Industrial and Laboratory Processing of Spent Li-Ion Batteries for Efficient Materials Recovery

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Abstract—As the number of electric vehicles on the road grows, so does the need for solutions to treat the batteries used in these vehicles. The need to handle these batteries stems from the important raw materials that make them up, as well as a need to avoid exploitation of such materials in politically dangerous areas. Recycling appears to be a viable solution to these issues.

The goal of this project was to design a battery deconstruction operation and a deactivation process for further treatment on an industrial scale, as well as a mass balance of components and materials that make up the battery's system peripheral. The goal of the work at the laboratory scale was to determine the percentage of each substance, the type of chemistry used, and morphologically define some of the cells, all while investigating the effects of the previously imposed treatments.

The most commonly employed technology, based on the analysis of ten cells, is lithium nickel manganese cobalt oxide (NMC), specifically NMC111. The cell accounts for 68.08% of the overall weight of the battery, followed by the module (18.66%), and the battery periphery system that makes up the pack (13.26%). In terms of deactivation techniques, ohmic discharge is probably the best option, as it was discovered that, while discharging with a caustic solution does not significantly alter the cell structure, it does necessitate effluent treatment and some lithium ion leaching occurs.

Index Terms—Lithium-ion batteries (LIBs), Dismantling, Deactivation, Recycling.

✦

1 INTRODUCTION

Europe is the world leader in the production of automobiles. This industry employs around 13,8 million people. Despite numerous technical advancements, transportation still accounts for a quarter of Europe's greenhouse gas (GHG) emissions.

The Strategy for Low Emission Mobility, released in 2016, highlighted the need for zero- and low-emission cars, as well as initiatives to aid in the transition. At the time, lithium-ion batteries (LIBs) are the most widely used technology in electric vehicles (EVs). They do, however, present a number of obstacles, including the use of a variety of raw materials, some of which pose supply risks and are vital to the economy. Furthermore, because the production of electric vehicles involves several steps, analysing the environmental impact of each stage, from cradle to cradle, becomes critical.

Recycling offers a way to boost resource efficiency and increase the amount of raw materials available in Europe. With this in mind, the Batteries Directive (2006/66/EC) has controlled batteries and waste batteries since 2006, while the Directive (2013/56/EU) mandated minimum collection rates for retired batteries in 2013 [1]. Deficient legislation, ineffective collection infrastructure (it is believed that 95% of LIBs produced around the world remain untreated in households [2]), and low practical recycling technology have all hampered recycling rates to date. The need for batteries, on the other hand, is expected to climb 14-fold by 2030.

As a result, the European Commission (EC) presented a proposal in December 2020 with the goal of increasing portable battery collection and recycling rates by 65% in 2025 and 70% in 2030 [3], [4]. Although its widespread use is crucial, lithium battery recycling processes are extremely complex, especially because elec-

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tric vehicle batteries come in a variety of architectures and cathode compositions, making it more difficult to execute a holistic approach to their disassembly and recycling [5]–[7].

The ultimate goal of this research is to provide plenty of recycling instructions. Furthermore, a physical-chemical evaluation of the battery components of end-of-life (EoL) lithium batteries will be performed in order to develop methods for processing and recycling management.

2 LIB RECYCLING

Recycling, in addition to lowering environmental toxicity from the production of virgin materials and resource mining, is a viable strategy for the future due to the placement of materials back to the supply chain. It poses as a chance for Europe to lessen its dependency on certain suppliers with poor social and environmental records. Furthermore, recycling is predicted to become a significant sector, generating billions of dollars in revenue, tax revenue, and job opportunities [1], [8].

The purpose of recycling operations is to divide the components of spent batteries into smaller fractions that can be reintroduced into the manufacturing of new materials [9]–[11].

2.1 Challenges of Recycling

In an ideal world, recycling would return materials to their original state in a closed-loop recycling cycle. However, most recyclers down cycle, which means that their output is sold to other businesses. Another important issue is the high expense of collection and recycling.

Because of the range of EVs on the market, multiple pack configurations and methods to remove these packs are required. Various issues arise as the battery packs are dismantled, such as the removal of electrical wirings, the high voltages until the modules are separated, and the potential for other safety concerns. Sealants and thermal glues make module disassembly difficult; cells may also be enclosed and soldered together, and the state-of-health (SoH) may be unknown, a clear separation of components, the fact that the chemistries are not specified, diverse designs, and so on.

