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Microwave-assisted calcination of spodumene for efficient, low-cost and environmentally friendly extraction of lithium

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ABSTRACT

To utilize the advantages of microwave-assisted calcination over conventional heating, this work studied the effect of microwave power on phase transformation and leachability of spodumene. X-ray diffraction (XRD) and scanning electron microscope (SEM) were utilized to characterize the phase transformation and structural changes of spodumene calcined by both microwave and conventional heating methods. Results showed that the effect of microwave power on both phase transformation and leachability of spodumene is significant. The optimum lithium recovery through the microwave calcination-acid roasting-leaching process reached 97% at 2.0 kW microwave power, comparable with that of conventional heating. Besides a more rapid, less energy-consuming, and free greenhouse-gas-emission calcination, microwave heating also resulted in lower recovery values of Fe, Na, and Ca in the leaching process, gaining an advantage in downstream purification processes. The microwave temperature profiles of spodumene, and characterization results confirmed that α-spodumene started to transfer into γ- and β-spodumene at around 900 °C.

1. Introduction

Lithium is one of the critical elements with widespread applications in next-generation technologies, including energy storage, electric mobility, and cordless devices [12,27,30,39,43]. Due to its unique applications, lithium cannot be substituted in most applications; therefore, a steady annual demand increase of 8–11% is anticipated [5,16,17]. Meeting such a rising demand for lithium for various applications requires prospecting and processing all viable resources, including brine sources and ores (e.g., spodumene mineral).

Spodumene mineral is the major source of high-purity lithium, which contains a theoretical chemical composition of approximately 8% Li_2O, 27.4% Al_2O_3, and 64.6% SiO_2 [3,32,44]. Spodumene can exist in α, γ, and β modifications [38]. The α-spodumene phase, which belongs to the pyroxene group, is the naturally occurring crystal structure. β-spodumene is a recrystallized product when α-spodumene is heated at 900–1100 °C [10,13,31], where the complete conversion occurs by heating at 1100 °C for 2 h [37], and has interlocked five-membered rings of $(Si,Al)O_4$. The γ-spodumene is a metastable phase that occurs when α-spodumene is heated at 700–900 °C [23]. The α-spodumene is a compact mineral with very low reactivity, and cannot be leached for lithium extraction. Therefore, modifying the crystalline structure of concentrated spodumene mineral to the porous, reactive, and leachable β-spodumene using conventional heating (calcination) at 1000–1100 °C for about 2 h is a current industrial practice [26]. This calcination process is very energy-intensive and has been the bottleneck of the economic extraction of lithium from spodumene ores. This process also significantly contributes to the high CO_2 emission of Li extraction from spodumene (i.e., 9 t of CO_2 per tonne of lithium carbonate equivalent (LCE) produced, nearly triple that of LCE from the brine sector) [36]. Microwave-assisted calcination of spodumene offers significant advantages over calcination through conventional heating.

Microwaves are electromagnetic waves with frequencies between 300 MHz and 300 GHz from which 915 and 2450 MHz are the most commonly used for industrial/commercial applications. Compared to the conventional heating, cost-saving, low processing time, better controllable heating process with no greenhouse gas emission, and direct, non-contact, selective, and volumetric heating are tangible benefits of microwave processing [1,11,33]. The latter results in enhancing the porosity of the host mineral, thereby reducing the required sintering time,

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and chemical reactions time and temperature, while enhancing the lixiviants diffusion in the mineral and improving elemental recovery values [9,22,24,33,34,46]. These advantages have increased the utilization of microwave heating in metallurgical processes for the extraction of various metals such as heavy, light, rare, and precious metals, especially from corresponding minerals [1,11,20,33,42].

As for the interaction with microwave, materials are categorized into three groups: transparent, conductor/opaque, and absorber [4,8,21,41]. The materials with dielectric loss factors that change rapidly with temperature during microwave processing can be susceptible to uneven heating and thermal runaway. A review of susceptor materials and their applications in microwave processing can be found in the literature [7]. On the other hand, materials with low dielectric loss factors, such as spodumene, are difficult to heat from room temperature due to their low absorption of the incident energy. However, these materials can absorb microwave at elevated temperature as their dielectric loss increases with temperature rise. Therefore, for microwave heating of these materials, instead of the direct method, hybrid microwave heating is generally used in such cases. In this method, the materials are preheated using another heating source or through a susceptor to a certain temperature, where the sample becomes an absorber and heats up directly in microwaves [11].

