Recovery of NMC-lithium battery black mass by microwave heating processes

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ABSTRACT

The exceptional microwave absorption capabilities of carbon-based materials make them highly effective for facilitating various chemical reactions, such as synthesis, reduction, exfoliation, doping, and decoration. In this study, we applied microwave irradiation to the thermochemical treatment of spent nickel manganese cobalt (NMC622) lithium-ion batteries, obtained as a mix of the cathode and the anodic graphite. This approach significantly accelerated the chemical reactions, leveraging carbon's robust ability to absorb microwave energy. As a proof-of-concept, we demonstrated that within just 5 min, the carbothermic reduction reactions in the black mass facilitated the formation of water-soluble Li_2O, and enhanced the solubility of Co, Mn, and Ni in a weak acid. More than 90% of Li and 87% of Mn are recovered. This method, which bypasses the need for cathodic and anodic separation, presents a promising new approach for recovering strategic metals from spent lithium-ion batteries.

1. Introduction

In the automotive industry, the most utilized lithium-ion battery (LiB) type is NMC, consisting of a cathode active material with a general composition of $LiNi_{1-x-y}Mn_xCo_yO_2$, indicating the presence of nickel, manganese, and cobalt metals in the lithium-based cathode. Considering that materials account for nearly 75% of the manufacturing expenses in LiB production, recovery of spent battery metals becomes essential not only to assure the availability of the strategic critical raw materials (that can be found in the list provided by the EU Commission in 2023, for the production of strategic products, especially in green energy, digital technology, and defense), but also for battery cost reduction [1]. Generally, recycling processes for LiB cells involve a series of pre-processing methods, mainly focusing on the mechanical process of dismantling the batteries, and concentrating active materials, based on their physical and magnetic properties [2]. These techniques allow to maintenance of the cell chemistry and obtain the so-called "black mass" (BM) resulting in a mixture of mainly the cathode and anode materials. These processes are generally followed by hydrometallurgical or pyrometallurgical approaches. Pyrometallurgy has the advantage of handling mixed waste with complex chemistries, more efficiently in comparison to chemical processes. In addition, the organic components of the batteries, including the electrolyte, PVDF, graphite, and plastic materials, can be burnt off [3,4]. For instance, some authors observed an enhancement in the extraction of both Co and Li after thermal treatment at 700 °C for 60 min, due to the cathode partial decomposition, with the formation of Co_3O_4 at 500 °C, and both Co_3O_4 and CoO at 700 °C [5,6]. A recent paper shows that a treatment performed at 600 °C for 90 min allows to reach a complete decomposition of the graphite and to obtain cobalt, manganese, nickel, and lithium oxides [7]. However, pyrometallurgy has the main significant drawback due to its high energy demand. Indeed, the literature associates thermochemical treatment processes with high energy demand. In addition, the transition metals (Mn, Co, and Ni) with high valence states present in NMC cathodes have a strong chemical bond with oxygen and are even adverse to their leaching behavior [8].

To overcome these problems, a rapid heating method that is not based on conventional furnaces is being developed, allowing

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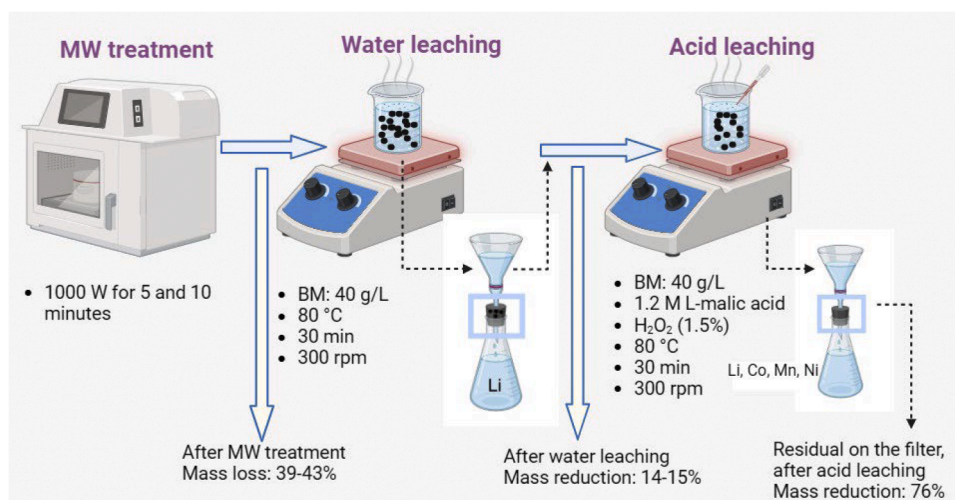*E-mail address:* elza.bontempi@unibs.it (E. Bontempi).

