

Hydrometallurgical Processing of Li-ion batteries from Electric Vehicles

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Abstract

The number Electric Vehicles (EVs) on the market is increasing as well as the number of end of life Lithium Ion Batteries (LIBs) that will enter the wastestream. It is of utmost importance to develop an efficient recycling process. A goal of this research is to suggest a process that can be implemented industrially. The several unit operations required, such as electrolyte removal, foil and powder separation and acid leaching were studied. Almost half of the electrolyte was removed, and the electrode parts were successfully separated using a brush. A mass balance of the components of the battery module and cell was carried out. The cell makes up 57% of the battery module, cathode and anode are 25% and 31% of the cell, respectively. The remaining are the outer aluminum case and organic materials.

Organic acid leaching is a rising process to attain a more sustainable recycling route, since it avoids acidic wastewater. The optimal leaching conditions were found to be citric acid at 1M as leaching agent, hydrogen peroxide 1%vol/vol as reducer, an L/S of 15L/kg, temperature of 90°C and leaching time of 3 hours. In the aforementioned conditions, 100% of cobalt and nickel and 90% of lithium could be leached. No more than 50% of aluminum was obtained.

Lastly, calcination temperature was evaluated, it was found that an increase in thermal treatment temperature, increases leaching efficiency due to an increase in powder reactivity.

Keywords: Lithium ion battery, Organic acid, Leaching, electrolyte removal

1. Introduction

In today's society, there is a growing concern about global warming and promoting a more sustainable industries and lifestyles. Energy generation accounts for 72% of global emission greenhouse gases (GHG) and transportation alone is responsible for 15% of overall energy consumption [?] [?].

In order to reduce the transportation share on this problem, internal combustion engine (ICE) vehicles are being replaced by hybrid and electric vehicles (EVs). Other motivation to move from ICE to EVs is the dependence on countries from the Middle East, that are the main suppliers of fossil fuels. In order to fully understand the potential of electric vehicles to overcome these problems a full lifecycle assessment that looks at the three stages, manufacturing, use and disposal must be analyzed. Several studies demonstrated that EVs are less pollutant during use, due to lack of tailpipe emissions, which decreases NO_x emissions and the power source is electricity as opposed to ICE vehicles that rely on fossil fuels. Nevertheless when considering electricity is also important to notice that if it is obtained from C instead of cleaner renewable sources the environmental benefit is less significant [?] [?] [?]. Considering the other two stages manufacturing

and disposal, EVs pollute more than ICEs due to the complex lithium ion battery design and exploration of raw materials required for its fabrication, specially cobalt. Also due to lack of design to dismantle, the recycling process becomes more complicated and expensive since an extensive number of unit operations are required to recover the valuable materials (Co, Ni, Li, C) [? ? ? ?].

Policies and European directives, namely the 2012/19/UE and 2006/66/CE from European Parliament and Council state that 25% and 45% of batteries and accumulators should be collected by the Member States by the end of 2012 and 2016, respectively and for batteries the minimum recycling rate was set to 50%. Due to the risk of explosion when batteries are exposed to high temperatures or damaged and the heavy metals present in the equipment that can leak and contaminate both the soil and groundwater, this waste was prohibit to be disposed in landfills. Nevertheless the residues from recycling are allowed to be sent there. Despite these directives and all the recycling processes already developed, the global collection rate of spent batteries is inferior to 10% and lithium recycling is below 1% [? ? ? ? ?].

Not only policies with regard to battery collection

but also incentives towards putting more electric vehicles on the road, namely the EV30@30, targeting a market share of 30% of EVs by 2030 have been proposed around the globe. If this goal is achieved, then an increase of LIBs in the waste stream is expected, which means that it is imperative to find a sustainable and economically viable recycling process to reduce the economic and environmental costs [? ?].

