# Physical Method for the Recycling of Lithium Iron Phosphate (LFP) Cells

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## 1. Introduction

In 2019, 74,900 t of lithium-ion batteries (LIBs) were placed on the European Union (EU) market, of which 51% were industrial and automotive batteries (European Commission, 2019). LIBs are seen as excellent secondary resources for the recovery of lithium, a critical raw material (European Commission, 2022). EU member states have the challenge to achieve a minimum collection rate for LIBs of 70%, and a recycling rate of 70% for lithium by 2030 (European Commission, 2020). Therefore, more efforts should be made to recover lithium and other metals through recycling methods (Castelvecchi, 2021). There are different types of currently used LIBs, distinguished by the chemical composition of the cathode material. A common type is the Lithium Iron Phosphate (LFP) cell (Zhao et al., 2021). Considering that solutions for an efficient recycling process of batteries urges, this study aims to characterize the LFP cells to the elemental composition, and propose a mechanical process to obtain rich fractions of valuable materials. The knowledge about the elementary composition of the batteries and the proposition of initial concentration steps to valorize the residue are essential to improve the efficiency of the entire recycling routes.

## 2. Material and methods

Lithium Iron Phosphate (LFP) cells were subjected to chemical characterization as well as to the mechanical separation process by milling and sieving. To characterize the elemental chemical composition of the cell studied, approximately 500 g of cells were comminuted in a Retsch model SM300 knife mill (rotation speed of 1500 rpm), passing once through each grid, from the largest to the smallest, whose opening sizes were 9.5, 5, 2, 1, 0.75, and 0.5 mm. This sequence of screens was used in order to not cause damage to the mill and to intensify the separation of the cathode materials from the cathode support. The ground material was quartered, and six samples of approximately 5 g were subjected to acid leaching with aqua regia (75% HCl and 25% $HNO_3$) for 2h, with heating (temperature 70-80ºC), and a solid-liquid ratio of 1/40. After filtration, the liquid fraction containing the solubilized metals was analyzed by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). These results were set as reference values to evaluate the subsequent recycling process.

The recycling by mechanical processing consisted in grinding about 500 g of LFP cells for only one cycle in the Rone SRB 2305 mill, for two hours. The equipment grid had a 9.5 mm opening, which already previously separated a material with a particle size greater than 9.5 mm. The rest of the ground material was also collected and passed through a sequence of bench sieves with a vibration system, whose openings were 9.5, 4.75, 2, 1, and 0.5 mm. During sieving, 30 g of ground material was put through the system every 15 minutes. After homogenization and quartering of the fractions obtained, 3 samples of 5 g (of each fraction, totalizing 18 samples), were leached and analyzed by ICP-OES in the same way as previously reported.

## 3. Results and discussion

The elemental increments and decreases of each fraction compared to the untreated LFP cell are shown in Table 1. The elemental composition of each fraction obtained by the mechanical treatment is shown in Figure 1, as well as the representativeness of each fraction in relation to the LFP cells, both in mass percentage.

Table 1. Comparison of the elemental increments or decrements of the fractions obtained by recycling via grinding and sieving, in relation to the untreated LFP cell.

| | Increase/decrease (%, by mass) compared to untreated LFP battery | | | | | |
|---|---|---|---|---|---|---|
| | n > 9.5 mm | 4,75 < n < 9.5 mm | 2 < n < 4.75 mm | 1 < n < 2 mm | 0.5 < n < 1 mm | n < 0.5 mm |
| *Al* | -69 | <u>95</u> | <u>74</u> | <u>26</u> | -75 | -95 |
| *Cu* | -83 | <u>50</u> | <u>156</u> | <u>102</u> | -56 | -89 |
| *Fe* | <u>86</u> | <u>74</u> | -26 | -26 | 0 | -7 |
| *Li* | -53 | -64 | -41 | -20 | <u>74</u> | <u>58</u> |
| *P* | -89 | 37 | 75 | 87 | <u>207</u> | <u>172</u> |

