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Article

Thermodynamic study for the recovery of lithium and cobalt from waste batteries

Estudio termodinámico para la recuperación de litio y cobalto a partir baterías de desecho

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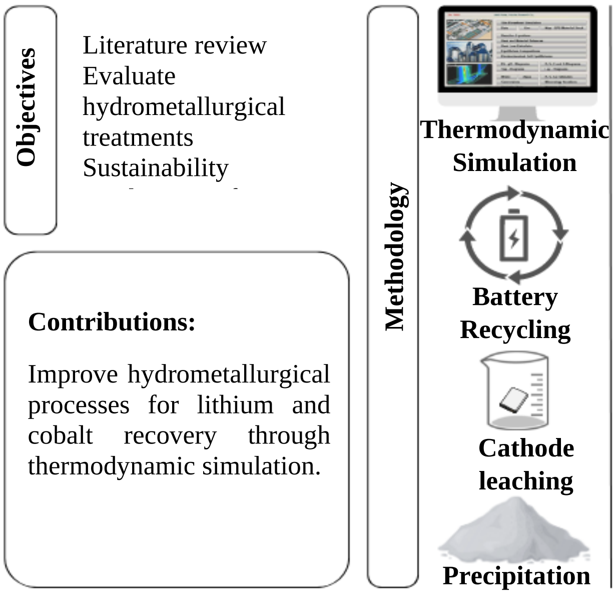
Abstract

Lithium and cobalt are a fundamental raw material for the manufacture of portable batteries, widely used in mobile phones, laptops, electric cars, etc., due to their properties in the accumulation of electrical energy. While it takes approximately 250,000 kg of lithium ore or 750,000 kg of brine to extract 1,000 kg of lithium, it takes only 28,000 kg of discarded lithium-ion batteries to obtain the same amount of metal. For this reason, this project seeks to improve and optimize the recycling process through the selective precipitation of lithium (Li) and cobalt (Co) from ammonium oxalate $(NH_4)_2C_2O_4$ and sodium carbonate Na_2CO_3, through a detailed study of variables such as reagent concentration, pH and temperature. This will be achieved through thermodynamic simulations, with the aim of maximizing the recovery of lithium and cobalt from discarded batteries.



Objectives

Literature review
Evaluate hydrometallurgical treatments
Sustainability

Contributions:

Improve hydrometallurgical processes for lithium and cobalt recovery through thermodynamic simulation.

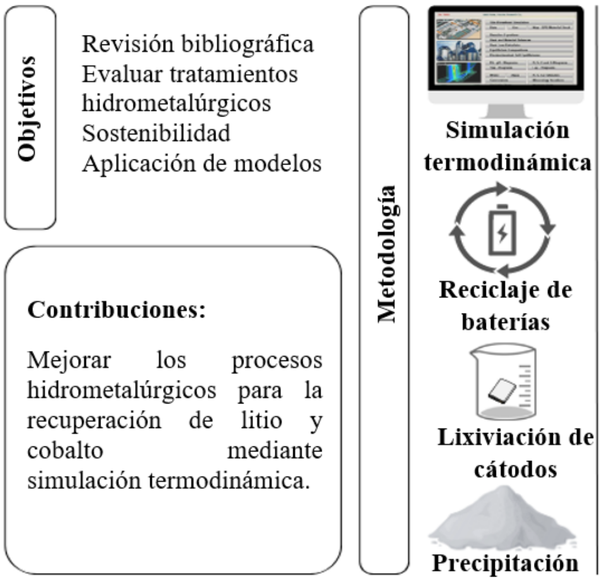
Methodology

Thermodynamic Simulation
Battery Recycling
Cathode leaching
Precipitation

Thermodynamic simulation, Batteries, Lithium

Resumen

El litio y cobalto son una materia prima fundamental para la fabricación de baterías portátiles, ampliamente utilizada en los teléfonos móviles, laptops, autos eléctricos, etc., debido a sus propiedades en la acumulación de energía eléctrica. Mientras que se necesitan aproximadamente 250,000 kg de mineral de litio o 750,000 kg de salmuera para extraer 1,000 kg de litio, se requieren solamente 28,000 kg de baterías de ion litio desechadas para obtener la misma cantidad de metal. Por esta razón, este proyecto busca mejorar y optimizar el proceso de reciclaje mediante la precipitación selectiva de litio (Li) y cobalto (Co) a partir de oxalato de amonio $(NH_4)_2C_2O_4$ y carbonato de sodio Na_2CO_3, mediante un estudio detallado de variables como la concentración de reactivos, el pH y la temperatura. Esto se logrará a través de simulaciones termodinámicas, con el objetivo de maximizar la recuperación de litio y cobalto de las baterías desechadas.



