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Article

Recovery of Separator from Battery Waste by Supercritical Carbon Dioxide Extraction: Removal of Electrolyte and Electrode Contaminants

Martin Östergren * , Philipp Mikšovský  and Burçak Ebin * 

Energy and Materials Division, Department of Chemistry and Chemical Engineering, Chalmers University of Technology, Kemivägen 4, SE-412 96 Gothenburg, Sweden; philipp.miksovsky@chalmers.se

* Correspondence: maroste@chalmers.se (M.Ö.); burcak@chalmers.se (B.E.)

Abstract

Hazardous compounds from used batteries pose a great threat to the environment. To prevent pollution and to recover critical materials from battery waste, efficient recycling is required. Until now, battery recycling has focused on the recovery of valuable metals from cathode materials, while organic fractions have often been neglected due to their low material value. New approaches to battery recycling are therefore necessary, where recycling methods based on supercritical carbon dioxide (SC-CO₂) extraction show great potential. In this work, a SC-CO₂ method was implemented to extract electrolyte solvents for the purification and recovery of a separator waste material (SWM) sorted out from lithium-ion battery (LIB)-based black mass. In addition, two other separation routes (ultrasonic washing and thermal treatment) were used for comparison. Based on the results from the three routes, mass balances revealed the gravimetric composition of the SWM, which includes separator, electrolyte, and electrode powder. The composition of electrolyte solvents was determined via Gas Chromatography-Mass Spectroscopy analysis. Furthermore, the polymeric separator was analyzed using Fourier Transform Infrared Spectroscopy, Thermogravimetric Analysis, and Differential Scanning Calorimetry analysis to evaluate the effects of SC-CO₂ extraction on the physicochemical properties. The recovery of electrolyte by the SC-CO₂ route is more efficient than the others, with extraction yields of 162 mg of electrolyte per gram of SWM. Moreover, no changes are observed in the analyzed properties of the polymeric separator material due to the SC-CO₂ extraction. Thus, the SC-CO₂ process proves to be a promising method for an efficient and sustainable recycling of electrolyte solvent and purifying of separator material from LIB waste.

Keywords: battery recycling; supercritical carbon dioxide extraction; electrolyte recycling; polymeric separator recovery



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1. Introduction

1.1. Battery Recycling

The use of battery-powered electric applications is rapidly growing, and the demand for efficient high-performance battery technologies has therefore become increasingly urgent. Owing to their high energy density, cyclability, and charge/discharge rates, lithium-ion batteries (LIBs) are currently the most used type of battery [1]. Therefore, production of LIBs is increasing exponentially, and the market is estimated to reach up to 4500 GWh by 2030 [2]. As a growing number of batteries reach their End-of-Life, the amount of battery waste increases and is estimated to reach 11 million tons by 2030 [3].

LIBs basically consist of an anode, cathode, electrolyte, and separator, along with current collectors and casing materials. Hence, battery waste is composed of a complex material blend, including alkali and transition metals, plastics, organic binders and solvents, as well as halogenated compounds [4]. Until now, battery recycling has mainly been based on pyro- and hydrometallurgical processes, targeting the recovery of valuable electrode materials such as cobalt, nickel, manganese, and lithium. However, organic fractions such as electrolyte and separator have largely been neglected due to low economic incentives [5].

To prevent hazardous and toxic materials from being released into the environment and to ensure the efficient utilization of material resources to produce new batteries, the development of methods for battery recycling is crucial. New approaches for battery recycling that include all parts of the battery can enable more complete material circularity and thereby meet the target set by the European Union of 70% LIB recycling efficiency by 2030 [6].

1.2. Supercritical Carbon Dioxide Extraction and Implementation to LIB Recycling

One novel approach to battery recycling is to utilize supercritical carbon dioxide (SC-CO₂) extraction, which previously has been used in pharmaceutical, cosmetic, textile, and food industries [7,8]. The technique exploits the physicochemical properties of supercritical fluids, which are between liquid and gas. A fluid becomes supercritical at its critical pressure (P_C) and temperature (T_C), which for carbon dioxide are relatively low ($P_C = 73.8$ bar, $T_C = 31.1$ °C) [9]. The solubility of different compounds in supercritical fluids can be controlled by adjusting pressure and temperature to enable selective extraction. Given the relatively low dielectric constant of carbon dioxide ($\epsilon = 1.18$ at equilibrium), non-polar species are more easily dissolved in SC-CO₂ than polar molecules [10,11]. The polarity of the SC-CO₂ system can be increased by adding polar co-solvents and thereby extending the range of extractable compounds [9,12]. Compared to conventional solvents, SC-CO₂ has many benefits, such as lower cost, toxicity, and flammability, along with a high availability of carbon dioxide [8]. With a low environmental impact, infinite reusability, and high selectivity, SC-CO₂ extraction provides an interesting alternative to other battery recycling methods [13].

In 2014, Grützke et al. [14] conducted proof of principle experiments, demonstrating the ability to extract electrolyte components from pristine separator materials and from commercial post-mortem LIBs. Electrolyte recovery rates of up to 73.5 wt% from pristine polyethylene fleece separator and 0.35 g from commercial 18650 battery cells were reported. In parallel, Liu et al. [15] optimized SC-CO₂ extraction parameters with response surface methodology, reaching electrolyte extraction yields of more than 85%. In a second study, Liu et al. [16] developed a method with the aim of closed-loop recycling of LIB electrolyte implementing SC-CO₂ extraction. The extracted electrolyte demonstrated high ionic conductivity (0.19 mS cm⁻¹) and electrochemical stability up to 5.4 V. Thus, the method showed promising results on the reusability of recovered electrolyte components.

Moreover, studies have also focused on the extraction of metals from battery waste by SC-CO₂ processes, exceeding extraction efficiencies of 90% for nickel, cobalt, and manganese and 73% for lithium [17]. Lithium extraction yields of 89% could be obtained by the addition of 4% hydrogen peroxide as a co-solvent [18]. Other research projects have shown the potential to combine electrolyte extraction with the exfoliation of active materials from spent LIBs by SC-CO₂ extraction, with a peeling efficiency close to 99% [19]. Graphite from commercially available LIBs has been recovered by combining subcritical carbon dioxide and SC-CO₂ extraction, demonstrating better electrochemical performance of the recycled graphite than pristine graphite anode material [20].

