



Recovery of lithium and copper from anode electrode materials of spent LIBs by acidic leaching

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Abstract

In view of the importance of environmental protection and resource recovery, recycling of spent lithium ion batteries (LIBs) is quite necessary. In the present study, lithium and copper are recycled to lithium carbonate and copper oxide from anode electrode material of the spent LIBs. The anode electrode material is firstly treated with hydrochloric acid to leach lithium (96.6%) and then with nitric acid to leach copper (97.6%). Furthermore, lithium and copper are recovered as lithium carbonate and copper oxide from their respective solutions using precipitation and calcinations. These synthesized products are further characterized using XRD, FE-SEM, and EDX analysis. Finally, a simple process is proposed for the recovery of lithium and copper from anode electrode material of spent LIBs.

Keywords Spent lithium ion batteries · Recovery · Recycling · Leaching · Anode

Introduction

Sony Corporation firstly introduced lithium ion batteries in early 1990s (Castillo et al. 2011; Paulino et al. 2008). Lithium-ion batteries (LIBs) are extensively used as a power source in many portable electronic devices such as digital cameras, mobile phones, computers, and electric vehicles (Nan et al. 2005, 2006; Cabral-Neto et al. 2023). They are used because of following features like high energy density, high cell voltage, lighter weight, low self-discharge rate, and wider operating temperature range (Jha et al. 2013; Zhang et al. 2014). Rapid urbanization and digitalization has stimulated the production and consumption of LIBs. By 2035, an amount of 6.76 million is expected for the global LIB market

(Palacin and deGuibert 2016; Zhang et al. 2018). With 1.2 billion users, the largest mobile phone market is India, leading to the huge consumption and demand of LIBs by 2030 (Upadhyay 2019; Trends in Lithium-ion Battery Reuse and Recycling 2019).

The bulk disposal or landfilling of waste LIBs causes soil and water contamination due to release of electrolytes and heavy metals, leading to a threat to the ecological and environmental sustainability (Chen et al. 2015). Moreover, health effect due to air pollution is resulted by the emission of gases due to discharge of lithium (Pathak et al. 2017). Therefore, waste management agencies as well as environmentalists are highly concerned toward the challenge posed for management of spent LIBs effectively (Meshram et al. 2020). Valuable metals, organic compounds, plastics, and other materials are included in spent LIBs; the contents vary depending on the manufacturer and kind of battery (Shin et al. 2005). Moreover, the metal values locked in spent LIBs can be of great interest as an alternative resource if recycled appropriately (Srivastava and Pathak 2020). The main focus of the treatment technologies is on cathode material in order to recover various precious as well as valuable metals (Islam et al. 2021; Porvali et al. 2019; Zhuang et al. 2019; Lu et al. 2022; Chen et al. 2023), whereas only scarce studies are available on recycling and recovery from anode materials (Natarajen et al. 2018; Guo et al. 2016; Yang et al. 2021).

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The lithium content is above the environmental standards in the anode material of the LIBs. In the long-term perspective, the lithium recovery is to obey environmental laws and ecological benefits, as the lithium content and market price are low (Meshram et al. 2014). On the other hand, copper is also present in the anode material of spent LIBs. With an annual increase of around 2.8%, since 1965, every 25 years the production of copper is doubled. Copper is essential for many applications such as in wires for distribution, transmission, and power generation. By 2050, an excess of copper will be required in order to serve the global electrical requirements to establish the system with low carbon energy (Henckens and Worrell 2020). Therefore, recycling of copper from waste products is also important.

Different recycling methods such as pyrometallurgical and hydrometallurgical methods have been used for the recovery of metals from spent LIBs and other matrices. The recovery of valuable metals by pyrolysis is disadvantageous of higher temperatures as well as partial separations only, which requires again processing and refining. Therefore, in view of low energy consumption and higher selectivity, researchers prefer hydrometallurgical methods over pyrometallurgical (Jha et al. 2013; Meshram et al. 2020; Kumar et al. 2023; Dhiman et al. 2024).

