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### Flotation Technique for Recycling Black Mass from Spent Lithium-ion Batteries

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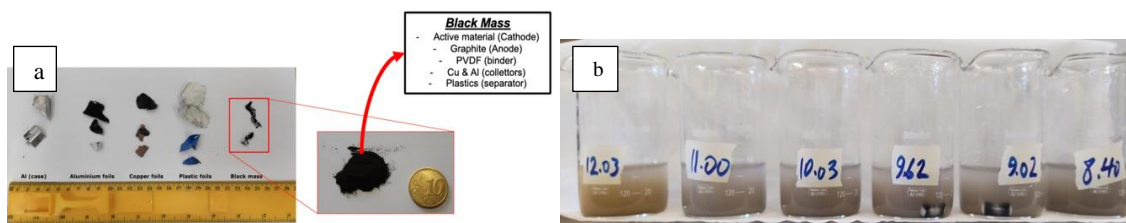
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The rapid increase in the adoption of lithium-ion batteries (LIBs) in consumer electronics and electric cars has highlighted the need for efficient recycling methods. This study, within the framework of the European Battery Innovation - EuBatIn initiative, aims to develop a sustainable and industrially scalable hydrometallurgical process for recycling spent LIBs. One of the many challenges to face in is to get an efficient separation of the components of the black mass (BM) – a fine, powdery material comprising cathode and anode active substances (typically metal oxides and graphite), as well as metallic collectors such as copper and aluminum, and residual plastics – produced from the shredding of batteries.

This research specifically investigates experimental conditions for an effective flotation process for separating BM components, particularly focusing on two key objectives: i) the isolation of graphite from the cathode active material, and ii) the separation of distinct cathode active materials (e.g., nickel manganese cobalt oxide (NMC), lithium iron phosphate (LFP)) in case of mixed-chemistry battery batches. The flotation process hinges critically on the surface and charge properties of the materials involved, making the determination of the point of zero charge (PZC) a crucial step for optimizing separation efficiency.

Given the limited availability of data on the PZC, especially for representative samples of BM of spent LIBs, this study aims to fill this knowledge gap to design robust and effective flotation protocols. A systematic comparison was conducted using three commonly employed methods for PZC determination (drift method, titration, and mass titration) to assess the results consistency and/or limitations. Experiments invariably revealed notable differences in PZC (~2.5 pH units) for pristine battery-grade LFP and NMC samples, indicating that charge-driven surface manipulations could be leveraged to enhance the efficiency of separation by flotation. Furthermore, PZC measurements for cathode and anode materials collected from spent LIBs have been performed to highlight the role of particles dimension and of matrix interferences.



**Figure 1.** a) Main components of a LIB, sorted after shredding; b) picture of a typical run for the measurement of PZC through the drift method.

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