Objetivos

Revisión bibliográfica
Evaluar tratamientos hidrometalúrgicos
Sostenibilidad
Aplicación de modelos

Contribuciones:

Mejorar los procesos hidrometalúrgicos para la recuperación de litio y cobalto mediante simulación termodinámica.

Metodología

Simulación termodinámica
Reciclaje de baterías
Lixiviación de cátodos
Precipitación

Simulación termodinámica, baterías, litio

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Introduction

Lithium and cobalt are fundamental raw materials for the manufacture of portable batteries, widely used in mobile phones, laptops, digital cameras, electric cars, and more. due to the advantages of light weight, large capacity and long life. As a result, the production and consumption of lithium batteries skyrocketed (Kaya, 2022).

The main use of lithium and cobalt in Mexico and globally is in battery manufacturing, accounting for 39% of total usage. Other uses include ceramics and glass (30%), lubricating greases (8%), flux powders for continuous casting and polymer production (5%), air treatment (3%), and miscellaneous applications (10%). In 2016, Mexican lithium exports were only valued at $658, while imports amounted to 219 tons, representing a value of $1.6 million. These figures show that, in 2016, Mexico's trade balance for lithium had a deficit of $1.6 million. Finally, it is noted that 90% of Mexico’s lithium imports came from Chile, 9% from Slovenia, and the remaining 1% from other countries (Secretaría de Economía, 2018).

In Mexico there are currently 36 foreign-owned mining projects focused on lithium extraction, controlled by 10 companies. These projects represent 97 thousand hectares of concessions, with an addition to 537 thousand hectares still in process. As a result, there is not yet a lithium or cobalt mine with significant productions, due to the low grades in the deposits, lack of adequate extraction technologies, in addition to the high costs of extraction (Geocomunes, 2021).

Advances in the commercial development of lithium-ion batteries have led to significant growth in demand for lithium (Li), cobalt (Co), manganese (Mn), and nickel (Ni). In 2019, lithium-ion batteries for electric vehicle production were estimated to consume around 19,000 mt (Million tonne) of cobalt, 17,000 mt of lithium, 22,000 mt of manganese, and 65,000 mt of nickel. With the projection of 245 million electric vehicles by 2030, the required demand for cobalt expands to around 180,000 mt/year, lithium to around 185,000 mt/year, manganese to 177,000 mt/year, and nickel to 925,000 mt/year (Kaya, 2022).

After hundreds of charge and discharge cycles, the expansion of the batteries and the capacity decreases until they are discarded, the lifespan of the batteries is around 3 to 15 years depending on the device. Consequently, the huge battery consumption also generates a surprising number of scrap batteries. (Kaya, 2022).

The largest source of lithium available for recycling is lithium-ion batteries. Once their capacity falls below 80% of their rated capacity, these batteries are considered unsuitable for electronic devices and are consequently discarded. The concentration of lithium in these discarded batteries ranges from 3% to 7%, which is significantly higher than the concentration found in natural sources. While around 250,000 kg of lithium ore, or 750,000 kg of brine are required to extract 1,000 kg of lithium, it takes only 28,000 kg of discarded lithium-ion batteries to obtain the same amount of the metal (Qiao et al., 2021).

There are methods for recycling lithium and other metals present in batteries, one of them is acid leaching from hydrometallurgy (H. Bae et al., 2021), however, this is not yet fully understood, as there is limited information on thermodynamic simulations. This lack of data prevents the identification of optimal conditions for maximizing the recovery of lithium and other metals.

As can be seen, a possible solution to meet the demand for lithium and avoid the accumulation of waste batteries is to improve recycling processes. Therefore, in this project he proposes from the collection of electronic scrap, to carry out a study to recover the lithium and cadmium present in these wastes, based on thermodynamic models, considering different concentrations of reagents, pH and temperature to maximize the release and recovery of metal species, in turn to make the extraction processes via hydrometallurgy more efficient.

