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Closed-loop cobalt recycling from spent lithium-ion batteries based on a deep eutectic solvent (DES) with easy solvent recovery

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ABSTRACT

Efficient recycling technology for the rapid growth of spent lithium-ion batteries (LIBs) is essential to tackle the resources and environmental crisis. Hydrometallurgical approach has attracted extensive research due to its potential to reduce the consumption of energy and threat to the environment. However, the simultaneous realization of green, efficient and closed-loop recycling is still challenging. Herein, we report a closed-loop and highly efficient approach to recycle lithium cobalt oxide from spent LIBs based on a choline chloride:oxalic acid (ChCl:OA) type deep eutectic solvent (DES). An ultrafast leaching process is observed at 180 °C for 10 s with no observable residues. The energy barrier during leaching is calculated to be 113.9 kJ/mol. Noteworthy, the solubility of cobalt ions can be reversibly tuned by simply adding/evaporating deionized water, thus avoiding the addition of precipitant and enabling the easy recovery of the leaching solvent for realizing a closed-loop recycling process. The simultaneous realization of high efficiency, green and closed-loop process is expected to push the DES into practical application for recycling the electrodes of LIBs.

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1. Introduction

In recent decades, there has been an explosive growth for rechargeable batteries, wherein lithium-ion batteries (LIBs) dominate the market due to their light weight, small size, high energy density and low self-discharge rate [1–5]. It's predicted that at least 145 million electric vehicles (EVs) powered by LIBs will be on the road by 2030, up from 11 million in 2020 [6–8]. The accelerated production and the coming of their end of life (EOL) will quickly translate into massive amounts of waste. The global LIBs recycling market is inferred to reach $23.72 billion by 2030 [9–12]. Within the spent LIBs, several metal elements including lithium, cobalt, nickel and copper are identified as expensive strategic resources [13–16]. In particular, cobalt has relatively low reserve in the earth crust (~20 ppm) and distributes in regions with unstable political situations [3,10,17,18]. Furthermore, the rational and effective recycling of cobalt resources is necessary to reduce water and soil contamination [19–21].

Currently, the mainstream industrialized approaches to recycle LIBs are based on pyrometallurgical processes [22–24], which generally need extreme temperature (>1400 °C) and the emission of highly toxic and corrosive gases like HF. Alternatively, hydrometallurgy strategy has been extensively studied due to its potential to achieve the extraction and separation of metal elements with high purity at lower temperature (25–200 °C) [25,26]. The types of hydrometallurgical processes mainly include inorganic acid leaching [27], organic acid leaching [28], as well as alkaline leaching with ammonia coordination [29]. Generally, inorganic acids approach exhibits relatively stronger acidity and higher leaching rate while they introduce severe secondary pollution [30,31]. On the contrary, organic acid leaching or alkaline leaching are environmentally more friendly while their leaching rate require moderate control of the reaction system [32,33]. Despite much progress, a relatively high temperature and high concentration of caustic leaching reagent are often inevitable [34–36]. Besides, to precipitate and separate the dissolved metal ions, it's difficult to reuse the leaching agent due to the addition of precipitant. Therefore, it's necessary to develop a leaching system with simultaneously environmental friendliness, mild reaction condition, high efficiency, as well as easy reusability.

Recently, deep eutectic solvents (DESs) have emerged as a new class of solvents which show unexpected interaction towards various metal compounds [37–39]. DESs are a series of eutectic mixtures of two or more inexpensive and safe components interacting via hydrogen bonds. Compared to conventional sol-

