



Study of the Dissolution and Leaching of Metallic Elements from the Cathode Powder of Mobile Phone Batteries

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Abstract: Spent lithium-ion batteries (LIBs) contain a significant amount of precious metals and are expected to be an important strategic resource in the future. Recycling these batteries is therefore crucial. This study investigates the dissolution of cathode powder and the leaching efficiency of metallic elements using various acids and a reducing agent. Specifically, organic acids (citric acid and acetic acid), inorganic acids (sulfuric acid and nitric acid), and hydrogen peroxide were examined. Results from Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) indicate that high dissolution of the cathode powder was achieved with nitric acid, reaching 66.8% when refluxed at 80°C. At room temperature, high dissolution rates were also observed with sulfuric acid (63.65%), nitric acid (62.90%), and citric acid (47.50%). The leaching efficiencies for sulfuric acid were 94.86% Ni, 87.99% Mn, 79.25% Co, and 65.45% Li; for nitric acid, 74.55% Mn, 74.05% Co, 69.26% Ni, and 64.78% Li; for acetic acid, 80.70% Li, 39.49% Mn, 32.53% Co, and 11.97% Ni; and for citric acid, 81.09% Li, 73.42% Mn, 55.36% Co, and 21.21% Ni. To balance environmental preservation and energy savings, dissolving at room temperature with a biodegradable acid would be preferable. In this context, citric acid emerges as a suitable choice for dissolving cathode powder. However, it should be noted that under these experimental conditions, strong acids remain the most effective leaching solutions, even though weak acids perform better for leaching lithium.

Keywords: Spent lithium-ion battery, metals, dissolution, leaching.

1. Introduction

Mobile phones are a crucial means of telecommunication, especially in remote areas where traditional landline infrastructure is lacking. They are used by billions of people around the world, and their demand is growing exponentially. According to the International Telecommunications Union (2011), there were 6 billion mobile subscriptions at the end of 2011, which is equivalent to 87% of the global population. This represents a significant increase from 5.4 billion in 2010 and 4.7 billion in 2009 [1,2].

The substantial progress in the manufacture of electrical and electronic equipment using batteries as power sources has resulted in large quantities of waste, particularly used batteries. Various types of batteries, such as zinc-carbon, nickel-cadmium (NiCd), nickel-metal hydride (NiMH), and lithium-ion, are widely used as electrochemical energy sources [3,4].

Lithium-ion batteries (LIBs) are particularly significant due to their characteristics, including high energy density, light weight, high cell voltage (three times that of NiCd and NiMH), short storage life, low self-discharge rate, and wide useful temperature range. Additionally, LIBs are preferred because they do not contain



toxic heavy metals such as mercury, cadmium, and lead [5]. Depending on the manufacturing process, LIBs are composed of metals, organic chemicals, and plastics in proportions of 5-20% cobalt, 5-7% lithium, 5-10% nickel, 15% organics, and 7% plastics [6,7]. Given that lithium, cobalt, manganese, and nickel are all strategic and precious metallic elements, recycling these batteries is highly beneficial from an environmental protection standpoint.

Recycling LIBs not only helps protect the environment but also enhances the utilization of natural resources and can reduce the cost of battery production. This waste represents an important secondary source of metals ([5], [8]). Furthermore, the price of cobalt has risen significantly in recent years, making it economically viable to recycle this metal [9]. In 2007, about 25% of global cobalt demand was for battery production [10]. LIBs consist of an anode, a cathode, electrolytes, a separator, and an outer casing ([11], [12], [13]). The anode is made of a copper foil coated with a layer of carbon graphite powder, while the cathode is made of an aluminum foil coated with a layer of metal oxide, such as lithium cobalt oxide (LiCoO₂) [9]. This compound is the most widely used cathode material in LIBs due to its good electrochemical performance. However, its disadvantages include high cost, limited cobalt availability, and toxicity ([11], [9]). Separators are made from polymer materials, and the outer casing is generally constructed from steel or aluminum ([14], [15]).

Recycling this electronic waste is therefore both a necessity and an obligation from both economic and environmental perspectives. Numerous studies have been dedicated to finding processes for recovering metals from used LIBs. Various pyrometallurgical, hydrometallurgical, or combined processes ([7], [12], [13], [3], [16]) have been explored to extract precious metals from used LIBs. Pyrometallurgical methods are adopted and commercialized by various organizations (Inmetco, Xstrata, and Umicore). In this process, the binder and organic electrolyte are generally burned before further processing, which is not economical due to high energy consumption and the emission of toxic gases (SO₂, Cl₂, CO, HF) [17]. The biometalurgical process is too slow for treating used LIBs, making battery development challenging, and its efficiency is low. Due to the advantages of high recovery efficiency, low cost, and low energy consumption, the hydrometallurgical process has been considered an effective method for recovering metals from used LIBs.