Background

The process of hydrometallurgical extraction, or chemical leaching, that is practiced commercially in China, for example, offers an alternative that consumes less energy and lower capital costs.

These processes employ reagents such as hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), and hydrogen peroxide (H_2O_2) to extract and separate metals from the cathode, generally operate below 100 °C, and can recover lithium in addition to the other transition metals (Jung et al., 2021).

Various hydrometallurgical methods have been developed over the past ten years to recycle lithium-ion battery cathode materials from various different battery chemistries, including lithium cobalt dioxide, $LiCoO_2$ (LCO), lithium manganese dioxide, $LiMn_2O_4$ (LMO), lithium nickel manganese cobalt oxide, $LiNiMnCoO_2$ (NMC), and lithium oxide, nickel, cobalt, and aluminum, $LiNiCoAlO_2$ (NCA), and lithium-iron phosphate $LiFePO_4$ (LFP), to recover cobalt, nickel, magnesium, and lithium (Jung et al., 2021).

The process can be classified into four main sections, which are leaching, impurity removal, metal recovery such as Ni, Co, Mn and lithium recovery, the spent cathode material is first suspended with weak acid and then transferred to the leach tanks. Acid and reducing agent are then added to the tank leaching to leach Li^+, Ni^{2+}, Co^{2+}, Mn^{2+}, Fe^{2+}, and Al^{3+}. In the impurity removal section, unwanted impurities will be removed by adjusting the pH. After impurity removal, the solution is transferred to the metal recovery section, where the metal can be recovered through crystallization precipitation using sodium carbonate.

For the hydrometallurgical treatment for the acid pathway in lithium and cobalt recovery, the leaching process of $LiCoO_2$ in a sulfuric acid (H_2SO_4) solution could be represented as follows (H. Bae et al., 2021):

$$2LiCoO_2(s)+3H_2SO_4(aq)+H_2O_2(aq)\rightarrow 2CoSO_4(aq)+Li_2SO_4(aq)+4H_2O(g)+O_2(g) \quad [1]$$

Subsequently, to selectively precipitate cobalt from the leachate liquor, ammonium oxalate is added to precipitate cobalt oxalate $CoC_2O_4{\cdot}2H_2O$. The precipitation process can be expressed by (H. Bae et al., 2021):

$$CoSO_{4(aq)}+(NH_4)_2C_2O_{4(aq)}\rightarrow CoC_2O_{4(s)}\downarrow + (NH_4)_2SO_{4(aq)} \quad [2]$$

After recovering CoC_2O_4, a high amount of sodium carbonate is added, to precipitate lithium carbonate (Li_2CO_3). The reaction in this system is shown below (H. Bae et al., 2021):

$$2Li^+ + Na_2CO_3 \rightarrow Li_2CO_3\downarrow + 2Na^+ \quad [3]$$

Methodology

Thermodynamic simulation of lithium and cobalt leaching

To thermodynamically model the leaching in different pH scenarios, Pourbaix and species distribution diagrams of the Li-H_2O, Co-H_2O systems will be constructed using the MEDUSA © software (Puigdomenech, 2010), considering the presence of sulfuric acid in different concentrations. This to find the most favorable conditions to maximize the release of lithium and cobalt from battery cathodes.

Thermodynamic simulation of lithium and cobalt precipitation

To thermodynamically model the leaching process under different pH scenarios, Pourbaix and species distribution diagrams for the Li-H_2O and Co-H_2O systems will be constructed using MEDUSA© software (Puigdomenech, 2010). This modeling will consider the presence of sulfuric acid at varying concentrations to identify the most favorable conditions for maximizing the release of lithium and cobalt from battery cathodes.

Leaching of battery cathodes

Based on conditions obtained from the thermodynamic simulation, the leaching of the cathodes of the waste batteries will be carried out; in order to release the lithium and cobalt they contain in solution. A simplified diagram of the process that will be carried out in the leaching of the battery cathodes is presented (Figure 1).