The battery main components are a steel and aluminum external case, print circuit boards and plastic cover. The cathode (positive side) that consists on an aluminum current collector foil layered with the cathode active material, sn LiMO_2 . The anode (negative side) which has a copper current collector foil coated with the anode active material. A binder, usually polyvinylidene fluoride (PVDF) that helps the adherence between the collector foil and electrode active material. A separator between the anode and cathode is needed to prevent contact between these two components and subsequent short-circuit. Lastly, the organic liquid electrolyte that contains a lithium salt usually LiPF_6 is present to allow lithium ions movement [? ? ?].

The current research focuses on recovering elements from a nickel-cobalt-aluminum (NCA) cathode from an electric vehicle. The increase of these vehicles, also increases the number of LIBs that go into the waste stream and require treatment. For environmental and economic reasons it is important to recycle the batteries, to avoid disposal in landfills, which would allow heavy metals, such as Co, to enter the groundwater and it enables the recovery of valuable raw materials with high purity [? ?].

Hydrometallurgy was the technology of choice, since it is less energy demanding than pyrometallurgy. Considering the design of a process as environmental friendly as possible, organic acid leaching will be implemented, instead of the common strong acids, which produce very acidic waste water and requires further treatment. Not only the chemical, but also the physical process will be studied.

Another goal is tantamount to close the cycle of lithium ion batteries, promoting a circular economy of this product. This will reduce the supply dependence from countries with geopolitical problems, such as Democratic Republic of Congo (DRC) and global availability of raw materials in the deposits.

2. Background

There are three different methodologies that can be used to recover value components or materials from lithium ion batteries and avoid the disposal of LIBs in landfills. These include direct recycling pyrometallurgy and hydrometallurgy [? ?].

2.1. Direct Recycling

Direct recycling uses physical processes to separate the different components present in the active material from the cells.

The main advantage of this process is that the materials can be recovered without chemicals, enabling its reuse with minimal treatment. Also it has a short recycling route, as it only requires discharging, disassembly, electrolyte removal, comminution (crushing) and physical separation (sorting). Besides this, it is a process with low energy consumption and high recovery. The major drawback relates with the absence of evidence that the recovered material will be able to produce batteries with the same performance, as the ones made out of virgin raw material. Being equal performance, a requirement for an efficient recycling route and circular economy. Even though it cannot be used in the manufacturing of new LIBs, it take part in production of goods for other distinct applications[? ?].

2.2. Pyrometallurgy

The second process that can be implemented to recycle LIBs is pyrometallurgy, which relies on high temperatures to obtain the valuable materials present in the batteries with a high recovery rate. The most commonly recovered raw materials are Ni and Co, the remaining, such as, Li and Al are lost in slag [? ?].

In 1998, the Japanese company Sony-Sumitomo developed a method in which the whole battery is calcinated at 1000°C forming an ash. This ash is rich in Co and Ni. After crushing and screening the powder can be further processed by hydrometallurgy to retrieve the metal oxides [? ? ?].

Alternatively, Umicore, a Belgian company, relies on smelting at high temperature, process with which natural ores are treated. The battery module is dismantled and the individual cells are fed in a shaft furnace (30-50%). Small quantities of coke, slag formers and limestone are added to ensure an economically viable operation [? ?].

Other industrial process was developed by the American company Inmetco and comprises four distinct steps. Initially, the individual cells are opened, prior to shredding the plastic parts are removed and the electrolyte is drained out.

Afterwards, reduction in a rotary hearth furnace at 1260°C takes place, the equipment is fed with the shredded cells mixed with the other solids (waste of Ni-Cd batteries and refractories with Cr, carbon and dolomite along with an organic reductant. The residence time is 20 minutes and the metal oxides are reduced to the metallic state. The metals are melted in a submerged electric arc furnace for smelting. An alloy containing Fe, Ni, Co and Mn is

formed.

Finally, the molten alloy is poured into molds which are cooled with water, "pig" alloy is produced and sold to the steel industry. "Pigs" usually have a composition of 10%Ni, 14%Cr, and 68%Fe. [?]

Similarly, to the other pyrometallurgical processes Li is lost in slag, due to its high reactivity and Ni, Fe, Cu and Co are recovered in an alloy [? ?].