Existing battery designs could be modified for recycling as a solution. Ecodesign is a method that pushes manufacturers to build products that have a low environmental effect throughout their life cycle. The Ecodesign Directive was enacted in the EU in 2005 (2005/32/EC) for energy-using products, although it was later extended (2009/125/EC) to include energy-related products. This Directive works in tandem with other legislation, allowing the EC to establish minimum performance criteria while encouraging innovation. The greatest place to begin optimizing arrangements for end-of-life products is during the product design phase, as components that make a battery more difficult to dismantle can be substituted.

3 EXPERIMENTAL METHODOLOGY

The experimental methodology of this thesis is divided into two main parts: industrial and laboratory treatments. The first section of this methodology was performed at Ambigroup and Palmiresíduos. The second portion covers the work done in the laboratories of both Técnico and Laboratório Nacional de Energia e Geologia (LNEG), mostly at the cell level, where battery cells were discharged, dismantled, and chemically and physically characterised using a variety of techniques.

3.1 Industrial Treatments

The process at both companies began by opening the containers in which the packs were brought. After the containers were opened, the batteries were examined using a voltmeter to see if any electrical current was flowing through their external case. Forklifts were used to transfer them to the workstation once they were declared safe. Following that, the top outside lid and all external screws were removed, and the pack was examined with a multimeter to determine its voltage (V).

If the battery pack was deemed activated (depending on the number of modules, but usually above 15V), the process was to deactivate them. Alternatively, the next step would be to dismantle them.

3.1.1 Deactivation

Deactivation can be achieved using several methods (ohmic discharge, brine solutions, etc) and it is made to ensure that the risk of explosion is reduced [9], [12].

Palmiresíduos and Ambigroup batteries were deactivated using ohmic discharge (electric heaters as resistors) and a brine solution. The parameters for determining which direction the pack would take were simple (Fig. 1): if the pack's terminals were easily accessible, the pack was discharged. If not, the modules were separated and removed from the pack's outer lid before being placed into the solution. At both locations, 200L tanks were provided and filled with water containing 0.3%wt NaCl.

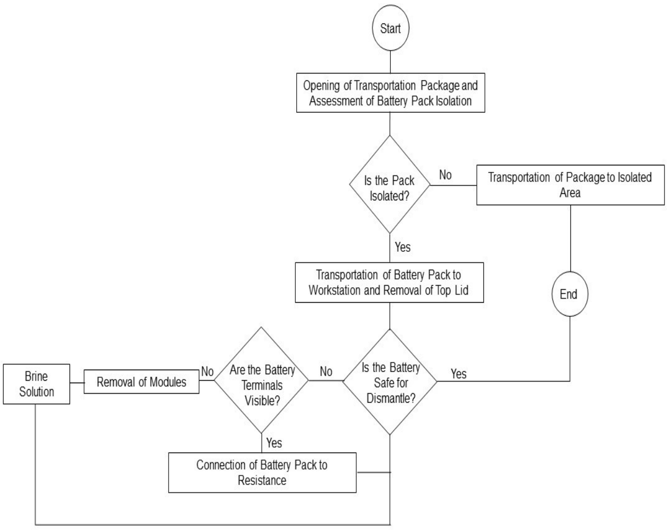

Figure 1. Deactivation procedure.

3.1.2 Dismantling

When the battery packs were deemed discharged (below 15V) or the modules' voltage fell below 6V, the disassembly process began. The goal of disassembling battery packs into modules and modules into cells is to ensure a clear separation of components, allowing valuable materials to be easily targeted for recovery.

This procedure begins by opening any remaining covers (some may need to be opened earlier to ensure access to the battery's terminals for discharging), followed by retrieving any electrical connectors. This procedure is seen in Fig. 2.

