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Efficient lithium recovery from industrial lithium iron phosphate (LFP) blackmass using formic acid-enabled hydrometallurgy

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A R T I C L E I N F O

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A B S T R A C T

As millions of LFP batteries reach their end of life, the challenge of selectively extracting lithium from industrial blackmass, loaded with impurities, has become a focal point for sustainable innovation in battery recycling. However, industrial-scale LFP recycling remains technically challenging due to the complex composition of real battery waste, including high graphite content, binders, and metallic inclusions that undermine conventional leaching processes. In this research study, a selective, green hydrometallurgical approach tailored for impurity-rich LFP blackmass is presented. Using only trace ("hint of acid") amounts of formic acid with hydrogen peroxide and two-step leaching, achieving ~94.5% lithium extraction in the two-step formic acid route, whereas the trace sulfuric acid + H_2O_2 condition delivers the highest selectivity, reaching >95% lithium recovery with negligible iron co-leaching, and producing lithium carbonate as an end product (confirmed by XRD and SEM) without the environmental burden of aggressive reagents or high-temperature treatment. Key to this process is careful control of solution pH and oxidation conditions, allowing a scalable, cost-effective route to close the loop on LFP materials, and proving that the high-yield lithium recovery and environmental responsibility can be achieved in the same process.

1. Introduction

The global transition to renewable energy and electric mobility has positioned LFP batteries as a cornerstone of sustainable energy storage. Their safety, longevity, and cost-effectiveness, free from cobalt and nickel, have driven market growth, with projections reaching USD 72.76 billion by 2030 (Arshad et al., 2020, Bhar et al., 2023, Bruno and Fiore, 2025). However, this boom has exposed a critical gap: the absence of scalable, environmentally sustainable recycling solutions for end-of-life LFP batteries. Existing recycling practices, dominated by pyrometallurgy and conventional hydrometallurgy, are either energy-intensive or generate hazardous waste, failing to meet both economic and environmental benchmarks. Furthermore, most research focuses on pristine LFP, neglecting the real-world complexity of industrial blackmass, which contains graphite, aluminum, copper, and fluorinated binders, complicating selective lithium extraction (Chen et al., 2024, Forte et al., 2020, Li et al., 2024).

Traditional recycling methods have struggled to meet these demands. Pyrometallurgical processes, while industrially established, require extreme temperatures (often above 1,400°C), leading to high energy consumption, substantial carbon emissions, and poor lithium recovery, rendering them environmentally and economically unsustainable for LFP chemistry. Conventional hydrometallurgical routes, though capable of high lithium recovery, typically rely on concentrated mineral acids such as sulfuric acid (Wang et al., 2022, Almahri and An, 2025, Bi et al., 2021).

Several recent studies offer important insights into selective lithium extraction from spent $LiFePO_4$ (LFP) batteries using a range of lixiviants, process conditions, and feedstock chemistries. Wang et al. (2025) (Wang et al., 2022) explored an air roasting step followed by acid leaching, using 0.5 M sulfuric acid for blackmass with approximately 4.47 % Li content; their process realized a lithium leaching efficiency of 97.48 %,

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though higher Fe dissolution (>15 %) was observed unless H_2O_2 was used as an oxidant to enhance selectivity. Müller et al. (2024) (Müller et al., 2024) assessed thermal pretreatment and flotation for industrial blackmass containing 1.91 % Li, indicating that subsequent leaching using 0.3–0.9 M sulfuric acid and H_2O_2 achieved roughly 95–97 % lithium extraction with Fe leaching below 0.5 %, thereby supporting the efficacy of low-molarity acid and oxidation for selectivity. Jin et al. (2022) (Jin et al., 2022) introduced a purely green approach by combining air oxidation with water leaching on a precursor with 4.47 % Li, obtaining 99.3 % lithium recovery and keeping Fe and P co-leaching below 0.02 % at pH 3.5, a highly selective system demonstrating near-complete lithium extraction in water.

Liu et al. (2025) (Liu et al., 2025) reported a rapid, highly selective process using 0.9 M H_2SO_4 and H_2O_2 for blackmass with 4.43 % Li, achieving 98.7 % lithium recovery within 10 minutes while Fe co-leaching was limited to below 0.3 %; the process relied on a nearly stoichiometric acid-to-lithium ratio, underscoring cost-effectiveness and environmental benefits. Li et al. (2017) (Li et al., 2017) used an optimized stoichiometric sulfuric acid leaching system (0.3 M H_2SO_4 with H_2O_2) for a spent cathode with 3.85 % Li, extracting 96.85 % lithium while restricting Fe leaching to just 0.027 %, another demonstration of high selectivity at low acid concentration. Bruno et al. (2024) (Bruno et al., 2024) examined 0.25–0.5 M H_2SO_4 for LFP production scrap containing 2.1 % Li, attaining up to 98 % lithium leaching and 98 % of Fe and P retained in solid residues; Fe co-leaching was kept low (<5 %) by addition of H_2O_2 as oxidant, and citric acid was far less effective for lithium recovery.

Recent advances in the field have begun to address these needs. Greener hydrometallurgical strategies, such as the use of organic acids (e.g., formic acid), hydrogen peroxide, and even deep eutectic solvents, are emerging as promising alternatives to harsh mineral acids. These approaches aim to maximize lithium recovery while suppressing the dissolution of iron and phosphate, thereby reducing waste generation and simplifying purification. Zhao et al. (2023) (Zhao et al., 2023) developed a direct selective leaching process utilizing 0.5 M formic acid (HCOOH) with 2.5 % H_2O_2 as oxidant, targeting industrial blackmass containing 1.65 % Li. Under these conditions, lithium extraction surpassed 97 %, while co-leaching of iron and other metals remained below 1 %. In a related study, Zhao et al. (2024) (Zhao et al., 2024) explored oxygen as a greener alternative oxidant, employing 2.5 M formic acid on blackmass with 4.18 % Li. Their process achieved over 99.9 % lithium leaching, with iron extraction limited to 1.7 %, and the selectivity remained high even when air replaced pure oxygen (Li leaching 97.8 %, Fe 4.8 %).

Mahandra and Ghahreman (Mahandra and Ghahreman, 2021) proposed a sustainable two-step recovery from cathode powders (4.35 % Li), using 1.0 M formic acid and H_2O_2 as oxidant. Their method resulted in lithium precipitation yields up to 99.98 %, with iron dissolution kept below 0.5 %, confirming the high selectivity of the formic acid route. Complementing these findings, Segura-Bailón et al. (Segura-Bailón et al.) compared tartaric and formic acids (using 0.5 M HCOOH with 2.5 % H_2O_2) on blackmass containing 1.81 % Li, achieving complete lithium leaching (100 %), while iron and phosphorus co-leaching were suppressed by over 90 %.