Pyrometallurgy does not require a specific pretreatment, permits mixed wastes, such as LIBs or other battery types (e.g. Nickel Metal Hydride batteries) to be processed and is robust, so large volumes of waste can be fed at the same time. However, some of the valuable materials are lost in slag which hinders the recovery and some additional steps are required to recover the distinctive metallic elements. Also, this route is expensive owing to the high temperatures required, increasing energy consumption and costs. Lastly, the emission of greenhouse effect gases causes pyrometallurgical processes to have a negative environmental impact. [? ? ?].

2.3. Hydrometallurgy

The last process that can be followed for recycling LIBs is hydrometallurgy. Theoretically it grants the recovery of all the metallic compounds present in the spent batteries. Contrary to pyrometallurgy, hydrometallurgy requires physical processing after discharging and dismantling to maximize the recovery rate. This includes thermal and mechanical pretreatments [? ? ?].

Leaching is usually, carried out at a temperature below 100°C, which reduces energy costs, nevertheless expensive chemicals are required and the waste water produced has negative environmental impacts, if not treated. Several companies are already taking this recycling route. Also considerable researchers are studying alternative pretreatments and chemicals to achieve a process as efficient and sustainable as possible. These improvements can be attained by switching from strong acids to organic ones and reducing energy cost in the physical processing stage [?].

The individual cells can be separated into cathode, anode and separator, usually manually. The electrolyte is either volatilized [? ? ?] or collected. The most promising technique which allows the collection of the electrolyte was proposed by M. Grutzke's group and relies on supercritical CO₂ [?]. It is worthwhile mention that the electrolyte removal is done previously to manual separation or crushing. Chemical processing includes leaching and recovery of the metals from the leachate solution and residue. In the present context the term *residue* is used to describe the solid that did not

dissolve in the solution.

The leaching step requires a reductant, usually a base (H₂O₂ or Na₂S₂O₅), and a leaching agent, more commonly strong acids (HCl or H₂SO₄), some recent studies are focused on milder organic acids (e.g. citric and ascorbic). The base is required to increase the leaching rate, by reducing the transition metal oxides to the metallic state which facilitates its dissolution in the acid solution. It is proven that lack of reducing agent hinders the leaching efficiency, specially when inorganic strong acids are used [? ?].

Throughout the years, several researchers have been exploring different combinations of acids and reducing agents at different concentrations and processing conditions.

Most of the research in the recycling of LIBs focuses on NMC batteries, and very little information is available on NCA, which are the ones of interest.

The most commonly used leaching agent is sulfuric acid. However, this acid produces very low pH wastewater, which is a secondary source of pollution so it requires suitable treatment and leads to higher recycling costs. Several alternatives have been investigated to replace the strong acids, such as organic acids and ammonia. Ammonia, is deemed to be an effective selective leachant for copper, nickel and cobalt, due to the formation of amine complexes. Nevertheless, the recovery rate obtained when using this chemical is less satisfactory when compared to strong acids. Organic acids, on the other hand, are more environmentally friendly and have satisfactory results on recovery efficiency [? ? ? ? ?].

The main advantage of organic acids is that, contrary to HCl and H₂SO₄, there is no release of toxic gases, such as Cl₂, SO₃ and NO_x nor the production of acid wastewater, which are harmful to the environment. These are the two main benefits of applying these chemicals, however organic acids are more expensive than inorganic.

3. Experimental Methodology

3.1. Electrolyte removal

To avoid the formation of fluoridric acid and facilitate the collection and quantification of the electrolyte solution an open cell was wrapped in paper and sand, placed in a jar and heated to 100°C during several time steps. The goal was to find the minimum time required to collect the majority of the electrolyte. The initial and final masses of the cell and system were weighted in the beginning and at the end. The difference in mass allows to quantify leakage of the gas from the system. The cell was heated with and without the metallic case as well as without the polyamide, and the results compared.

3.2. Mass Balance

The individual components were separated by material type, carefully weighted and chemically analyzed to study the battery composition and compare the results with the ones available in literature.