Some researchers synthesized LIB anode from nanofibers of porous carbon coated with poly(3,4-ethylenedioxythiophene/manganese oxide (Abdah et al. 2021). Amici et al. (2021) employed polymer electrolyte based on nanosponge composite gel for anode material in LIBs. A dangerous polarization was reported, when the liquid electrolyte in the standard cell was placed with a commercial separator. The sulfur cathode and silicon anodes were developed using micro-mesoporous carbons of cyclodextrin nanosplices (Alidost et al. 2021). A few studies are also focused on the development of membranes using biobased gel polymer electrolyte in order to fabricate potassium ion batteries. It is useful for next generation technology for sustainable batteries (Manarin et al. 2022). Zhang et al. (2024a, b) applied metal organic framework on the enhancement of performance of LIBs. The results suggest that the framework not only enhances battery life cycle and cycle stability but also inhibits formation of lithium dendrites. Similarly, high-performance batteries of zinc-metal were developed using cross-linked cellulose hydrogel electrolyte (Zhang et al. 2024a, b).

A thorough literature survey on the lithium and copper recovery from anodic substances of spent LIBs has indicated that none of previous studies has investigated two acids in a single leaching process and none of studies recovered lithium and copper as lithium carbonate and copper oxide from anode materials of spent LIBs. Henceforth, in the present study a simple method was developed for the recovery of lithium and copper from the

anode material of spent LIBs. After the pre-treatment of spent LIBs, anode material was separated from the copper layer from anode. The anode electrode material was leached with different mineral acids to optimize best leaching agent for lithium and copper. Finally, the solution of lithium after leaching with HCl was used to precipitate lithium as lithium carbonate. Thereafter, copper recovered as copper oxide from the solution of copper after leaching with HNO_3 in the next step. The prepared materials have been characterized through XRD, FESEM, and EDX.

Experimental

Material and methods

The spent LIBs used in this research were collected from the local market. Sodium hydroxide, sodium carbonate, nitric acid (HNO_3), sulfuric acid (H_2SO_4), and hydrochloric acid (HCl) employed in the study are analytical grade and procured from Merck. All the solutions have been prepared using ultrapure water. For the assurance of reproducibility, the experiments were conducted in triplicates.

Instrument

Inductively coupled plasma–mass spectrometer (ICP-MS, Agilent 4650) was employed to check metal ion concentrations in digested sample and leach liquors. The crystal structure of synthesized lithium carbonate and copper oxide was analyzed by X-ray diffraction (XRD), using Bruker diffractometer (AXS D8) with copper- $\text{K}\alpha$ ($\lambda = 1.54 \text{ \AA}$) in the range of $10\text{--}80^\circ$ (2θ) at a scanning rate of 2° per minute. A scanning electron microscope (Jeol 6380LV) with 15 kV accelerating voltage was applied to examine the morphology of the synthesized products. EDX spectrometer connected to SEM was employed to measure the chemical composition.

Dismantling, digestion, and leaching methods of spent LIBs

Dismantling

The different parts (anode, cathode, and separator) were separated after mechanical dismantling of spent LIBs of mobile phones collected from local market. The anode materials were then collected from anode (copper foil) and dried for 12 h at 60°C to remove the moisture (Natarajan et al. 2018). Thereafter, a sieve of $75 \mu\text{m}$ was used to get the fine powder of the crushed residue.

Digestion

To quantify the metal contents in anode materials, 1 g of anode powder was digested twice with 20 mL aqua regia ($\text{HCl}/\text{HNO}_3 = 3/1$, v/v) for 2 h at 90 °C (Chao et al. 2018) until near dryness. The solution obtained after digestion was cooled, filtered, and diluted to 100 mL using ultrapure water and analyzed by ICP-MS for the metal concentrations (Table 1).

Leaching

For the recovery of lithium and copper from the anode materials, different inorganic acids such as nitric acid (HNO_3 , 65%), sulfuric acid (H_2SO_4 , 98%), and hydrochloric acid (HCl , 37%) were used as a leaching agents. The leaching experiments were conducted in flasks of 500 mL capacity fitted with reflux condenser (for recirculation of any evaporated liquid) and placed over a ceramic hot plate with magnetic stirring system under a fume hood. For each experiment, 1 g sample was placed into flask and 50 mL of acid solution (appropriated concentration) was added into the flask then heated at 70 °C for a 2 h. The contents were stirred at 400 rpm. After leaching of 2 h, the samples were withdrawn, filtered, diluted, and analyzed by using ICP-MS to read the metal ion concentration in the solution after appropriate dilutions (Wang et al. 2009). The percent leaching is calculated as follows:

$$\text{Percent leaching}(\%) = C_O(\text{mg/g})/C_S(\text{mg/g}) * 100 \quad (1)$$

where C_O (mg/g) is the concentration of metals leached to the solution per gram of the added anode material, and C_S (mg/g) is the corresponding total amount of metals determined by digestion in aqua regia (mg/g).