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vents, DESs show unexpected properties including low vapour pressure, high thermal and chemical stability, high tunability and are easy to prepare [40–42]. The properties of DESs are considered to be similar to ionic liquids (ILs) [43], while DESs are distinguished by the presence of a large amount of molecular components (typically hydrogen bond donors) [44]. Noteworthy, deep eutectic solvents are found to show high solubility for various metal oxides [45–47]. It was suggested that the hydrogen bond donor (HBD) species in the DESs can act as oxygen acceptors, thus breaking the metal-oxide bonds [46]. In 2019, a DES composed of choline chloride:ethylene glycol (ChCl:EG) system was reported to extract cobalt ions from lithium cobalt oxides (LCOs) without the need for supporting chemicals [10], providing a proof of concept for the use of these sustainable solvents as vehicles for recycling spent LIBs. It's reported that 94.1% leaching rate was achieved at 220 °C for 24 h. Thereafter, DESs with various compositions and optimized reaction condition have been investigated to further improve the efficiency. For example, ChCl:EG (2.3:1) was reported to dissolve the $LiCoO_2$ at 190 °C for 15 min to reach 99.86% leaching [48]. ChCl:Urea (1:2) could dissolve the $LiCoO_2$ at 180 °C for 12 h to reach 95% leaching [49]. However, the simultaneous achievement of high efficiency, mild reaction condition, and easy solvent recovery is still challenging.

Herein, we report the recycling of LCO using a choline chloride: oxalic acid (ChCl:OA) type DES with high efficiency and easy solvent recovery. Compared with EG based DES, the carboxylate group in OA provides stronger activity as hydrogen bond donor and accelerates the dissolution process, achieving a low energy barrier of 113.9 kJ/mol during leaching LCO. An ultrafast leaching process can be achieved in only 10 s at 180 °C. Alternatively, a leaching rate of nearly 100% can be achieved at 90 °C in 2 h, meeting the demand for the design of mild yet efficient reaction process. Furthermore, the solubility of cobalt ions can be reversibly tuned by adding or evaporating deionized water (DIW) in the solution, enabling the direct precipitation of metal ions and reversible recovery of the leaching solution. The cobalt ions can be precipitated as cobalt oxalate phase, and further be converted back LCO after annealing. The recycled LCO material shows good crystallinity and its electrochemical performance is comparable with the commercialized sample. The simultaneous achievement of high efficiency, mild reaction condition, and easy recovery in such proton-active DES is expected to provide practical pathway for the recycling of LIBs.

2. Experimental

2.1. Preparation of DES

Various types of DESs were obtained by mixing ChCl ($C_5H_{14}ClNO$; 98%; Shanghai Saen Chemical Technology Co., Ltd.) with different hydrogen bond donors at given molar ratio. ChCl:OA ($C_2H_2O_4$; 98%; Shanghai Saen Chemical Technology Co., Ltd.) (1:1) was obtained by mixing and stirring (300 r/min) at 70 °C for 4 h. As control groups, ChCl:EG ($C_2H_6O_2$; 99%; Shanghai Saen Chemical Technology Co., Ltd.) (1:2) and ChCl:Urea (CH_4N_2O; 99%; Shanghai Saen Chemical Technology Co., Ltd.) (1:2) were prepared by mixing the corresponding solids (300 rpm) at 70 °C for 2 and 4 h, respectively (Fig. S1). The obtained DESs were all transparent liquids at room temperature.

2.2. Leaching experiment for evaluating the efficiency

For evaluating the leaching rate of the DESs, 20 mg of cathode material LCO powder ($LiCoO_2$; 99%; Guangdong Canrd New Energy Technology Co., Ltd.) was added to 3 g of the tested DES in a glass vial. The vial was then heated in an oil bath (60–110 °C, 5–480 min). The solution quickly turned blue, indicating the rapid leaching of the solid. To exact the supernatant for quantifying dissolved cobalt elements at given condition, the vial was quickly cooled and then centrifuged at 10000 rpm for 10 min. Ultraviolet-visible spectrometry (UV–Vis, Shimadzu UV-2700) was used for quantifying the concentration of the cobalt elements in the supernatant.