3.3. Thermal Treatment

To study the best conditions to efficiently separate electrode and foil Hanisch's work was taken in consideration [?]. After unrolling the cell, 16 squares of 9cm were cut from the anode and cathode. Each square was then cut in four equal parts, which were placed in a crucible and thermal treated in a *Carbolite* and *Prolabo* muffles. Five temperatures were studied 150°C; 200°C; 300°C; 400°C and 500°C for 1, 5 and 10 hours.

3.4. Mechanical Treatment

After thermal treatment, mechanical treatment was carried out and five techniques were evaluated: ball, disk and rasp milling, ultra sounds and manual brushing.

3.5. Characterization

Prior to the chemical processing, it was of interest to characterize the collected powder chemically, morphologically. In order to achieve the aforementioned goal, Scanning Electron Microscopy (SEM) with Energy Dispersive Scanning (EDS). Inductive Coupled Plasma (ICP) spectroscopy and X-Ray Diffraction (XRD) were used.

3.6. Chemical Processing

Lastly, the recovered powder was leached. Three different organic acids were tested as leaching agent, citric acid (99.8%), gluconic acid (prepared from 98% grade salt) and oxalic acid (99.5%). The chosen reducer was H_2O_2 , since it is the most commonly used in organic leaching.

Processing conditions were varied, using a Factorial Design of Experiments (FDE) The parameters of study were acid leaching temperature, time and liquid solid ratio.

Sampling was carried out using the *Spinning rifle* from *Microscal Ltd*, 17 samples of 2.5g of cathodic powder were obtained.

In the first set of experiments the acid concentration, was kept at 1 M, in a 25mL round flask, temperature was set to 60°C and the liquid solid ratio was maintained at 10L/kg. Finally, to understand the behavior of thermal treatment in the leaching efficiency 0.5g of cathodic powder calcinated at 150°C, 200°C, 300°C and 400°C were prepared and leached under the optimal conditions.

4. Results

4.1. Electrolyte removal

The electrolyte removal was quantified weighing the cell and the jar with the paper, sand and cell alto-

gether, both in the beginning and at the end of the process.

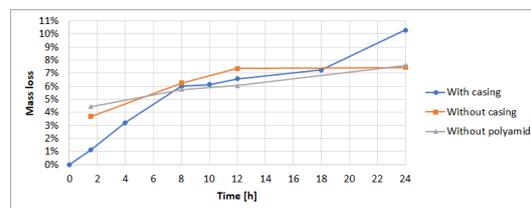


Figure 1: Mass loss of the cell with metal casing over time, when heated at 100°C.

As it can be seen in Figure ??, the mass loss reached a plateau after 8 hours, in the three conditions tested (cell with the outer metallic casing, without it and depleted from casing and polyamide). was taken in consideration, the author states that the volatile components of the cell make up 16% of its total mass Diekemann's work [?]. With the strategy followed on this research only 7% of volatile components could be removed.

4.2. Mass Balance

As it can be seen in Figure ??, the aluminum cell is the component that weights the most, which is in concordance with what has been reported in literature [?]. The outer metallic box of the battery that protects the cells and contains the back up battery and Print Circuit Board (PCB) are mainly produced from aluminum alloys and steel. Regarding the cell, which is the component of interest in the current research, the anode makes up 31% of the overall composition while the cathode accounts for 25%, the remaining 45% are distributed between the cell housing, electrolyte, separator and other organic parts. The materials were identified using XRD and an infrared gun.