(1)

(2)

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Box 1

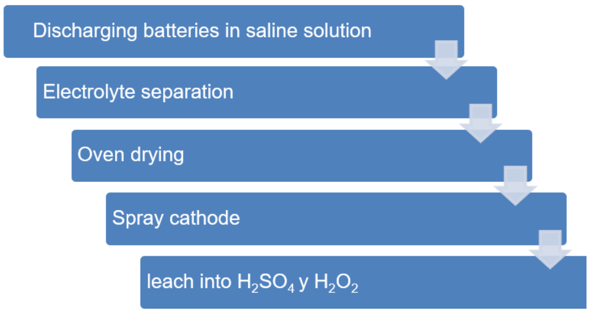

Figure 1

Process used for leaching cathodes from mobile phone batteries

Source: Own elaboration

Precipitation of Li_2CO_3 and CoC_2O_4

Once the leaching of the battery cathodes has been carried out and based on the conditions obtained in terms of reagent concentration, pH and temperature in the thermodynamic simulation, the recovery from the precipitation of lithium and cobalt will be carried out as follows:

Addition of sodium hydroxide to the leachate solution to adjust pH to maximize cobalt recovery.

Addition of ammonium oxalate in the solution to selectively recover cobalt, precipitating as cobalt oxalate (CoC_2O_4).

Filter the (CoC_2O_4) precipitates from the solution and dry them in the oven.

Add sodium carbonate to the cobalt-free solution to precipitate lithium in the form of lithium carbonate (Li_2CO_3).

Filter the Li_2CO_3 lithium carbonate precipitates from the solution and dry in an oven and discard the solution.

Results

One of the easiest ways to determine if it is possible to form a chemical compound is by employing the Nernst equation (Eq. 4) from the dissolution reaction, where $aO + ne^- \rightarrow bR$ the pH range for its formation can be calculated.

$$E_{Rxn} = E^{\circ}_{Rxn} + \frac{RT}{nF} ln \frac{{a_O}^a}{{a_R}^b} \qquad [4]$$

In this way, in the present work, thermodynamics was used through the construction of Pourbaix diagrams to identify the possible species that can be formed, in addition to the use of species distribution diagrams to identify the conversion fraction associated with a certain pH.

It should be noted that these thermodynamic simulations are carried out at room temperature (25 °C) which would lead to a more cost-effective process.

The Pourbaix diagram was constructed for the Co-Li system by adding the oxalate ion, in order to elucidate the formation of cobalt oxalate, Figure 2 presents this diagram with an oxalate concentration of 1 M, highlighting the different phases that predominate in the system. Notably, cobalt oxalate stands out, which is formed only in the acidic window of pH 0.1 – 5.

Box 2

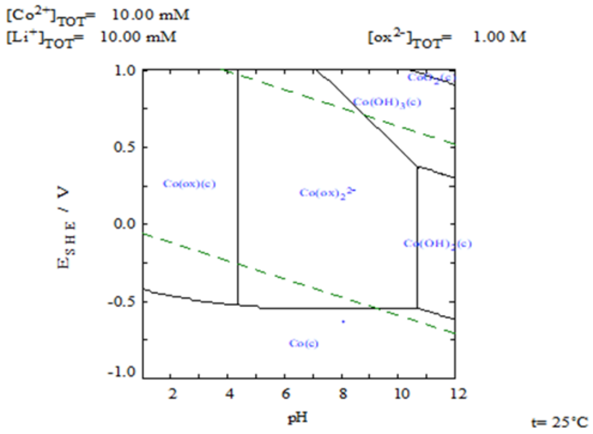

Figure 2

Pourbaix diagram of the Co-Li system with the oxalate ion.

Source: Own elaboration

According to the molar fraction diagram at different pH values of this same system (Figure 3), it can be elucidated that the most appropriate pH to maximize the formation of this cobalt oxalate compound is 2.85, since it is there where the maximum conversion peak is 0.96.

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Box 3

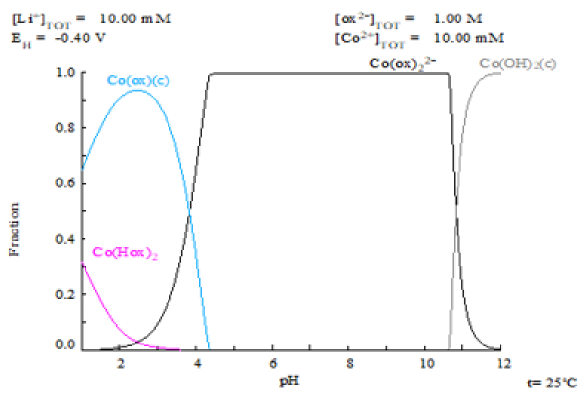

Figure 3

Distribution diagram of species of the Co-Li system with the oxalate ion.