Fig. 1. Procedure steps: after a MW treatment of 5 or 10 min at 1000 W, the sample was subjected to water and acid leaching. Both leaching steps were carried out in a bath heated to 80 °C for 30 min on a magnetic stirrer set to 300 rpm. The same S:L ratio (40 g sample/L leaching solution) was used. Specifically, acid leaching was performed using 1.2 M of L-malic acid solution and 1.5% H_2O_2. The MW procedure led to 39.2 and 43.0% mass loss for the treatment at 5 and 10 min respectively. Then, water leaching and acid leaching of the residuals accounted for mass reductions of 14–15% and 76% respectively. Fig. 1 is created with BioRender.com.

carbothermic reactions using incineration, for NMC cathode recovery, is proposed. The recovery of this kind of LIBs was investigated in a limited amount of works [9]. Recently we have introduced an advance in thermochemical processes, through the integration of microwave (MW), allowing a reduction of energy consumption in comparison to classical thermal treatments [10]. The efficiency of MW heating in carbon-based materials is particularly high due to their strong ability to convert electromagnetic energy into thermal energy. Unlike traditional heating methods that typically heat from the surface inward, MW energy penetrates the material and causes heating throughout the volume of the material simultaneously [11]. This leads to quicker and more uniform heating. MW usage offers two primary advantages: it saves energy that would typically be needed to heat the reaction chamber and the heating process is faster and requires less energy compared to traditional heating technologies [12]. It was recently discussed the potentialities of the new technology to enhance the reactivity of carbon-based materials [13]. It should be also pointed out that MW heating does not require the use of fuel, often necessary to support conventional processes. Another advantage of the treatment is due to the possibility to treat the combination of anodic and cathodic battery materials, avoiding their separation.

In recent years, MW irradiation has become a popular method for synthesizing carbon materials in various morphologies using diverse precursors. Although recent studies have proposed MW treatments for NMC recovery [14], offering an efficient and low-carbon approach to recycling spent LIB, they have been limited to using strong acids and lengthy treatment times of up to one hour. Our work has successfully addressed and improved upon these limitations.

In this work, for the first time, the incineration of NMC-LIBs was performed, based on MW use, also supported by a susceptor, for only a few minutes. A susceptor material absorbs energy from the MW, converting it into heat energy that is then transferred to the processed material. For example, carbon can be used as a susceptor. This material possesses dielectric properties that make it responsive to electromagnetic fields [15,16]. The heating mechanism primarily involves electron conduction due to the delocalized electrons in carbon atoms.

The susceptor-assisted MW heating is known as hybrid MW heating. In the present case, the use of a chamber enhances the MW heating efficiency, with several advantages: more uniform heat distribution and reduced energy consumption (better energy transfer), reduced heat loss, enhanced process reproducibility, rapid heating, non-contact heating, quick start-up and stopping, the possibility to optimize the susceptor and

chamber design, less manual handling of dangerous substances (as heavy metals), the ability to treat waste in-situ, and the portability of equipment and processes. An increase in carbon content can allow for a reduction in the time and/or power for MW carbothermal reduction reactions [17]. The novelties of this study are also related to the use of real waste material, derived from spent NMC-LIBs (literature often refers to model materials) and the direct carbothermic reactions are obtained in ambient conditions (no controlled atmosphere), and without the addition of any chemical. Implementing recovery technologies that avoid the use of costly commercial chemicals is essential for both economically retrieving precious metals and ensuring environmental safety by reducing the spread of toxic compounds.

Emerging technologies, driven by recent advancements, offer significant transformative potential, particularly in addressing major industrial and environmental challenges including sustainability.

2. Experimental section

2.1. Methods

The NMC-BM was produced by an industrial plant devoted to managing waste batteries from electrical vehicles. The sample was previously subjected to a mechanical pre-treatment, devoted to separating plastics and metallic cases, reducing the BM in a powder, and selecting the finest battery fraction containing the anodic and cathodic materials. The resulting powder passes through a 300-μm sieve.

To investigate the chemical composition of NMC, 0.1 g of NMC as-received sample was digested on the heating plate at 300 rpm in 15 mL of aqua regia (3:1, HCl:HNO$_3$) for 6 h (1 h at room temperature and 5 h at 70 °C). The same procedure was applied to investigate the composition of residuals obtained from the different recovery procedure steps.

X-ray powder diffraction (XRD) analysis of crystalline compounds was performed using a X'Pert PRO diffractometer (PANalytical, Malvern, UK), equipped with the X'Celerator detector and Cu anode (CuKalpha 1.5406 A) operating at 40 KV and 40 mA.

The distribution of the temperature field inside a sample was determined using a thermal imaging camera (Testo 890, Settimo Milanese (MI), Italy).

Scanning electron microscopy (SEM, SNE-ALPHA, Nanoeye-sec, South Korea), equipped with Xplore compact EDS detector (Oxford Instruments), was performed to evaluate the morphology and chemical

Table 1

As received NMC-BM chemical composition. The powder composition was also evaluated after the MW treatment resulting in the organic part loss, and the results were normalized to the original BM composition. The concentrations were evaluated by ICP-MS.