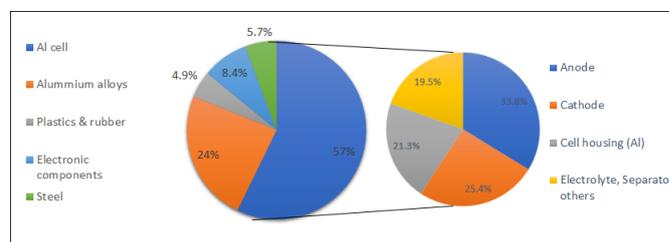
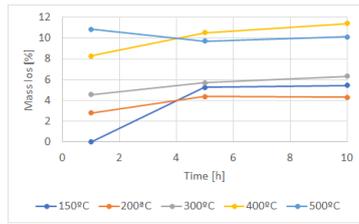


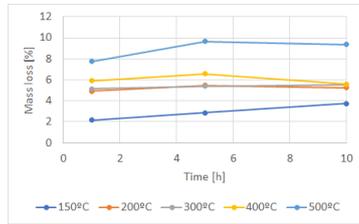
Figure 2: Mass balance by overall material and individual cell component

4.3. Thermal Treatment

The results of the thermal treatment carried out as explained in Section ??, are summarized in Figure ??.



(a) Cathode



(b) Anode

Figure 3: Mass loss of electrodes over time at five different temperatures.

In both electrodes, the trend is that with the increase in temperature and exposure time, the mass loss also increases. However, not all the conditions studied have this behavior. In the cathode the highest mass loss is after 10 hours at 400°C and not, as expected at 500°C. One explanation for this phenomena is that when placing the samples in the crucible, they are not evenly located, so some are more exposed to air and oxygen than others, which hinders the decomposition of the organic binder by combustion. Another explanation can be the oxidation of Al.

4.4. Mechanical Treatment

Disk mill was considered to be too energetic and the first samples were damaged, being the powder pressed against the equipment only after 3 seconds, so the next ones were treated by hand scratching and there are no results to present. All samples processed at 150°C and the sample treated at 200°C for 1 hour could not be completely separated with the spatula. On the other hand the cathode samples calcinated at 200°C for 5 and 10 hours were entirely separated with the spatula. The powder from the foils calcinated at 300°C could be entirely removed by hand scratching, while in the samples treated at 400°C and 500°C for 1 hour the two components were separated just by handling. This method was only valid for the cathode. Considering that the energy required to heat up from 400°C to 500°C is smaller than the one of leaving the equipment working for 5 hours, instead of 1, the optimal condition was selected as 500°C for 1 hour. With this in mind it was investigated if the calcination time could be further reduced. Time steps of 15, 30, 45 and 60 minutes were considered.

Similarly, the mass loss was calculated and is

graphically represented in Figure ?? . The mass loss of the cathode is higher after 30 minutes than after 15 minutes, as expected. However, after 45 minutes it drops, which can be due to a phenomenon of oxidation of the Al foil and then it increases again.

Considering the anode, there is an increase of mass loss until after 45 minutes, dropping after 60 minutes, which can also be explained by oxidation of the Cu foil. The mass loss increases, since the organic binder is being volatilized, after oxidation of the foil this parameter is influenced not only by decomposition of the binder, but also by the oxide being formed.

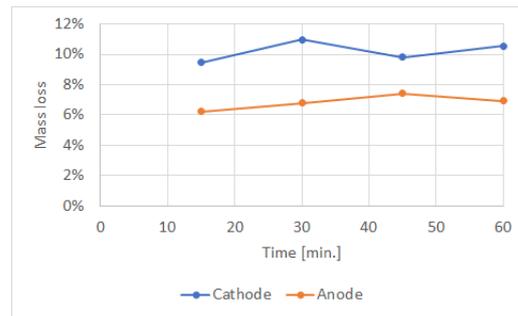


Figure 4: Mass loss of the cathode and anode when calcinated at 500°C for 15, 30, 45 and 60 minutes.

From Figure ?? it can be observed that rasp mill is the technique that showed the highest in three out of the four conditions being studied. The optimum condition is calcination for 30 minutes, with an efficiency of 94%. Furthermore, when using the hand brush, an efficiency of 90% could be achieved in all the conditions. It is worthwhile mention that as the force being applied to the cathode electrode decreases, the better the separation between foil and electrode active material. Alongside when hand brushing is the mechanical treatment of choice, time has almost no influence in the efficiency. For the leaching operation, the pre-treatment selected was calcination at 500°C for 30 minutes followed by hand brushing.