The mechanism and effect of hybrid microwave process parameters on the structural change of spodumene have not been well understood. Previous limited studies on the phase transformation of spodumene through microwave treatment show inconsistent mechanism and results such as the reaction sequence of $\alpha \rightarrow \gamma + \beta$-spodumene [31] versus the formation of γ-spodumene as an intermediate phase, i.e., $\alpha \rightarrow \gamma \rightarrow \beta$-spodumene [32] or the formation of γ-spodumene during calcination of finely ground, and partly amorphized α-spodumene [6,18,19,23]. The differing observations of the phase transformation mechanism of spodumene polymorphs at high temperatures arise as a likely consequence of variable grain sizes, concentration of impurities, amorphous material formed during the grinding, different heating techniques, experimental setup, and temperature measurement techniques [31]. Nevertheless, the effect of process parameters (mainly microwave power) on the phase transformation of spodumene and its leachability has not been yet studied.

Therefore, to utilize the potential benefits of microwave and hybrid processes, the current research studied the microwave-assisted calcination of spodumene, evaluated the effect of microwave power on phase transformation and leachability of this mineral, and compared the results with that of calcined via conventional heating.

2. Materials and methods

2.1. Materials

The spodumene ore sample was provided by Piedmont Lithium Inc., collected from the Carolina Tin-Spodumene Belt near Kings Mountain, NC, which was once the heart of lithium production in the USA from the 1960s to the 1980s. The mineralogical studies on the sample revealed that the ore contains ~20% spodumene (~1.6% Li_2O), ~30% quartz, ~43% feldspar, ~5% mica, and trace amounts of biotite, other silicates, calcite, pyrite, chlorite, and apatite.

Traditionally, spodumene ores are upgraded to a concentrate with a Li_2O content of >6.0% and a Fe_2O_3 content of <1%, as the feed for the downstream calcination and lithium extraction processes. In this study, to prepare the spodumene concentrate for microwave processing, the ore sample was first crushed to −3.35 mm top size and ground into a size of −250 μm, followed by desliming at 20 μm. Then, the spodumene mineral was floated in a series of rougher, cleaner and re-cleaner flotation tests, conducted using a Denver lab-scale flotation cell. MIBC and AERO-727 (Solvay) were used as flotation frother and collector reagents, respectively. Finally, the spodumene concentrate was passed through a Wet High-Intensity Magnetic Separator (WHIMS) to remove the iron-bearing minerals. Quantitative analysis of the X-ray diffraction (XRD) pattern showed that the spodumene concentrate consists of 92% spodumene and 8% quartz. This finding was supported by the lithium, aluminum and silica oxide contents shown in Table 1 as determined by XRF analysis.

Table 1
Chemical composition of the spodumene concentrate analyzed by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES).

Component	Li_2O	Al_2O_3	SiO_2	Fe_2O_3	Na_2O	CaO	Total
Content (%)	5.64	25.10	65.70	0.73	1.06	0.66	98.90

2.2. Methods

2.2.1. Calcination

The effectiveness of hybrid microwave calcination and the effect of microwave power on phase transformation of spodumene concentrate was investigated using a multimode cavity microwave chamber, model HM-X06–10, Cober Electronics. The schematic of the microwave system is shown in Fig. 1. The dimensions of the processing chamber were 61 cm in width, 64 cm in length, and 43 cm in height. The chamber was equipped with independent magnetrons and a monitoring system that allowed power and temperature control. The power was adjustable from 0.1 to 6.0 kW with a microwave frequency of 2.45 ± 0.05 GHz. For each test, a sample of 5 g of spodumene concentrate was placed in an alumina crucible with dimensions of 32 mm diameter and 35 mm height. Alumina crucible is transparent to microwave energy; therefore, it did not interfere with the microwave heating process. Blocks of Fiberfrax® (porous alumina-silicate) were used for the thermal insulation of the sample. For hybrid heating of the samples, six silicon carbide (SiC) rods with a diameter of 12 mm and 27 mm in height were placed around the crucible as susceptors. The power was turned on and adjusted to achieve target temperature values, and the temperature and applied power at different times were monitored continuously.

