

SUPPLEMENTARY INFORMATION

Spent Lithium Ion Battery Recycling Using Flotation Technology: Effect of Material Heterogeneity on The Separation Performance

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This supplementary information document provides the comprehensive details for the processing of battery materials previous the flotation stage that was not included in the main document and the second section contains the method to calculate the flotation time and collector dosages used in this study.

S1. Sample Preparation Diagram of Feed Materials

S1.1. Sample Preparation Diagram of Feed Materials

The sample preparation procedure used for all materials tested in this study is summarised in Figure S1 and detailed in the following sections of this document. Note that for the spent mixed material, the processing starts on the pyrolysis stage.

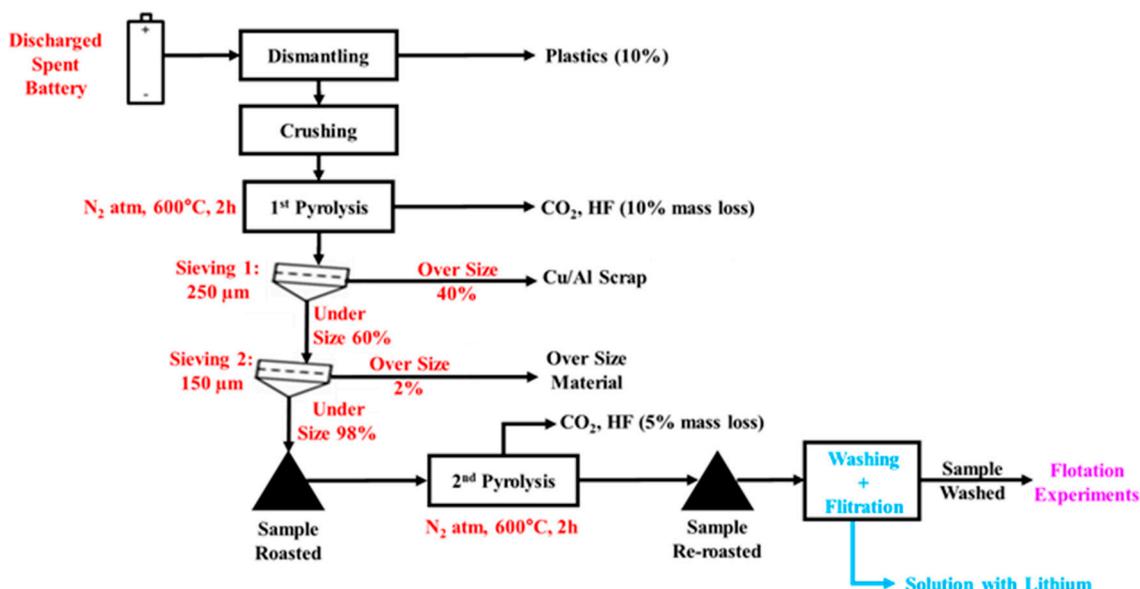


Figure S1. Sample preparation procedure for all materials tested.

S1.2. Dismantling of Battery Pads

Spent single battery pads were dismantled manually and anode and cathode materials were separated from the plastic separator, resulting anode and cathode plates. This is shown in Figure S2.

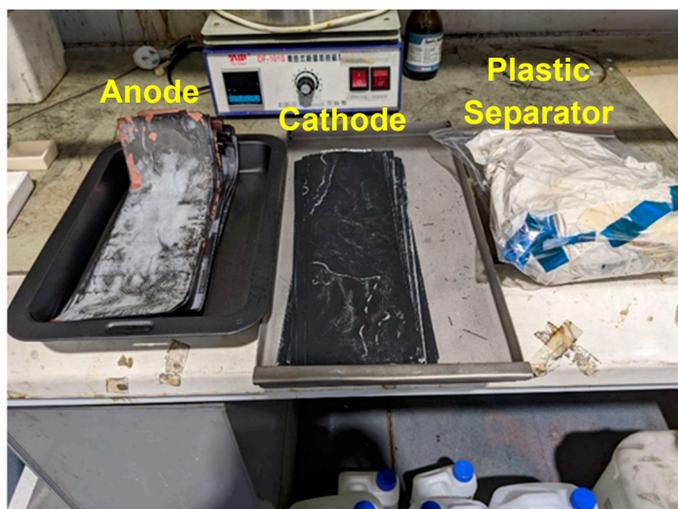


Figure S2. Anode and cathode electrodes separated from battery pads.