Result and discussion

Characterization of anode material of spent LIBs

The anode of spent LIBs mainly consists of a copper foil coated with active materials mainly containing graphite. The XRD spectrum of anode material also showed the presence of graphite reported by Natarajan et al. (2018). The graphite also contained sufficient amount of lithium. The high content of lithium maybe due to the process of lithium-ion traveling between the cathode and anode electrode for energy storage and release as well as some of the lithium-ion were inserted into the pores of graphite. Therefore, anode material was further analyzed by ICP-MS to determine its metal contents.

Table 1 shows the anode material composition, indicating that copper and lithium are major part of anode material of spent LIBs. Some amount of cobalt, iron, and nickel is also present in the sample. The concentration of cobalt (0.009%), iron (0.007%), and nickel (0.004%) in anode material of spent LIBs is very low, as cobalt, iron, and nickel are not the main constituents of anode material. Therefore, the authors have not focused on these metals.

Leaching

To select the best leaching agent for the leaching of lithium and copper from the anode material of spent LIBs, leaching was carried out with HNO_3 , H_2SO_4 , and HCl under similar conditions. For the assurance of reproducibility, the experiments were conducted in triplicates with an experimental error of $< 5\%$ in the present study.

Effect of hydrochloric acid concentration

HCl concentrations were varied in the range 1–5 mol/L, keeping other parameters constant at 70 °C, 2 h, S/L ratio 1 g/50 mL. Figure 1 shows that the leaching efficiency of lithium was enhanced with rise in HCl concentration from 1 to 5 mol/L. The leaching efficiency of lithium was $52.5 \pm 0.31\%$ at 1 mol/L HCl and it increases to $96.6 \pm 0.39\%$ at 5 mol/L HCl , while leaching of copper was negligible at low concentration of HCl and at high concentration of HCl (5 mol/L) only $16.2 \pm 0.64\%$ copper was leached.

Effect of sulfuric acid concentration

The H_2SO_4 concentration was varied in the range 1–5 mol/L, keeping other parameters constant at 70 °C, 2 h, S/L ratio 1 g/50 mL. Figure 2 shows that leaching efficiency of lithium was enhanced from 28.5 ± 0.68 to $68.7 \pm 0.19\%$ as the concentration of H_2SO_4 increased from 1 to 5 mol/L. In case of copper leaching, the leaching efficiency was increased from 4.8 ± 0.17 to $31.5 \pm 0.61\%$.

Effect of nitric acid concentration

HNO_3 concentration was varied in the range 1–5 mol/L, keeping other parameters constant at 70 °C, 2 h, S/L ratio 1 g/50 mL. From Fig. 3, it is evident that the leaching efficiency of lithium was lower in HNO_3 medium as compared to HCl and H_2SO_4 medium. The leaching efficiency of lithium was increased from 14.4 ± 0.15 to $59.3 \pm 0.36\%$ with increasing

Table 1 Composition of anode material of spent LIBs

Metals	Li	Co	Fe	Ni	Cu
mg/g	33.49 ± 0.18	0.09 ± 0.008	0.07 ± 0.002	0.04 ± 0.003	15.47 ± 0.23

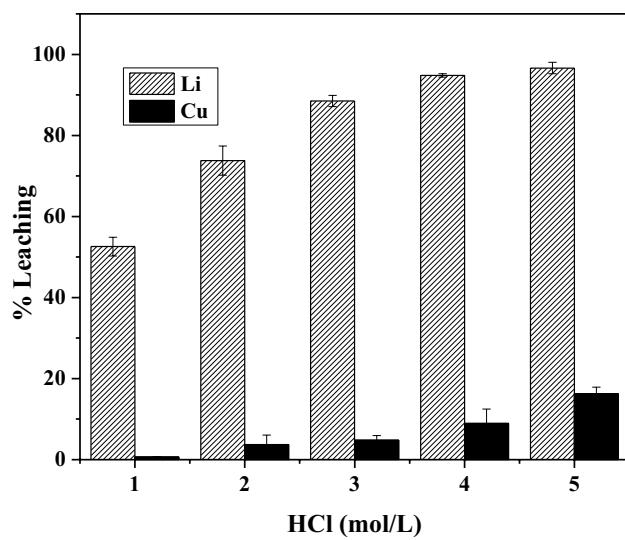


Fig. 1 Effect of HCl concentration on the leaching of lithium and copper from anode material of spent LIBs. Conditions: temp. = 70 °C, time = 2 h, S/L ratio = 1 g/50 mL

