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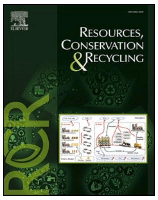

Full length article

Recycling spent lithium ion batteries by flash joule heating: preferential lithium recovery and Li-phase conversion mechanism under ultra-temperature

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ABSTRACT

The recovery of spent lithium-ion batteries is crucial for sustainable renewable resources and environmental protection. Low lithium recovery efficiency and high energy consumption are the main problems in current recycling technologies for black mass of spent NCM batteries. This study innovatively applies flash joule heating (FJH) to preferentially recover lithium from industrial raw materials. We firstly revealed the Li-phase conversion behavior under nonequilibrium high temperature state. At the optimal FJH condition (1400 °C, 90 s), main Li-phases convert into Li_2O and Li_5AlO_4, while the transition metals reduce to elemental forms and low-valence oxides. The Li-phase conversion behavior and FJH characteristics matched excellently, enabling 91.8 % selectively lithium extraction by water. Ni, Co, and Mn are recovered via sulfuric acid leaching with over 98 % efficiencies. This innovative method offers a new pathway for recycling spent LIBs in both theory and technology, and extremely appealing for its energy conservation and high recovery efficiency.

1. Introduction

Lithium-ion batteries (LIBs) have been widely applied in various fields due to their durability, rapid charging, and high energy density (Ciez and Whitacre, 2019; Liu et al., 2023; Li et al., 2011). Driven by the pursuit of carbon neutrality, the surge in the use of LIBs has brought a scarcity of strategic metal resources, such as lithium, cobalt and nickel (Nykvist et al., 2019; Lai et al., 2021; Fan et al., 2020). In the meantime, the average lifespan of these batteries is limited to 8 − 10 years, leading to a rapid increase in the number of spent LIBs (Swain, 2017; Chen et al., 2019). The proper disposal of spent LIBs not only mitigates environmental pollution, but also enables the recycling of valuable metals, which can be reinstated as raw materials for new battery production (Meshram et al., 2014; Xu et al., 2008). Therefore, the recovery of spent LIBs has become a significant issue of global concern.

Three main recycling approaches have been employed to recover the spent ternary LIBs, including pyrometallurgical (Makuza et al., 2021; Holzer et al., 2023; Rinne et al., 2022; Hu et al., 2021; Zhou et al., 2021), hydrometallurgical (Zhu et al., 2022; Li et al., 2017; Wang et al., 2020; Jo and Myung, 2019), and pyro-hydro combination technologies (Su et al., 2023; Yang et al., 2021; Zhao et al., 2020; Huang et al., 2022; He et al., 2023). The pyrometallurgical process is characterized by the direct application of high temperature, which promotes the separation of slag phase and molten metal. However, lithium tends to enter into the slag phase and is difficult to recover (Georgi-Maschler et al., 2012). In addition, this process is highly energy-intensive (Xu et al., 2022). In the hydrometallurgical process, strong acids and reducing agents are commonly employed in industry to leach metals from cathode materials. However, the leaching process lacks selectivity for metals, resulting in the intricate separation and purification steps, and the generation of substantial wastewater which is difficult to manage properly (Lv et al., 2018). The pyro-hydro combined method is currently the most widely used in industrial applications. In the typical carbon thermal reduction process, the mixture of cathode and anode active material (black mass) is usually first roasted in the temperature of 700−750 °C to dissociate and change the phase state, so that priority lithium extraction and efficient leaching of Ni, Co, Mn could be achieved in the water-leaching and subsequent acid-leaching processes respectively. However, the presence of Al in the raw materials can result in the generation of water-insoluble $LiAlO_2$ during roasting period, which will reduce the

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lithium recovery rate (Ding et al., 2023). To solve this problem, Yang et. al used pre-alkali leaching method to achieve preferentially remove of Al foil in spent NCM black mass, and the Li recovery rate increases from 70 % to 88 % (Yang et al., 2021). However, the priority pre-alkali leaching of Al leads to the lithium loss, and the $NaAlO_2$ leachate is difficult to treat. Additionally, the carbon thermal reduction process still requires a considerable input of energy. Hence, it is urgent to explore an effective method for recycling spent LIBs with less energy consumption and higher lithium recovery rate.

Currently, the electrical heating techniques including flash joule heating and carbothermal shock have attracted significant interest, owing to their ultrafast high temperature, high heating rates, and low energy consumption (Wang et al., 2020; Deng et al., 2022). In the domain of LIBs recycling, the FJH process was mainly investigated for the separation and regeneration of cathode or anode active materials from spent lithium batteries (Yin et al., 2023; Zheng et al., 2023; Li et al., 2023; Chen et al., 2023; Zhang et al., 2023; Li et al., 2024; Ji et al., 2024). Except that, the FJH strategy was employed to heat the black mass, and high recovery efficiencies of Li, Ni, Co and Mn were achieved by the followed HCl leaching. However, lithium is difficult to efficiently recover from this metals-mixed solution (Chen et al., 2023). Zhu et.al developed a carbothermal shock strategy to treat NCM cathode materials which are preferentially separated from the Al foils by manual disassembly, and 90.8 % of Li was extracted in the subsequently water leaching (Zhu et al., 2023). However, aluminum was absent in the manual dismantled cathode material, so the feasibility of this method for the more common black mass and particularly the effect of aluminum on the lithium recycling efficiency are still questionable.

In this study, the FJH equipment was firstly employed to treat black mass of spent LIBs. The various valuable metals were then recovered by selective water leaching and acid leaching. A comprehensive investigation was designed to analyze the impacts of flash joule heating conditions on lithium recovery rate, including temperature and time. And the phase transition of flash heating process was elucidated by comparative experiments, thermodynamic analysis and various characterizations, with a particular focus on the aluminum-lithium phases. This method provides an innovative strategy for the rapid, energy efficient and effective recovery of spent LIBs.