In recent studies, battery recycling based on SC-CO₂ extraction has proven to be an efficient method in the recovery of organic fractions from shredded and ground battery material, the so-called black mass. Zachmann et al. [21] extracted electrolyte from industrially produced black mass, with yields exceeding 95% for cyclic carbonates and more than 99% for linear carbonates. Multiple aging products from the decomposition of the electrolyte were also detected. In parallel, Akbas et al. [22] demonstrated an efficient extraction of polyvinylidene fluoride (PVDF), with extraction yields of 56% from industrial black mass. However, to the best of our knowledge, recycling of polymeric separator separated from black mass using SC-CO₂ extraction has not been studied.

1.3. Polymeric Separator Recycling

The purpose of the separator is to prevent direct contact between the anode and the cathode in the battery cell, while allowing the electrolyte to access both electrodes and thereby enabling ionic transportation [23]. Besides requirements on chemical stability and mechanical integrity during charge and discharge throughout the battery's life, the separator material must also maintain the porous structure and be chemically compatible with the electrolyte [24].

Different approaches have been used to recycle polymeric separator materials, mainly focusing on materials recovery. In a study by Hou et al. [25], polymeric separator material was used as a reductant in the refining of cathode materials in a pyrometallurgical process. Bhar et al. [26] developed a method to combine carbon derived from polymeric separators with graphite, to enhance the efficiency of recycled graphite anodes. Pang et al. [27] developed a method to transform separator polymers into high-value carbon materials through urea-assisted pyrolysis process. While these strategies utilize the molecular components of the polymeric material, the separator is not recycled to be used in the production of new batteries.

Recently, techniques based on direct recycling of separators have received more attention, where the different layers of the batteries are manually disassembled, followed by restoration of the components before reassembly. Natarajan et al. [28] developed a method to manually separate and clean the polymeric separator from spent LIBs, followed by characterizing the material and finally assembling new batteries using the recovered separator. With a capacity of approximately 123 mAh g⁻¹ at 25 mA g⁻¹, the cell with the recycled separator performed close to the reference and achieved a better cycling life. Although sufficient recovery and cleaning of the separator is possible, direct recycling of separator materials is still in an early phase, and challenges remain with efficiency, upscaling, and commercialization [29]. Thus, alternative approaches to recycling of battery separator materials should be evaluated. This study aims to explore the implementation of SC-CO₂ extraction for the recycling of LIBs, with a specific focus on recovering the polymeric separator from black mass residuals and the absorbed electrolyte solvent.

2. Materials and Methods

2.1. Materials

The material analyzed in this study is a shredded separator fraction from black mass provided by SK TES (Domène, France), originally from used vehicle batteries. The separator waste material (SWM) consists of different polymeric materials (blue, green and gray pieces), pieces of current collectors, a powder fraction consisting of residual electrode material and electrolyte absorbed in the separator. Since the material comes from a treated waste stream, the exact composition of the separator waste is unknown and variations within the material are expected. Based on the multiple colors of the SWM, only parts of the polymeric content

can be assumed to originate from pristine white-colored separator material, whereas the rest may come from casing and other support materials [30].

2.2. Experimental Setup

To evaluate the efficiency of the SC-CO₂ extraction, the amount of electrolyte in the SWM must first be determined. Three routes have been used to complete the mass balance of the SWM: ultrasonic washing (R1), thermal treatment (R2) and SC-CO₂ extraction (R3) (Figure 1a–c).

