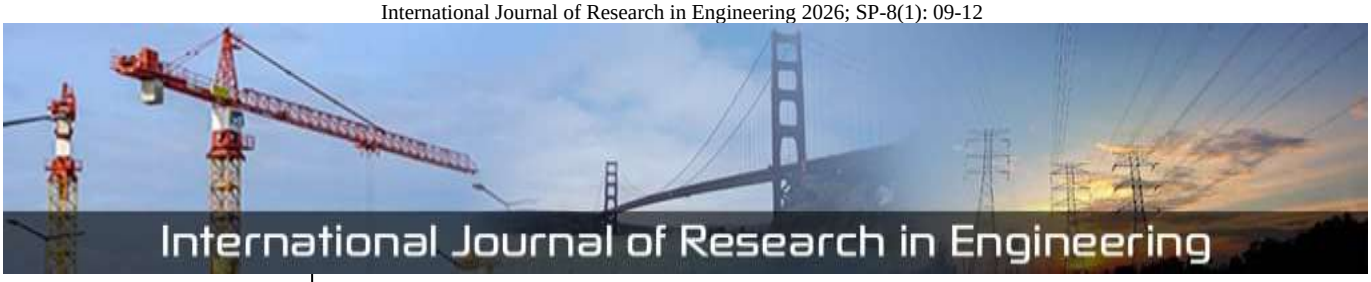

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Selective extraction of lithium from spent EV batteries by using organic acid

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Abstract
Rising demand for electric vehicles (EVs) has intensified the need for effective lithium-iron-phosphate (LFP) battery recycling methods. This research presents an oxalic acid-hydrogen peroxide leaching process for selective lithium recovery from spent EV batteries. Optimization of acid concentration, H_2O_2 dosage, pulp density, reaction time, and temperature achieved 99.4% lithium extraction efficiency.
Optimal conditions comprised 10% (w/v) oxalic acid, 5% (v/v) H_2O_2, 100 g/L pulp density, 300 rpm stirring speed, and 60°C for 4 hours. XRD and SEM analyses verified the purity and morphology of the recovered lithium material. This method enables complete LFP battery recycling, advances circular economy principles, minimizes environmental impact, simplifies waste management, and provides substantial benefits to the EV sector.

Keywords: Blackmass, Hydrometallurgy, LFP battery, Selective leaching, Oxalic acid

Introduction
Lithium iron phosphate (LFP) batteries have become dominant in electric vehicles (EVs) due to their advantages, like low cost, excellent power density, and long cycle life (Li *et al*., 2024) [3]. This surge in popularity has boosted production dramatically, with the global LFP market valued at $17.54 billion in 2023 and projected to reach $48.95 billion by 2031 (Lu *et al*., 2024) [4]. However, widespread LFP battery deployment raises critical challenges in resource conservation, economic viability, and environmental protection.
Improper disposal of spent LFP batteries risks serious ecological damage, including soil and groundwater contamination (Alipanah *et al*., 2021) [1]. Recycling emerges as a key solution, recovering valuable metals while minimizing the financial and environmental costs of primary mining. The process typically involves mechanical pretreatment to obtain black mass, followed by leaching via pyrometallurgy, hydrometallurgy, or bio-hydrometallurgy (Mrozik *et al*., 2021) [8].
Hydrometallurgy stands out for its superior metal recovery rates, lower energy demands, and reduced emissions. Traditional approaches use aggressive inorganic acids (HCl, HNO_3, H_2SO_4) to extract metals from spent LFP batteries (Yadav *et al*., 2020) [9]. Yet, their environmental drawbacks have driven exploration of organic acids—such as oxalic, tartaric, ascorbic, citric, and malic acids—as sustainable alternatives (Mahandra & Ghahreman, 2021) [6].
This study develops an innovative oxalic acid-hydrogen peroxide system for selective lithium leaching. This method offers high efficiency, specificity, and environmental compatibility, with low costs and simple operations. Such advancements in recycling technology promote industry sustainability and address waste management pressures.