These studies underline that formic acid, applied at concentrations from 0.5 to 2.5 M and tailored to blackmass lithium content, enables highly efficient and selective lithium extraction towards LFP waste.

Despite the significant advancements highlighted in current literature, a critical knowledge gap remains between laboratory-scale innovation and industrial reality for sustainable LFP recycling. Most reported processes (Biswas et al., 2023, Dai et al., 2020, Gong et al., 2022) demonstrate impressive selectivity and efficiency using formic acid or other organic lixiviants on relatively pure/laboratory-simulated blackmass or only-cathode based blackmass, often under tightly controlled conditions with minimal impurity interference. However, actual industrial LFP blackmass is far more heterogeneous, packed with considerable amounts of graphite, metals like aluminum and copper, as well as binders and other contaminants introduced during large-scale mechanical shredding. Because of this complexity, many established methods struggle with selectivity, suffer from unexpected co-dissolution of non-target metals, and present challenges for downstream purification on a practical scale. Furthermore, regulatory shifts demanding higher lithium recovery rates and reduced secondary pollution stress over the need for scalable, environmentally benign solutions that are pragmatic in the face of real-world impurity profiles. (Costa et al., 2021, Rehman et al., 2025, Windisch-Kern et al., 2022)

Although trace sulfuric acid combined with hydrogen peroxide demonstrates the highest selectivity in our comparative tests, its use produces sulfate-rich effluents that require additional neutralization and generate larger secondary waste streams. In contrast, formic acid offers important environmental and operational advantages. It decomposes into carbon dioxide and water, produces no persistent sulfate residues, and can be used in significantly smaller quantities in the two-step leaching route developed in this study. These characteristics reduce effluent load, simplify downstream treatment, and align better with emerging sustainability requirements for hydrometallurgical recycling processes. For these reasons, formic acid was selected as the primary focus of this work, despite the superior selectivity of the trace sulfuric acid condition.

Therefore, this research aims to determine whether a hydrometallurgical process using minimal ("hint of acid") amounts of formic acid and hydrogen peroxide can achieve high lithium extraction efficiency and selectivity from impurity-rich industrial LFP blackmass. It also investigates how critical operational parameters, particularly the solid-to-liquid ratio and temperature, affect the efficiency, selectivity, and impurity levels during the leaching of real-world LFP blackmass. Additionally, it examines the effectiveness of sequential impurity removal and controlled precipitation techniques in producing lithium carbonate directly from complex industrial leachates. Finally, it assesses how impurities of industrial LFP blackmass such as graphite, aluminum, copper, and binder residues influence the kinetics and selectivity of lithium leaching, as well as downstream precipitation.

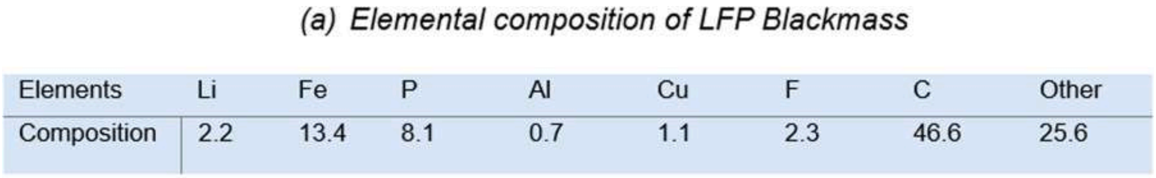
2. Materials and Methods

2.1. Materials

The LFP blackmass used in this study was sourced from industrial battery recycling streams: a mixture of cathode material $LiFePO_4$, anode material graphite (30–45 %), and impurities such as Al, Cu, and possibly PVDF binder residues. The sample was sieved to <90 µm for uniform leaching performance, and its chemical composition can be observed in Fig. 1(a). The majority presence of graphite and $LiFePO_4$ phases is shown in an X-ray diffraction in Fig. 1(b).

This modest lithium content emphasizes the importance of achieving high extraction efficiencies, as every percentage point of recovery directly impacts the economic viability of the recycling process. The iron content of 13.4 % and phosphorus at 8.1 % align well with the expected Fe:P ratio in $LiFePO_4$, confirming that the olivine cathode material is the primary source of these elements ("A Comparative Analysis of Lithium-Ion Batteries Using a Proposed Electrothermal Model Based on Numerical Simulation", "Navigating battery choices: A comparative study of lithium iron phosphate and nickel manganese cobalt battery technologies - ScienceDirect"). The fact that iron significantly outweighs lithium by mass (6:1 ratio) highlights why achieving selective leaching that minimizes iron dissolution while maximizing lithium recovery is so critical for downstream processing efficiency. Based on the Li-content, it is possible to estimate theoretically the oxygen content of the blackmass. Following the assumption that all Li in the sample is in the form of $LiFePO_4$, then the O-content is 20.1 %, which leaves 5.51 % of unidentified compounds.

The XRD pattern obtained using a Bruker D8 Advance diffractometer



(a) Elemental composition of LFP Blackmass

Elements	Li	Fe	P	Al	Cu	F	C	Other
Composition	2.2	13.4	8.1	0.7	1.1	2.3	46.6	25.6

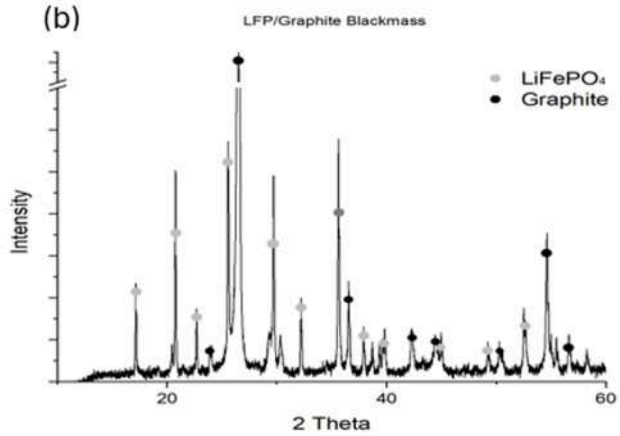


Fig. 1. (a) Elemental Composition of Industrially shredded Blackmass (b) XRD Pattern of LFP Blackmass, comprising of LFP and graphite.

with Cu-Kα radiation (λ = 1.54 Å) over a 2θ range of 10–80° at a 0.02° step size (Fig. 1(b)) clearly shows the heterogeneous nature of the LFP/graphite blackmass, confirming the presence of both target and non-target phases. The most prominent peak at ~26.5° (2θ) corresponds to the (002) reflection of graphite, indicating that graphite forms a major fraction of the blackmass, consistent with ICP analysis of shredded battery material, where anodes and cathodes are mixed. The $LiFePO_4$ phase is identified through its characteristic peaks at ~20.8°, 25.6°, 29.7°, and 35.6° (2θ), corresponding to the olivine (011), (111), (020), and (002) reflections, respectively ("Characterization of Industrial Black Mass from End-of-Life LiFePO4-Graphite Batteries", Assi and Amer, 2025). The sharp, well-defined nature of these peaks confirms that $LiFePO_4$ maintains good crystallinity despite mechanical shredding, which is favorable for leaching processes given the more predictable behaviour of crystalline phases.