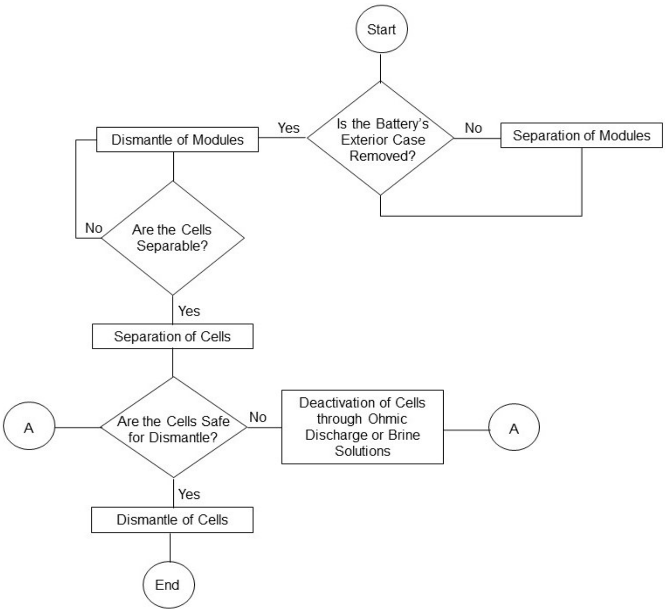

Figure 2. Dismantling procedure.

3.1.3 Material Balance

As the batteries are being dismantled a variety of components and materials can be obtained. The aim of this step is to determine the average weight of different components to contribute to a more assertive report on the possible profit for recyclers.

3.2 Laboratory Treatments

When all of the battery modules had been removed and the cells separated, they were transported to the laboratory to be deactivated, dismantled and characterised.

3.2.1 Deactivation

To discharge the cells, bulbs were used to perform an ohmic discharge and different caustic solutions were used. Using a voltmeter, the voltage was measured during deactivation to ensure that the values tended to zero.

Each pack was assigned a different digit to properly distinguish them. From now on, the first digit (which identifies the packs) will be followed by a second digit indicating the method of discharge used:

- 1 means deactivation with brine solution at a concentration of 20%wt NaOH where the cells' voltage was measured hourly;

- 2 means deactivation with brine solution at a concentration of 20%wt NaOH but the cells's voltage was only measured after 24H and 48H;
- 3 means deactivation with brine solution at a concentration of 10%wt NaOH where the cells' voltage was measured hourly;
- 4 means deactivation with brine solution at a concentration of 15%wt NaOH where the cells' voltage was measured hourly;
- 5 means that the cell was deactivated using ohmic resistance;
- 6 means deactivation with brine solution at a concentration of 0.3%wt NaCl;

3.2.2 Dismantle

After the cells are considered deactivated (below 1V), the next step is to disassemble them (as with packs and modules).

If it's a pouch cell, simply slit it open to expose the electrodes. Cylindrical cells, on the other hand, must be sawn to expose the electrodes, which is a rather simple process due to their soft shell. Prismatic cells are the most difficult to open due to their hard external shell, hence spinning discs were used in the laboratory to access their electrodes. After the cells are opened, the components are dried in a vented furnace to guarantee that all electrolyte and bath residues (if any) are removed.

3.2.3 Characterisation

Elemental Composition by ICP-AES
In this technique the emitted spectra is used to determine the sample's qualitative or quantitative elemental composition [13]. To analyse the electrode samples (solid), they must go through an acid digestion process to produce liquid samples. For this objective, an *Aqua Regia* solution was utilized, which is composed of hydrochloric acid (HCl) and nitric acid (HNO_3) in a 3:1 ratio. The samples are immersed in the solution and allowed to react for an hour. The mixture is next filtered, and the resulting solution is sent to be analysed.

After a complete solubilisation of the electrodes, the weight percentage (%w/w) of the elements was calculated according to the Eq. 1.

$$(\%w/w) = \frac{Concentration[mg/L]*InitialVolume*DilutionFactor}{Weight[mg]} \tag{1}$$

X-Ray Powder Diffraction (XRPD)
This technique uses the elastic scattering of x-rays by atoms in a periodic lattice to determine an unknown material. Using Bragg's Law (Eq. 2), this approach produces the lattice spacings.

$$n\lambda = 2dsin\Theta \tag{2}$$

The anode and cathode were scrubbed individually until the black matter detached from the foils in order to analyse the electrode samples. The black matter was crushed with a mortar until it became powder, then placed in a sample holder and scanned between 5°and 80°of 2Θ using CuKα radiation with a producing step duration of 74.0066s and generating settings of 35mA and 40kV.