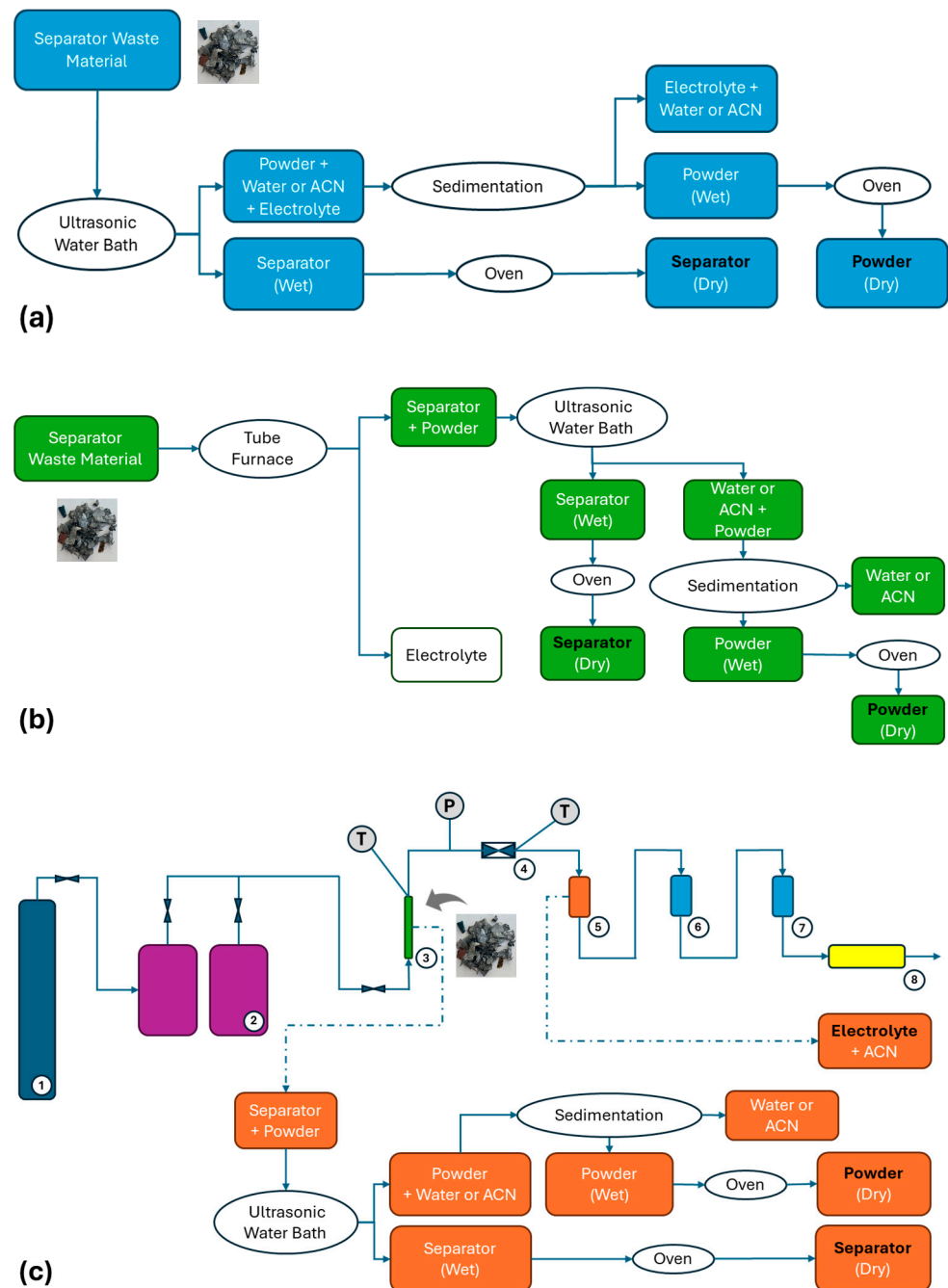


Figure 1. Implemented routes to determine the mass balance, (a) Route 1: ultrasonic washing (R1), (b) Route 2: thermal treatment (R2), and (c) Route 3: SC-CO₂ extraction (R3). R3 (1) CO₂ cylinder, (2) syringe pumps, (3) extraction vessel, (4) pressure regulation valve, (5) electrolyte trap, (6) and (7) gas washing bottles, (8) gas flowmeter. Note that washing of the separator after the tube furnace and the SC-CO₂ extraction follows the ultrasonic treatment of R1.

Route 1 (R1) consists of ultrasonic washing of SWM in water or ACN, followed by sedimentation and drying of the powder and separator fractions. In R1, 0.3 g of SWM was mixed with 50 mL acetonitrile (ACN, LiChrosolv[®] Hypergrade, Merck KGaA, Darmstadt, Germany) or 50 mL Milli-Q water, respectively, and then treated in an ultrasonic water bath (USC-THD/HF, VWR, Radnor, PA, USA) for 20 min at 40 °C. After the ultrasonic treatment, the wet separator and the liquid fraction were separated using a 125 µm sieve (VWR). The wet separator was dried in an oven (FD 115, BINDER GmbH, Tuttlingen, Germany) for 18 h at 60 °C. The liquid fraction was left to sediment for several days, until the powder fraction had separated completely from the liquid. The clear liquid was then removed using a pipette, leaving only a small amount of ACN or water plus the powder at the bottom of the test tube. The remaining liquid was then evaporated in an oven (BINDER FD 115; 60 °C, 18 h for ACN, several days for water), resulting in a dry powder fraction. All fractions were weighed for both ultrasonic treatment methods (ACN and water). The samples were prepared at ambient conditions, and all experiments were performed in triplicate.

Route 2 (R2) is based on thermal treatment of SWM to remove the electrolyte, followed by the ultrasonic treatment according to R1. In R2, 0.3 g of SWM was placed in an open alumina container and then heated in a quartz tube oven (P330, Nabertherm GmbH, Lilienthal, Germany) under constant argon flow (Air Liquide Gas AB, Gothenburg, Sweden, 1000 mL/min) for 1 h at 150 °C. After the thermal treatment, the remaining separator fraction was washed according to R1. All fractions were weighed for both ultrasonic treatment methods (ACN and water). The samples were prepared at ambient conditions, and all experiments were performed in triplicate.

Route 3 (R3) builds on the removal of the electrolyte by SC-CO₂ extraction, after which the separator is cleaned by ultrasonic treatment according to R1. In R3, carbon dioxide (CO₂, purity 99.9%, ≤5 ppm *w/w* H₂O, Air Liquide Gas AB, Gothenburg, Sweden) (Figure 1c, (1)) was pressurized to 140 bar using two syringe pumps (260D, TELEDYNE ISCO, Lincoln, NE, USA) (2). The pressurized gas was then led through a 11.5 mL extraction vessel of stainless steel (3), loaded with 0.3 g of SWM. The extraction vessel was heated by a water heater (F12, JULABO GmbH, Seelbach, Germany) to 40 °C measured by a K-type thermocouple (Pentronic AB, Västervik, Sweden), to ensure a supercritical state of the CO₂. After 10 min of static extraction, the pressure regulation valve after the extraction vessel (4) was opened, ensuring a dynamic extraction at a flow rate of 300 mL CO₂/min (measured by a RED-Y RTU gas flowmeter (Vögtlin Instruments GmbH, Muttenz, Switzerland) (8) at ambient conditions). After the pressure regulation valve (4), the pressure was reduced to ambient conditions. To prevent the expanding gas from forming ice and potentially clogging the pipelines, the temperature was kept at 40 °C using electric heating bands (Winkler AG, Heidelberg, Germany). The gas was then led through the electrolyte trap (5), consisting of a gas washing bottle containing 20 mL ACN, cooled in a water/ice bath. Passing through the electrolyte trap, the electrolyte dissolved in the ACN, while the CO₂ continued through the washing bottle. The CO₂ was then led through two additional gas washing bottles, where the first (6) was empty and the second (7) was filled with 50 mL Milli-Q water, to lower the risk of contamination of the gas flowmeter (8). The extraction was terminated after a total of 5, 10, 20 and 40 L CO₂ respectively (measured at ambient conditions) had passed through the SWM sample (3). After the extraction, the separator fraction was removed from the extraction vessel and subsequently washed according to R1. In all three routes, the ACN containing the electrolyte (after the ultrasonic treatment) was filtered (using a 0.45 µm PTFE syringe filter from Thermo Fisher Scientific, Waltham, MA, USA), to remove any remaining powder residuals. The samples were prepared at ambient conditions, and all experiments were performed in triplicate.

2.3. Analytical Methods

The organic electrolyte compounds were analyzed by Gas Chromatography-Mass Spectroscopy (GC-MS, 7890A GC System/5977A MSD, Agilent Technologies, Santa Clara, CA, USA), equipped with an Agilent Technologies HP 5MS (30 m × 250 μm × 0.25 μm) column. 1 μL sample volumes were injected with a split ratio of 1:20. The temperature was ramped from 40 to 60 °C at a rate of 3 °C/min, followed by a second heating to 270 °C at 20 °C/min. The carrier gas was helium, with a flow rate of 1 mL/min. To determine the decomposition of the lithium salt, the amounts of lithium and phosphorus in the water-based samples were measured by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES, iCAP PRO XP Duo ICP-OES, Thermo Fisher Scientific). Standard series containing 0.625 to 40 ppm lithium and phosphorus were prepared starting from 1000 ppm single elemental standards (Teknolab sorbent, Kungsbacka, Sweden), and 1 ppm yttrium was used as an internal standard. To analyze the composition of the SWM and to evaluate the effects of SC-CO₂ extraction on the separator material, Attenuated Total Reflectance—Fourier Transform Infrared Spectroscopy (ATR-FTIR, Hyperion 3000/Vertex 70V, Bruker Corporation, Billerica, MA, USA) was used, scanning 16 times over a wavenumber range from 400 to 4000 cm⁻¹. Furthermore, Thermogravimetric Analysis (TGA, TGA/DSC 3+, Mettler-Toledo International Inc., Columbus, OH, USA) and Differential Scanning Calorimetry (DSC, DSC 2, Mettler-Toledo International Inc.) analysis were performed. TGA was carried out from 35 °C to 700 °C with a heating rate of 10 °C/min, under a nitrogen flow of 60 mL/min. DSC measurements were conducted in air from 25 °C to 300 °C and back to 25 °C, at 10 °C/min. Additionally, the surface of the separator material was analyzed by Optical Microscopy (AxioCam MRc 5, Carl Zeiss AG, Oberkochen, Germany).