Accurate temperature measurement is of major importance in controlling the microwave heating process and accurately interpreting the material processing. Therefore, a two-color E2RL optical pyrometer, manufactured by Fluke Process Instruments, was installed at the top of the chamber for continuous monitoring of the sample temperature in the range of 350–1200 °C. Taking into account that the apparent emissivity depends on the temperature, the pyrometer was first calibrated using the same target material in a conventional furnace, model HM-X06–10, in a temperature range of 500–1100 °C. After the measured temperature of the sample reached 1200 °C, the microwave system was turned off, the system was cooled down, and the sample was removed for characterization and further experiments.

A baseline calcination experiment was carried out in a conventional furnace, model HM-X06–10. The purpose of this experiment was to compare the results of completely converted spodumene concentrate into β-spodumene using conventional heating with those obtained from the microwave experiments. For the baseline experiment, 5 g of spodumene sample was placed in an alumina crucible and heated at

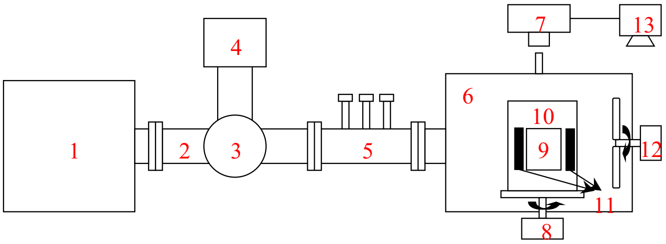

Fig. 1. Schematic of the microwave system (multimode, 2.45 GHz, 6 kW).

1100 °C for 8 h. All conventional and microwave calcination experiments were performed at ambient pressure.

2.2.2. Sample characterization

Solid samples (including spodumene concentrate and leaching residues) were digested according to the ASTM D6357-11 method. Elemental content of digested samples and leachate samples were analyzed using Thermo iCAP 7400, Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES) at the Penn State Laboratory for Isotopes and Metals in the Environment (LIME). Mineralogical characterization of the spodumene concentrate and the calcined samples was conducted through X-ray diffraction (XRD) analysis using Malvern Panalytical Empyrean III. The data were collected over a 2θ range of 5–80° with Cu K_α radiation. The morphology of the samples was also studied by Apreo (Thermo Fisher Scientific) scanning electron microscopy (SEM). For SEM analysis, each sample was coated with iridium (Ir) using Leica Sputter Coater to reduce the effect of charging on the particles. XRD and SEM analyses were performed at the Material Characterization Lab (MCL) at Penn State's Materials Research Institute (MRI).

2.2.3. Acid roasting and leaching

The leaching characteristics of microwave and conventional calcined samples were compared. To extract lithium from the calcined samples (i.e., β-spodumene), conventional acid roasting followed by water leaching of β-spodumene was performed [15]. Two grams (i.e., 1.2 mL) of H_2SO_4 (C = 96.2 wt%) solution was added to one gram of each calcined sample and manually stirred with a polytetrafluoroethylene (PTFE) stick so that the sample is properly impregnated by the acid. The mixture was then heated in a furnace at 250 °C for 60 min. After acid roasting and cooling to room temperature, 100 mL of distilled water was added to the acid roasted sample. The mixture was then stirred for 120 min at room temperature at a speed of 450 rpm to leach the soluble Li_2SO_4 generated during the acid roasting. The leachate was then separated from the solid residue by filtration, using a Buchner funnel and a 0.45 μm filter paper. The residue particles were rinsed with DI water and dried overnight at 100 °C. Finally, the leachates, which were diluted with DI water, and the digested leaching residues were analyzed for Li, Al, Si, Fe, Na, Ca contents using ICP-AES. The elemental recovery values were calculated using the following equation, where C_l and V_l are the concentration (mg/L) and volume (L) of the leachate; and C_s and M_s are the concentration (mg/g) and mass (g) of the residue, correspondingly.