Scanning Electron Microscopy (SEM/EDS)
The most common SEM modes involve the capture of secondary and backscattered electrons by a photomultiplier and the formation of a picture that provides chemical compositional data (EDS technique) and as long as the sample is conductive, it can be scanned.

The samples used in the XRPD analysis were also utilised in this analysis, with the key difference being that the samples were coated by sputtering with gold (Au) and palladium (Pd) to ensure that they were electrically conductive.

4 RESULTS & DISCUSSION

4.1 Industrial Treatments

4.1.1 Deactivation

The first step is to deactivate the battery, however, since no measurements were taken at the facilities regarding the rate at which they discharged, this step will be ignored for now.

4.1.2 Dismantle

The problems of this process begins with the transfer of the battery to the work station, which requires the use of forklifts and thus a specialised worker. Following that, the tools used to remove the external case must be planned. The second step in this procedure is to remove all screws and bolts. Because the sizes of packs and modules fluctuate, different hand tools have to be used. It is not a difficult task, but it can take up to 10 minutes to remove all exposed screws and attachments. This task is hampered when manufacturers employ adhesive to prevent screws from getting loose, making it more difficult to remove them.

After that, the modules must be removed from the pack. This is apparently a simple step; however, some manufacturers utilise a type of thermal glue that allows heat to escape through the structure while also attaching the modules to the external lid. Another issue discovered was that when pieces were welded together, they were impossible to separate using merely force, necessitating the employment of additional equipment such as rotating discs for cutting. When the modules are separated, they must also be dismantled, which necessitates the use of additional and smaller tools to remove all screws. To avoid trepidation of the cells, they are frequently housed in plastic frames that must be cut or broken.

Table 1 summarises the information obtained when all cells were retrieved.

4.1.3 Material Balance

Batteries are made up of various components and materials, each with its own weight and monetary value. For recyclers, the most attracting components are the external cases made of metals, the battery management system (BMS), printed circuit boards (PCB) and all of the electrical cables. These components are made of valuable and critical metals such Au, Pd, silver (Ag) and copper (Cu). Modules have a lower monetary value because of their increased plastic fraction.

Table 1
Summarized information of the LIB modules used.

Battery Pack	Enterprises	Cell Type	Cell Weight (Kg)	Number of Cells per Module
1	Palmiresíduos	Prismatic	1.702	8
2	Palmiresíduos	Prismatic	0.722	13
3	Palmiresíduos	Pouch	0.530	9
4	Palmiresíduos	Prismatic	0.563	8
5	Palmiresíduos	Pouch	0.698	50
6	Ambigroup	Pouch	0.745	48
7	Ambigroup	Pouch	1.269	30
8	Ambigroup	Pouch	0.965	24
9	Palmiresíduos	Pouch	0.745	48
10	Técnico Laboratory	Cylindrical	0.437	196

4.2 Laboratory Treatments

4.2.1 Deactivation

The majority of experiments employ NaCl saline solutions to discharge the batteries. When electrolyzed, it creates hydrogen and chlorine gas, and chlorine ions promote the aqueous corrosion of steel [14]. With this in mind, solutions containing 10%wt to 20%wt of NaOH and distilled water were tested.

Conductivity depends on the ionic nature of the compounds dissolved, and its corresponding capacity to dissociate into charged ions to carry the charge (Eq. 3). If a solution has higher conductivity the lower its resistance, consequently, the faster the ions move, the quicker the discharge of the cell is until the equilibrium of components is reached [14].

$$R(\Omega) = \frac{1}{Conductivity(S.m^{-1})} * k(m^{-1}) \quad (3)$$

K stands for the cell constant, which is equal to the distance in metres (m) between the probe's electrodes divided by the surface area of the electrodes in m^2.

Depending on the shape of the cell, cells with the greatest distance between the electrode probes and the smallest surface area have higher resistance, will have different discharging profiles. In Fig. 3, 4, and 5 the behaviour of each cell in different solutions is analysed.