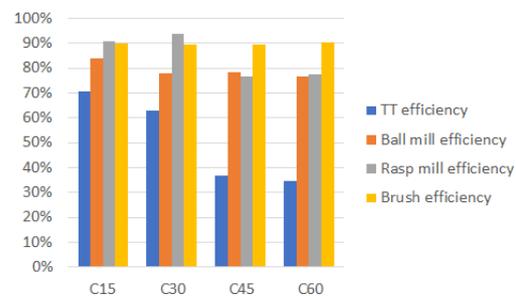


Figure 5: Efficiency of thermal and mechanical treatments for the collection of powder from the cathode.

4.5. Characterization

4.5.1 Phase Identification

From Figure ?? it is possible to confirm the oxidation of the copper foils, by the presence of CuO . Also graphite from the active material was detected on the foil. From diffractogram 2, it can be observe that on the cathodic foil after 15 minutes of thermal treatment at 500°C only Al is present. Regarding the cathodic powder (diffractogram 3) the presence of the NCA chemistry was confirmed.

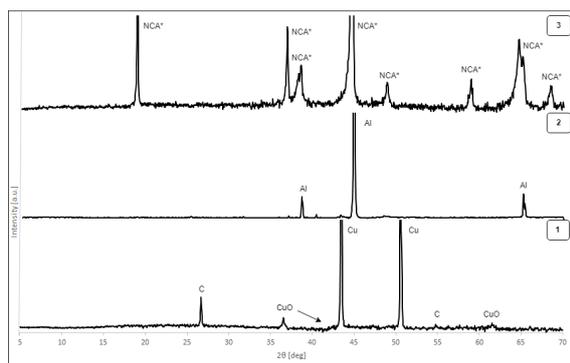


Figure 6: X-Ray Diffraction of the 1) anodic foil, pretreated at 500°C for 1 hour; 2) cathodic foil, pretreated at 500°C for 15 minutes; 3) cathodic powder, pretreated at 500°C for 15 minutes.

4.5.2 Elemental Characterization

In Figure ?? it is possible to observe the composition of the cell both in terms of principal components and the elemental composition of the two electrodes. The inner circle represents the components, while the outer circle is the elemental composition of the electrodes, since they are the objects of study in the current research. It is interesting to point out that in the anode and cathode, foil and powder have approximately the same contribution of 65% electrode active material and the remaining current collector foil. Regarding the anode, it should be noted that some researchers found that the contribution is fifty-fifty for copper and graphite, this difference in results can be explained by the fact that the amount of powder deposited in the depends from manufacturer to manufacturer, as well as the thickness of the foil.

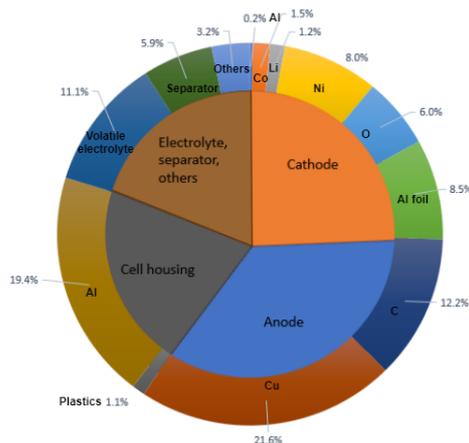


Figure 7: Cell composition by component and element

4.5.3 Chemical and Morphological Characterization

The cathodic powder is where most of research was carried out, for this reason only the SEM image of it will be displayed. Figure ?? is the cathodic powder from the most representative fraction, the infra $125\mu\text{m}$. Two different morphologies are identified, area 1 composed of very small spheres agglomerated and area 2, where larger spheres are identified. Regarding its elemental composition they are very similar and the detected elements are C, O, Ni, Al and P. Alongside these elements, area 1 also contains F.