The lixiviants included deionized water (resistivity >18 MΩ·cm), sulfuric acid (H_2SO_4, 95–98 %, Sigma-Aldrich), and formic acid (HCOOH, ≥88 %, Merck). Hydrogen peroxide (H_2O_2, 30 % w/w, VWR Chemicals) served as the oxidizing agent. All reagents were of analytical grade to minimize contamination risks.

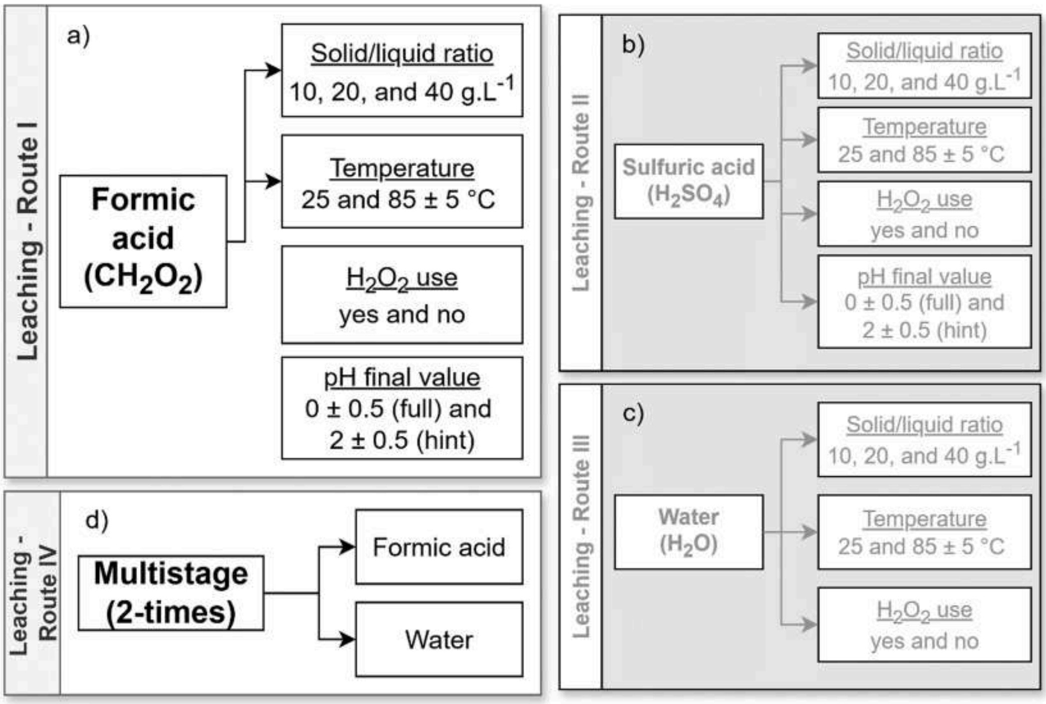


Fig. 2. Experimental plan a) Leaching conditions and parameters for formic acid; b) Sulfuric Acid; c) Water; d) Multi-stage leaching using Formic acid and Water.

2.2. Experimental Setup and Method

2.2.1. Leaching Experiments

The assessment of leaching step considered four parameters: solid/liquid ratio, temperature, presence or absence of H_2O_2 as oxidizing agent, and final pH value (differentiating between "hint" or dilute and fully concentrated solutions) as shown in Fig. 2. Other parameters were kept constant throughout the trials: the agitation at 350 rpm, duration of 90 min, the reactor, and the acid concentration of 2 $mol.L^{-1}$. The setup of the trial was a 250 mL glass reactor equipped with an overhead stirrer and temperature controllers. For each trial, 2.5–10 grams of LFP blackmass were added to 250 mL of solution. After each trial, the leachate was filtered, and the lithium concentration was analyzed using inductively coupled plasma optical emission spectroscopy (ICP-OES) with a PerkinElmer Avio 500 spectrometer. All the experiments were repeated at least three times for reproducibility.

The first set of experiments aimed to determine the baseline leaching ability of water, both in its pure form and when improved with an oxidant, for extracting lithium from industrial LFP blackmass, as shown in Fig. 2(c). A multi-step water leaching process and formic acid (Fig. 2 (d)) was also tested to see if sequential treatments could gradually release more lithium from the solid residue remaining after the initial leach stage.

Considering sulfuric acid's strong acidity and widespread industrial use, its performance was thoroughly evaluated as a benchmark in this research (shown in Fig. 2(b)), both as a standalone lixiviant and in combination with an oxidant. Additionally, a novel approach called the "hint" of sulfuric acid was tested, where just 5–6 mL of concentrated acid (2 M) was added to 250 mL of deionized water to gently lower the pH to nearly 2. This minimal acidity aimed to subtly destabilize the LFP structure while avoiding the harshness and waste associated with traditional high-acid systems. The concept of a 'Hint of Acid' directly supports the paper's main goal: to deliver a selective, greener, and low-waste hydrometallurgical process suited to real, impurity-laden LFP blackmass from industrial waste streams.

Building on an understanding of LFP blackmass behavior in neutral (pH 7), highly acidic (pH 0), and mildly acidic (pH 2) environments, formic acid has been chosen as a greener alternative to conventional mineral acids. As a strong organic acid (stronger than commonly used organic acids such as acetic acid ($pKa \approx 4.76$) and oxalic acid ($pKa_1 \approx 4.22$), and natural chelating agent, formic acid offers biodegradability and reduces environmental impact without compromising leaching potential. Tests followed the same parameter framework as previous experiments, except for an elevated temperature of 88°C, selected based on pre-trial data showing improved lithium extraction as shown in Fig. 3 (a). Four approaches were examined using formic acid: (i) baseline leaching with 2 M formic acid to determine inherent efficiency; (ii) 2 M formic acid combined with H_2O_2 to leverage synergistic oxidizing effects for better selectivity and yield; (iii) a small amount of formic acid (5–6 mL in 250 mL DI water, pH ≈ 2) to test the minimum effective dose for $LiFePO_4$ breakdown; and (iv) multi-step leaching with repeated mild formic acid treatments to evaluate cumulative lithium recovery benefits.

