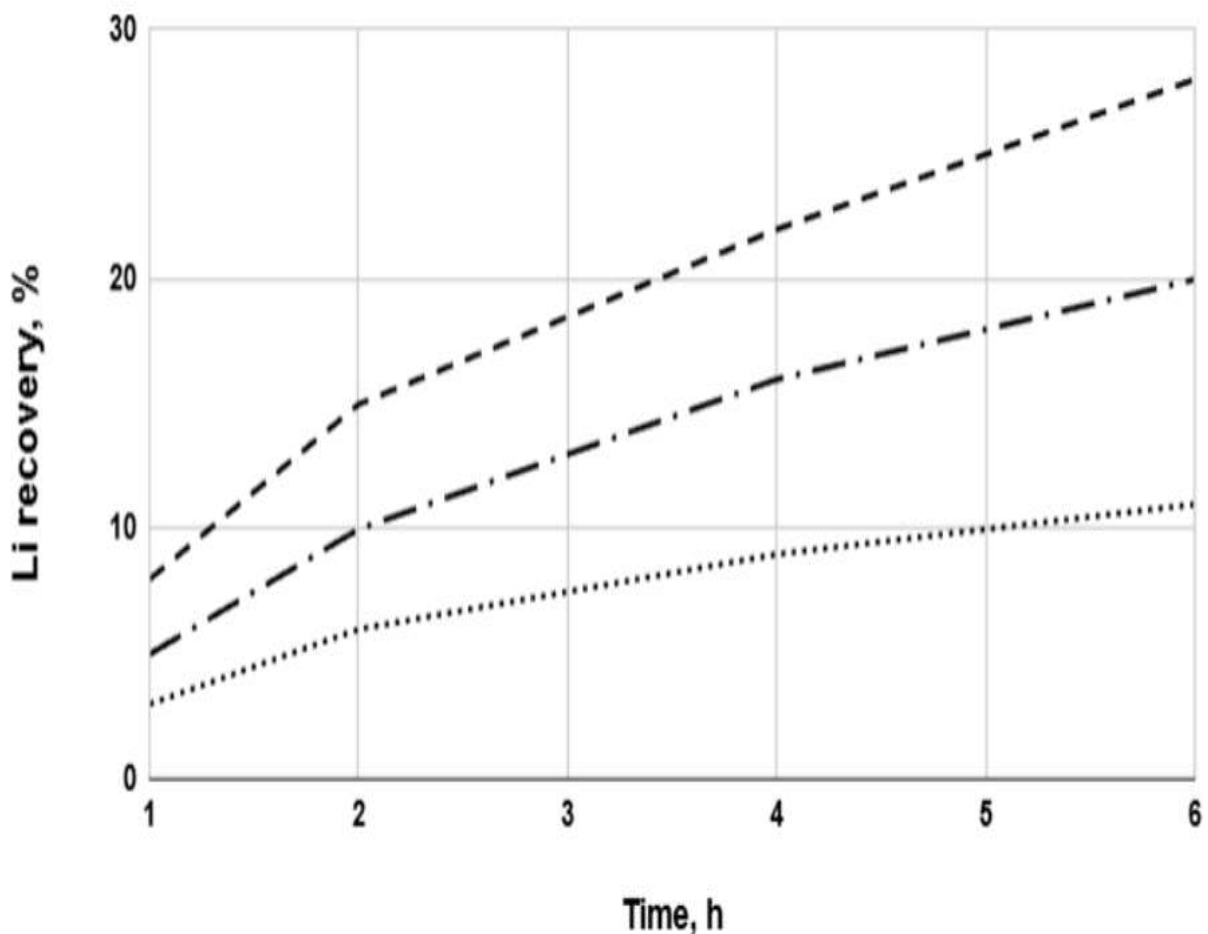