3. Results

3.1. Materials Content

The composition of the SWM (separator, powder, electrolyte) derived from R1, R2, and R3 is shown in Figure 2. In R1, the amount of removed powder is higher using ACN (7.9 wt%) compared to water (3.2 wt%), which indicates a more efficient separation between powder and separator using ACN. This could be explained by the hydrophobicity of the graphite in the powder fraction [31]. Also, partial swelling of the PVDF binder by ACN may reduce the adhesion between powder and separator [32]. As the electrolyte remains in the liquid fraction, it is not possible to gravimetrically determine the exact amount of electrolyte by R1, which was calculated using the total mass and other components' measured amounts.

After heating the SWM in R2, the mass loss derived from the evaporation of volatile electrolyte compounds accounts for 13.9 to 15.9 wt%. As the conductive salt lithium hexafluorophosphate (LiPF₆) starts to decompose at 107 °C forming phosphorus pentafluoride (PF₅) and lithium fluoride (LiF), the gaseous PF₅ is removed by the gas flow while the LiF remains in the separator fraction [33]. The total mass of the electrolyte is therefore higher than the measured values. After washing the separator fraction according to R1, the higher powder fraction using ACN (8.7 wt%) again indicates a more efficient separation compared to washing with water (3.0 wt%). When completing the mass balance, the material loss accounts for approximately 10 wt%, possibly due to transferring the material between different containers during the ultrasonic treatment and the drying steps.

In R3, up to 16 wt% of electrolyte is extracted. Due to the low polarity of SC-CO₂, extraction of the polar LiPF₆ is limited [34]. The total amount of electrolyte is therefore higher than the measured values. The trend of higher separation efficiency after ultrasonic treatment using ACN is not observed in R3. However, the standard deviation for the powder fraction is relatively high after ultrasonic treatment with water, which could

explain this difference in the trend. Besides previously given reasons explaining the mass loss of 12 wt% in R3, some additional material may have been stuck in the filters and pipelines of the SC-CO₂ system.

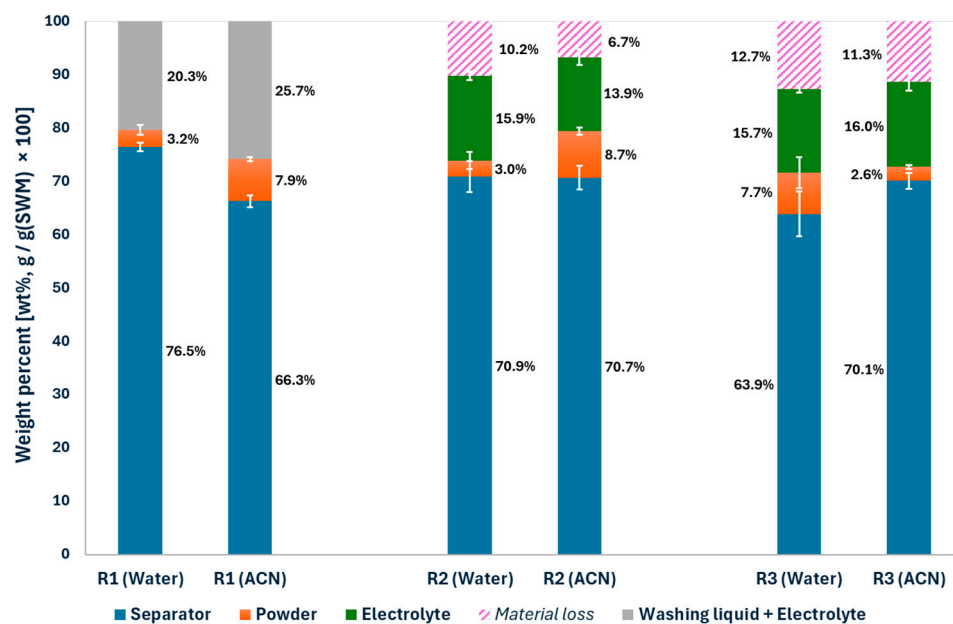


Figure 2. SWM composition, based on the results from R1, R2 and R3. In R1, the electrolyte fraction is inseparable from water and ACN (gray), why the mass balance over R1 is not possible to complete gravimetrically.

Given the heterogeneity of the SWM, variations in weight distribution are expected. Based on the results from R2 and R3 routes (Figure 3), the separator accounts for approximately 68.9 wt%, the powder fraction 5.5 wt% and the electrolyte 15.4 wt% of the SWM. On average, 10.2 wt% of the sample weight is lost during the whole process, due to the low weight of the samples. When comparing the results of R1, R2 and R3, SC-CO₂ extraction of the electrolyte from the SWM proves to be the most efficient separation method, with yields of up to 162 mg of electrolyte per gram of SWM.

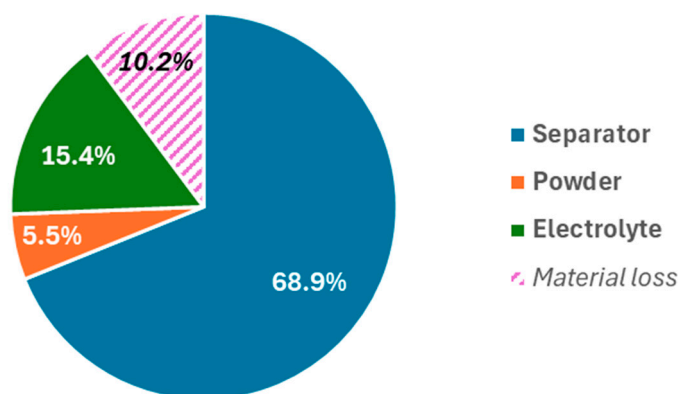


Figure 3. Average SWM composition (wt%), based on R2 and R3.