Element	As-received NMC-BM (g/kg)			Treated powder (1000 W 10 min) composition normalized to original BM (g/kg)		
Li	47.7	±	9.5	46.3	±	2.4
Al	9.1	±	1.8	9.6	±	0.5
Mn	70.6	±	14.1	72.1	±	3.7
Co	72.2	±	14.4	72.5	±	3.7
Ni	227.6	±	46	216.7	±	11
Cu	20	±	4	23.5	±	1.2

composition of samples.

Water and L-malic acid leaching of the reduced sample powder was carried out to evaluate the amount of recovered Li, Mn, Co, and Ni [10,18].

The digested and leached solutions after water and L-Malic acid leaching were evaluated by ICP-MS. Elemental analyses of leaching solutions properly diluted with ultrapure 1% wt. nitric acid was performed with a NEXION 2000 ICP-MS (Perkin Elmer) in He mode with kinetic energy discrimination. The instrument was calibrated over the analytical range of interest with appropriate multielemental standard solutions (I.V. Labs, Inc.). The internal standard and standard addition methods were employed to cope with possible matrix effects. The calibration data for the ICP are reported in the Supporting (S1).

2.2. Experimental tests

The technology used in this work was patented (PCT/IB2023/051034). NMC-BM samples (5.505 g) were treated in a PYRO Advanced Microwave Muffle Furnace (Milestone s.r.l., Bergamo, Italy), working at 2.4 GHz. BM samples were put in a quartz crucible and inserted into a refractory small chamber system with the presence of a susceptor (see Fig. 3) [10,13,18]. Preliminary tests were performed to verify the reproducibility and the most suitable conditions to reach the temperature of at least 600 °C, to obtain the reduction of the metals, with a treatment at 1000 W.

In particular, the reproducibility of the treatment hinges on a uniform temperature distribution, which is achieved through consistent MW absorption and the use of a constant mass amount in all the experiments. Indeed, we verified that a slight change in sample mass resulted in a different amount of excited samples, with resulting different temperatures. These data have been recently published [17].

As a consequence, it was crucial to replicate the experiments 10

times to ensure the accuracy and reliability of error evaluation.

In this work, only the results obtained after the treatments at 1000 W for 5 and 10 min are reported.

After MW treatments the samples are subjected to water leaching, essentially to recover Li. Then, the residual mass is treated by acid leaching to recover all the strategic metals. Fig. 1 shows the basic steps of the procedure, with all the experimental conditions. Both leaching steps were carried out in a bath heated to 80 °C for 30 min on a magnetic stirrer set to 300 rpm. The same S:L ratio (40 g sample/L leaching solution) was used. Specifically, acid leaching was performed using 1.2 M of L-malic acid solution and 1.5% H_2O_2 [19].

The MW treatment led to significant mass loss (39–43%). Then, water leaching and acid leaching of the residuals accounted for mass reductions of 14–15% and 76% respectively, culminating in a total mass decrease of about 88% by the end of the process for both samples (treated for 5 and 10 min).

Table 1 shows the NMC sample composition, evaluated after sample digestion. It also reports the composition of the BM, after the 10 min of MW treatment, to evaluate the eventual loss of metals due to the annealing. The table shows that the variation in metal composition after MW treatment is limited, and within the uncertainty associated with the measurement results.

Indeed, the BM weight loss occurring till about 570 °C is ascribed to the volatilization of organic matter, such as some residual plastics and the organic binder (PVDF) [20]. After this temperature, the reaction of carbon and oxygen occurs.

It can be inferred, by the analysis of data reported in Table 1, that the main component of the active cathode is $LiNi_{0.6}Co_{0.2}Mn_{0.2}O_2$ (also known as NMC622). Some small percentages of Al and Cu were present, derived from anode and cathode collectors. These metals are not the target of the present recovery process, because they are supposed to be recovered by pre-treatments.

3. Results and discussion

Fig. 2 shows the comparison between XRD patterns of the as-received NMC622-BM (a) and the samples treated at 1000 W for 5 (b) and 10 (c) min, respectively.

The peaks at 18° and 44° are typical of $LiNi_xCo_yMn_zO_2$ phases. The graphite main peak can be found at about 26.5°. The $LiNi_xCo_yMn_zO_2$ and graphite peaks decrease after both treatments and new peaks appear, indicating that some reactions occurred. The newly detected phases are NiO, Co_3O_4, Li_2O, MnO, and Ni. The XRD pattern of the sample treated for only 5 min (b), is quite like that treated in 10 min (c). However, the relative intensities of the graphite and $LiNi_xCo_yMn_zO_2$ peaks, in comparison for example to the main NiO peak are higher, due to the less