$$\text{Recovery } (\%) = \frac{C_l V_l}{C_l V_l + C_s M_s} \times 100\% \tag{1}$$

3. Results and discussion

3.1. Microwave heating characteristics of spodumene

To study the effectiveness of microwave calcination on spodumene concentrate and the effect of microwave power, calcination experiments were conducted at various power levels of 0.5 to 3.0 kW with an increment of 0.5 kW. The sample temperature vs. microwave power profiles are shown in Fig. 2. It can be seen that at 0.5 kW, the temperature of the sample could not reach 600 °C even over 70 min. When the microwave power was raised to 1.0 kW, the peak temperature of the sample could reach over 1200 °C in 30 min, and with 2.5 kW or higher in less than 10 min. Close examination of the temperature profiles of spodumene at the microwave powers of 1.0, 1.5, and 2.0 kW revealed that the heating process of spodumene could be divided into four stages in terms of heating rate: 1. medium heating (50–70 °C/min on average) up to around 600 °C, 2. a plateau (~0 °C/min), 3. slow heating (20–30 °C/min) from 600 to 900 °C, and 4. very rapid heating (>300 °C/min) after 900 °C.

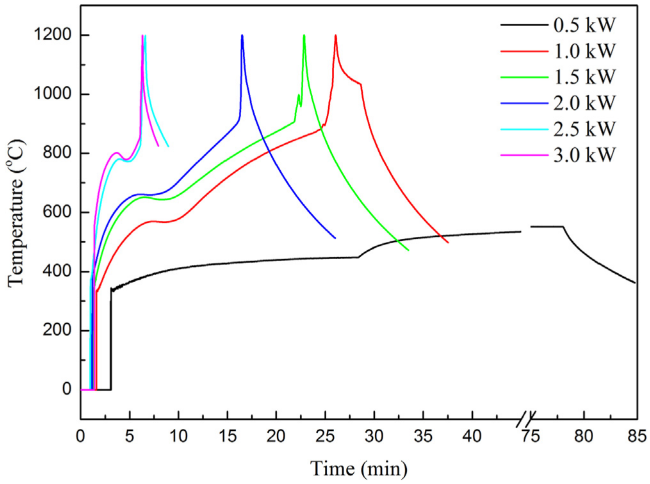

Fig. 2. Measured spodumene temperature profiles under different microwave powers (0.5–3 kW).

Microwave heating depends on the thermo-physical properties of the sample [33]. Spodumene is transparent to microwave at low temperature, and the heating mechanism at the first stage is due to the radiation of SiC, which has a high loss factor and strongly absorbs microwaves. Therefore, the temperature at the first stage increased at a relatively rapid rate of above 50 °C/min. For a given microwave power, the temperature will reach the maximum value after a certain time. After this, a plateau is observed as the second stage of the temperature profile in Fig. 2. By comparing the temperature profiles, it can be seen that as the microwave power increases, the maximum temperature for the first stage increases [25,28,33]. At the microwave power of 0.5 kW, the maximum temperature which spodumene can reach through heating by SiC rods was around 550 °C. For 1.0–2.0 kW, the temperature of the second stage was around 600 °C, while it reached around 800 °C at 2.5–3.0 kW powers. By increasing the power, heat generation at the surface of materials (W/m^3) increases; therefore, a higher temperature in shorter periods of time can be achieved [14].

Apart from 0.5 kW power, the temperature profiles at applied microwave powers more than 1.0 kW continued to rise after the corresponding plateaus. The critical temperature after which α-spodumene absorbs microwave energy and is reported in the literature to be around 634 °C [37]. This explains the phenomenon that at the microwave powers of over 1.0 kW the temperature of spodumene started to climb after the plateau stage. However, the critical temperature for α-spodumene observed in this study under 1.0–2.0 kW microwave power was in the range of 570–660 °C, which is about 0.4–0.5 times its melting point of around 1400 °C [29]. It can be seen that the microwave power affects the critical temperature of spodumene. Although α-spodumene started from 570 °C to absorb microwave energy in this study, the accurate critical temperature for α-spodumene needs further study. At the third stage, the heating rate was smaller (i.e., about 20–30 °C/min) than that of the first stage (an average of 50–70 °C/min), probably due to the reason that even at above critical temperature the dissipation factor of α-spodumene ($\tan\delta \approx 0.0002$–$0.0007$) is smaller than that of SiC ($\tan\delta \approx 0.1$) [2,40,45].