Source: Own elaboration

Additionally, the Pourbaix diagram was elaborated by decreasing the oxalate concentration to 0.5 M (Figure 4), in which it is observed that the cobalt oxalate formation window was greatly expanded, in a pH range of 1-10, with respect to that found in Figure 2.

Box 4

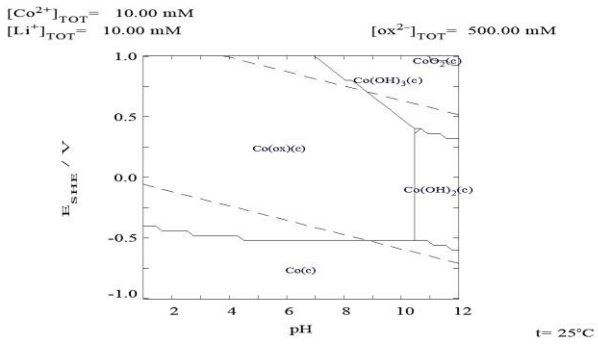

Figure 4

Pourbaix diagram of the Co-Li system with oxalate ion

Source: Own elaboration

Regarding the distribution diagram of species with the concentration of 0.5M oxalate (Figure 5), the maximum fractional conversion of cobalt oxalate crystals is achieved at a pH of 2.85, obtaining a conversion of 0.98 greater than that of 1 molar (Figure 3).

Box 5

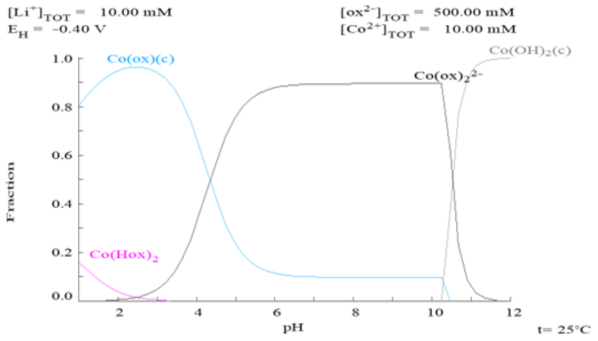

Figure 5

Distribution diagram of species of the Co-Li system with the oxalate ion.

Source: Own elaboration

Similarly, the Pourbaix diagram was constructed by increasing the concentration of oxalate to 10 M (Figure 6), in which it is observed that the window of cobalt oxalate formation was greatly decreased, in a pH range of 2-3, in this sense the more the oxalate concentration is increased, the cobalt oxalate formation window will decrease.

Box 6

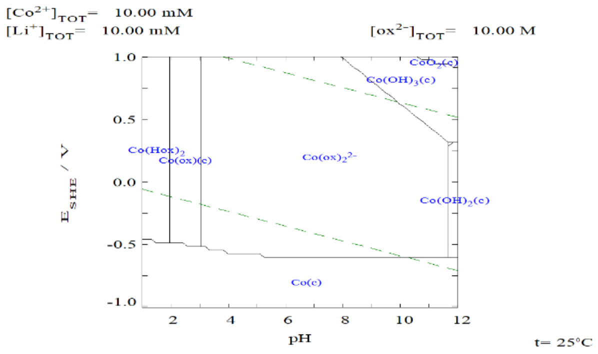

Figure 6

Pourbaix diagram of the Co-Li system with the oxalate ion.

Source: Own elaboration

As for the distribution diagram of species with the concentration of 10 M, it is observed that the maximum peak of fractional conversion to cobalt oxalate is observed to be 0.4, which is reached at a pH of 2.8 (Figure 7).