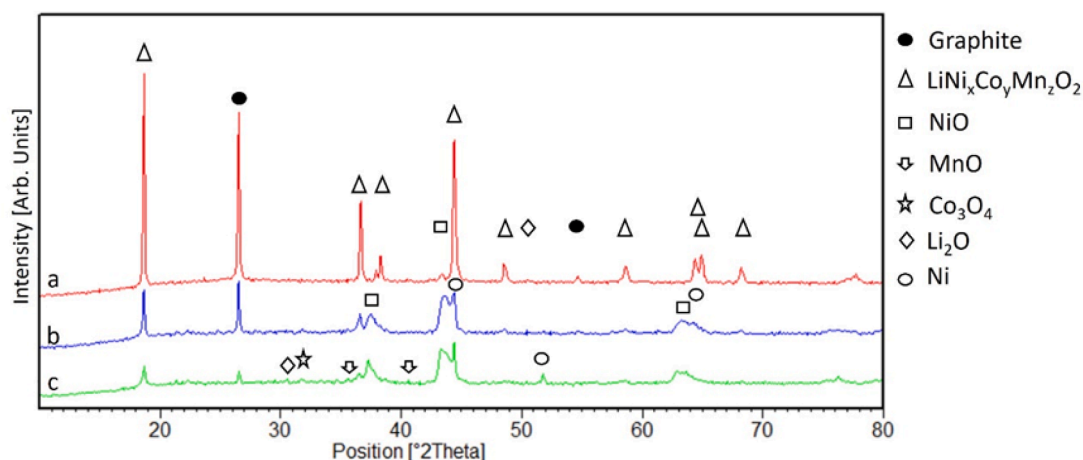**Fig. 2.** XRD patterns of a) as-received NMC black mass sample, b) sample treated at 1000 W for 5 min, and c) sample treated at 1000 W for 10 min.
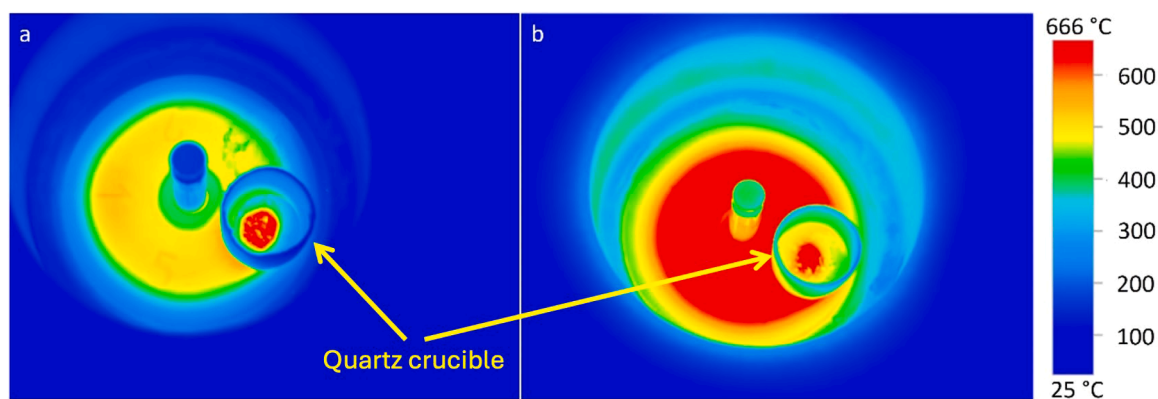
Fig. 3. Thermal imaging camera images, showing the chamber containing the samples (with the susceptor located on the basis, under the crucibles) a) after the treatments at 1000 W 5 min; b) after the treatment at 1000 W 10 min. The lower temperatures are associated with the isolating system, outside of the oven camera. Samples are inserted in a quartz crucible, and they are visible because their temperature is higher than 650 °C. It is very interesting to notice that for the sample treated for 5 min, the final reached temperature is comparable to that of the sample treated for 10 min, due to the MW-induced carbon excitation.

Table 2
Concentration of the recovered metals, after the two leaching steps (in water and L-malic acid) evaluated for the samples treated for 5 and 10 min at 1000 W, with corresponding recovery (%). The compositions were calculated by considering the weight loss (due to the organic part incineration and mass reduction due to leaching), to refer to the original BM composition. The concentrations were evaluated by ICP-MS. For comparison, the results obtained after the same leaching steps on the BM, with no MW treatment are also reported.