To evaluate the dependency of the amount of CO₂ used in R3 on the electrolyte extraction, the effects of different volumes were analyzed. As displayed in Figure 4, no major differences in electrolyte extraction yields were observed for 10, 20 and 40 L of CO₂. It was therefore concluded that 10 L of CO₂ is sufficient to ensure complete electrolyte extraction. In the study by Zachmann et al. [21], an extraction yield of 18 mg of electrolyte

per gram of black mass was achieved using 42 g of CO₂ per gram of black mass. In this work, 66 g CO₂ per gram of SWM was used for the electrolyte extraction. The higher amount of CO₂ required for the extraction of electrolyte from SWM is expected, given the larger (wt%) quantity of electrolyte in the SMW compared to the amount of electrolyte in the black mass. This may be due to the low density of the porous polymeric separator [35], which leads to a larger surface area extracting electrolyte. Considering the amount of extracted electrolyte per gram of CO₂, the electrolyte load for SWM (2.5 mg/g) is five times higher than for the black mass (0.5 mg/g) given in the previous study [21].

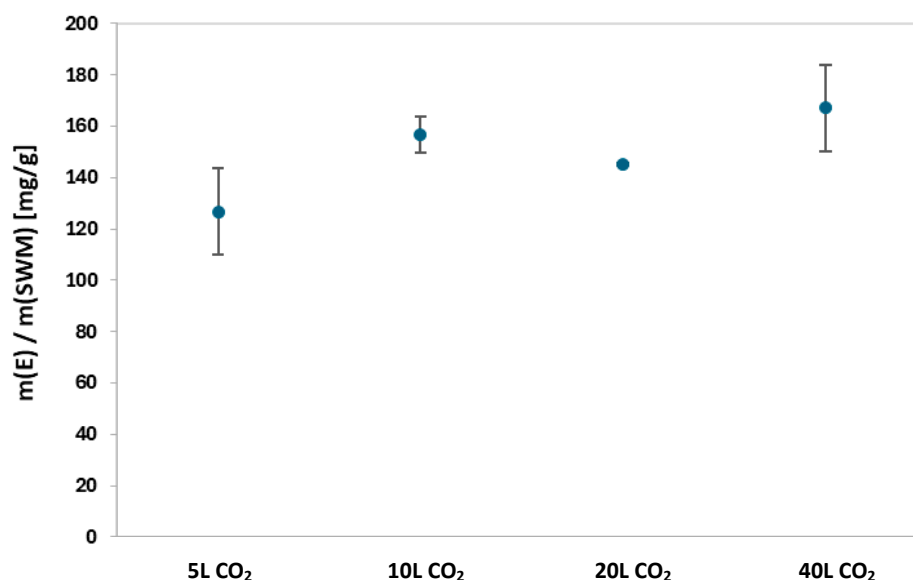


Figure 4. Total amount of extracted electrolyte per gram of SWM by R3, using different volumes of CO₂.

LIBs contain approximately 15 wt% electrolyte and around 5 wt% separator material [36,37]. Even though the SWM has an electrolyte content that is almost three times higher than that for black mass from the same supplier, only 5% of the total electrolyte content can be found in the SWM [21]. Therefore, the motivation to remove the electrolyte from the SWM is to enable further recycling of the separator material and to reduce the risk of exposure of hazardous components, rather than for economic incentives.

3.2. Electrolyte Recovery

Pristine LIB electrolyte typically contains organic solvents such as ethylene carbonate (EC), dimethyl carbonate (DMC), diethyl carbonate (DEC), and ethyl methyl carbonate (EMC) [38]. Additives such as propylene carbonate (PC), biphenyl, and vinylene carbonate are also commonly used [21]. During the usage of the battery, the electrolyte components gradually decompose, e.g., as a result of thermal degradation or due to reactions with water, forming a wide range of aging products, as reported in several studies [39–41]. By the decomposition of cyclic carbonates and reactions with decomposition products of the conductive salt, dicarboxylates, diols, inorganic and organic phosphates, and alkyl fluorides make up many of the identified compounds [42,43]. Moreover, studies suggest a reaction between different carbonates and the formation of carbon–carbon bonds, resulting in larger carbonates [44].

In Figure 5, GC-MS analysis of the extracted electrolyte from R1 is presented. Besides EC and PC, a majority of the detected compounds are identified as linear hydrocarbons, including decane, trimethyldodecane and dodecane. Additionally, 1,3-ditertbutylbenzene is identified. These linear and cyclic hydrocarbons likely originates from additives in

the electrolyte, where 1,3-ditertbutylbenzene is used as an overcharge protection additive and decane can increase electrolyte conductivity [45,46]. Lighter electrolyte solvents and degradation products are absent in the GC-MS results, likely due to evaporation during the processing of the SWM by the supplier.

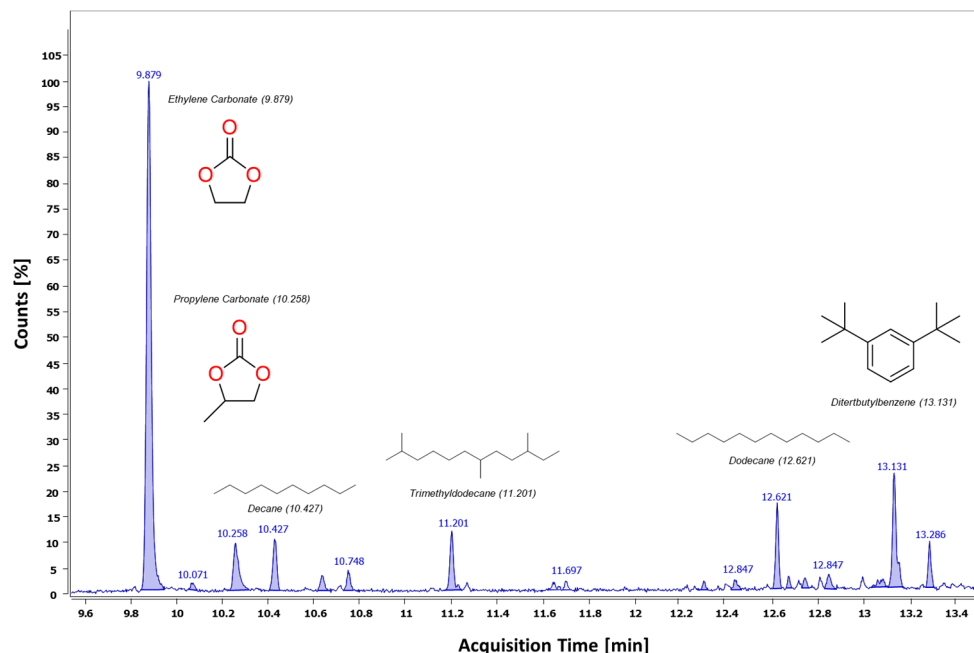


Figure 5. GC-MS results of electrolyte in ACN from R1, displaying multiple additives in the electrolyte together with EC and PC.