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Box 7

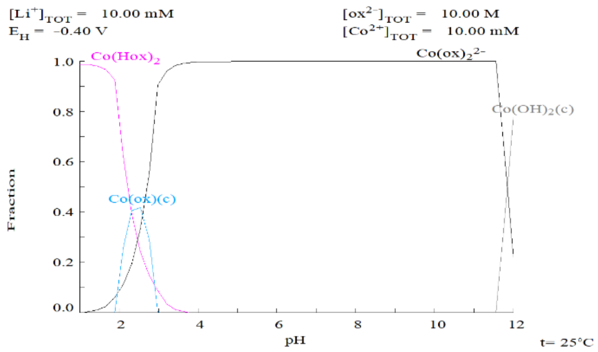

Figure 7

Distribution diagram of species of the Co-Li system with the oxalate ion.

Source: Own elaboration

It is interesting to mention that (Chen et al., 2015; Li et al., 2023; Zhu et al., 2012) carried out research where they recovered cobalt in lithium-ion batteries, precipitating it in the form of cobalt oxalate, obtaining good results with a recovery close to 90% with an oxalate concentration of 0.5 M at a pH of 2, however, according to the results of the thermodynamic simulation, an increase of 8% can be achieved if a pH of 2.85 is used (Figure 8). Additionally, the simulation indicated that regardless of the concentration, the pH that maximizes the conversion or recovery of cobalt oxalate is around 2.8-2.85.

Thermodynamic Modeling for Lithium and Cobalt Recovery

On the other hand, the Pourbaix diagram was constructed for the Li-Co system by adding the carbonate ion, in order to find the formation of lithium carbonate. Figure 8 presents this diagram with an oxalate concentration of 1 M, where the different phases that predominate in the system can be appreciated. In this the presence of lithium carbonate is highlighted, which is formed in a wide window of pH 1 – 14, it should be noted that the Pourbaix diagrams remain unchanged regardless of variations in the carbonate ion concertation.

Box 8

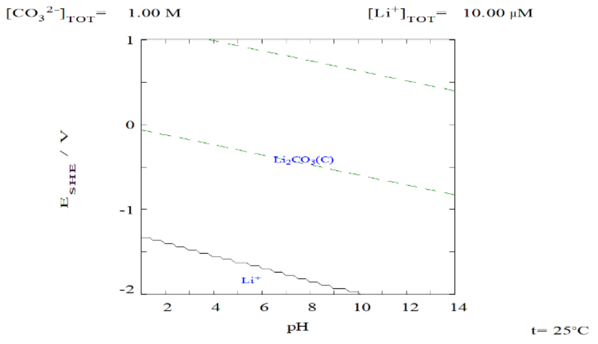

Figure 8

Pourbaix diagram of the Li system with the carbonate ion.

Source: Own elaboration

According to the molar fraction diagram at different pH values with a carbonate ion concentration of 0.001M (Figure 9), it can be elucidated that at low carbonate concentrations the formation of lithium carbonate is low, only 0.2 (20%) in a pH range of 10-14.

Box 9

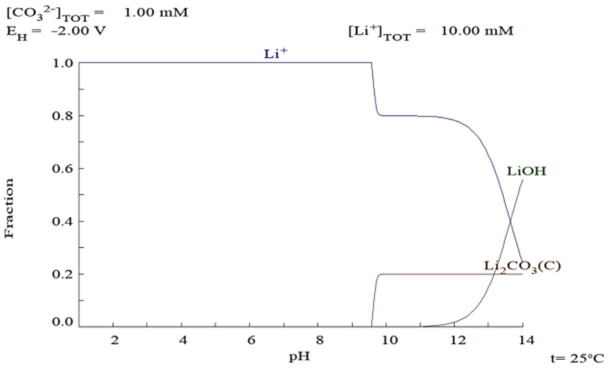

Figure 9

Distribution diagram of species of the Li system with the carbonate ion.

Source: Own elaboration

Regarding the distribution diagram of species with the concentration 0.5M of the carbonate ion (Figure 9), the maximum fractional conversion of sodium carbonate crystals is reached at a pH greater than 10, obtaining a conversion of 0.98, higher than that of 0.001 molar (Figure 8).

