

Microwave Pre-Treatment of Spent Lithium-Ion Battery Electrode Materials: Mass Loss and Morphological Response

AKINYELE, O. S¹, BORODE, J. O², ALABI, O. O³, OLORUNTOBA, D. T⁴

^{1, 2, 3, 4} Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, Nigeria.

Abstract- The growing volume of spent lithium-ion batteries has increased the need for simple and effective pre-treatment steps that improve material liberation before downstream recycling. In this study, dismantled electrode materials recovered from spent commercial lithium-ion battery cells were subjected to microwave pre-treatment after removal of the casing and separator. Microwave irradiation was conducted at 440, 620 and 800 W for exposure times of 5, 10 and 20 minutes (min). The response of the electrode materials was evaluated using percentage mass loss and visual morphological assessment of delamination, powder formation, foil distortion and structural fragmentation. Mass loss ranged from 4.36 to 12.14%, depending on the microwave condition. At 440 W, mass loss remained within a narrow range of 5.05 to 5.35%, indicating mild thermal action and early removal of readily volatile constituents. At 620 W, mass loss decreased slightly with increasing residence time, which is attributed to the heterogeneous distribution of electrode constituents in the dismantled material. At 800 W, mass loss increased from 5.04% at 5 min to 12.14% at 20 min, showing a stronger thermal response and greater removal of volatile or organic fractions. However, visual inspection showed that the most severe condition also promoted brittleness, wrinkling and thermal distortion of the electrode fragments. The results indicate that microwave pre-treatment can assist the physical disintegration and partial liberation of spent battery electrode materials, but treatment severity must be controlled to avoid excessive thermal damage. For conservative material preservation, 440 W for 20 min gave visible delamination with limited foil distortion, while 800 W for 10 min provided greater mass reduction but with a higher risk of thermal damage. The study provides a process screening basis for selecting microwave pre-treatment windows before flotation, leaching or other recovery operations.

Keywords: Lithium-Ion Battery Recycling, Microwave Pre-Treatment, Electrode Liberation, Mass Loss, Electrode Morphology, LiFePO₄

I. INTRODUCTION

Lithium-ion batteries are now central to portable electronics, electric mobility and renewable energy storage. Their rapid deployment has created a parallel increase in spent battery generation and has strengthened the need for recycling routes that can recover valuable materials while reducing environmental burden (Harper et al., 2019; Mrozik et al., 2021; Wei et al., 2023; Giza et al., 2023). Lithium iron phosphate battery systems are especially important because of their safety, long cycle life, thermal stability and lower toxicity when compared with several cobalt rich cathode chemistries (Nitta et al., 2015; Zhang et al., 2023; Cao et al., 2024).

Although these advantages support their large-scale use, spent cells still contain recoverable lithium, graphite, aluminium, copper and other constituents embedded within a strongly bonded electrode structure.

Recycling of spent lithium-ion batteries commonly involves dismantling, mechanical separation, thermal treatment, flotation, leaching or combinations of these operations. A major difficulty is that active material particles are held to aluminium and copper current collectors by polymeric binders and residual electrolyte species. If this adhesion remains strong, the efficiency of separation, flotation and chemical extraction can be reduced (Wang et al., 2018; Zhong et al., 2020; Renier et al., 2023; Müller et al., 2024). Effective pre-treatment is therefore required to weaken electrode adhesion and improve liberation before downstream recovery.

Microwave pre-treatment offers a promising route because microwave energy can generate rapid internal heating in microwave susceptible components of the electrode mixture. Graphite rich regions can absorb microwave energy strongly, while organic constituents and residual electrolyte phases may respond through localized thermal effects. This makes microwave treatment attractive as a potentially faster and more selective alternative to conventional heating when it is properly controlled (Hou et al., 2021; Sun et al., 2023; Cornelio et al., 2024).

However, severe heating can also damage electrode fragments, distort current collectors and alter surface characteristics that are important for downstream separation.

Previous studies have shown that thermal and microwave assisted treatments can promote the removal of volatile species, weaken binder adhesion and enhance liberation of spent battery electrode materials (Ross et al., 2020; Wang et al., 2023; Liu et al., 2024). Nevertheless, practical process screening data are still needed for dismantled electrode materials, particularly where the aim is to identify conditions that promote useful mass loss and delamination without excessive thermal degradation.

This study therefore investigates the influence of microwave power and exposure time on the mass loss and visible morphological response of spent lithium-ion battery electrode materials. The objective is to identify a practical microwave treatment window that can support material liberation before beneficiation or chemical recovery.