the HNO_3 from 1 mol/L to 5 mol/L. Similarly, copper was showed efficient leaching in HNO_3 medium. The leaching of copper was increased significantly with increasing the HNO_3 from 1 to 3 mol/L then changes only 2 to 3% from 3 to 5 to 5 mol/L HNO_3 .

Mechanism of leaching

Leaching of lithium and copper by mineral acid is based on solubility of lithium and copper in hydrochloric acid, sulfuric acid, and nitric acid. Lithium has high solubility in hydrochloric acid and form stable chloride complexes of lithium (Mubarok et al. 2021). In sulfuric acid medium, when lithium reacts with sulfuric acid, lithium sulfate forms, which is less soluble in sulfuric acid compared to hydrochloric acid, and it may result in the lower leaching efficiency in sulfuric acid. In nitric acid medium, lithium nitrate is formed during the leaching process (Maulidia et al. 2023). Lithium nitrate is also soluble in water. Therefore, lithium is also leached with nitric acid, but overall lithium is more leached with hydrochloric acid due more stability and solubility toward chloride medium as compared to sulfuric acid and nitric acid (Figs. 1, 2, and 3).

In case of copper, the order was reversed. The highest leaching of copper was with HNO_3 then followed by H_2SO_4 and HCl. This is mainly due to strong oxidizing power of nitric acid. Nitric acid easily oxidizes the copper metal from the waste and solubilizes it into the aqueous solution. Hydrochloric and sulfuric acid are not effective for copper leaching, as these require the presence of suitable oxidants (H_2O_2 , Cl_2 , O_2 , and bacteria) (Kim et al. 2011; Bas et al. 2014).

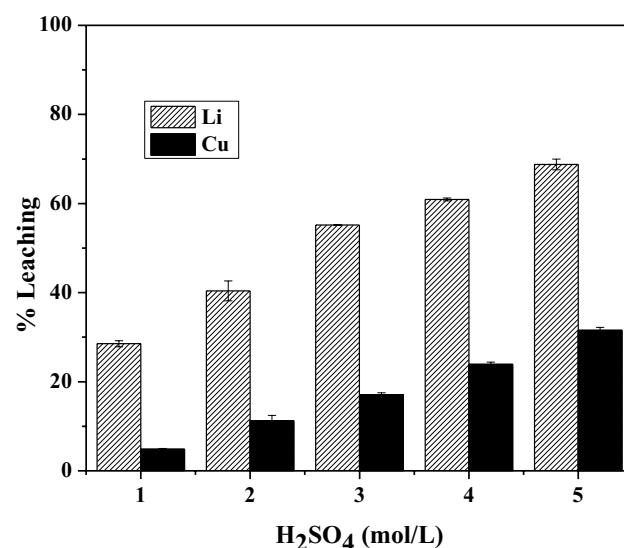


Fig. 2 Effect of H_2SO_4 concentration on the leaching of lithium and copper from anode material of spent LIBs. Conditions: temp. = 70 °C, time = 2 h, S/L ratio = 1 g/50 mL

Therefore, HCl and HNO_3 were found to be best leaching agent for lithium and copper, respectively.