| Element | 5 min of BM treatment | | | | | | | | | | | |
	mass recovered after water leaching (g/kg BM)			mass recovered after water and L-malic acid leaching (g/kg BM)			TOTAL (g/kg BM)			Recovery %		
Li	15.8	±	0.8	31.6	±	0.8	47.5	±	1.6	99.5	±	6.0
Mn	0.0	±	0.0	62.7	±	1.6	62.7	±	1.6	88.7	±	5.6
Co	0.0	±	0.0	47.3	±	1.2	47.3	±	1.2	65.5	±	5.6
Ni	0.0	±	0.0	139	±	4	139	±	4	60.6	±	5.6
Cu	0.00	±	0.00	11.6	±	0.3	11.6	±	0.3	58.4	±	5.7
Al	0.040	±	0.003	4.6	±	0.1	4.64	±	0.12	51.7	±	6.1

| Element | 10 min of BM treatment | | | | | | | | | | | |
	mass recovered after water leaching (g/kg BM)			mass recovered after water and L-malic acid leaching (g/kg BM)			TOTAL (g/kg BM)			Recovery %		
Li	17.8	±	0.9	25.5	±	1.3	43.4	±	2.2	90.9	±	7.1
Mn	0.0014	±	0.0001	62	±	3	62	±	3	87.1	±	7.1
Co	0.0028	±	0.0001	59	±	3	59	±	3	82.1	±	7.1
Ni	0.0	±	0.0	164	±	8	164	±	8	72.0	±	7.1
Cu	0.053	±	0.003	14.5	±	0.7	14.6	±	0.7	73.3	±	6.9
Al	1.7	±	0.1	6.2	±	0.3	7.9	±	0.4	86.6	±	7.4

| Element | no MW treatment | | | | | | | | | | | |
	mass recovered after water leaching (g/kg BM)			mass recovered after water and L-malic acid leaching (g/kg BM)			TOTAL (g/kg BM)			Recovery %		
Li	5.6	±	0.3	0.77	±	0.04	6.39	±	0.32	13.4	±	7.1
Mn	0.025	±	0.001	1.2	±	0.1	1.3	±	0.1	1.8	±	7.1
Co	0.018	±	0.001	1.2	±	0.1	1.2	±	0.1	1.7	±	7.1
Ni	0.035	±	0.002	3.8	±	0.2	3.9	±	0.2	1.7	±	7.1
Cu	0.300	±	0.017	0.30	±	0.01	0.60	±	0.03	3.0	±	7.2
Al	0.300	±	0.015	0.033	±	0.002	0.333	±	0.017	3.6	±	7.4

complete reactions, that better occurred for the sample treated for 10 min.

The LiNi$_x$Co$_y$Mn$_z$O$_2$ can be decomposed into stable NiO, CoO, Li$_2$O, and MnO phases at high temperatures, in accordance with the Ellingham diagrams [21]. The presence of carbon (graphite) enables carbothermic reductions, which are well-known in the literature [3], with a decrease in the theoretical decomposition temperature. Fig. 3 shows the temperatures reached by the treated samples, after the MW treatments. The result is that both 5 and 10-minute treatments allow the sample to reach temperatures higher than 650 °C at the end of the process (to induce the reactions a temperature greater than 570 °C is necessary). This is because the efficiency of MW heating in carbon-based materials is particularly high because of their strong ability to convert electromagnetic energy into thermal energy [22]. In addition, Fig. 3 shows that for

the sample treated for 5 min, the final reached temperature is comparable to that of the sample treated for 10 min, and higher to that of the surrounding materials in the camera, due to the MW-induced carbon excitation.