2.3. Extraction of metal elements and the easy recovery of DES

After leaching, the dissolved cobalt elements can be precipitated by directly adding suitable amount of deionized water into the solution. Flocculent precipitate was formed while the solution turned from clear blue to opaque white. After 2 h, the precipitate turned brown and the supernatant turned transparent. The precipitate was centrifuged, washed and dried before being annealed to Co_3O_4 precursor at 400 °C for 5 h as the raw material for $LiCoO_2$ regeneration. The mole ratio of lithium and cobalt content of Li_2CO_3 (98%; Shanghai Saen Chemical Technology Co., Ltd.) and Co_3O_4 is 1.05:1. X-ray diffraction (XRD, PANalytical B. V. Empyrean) was used to characterize the structure of the precipitated powder before and after calcining. Scanning electron microscopy (SEM, HITACHI SU8010) was used to characterize the particle size distribution and surface morphology of the materials before and after precipitation. ^{13}C nuclear magnetic resonance (NMR, JNM-EC400S/L1) was used to study the composition of the leaching solution after repeated leaching/recovering cycles. The easy recovery of the DES can be easily achieved by reversibly controlling the water content, which means the supernatant can be reused after evaporating the deionized water at 90 °C for 18 h.

2.4. Electrochemical tests

The working electrodes were prepared by slurry coating process. The slurry contained 80 wt% active materials, 10 wt% acetylene black (AB) and 10 wt% poly vinylidene fluoride (PVDF) dissolved in *N*-methyl pyrrolidinone (NMP). The slurry was coated on aluminum foil, and then vacuum-dried at 90 °C for 12 h. They were then punched using a 12.7 mm φ disc punch (Ted PELLA, INC) and weighted with a microbalance with the readability of 0.001 mg (MSA6.6S-000-DM, Sartorius). The average loading of the electrode is 1.0 ± 0.2 mg. The punched electrodes were then assembled in an argon-filled glove box (Mikrouna Universal). The coin-type cells (CR2032, Panasonic) used lithium sheets as counter electrodes, microporous polypropylene film (Celgard 2400) as the separators, and 1.0 M $LiPF_6$ assembled in EC:DMC (3:7) as the electrolyte were used for electrochemical tests. All electrochemical tests were carried out at 20 °C. The cycle test and rate capability were performed on LANHE CT3001A battery tester. The cyclic voltammetry (CV) and EIS were tested on CHI660E electrochemical workstation.

3. Results and discussions

3.1. Green, efficient and closed-loop recycling of cobalt

The simultaneous realization of being green, efficient and reusable is desirable yet challenging for the hydrometallurgical recycling of LIBs [50–54]. This work reports the reversible dissolution/precipitation of cobalt elements in ChCl:OA type DES with mild property (without strong acid) yet high efficiency, as shown in Fig. 1 (The main reactions involved in the recovery process are given in the Supporting Information). The commercial LCO powder with ellipsoidal morphology and size of about 10 μm was used as the standard sample for studying the leaching