II. MATERIALS AND METHODS

2.1 Materials and manual dismantling

Spent commercial lithium-ion battery cells obtained from a Zinox laptop power bank were used for the experiment. The cells were manually opened to remove the external casing and separator, while the remaining cathode and anode rich electrode materials were recovered for microwave treatment. The separated components were visually examined and weighed before treatment. The separator mass was 1.15 g, the battery terminals each weighed 1.54 g, the

cathode rich electrode fraction weighed 19.79 g and the anode rich electrode fraction weighed 13.68 g.

The present study is framed as a physical process screening study. The electrode chemistry was treated according to the supplied battery type and visible electrode architecture, but no independent X ray diffraction, inductively coupled plasma analysis, X-ray fluorescence, Raman spectroscopy or electron microscopy confirmation was performed within the scope of this manuscript. This limitation is important when interpreting the mechanistic discussion and is addressed conservatively in the discussion section.

Table 1: Recovered components from the dismantled spent lithium-ion battery cells.

Component	Observed role in cell	Mass or dimension	Comment
Separator	Electrode separation and ion transport	66 cm length; 1.15 g	Thin polymeric separator removed before microwave treatment
Battery terminals	Electrical connection	1.54 g each	Lightweight conductive connector: removed before microwave treatment
Cathode rich electrode fraction	Active material coated on aluminium current collector	19.79 g	Higher electrode mass fraction
Anode rich electrode fraction	Graphite coated on copper current collector	13.68 g	Copper foil remained visually more stable during treatment
Two Insulating gasket	critical safety component used in	0.15 grams each	Removed before microwave treatment

	battery assembly to prevent short circuits		
Stainless steel shell	provide the mechanical integrity of the lithium-ion battery upon external mechanical loading		Removed before microwave treatment

The samples were placed in clean evaporating dishes before loading into the microwave chamber. Initial mass was measured before irradiation and final mass was measured after treatment and cooling. Percentage mass loss was calculated using Equation 1. Because the work was designed as a preliminary process screening study, interpretation of the mass loss results was supported by visual observation of surface condition, delamination, brittleness, powder formation and foil distortion.

$$\text{Mass loss (\%)} = [(M_i - M_f) / M_i] \times 100 \quad (1)$$

where M_i is the initial mass of the electrode material before microwave treatment and M_f is the final mass after the selected exposure time.

III. RESULTS AND DISCUSSION

3.1 Physical characteristics of dismantled battery components

The dismantled cells showed the typical layered configuration of commercial lithium-ion battery cells, with electrode sheets separated by a thin polymer membrane. The low separator mass and long strip geometry are consistent with the lightweight polyethylene or polypropylene type separators widely reported for liquid electrolyte lithium-ion batteries (Zhang, 2007; Deimede and Elmasides, 2015; Lee et al., 2014; Li et al., 2020). Removal of the separator before microwave treatment was necessary to reduce the risk of polymer melting, uncontrolled fumes and interference with the interpretation of electrode mass loss (Tong and Li 2024; Bläubaum et al., 2024).

The cathode rich fraction had a higher mass than the anode rich fraction. This is reasonable because cathode coatings usually contain a comparatively dense active material layer on aluminium foil, whereas the anode consists largely of graphite coated on copper foil (Neumann et al., 2022; Nitta et al., 2015). From a recycling perspective, the different behaviour of cathode rich particles, graphite rich particles and current collector foils is important because downstream recovery depends on effective liberation and separation of these fractions (Badenhorst et al., 2023; Renier et al., 2023; Müller et al., 2024).



Figure 1: Spent battery cells and recovered components after manual dismantling

2.2 Microwave pre-treatment

Microwave heating was applied as a thermal pre-treatment step to promote removal of volatile constituents and weaken the adhesion between electrode coatings and current collectors. The treatment was carried out using a Sharp R-600ww microwave system with a rated power of 800 W and a frequency of 2450 MHz. The operating powers selected for the experiment were 440, 620 and 800 W. For each power level, electrode materials were exposed to microwave irradiation for 5, 10 and 20 minutes.

3.2 Morphological response to microwave pre-treatment

The untreated electrode sheets were comparatively smooth, continuous and mechanically stable.

According to Liu et al., (2019) describe the untreated electrodes materials (active-material coatings bound into intact sheets) as continuous and mechanically stable. After microwave exposure, the materials generally became darker, flaky and more fragmented, with visible powder formation and detachment of active material from the current collector foils as shown in the circled portion of Figure 2a, 2b, 2c, 3b, and 3c. These changes suggest that microwave irradiation weakened the electrode structure and promoted physical liberation. The response became more pronounced as treatment severity increased, although severe treatment also increased the risk of foil wrinkling, brittleness and thermal distortion. Wu et al., (2026) confirms increasing microwave power/time intensifies delamination/fragmentation while also increasing risks of overheating-related damage (e.g., foil deformation or thermal distortion).