However, the most interesting result reported in Fig. 3a is the evidence that the sample locally reached temperatures higher than that of the susceptor, indicating that the graphite contribution is essential not only for the carbothermic reduction but also for heating the sample, which leads to rapid heating of BM particles. Indeed, MW radiation is particularly effective at exciting carbon, primarily due to carbon's excellent MW absorption capabilities. This is an advantage of a MW treatment of a carbon-based material because only a few minutes are necessary to reach the reaction conditions. Water leaching recovers more than 53% of Li, in the sample treated for 5 min at 1000 W. In
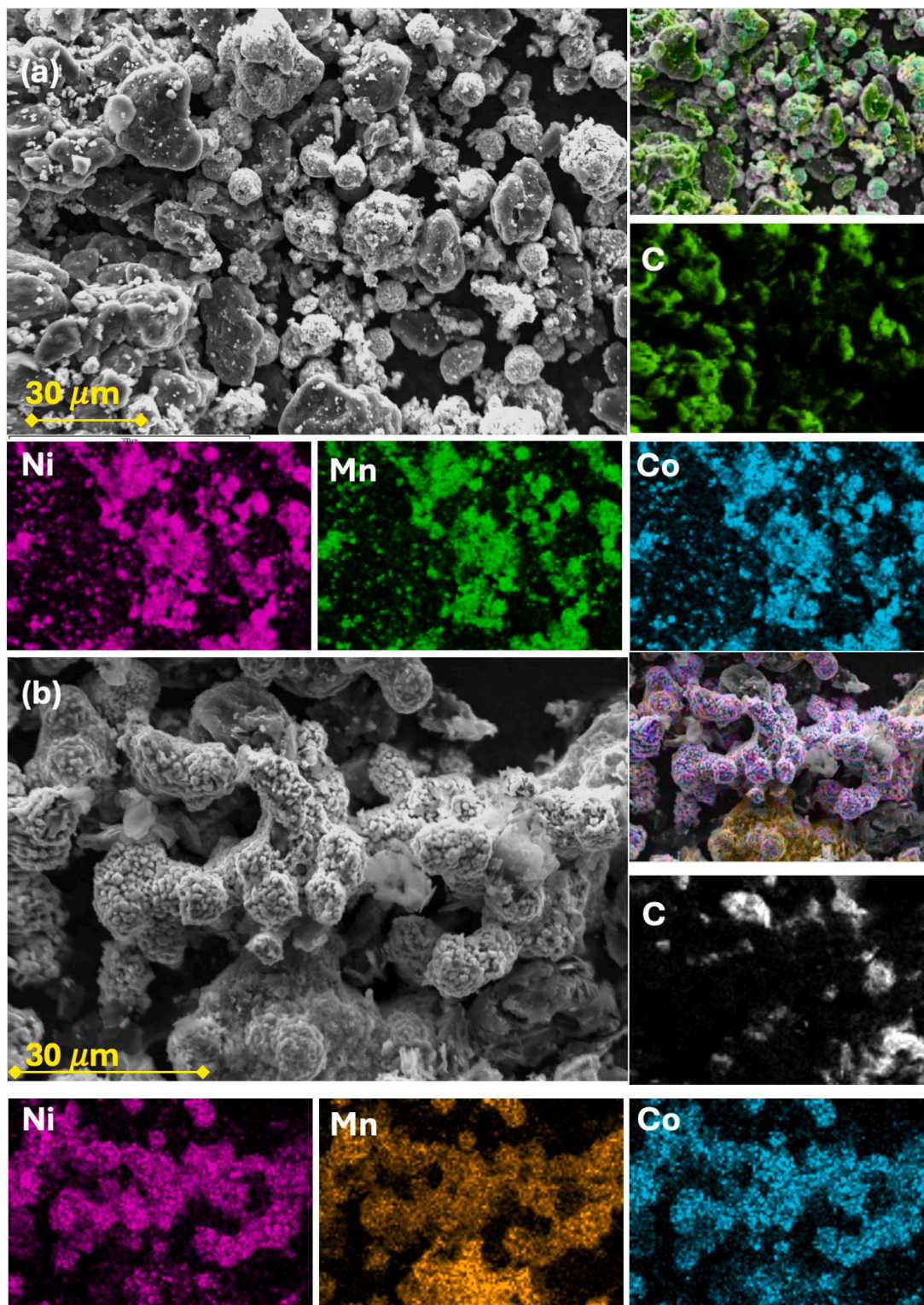
Fig. 4. The scanning electron microscopy (SEM) analysis, with the associated energy-dispersive X-ray spectroscopy (EDS) mapping, of the BM (a), and the BM after a MW treatment of 10 min (b). SEM images (15 kV) were performed at a) 4k and b) 3.5k magnification.

Table 2 the total recovered metals concentrations in the solutions, after water and acid leaching tests, and after the MW treatments for both samples treated for 5 and 10 min are reported. Based on these results, it was possible to evaluate the percentage of the metals recovery, considering the mass balance due to the volatilization of carbon (in the form of carbon dioxide), and other residual components (residual plastics, binder, and electrolyte, for example, during the thermal treatment)

[18]. As a comparison, Table 2 also reports the metal recovery after the same leaching steps (with water and L-malic acid, but no MW treatments), on the as-received BM, as a control experiment.