When washing the SWM with ACN following R1, 42.2 mg of EC and 2.6 mg of PC are measured by GC-MS, accounting for 26 wt% of the electrolyte in the SWM. Extracting the electrolyte with SC-CO₂ following R3, only a fraction of the EC is detected. However, no EC was observed when washing the separator with ACN after SC-CO₂ extraction and analyzing the solution using GC-MS, proving that all of the EC is removed from the SWM by the SC-CO₂ extraction. The low amount of EC obtained in the electrolyte trap is likely due to partly crystallization of EC in the tubing of the setup after the reduction in pressure and temperature to ambient conditions [21].

ICP-OES analysis of the water fraction after ultrasonic treatment (R1) reveals a lithium content of 3.8 mg per gram of SWM, as shown in Table 1. The results for separator material after thermal treatment and ultrasonic treatment in water present a Li amount of 2.5 mg/g (R2). ICP-OES analysis for separator material after SC-CO₂ extraction followed by ultrasonic treatment in water results in 5.4 mg/g (R3). The lower lithium content for R2 may be explained by the evaporation of electrolyte solvents and the concurrent removal of dissolved LiPF₆ or its decomposition products, which could be carried away by the gas flow during the thermal treatment [47]. The higher amount of lithium from the ultrasonic treatment after the SC-CO₂ extraction (R3) is nearly twice as large as for R1, indicating a better separation of lithium from the separator. This may be due a more efficient removal of binders or minor surface modifications caused by swelling of the polymer during the SC-CO₂ extraction [48]. From the measured amounts of phosphorus, the mole ratio to lithium (Li/P) is calculated. For all three routes, more lithium than phosphorus is detected. The higher amount of lithium likely originates from degradation of cathode material, while the lower amounts of phosphorus could be explained by evaporation of phosphorous compounds (e.g., POF₃) formed by hydrolysis or during thermal degradation of LiPF₆ [49].

Table 1. ICP-OES results for R1, R2 and R3, displaying the lithium (Li) and phosphorus (P) content and the lithium–phosphorus mole ratio (Li/P) in water samples after ultrasonic treatment.

	Li [mg/g SWM]	P [mg/g SWM]	Mole Ratio (Li/P)
R1 (Water)	3.8	10.8	1.6
R2 (Water)	2.5	3.4	3.3
R3 (Water)	5.4	9.1	2.6

The water fraction of R1 contains more LiPF_6 than water samples from R2 and R3. In R2, only parts of the conductive salt are accounted for, as LiF remains as a solid in the separator fraction. In R3, LiPF_6 is not efficiently removed by the SC-CO_2 extraction, due to the low polarity of carbon dioxide [11]. Therefore, the relative amount of extracted electrolyte is higher for R2 and R3 than for R1.

3.3. Effects of SC-CO_2 Extraction on the Properties of the Polymeric Separator

To evaluate the effects of the SC-CO_2 extraction on the separator material, the separator surface was analyzed by optical microscopy before and after R3. As displayed in Figure 6a,b, the SWM displays white crystals which the separator fraction lacks after the SC-CO_2 extraction. This is likely due to the absence of EC after the SC-CO_2 treatment. The images also show the presence of electrode residuals (black powder) both before and after SC-CO_2 extraction, demonstrating the challenges of a complete separation between separator and powder fractions using the ultrasonic treatment of R1.

The FTIR spectrum of the SWM before and separator after SC-CO_2 extraction (Figure 7) displays a clear trend, where most of the bands between 500 cm^{-1} and 1900 cm^{-1} in the results for SWM are absent in the spectra for the separator. In the spectra for SWM, the bands at 1790 cm^{-1} and 1770 cm^{-1} correspond to the stretching of the C=O bond in EC. The bands at 1470 cm^{-1} and 1390 cm^{-1} are related to bending and wagging vibrations of C-H bonds in CH_2 groups in EC [50]. After the SC-CO_2 extraction, these bands are absent, proving that EC has been removed from the SWM. Bands characteristic for polyethylene (PE) at 2916 cm^{-1} and 2849 cm^{-1} corresponding to stretching of C-H bonds in CH_2 and CH_3 groups and bands at 1473 cm^{-1} and 717 cm^{-1} attributed to rocking vibrations of C-H bonds remain unchanged in the spectra [51]. This proves that besides the removal of EC from the SWM, the polymeric material is not affected by the SC-CO_2 extraction.

The TGA graphs (Figure 8) also confirm that the separator is not affected by the SC-CO_2 extraction, as decomposition of the polymeric material occurs at the same temperature (between 420 and $500\text{ }^\circ\text{C}$) before and after the SC-CO_2 extraction. For the separator after the SC-CO_2 extraction, the only difference is the absence of the mass loss starting at $70\text{ }^\circ\text{C}$, related to the vaporization of volatile electrolyte compounds. In accordance with the results from R2 and R3 (Figure 3), the weight change from 70 to $180\text{ }^\circ\text{C}$ accounts for approximately $15.7\text{ wt}\%$.

In correlation with the FTIR and TGA results, the DSC data of melting temperatures (Figure 9a) and crystallization temperatures (Figure 9b) for the SWM before and separator after the SC-CO_2 extraction are well fitted to each other. This implies that the recycled separator has the same crystal structure and material properties as the SWM, which indicates that the SC-CO_2 extraction does not affect the significant polymer properties. The difference in peak area of the SWM and the separator after SC-CO_2 extraction likely relates to the variations within the material, since samples originate from different batteries and thus exhibit different levels of crystallinity, resulting in different peak areas.