2.2. Precipitation of Li_2CO_3 and Removal of Impurities

With the green lixiviant leaching route of maximum Lithium extraction, the pregnant leaching solution was processed targeting Li_2CO_3 as an end-product. This was achieved by first adding 2 $mol.L^{-1}$ NaOH at 90°C to remove impurities like Fe, Al and Cu, then adding 2 $mol.L^{-1}$ of Na_2CO_3 at 80°C. The precipitated compound was characterized using ICP, XRD, and FESEM (Zeiss Ultra 55 Field Emission Scanning Electron Microscope) with an acceleration voltage of 3 kV.

4. Results and Discussion: Efficient, Selective Lithium Leaching

4.1. Effect of parameters: Temperature and S/L ratio

Understanding how lithium behaves under different process settings was the focus of the initial parameter screening. Optimized values from these trials formed the basis for our leaching studies. Starting with reaction temperature, it strongly influences leaching kinetics, but its benefits are lixiviant-dependent. Literature reports (Li et al., 2017) up to 95 % Li recovery in just 30 min at 80°C using H_2SO_4/H_2O_2, compared to 60 % at 25°C, but with increased energy demands and accelerated H_2O_2 decomposition above 60°C. Preliminary experiments conducted at a fixed S/L ratio of 20 g/L and constant reagent concentrations showed that raising the temperature had little effect on both water and sulfuric acid-based leaching. In contrast, formic acid + H_2O_2 exhibited a marked 24 % improvement at 88°C, reaching 89 % Li recovery, indicating enhanced reactivity at elevated temperatures despite potential peroxide losses. Importantly, temperature did not significantly affect impurity metal dissolution, emphasizing its primary role here in facilitating lithium release, particularly with organic acid systems ("Parameter Study on the Recycling of LFP Cathode Material Using Hydrometallurgical Methods"), (Li et al., 2024), (Kumar et al., 2020).

S/L ratio governs reagent accessibility and slurry dynamics, with literature studies (Yongxia et al., 2018, Jing et al., 2019) commonly associating lower pulp densities (10–20 g/L) with higher recovery due to

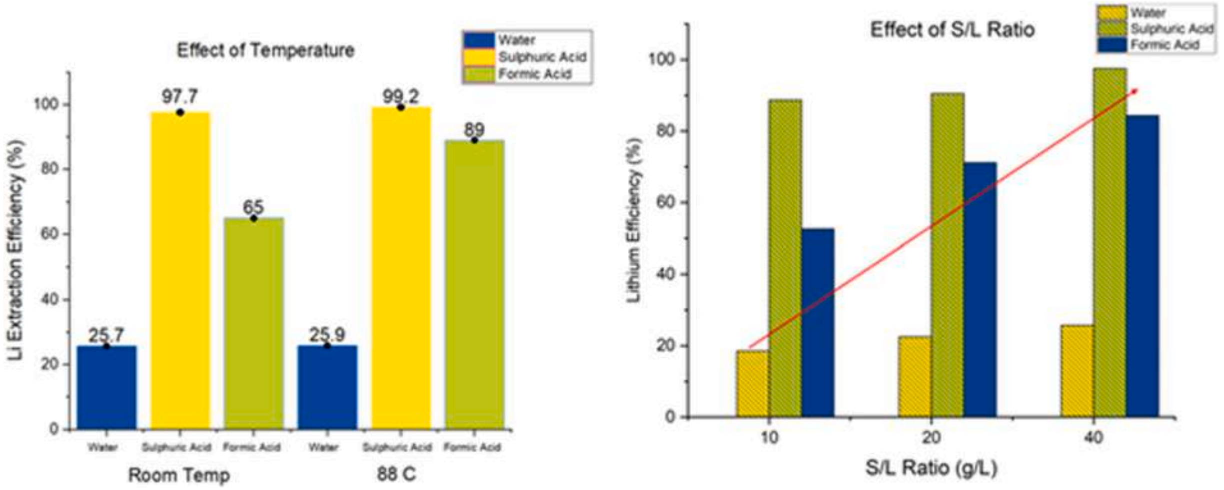


Fig. 3. (a) Influence of temperature on lithium extraction performance using water, sulfuric acid, and formic acid systems (b) Variation in lithium extraction efficiency with changing pulp density under constant reaction conditions of 90 mins.

reduced crowding and improved diffusion. Surprisingly, pretrials on industrial LFP blackmass (water + H_2O_2, room temperature) revealed the opposite with 40 g/L outperforming both 10 and 20 g/L similarly for sulfuric and formic acid as well (Fig. 3(b)). In our understanding, this behaviour is linked to the complex feed composition where excess liquid at low S/L diluted active reagents and promoted graphite flotation/agglomeration, limiting contact with the active material. At 40 g/L, a higher local concentration of acid/oxidant around particles, improved mixing, and the early precipitation of impurities such as Al reduced side reactions and interference. In addition, graphite at this density may have aided redox pathways, selectively oxidizing Fe^{2+} to Fe^{3+} and stabilizing Fe and PO_4 as insoluble phases thereby favoring lithium dissolution. These findings highlight the need to tune operational parameters specifically for *real* blackmass, as trends from pure materials do not always translate to industrial feedstocks, as evident from research ("Effect of Oxidative Roasting on Selective Leaching of Lithium from Industrially Shredded Lithium Iron Phosphate Blackmass", Zhou et al., 2025). The leachability of LFP and formation of water-soluble lithium compounds need to be addressed for industrially shredded blackmass.