Materials & Methodology
Materials and Reagents
This research used 10 kg of spent prismatic LFP batteries obtained from a local supplier in Pune, India. Commercial-grade oxalic acid ($C_2H_2O_4 \cdot 2H_2O$) and hydrogen peroxide (H_2O_2) were employed as leaching reagents. Ultrapure deionized water served for sample

preparation, dilutions, and washing procedures.

Disassembly and Pre-processing of LFP Batteries

Spent LFP batteries were first discharged safely in 15% NaCl solution to eliminate hazards (Mishra *et al*., 2025). Electrode materials were then subjected to thermal treatment followed by hot water rinsing to isolate purified black mass.

Leaching of Black Mass Powder

Oxalic acid was selected for its eco-friendly nature and selective lithium extraction properties. A specific quantity of black mass was added to a solution containing oxalic acid, deionized water, and H_2O_2 under controlled temperature conditions. The slurry was filtered through Whatman No. 42 filter paper, with the solid residue rinsed using deionized water and dried at 130°C for 24 hours in an oven.

Metal leaching efficiency was calculated using Equation (1):

$$\%\ Leachikg\ efficiekcy = CE * V / Co * m * 100 \quad (1)$$

The formula uses CE to represent the metal concentration in the leachate solution (g/L), L to represent the leachate's total volume, Co to represent the original metal content in the black mass (weight%), and m to represent the mass of the black mass that is being leached (g). ICP-OES and AAS were used to analyze the leachate solutions, and XRD and thorough chemical analysis were used to characterize the residues. All trials were carried out in duplicate to guarantee accuracy, and the final metal recovery percentages were calculated using the average results.

Result and Discussion

Black Mass Composition Analysis

Black mass powder was characterized for elemental and structural properties. ICP-OES (Thermo Scientific iCAP 7600) and AAS (Shimadzu AA-7000F) identified lithium (3.13%), iron (13.7%), and phosphorus (7.3%). XRD detected $LiFePO_4$ and $FePO_4$ phases (Figure 2). SEM showed irregular particles with heterogeneous size distribution (Figure 1).

These findings confirm the material's complex multi-phase nature, critical for leaching process design.

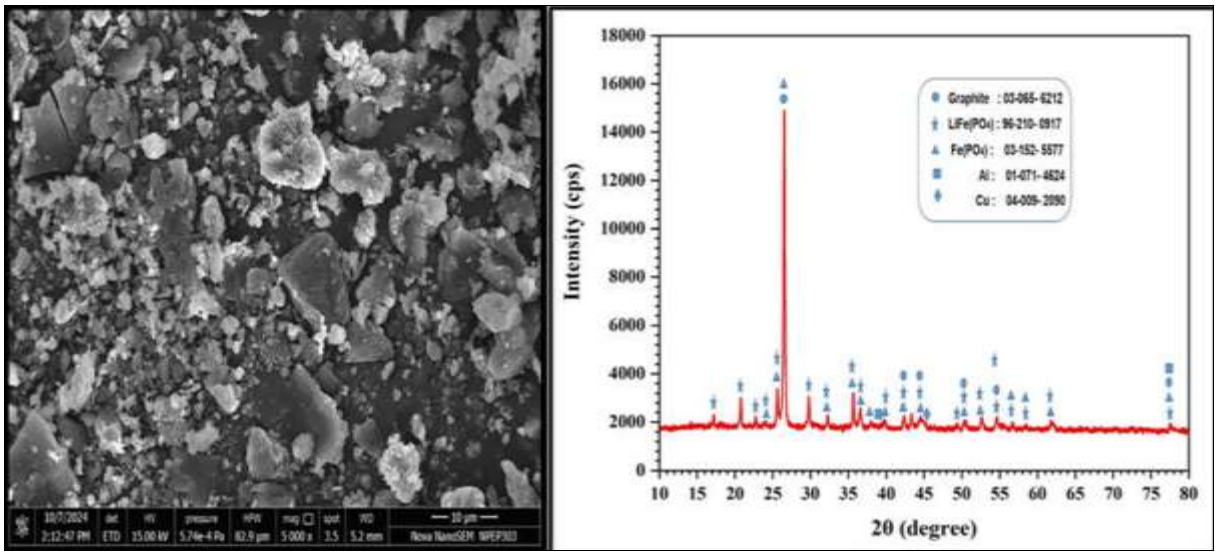

Fig 1: SEM & XRD of spent LFP black mass powder.