After selective leaching of lithium, copper was mostly presented in the leach residue, which was recovered by the leaching with HNO_3 . However, HNO_3 is more corrosive among all the studied acids, but due to strong oxidizing nature of HNO_3 , it is used for the recovery of copper from the leach residue after leaching of lithium. From Fig. 3, it was shown that the leaching efficiency of copper was

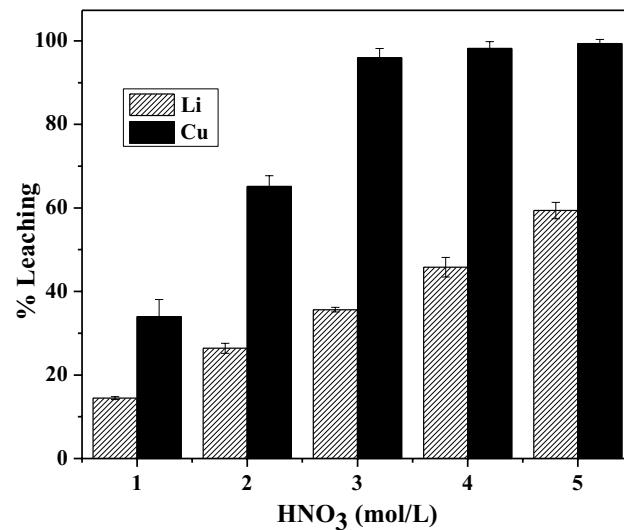


Fig. 3 Effect of HNO_3 concentration on the leaching of Li and Cu from anode material of spent LIBs. Conditions: temp. = 70 °C, time = 2 h, S/L ratio = 1 g/50 mL

approximately same at 3 mol/L (95.96 ± 2.19) and 4 mol/L (98.14 ± 1.65) HNO₃. Therefore, 3 mol/L HNO₃ was selected for the leaching of copper.

Separation of lithium and copper from the anode material of spent LIBs

Lithium and copper were separated by two-step leaching from the anode material of spent LIBs. In the first step, 5 g powder was leached with 4 mol/L HCl at 70 °C, with leaching time of 2 h and solid/liquid ratio 1 g/50 mL. Results are presented in Table 2. Around 95% lithium was leached with <10% leaching of copper.

Furthermore, the leach residue was washed with ultrapure water then dried at 60 °C in oven for 12 h. The dried leach residue was further leached with 3 mol/L HNO₃ at 70 °C, with leaching time of 2 h and solid/liquid ratio 1 g/50 mL. Table 2 shows the results; it was evident from the results that >95% copper was leached.

Recovery of lithium and copper from leach liquor

Recovery of lithium

The lithium solution obtained in the “Separation of lithium and copper from the anode material of spent LIBs” section was used to recover lithium as lithium carbonate. The pH of the solution was adjusted to 11 using solution of sodium hydroxide. Lithium was precipitated as lithium carbonate upon addition of saturated sodium carbonate solution at 100 °C with continuous stirring. The obtained white precipitate undergone filtration, washing by hot water, as well as drying in an oven for 24 h at 60 °C (Dhiman and Gupta 2019).

Recovery of copper

The copper solution after leaching of lithium in the “Separation of lithium and copper from the anode material of spent LIBs” section was used to prepare copper oxide. The solution pH was raised to 8.0 using sodium hydroxide solution. A light blue precipitate of copper hydroxide thus obtained was filtered, washed with ethanol, and dried at 60 °C in

an oven. The solid material was kept in muffle furnace at 350 °C for 4 h to obtain copper oxide (Singh et al. 2009).

The complete process for the recovery of lithium and copper from anode material of spent LIBs leach liquor is presented as a flow sheet in Fig. 4. Percentage recovery of lithium and copper from anode material of spent LIBs as oxide/carbonate is reported in Table 3.

Characterization of the synthesized products

XRD study

XRD patterns of synthesized lithium carbonate and copper oxide along with the bars representing reference spectra are given in Fig. 5(a) and (b). Maximum peak intensities of lithium carbonate and copper oxide were found at (002) and (111) planes, respectively. The prepared materials show good agreement between the observed and reference peaks (JCPDS No. 00-022-1141 and JCPDS No. 00-006-0416).

The crystallite sizes (D) are 57.7 nm and 10.4 nm for lithium carbonate and copper oxide, respectively, which were evaluated by Scherrer's equation,

$$D = 0.9\lambda / \beta \cos \theta$$

where the diffraction peak full width at half maximum is β , wavelength of X-ray beam is λ , and Bragg's diffraction angle is θ .