4.2. Effect of Lixiviants on Efficiency and Selectivity

Group A: Water-Based Leaching

Neutral pH, water-based leaching proved highly ineffective for industrial LFP blackmass. Pure water extracted just 18.15% of lithium with negligible iron dissolution. The addition of H_2O_2 improved Li recovery only marginally (to 19.5%), demonstrating that the stable olivine structure of $LiFePO_4$ resists aqueous dissolution. Even after a two-step water leaching, cumulative Li extraction reached just 30.2%, confirming that neutral routes cannot leach most of the lithium (Fig. 4 (a), Eq. i).

$$LiFePO_4 + H_2O + H2O2 \rightarrow Li^{+}(aq) + FePO_4(s) + O2 + H_2O \tag{i}$$

Group B: Sulfuric Acid as a Benchmark

Conventional sulfuric acid leaching (2 M) achieved near-complete Li extraction (95.93%) but dissolved almost all iron as well (95.96%), revealing a major selectivity problem. Adding H_2O_2 halved iron dissolution (50.3%) while maintaining strong Li recovery (81.5%), showing the oxidant's key role in improving, but very low selectivity (Fig. 4(b), Eq. ii)

$$2LiFePO4(s) + H2SO4 + H2O2(aq) \rightarrow 2Li^{+} + 2Fe^{+3} + SO4^{-2} + 2PO4^{-3} + 2H2O \tag{ii}$$

Group C: Formic Acid Approaches

Formic acid alone displayed moderate Li extraction (71.2%) and significant iron leaching (61.0%). However, combining formic acid with H_2O_2 provided a dramatic leap in selectivity: lithium recovery rose to 83% while iron dissolution dropped to only 2.01%. This demonstrates that formic acid, in synergy with an oxidant, is uniquely effective at differentiating between Li and Fe in real blackmass systems (Fig. 4(c), Eq. iii)

$$2LiFePO_4 + H_2O_2 + 2HCOOH \rightarrow 2Li^{+}(aq) + 2Fe^{+3} + 2PO_4{}^{-3}(aq) + 2HCOO^{-}(aq) + 2H_2O \tag{iii}$$

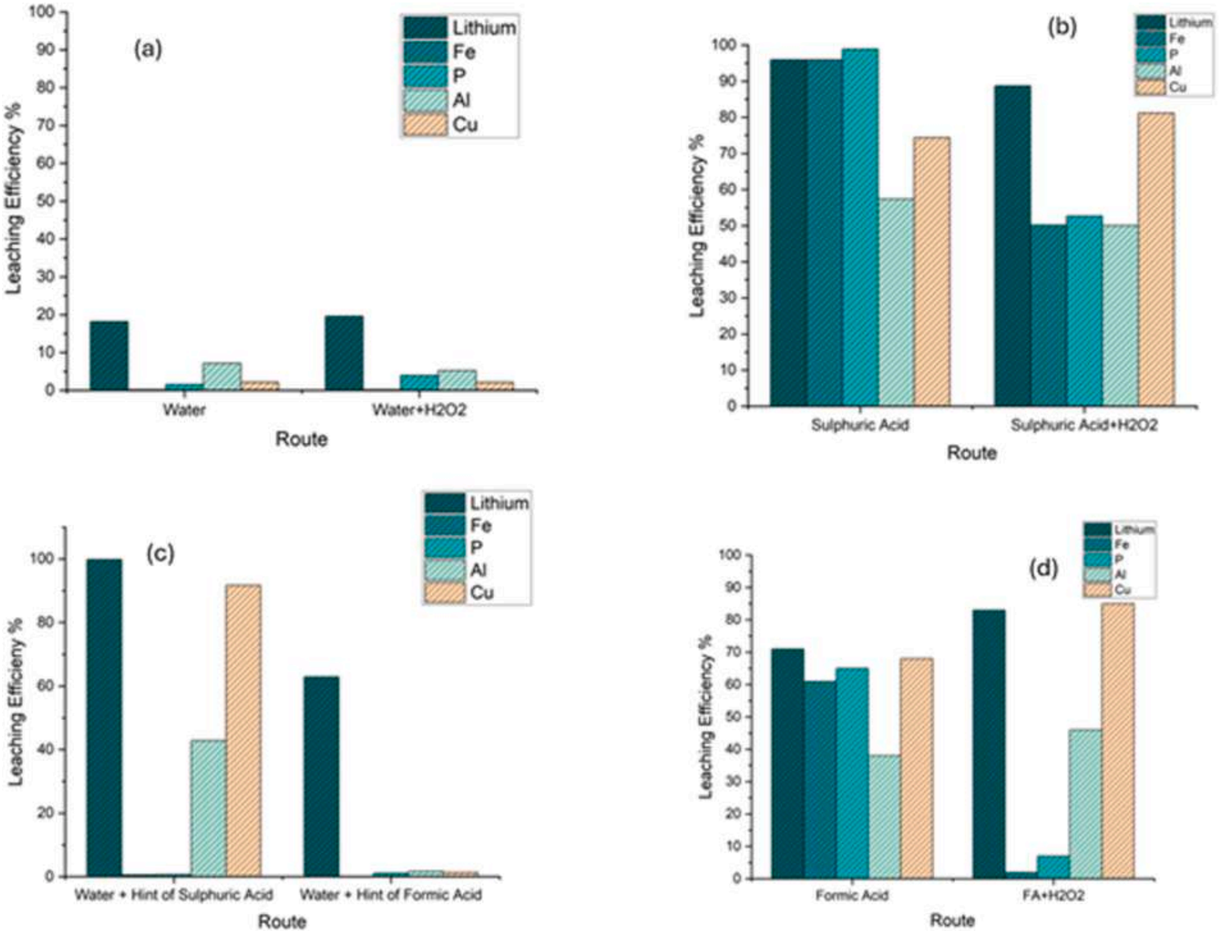


Fig. 4. Leaching Efficiency for different approaches using lixiviants (a) Water (b) Sulfuric acid (c) Formic Acid (d) 'Hint of Acid' Approach.

Hint of Acid Strategies

The “hint of acid” approach emerged as one of the most significant findings of this work, demonstrating that near-complete lithium extraction from industrial LFP blackmass can be achieved without resorting to harsh chemical conditions. In the first approach, the use of only trace amounts of sulfuric acid in combination with hydrogen peroxide enabled 97.81 % lithium recovery while simultaneously restricting iron dissolution to an exceptional 0.55 %. This level of selectivity, almost complete target-metal recovery with negligible co-leaching, represents what is often considered the gold standard of hydrometallurgical recycling. Whereas the second approach, using a similarly mild dosage of formic acid (88%) with peroxide, achieved 62.7 % lithium recovery at 0.35 % Fe dissolution, shown in Fig. 4(d), confirming that even minimal organic acid inputs can substantially enhance lithium release when paired with an oxidizing environment.

While the trace $H_2SO_4 + H_2O_2$ condition achieves the most selective lithium dissolution, its higher sulfate load and associated treatment requirements make it less attractive for large-scale closed-loop recycling, motivating the development of the low-acid formic-acid route explored here.

The success of this method lies in the precise electrochemical behaviour of LFP in the lixiviant. It has been studied using the E-pH diagram to understand that at neutral pH, $LiFePO_4$ remains in a stable region, resistant to dissolution. Reducing the pH only to ~2–4 pushes lithium into its soluble regime without destabilizing the Fe–PO_4 framework. Hydrogen peroxide plays a dual role: oxidizing Fe^{2+} to Fe^{3+}, which locks iron into insoluble $FePO_4$, while maintaining lithium in solution, depicted in Eq. iv and v.