1.1 Effect of Different Leaching Parameters

Oxalic acid's environmentally friendly and cost-effective properties make it suitable for selective lithium leaching. A parametric optimization study examined lithium recovery from scrap LFP black mass by varying oxalic acid concentration, H_2O_2 dosage, pulp density, reaction time, and temperature, as shown in Figure 2.

Researchers examined how oxalic acid concentration affects the leaching efficiencies of key metals. As shown in Figure 2(a), raising the concentration from 5% to 10% (w/v) boosted lithium recovery from 39% to 66%, iron from 2.5% to 4.2%, and phosphorus from 1.5% to 2.5%. The 10% (w/v) level proved optimal, providing ample protons and acid radicals for effective metal extraction. Concentrations above 15-30% (w/v) yielded only slight gains, likely due to higher ionic strength impeding further leaching (Li *et al*., 2018). [2]

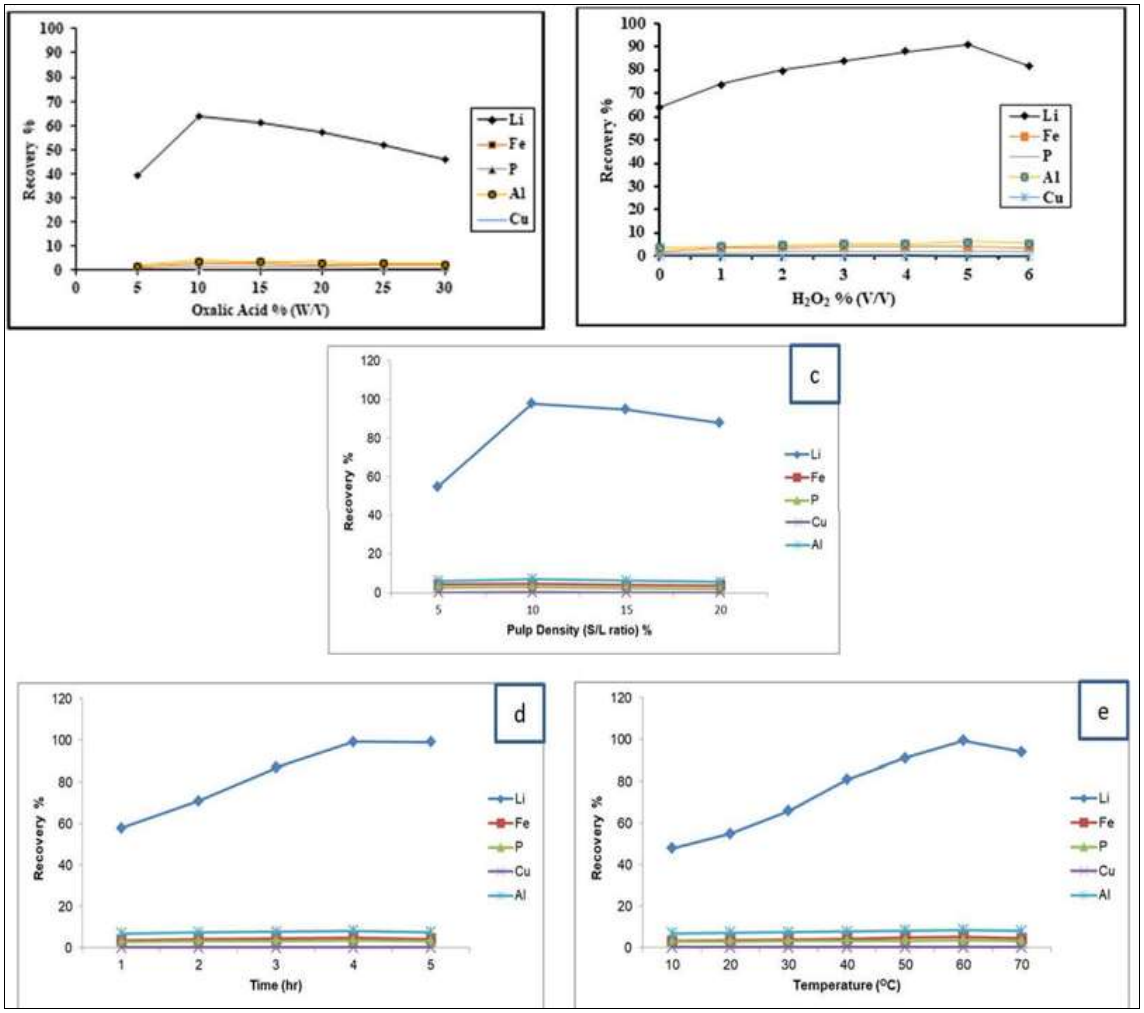

Fig 2: Influence of oxalic acid concentrations(a), H2O2volume(b), pulp density(c), reaction time(d), and temperature(e) on leaching efficiency.

H_2O_2 Dosage Effects

Figure 2(b) illustrates the impact of H_2O_2 dosage on leaching efficiency. Higher concentrations markedly enhanced recovery, with 5% (v/v) emerging as optimal. This stems from H_2O_2's role in degrading organic matter, oxidizing Fe^{2+} to Fe^{3+}, and breaking down mineral structures to improve metal release. Excess beyond 5% (v/v), however, risks forming insoluble compounds that lower yields (Mahandra & Ghahreman, 2021) [6].

Pulp Density Effects