2. Experimental

2.1. Materials

The black mass used in this work was provided by a battery recycling company. This material was sourced from spent NCM batteries through discharge, disassembly and crushing. Fig. S1 displays the XRD pattern of the black mass, indicating that its main components are $LiNi_xCo_yMn_zO_2$ and graphite. The aqua regia solution (HCl: HNO_3=3:1) was used to dissolve the black mass, and the composition is presented in Table S1. The all chemicals employed in this study were of analytical grade, and all solutions were prepared by using ultrapure water.

2.2. Experiment procedure

2.2.1. Flash joule heating reduction

To examine the influence of various FJH conditions on the phase transformation of spent NCM materials and Li recovery efficiency, we first loaded 1.5 g of black mass into a quartz tube lined with graphite paper which can achieve uniform heating. The rapid heating reduction experiments were performed in the FJH equipment (FJH-2023A, SAIYIN, China). FJH conditions such as temperature (1000−2000 °C) and time (30−120 s) were investigated systematically to determine the optimal parameters.

2.2.2. Water leaching

After FJH process, to extract lithium from FJH products, the product was mixed with water in a beaker placed on a magnetic stirrer, the experimental conditions are room temperature and liquid/solid ratio of 50 mL/g for 3 h. The obtained suspension was filtered, and the leaching residue was dried at 80 °C. The concentrations of different elements in the leachate and residue were analyzed by ICP-OES. The leaching rate of different metals can be calculated by:

$$\eta = \left(\frac{c_i V}{c_i V + m w_i}\right) \times 100\% \tag{1}$$

the V and c_i in this formula are the volume of leaching solution and the concentration of element “i”, respectively, and m and w_i are the mass of leaching residue and the weight content of element “i”, respectively.

2.2.3. Acid leaching

The Mn, Ni and Co retained in water-leaching residue were leached through H_2SO_4 solution. And the contents of Ni, Co and Mn in acid leaching solution were also measured by ICP-OES. The leaching rate can be calculated as follows:

$$\eta = \left(\frac{c_i V}{m w_i}\right) \times 100\% \tag{2}$$

where m and w_i are the mass of water-leaching residue and the weight content of element “i”, respectively, and c_i and V are the concentration of element “i” and the volume of leachate, respectively

2.3. Characterization

The concentration of various metals in solution was measured through inductively coupled plasma optical emission spectrometer (ICP-OES). The carbon content of black mass was analyzed by carbon and sulfur analyzer (EMIA-820 V, Horiba, Japan). X-ray diffractometer (XRD, D8 ADVANCE, BRUKER, Germany) was applied to characterize the phase compositions of solid samples. The X-ray photoelectron spectroscopy (XPS, Thermo escalab 250Xi, Thermo Fisher, America) was used to analyze the elemental valence states in solid powders. Scanning electron microscope (SEM, SUPRA55, ZEISS, Germany) with an energy dispersive Xray spectroscopy (EDS, SUPRA55, ZEISS, Germany), and time of flight secondary ion mass spectrometry (TOF-SIMS, S9000X, TESCAN, Czechia) were applied together to characterize microscopic morphology and elemental distribution of all samples.

3. Results and discussion

3.1. FJH reduction roasting

3.1.1. Thermodynamic analysis

Thermodynamic calculation was used to investigate the theoretical phase compositions in the temperature range of FJH process. Here, we used the “Equilib-module” of FactSage 8.3 software with the database FToxid and FactPS calculate the equilibrium contents of the possible products. Fig. 1(a) shows the Li-related phases in the roasted products at different temperatures. As observed, the main existence forms of lithium in the roasting products include Li_2CO_3, $LiAlO_2$, Li_2O and Li_5AlO_4. At low temperature (<800 °C), the main Li-related phases are Li_2CO_3 and $LiAlO_2$. As the temperature increases, Li_2CO_3 and $LiAlO_2$ are converted to Li_2O and Li_5AlO_4. However, excessive temperatures result in the formation of gaseous lithium, which leads to the loss of lithium and is not conducive to lithium recovery.

Due to the fact that $LiAlO_2$ is insoluble in water while Li_5AlO_4 exhibits solubility, what phase Li exists in has a significant impact on the water leaching rate. Fig. 1(b) shows the delta G-T relationship of the chemical reactions that may result in the formation of lithium aluminates. As illustrated, the generation of lithium aluminates is mainly due to the reaction of Li_2CO_3 with Al_2O_3 during the roasting process. Specifically, the generation of $LiAlO_2$ can only occur at temperatures above