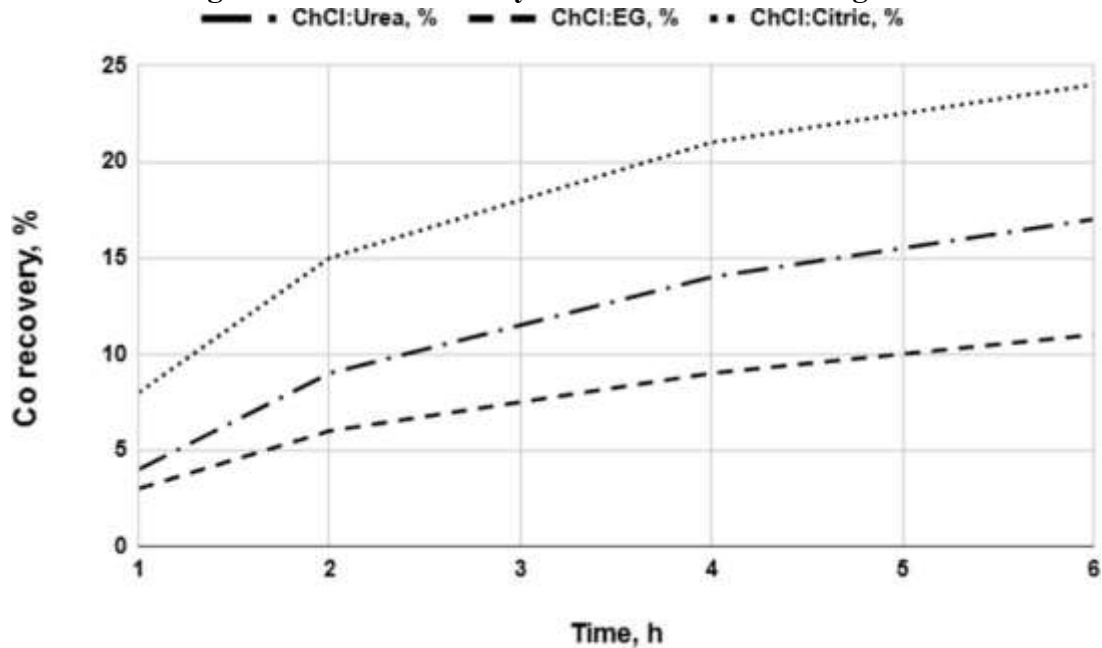