Lower pulp densities increase oxalic acid availability in solution. Figure 2(c) indicates 10% pulp density as optimal, striking a balance between acid supply and metal ion levels. Elevated densities reduce efficiency by raising pH and limiting metal solubility (Zheng *et al*., 2016) [10].

Reaction Time Effects

Leaching efficiencies for various metals rose with reaction time, as depicted in Figure 2(d), peaking at 4 hours. This duration allows sufficient diffusion of reactants into the black mass and release of products from the solid phase (Luo *et al*., 2021) [5].

Temperature Effects

Figure 2(e) highlights the temperature's role in leaching, identifying 60°C as optimal. At this point, heightened molecular energy and diffusion rates maximize metal recovery. Higher temperatures diminish performance through reductant breakdown and rapid H_2O_2 decomposition (Mishra *et al*., 2025). Overall optimal conditions include 10% (w/v) oxalic acid, 5% (v/v) H_2O_2, 10% pulp density, 4 hours, and 60 °C.

Leaching Residue Analysis

Post-leaching, the slurry underwent filtration, rinsing, and drying. The resulting leach liquor was lithium-rich, while the residue contained primarily iron (13.02%) and phosphorus (7.03%). SEM imaging (Figure 4) showed marked particle morphology changes, including prismatic aggregate formation. XRD patterns (Figure 3) confirmed $FePO_4$ and $FeC_2O_4 \cdot 2H_2O$ phases, signaling effective $LiFePO_4$ dissolution. These findings demonstrate successful lithium separation, leaving a residue dominated by $FePO_4$ and $FeC_2O_4 \cdot 2H_2O$.