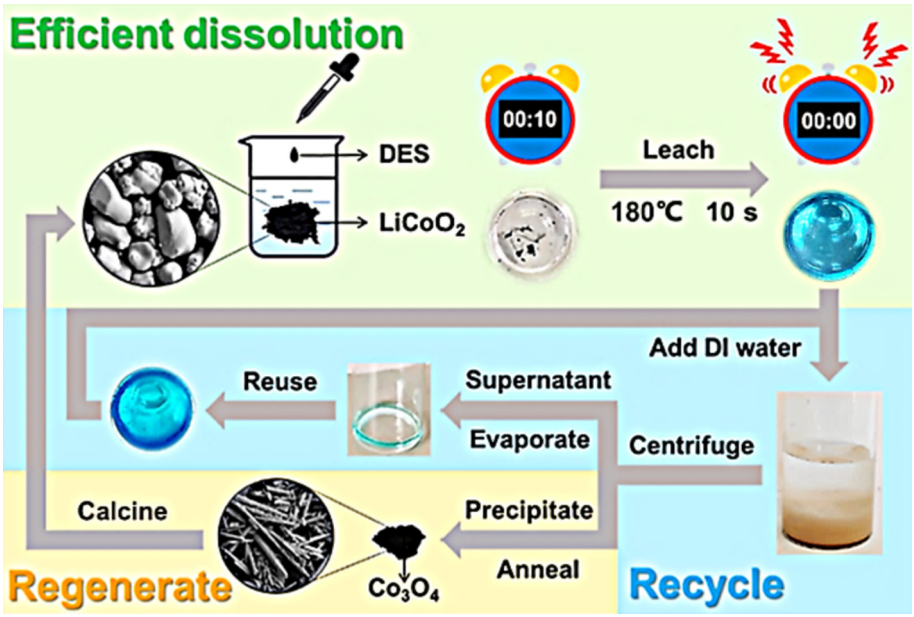

Fig. 1. Schematic illustration of the highly efficient recycling process with solvent reusability enabled by the ChCl:OA DES system. Noteworthy, ultrafast leaching of LCO can be achieved in 10 s at 180 °C, indicating an excellent kinetics. Meanwhile, the solubility of cobalt ions can be reversibly tuned by adding/evaporating deionized water, realizing the precipitation of cobalt ions without the introduction of other precipitant, thus enabling the easy recovery of the supernatant solvent for reusability.

process. While previously DESs were reported to dissolve LCO in hours or days [10,48,49], we observed that 0.02 g LCO powder can be completely dissolved in 3 g ChCl:OA DES solvent (preheated to 180 °C) in an ultra-short time of 10 s. A large number of small bubbles were observed upon the addition of LCO powder (Movie S1 and Fig. S2), indicating a violent reaction kinetics. The solution instantly changed from colorless to transparent blue. Alternatively, the leaching process can also be performed at more controllable condition with reasonable efficiency (90 °C, 2 h). After leaching, to precipitate the cobalt ions from the solution, it's necessary to maintain the reusability of the leaching solution. DESs are known to provide a rich variety of complexing environment for a series of transition metal ions and tune their properties [38,47,55–57]. In the ChCl:OA system, we observed that the solubility of cobalt ions can be reversibly controlled by directly adding or evaporating deionized water (DIW). The transparent blue solution obtained by leaching LCO, once being added with 13 mL DIW, turned opaque immediately. The light pink floccule gradually precipitated while the supernatant solution turned colorless (Movie S2 and Fig. S3), indicating the separation of the cobalt ions from the solution.

After centrifugation, the solids were washed, dried, and then converted to Co_3O_4 by annealing in the air at 400 °C for 5 h, and further recovered to pristine LCO by annealing with Li_2CO_3 at 600 °C for 5 h and then 900 °C for 10 h in a muffle furnace. The reversible extraction of Li can be achieved by pumping CO_2 into the solution after the extraction of Co (Fig. S4). This process generates white crystals, which can be easily filtered. For the supernatant liquid, the leaching capability can be recovered by simply evaporating the excessive deionized water at 90 °C. The recovered ChCl:OA DES showed well solubility towards LCO, endowing the realization of a closed-loop recycling protocol.

3.2. Kinetics analysis of the cobalt leaching process in ChCl:OA

In order to quantify the leaching kinetics of LCO in ChCl:OA DES, we systematically studied the leaching process at various conditions. Briefly, 0.02 g LCO powder was added into 3 g ChCl:OA DES in a glass vial and then heated at various temperature for a given time period (60–110 °C for 5–480 min) as shown in Fig. 2 (a). Accompanied with the dissolution process of the cobalt elements, the solution turned from colorless to light green and then to blue, indicating the conversion of the cobalt elements in solid