When the temperature increases up to around 900 °C, the heating profiles rapidly become almost exponential, reaching 1200 °C in about 1 min from 900 °C. The Gibbs free energy vs. temperature for the phase transformation of spodumene from α to β calculated by HSC Chemistry-6.0 (shown in Fig. 3) reveals that this transformation can occur spontaneously at temperatures above 800 °C. Recent studies also reported that α-spodumene started to be converted into γ- and β-spodumene at around 900 °C [10,31,32,38]. The dissipation factor of β-spodumene ($\tan\delta = 0.004$) is higher than that of α-spodumene

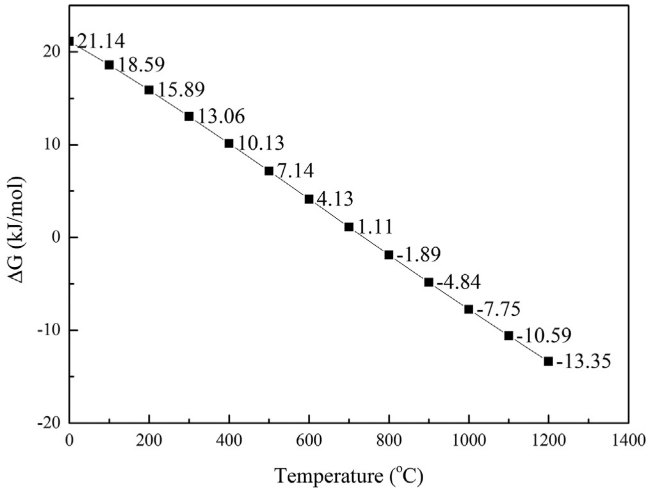

Fig. 3. Ellingham diagram for the conversion of α to β-spodumene. ΔG values were calculated in HSC Chemistry-6.0 [35].

[37,40]. In addition, spodumene might go through an amorphous phase which has a much higher dissipation factor and energy absorption (tan $\delta = 0.047$) [32,37,40]. These two scenarios result in the fourth stage of very rapid heating (>300 °C/min) after 900 °C. The results showed that the time needed to reach the fourth stage was much less with higher applied microwave power. At 2.5 kW or higher, the temperature of spodumene reached 1200 °C from room temperature in about 6 min, as increasing the power results in achieving higher temperature in a shorter time. The fact that the dielectric loss increases with the temperature explains the rapid increase of the sample temperature as the microwave power increases. The temperature profiles (shown in Fig. 2) indicated the success of the microwave hybrid heating of spodumene. The process can be further modified by mixing the samples with susceptor materials which can be readily recovered from the treated spodumene after calcination, utilizing the differences in physical properties such as size.

3.2. Characterization of the calcined samples

Mineralogical analysis of the microwave and conventionally calcined samples were conducted to understand the phase transformation of the sample during the calcination and to investigate the corresponding effects of microwave power (Fig. 4). Upon calcination at 1100 °C for 8 h via a conventional heating furnace, the α-spodumene was completely converted to β-spodumene. This complete transformation was expected since the required amount of time in other studies, and practical application is reported as 2 h.