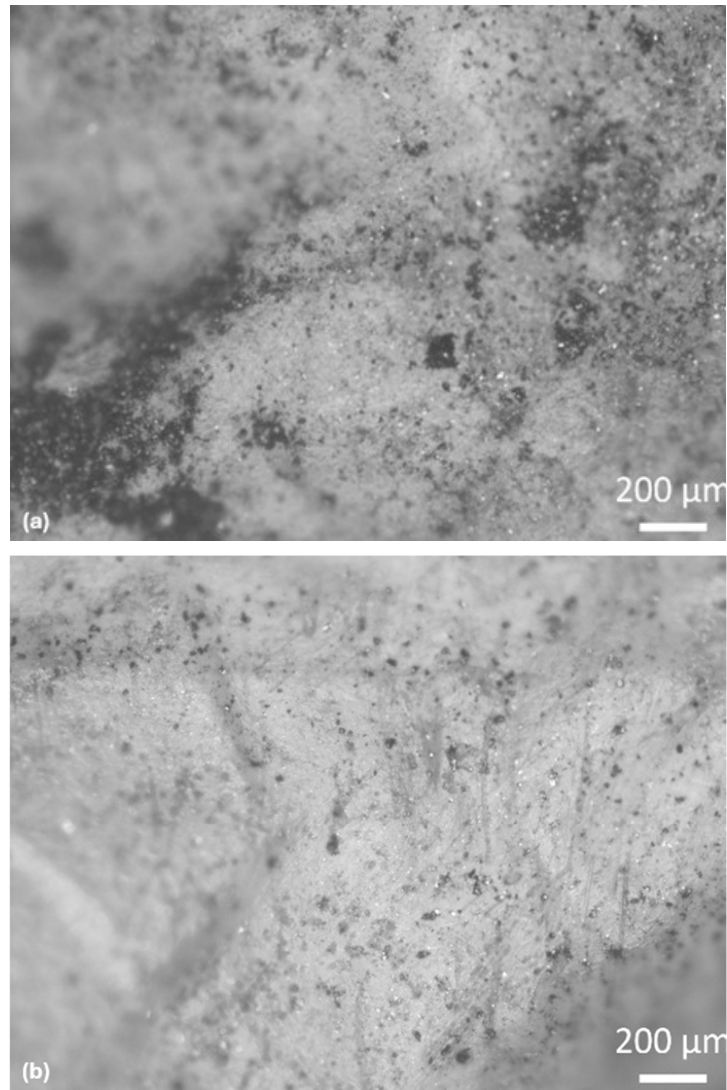


Figure 6. Optical microscopy images of (a) SWM before and (b) separator after SC-CO₂ extraction.

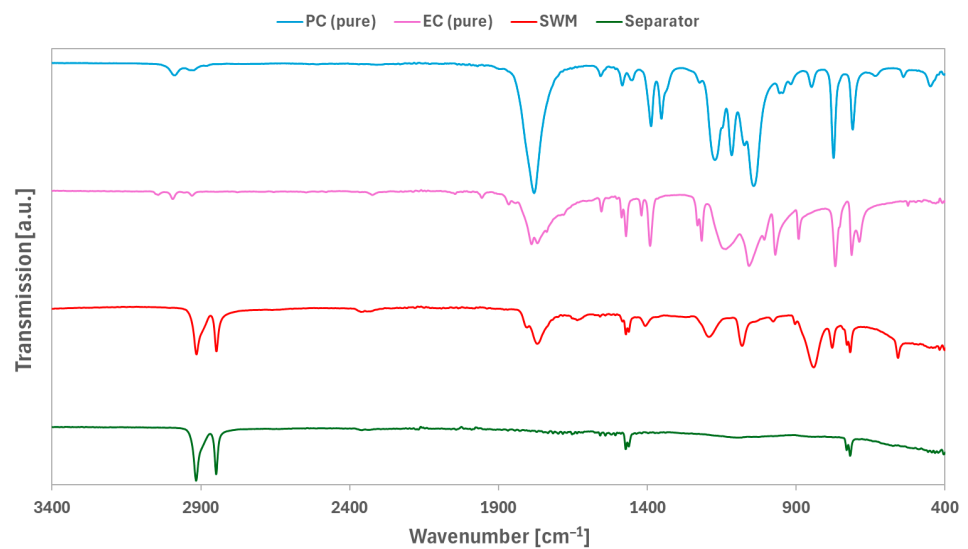


Figure 7. FTIR spectra for PC (pure), EC (pure), SWM before and separator after SC-CO₂ extraction.

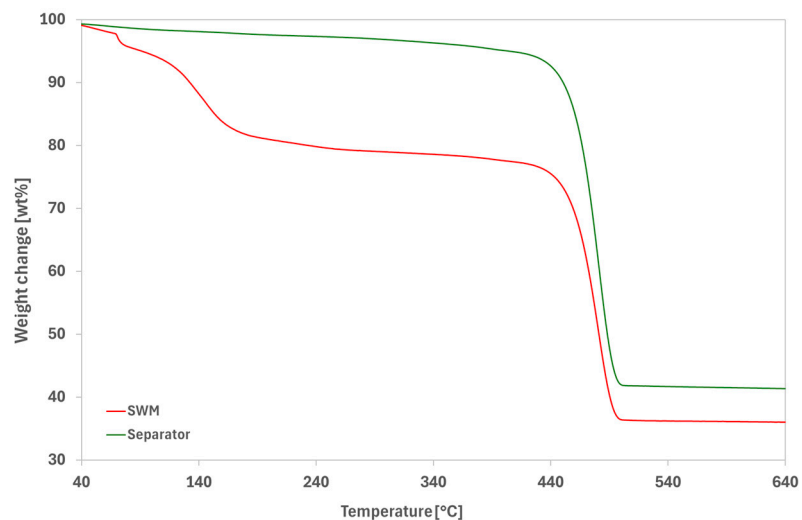


Figure 8. TGA results for SWM before and separator after SC-CO₂ extraction.