The discharging profile of each cell type is visible, indicating the relationship between cell form and discharge efficiency. It can be seen that the cells projected to have the highest resistance (type cell 1) have more difficulty discharging. The cells that attained lower charge values the fastest (type cell 2), on the other hand, had the lowest theoretical resistance, all of this because of the way they each were assembled.

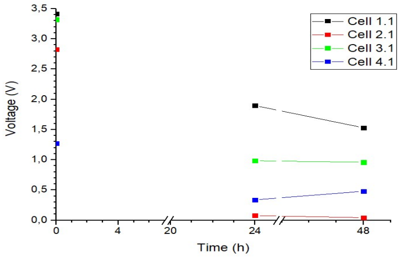

Figure 3. Brine solution 20%wt NaOH.

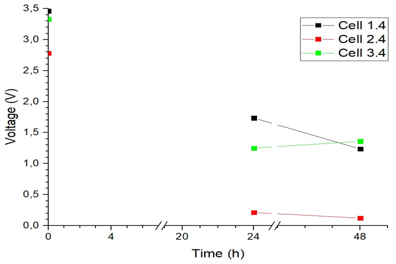

Figure 4. Brine solution 15%wt NaOH.

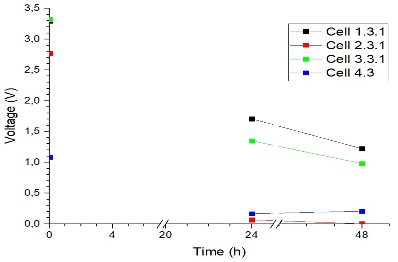

Figure 5. Brine solution 10%wt NaOH.

4.2.2 Balance of Components and Materials

Given the work done in the laboratory, it is also necessary to offer an estimate of the components and materials at the cell level. Most cells use metals as their external casing, the separator is a porous polymer membrane that has been impregnated with the electrolyte and is dried after opening. Plastics are used as wrappings in prismatic cells to close the rolling electrodes. In reality, disassembling these cells revealed that prismatic and cylindrical cells are essentially identical in their assembly, with the only difference being the cross section caused by the rolling electrodes.

The anode's components are most likely graphite and Cu at the current collector foil. The alternatives at the cathode are substantially broader, and the value of cells might vary depending on the metals utilised. This means that different chemistries have varied costs; for example, an LFP battery will be less expensive than an NMC battery because it needs less critical metals. Regarding recyclers, the only thing that can be said is that extracting metals from black mass is still a time-consuming and inefficient process, which means that at this stage, recyclers should focus on disassembling packs and processing the materials used in the external case and modules, and selling the cells to second-life businesses after assessing them and their SoH.

Following completion of this portion of the task, an average composition of a battery can be provided (Fig. 6). The outside case accounts for 13% of the weight, the module for 19%, and the cells for 68%. The anode accounts for 22% of this 68 percent, the cathode for 26%, the electrolyte for 11%, and the outer case for 6%, with the separator and plastics accounting for less than 5% of the cell's weight.

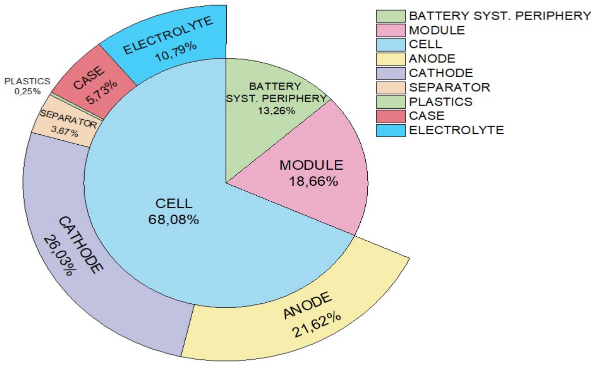

Figure 6. Average battery pack components.

4.2.3 *Characterization*

Elemental Chemical Composition

The results coming from this analysis give the concentration of each element in each sample using Equation 1, and the results are shown in Table 2.

Table 2
Cathodic material composition of NMC cell samples.