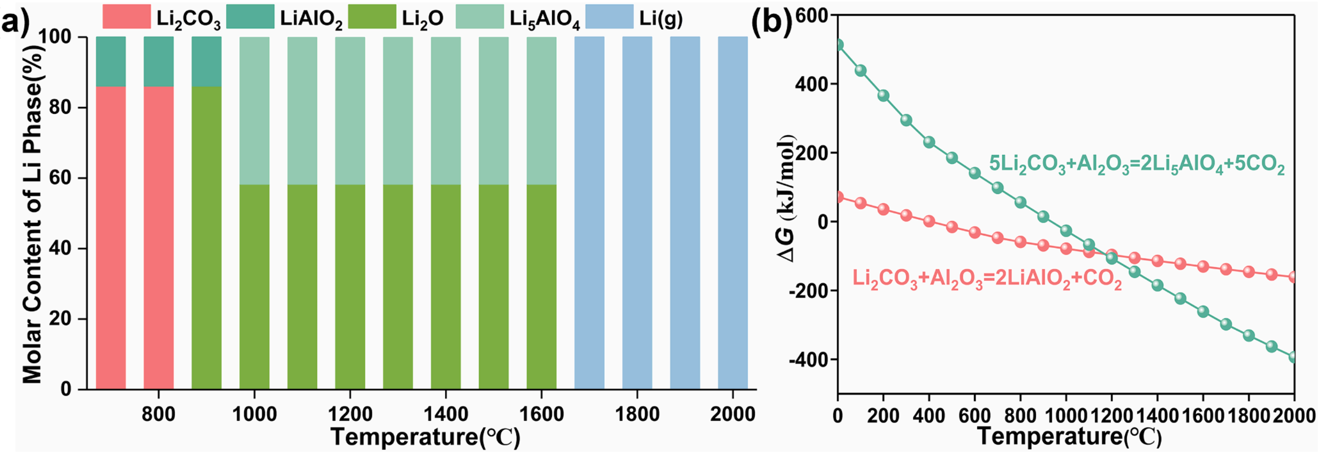

Fig. 1. (a) Calculated phase distribution of reaction products after reduction roasting (initial molar ratio of reactants: $n(Li_2O)$: $n(NiO)$: $n(CoO)$: $n(MnO_2)$: $n(Al)$: $n(C)$ = 0.24: 0.36: 0.06: 0.038: 0.04: 1.8; constant value: pO_2 (Pa) = 10^{-20}); (b) Ellingham diagrams for possible reactions to generate different lithium aluminates.

500 °C, while Li_5AlO_4 requires higher temperatures (>970 °C); As the temperature rises, the Gibbs free energy for the reaction to form Li_5AlO_4 displays increasingly negative values, suggesting that a portion of the Li_2CO_3 is more prone to convert to Li_5AlO_4.

Thermodynamic calculations indicate that above 1100 °C, water-insoluble $LiAlO_2$ can be converted into water-soluble Li_5AlO_4. Therefore, increasing the temperature of reduction conversion process has the potential to address the challenge of low lithium recovery caused by $LiAlO_2$. However, excessive temperatures will lead to significant lithium volatilization loss. The FJH method has been demonstrated to rapidly elevate temperatures to over 1000 °C and complete high-temperature reactions in an ultra-shot period of time. This technological characteristic is well-suited to facilitate the conversion of Li_5AlO_4, as well as to avoid the loss of lithium.

3.1.2. *Effects of FJH temperature and time*

Based on the results of thermodynamic analysis, a higher heating temperature was selected and the FJH method was adopted instead of the conventional muffle furnace heating. The effects of FJH temperature and time on Li leaching rate and the phase morphology of the product were systematically investigated. Fig. S2 shows the real-time temperature measurement under various conditions. The material rapidly heated to high temperature within approximately 25 s, which is hundred-folds faster than that of conventional heating methods.

As present in Fig. 2(b), the diffraction peak of $LiNi_xCo_yMn_zO_2$ remains detectable at 1000 °C, indicating incomplete dissociative reduction during the FJH process at this temperature. This corresponds to a lithium leaching rate of only 75.1 % (Fig. 2a). When the temperature increases, the diffraction peak of $LiNi_xCo_yMn_zO_2$ completely disappears

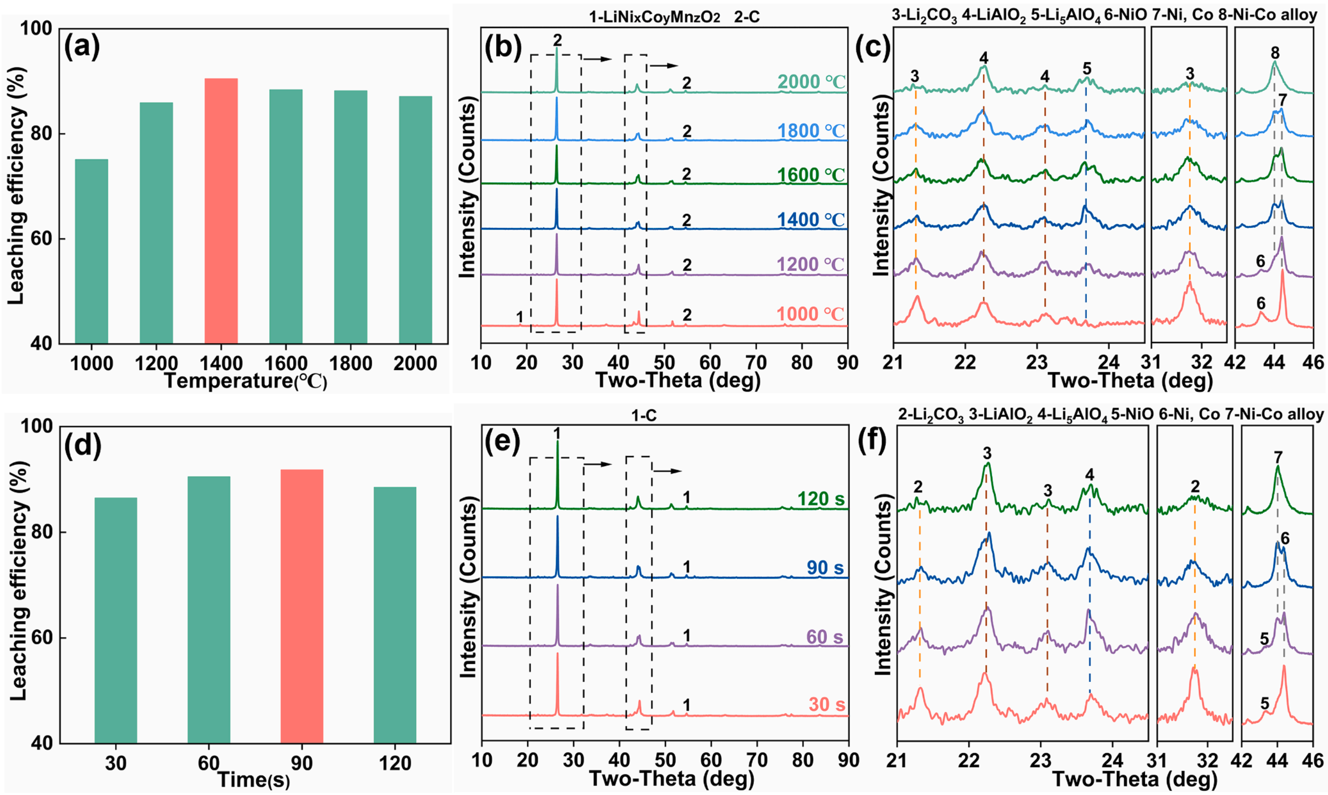

Fig. 2. Water leaching rate of lithium and XRD patterns of FJH products: ($a-c$) effect of FJH temperature (t = 60 s), and ($d-f$) effect of FJH time (T = 1400 °C).

