Supporting Information: Using Ultrasonic Oil-Water Nano-Emulsions to Purify Lithium-Ion Battery Black Mass

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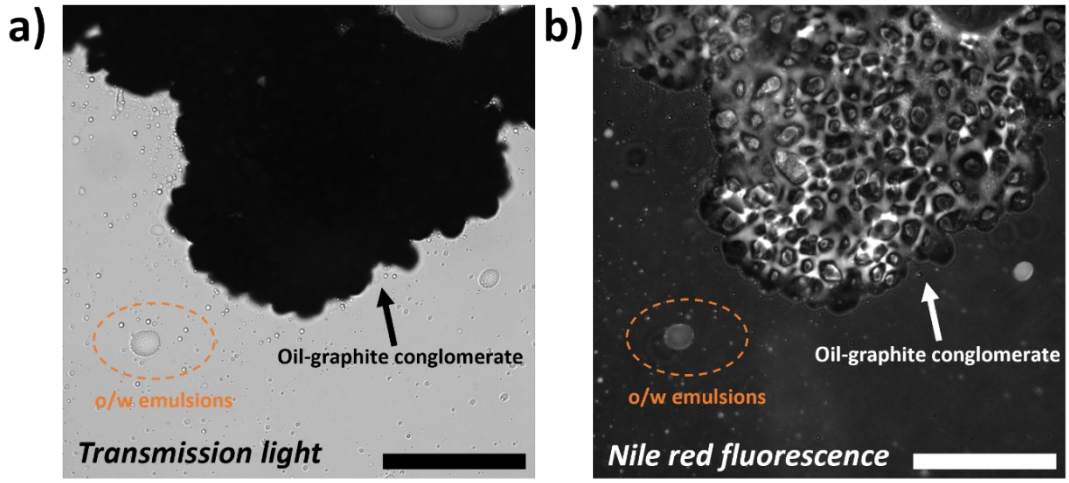

Figure S1. a) Microscopy images showing an oil-graphite conglomerate after one minute of insonation of the pristine NMC and graphite blend with 1% vegetable o/w emulsion. 1mM of Nile red is added to the oil phase prior to insonation in water. a) bright-field image taken using transmission light microscope. b) Fluorescence image of Nile red (λ_{ex} = 490 ± 7 nm and λ_{em} = 560 ± 10 nm). Scale bar is 100 µm.

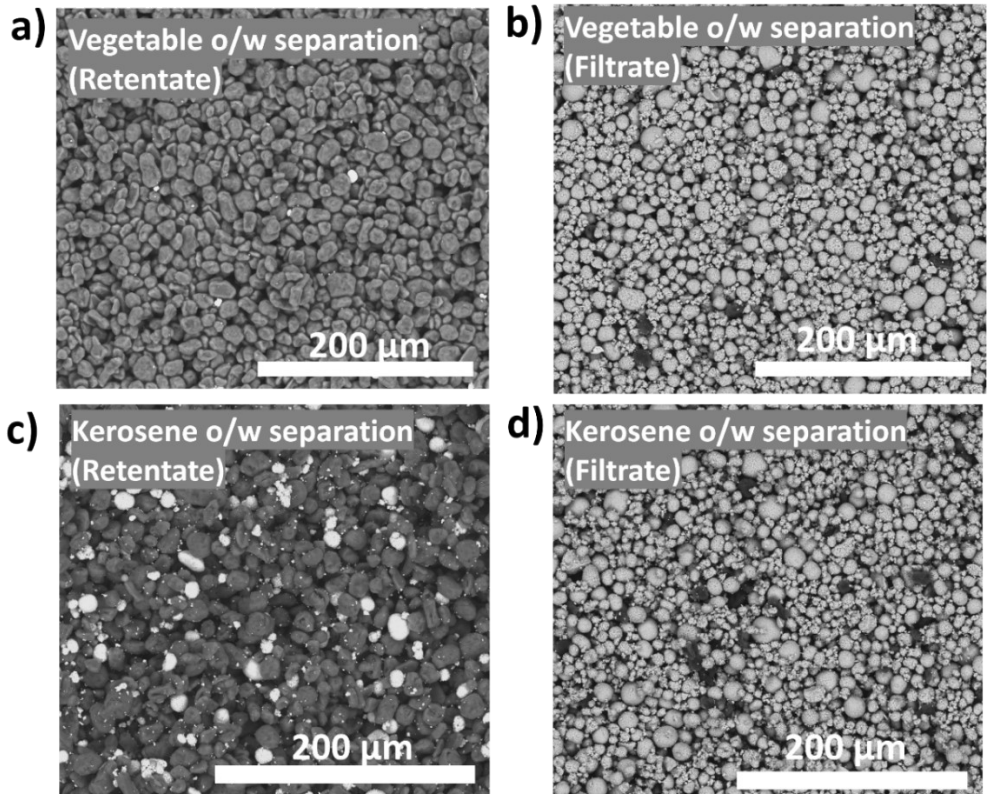

Figure S2. SEM images of retentate and filtrate after the o/w separation process of the binder-free pristine black mass using 1% of vegetable oil (a, b) and kerosene (c, d). The separation process leading to retentate and filtrate is illustrated in Figure 3 and discussed in the main text.