To recover metal elements from spent LIBs using the hydrometallurgical process, it is necessary to transfer as many metal elements as possible from the cathode powder into the leach solution. In recent years, many researchers have developed the dissolution of metal elements using strong (inorganic) acids such as H₂SO₄ ([18], [7], [5]), HCl [16], and HNO₃ [19]. In addition to inorganic acids, organic acids such as malic acid, oxalic acid, ascorbic acid, and citric acid, with or without H₂O₂, have also been used ([20], [21], [22], [23]) as alternatives to inorganic acids due to the environmental pollution associated with the production of gases like Cl₂, SO₃, or NO_x during leaching.

This work focuses on used lithium-ion batteries from mobile phones. The aim was to study the dissolution of the cathode powder and the leaching of the metallic elements with acids and hydrogen peroxide (as a reducing agent) to recover lithium, cobalt, nickel, and manganese from the cathode active material. The active cathode material obtained after dismantling was leached using pre-prepared acid solutions. The contents of the various metal elements extracted were determined after leaching. A new approach has been proposed to develop an ecological and economical recycling process.

2. Methodology

Preparation and Characterization of the Cathode Powder

The batteries used in this study were spent lithium-ion mobile phone batteries collected from second-hand dealers in the port area of Lomé.

After discharging and dismantling the batteries, the cathode was treated with a 1 M NaOH solution (in a 1:5 ratio) for 2 hours under magnetic stirring to dissolve the aluminum foil used as an electron collector. The cathode material, separated from the aluminum, was recovered by filtration and washed to a constant pH to remove residual soda. It was then dried in an oven at 105°C for 24 hours and ground to a particle size of 75 µm. The powder was stored for subsequent leaching studies and analysis.

Regal water (a mixture of hydrochloric acid (HCl) and nitric acid (HNO₃) in a 3:1 ratio) was used for the complete digestion of the LIB cathode powder before quantifying the metal content. The metal elements contained in the cathode powder, both before leaching and in the filtrates obtained after leaching, were



determined using an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES), model Thermo Fisher X Series II, from Thermo Fisher Scientific, Bremen, Germany.

Leaching Experiments

Reagents Used

All reagents were of analytical grade and were used as received without further purification. Sulfuric acid (H₂SO₄), nitric acid (HNO₃), citric acid (C₆H₈O₇), and acetic acid (C₂H₄O₂) were tested as leaching agents. Hydrogen peroxide (H₂O₂) was used as a reducing agent. Demineralized water was used for the preparation of all solutions.

Leaching of Metallic Elements

The cathode powder, which is the active material in lithium-ion batteries, contains lithium, cobalt, manganese, nickel, and other minor elements. Hydrometallurgy can be employed to recover these metals from used batteries.

To solubilize the metallic elements, an acid reductive leaching method was employed [24, 25]. Leaching was carried out using molar solutions of organic or inorganic acids in the presence of hydrogen peroxide (H₂O₂) as a reducing agent.

A quantity of 5 g of cathode powder was treated with 100 ml of acid solution, to which 5 ml of H₂O₂ was added in a glass beaker. The mixture was stirred at 1500 rpm for 4 hours at room temperature. The same experiment was repeated under reflux conditions at 80°C. The solutions obtained from each test were then filtered. The procedure was conducted three times to maximize the extraction of metallic elements. The final residue was washed, dried, and weighed to calculate the dissolution rate by the difference in mass.

$$\% \text{Dissolution} = \frac{M_1}{M_2} \times 100 \quad (E_1)$$

M₁: mass of final residue,

M₂: mass of cathode powder

The inorganic acids used in this study were sulfuric acid and nitric acid, while the organic acids tested were citric acid and acetic acid. The filtrates obtained after leaching were analyzed using an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) to determine their cobalt, manganese, nickel, and lithium content.

3. Results and Discussion

This section presents the results of the study on the dissolution of the cathode powder, aiming to identify the most effective leaching solution. The goal is to find a solution that consumes the least energy and causes the least environmental impact.

Dissolution of Cathode Powder in Acid Solutions

The cathode powder, which is the active material in mobile phone batteries, contains most of the metallic elements (cobalt, lithium, manganese, and nickel) that can be recovered through hydrometallurgical processes. The initial step involves dissolving the cathode powder in an acid solution to separate the metallic elements into ions, which can then be selectively precipitated.