and optimal lithium leaching rate of 90.5 % is achieved at 1400 °C. The partial enlargement of XRD (Fig. 2c) indicates that as the temperature increases, the diffraction peaks of Li_2CO_3 weaken gradually, while the diffraction peaks of Li_5AlO_4 have a gradual enhancement. This phenomenon may be attributed to the fact that under high temperature, the thermal decomposition of Li_2CO_3 is severe, and the increase in its product Li_2O promotes the formation of soluble lithium salts Li_5AlO_4 rather than $LiAlO_2$, which corresponds to an increase in the lithium leaching rate. However, further increase in temperature from 1600 °C to 2000 °C leads to the decrease in intensity of Li_5AlO_4 peak, which is due to the generation of gaseous Li phase. This is the reason for the decrease in the lithium leaching rate. The physical phases of Ni and Co are also further changed with the increase of flash heating temperature (Fig. 2c). The diffraction peaks of NiO are gradually disappeared at 1400 °C, indicating that the metals have been completely reduced to the lower valence state under this condition. Subsequently, the diffraction peak corresponds to the Ni and Co crystallographic planes (111) exhibits a split in the lower angle region. This phenomenon is indicative of the generation of Ni-Co alloy under elevated temperatures (You et al., 2012). And as the temperature rises, the diffraction peaks of alloy gradually intensify until completely convert. Additionally, the diffraction peaks for Li_2O and MnO are weak or even absent, which is primarily

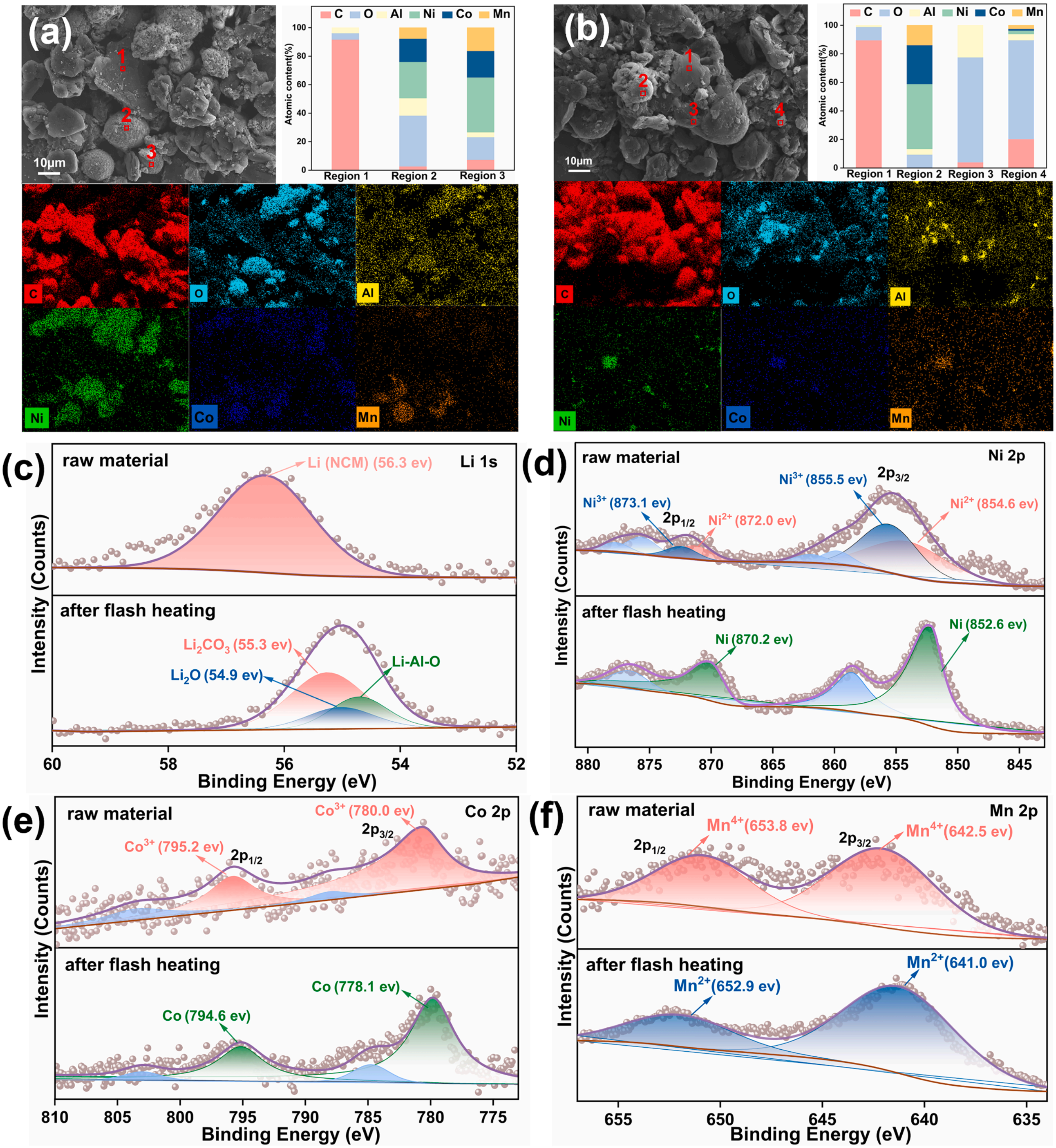

Fig. 3. (a, b) SEM-EDS analysis of the samples before and after FJH process; (*c* − *f*) XPS spectra of raw material and FJH product.

attributable to the low content of raw materials and its poor crystallinity (Zhang et al., 2023).