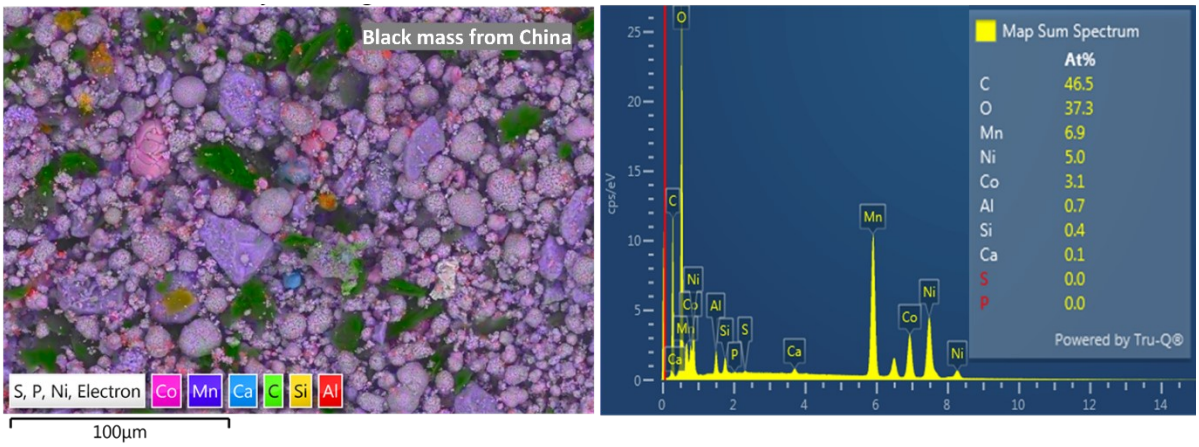

Figure S3. SEM and EDX analysis of commercial black mass.