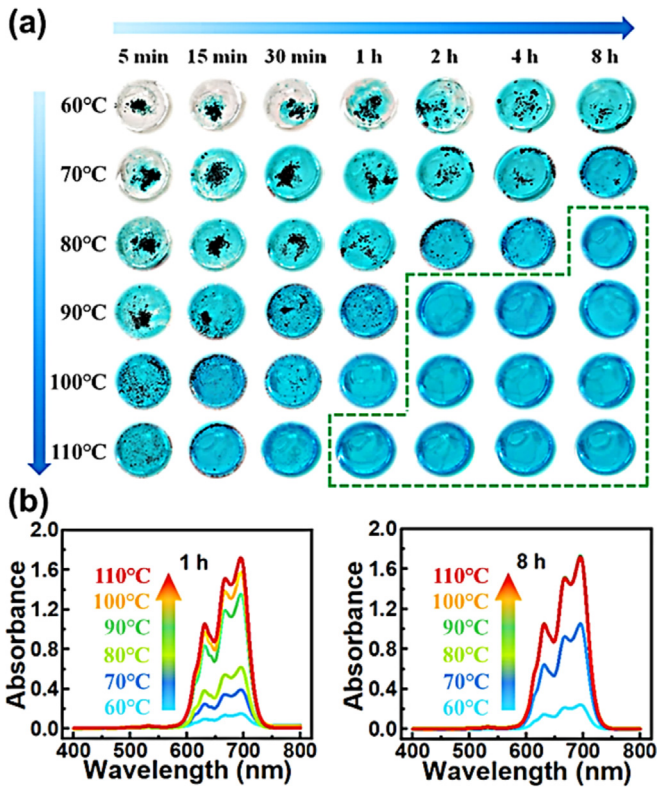

Fig. 2. Leaching of LCO in ChCl:OA DES at various conditions. (a) The digital pictures of the vial bottoms containing the reactants (LCO powder in ChCl:OA DES solvent) reacted at various conditions (60–110 °C, 5–480 min). The dotted marks the critical condition for complete dissolution. (b) The UV-Vis absorbance of the leaching solution at given conditions (60–110 °C, 1 and 8 h).

LCO to ionic cobalt (II) chloride complex [55,58,59]. UV-Vis was used to quantify the concentration of the dissolved ions. The solutions showed three characteristic bands (630, 667, and 696 nm) in the UV-Vis spectra, which corresponded to the absorption of $[CoCl_4]^{2-}$ species [10,60]. The intensity of the peak was used to quantify the concentration of the cobalt elements. Firstly, solution with various known concentration was used to obtain the standard concentration curve (Fig. 3a). The UV-Vis spectra of reacted solutions at different stages (Fig. 2b and Fig. S5) were then measured and used to calculate the concentration of dissolved elements. It's found that the absorption spectra of samples with sufficient reaction driven forces (80 °C 8 h, 90 °C 2 h, 100 °C 2 h, and 110 °C 1 h) overlapped with each other, indicating the complete dissolution states. It's consistent with the results from direct visual observation (dotted line in Fig. 2a). The leaching rate can be further improved by mechanical stirring (Fig. S6). In addition, the viability of our leaching protocol for practical electrodes containing additives (for example, 80 wt% cathode materials, 10 wt% binder and 10 wt% conductive agent) was confirm (Fig. S7). The leaching process of the LCO component is similar to pure LCO powder, while insoluble additives exist as solids and can be easily filtered. Based on the UV-Vis spectra, the leaching rate (ratio between the dissolved and input cobalt elements) can be calculated by Formula S1 (Fig. 3b). It's seen that at temperature below 70 °C, the leaching process is slow while at 90 °C or above the leaching is considerably accelerated.