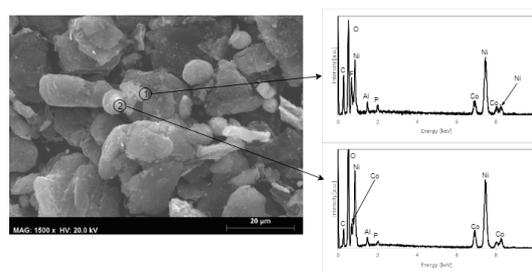


Figure 8: SEM and EDS analysis of cathodic powder fraction $> 125\mu\text{m}$.

4.6. Chemical Processing

The anodic powder and foil alongside cathodic foil were also characterized.

4.6.1 Acid Leaching

A preliminary acid selection in the leaching conditions of acid [1M], hydrogen peroxide [1%vol/vol], temperature of 60°C , time of 3 hours and an L/S of 10 L/kg, allowed to understand that citric acid has the capability of leaching Li, Co, Ni and Al. Oxalic

acid only leaches Li and Al. While gluconic acid does not react with Al. Therefore, acid citric and oxalic were selected for the FDE.

From Figure ?? it is possible to evaluate the behavior of each metal, over time when using citric and oxalic acid as leaching agents.

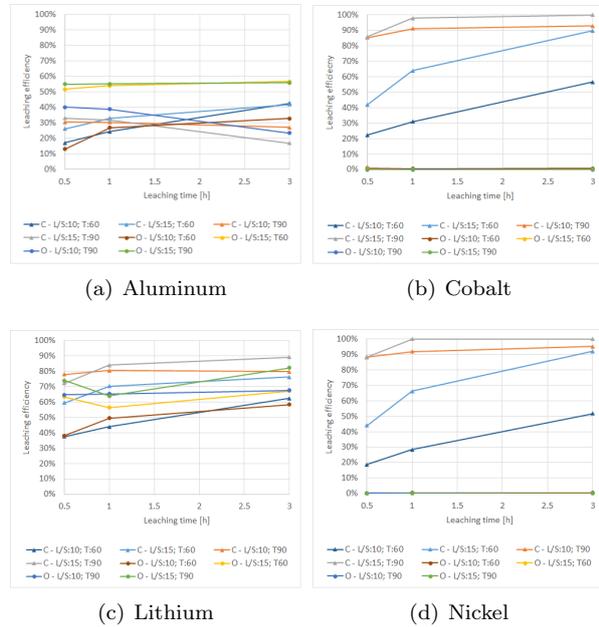


Figure 9: Leaching efficiency over time of the four elements of interest, using citric and oxalic acid. Temperature and L/S were varied as seen in the graphs. Acid concentration: 1M; H₂O₂ concentration: 1%vol/vol

It can be seen that cobalt and nickel have a similar behavior. For both metals, only citric acid is an effective leaching agent and 100% of leaching efficiency was attained after 1 hour. The best leaching conditions are 90°C, with an L/S of 15L/kg. After 3 hours all the cobalt was leached out. For nickel 1 hour was found to be enough to dissolve all the metal in solution. It can also be observed from Figure ?? that leaching efficiency with citric acid, increases with temperature for Co and Ni as well as with the L/S ratio.

Regarding aluminum, oxalic acid was the most efficient leachant. The leaching yield was the highest when using an L/S of 15. The increase in temperature from 60 to 90°C produced no change in the efficiency and only 55% of Al could be recovered from the cathodic powder.

When the L/S used was 10L/kg, at 90°C the efficiency dropped considerably after 1 hour. At 60°C there was a slight increase, of about 10% between 1 and 3 hours. Considering the citric acid at 60°C the yield after three hours was 42% for both L/S, however the ratio of 15L/kg had a higher rate than the one of 10L/kg.

Lastly, lithium was studied. The highest efficiency was obtained with citric acid at 90°C, being 90% leached after 3 hours with a ratio of 15L/kg. At the same temperature with the lower ratio the efficiency dropped to 80% this value is kept constant over time. It should also be noted that the in the least severe conditions with citric, the leaching rate is the highest. Considering the oxalic acid at 90°C with the L/S of 10kg, the leaching efficiency is found to be constant and of 65%. It is worthwhile mention that when a ratio of 15kg was used after 1 hour the efficiency dropped and increased again at the end of 3 hours. This oscillation can be explained by experimental errors, since several steps and measuring equipment are required in the leaching operation. To note that at 90°C the efficiency raised to 82%.