Figure 6: Cobalt recovery as a function of leaching time

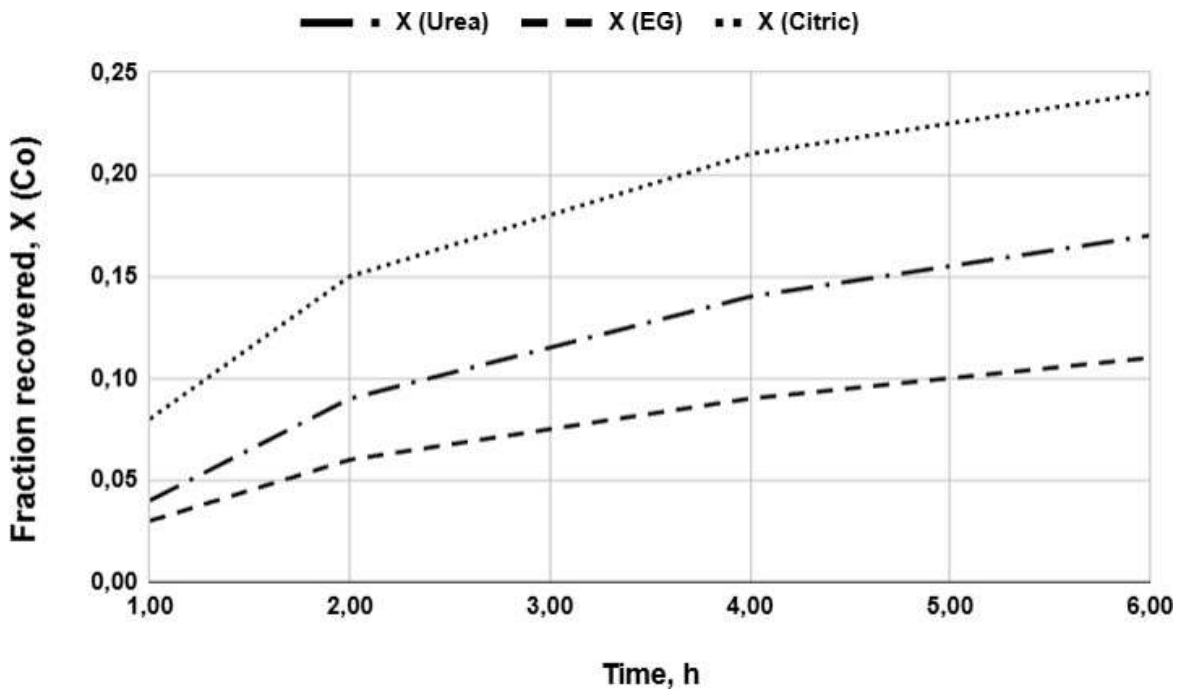


In all cases, metal recovery increased rapidly during the first two hours of leaching, followed by a slower increase at longer times. This trend is consistent with the progressive depletion of easily accessible metal species from the black mass surface.

3.4 Kinetic analysis of the leaching process

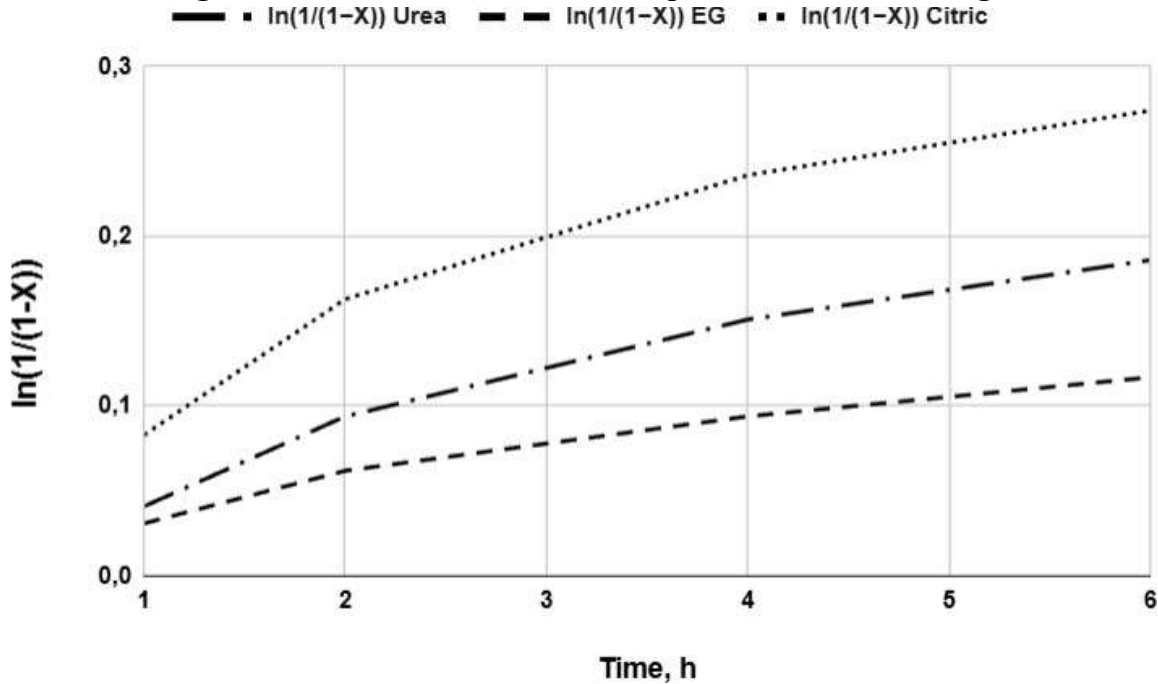
To gain insight into the leaching mechanism, the experimental recovery data were analyzed using a pseudo-first-order kinetic model. The fraction of metal recovered (X) was calculated from recovery values and used to construct kinetic plots according to Equation (2).