To further understand the control steps and the fundamental mechanism of the leaching process, it is necessary to analyze the leaching kinetics of the system. The leaching of LCO in the DES system can be considered as a solid–liquid leaching process, within which the cobalt elements convert from solid state crystal cobalt to ionic $[CoCl_4]^{2-}$ complex [61]. The particle size decreases gradually until complete dissolution. This reaction process can be described with the model of unreacted nucleus contraction [62]. The control steps of leaching rate in this model are mainly divided into diffusion control, surface chemical reaction control and mixture control. Based on the leaching rate at various reaction conditions, the apparent reaction rate at each temperature can be calculated (Formula S2). Therefore, the corresponding activation energy (E_a) can be calculated (Formula S3) to be about 113.9 kJ/mol by fitting -ln$K$ to 1000/T (Fig. 3c and d), which is essential for realizing efficient leaching. Meanwhile, in the tested temperature range of 60–110 °C (333–383 K), the intercept corresponds to the negative logarithm of the pre-exponential factor (–32.1). Noteworthy, the E_a controlled by chemical reactions is generally higher than 50 kJ/mol. Mathematically, the E_a can be used to describe to what degree the reaction rate is influenced by temperature. It was reported that in 1.5 mol/L lactic acid:0.5 vol% H_2O_2 systems (70 °C for 20 min), the activation energies for the leaching of Li, Ni, Co, and Mn are 62.81, 63.96, 62.83, and 70.62 kJ/mol, respectively [63]. In another report, the leaching behavior of Ni and Co in ammonia, ammonium carbonate and ammonium sulfite appeared to follow the reaction controlled shrinking core model with the activation energy of Ni and Co being 57.4 and 60.4 kJ/mol respectively (for 40 min) [64]. Our reported E_a is apparently larger, indicating that the leaching of Co in ChCl:OA system can be easily controlled by adjusting the temperature, providing a greener and more biodegradable DES as controllable leaching solution without additional reducing agent. Compared to other reported DES leaching solutions, the selected DES in this work contains oxalic acid which is reductive and has active protons. The dissolution reaction process at the interface between the LCO and the ChCl:OA can be roughly considered as two stages: the leaching process which converts the cobalt elements in the solid lattice into ionic cobalt, as well as the reduction process which continuously transform Co^{3+} to Co^{2+} whose complexing compounds have higher solubility [65,66]. The active protons and the reductive characteristics of OA are inferred to promote the breaking of the original metal-oxide bonds and benefit the leaching rate.

3.3. Closed-loop recycling realized by reversibly tuning the solubility of the cobalt ions

One key challenge of conventional hydrometallurgical approach for recycling LIBs is the difficulty to achieve the recovery of the leaching solvent due to the addition of precipitant to extract the

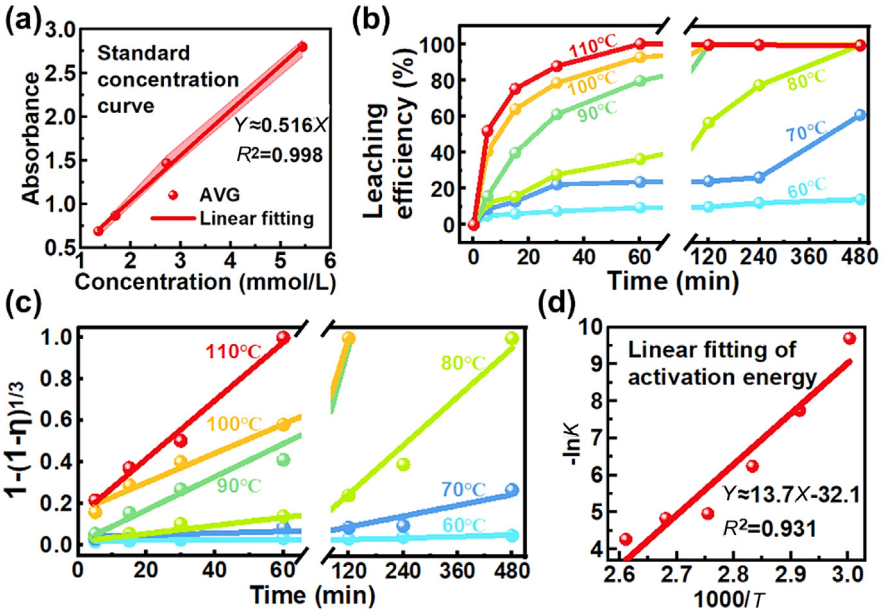

Fig. 3. (a) The variation range of absorbance of LCO powder completely dissolved under many experiments, and takes the average absorbance of different concentrations to make the linear fitting curve. (b) Expresses the leaching rate of solution under different conditions (60–110 °C, 5–480 min). (c and d) The fitting of activation energy in the leaching process.