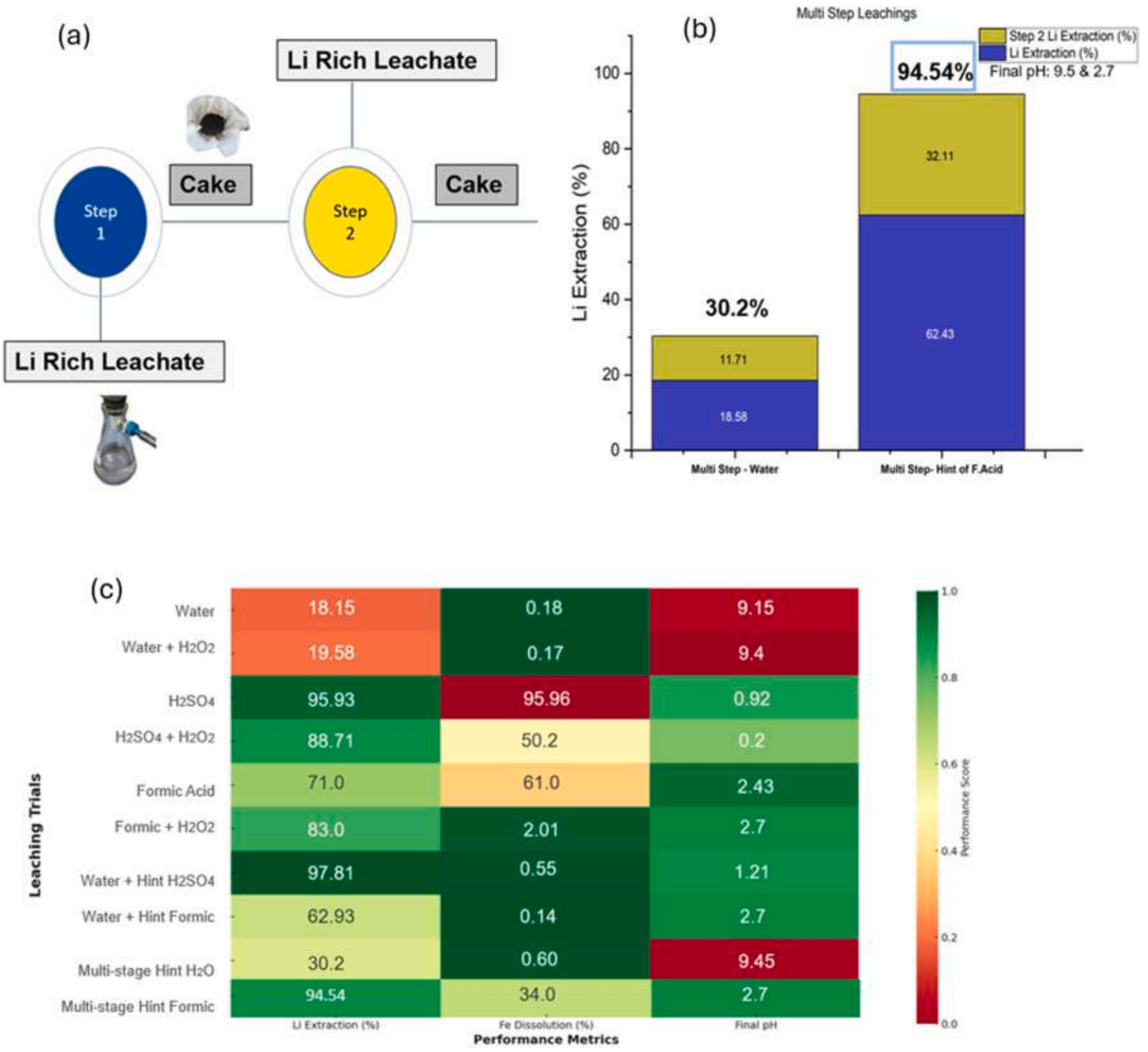
$$2Fe^{2+} + H_2O_2 + 2H^{+} \rightarrow 2Fe^{3+} + 2H_2O \tag{iv}$$

$$Fe^{3+} + {PO_4}^{3-} \rightarrow FePO_4\ (s) \tag{v}$$

Unlike conventional high-acid leaching, which indiscriminately forces both lithium and iron into solution, this tuned system operates in a narrow electrochemical window where lithium dissolution is thermodynamically favored, yet $FePO_4$ stability is preserved. Though the kinetics are gentler than strong acid routes, this slower pace fosters controlled $FePO_4$ layer formation, avoids secondary phase traps, and, critically, slashes chemical consumption and effluent load. The result is a greener, safer, and industrially scalable pathway, illustrating that selective, high-yield lithium recovery does not require environmentally aggressive leaching chemistry and hence answers our main research objective.

Multi-Step Leaching

Multi-step leaching with a hint of formic acid approach, drove total



	Li Extraction (%)	Fe Dissolution (%)	Final pH
Water	18.15	0.18	9.15
Water + H2O2	19.58	0.17	9.4
H2SO4	95.93	95.96	0.92
H2SO4 + H2O2	88.71	50.2	0.2
Formic Acid	71.0	61.0	2.43
Formic + H2O2	83.0	2.01	2.7
Water + Hint H2SO4	97.81	0.55	1.21
Water + Hint Formic	62.93	0.14	2.7
Multi-stage Hint H2O	30.2	0.60	9.45
Multi-stage Hint Formic	94.54	34.0	2.7

Fig. 5. (a) Experimental approach for 2-step leaching process (b) Leaching efficiency of Lithium in water and Hint of Formic acid 2-step leaching, with blue bar indicating first step Li extraction efficiency and green bar indicating second step leaching efficiency of lithium (c) Heatmap showing Leaching routes on y-axis and targeted Lithium extraction along with Fe dissolution and final pH of leaching solution on x-axis.

lithium recovery to 94.6%, as shown in Fig. 5; however, iron dissolution reached 30.21%. This increase is attributed to the gradual erosion of the protective $FePO_4$ layer shown in the Supplementary material. Critically, the two-step method is invaluable for complex industrial blackmass. The first leach neutralizes reactive impurities and clears away metals (like Al, Cu) and some graphite. The residue is then filtered, reducing physical barriers and reagent competition. The second step, using fresh lixiviant, targets any remaining LFP, ensuring high lithium recovery by optimizing contact and minimizing side reactions.