Fig. 2($d - f$) shows the leaching rate of lithium extracted by water leaching and the phase morphology of the products at different flash heating times. The leaching rate of lithium exhibits an increase followed by a subsequent decrease in accordance with the prolongation of flash reduction time (Fig. 2d). However, specifically, the flash reduction time has a less pronounced effect on the leaching rate of lithium when the temperature is 1400 °C. XRD partial enlargement demonstrates that with the increase of flash heating time, more Li_2CO_3 decomposes due to high temperature, and the diffraction peaks corresponding to it gradually weaken, while the diffraction peaks of Li_5AlO_4 gradually strengthen (Fig. 2f). These phenomena suggest that the extension of FJH time promotes the Li_2CO_3 convert to Li_5AlO_4 rather than $LiAlO_2$. However, it is recommended that the FJH time should not be excessively prolonged, as this may result in a greater loss of lithium due to volatilization, which will decrease the leaching rate of lithium. In summary, a high lithium recovery efficiency of 91.8 % can be achieved within only 90 s by the FJH method.

3.1.3. *Phase transition mechanism*

Fig. 3(a, b) and Table S2 display the SEM-EDS analysis results of initial black mass and FJH product. Prior to the FJH process, the cathode material particles exhibit a more regular spherical shape, with all valuable metals distributed evenly across the particle surfaces. Moreover, the particles that assume an irregular flake are identified as graphite. Based on these characteristics, and with the confirmations provided by EDS and XRD analyses (Fig. S1), the primary constituent of the cathode material is $LiNi_xCo_yMn_zO_2$. After FJH reduction, the metals in raw material tend to be irregularly agglomerated together to form larger ones (Fig. 3b), which was mainly due to a short period of high temperature smelting. To follow up, EDS was employed to quantify the elemental composition of four distinct regions within the SEM image. As can be seen from the results, the C-rich phase with an irregular morphology in region 1 is graphite. Region 2 is primarily enriched in nickel, cobalt, and manganese. It may also contain a mixture of monomeric and low valence oxide forms of metal. In region 3, the oxide of impurity aluminum is present, and the atomic ratio of Al to O is approximately 1: 4, so it is conjectured that this region corresponds to Li_5AlO_4. Region 4 exhibits a phase abundant in carbon and oxygen, where the atomic ratio of C to O is nearly 1: 3, suggesting the presence of Li_2CO_3. These observations align with the XRD analysis outcomes.

Fig. 3($c - f$) shows the surface XPS spectra of the raw material and FJH product. As observed in Li 1 s energy spectrum, the peak at 56.3 eV belongs to the oxidation state of Li^+ disappeared while three peaks emerge at 55.3, 54.9 and 54.76 eV in the FJH product, which is ascribed to Li_2CO_3, Li_2O and lithium aluminum oxide, respectively. (Ota et al., 2004; Dedryvère et al., 2005). In the Ni 2p spectra analysis results, peaks at 873.1 eV and 855.5 eV are indicative of Ni^{3+} states within the Ni $2p_{1/2}$ and Ni $2p_{3/2}$ orbitals (Mansour, 1994). The binding energy peaks at 872.0 eV and 854.6 eV confirm the presence of Ni^{2+} (Shaju et al., 2002). After flash heating process, the binding energy peaks associated with Ni^{3+} and Ni^{2+} disappeared while the peaks belonging to metallic Ni appeared at 870.2 eV and 852.6 eV (Prieto et al., 2012). Fig. 3(e) shows the Co 2p energy spectra, two main peaks at 795.2 eV and 780.0 eV can be clearly detected in the raw material, suggesting that the presence of Co^{3+} (Nithya et al., 2011). For the FJH products, the peaks are appeared at 794.6 eV and 778.1 eV, signifying the presence of metallic Co (Hyman and Vohs, 2011). The Mn 2p energy spectra in Fig. 3f shows that Mn^{4+} (642.5 eV, 653.8 eV) in the black mass is converted to Mn^{2+} (641.0 eV, 652.9 eV) during the FJH reduction (Treuil et al., 1999; Regan et al., 1999). The XPS analysis results verified the XRD and SEM-EDS analysis, which demonstrate that the FJH process achieves complete dissociation and reduction of cathode material.

Fig. 4 illustrates the phase transformation mechanism of spent NCM material in the FJH process deduced by both thermodynamic and characterization analysis. The covalent bond between the metal and oxygen is firstly broken at a low temperature about 400 °C, causing the