Sample	Cathodic Composition
1.1	LMO-NMC111
2.1	NMC333 / NMC111
3.2	LFP
5.5	NMC333 / NMC111
6.6	NC532
7.5	NMC333 / NMC111
8.5	NMC622
9.6	NMC532
10.5	NMC811

Cell 3.2 uses LFP technology. With the exception of cell 3.2, the cathode samples demonstrate that the most commonly employed technology is NMC due to the continual presence of elements such as Ni, Mn, and Co. It is important to remember that no data is given for pack manufacturer 4 since the sample regarding this type of battery was very damaged after exiting the bath and the conclusions would always be hindered due to the impossibility of separating the anodic and cathodic material.

Morphological and Chemical Characterisation of Phases

Graphite, aluminum, corundum, and lithium nickel oxide are the most common chemicals discovered at the anode of cell 2 (Fig. 7). Graphite was found as the anode material, which is not surprising given its outstanding electrochemical performance and specific capacity.

Aluminium is present because it was used to make the external connectors at the cell, which deteriorated during the NaOH bath and ended up in the electrode material. Corundum, or Al_2O, is a well-known crystalline form of aluminium oxide that could have formed as a result of the interaction of aluminium with NaOH atoms and water because as an amphoteric substance it can react with both acids and bases. Finally, lithium nickel oxide, or Li_2NiO_2, is widely employed because cathodes layered with lithium transition metals oxides present higher theoretical capacity (≈ 270mAh/g), and higher average operating voltages (≈ 3.6V vs Li^+/Li) [15]. The fact that it is displayed in the anode material rather than the cathode material is most likely due to some cross contamination while handling the samples for testing.

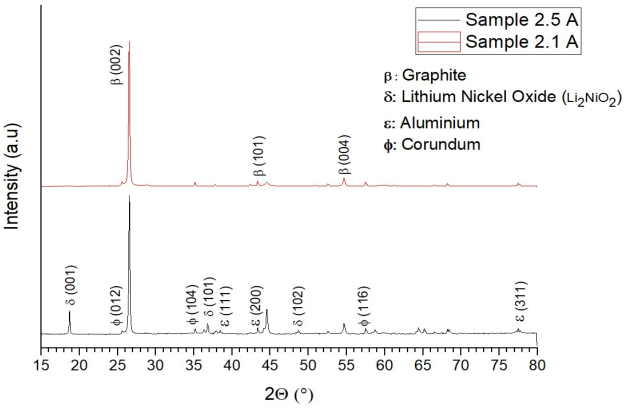

Figure 7. XRPD analysis of sample 2 anode.

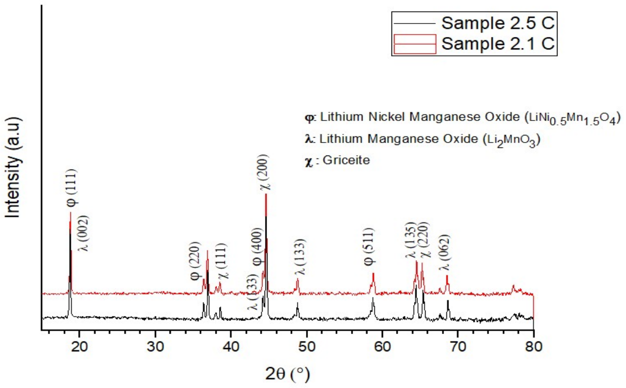

Figure 8. XRPD analysis of sample 2 cathode.

The presence of lithium nickel manganese oxide in the cathode sample (Fig. 8) is explained by the nature of this battery, which utilises NMC cathode technology.

Griceite, LiF, is utilized as an additive to increase metal surface diffusion at the SEI layer during electrodeposition, as well as a source of ^{6}Li isotopes. Another explanation for its appearance is that it is caused by the leaching of anode material coupled with HF [14].

The anode samples of cell 2 may be shown in Figure 9 and 10. The EDS spectra given supports the XRPD study, in which carbon is the most prominent element, followed by oxygen, which is related to the discharging process used, and aluminum, which is derived from the foils on which the black material is deposited. The presence of gold is explained by the fact that it is used as a conductor in SEM examination. One key point to note is that in Figure 10, two spectras are shown. This occurs because a contamination was discovered in the anode sample, the presence of cathode material (Co, Ni and Mn).