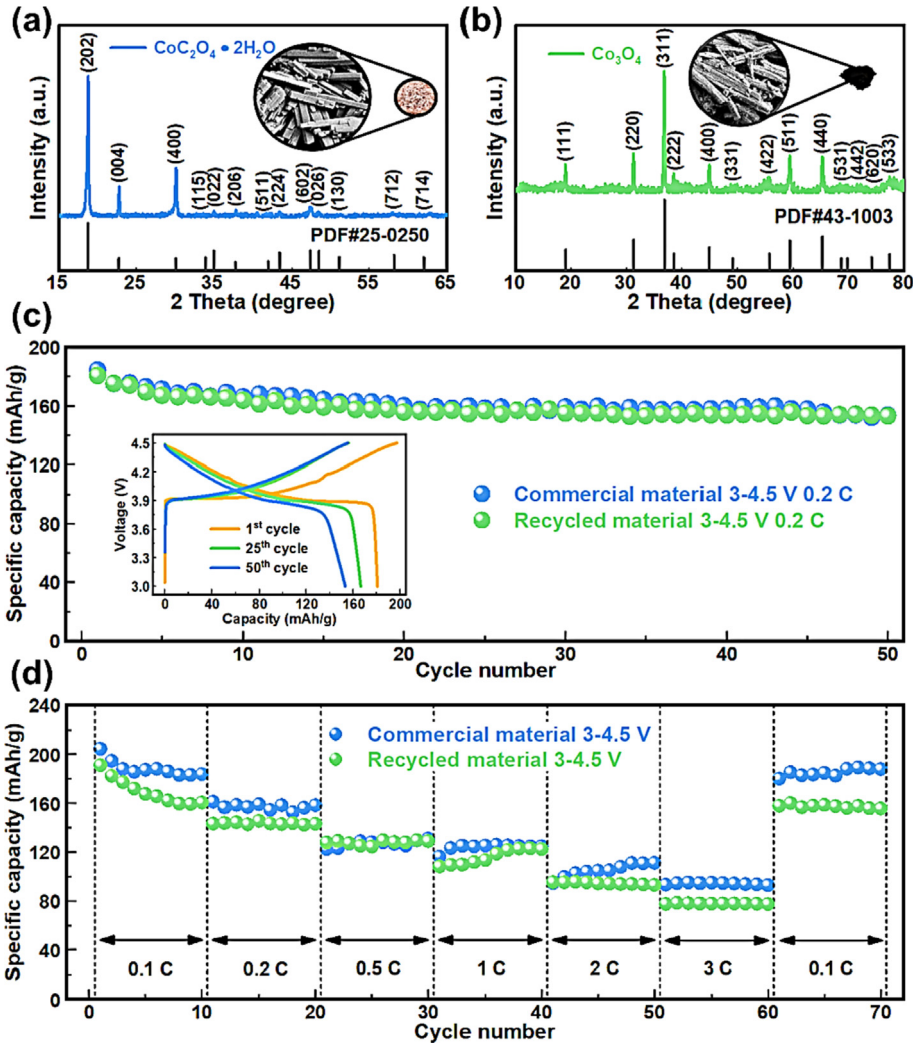

Fig. 4. (a and b) The XRD and SEM of the precipitate before or after calcination. (c) The cycle test of the recycled material and the commercial material. (d) The rate capability of the recycled material and the commercial material.