Table 1 presents the dissolution rates of the cathode powder in various acid solutions. At room temperature, the dissolution rates are as follows: 63.65% for sulfuric acid, 62.90% for nitric acid, 47.50% for citric acid, and 27.31% for acetic acid. The high dissolution rates observed with sulfuric and nitric acids can be attributed to their strong acidity. In contrast, citric acid, though weaker than sulfuric and nitric acids, shows a higher dissolution rate compared to acetic acid due to its nature as a triacid. These results indicate that inorganic acids generally solubilize metallic elements more effectively than organic acids [26,27].

In alignment with studies by other researchers [28,29] who conducted experiments at reflux temperatures of 80°C, our experiments were repeated under the same conditions. The results showed improved dissolution rates: in acetic acid, the rate increased from 27.31% to 47.33%, and in nitric acid, it increased from 62.90% to 66.80%. Conversely, dissolution rates in sulfuric and citric acids decreased, with citric acid showing precipitation, where the mass of the residue exceeded the mass of the initially introduced powder.



Table 1: Dissolution Rate of Cathode Powder

Conditions	Acetic Acid	Sulfuric Acid	Nitric Acid	Citric Acid
Ambient Temperature	27.31%	63.65%	62.90%	47.50%
Reflux at 80°C	47.33%	49.08%	66.80%	-7.22%

The dissolution rates of the cathode powder reveal the following insights:

Dissolution Efficiency at Ambient Temperature:

Sulfuric Acid and Nitric Acid show high dissolution rates of 63.65% and 62.90%, respectively.

Citric Acid has a moderate dissolution rate of 47.50%.

Acetic Acid exhibits the lowest dissolution rate at 27.31%.

Effect of Reflux at 80°C:

Nitric Acid achieves the highest dissolution rate of 66.80% when refluxed.

Acetic Acid also shows a significant improvement at reflux, with a dissolution rate of 47.33%.

Sulfuric Acid shows a decrease in dissolution efficiency, with a rate of 49.08%.

Citric Acid displays a negative dissolution rate of -7.22%, indicating potential precipitation or side reactions at this temperature.

Environmental Considerations:

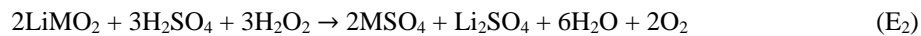
To minimize environmental impact and energy consumption, it is preferable to use biodegradable acids at room temperature. Among the acids tested, **Citric Acid** is a promising option due to its moderate dissolution rate and eco-friendly profile. This makes it a suitable alternative for achieving effective dissolution while reducing environmental pollution.

Leaching of Chemical Elements

Leaching with Strong Acids

Figures 1 and 2 below illustrate the efficiency of leaching metallic elements with sulfuric and nitric acids.

With sulfuric acid, nickel was leached to the extent of 94.86%, manganese to 87.99%, cobalt to 79.25%, and lithium to 65.45%. These results show that sulfuric acid exhibits a high leaching efficiency for the metallic elements contained in mobile phone batteries. The leaching reaction can be illustrated by the following equation (E₂):



Where M = (Co, Ni, Mn)

These results are quite similar to those obtained by Yang et al. [30], who leached 62.11% Li, 98.41% Ni, 91.86% Co, and 93.54% Mn from cathode powder using sulfuric acid, and by Meshram et al. [31], who leached 63.4% Li, 90% Co, 96.3% Ni, and 70.2% Mn from cathode powder with the same acid.

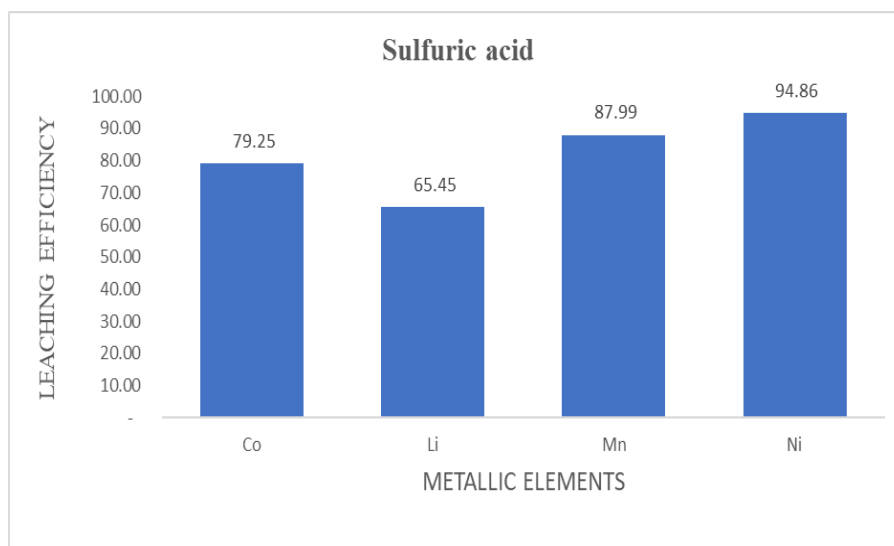
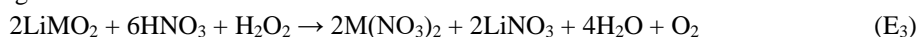