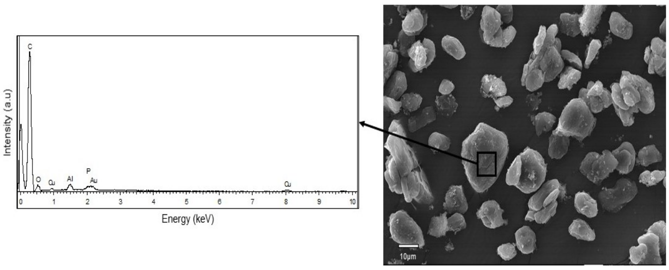

Figure 9. SEM/EDS analysis of sample 2.1 anode.

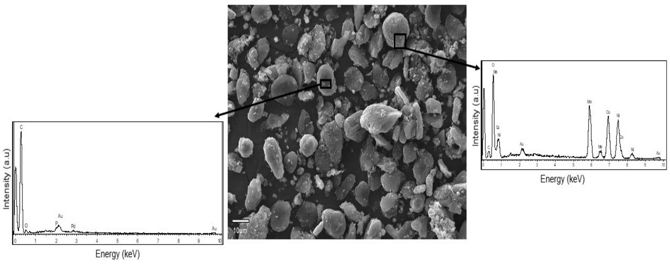

Figure 10. SEM/EDS analysis of sample 2.5 anode.

The patterns obtained for battery pack 3 are consistent with the elemental chemical analysis performed. The primary compounds detected at the anode (Figure 11) are graphite and griceite, which presence was previously described.

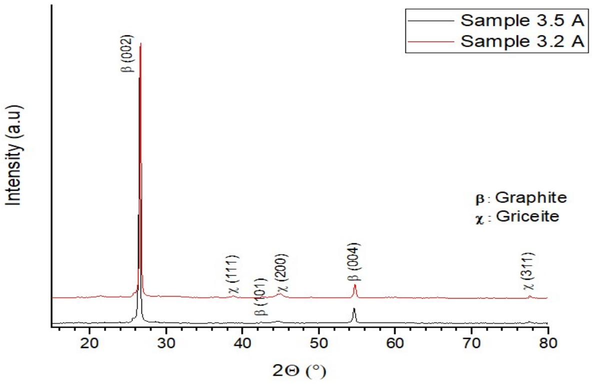

Figure 11. XRPD analysis of sample 3 anode.

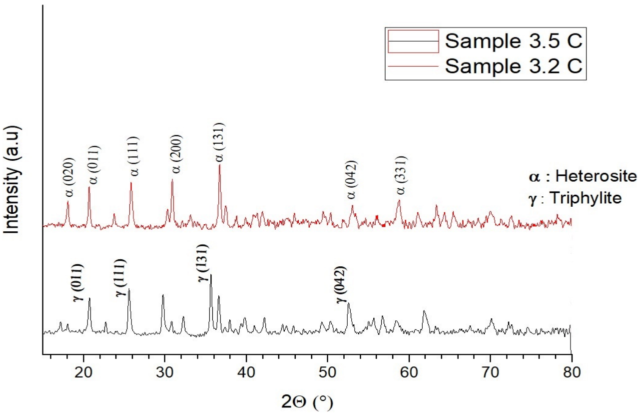

Figure 12. XRPD analysis of sample 3 cathode.

The EDS spectra revealed not only the primary elements (Fe, P, O), but also V (Fig. 14 and 15). Some works have suggested that if a trivalent cation could be incorporated on the Fe site in heterosite, then that would lead to vacancies on the lithium site in order to balance charge. Consequently having enhanced lithium mobility and higher power capabilities. Vanadium would then be a great candidate, and was found to enhance the electrochemical performance of the cell, even though it occupied the P site and not Fe, decreasing the unit cell in size [17]. However, this hypothesis cannot be proven as V was not detected during XRPD analysis nor within the structure.