At 440 W, the treated material showed visible delamination and powder formation while the foil fragments appeared comparatively less distorted. This condition therefore represents a mild treatment range where the electrode structure was weakened without the most severe visual signs of overheating. At 620 W, brittleness and fragmentation increased, and at longer exposure time the electrode pieces became more distorted. At 800 W, the treated materials showed the strongest fragmentation and the most visible signs of thermal damage, especially at the longer exposure times. These observations support the need to balance liberation against the risk of excessive thermal degradation.



Figure 2: Representative morphology of electrode materials treated at 440 W for 5, 10 and 20 min



Figure 3: Representative morphology of electrode materials treated at 620 W for 5, 10 and 20 min

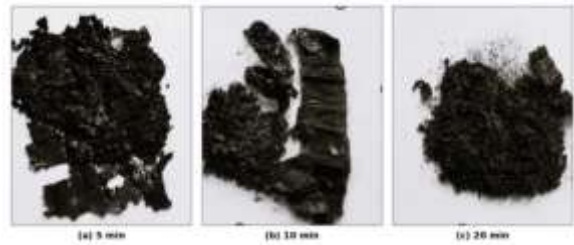


Figure 4: Representative morphology of electrode materials treated at 800 W for 5, 10 and 20 min

Table 2: Summary of observed morphological response after microwave pre-treatment

Condition	Surface appearance	Delamination	Foil condition	Interpretation
Untreated	Smooth black surface with metallic sheen	Active material strongly adhered	Smooth rolled sheets	Baseline electrode condition before thermal action
440 W, 5 to 20 min	Flaky and powdery surface	Severe visible detachment from foil	Foils mostly preserved	Mild to moderate liberation with limited visual foil damage
620 W, 5 to 20 min	More fragmented and brittle	Strong detachment from foil	Wrinkling and distortion increased	Greater thermal effect and higher brittleness
800 W, 5	Highly fragmented	Strong detachment	Clear thermal	Strong treatment

to 20 min	ted and powder rich	nt and particle liberation	l distorti on at severe exposu re	response but increased risk of overheati ng
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3.3 Percentage mass loss during microwave pre-treatment

The percentage mass loss of the dismantled electrode materials varied with microwave power and exposure time, as shown in Table 3 and Figure 5. At 440 W, mass loss values were 5.05, 5.35 and 5.14% after 5, 10 and 20 min, respectively. The narrow range suggests that most readily removable volatile constituents were released early in the treatment, while longer exposure under this mild condition produced only limited additional mass reduction.

At 620 W, mass loss values were 5.68, 5.04 and 4.36% after 5, 10 and 20 min, respectively. The downward trend does not necessarily imply that mass was regained during treatment. Rather, it most likely reflects heterogeneity in the dismantled electrode mixture, including variations in the amount of residual electrolyte, binder-rich fragments, graphite-rich particles, and current collector pieces present in the sample portion used for each condition. Zachmann et al., (2023) similarly reported that thermal pre-treatment and controlled electrolyte removal significantly influence the behaviour of spent lithium-ion battery materials, since residual electrolyte represents one of the most variable constituents in dismantled electrode mixtures and can strongly affect the measured mass loss under nominally identical treatment conditions.

At 800 W, mass loss increased from 5.04% at 5 min to 7.89% at 10 min and 12.14% at 20 min. This trend indicates a stronger thermal response at the highest microwave power. The increase is consistent with greater removal of volatile or organic constituents and with partial thermal degradation of binder containing regions. This finding is consistent with Mannu et al., (2026) and Mousa et al., (2022), stating that microwave thermal treatment removes of VOCs/fluorinated species and higher power increases volatile/organic release from spent LIB black mass.

However, the accompanying visual damage at 800 W, especially after prolonged exposure, shows that higher mass loss should not automatically be interpreted as a better treatment outcome. Excessive thermal severity may change surface properties, increase brittleness and reduce the quality of recovered electrode fractions as confirmed by Liu et al., (2025).

Overall, the results show that microwave power and residence time influence the physical response of spent electrode materials. The most useful process condition depends on the downstream objective. If the priority is conservative material preservation and foil integrity, 440 W for 20 min is preferable because it produced visible delamination with less severe distortion. If the priority is greater volatile removal, 800 W for 10 min may be useful, although it should be applied with temperature control and follow-up separation performance testing. The 800 W for 20 min condition gave the highest mass loss but is less attractive as a balanced pre-treatment because of the risk of overheating and structural damage.