Box 10

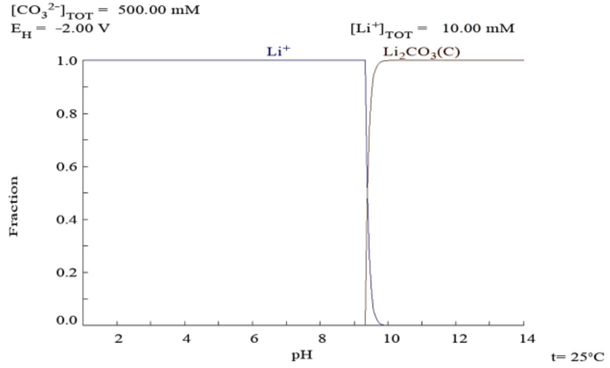

Figure 10

Li System Species Distribution Diagram with Carbonate ion.

Source: Own elaboration

Finally, the simulation was carried out by increasing the concentration of the carbonate ion up to 10 M (Figure 11), obtaining results very similar to those obtained with a lower concentration 0.5 M (Figure 10), based on this it can be observed that it is not convenient to use a high concentration of carbonate ion, since it does not reflect an improvement in the recovery of lithium carbonate.

Box 11

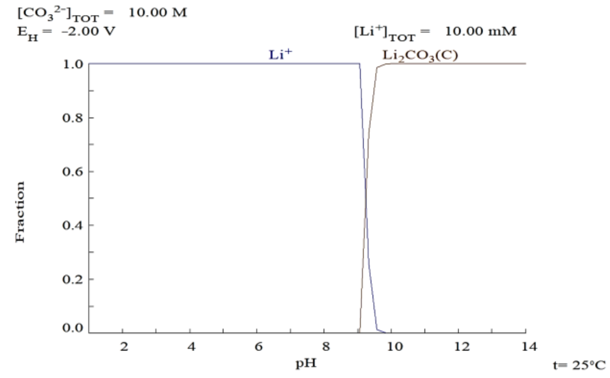

Figure 11

Distribution diagram of species of the Li system with the carbonate ion.

Source: Own elaboration

Additionally, studies carried out by different authors , where they recovered lithium in lithium-ion batteries, precipitating it in the form of lithium carbonate, obtaining recoveries around 70%, the concentration of carbonate, nor the pH used for such recovery is not mentioned, according to the results obtained in the present study 25% more can be recovered, if a concentration of 0.5 M and a pH close to 10 are used. (Chen et al., 2015; Li et al., 2023; Zhu et al., 2012).

Acid leaching of battery cathodes

Initially, the dismantling, electric discharge in saline solution, separation and pulverization of the cathodes present in the batteries, obtained in the recycling campaign, was carried out, then the leaching was carried out to release the cobalt and lithium with a solution a concentration of 3M, agitation of 300 rpm, time of 2 hours and a temperature of 25 °C. these conditions were used because, according to the authors (Chen et al., 2015; Zhu et al., 2012; Kaya, 2022), it is possible to obtain a 100% release of the cobalt and lithium present in the cathodes of the battery H_2SO_4 (Figure 12).

Box 12

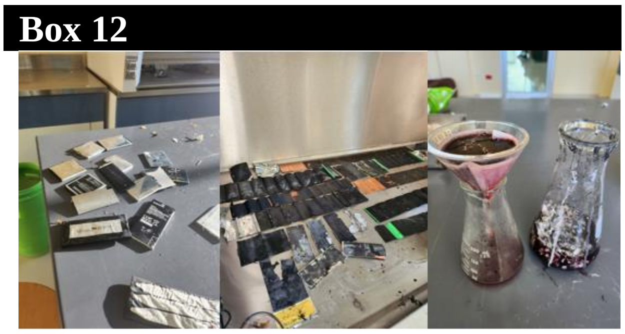

Figure 12

Disassembly, leaching, and filtering of battery cathodes.

Source: Own elaboration

Recovery of cobalt from leachate solution

After leaching the cathodes, the solution rich in cobalt and lithium was filtered to add ammonium oxalate $(NH_4)_2C_2O_4)$ at different concentrations and pH (Figure 13), to evaluate the results obtained in the thermodynamic simulation (Table 1).

Box 13

Figure 13

Addition of ammonium oxalate and precipitation of cobalt oxalate.

Source: Own elaboration

Box 14

Table 1

Cobalt recovery results at different concentrations and pH.