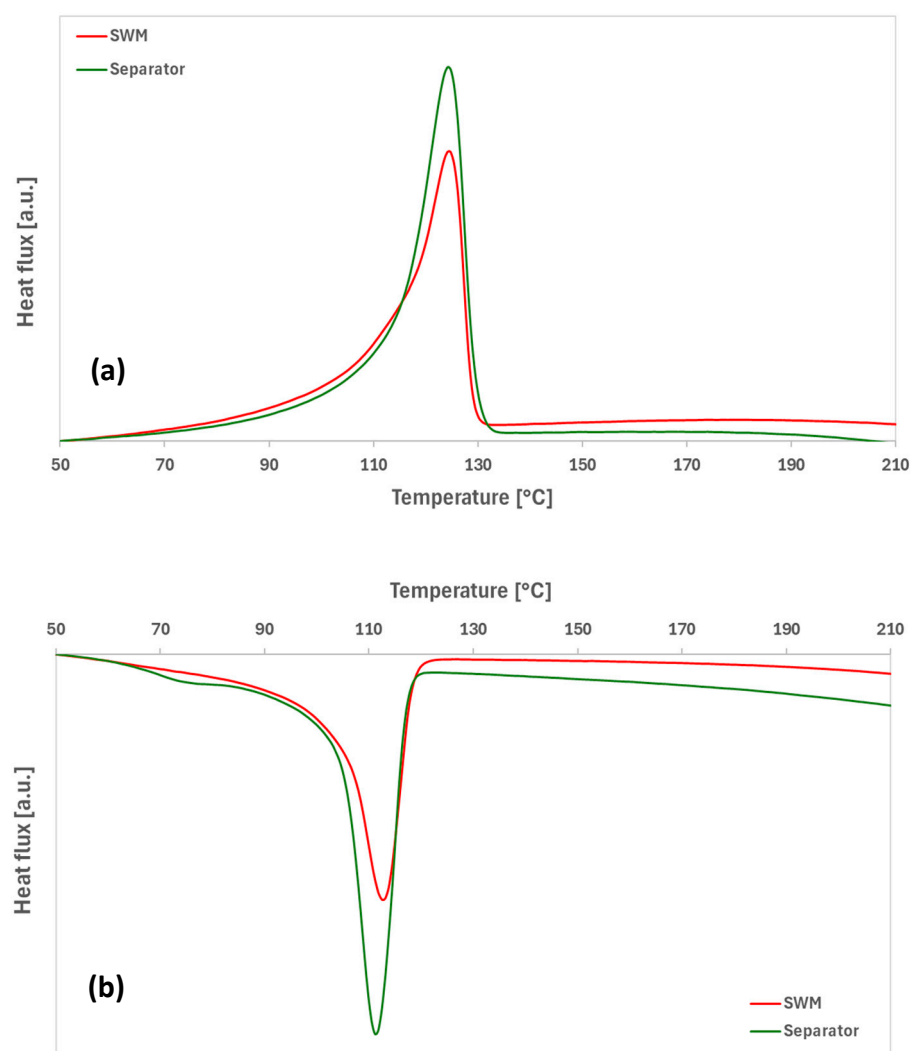


Figure 9. (a) Melting temperatures and (b) crystallization temperatures for SWM before and separator after SC-CO₂ extraction.

Based on the FTIR, TGA and DSC results, the polymeric separator material is not affected by the SC-CO₂ extraction. This conclusion correlates well with data in the literature

from tests performed on pristine crystalline polymers in SC-CO₂ [52]. The difference in the FTIR spectra for SWM before and separator after SC-CO₂ extraction are directly associated with the removal of EC, according to the correlating bands of pure EC. The characteristic bands of PE are unchanged, proving that the separator material is not affected by the SC-CO₂ extraction. The TGA results support this statement, where the only difference between the two curves is the weight change associated with the removal of electrolyte. The DSC results confirm the outcome of the TGA, as melting and crystallization temperatures remain the same before and after SC-CO₂ extraction.

The solubility of electrolyte solvents in SC-CO₂ has been accounted for in several studies, which demonstrate the solubility dependency of pressure, temperature and intermolecular interactions between the solutes and SC-CO₂ [14,16,34]. Where smaller molecules with low polarity are easily dissolved, non-linear and more polar compounds have a lower affinity for SC-CO₂ [21]. To dissolve polymeric materials in SC-CO₂, the cohesive forces between the polymer segments must be broken, why factors such as temperature, pressure and polarity become even more crucial. While semi-crystalline, non-polar PE is insoluble in SC-CO₂, more polar polymers can be dissolved at elevated temperatures and pressures if a polar co-solvent such as acetone or ethanol is added [53]. For polymers possessing electron-donating functional groups, Lewis acid–base interactions with the carbon atom of CO₂ are possible, forming complexes with bonds slightly stronger than the intermolecular forces between the polymer chains [54]. Where polar interactions are more dominant at lower temperatures, the free volume of a polymer plays a more significant role for the solubility at higher temperatures. Higher free volume implies higher solubility, why stiffer polymer structures are more difficult to dissolve in SC-CO₂ [54]. Though the composition of the SWM is initially unknown, the FTIR, TGA and DSC results indicate that the polymeric separator consists of PE. With a PE-based separator material, only electrolyte components can be extracted by SC-CO₂ considering the solubility of PE under the experimental conditions, thereby enabling a selective separation between electrolyte and separator. Thus, SC-CO₂ extraction is a feasible method to extract electrolyte from battery separator waste.

4. Future Perspective

Considering the current abundance of low-cost, industrially produced polymeric materials, the immediate demand for recycled polymeric separators may not be very high. In the future, however, the need for recycling of high-grade polymers may grow, as availability and costs of polymeric separator materials might change when moving away from petroleum-based raw materials [55,56]. Besides possibilities to recycle and reuse the polymers, an immediate benefit of an electrolyte-free separator fraction is the elimination of risks and high costs associated with handling and transportation of the material, as this no longer would be treated as hazardous waste.

5. Conclusions

In this study, electrolyte from a separator waste material recovered from LIB black mass was extracted by SC-CO₂. In addition to SC-CO₂ extraction, two other separation routes (based on ultrasonic washing and thermal treatment) were employed to evaluate the extraction efficiency of the SC-CO₂ method. Featuring extraction yields of up to 162 mg of electrolyte per gram of SWM, SC-CO₂ extraction performed better than the other evaluated methods. Furthermore, GS-MS results revealed that 26% of the extracted electrolyte consisted of EC, while several of the detected compounds were associated with electrolyte additives. Highly volatile electrolyte compounds were not detected in the extracted electrolyte, likely due to evaporation during the pre-treatment of the SWM. FTIR analysis of the SWM showed that bands characteristic of PE remained the same before and

after the SC-CO₂ extraction, proving that the polymer was not affected by the extraction process. TGA and DSC results confirmed the outcome of the FTIR, as decomposition temperature, melting point and crystallization temperature all remained unchanged before and after the electrolyte separation by the suggested method. Thereby, the study proves that SC-CO₂ extraction offers an efficient, non-destructive and environmentally friendly alternative to conventional battery recycling techniques, enabling complete and sustainable recycling of batteries.

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Abbreviations

The following abbreviations are used in this manuscript:

ACN	Acetonitrile
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy
DEC	Diethyl carbonate
DMC	Dimethyl carbonate
DSC	Differential Scanning Calorimetry
EC	Ethylene carbonate
EMC	Ethyl methyl carbonate
GC-MS	Gas Chromatography-Mass Spectroscopy
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectroscopy
LIB	Lithium-ion battery
LiF	Lithium fluoride
LiPF ₆	Lithium hexafluorophosphate
PC	Propylene carbonate
P _C	Critical pressure
PE	Polyethylene
PF ₅	Phosphorus pentafluoride
PVDF	Polyvinylidene fluoride
SC-CO ₂	Supercritical carbon dioxide
SWM	Separator waste material
T _C	Critical temperature
TGA	Thermogravimetric Analysis

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