Fig. 4a shows that the BM particles do not form large agglomerates, with dimensions generally falling in the range of 5–30 μm. Their surface appears quite smooth. The chemical analysis (EDS) highlights that Mn, Co, and Ni can be detected in the same positions, corresponding to the
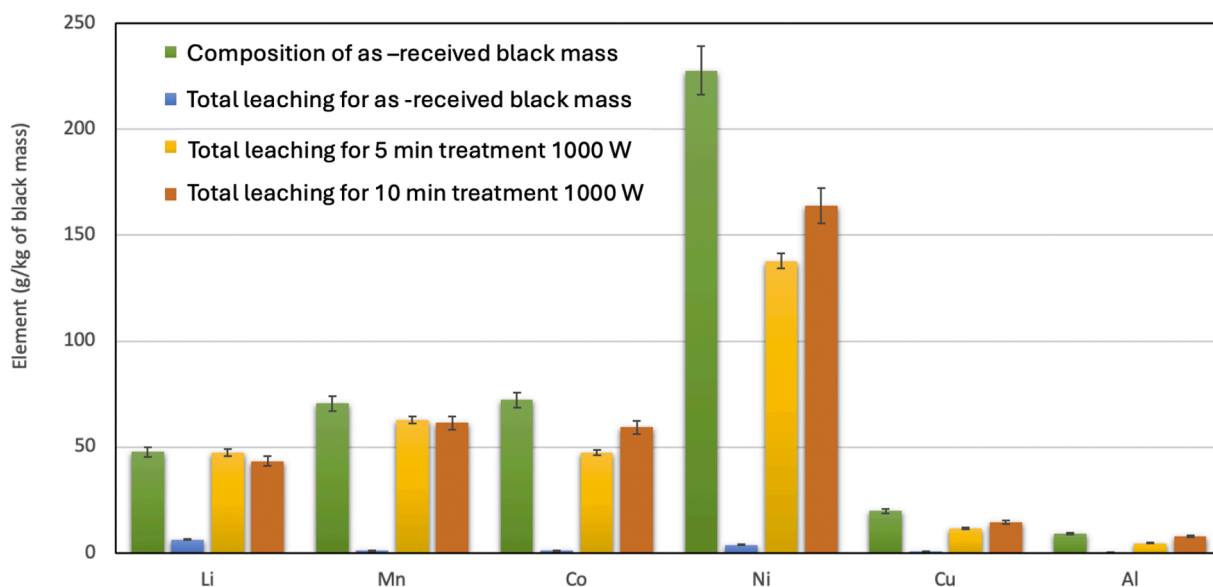
Fig. 5. Mass of Li, Mn, Co, Ni, Cu, and Al expressed in g of the element/kg of black mass, after water and L-malic acid leaching for black mass treated at 1000 W for 5 min (yellow) and after water and L-malic acid leaching for black mass treated at 1000 W for 10 min (orange). The composition of the as-received sample is reported in green. The data for water and L-malic acid leaching for black mass without any MW treatment are also reported as a control experiment. In this case, the metal recovery is quite limited.
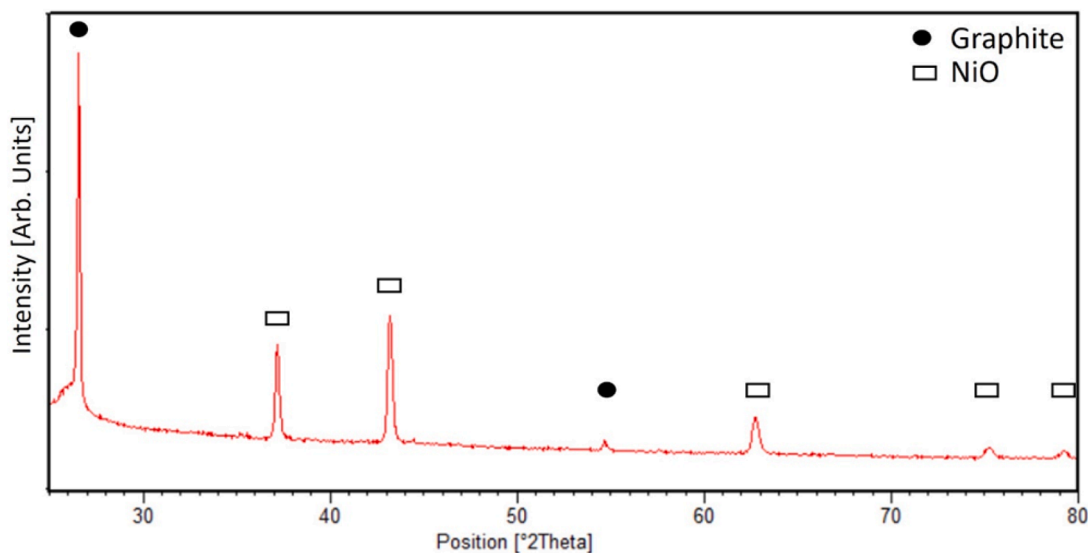

Fig. 6. XRD pattern of the NMC BM residual collected on the filter, after the treatment at 1000 W 10 min, and water and acid leaching.

same phase. The MW treatment of BM results in significant changes in particle morphology and structure. After the MW treatment (Fig. 4b), SEM images show that a mix of small (few μm) and large aggregates can be found. In particular, the MW-treated BM shows a significant aggregation of low dimensions particles, forming larger and more complex structures, in comparison to as received BM. In this case, the EDS mapping highlights that Mn distribution does not correspond in the bottom part with the Co and Ni location, showing that it can be found in a separate oxide phase, in accordance with the XRD results (see Fig. 2). The treated sample exhibits increased aggregation, reduced particle size uniformity, and a rougher surface texture, which are indicative of the phase transformations that occur during the process.

The recovery of Li is higher than 90% for both treatments. More than 82% Co recovery can be obtained after 10 min of MW treatment. Ni results in lower recovery, even if Ni may be also recycled from the residual on the filter. Very good results were obtained for Mn (see also

Fig. 5), reaching a recovery amount of about 87–89%.

This corresponds to an improvement in comparison to the literature, reporting that Mn remains incompletely reduced during NMC carbothermic reactions and exists in the forms of +3 or +4 valence states in the final product, resulting in a lower Mn leaching efficiency in comparison to Li, Ni, and Co [23]. In the present case, Mn is available as MnO, after MW treatment, as shown in the XRD pattern of Fig. 2. The comparative results found for both treatments can be justified considering that the temperature for reactions was attained in the two cases (considering 5 and 10 min), as shown in Fig. 3.