Temperature (°C)	pH	Concentration (M)	Recovery (g)
25	2.85	0.5	3.4172
25	2	0.5	3.1774
25	2.85	10	2.1178
25	2	10	1.8733

Source: Own elaboration

By comparing the recovery of cobalt oxalate with the following conditions: pH 2.85 and a concentration of 0.5 M oxalate, with that used by with a pH of 2 and an oxalate concentration of 0.5 M, it was possible to increase recovery by 7.017% (Chen et al., 2015; Li et al., 2023; Zhu et al., 2012).

This same phenomenon occurs when the oxalate concentration is increased to 10 M. Comparing pH 2 and pH 2.85 under these conditions, recovery increases by 11.54% at pH 2.85. The thermodynamic simulation results are conclusive for the recovery and precipitation of cobalt as cobalt oxalate. It was identified that the pH maximizing cobalt recovery is 2.85 at any concentration. Moreover, with an oxalate concentration of 0.5 M, it is possible to recover up to 98% of cobalt.

Recovery of lithium from leachate solution

Finally, after recovering the cobalt, sodium carbonate was added to the solution without cobalt to precipitate the lithium in the form of lithium carbonate (Li_2CO_3) using different concentrations of sodium carbonate (Figure 14), it should be noted that fixed at a pH of 10, this because according to the thermodynamic simulation at this pH the highest lithium recovery is obtein.

Box 15

Figure 14

Lithium carbonate recovery

Source: Own elaboration

When comparing lithium carbonate recovery with different concentrations of sodium carbonate (Table 2), it was found that low concentrations greatly reduce lithium recovery.

Box 16

Table 2

Lithium recovery results at different concentrations

Temperature (°C)	pH	Concentration (M)	Recovery (g)
25	10	0.01	1.5784
25	10	0.5	3.8557
25	10	10	3.8487

Source: Own elaboration

In addition, it was obtained that it is not necessary to use large concentrations of sodium carbonate to increase recover, since by using 10 M or 0.5 M, very similar recoveries were obtained, furthermore, it was also found that it is not necessary to use high temperatures to obtain good recovery results.

The thermodynamic simulation results are conclusive for the recovery and precipitation of lithium, in the form of lithium carbonate, since it was possible to identify that the pH that maximizes lithium recovery is 10 in any concentration, however, it was found that it is not necessary to use high concentrations of sodium carbonate to have a good lithium recovery, a low concentration 0.5 M is enough, these results contrast with those obtained by various authors (Chen et al., 2015; Li et al., 2023; Zhu et al., 2012), where they suggest using saturated lithium carbonate solutions to obtain satisfactory lithium carbonate recoveries.

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Conclusions

Based on the objectives set, it was possible to carry out the thermodynamic study for the recovery of lithium and cobalt, from lithium-ion batteries, collected through an electronic scrap recycling campaign.

The optimal conditions that maximize cobalt recovery were shown to be pH 2.85 and 0.5 M oxalate. For lithium recovery it was found that low concentrations of sodium carbonate of 0.5 M and pH 10 are required to efficiently recover lithium.

It was possible to make the lithium and cobalt extraction process more efficient from the precipitation of lithium carbonate and cobalt oxalate, based on the data obtained by the thermodynamic simulation and the experiments carried out.

Finally, the present study collaborates with an innovative contribution in the area of lithium and cobalt extraction processes in lithium-ion batteries, since there is no reported history to make these processes more efficient.

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

Farias-Gonzalez, Miguel Angel: Contributed with the main idea of the project and carry out thermodynamic simulations to find the operating conditions for the experiments.

Farias-Gonzalez, Francisco Javier: Contributed by interpreting the data obtained through thermodynamic simulation, as well as carrying out experiments for the recovery of lithium and cobalt.

Perez-Castro, Laura Luz: Helped in the review of the systematization of the background for the state of the art, the research design, the type of research, the approach, the method and the writing of the article.

Availability of data and materials

The lithium-ion batteries were collected from a recycling campaign, and the experimentation was carried out within the institution's laboratories.

Funding

The research did not receive any funding.

Abbreviations

Co	Cobalt
CoC_2O_4	Cobalt oxalate
Li	Lithium
Li_2CO_3	Lithium carbonate
M	Molarity
pH	Hydrogen Potential
mt	Million tonne

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