FE-SEM and EDX analysis

The morphology of any substance is mainly useful to understand the characteristics of the material such as size and shape. Based on the size and shape of the material, it can be applied for various applications. The particle sizes in nano-range are also known to enhance the performance and efficiency of the material. The morphology of obtained lithium carbonate and copper oxide was studied with FE-SEM analysis (Fig. 6). It is clear that the morphology of lithium carbonate is primary sheets. The synthesized copper oxide shows formation of spherical nanoparticles with small degree of agglomeration. Aggregations in nanoparticles were observed due to high surface area of contact and high surface energy. The mean sizes of lithium carbonate and copper oxide nanoparticles calculated by the software ImageJ were 165.89 ± 51.54 nm and 38.97 ± 7.89 nm,

Table 2 Selective separation of lithium and copper from anode material of spent LIBs by two step leaching

Metals	I step leaching (4 mol/L HCl), mg/L	% Leaching	II step leaching (3 mol/L HNO ₃), mg/L	% Leaching
Li	647.24 ± 28.65	96.63	7.25 ± 0.95	31.52
Cu	25.71 ± 1.24	8.31	277.43 ± 11.79	97.68

Fig. 4 Flow sheet for the recovery of lithium and copper from anode material of spent LIBs

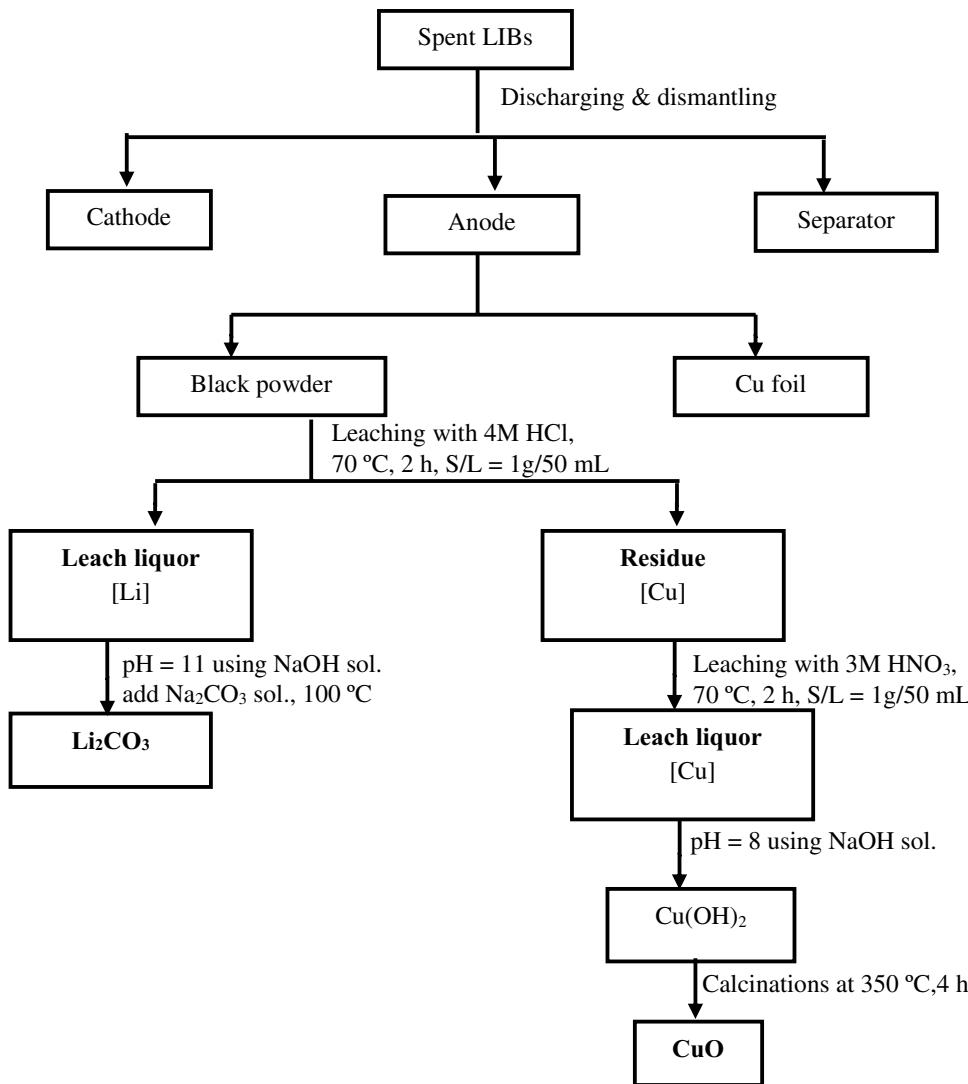


Table 3 Recovery of lithium and copper as lithium carbonate and copper oxide from the anode material of spent LIBs