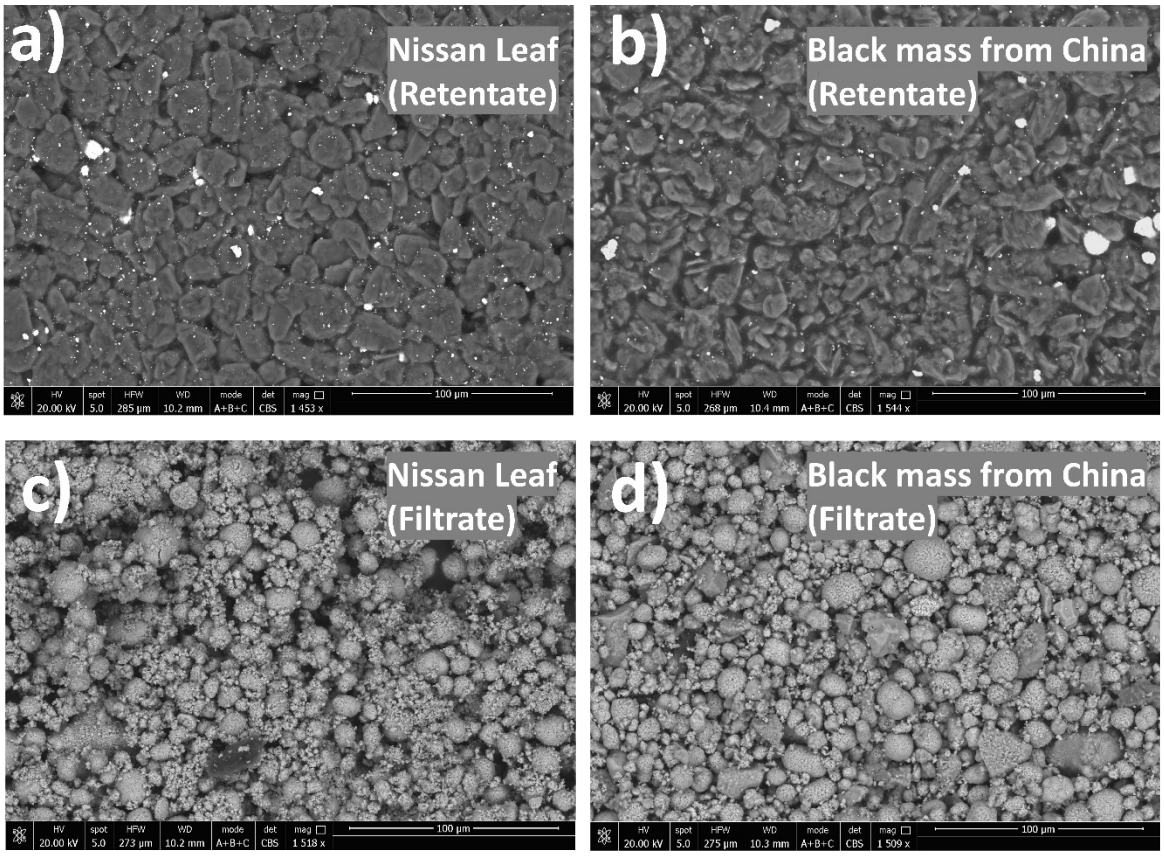

Figure 4. SEM images of oil-in-water purification of two grams of commercial black mass. These are complementry images to the SEM images shown in Figure 4 b) and c) in the main text. The wider viewing window allows the purity of separation to be appreciated. a) and c) are SEM images of the rententate and filtrate obtained from o/w separation of Nissan Leaf black mass, respectively. b) and d) are SEM images of the rententate and filtrate obtained from o/w separation of commercial black mass, respectively. The o/w separation process is shown in Figure 4 a).

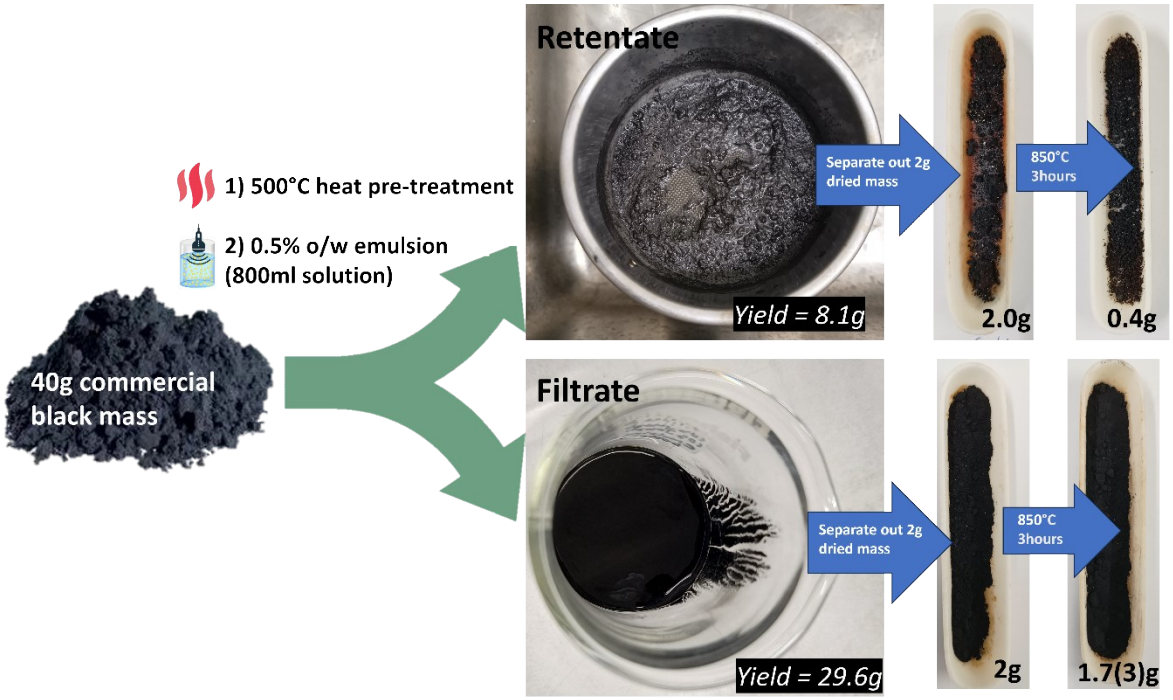

Figure S5. Separation of 40g of commercial black mass using 0.8L of 0.5% oil-in-water emulsions. The mass of the retentate and filtrate obtained after sieving, rising off the oil residue, and drying, were 8.1g and 29.6g, respectively. Furthermore, 2g of the retentate and filtrate were heated separately at 850°C for 3 hours and the resultant mass of materials remaining on the incombustible crucible was 0.4g and 1.7(3)g, respectively. Note that in this scale-up o/w separation (40g of commercial black mass), the size of the ultrasonic horn remained the same as the laboratory scale separation (2g of black mass). Therefore the following parameters were optimised to ensure a high-purity of separation at a larger scale of operation: ultrasonic power increased to 1000w, a higher black mass loading (40 grams in 800 ml of o/w emulsion) and a slightly decreased oil content (0.5 v/v%). The ultrasonic duration of one minute was unaltered from the 2g separation procedures.

Table S1. Energy consumption for laboratory-scale ultrasonic and heating processes. Energy consumption during the individual processes were measured using an energy metre.

Processes	Duration (minutes)	Measured energy consumption (Wh)
Ultrasonic emulsification and agitation of 0.5% oil in 800ml water	2	5.6
Binder removal (500 °C)	60	453
Direct incineration of graphite and binder (850 °C)	60	821