Adopting a multistep leaching strategy is an effective way to navigate this complexity. The first mild acid stage acts as an “impurity scavenging” step, quickly neutralizing and removing most reactive electrolyte salts and Al/Cu impurities into the solution, where they can be filtered away. Once these competitive compounds are out of the way, a fresh acid/oxidant is introduced in the second leaching stage, now able to target $LiFePO_4$ with far greater selectivity and efficiency. Lithium can thus be extracted under controlled conditions, maximizing recovery and minimizing unwanted dissolution of iron or phosphorus. This staged approach, tailored for real-world blackmass composition, transforms a messy, interfering waste stream into a process where every reagent addition counts, making high-yield, selective lithium recycling practically attainable for industrial feedstocks.

The heatmap in Fig. 5 fractionate complex leaching data into a clear visual comparison of lithium recovery and selectivity across all tested routes. Color intensity conveys performance at a glance, highlighting conditions, most notably the “hint of acid” + H_2O_2 approach, that achieve high lithium yields with minimal iron dissolution. Conventional strong-acid systems deliver efficiency but at the expense of selectivity, whereas greener, milder strategies consistently balance both. This visual tool was included to enable rapid identification of the most promising, environmentally aligned leaching pathways, making the rationale and impact of our approach immediately accessible to both academic and industrial audiences.

5. Precipitation and Product Purification

Following optimized leaching, lithium was selectively recovered from the purified pregnant leaching solution (PLS) using Na_2CO_3 as the precipitating agent. Before precipitation of lithium carbonate, impurities such as iron, aluminum, and copper were removed by sequential pH adjustment with NaOH (2M to pH 4–5, then 2 M to pH 5–7), leading to efficient precipitation of their respective oxides.

Both Jai Kumar et al. and Zheng et al. (Kumar et al., 2022, Zheng et al., 2016) employed precipitation methods closely matching, where iron and other impurities were first removed from the leachate by adjusting the pH with NaOH, as indicated by Fig. 6(a), followed by lithium recovery through precipitation with Na_2CO_3 to obtain high-purity Li_2CO_3.

For the last step of precipitation, a stoichiometric Na_2CO_3 solution was slowly added to the concentrated PLS, impurity-free leachate at 80–90°C

The optimal precipitation conditions reported in (Cai et al., 2014, Jing et al., 2024) typically use CO_3^{2-}/Li^+ ratios ranging from 0.5 to 2, with the stoichiometric 0.5 ratio being the theoretical minimum needed

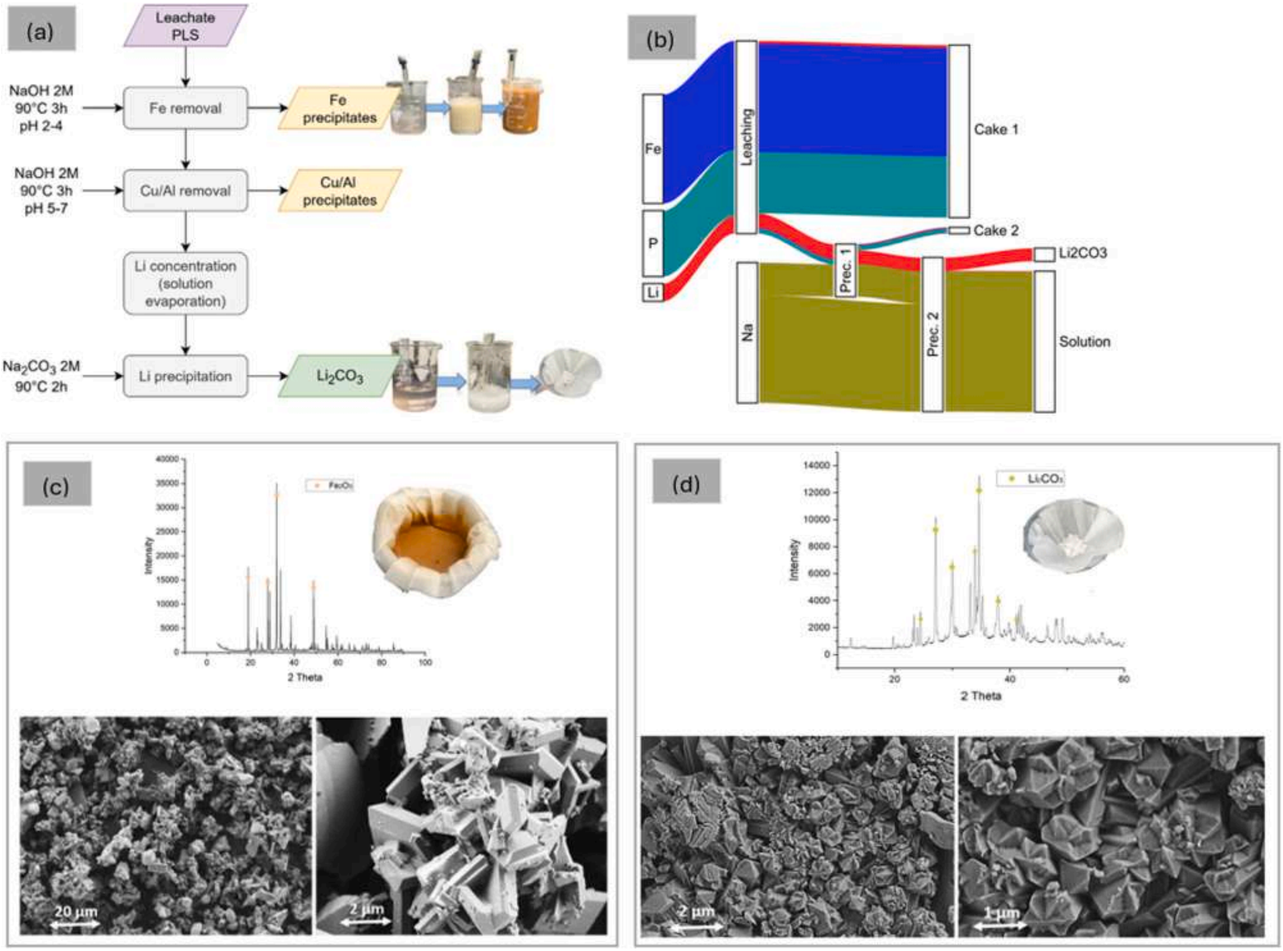


Fig. 6. (a) Experimental Flowchart for precipitation using Pregnant leaching solution (PLS), along with precipitated products of iron and lithium, (b) Sankey diagram showing process mass flow for lithium extraction research route, along with Li losses along the pathways and XRD and SEM Analysis of (c) Iron oxide, (d) Precipitated Lithium carbonate.

for complete precipitation. Slight excess sodium carbonate is often used to drive precipitation to completion without forming excess sodium carbonate impurities, the reason Li: Na of 0.93 is being used in this research.