Metals	Leached metal (mg/g) [#]	Recovered metal (mg/g) ^{\$}	Recovery (%)
Li	32.31±0.19	31.98±0.21	98.9
Cu	15.11±0.23	14.97±0.17	99.1

[#] Metal leached per gram of anode material

^{\$} Metal recovered per gram of anode material (calculated from recovered carbonate/oxide)

respectively. In order to study the distribution of particle sizes, curves were sketched for prepared materials. The Gaussian fit confirms the narrow size particle distribution (Fig. 6a and b). EDX spectrum for copper oxide indicates that it is composed of Cu and O only (Fig. 7).

Conclusion

The electrode scraps and spent LIBs are usually disposed improperly, which results in wastage of major resources as well as long-term impact on the environment. In this study, simple process was developed to recover lithium and copper from anode electrode material of spent LIBs. The optimum leaching conditions for lithium was HCl concentration of 4 mol/L, temperature of 70 °C, solid-to-liquid ratio of 20 g/L, and leaching time of 2 h. Under these conditions, the leaching efficiency for lithium was 96.6%. Around 97.6% copper was leached under the optimized conditions with HNO₃ concentration of 3 mol/L,

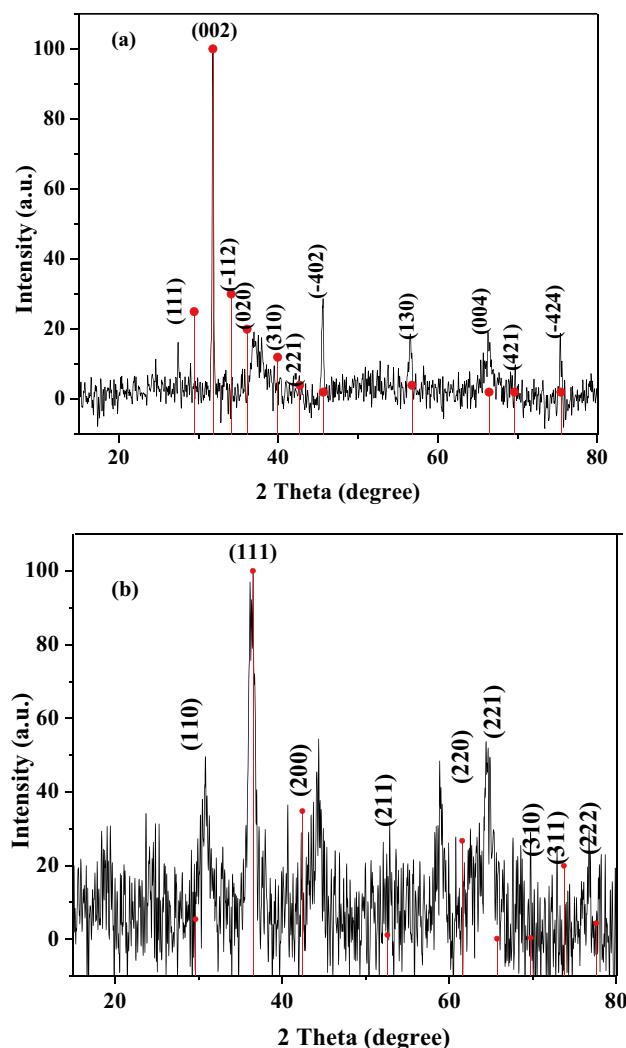


Fig. 5 XRD spectra of **a** lithium carbonate and **b** copper oxide

temperature of 70 °C, solid-to-liquid ratio of 20 g/L, and leaching time of 2 h after leaching of lithium. Furthermore, lithium carbonate and copper oxide were synthesized from their recovered respective solution using cost-effective precipitating agents. Recovery of copper as copper oxide and lithium as lithium carbonate is 99.1% and 98.9%, respectively. A simple process for the recycling of anode material of spent LIBs is thus proposed. Based on the batch scale study, a flow sheet is proposed, which can be further extended for industrial application on commercial scale.

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Author contribution All authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by Shubhangee Agarwal, Soniya Dhiman, and Himanshu Gupta. The first draft of the manuscript was written by Shubhangee Agarwal and Soniya Dhiman, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Data availability All data are true and valid and can be available.