4.6.2 Influence of Calcination Temperature

The results, as seen in Figure ??, display that the four metals follow an identical trend and the efficiency increases proportionally with the calcination temperature. This allows to understand that the removal of the binder enhances the reactivity of the cathodic powder, probably due to an increase in contact area.

Also to note that at 300°C there is a decrease in efficiency, which is more relevant at the lowest yields, such as the ones attained for Al. This can be either due to an error during the experiment or a physical or chemical phenomena that happens at this temperature. It could be of interest to repeat these tests, to be sure unfortunately there was not enough material available to allow it. Additionally, there is very scarce on hydrometallurgical processing of NCA. As well as on the influence of thermal treatment in the chemical processing, therefore comparison with literature was not possible to fully explain the presented results.

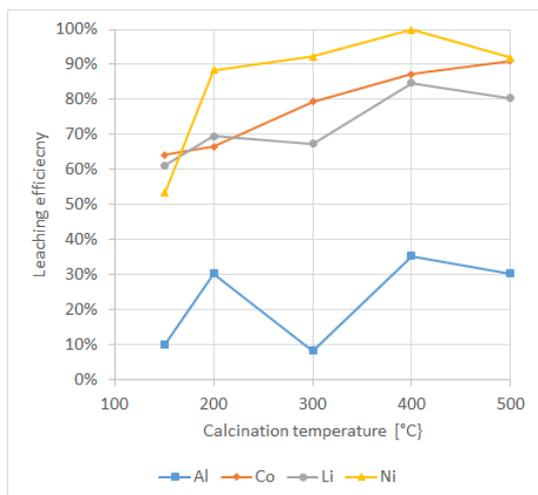


Figure 10: Leaching efficiency of each metal from the cathodic powder thermal treated at different temperatures

It is worthwhile mentioning that at 400°C the efficiency seems to be higher than at 500°C. This result can be explained by the different amount of powder used to perform the test at 500°C, 2.5g and at the remaining, 0.5g. Nevertheless, for cobalt as the temperature increases so does the efficiency and the aforementioned effect at 300°C is not present.

5. Conclusions

With this work, it was possible to fully characterize a lithium ion battery and produce a mass balance, which contemplates all the components in the module and cell. It was found that cell makes up 57% of the overall weight of the battery pack when the 35 cells are considered. The cell was further studied and it was concluded that the anode weights 34%, of which 22 percentual points are the graphite and the remaining 12 the copper foil. The cathode weights 25% of which 8 points are the aluminum foil, while the other 17 are the cathodic powder.

A method to recover the electrolyte was proposed, even though improvements are required. A system that combines the cell and an absorbent material capable of retaining and quantify the amount of electrolyte recovered, while preventing the emission of harmful gases can be implemented.

Foil and powder from the cathode were effectively separated using a brush. From an industrial point of view, the apparatus used in an automated car wash may be taken in consideration as an option to design a possible machine with a similar processing principle. The foil would be on two moving trails, brushes would contact the electrode in the two sides, to removal of the powder. The electrode and foil would be moving with opposite speeds, this would allow a constant movement of the brushes and a uniform powder collection rate.

Calcination was studied as thermal pre-treatment and it was found that heating at 500°C for 30 minutes is the best condition for an efficient mechanical treatment.

Leaching was investigated and the optimal conditions are citric acid [1M] as leachant at 90°C for 1 hour, using an L/S of 15L/kg and 1%vol/vol of H₂O₂ as reducing agent. The leaching efficiency in the aforementioned conditions was 35% for Al, 100% for Co and Ni and 85% for Li. The increase of calcination temperature improves the leaching efficiency in 20% to 30% depending on the metal.

It can also be concluded that oxalic acid is a selective organic reagent that can effectively leach lithium, while promoting the formation of oxalates of nickel and cobalt that can be precipitated and, therefore, recovered.

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