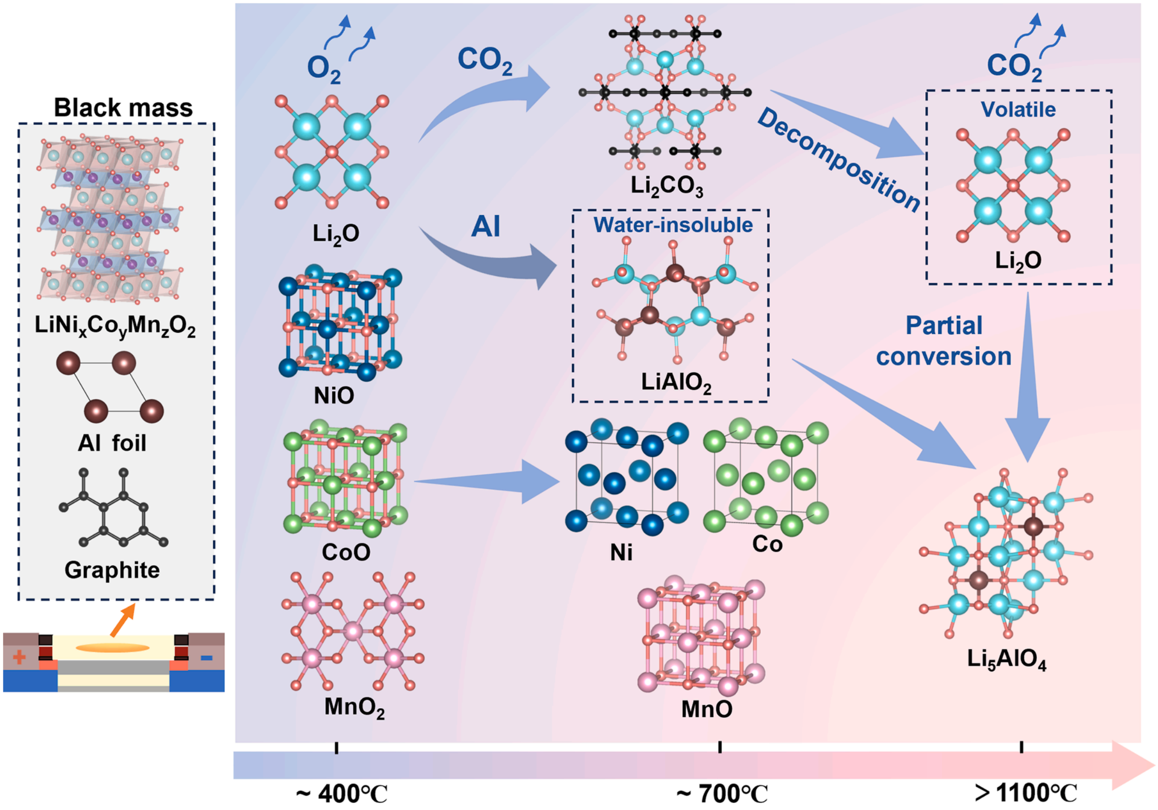

Fig. 4. Phase transformation mechanism of cathode material of ternary LIBs in FJH process.

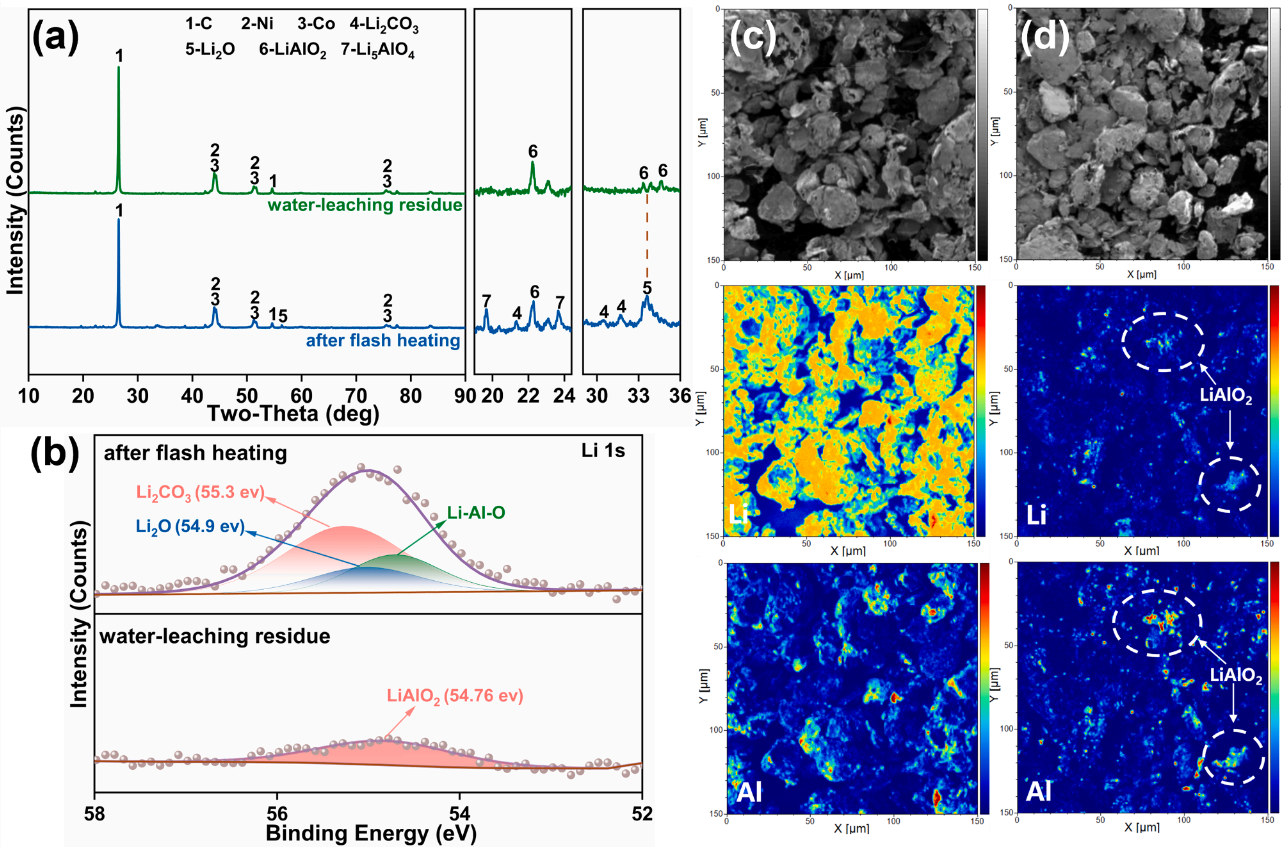

Fig. 5. (a) XRD pattern, (b) XPS analyses and (c) TOF-SIMS mapping of Li, Al elements distribution in the FJH product and water-leaching residue.