Declarations

Ethics approval I would like to declare on behalf of my co-authors that the work described was original research that has not been published previously.

Consent to participate All the authors listed consent to participate.

Consent for publication All the authors listed have approved the manuscript that is enclosed.

Competing interests The authors declare no competing interests.

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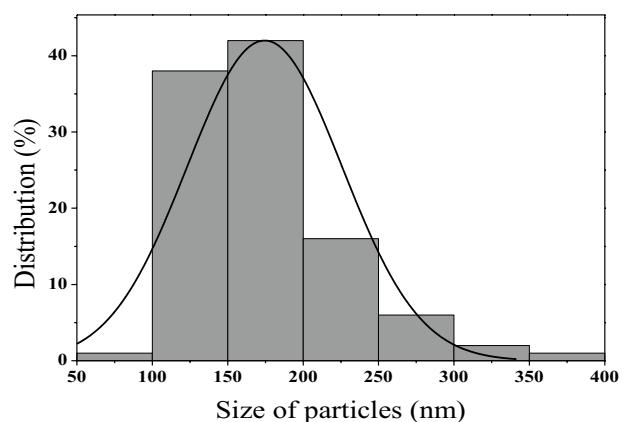
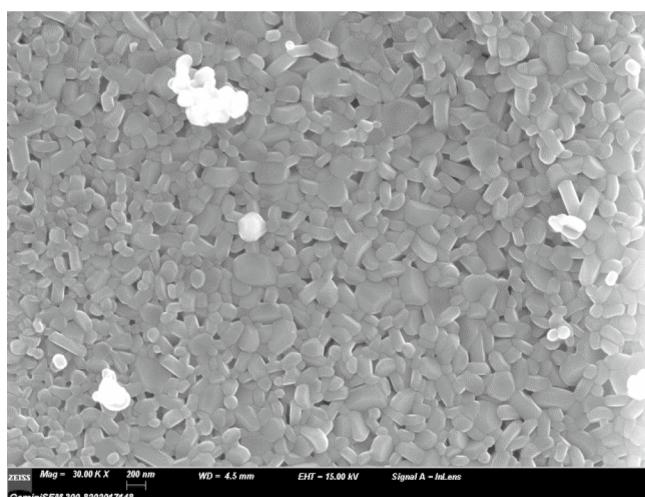
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(a)



(b)

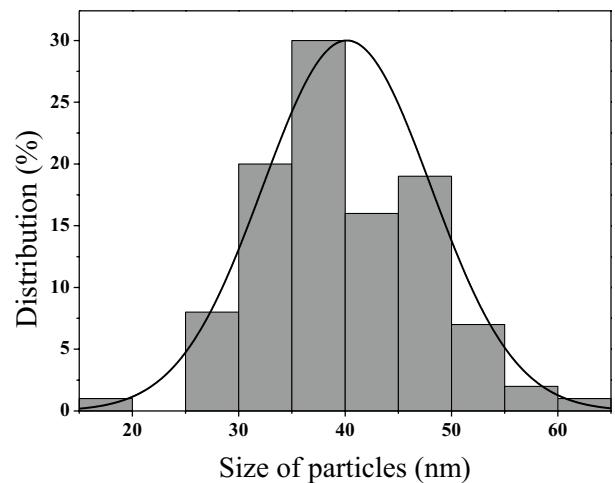


Fig. 6 FE-SEM spectra and size distribution curve for **a** lithium carbonate and **b** copper oxide

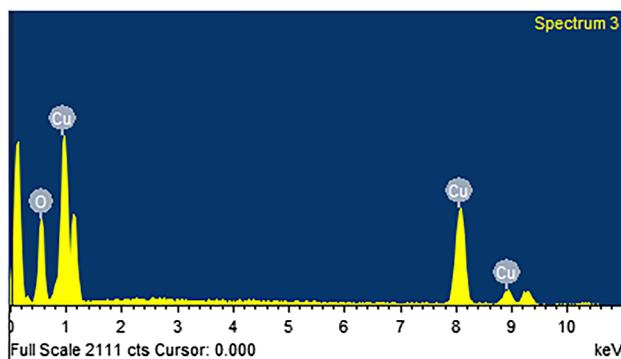


Fig. 7 EDX spectrum of copper oxide

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