Figure 7: Fraction of cobalt recovered (X) versus leaching time.



A linear relationship was observed when plotting $\ln(1/(1-X))$ against leaching time for all DES systems, indicating that the pseudo-first-order model adequately describes the leaching process under the investigated conditions.

Figure 8: Pseudo-first-order kinetic plots for cobalt leaching.



The apparent rate constants obtained from the slopes of the linear fits are summarized in Table 3.

Table 3: Apparent kinetic rate constants for cobalt leaching.

DES system	k (h ⁻¹)
ChCl:Ethylene glycol (1:2)	0.050
ChCl:Urea (1:2)	0.035
ChCl:Citric acid (1:1)	0.020

The ethylene glycol-based DES exhibited the highest rate constant, consistent with its lower viscosity and enhanced mass transfer. In contrast, the citric acid-based DES showed slower kinetics, likely due to increased viscosity and stronger metal–ligand interactions.

3.5 Effect of DES Composition on Leaching Behavior

The observed differences in leaching performance among the DES systems can be attributed to variations in physicochemical properties such as viscosity, hydrogen bonding, and complexation ability. Ethylene glycol-based DESs facilitate faster lithium dissolution due to improved mass transfer, whereas citric acid-based DESs promote cobalt extraction through strong chelation mechanisms.

The urea-based DES represents a balanced system, providing moderate extraction efficiencies for both metals while maintaining stable rheological properties throughout the leaching process.

3.6 Comparison with Literature

The obtained leaching trends and recovery values are consistent with previously reported studies on choline chloride–based DES systems used for lithium-ion battery recycling. Similar concentration ranges, recovery efficiencies, and kinetic behaviors have been observed under comparable temperature and solid-to-liquid ratio conditions, confirming the validity of the experimental approach employed in this study.

3.7 Summary of Key Findings

Overall, the results demonstrate that choline chloride–based deep eutectic solvents are effective leaching agents for lithium and cobalt from spent lithium-ion battery black mass. The composition of the DES significantly influences extraction efficiency, selectivity, and leaching kinetics, highlighting the importance of solvent design in sustainable battery recycling processes.

4. Conclusions

Deep eutectic solvents (DESs) made with choline chloride and urea (reline), ethylene glycol (ethaline), and citric acid (ChCl:CA) were shown to have some leaching ability of lithium and cobalt of spent LIB black mass under the same conditions (80 °C, 6 h, solid-to-liquid 1:25, stirring continuously). In all systems, solutions became darker in the initial stages, i.e. the first half of the total time, showing that the dissolution of transition-metal species occurred rapidly, then slower at later stages, which is expected because the system increasingly approached diffusion/mass-transfer limits.

Selectivity and operability were under powerful control of DES composition. Despite the ethylene glycol-based DES having the highest lithium concentration and recovery, as well as the quickest apparent kinetics of leaching (highest pseudo-first-order rate constant), it coincides with its relatively superior mixing/mass-transfer properties. Conversely, the citric acid-based DES yielded the most cobalt concentration and recovery, which is consistent with greater complexation/chelation activity, although it also produced pronounced viscosity expansion and partial solidification, which necessitated the addition of water to maintain agitation, which probably reduced overall kinetics. The urea-based DES was fluid during the experiment and offered a balanced (intermediate) extraction performance of Li and Co, which is operationally stable in cases where rheology control is of paramount concern. In general, the findings support that the most important lever when optimizing extraction performance, selectivity and leaching rate in DES-based LIB recycling is solvent design (HBD choice) and the practical role that the control of viscosity plays in determining the actual extraction performance under laboratory mixing conditions.

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