Table 3: Mass loss response of electrode materials after microwave pre-treatment

Microwave power (W)	Exposure time (min)	Mass loss (%)	Process interpretation
440	5	5.05	Mild treatment response with limited additional mass reduction
440	10	5.35	Mild treatment response with limited additional mass reduction
440	20	5.14	Mild treatment response with limited additional mass reduction
620	5	5.68	Moderate response affected by sample

			heterogeneity
620	10	5.04	Moderate response affected by sample heterogeneity
620	20	4.36	Moderate response affected by sample heterogeneity
800	5	5.04	Stronger volatile removal with increasing thermal effect
800	10	7.89	Stronger volatile removal with increasing thermal effect
800	20	12.14	Highest mass loss but greater risk of thermal damage

gravity separation, sieving and hydrometallurgical leaching because liberation affects both recovery efficiency and product purity.

However, the results also show that treatment severity must be controlled. A high mass loss condition may remove more volatile material, but it may also promote excessive brittleness, foil distortion and surface modification of graphite rich fractions. These effects can alter flotation behaviour or leaching response. For this reason, microwave pre-treatment should be selected together with downstream performance data rather than from mass loss alone.

3.5 Limitations of the present study

The study provides useful preliminary process screening data, but some limitations should be recognized. The chemistry of the electrode material was not independently confirmed by X-ray diffraction, inductively coupled plasma – optical emission spectrometry (ICP-OES), ultimate carbon analysis, and Scanning Electron Microscopy with energy dispersive spectrometer (SEM/ EDS). The microwave temperature history was also not measured, and the mass loss values are presented as condition responses rather than statistically optimized mean values. These limitations do not invalidate the observed physical response, but they restrict the strength of mechanistic claims that can be made from the data.

Future work will include particle size analysis after treatment, TGA assessment of organic and binder removal, XRD and Raman evaluation of structural change, SEM and flotation or leaching tests to connect pre-treatment response with actual recycling performance.

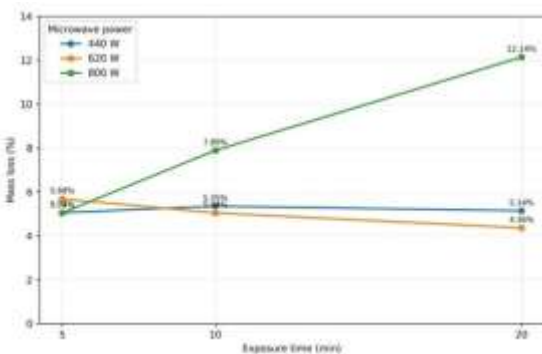


Figure 5: Effect of microwave power and exposure time on percentage mass loss of dismantled spent lithium-ion battery electrode materials.

3.4 Implications for downstream recycling

The main practical value of microwave pre-treatment is not simply mass reduction. Its value lies in weakening the bonding between electrode coatings and current collector foils so that subsequent separation steps can proceed more efficiently. Moderate treatment can assist detachment of active materials, generation of liberated particles and separation of foil fragments from black electrode coatings. These changes are relevant to flotation,

CONCLUSION

Microwave pre-treatment influenced the mass loss and visible morphology of dismantled spent lithium-ion battery electrode materials. The measured mass loss ranged from 4.36 to 12.14%, depending on microwave power and exposure time. At 440 W, mass loss remained within a narrow range, indicating mild thermal action and early removal of readily volatile constituents. At 620 W, the non-monotonic trend was most likely related to heterogeneity in the

dismantled electrode mixture. At 800 W, mass loss increased clearly with exposure time, reaching the highest value at 20 min.

Visual examination showed that microwave treatment promoted delamination, fragmentation and powder formation, which are desirable for electrode liberation. However, severe exposure, particularly at 800 W for 20 min, also caused stronger brittleness and thermal distortion. Therefore, the most suitable condition depends on the intended downstream process. For preservation of material integrity, 440 W for 20 min appears more conservative, while 800 W for 10 min may be useful where greater volatile removal is required but should be applied with care. The highest severity condition should not be selected solely because it gives the highest mass loss.

The findings support microwave pre-treatment as a useful preliminary step for improving the physical liberation of spent lithium-ion battery electrode materials before flotation, leaching or related recovery operations. Further characterization and downstream recovery tests are required to establish the optimum condition with stronger statistical and mechanistic confidence.

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