S1.3. Crushing of Electrode Samples

Firstly, anode and cathode electrodes were reduced in size using a paper shredder (J.Burrows, model S360A, China), resulting materials with size (7mm (W) x 50 mm (L)) respectively. Then these materials were crushed up to 1 mm particle size using a knife mill blender (Anko, model BL9005-CB, China). Figure S3 shows materials after shredding and crushing stages.



Figure S3. Photographs of materials after shredding (a) and after crushing process (b).

S1.4 Pyrolysis and Sieving of Electrode Materials

All pyrolysis of black mass materials (single battery and mixed battery materials) was conducted in a lab muffle furnace (Modutemp Laboratory Furnace, Australia). For this purpose, 300g of raw crushed sample was used in each experiment and pyrolysis was carried out at 600°C for 2h twice using 100% Nitrogen atmosphere in order to avoid oxidation of the anode material. The equipment used in the experiments is shown in Figure S4.



Figure S4. Photograph of the muffle furnace used for the sample pyrolysis.

After the first pyrolysis is complete, the electrode materials are sieved in order to separate the electrode materials from the copper and aluminium foils. For this purpose, the sieving process was carried out in two stages. The first stage was using a sieve with a mesh size of 250 μm (woven wire mesh sieve, Endecotts Ltd, England), this removes the metallic foils from electrode materials. Next, a sieve with a mesh size of 150 μm (woven wire mesh sieve, Endecotts Ltd, England) is used in order to remove agglomerated material. Finally, all the material below 150 μm (undersize) is submitted to a second pyrolysis prior the flotation stage shown in Figure S1.

S1.5 Sample Washing

Finally, the last stage of this procedure before flotation stage is the sample washing. The purpose of this stage is to remove the soluble lithium that impacts negatively the entrainment of cathode materials in anode concentrates.

S2. Flotation Time and Collector Dosage Calculation Method.

Collector dosage and flotation time for the rougher stage was justified using experimental information taken from literature as reference and with good results [13,18]. However, for scavenger and cleaner stages both processing variables were not available or not mentioned commonly for the processing of these materials. Therefore, collector dosage and flotation time were inferred from other works where scavenger and cleaner stages are used.

In case for collector dosage, the values used were calculated based on the work of Muzenda et al [21]. In this work the authors show how to optimise typical reagents dosages used for rougher, scavenger and cleaner stages of a xanthate collector used in platinum ore processing. The ratio of collector dosage between each stage was used as reference and with these ratios and based on the rougher dosage used in this work, the scavenger and cleaner collector dosages were calculated. Another work used as reference was reported by Wakamatsu and Numata [19], where different processing approaches for graphite processing are reported. This work indicates typical reagent dosages used in rougher, scavenger and cleaner stages. Based on this information, the mass ratios used for the dosage calculation in this work are shown and collector dosages are calculated in Table S1.

Table S1. Mass collector ratios used for dosage calculation.

Stage	Units	Stage/Rougher Ratio [13]	Collector Dosage, g/t
Rougher	[-]	1.00	$500 \times 1 = 500$
Scavenger	[-]	0.50	$500 \times 0.50 = 250$
Cleaner	[-]	0.33	$500 \times 0.33 = 167$

Regarding the flotation time, Wakamatsu and Numata [19] report the processing time used in rougher, scavenger and cleaner stages for graphite processing. Based on this processing time, a time ratio was calculated in order to recalculate the processing time needed for scavenger and cleaner stages and using the rougher stage as reference. Based on this information, the time ratios used are shown and flotation time is calculated in Table S2.

Table S2. Mass collector ratios used for time calculation.

Stage	Units	Stage/Rougher Ratio [18]	Flotation Time, min
Rougher	[-]	1.00	$8 \times 1 = 8$
Scavenger	[-]	0.67	$8 \times 0.67 = 5$
Cleaner	[-]	0.67	$8 \times 0.67 = 5$