Figure 1: Metallic Element Contents in the Liquor Obtained with Sulfuric Acid



With nitric acid, manganese was leached at 74.55%, cobalt at 74.05%, nickel at 69.26%, and lithium at 64.78%. Nearly all the metallic elements were leached at a high rate of over 60% using nitric acid. These results indicate that nitric acid is also an effective leaching agent for metallic elements in mobile phone batteries. The equation for the leaching reaction is as follows:



With M= (Co, Mn and Ni)

These results are fairly consistent with those of Aaltonen *et al.* [32], who leached 65% Li, 70% Ni, 89% Co, and 80% Mn from cathode powder using nitric acid.

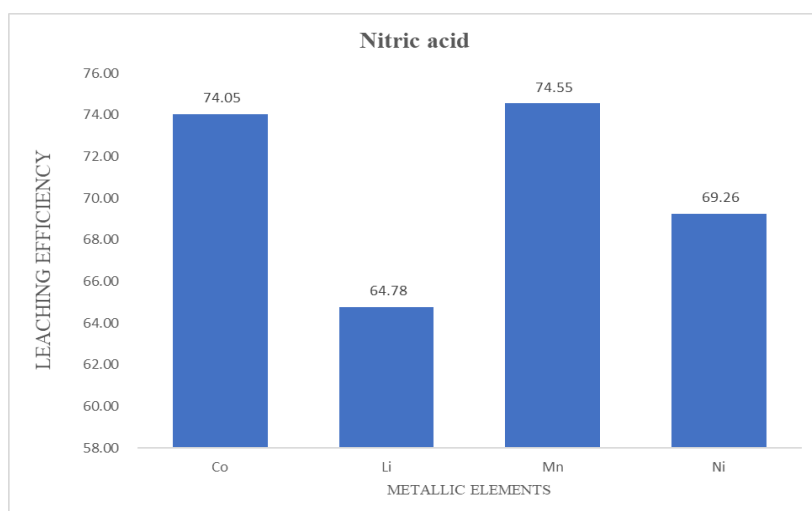


Figure 2: Content of Metallic Elements in the Liquor Obtained with Nitric Acid

These results indicate that strong acids leach less lithium. Additionally, the leaching efficiency of metallic elements is higher with sulfuric acid compared to nitric acid, which can be attributed to sulfuric acid being a diprotic acid.

Leaching with Weak Acids

Figures 3 and 4 displays the results of the leaching efficiency of metallic elements from the cathode powder using acetic and citric acids.

With acetic acid, the leaching rates for the metallic elements are as follows: 80.70% for lithium, 39.49% for manganese, 32.53% for cobalt, and 11.97% for nickel. The leaching reaction can be represented by the following equation (E₄), with M = (Ni, Co, and Mn):

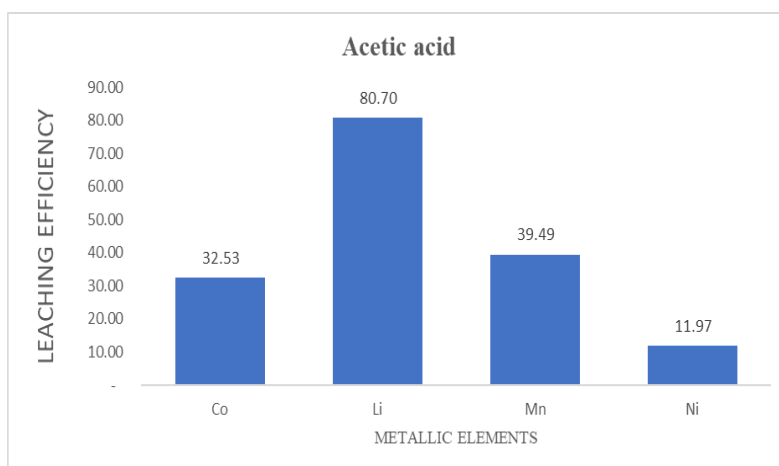
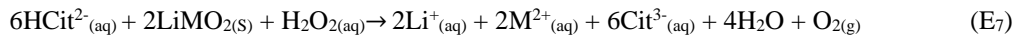
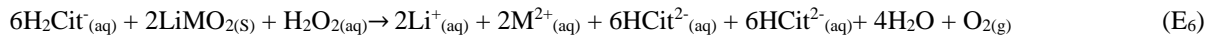
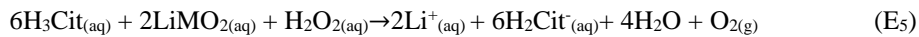