decomposition of $LiNi_xCo_yMn_zO_2$ in the black mass and be transformed into lower valence oxides such as Li_2O, NiO, CoO and MnO_2, releasing O_2 (Eq. (3)) (Makuza et al., 2021). With the increases in temperature and time, the graphite reacts with escaping O atoms to generate CO_2.

$$LiNi_xCo_yMn_zO_2 \xrightarrow{\sim 400\ ^\circ C} Li_2O + NiO + CoO + MnO_2 + O_2(g) \quad (3)$$

$$Li_2O + NiO + CoO + MnO_2 + C + Al + O_2(g) \xrightarrow{\sim 700\ ^\circ C} Li_2CO_3 + LiAlO_2 + Ni + Co + MnO + CO_2(g) \quad (4)$$

Meanwhile, these transition metal oxides are further reduced by graphite, resulting in the formation Ni, Co and MnO at about 700 °C (Rostami et al., 2022). Li_2O is mainly combined with CO_2 and impurity Al to form Li_2CO_3 and $LiAlO_2$ in this stage (Eq. (4)). In the ultra-temperature stage of FJH process (>1100 °C), Li_2CO_3 is completely decomposed to form Li_2O and CO_2 is released (Zhou et al., 2025), portion of Li_2O will combine with $LiAlO_2$ to form Li_5AlO_4 under high temperature conditions (Eq. (5)). The lithium salts generated at this stage are all soluble, which can be extracted by selective water leaching. Besides, the saturation vapor pressures of Li_2O were higher than those of the TM oxides, indicating that Li has a greater tendency than TM oxides to volatilize at high temperatures. However, due to the ultrafast heating/cooling rate and short reaction duration of the FJH method, the volatilization loss of Li can be greatly minimized (Chen et al., 2023; Zhang et al., 2025). Despite the fact that some of the Li_2O still evaporate at high temperatures, it will recrystallize rapidly on the surface by the FJH method, thus avoiding diffusion losses (Zhu et al., 2023). As a result, the ingenious combination of the lithium phase conversion behavior in high temperature and FJH characteristics has broken through the problem of low lithium recovery rate from Al-contained black mass.

$$Li_2CO_3 + LiAlO_2 \xrightarrow{> 1100\ ^\circ C} Li_2O + Li_5AlO_4 + CO_2(g) \quad (5)$$

3.2. Step recovery of Li and Ni, Co, Mn

3.2.1. Lithium recovery by selective water leaching

After the FJH reduction process at 1400 °C for 90 s, we used simple water leaching method to selectively recover lithium. The concentration of various metals in leachate were measured by ICP-OES, and the specific results are shown in the Table S3. It shows that the leaching rate of Li is 91.8 %, and the contents of other metals are extremely low. This leachate can be prepared for battery grade lithium products following by the process of enrichment and simple purification. Interestingly, the pH value of lithium leaching solution increased to above 13 after the water-leaching process, which indicates that Li_2O is the main product in the FJH treated black mass. Fig. 5(a) exhibits the XRD analysis of FJH product and water-leaching residue. After flash heating at 1400 °C for 90 s, lithium exists mainly as Li_2O, Li_5AlO_4 and micro amounts of Li_2CO_3, $LiAlO_2$. And after water leaching, the lithium-containing phase is only

$LiAlO_2$. Li_2O, Li_2CO_3 and Li_5AlO_4 can be effectively leached by water, which explains the high leaching rate obtained by this method. In addition, the diffraction peaks of transition metals remain unchanged, indicating the selectivity of lithium extraction.

XPS and TOF-SIMS were also used to analyze the changes of components during FJH product and water-leaching residue. As shown in Fig. 5(b), three binding energy peaks in Li 1 s XPS spectra of FJH product can be observed at 55.3, 54.9 and 54.76 eV, respectively, corresponding to Li_2CO_3, Li_2O and Li-Al-O bond respectively. In contrast, these peaks could not be detected in the spectra of water-leaching residue, with the exception of $LiAlO_2$ (Ota et al., 2004; Dedryvère et al., 2005). In the TOF-SIMS mapping of Li and Al elements (Fig. 5c, d), obviously, the signal for the Li element in the FJH product is exceptionally intense. While in the leaching residue, the signal of element Li is almost undetectable. In addition, the signal of elemental Al is also attenuated due to the leaching of water-soluble lithium salt Li_5AlO_4 in the FJH process. The signal distribution of Li and Al in the water-leaching residue locates similar region, presumably the un-leached $LiAlO_2$. These results are extremely consistent with the XRD results, and further demonstrating the high recovery rate and high selectivity via FJH and water leaching method.

3.2.2. *Ni, Co, Mn recovery by acid leaching*

After selective leaching of Li, the transition metals were recovered through acid leaching. 98.7 % of Co, 98.2 % of Ni, and 98.6 % of Mn were leached from the water-leaching residues by using 3.5 M H_2SO_4 at 3 h, 85 °C and 10 g l^{-1} (Table S4). This leachate is suitable for preparing $Ni_xCo_yMn_z(OH)_2$ precursor after the purification process. Through the process of ultrafast high temperature reduction, transition metals can be rapidly converted into Ni, Co and MnO with high activity, so besides lithium, the efficient leaching of other valuable metals can also be realized.