The resultant Li_2CO_3 was collected by vacuum filtration, thoroughly washed with hot deionized water to remove residual sodium, and oven-dried to constant mass. XRD and SEM analysis of the white precipitates confirmed lithium carbonate product formation with removal of sodium carbonate after washing.

5.1. Iron Oxide Precipitates

The iron-rich precipitate obtained during the impurity removal step was characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The XRD pattern, Fig. 6(c), revealed distinct peaks corresponding to crystalline hematite (α-Fe_2O_3), as indexed by the ICDD reference PDF# 33-0664. These sharp reflections confirm the complete and well-ordered precipitation of iron as iron oxide (Ding et al., 2023, Gerold et al., 2021).

SEM analysis further illustrated the microstructure of the precipitate, showing densely aggregated, irregular particles with rough surfaces. This morphology is typical for iron oxide phases formed under aqueous precipitation conditions, reflecting effective nucleation and growth. Together, these results confirm successful iron removal as a stable, crystalline iron oxide phase, supporting minimum impurity downstream lithium recovery in line with best practices reported in recent recycling literature (Zou et al., 2024, Kumar et al., 2022, Ferreira et al., 2009, Jie et al., 2020).

5.2. Lithium Carbonate Precipitates

X-ray diffraction (XRD) analysis, shown in Fig. 6(d), of the lithium carbonate (Li_2CO_3) precipitates obtained during the recycling process displays sharp, well-defined peaks that are in excellent agreement with the standard Li_2CO_3 diffraction pattern (ICDD PDF No. 22-1141). These results confirm the high crystallinity and phase purity of the recovered lithium carbonate, with less discernible secondary phases present. Trace iron impurities, if any, would be identified as iron oxide reflections, commonly hematite (α-Fe_2O_3, ICDD PDF No. 33-0664) or magnetite (Fe_3O_4, ICDD PDF No. 19-0629), none of which are observed in the XRD scan, depicting the effectiveness of the impurity removal steps (Gao et al., 2017, Kim et al., 2024, Bian et al., 2016).

Scanning electron microscopy (SEM) images reveal that the Li_2CO_3 product consists of densely packed, well-faceted prismatic crystallites, average of 74 μm. The uniform morphology and clean surfaces further validate the XRD findings, indicating minimal contamination and successful removal of residual Na_2CO_3 or other potential process-related impurities like Na (Jing et al., 2024). This microstructural feature is consistent with reports on recycled lithium carbonate from battery waste of LFP.

Lithium Recovery Rate & Mass Flow

The process mass balance, illustrated in the Sankey diagram (Fig. 6 (b)), captures lithium flows from leaching through precipitation, providing a clear view of recovery efficiencies and loss points. Starting with 218 mg of lithium in the original LFP blackmass, the two-step leaching successfully transferred 206 mg into solution, a 94.5 % leaching efficiency, demonstrating the high selectivity and effectiveness of the chosen lixiviant system. During precipitation, 180 mg of lithium was recovered as impurity free Li_2CO_3, representing 82.6 % overall recovery from the feed, or ~87 % of the lithium that was leached, using Eq. vi. Loss analysis reveals that 15.6 mg (7.2 %) remained in solid residues, likely bound within secondary phases or unreacted material, while 10 mg (~5 %) was lost during pH adjustment, washing, or handling. Along with Li, Fe and P, metallic inclusions flow was also evaluated throughout, starting with 0.74% Al and 1.10 % Cu in blackmass and leached in a small amount of 0.5 % and 1 % in PLS, which was further removed via NaOH during the precipitation stage, leaving behind less than 0.01 % in Li-rich PLS. These results confirm that while leaching performance is already near optimal, the main opportunity for process enhancement lies in refining the precipitation and solid–liquid separation steps to further minimize lithium losses and push the overall recovery closer to the theoretical maximum.

$$Recovery(\ \%) = \frac{(Mass\ of\ Li\ in\ precipitate)}{(Mass\ of\ Li\ in\ feed)} \cdot 100 \tag{vi}$$

6. Conclusion and Outlook

This research establishes the two-stage diluted formic acid leaching approach, combined with hydrogen peroxide, as a significant advancement in sustainable hydrometallurgical recycling of industrial LFP blackmass. This methodology effectively overcomes the inherent challenges posed by complex, impurity-laden feedstocks, achieving over 95 % in the two-step formic acid route, while the trace $H_2SO_4 + H_2O_2$ pathway provided the highest selectivity (>95% Li with negligible Fe co-leaching) producing low-impurity lithium carbonate confirmed by XRD and SEM. Our experimental findings show that optimizing parameters such as temperature and solid-to-liquid ratio is crucial, as the complex nature of industrial blackmass demands precise process control to manage impurities and slurry behaviour. Incorporating multi-step leaching and sequential impurity removal successfully tackled contaminants like graphite, aluminum, copper, and binders, improving scalability and reducing waste. Mass balance analysis reveals that while 80 % of Li from LFP production scrap is recovered as carbonate following the hint of formic acid path, further refinement in precipitation and separation steps is needed to push lithium recovery toward theoretical maxima and minimize loss pathways. Visual tools like heatmaps proved invaluable for evaluating performance and guiding optimization.

The scope of this study is to work provide bench-scale proof of concept, further study is required to support industrial adoption. Future efforts will include larger-scale trials, time-resolved kinetic measurements, and a full techno-economic comparison of the sulfuric-acid and formic-acid routes. Advancing water recirculation and effluent treatment strategies will also be important for improving process sustainability. These developments will help move LFP recycling toward scalable, resource-efficient, and low-impact operation.

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CRediT authorship contribution statement

Ayesha Tasawar: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Daniel Dotto Munchen:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Data curation, Conceptualization. **Alexander Birich:** Writing – review & editing, Resources, Funding acquisition. **Rungsima Yeetsorn:** Writing – review & editing, Supervision, Funding acquisition. **Walter Sebastian Scheld:** Visualization, Investigation. **Waritnan Wanchan:** Visualization. **Benjamin Butz:** Resources. **Bernd Friedrich:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Ayesha Tasawar reports administrative support, equipment, drugs, or supplies, and writing assistance were provided by RWTH Aachen University Department of Process Metallurgy and Metal Recycling. Ayesha Tasawar reports a relationship with RWTH Aachen University Department of Process Metallurgy and Metal Recycling that includes: employment. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.resconrec.2025.108766.

Data availability

Data will be made available on request.

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