metal elements. This is even less studied for DES system. Herein, we show that the solubility of cobalt elements in ChCl:OA can be reversibly tuned by adding/evaporating deionized water in the system, thus enabling the precipitation of the cobalt ions while not introducing additional precipitant (Fig. S8a). By adding deionized water into the solution after leaching, flocculent $CoC_2O_4 \cdot 2H_2O$ can be observed. After centrifugation, for the supernatant solution, the deionized water content can be directly evaporated. The regenerated DES after evaporating DIW is light-green. It shows restored capability for LCO leaching. To understand the underlying mechanism for the reusability of the system, we used ^{13}C NMR to reveal the evolution of composition for the leaching solution (Fig. S8b) [67–69]. Compared with the pristine ChCl:OA DES, the absorption peak for the stretching vibration of OA's carboxyl group weakened and shifted with the increase of the cycle numbers during the repeated leaching/recovering process, indicating that the hydrogen bond effect of OA was weakened, revealing the consumption for certain amount of the OA. Meanwhile, no observable changes occur for other signals, indicating that the structure of the ChCl remains stable, which provides essential chemical mechanism for the reversible recycling. As a result, the leaching/extraction of the cobalt elements can be realized without the introduction of additional precipitant, enabling the DES to be easily restored for repeated usage, which is necessary for the realization of a closed-loop recycling process with practical value.

3.4. Structure and electrochemical properties of the recycled LCO

To evaluate the quality of the recycled LCO, the precipitate was repeatedly washed, centrifuged, and dried in oven to generate a light-pink powder. XRD and SEM analysis (Fig. 4a and Fig. S9) showed that the powder was $CoC_2O_4 \cdot 2H_2O$ with rod-like morphology. By annealing the $CoC_2O_4 \cdot 2H_2O$ at 400 °C for 5 h in air, black powder was obtained with Co_3O_4 phase (Fig. 4b and Fig. S10). The morphology of the Co_3O_4 resembles that of the $CoC_2O_4 \cdot 2H_2O$ precursor, while pores were formed due to the topological shrinkage of the lattice. Afterward, the Co_3O_4 was annealed with Li_2CO_3 at 600 °C for 5 h and then 900 °C for 10 h. The recycled LCO material has high purity, good crystallinity (Fig. S11) and bulk morphology (Fig. S12). The electrochemical performance of the recycled LCO was evaluated using coin cells. The CV and EIS spectrum (Fig. S13) of the recycled LCO has similar redox pairs and impedance with the commercial LCO, indicating the electrochemical kinetics of the recycled LCO is similar to the commercial materials. As shown in Fig. 4(c), at the current density of 0.2C and voltage range of 3.0–4.5 V, the recycled LCO shows an initial discharge capacity of 180.7 mAh/g. After 50 cycles, it retains 84.8% of its initial capacity. The rate capability is tested by cycling the cells at various current densities, as shown in Fig. 4(d). It's observed that the regenerated LCO has lower capacity than the commercial LCO at low current density of 0.1C and 0.2C, while their capacity at higher

current density of 0.5C–3C is similar. This indicates the smaller particle size of the regenerated LCO is beneficial for its rate capability. Meanwhile, at a charge/discharge rate of 3C, the recycled LCO shows a discharge capacity of about 94 mAh/g, indicating a decent electrochemical kinetics. The high efficiency and closed-loop recycling, as well as the satisfying electrochemical performance of the recycled LCO are expected to enable the DES system for the practical recycling of LIBs.

4. Conclusions

This work demonstrates the successful realization of a closed-loop, green and highly efficient recycling process for lithium cobalt oxide from wasted LIBs based on a ChCl:OA type DES. The reusability of the solvent after leaching relies on the tunable complexing environment of the system, which allows the reversible regulation of the solubility for the cobalt ions by simply adding/evaporating deionized water and avoids the addition of precipitant. Besides, the ChCl:OA type DES contains no inorganic strong acid while the leaching rate is high with a reaction barrier of 113.9 kJ/mol. The recycled LCO has a comparable electrochemical performance with commercial sample. The simultaneous achievement of closed-loop, green and high efficiency is expected to push DES into practical applications.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jechem.2022.05.008.

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