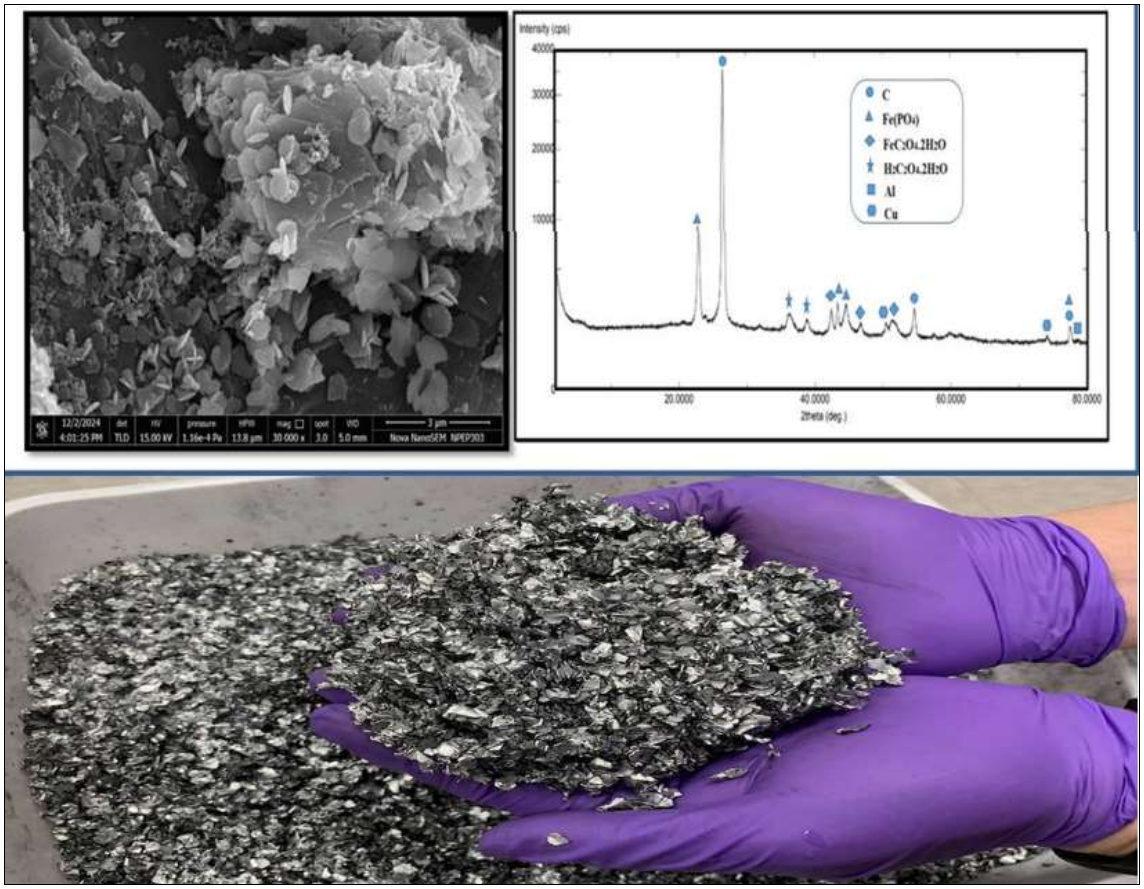

Fig 3: SEM &XRD of black mass powder after leaching.

Conclusion

This research introduces an innovative, environmentally sustainable method for recovering lithium from spent $LiFePO_4$ batteries. Optimal parameters included 10% (w/v) oxalic acid with 5% (v/v) H_2O_2, 100 g/L pulp density, 300 rpm stirring speed, 60°C temperature, and 4-hour duration. These conditions yielded an impressive 99.4% lithium extraction efficiency, with negligible co-extraction of other metals. The residues, mainly $FeC_2O_4 \cdot 2H_2O$, serve as a useful precursor for regenerating $LiFePO_4$. ICP-OES confirmed elemental composition in the black mass, while XRD verified residue phases, demonstrating high-purity lithium recovery. Overall, this approach advances circular economy principles and minimizes environmental harm and supports sustainable energy storage, alleviates waste management challenges, and paves the way for an eco-friendly future.

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