3.3. *Technical evaluation*

Comparative experiments were conducted for the same black mass under conventional heating and FJH process (Fig. 6a and Fig. S3). After the conventional reduction roasting at 700 °C, the water leaching rate of Li is only 70.1 % due to the formation of $LiAlO_2$. When the heating temperature increases to 1400 °C, the lithium leaching rate is only 24.4 %, and the diffraction peaks of Li-related phases are weak or even disappeared (Fig. S3b), which can be attributed to the gaseous volatilization loss of Li. While the rapid heating and reaction rates of FJH method can effectively avoid the loss of lithium, and the conversion from $LiAlO_2$ to Li_5AlO_4 further increases the Li leaching rate to 91.8 %. The energy consumption of FJH method is only 31.5 kJ/g, which is approximately 700-fold lower than conventional method. Furthermore, Fig. 6(b) and Table S5 show the comparison of FJH method and the available typical lithium recycling methods (Hu et al., 2017; Zhang et al., 2023; Liu et al., 2019; Liu et al., 2019; Yuan et al., 2023; Ma et al., 2021; Yan et al., 2023; Yang et al., 2020; Yang et al., 2024; Chen et al., 2021; Deng et al., 2024; Liu et al., 2023; Di et al., 2020; Zhu et al., 2024; Xiao et al., 2021; Yu et al., 2024; Peng et al., 2019). Our method has sufficient advantages in terms of energy conservation, and the recovery rate of lithium is also at a comparatively high level.

In summary, the method we proposed has the following advantages: 1) Ultrafast high temperature process enables the rapid dissociative reduction of NCM material, and prevents the thermal volatilization loss of metals. 2) The Li-phase exhibits distinctive conversion behavior which leads to high leaching rate of Li through selective water leaching, and the efficient of Ni, Co and Mn can also be realized without the addition of reductant. 3) The efficient and energy-saving technological features of the FJH method are conducive to reducing greenhouse gas emissions and improving the economic benefits of the battery recycling.

4. Conclusion

Low lithium recovery efficiency and high energy consumption are the bottleneck problems in the current recycling technologies for black mass of spent NCM batteries. In this work, the thermodynamic analysis indicates that the water-insoluble $LiAlO_2$ can be transformed into water-soluble Li_5AlO_4 when the temperature is above 1100 °C. This inspires us to employ flash joule heating (FJH) method to preferentially recover lithium from actual raw material in industry. After the optimal FJH condition of 1400 °C for only 90 s, 91.8 % of lithium is extracted by simple water leaching, and extremely low impurities were simultaneously leached. Further characterization analysis indicates that Li_2O and Li_5AlO_4 can be effectively leached by water, which explains the high leaching rate obtained by this method. In addition, over 98 % of Ni, Co, Mn retained in the water-leaching residue can be recovered through sulfuric acid leaching method. The phase transformation mechanism provides a more detailed explanation of the conversion behavior during the FJH process. At low temperature stage (700 °C), Li_2CO_3 and $LiAlO_2$ are the main phases. While in the ultra-temperature stage (>1100 °C), Li_2CO_3 is completely decomposed to form Li_2O, and the portion of Li_2O will combine with $LiAlO_2$ to form Li_5AlO_4 under this condition. Besides, due to the ultrafast reaction by FJH process, the volatilization loss of lithium can be efficiently inhibited. Compared to conventional methods,

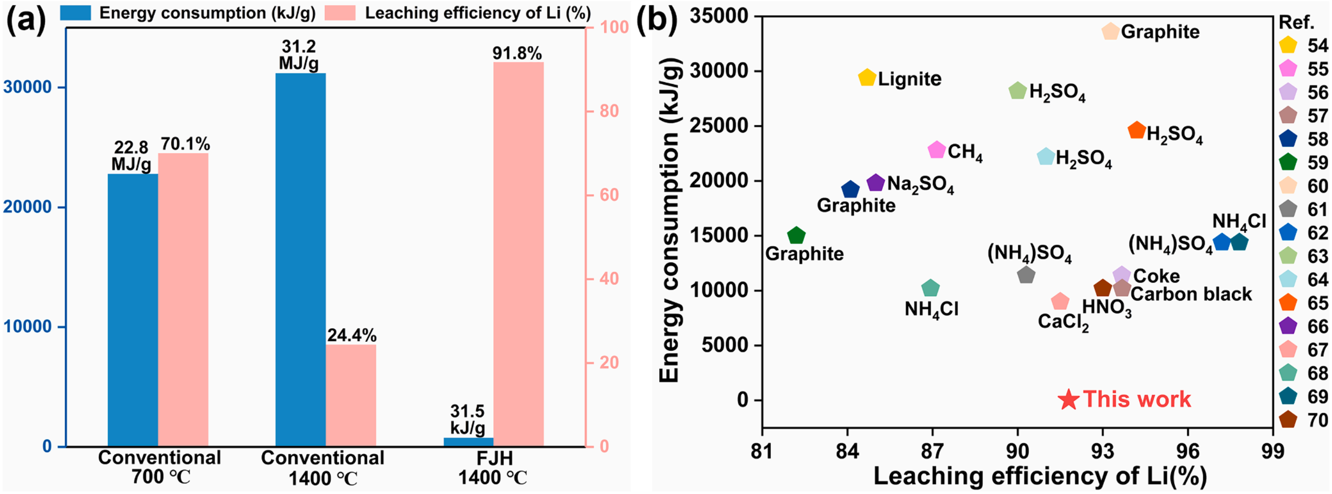

Fig. 6. (a) Energy consumption and lithium leaching rates of the same black mass under different roasting conditions; (b) Comparison of FJH method and conventional recycling methods.

the FJH method has extremely low energy consumption (31.5 kJ/g) and greenhouse gas emissions, further promoting sustainable development of the new energy industry under "carbon peaking and carbon neutrality" backdrop.

CRediT authorship contribution statement

Ao Shen: Writing – original draft, Methodology, Investigation, Data curation. **Jialiang Zhang:** Writing – review & editing, Funding acquisition, Formal analysis, Conceptualization. **Yongqiang Chen:** Writing – review & editing. **Chengyan Wang:** Supervision, Funding acquisition, Conceptualization.

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.

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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.2025.108433.

Data availability

Data will be made available on request.

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