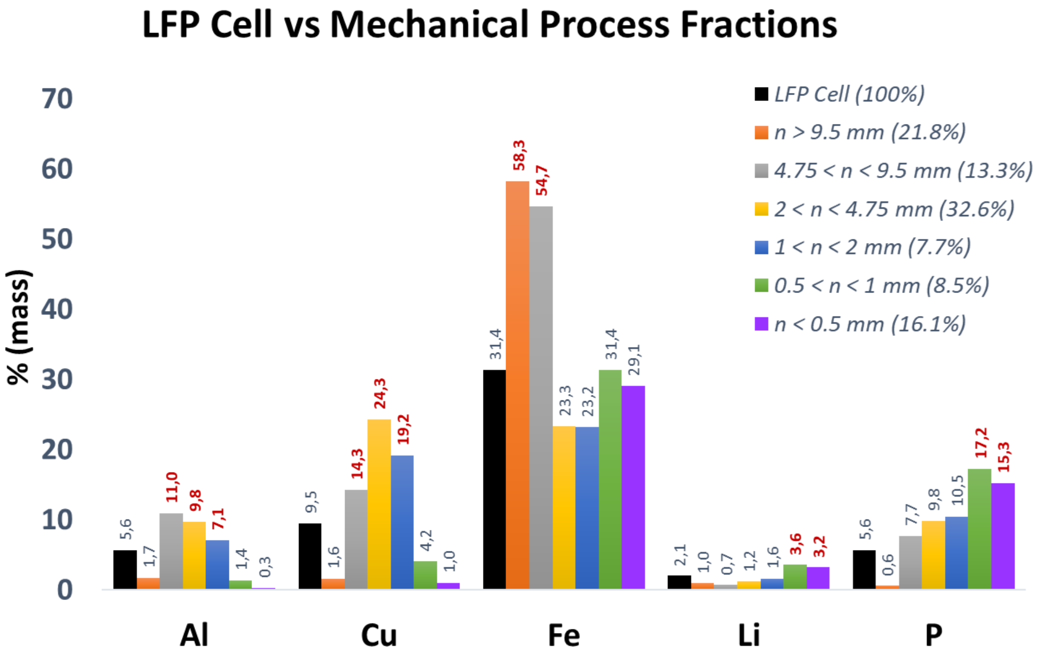


**Figure 1.** Chemical composition of the LFP batteries and the fractions obtained by milling and screening.

The mechanical treatment enabled the lithium to be 74% and 58% more concentrated at 0.5 < n < 1 mm and n < 0.5 mm, respectively, compared to the untreated LPF cell. This lithium concentrate (n < 1 mm) represented 24.6% of the battery mass and has more than 3% lithium. It was observed that aluminum (cathode support) and copper (anode support) tended to have intermediate particle sizes (4.75 < n < 1 mm). Relatively to the unprocessed cell, the 4.75 < n < 9.5 mm fraction had 95 % more aluminum, and the 2 < n < 4.75 mm fraction had 156 % more copper. In addition, more than 50 % of the fractions 4.75 < n < 9.5 and n > 9.5 mm are composed of iron (from the casing), which add up to 31.5 % of the LIB mass.

## 4. Conclusions

Despite being a simple process, the mechanical treatment evaluated was promising as a first step in recycling lithium iron phosphate cells. Lithium was concentrated 58-74% more at particle size n < 1 mm. Aluminum was concentrated 74-95% more at particle size 2< n < 9.5 mm. Copper was concentrated by 102-156% more in particle size 1 < n < 4.75 mm. Iron from cell casing was concentrated 74-86% more at particle size n > 4.75 mm. The developed process had only 1 grinding cycle and 1 screening cycle, which are easily applicable at an industrial scale with relatively low investment and lower environmental impact compared to pyrometallurgical or chemical processes.

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