The most common compounds found in the cathode were heterosite and tryphilite (Figure 12). These minerals form in the cores of granite pegmatites (heterosite-purpurite and triphylite-lithiophilite series, respectively). The thriphylite series phosphate is an olivine-type mineral with a grayish-blue color. These minerals are frequently exposed to aqueous rest liquid and undergo step-wise changes. Heterosite, which is found in secondary, weathered pegmatites, is one of these altered minerals. The creation of these isomorphous heterosite-purpurite series is caused by the oxidation of Fe^{2+}, accompanied with a reduction in lithium concentration, followed by the oxidation of

Mn^{2+} and total depletion of lithium [18]. The hexagonal dense packing of oxygen atoms in heterosite's crystal structure is similar to that of tryphilite, but with ordered vacancies in the cation position left by the lithium (Figure 13).

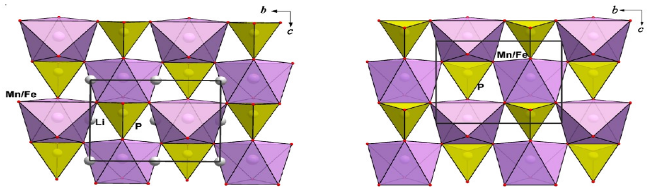

Figure 13. Layers of Mn/Fe centred octahedra and PO_4 tetrahedra parallel to *yz* plane in lithiophilite [left] and purpurite [right]. Reproduced from ref. [18]

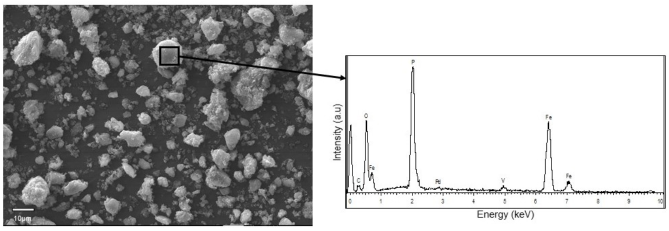

Figure 14. SEM/EDS analysis of sample 3.2 cathode.

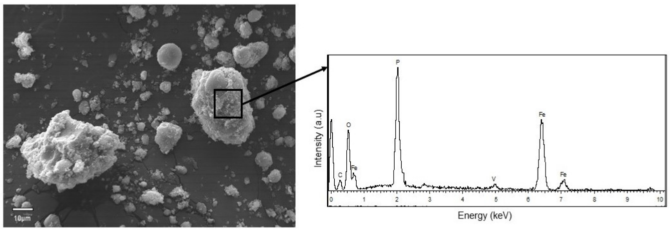

Figure 15. SEM/EDS analysis of sample 3.5 cathode.

5 CONCLUSION

Working directly with recyclers provided a unique view on the issues related to batteries and how to design an effective disassembly line. The direct interaction between recyclers' collaborators (and the bibliographic research supplied) allowed for a process to properly discharge the batteries, followed by a dismantling sequence.

Eco-design should not be limited to end-of-life businesses. One of the tenets of the circular economy is the reuse or reconditioning of parts in the same or other uses. When it comes to LIBs derived from EVs, they are typically used in a different field. This means that there are a lot more other businesses that rely on battery dismantling and reconditioning. As a result, it is critical that manufacturers reconsider their assembling strategy in order to assist downstream businesses that rely on the materials, and components that come their way.

In this study, the module, which is made up of screws, metals, plastics, circuit plates, and cables, accounts for 18.66% of the overall weight, while the battery system periphery, which includes the lids, BMS, and some screws, accounts for 13.26%. When it comes to the profit that will be made by recyclers, no view was formed due to the impossibility of reaching an exact conclusion. The materials collected at the firms could not be accurately recognised, and at the cell level, the fact that this thesis does not cover the recovery of metals in the black mass hampered the possible findings. For the time being, recyclers should concentrate on recovering the materials that make up the exterior battery casing, which are primarily metals and have a higher commodity price. Furthermore, retrieving PCBs, electrical cables, and the BMS may present as a better opportunity.

Concerning the morphological characterisation and the caustic solution influence, in terms of new compound forms some new minerals were formed. The NMC cell batteries were as affected by the baths as the LFP samples, independently of the shape of the cells (one are prismatic and the other pouch) and their components being more prone to react than others. The discharging method, is safe to say, is the one that will allow batteries to maintain their most original form, even with some delithiation. Another concern regarding the deactivation through caustic solutions is what to do with the liquid effluents.

6 ACKNOWLEDGMENTS

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