This is the first time that carbothermic reduction was observed for MW-treated NMC-BM. Indeed, the literature reports the possibility of having carbothermic reductions for this kind of BM, but in the vacuum or inert atmosphere [19,24] and with prolonged exposition to high temperatures. In particular, similar results are reported for NMC622, but they were obtained after conventional treatments requiring much more

Table 3

Mass balance evaluation for the sample treated 10 min at 1000 W: the concentration of the metals in the recovered solutions and on the residual allows to verify that the global amount corresponds with the original BM composition. The compositions were calculated considering the weight loss (due to the organic part incineration and mass reduction due to leaching), to refer to the original BM composition. The concentrations were evaluated by ICP-MS and all the data are available in the Supporting Table S2.

Element	Total recovered metals (g/kg BM)			Residual on the filter (g/kg BM)			TOTAL (recovered + residual on filter) (g/kg BM)		
Li	43.4	±	2.2	0.6	±	0.1	44	±	2.3
Al	7.9	±	0.4	0.7	±	0.1	7.9	±	0.5
Mn	62	±	3	3.7	±	0.4	65.7	±	3.4
Co	59	±	3	8.4	±	0.9	67.7	±	3.9
Ni	164	±	8	49	±	5	213	±	13
Cu	14.6	±	0.7	7.9	±	0.8	14.6	±	1.5

time [23]. Generally, at least 1 h is required for efficient carbothermic reduction of NMC batteries. Fig. 6 shows the XRD pattern of the BM residual, collected on the filter, after water and acid leaching for the sample treated for 10 min in the MW oven.

In this case, it appears that the only detectable crystalline phases are graphite and NiO. This agrees with the chemical analysis, indicating that almost all Co, Li, and Mn can be detected in the leached solution (see Supporting Table S2). On the contrary, some amount of Ni is still in the insoluble fraction, in the nickel oxide form, with residual graphite. In particular, the residual, recovered on the filter, contains a concentration of about 48.9 g Ni/kg of BM, corresponding to about 22% of the initial Ni amount (see Table 3). This justifies the lower amount of Ni recovered by leaching. Moreover, the residual Ni may be recovered, for example, by graphite separation by floatation. This may also allow to recovery of residual graphite that was not necessary for the reaction.

Table 3 shows the mass balance evaluated taking into account the metals recovery after the whole process, and considering the residual on the filter, for the sample treated for 10 min. It shows the results of the chemical tests, verifying that the total amount of the critical metals (also considering the residual of Ni on the filter) can be found in the recovered samples, in accordance with the XRD data.

In summary, throughout this work, we demonstrated for the first time the effectiveness of MW for carbothermic reduction of NMC batteries, making a treatment of a few minutes.

However, some challenges are still open, to optimize the recovery conditions, also depending on the spent batteries' chemistry. Indeed, despite that in the present work it is shown that treatment of a few minutes can allow proceeding with a weak acid to recover the starting metals in spent NMC622 batteries, the process optimization can still be realized, as the decrease of the MW power and the increase of the treatment time. Future studies may be also devoted to different cathode chemistry, with great attention not only to optimizing the energy demand but also to increasing the availability of the water-soluble Li_2O phase. Another interesting optimization possibility concerns the use of food wastes to produce weak acids, for example, L-malic acid. This may allow to obtain a process based on the use of all waste materials, and whiteout any addition of chemicals.

4. Conclusion

This paper introduces an innovation in the integration of thermo-chemical processes with MW, marking the proof of concept of the capability to recover strategic metals from an NMC622 BM. It holds the potential to significantly decrease energy consumption compared to traditional thermal treatments, offering a viable approach for recovering metals from spent NMC-Li batteries. The utilization of MW presents two primary advantages: a) conservation of energy typically required to heat the entire chamber (as only the sample is heated), and b) a faster and

more energy-efficient heating process compared to conventional methods. MW heating emerges as a promising, eco-friendly, key enabling technology, aligning with the prevailing trend toward sustainable development. The spotlight on this application gives an overview of the possible progress in LIBs recovery offered by MW, suggesting more research endeavors in this important field. We are currently investigating the conditions contributing to support the technology TRL increase, intending to reach its integration into a LIB industrial recovery facility, to boost the scalable recovery of critical raw materials. This is particularly advantageous for applications in energy sectors to support energy transition.

Supporting Information

S1: Calibrations curves for the ICP.

Table S2: Concentrations of metals in water and L-malic acid leaching for as-received black mass, black mass treated at 1000 W for 5 min, and black mass treated at 1000 W for 10 min evaluated by ICP-MS, expressed as g/L.

CRediT authorship contribution statement

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The technology used in this work was patented (PCT/IB2023/051034).

Data availability

Additional data are available in the Supporting information.

Acknowledgments

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ensm.2024.103703.

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