Figure 3: Content of Metallic Elements in the Liquor Obtained with Acetic Acid



With citric acid, the leaching rates are as follows: 81.09% for lithium, 73.42% for manganese, 55.36% for cobalt, and 21.21% for nickel.

The leaching reaction of metallic elements from the cathode powder with citric acid is represented by the following equations (E₅), (E₆), and (E₇):



Where M = (Ni, Co and Mn)

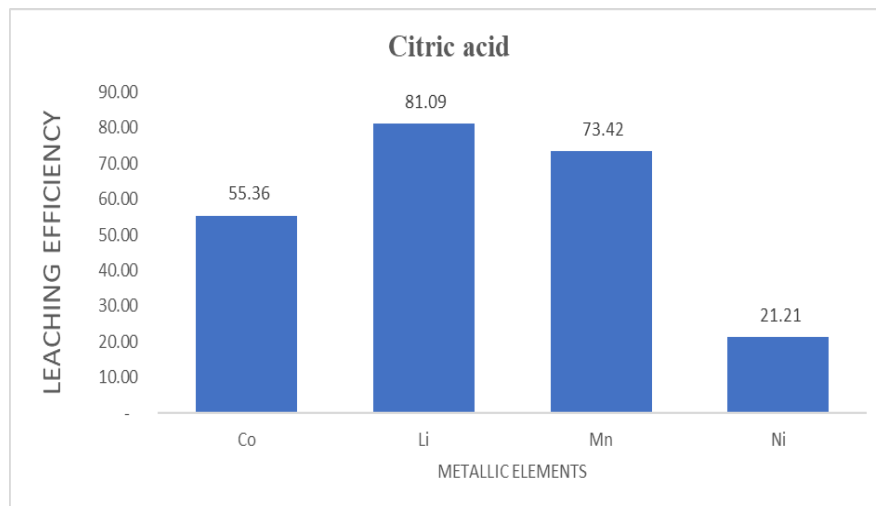


Figure 4: Content of Metallic Elements in the Liquor Obtained with Citric Acid

These results indicate that the leaching efficiency of metallic elements is higher with citric acid than with acetic acid, which can be attributed to citric acid being a tricarboxylic acid. Lithium is the most effectively leached element by both acetic and citric acids, with a rate of nearly 81%. Conversely, cobalt and nickel exhibit the lowest leaching rates.

The low recovery rates for cobalt, manganese, and nickel, despite the addition of hydrogen peroxide (H₂O₂), may be due to the absence of additional heat treatment for the cathode powder, which could have removed impurities such as organic binders (PVDF or PTFE) and graphite carbon.

These leaching efficiency results corroborate the dissolution rate findings, which show that sulfuric and nitric acids are superior leaching agents for cathode powder compared to weak acids (acetic and citric acids).

Under these experimental conditions, it is evident that strong acids remain the most effective leaching solutions, although weak acids perform better in leaching lithium, even with the use of hydrogen peroxide.

4. Conclusion

The results of the experiments conducted on the acid leaching of cathode powder from used lithium-ion batteries demonstrate that an ecological approach has been adopted for leaching the metallic elements (Li, Mn, Ni, and Co) contained in this type of waste.

High dissolution rates were achieved at room temperature with sulfuric acid (63.65%), nitric acid (62.90%), and citric acid (47.50%) without the need for prior calcination of the cathode powder. The best results were obtained using strong acids in combination with hydrogen peroxide as a reducing agent for Co(III) and Mn(IV).

Calcining the cathode powder and adjusting the leaching conditions can enhance the leaching rates of bivalent metal elements with organic acids. Therefore, the hydrometallurgical method employed at room temperature is the most effective for recovering metallic elements. Citric acid presents a viable alternative to strong inorganic acids as a leaching agent for dissolving cathode powder and extracting metal elements.

However, processes involving citric acid require appropriate modeling and pre-treatment of the cathode material to maximize the recovery efficiency of the metallic elements.



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Conflicts of Interest

The authors declare no conflicts of interest.

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