For the microwave calcined samples, the mineral phase of the sample after heating at 0.5 kW power remained as α-spodumene. As shown in Fig. 2, the maximum temperature the sample reached at 0.5 kW power was around 550 °C, which is not high enough for the conversion of α to β-spodumene. At 1.0, 1.5, and 2.0 kW powers, the temperature of the samples could reach over 1200 °C. The comparison of XRD peaks at 25.5°, 22.7°, and 19.2° of 2θ angles of the calcined samples at 1.0–2.0 kW power range with those of the spodumene concentrate (i.e., α- spodumene) indicated the α to β-spodumene conversion for these microwave powers. However, the presence of a relatively low-intensity peak at a 2θ angle of 25.5° revealed that the conversion of α-spodumene was not complete at 1.0 kW. The small peak at $2\theta = 32°$ also implied that α-spodumene was not completely converted into β-spodumene at 1.5 kW. At all the three different microwave powers, the small peak at a 2θ angle of 19.6° could be observed in the XRD patterns. This peak is the evidence of the formation of γ-spodumene [32], of which the appearance is consistent with recent studies. At 2.0 kW power, the α-spodumene was completely converted to β/γ-spodumene. At the microwave powers higher than 2.5 kW, XRD patterns showed that the composition of the calcined samples remained as α-spodumene. This unexpected result was attributed to the relatively short heating time discussed previously, which was not long enough to calcine the sample for the α-spodumene phase transformation.

The morphology of the samples was also characterized using SEM analysis (Fig. 5). Comparison of SEM image of spodumene concentrate (Fig. 5(a)) with that of calcined via conventional heating (Fig. 5(b)) shows that the flat and smooth surfaces of α-spodumene were wrinkled

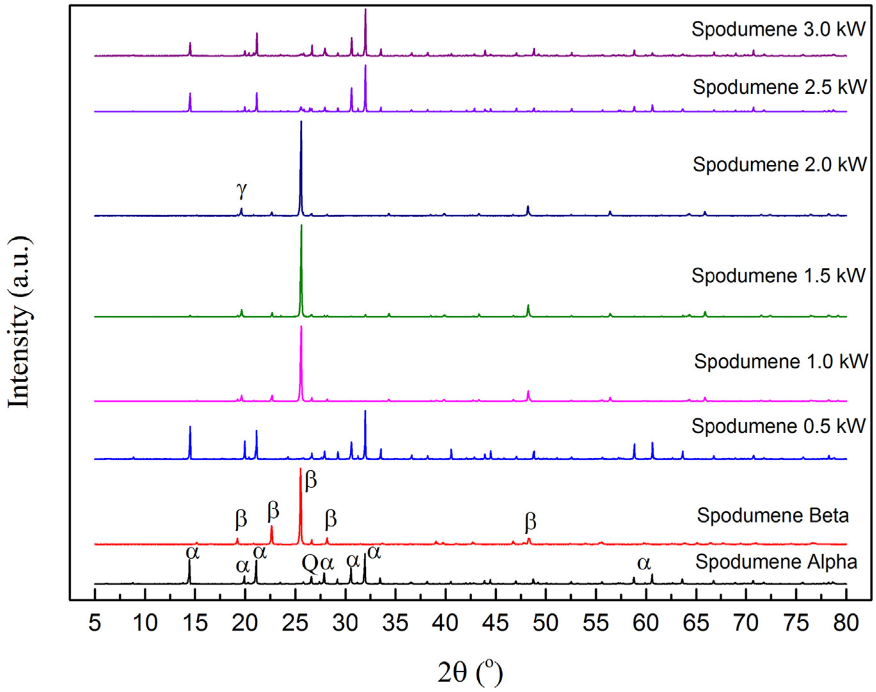

Fig. 4. XRD patterns of the spodumene concentrate (i.e., spodumene Alpha) and calcined concentrate samples via conventional heating (i.e., Spodumene Beta) and hybrid microwave heating at different microwave powers of 0.5–3.0 kW. α—α-spodumene; β—β-spodumene; γ—γ-spodumene; Q—quartz.

and fully fractured after conventional heating at 1100 °C for 8 h (i.e., conversion to β-spodumene). Fig. 5(c)–(h) shows the SEM images of calcined spodumene via microwave heating at different powers. At 0.5 kW microwave power, the form and appearance of spodumene were unchanged, while at 1.0 kW power, small particles of spodumene were cracked and decrepitated, though large particles remained relatively intact. As the microwave power increased, large particles were also decrepitated, and the cracks became wider. The morphological changes indicate that the structure of spodumene was transferred to a higher degree with increasing microwave power. However, at 2.5 kW and 3.0 kW, most spodumene particles were remained unchanged due to low sintering time. Interestingly, in these two samples, few small and

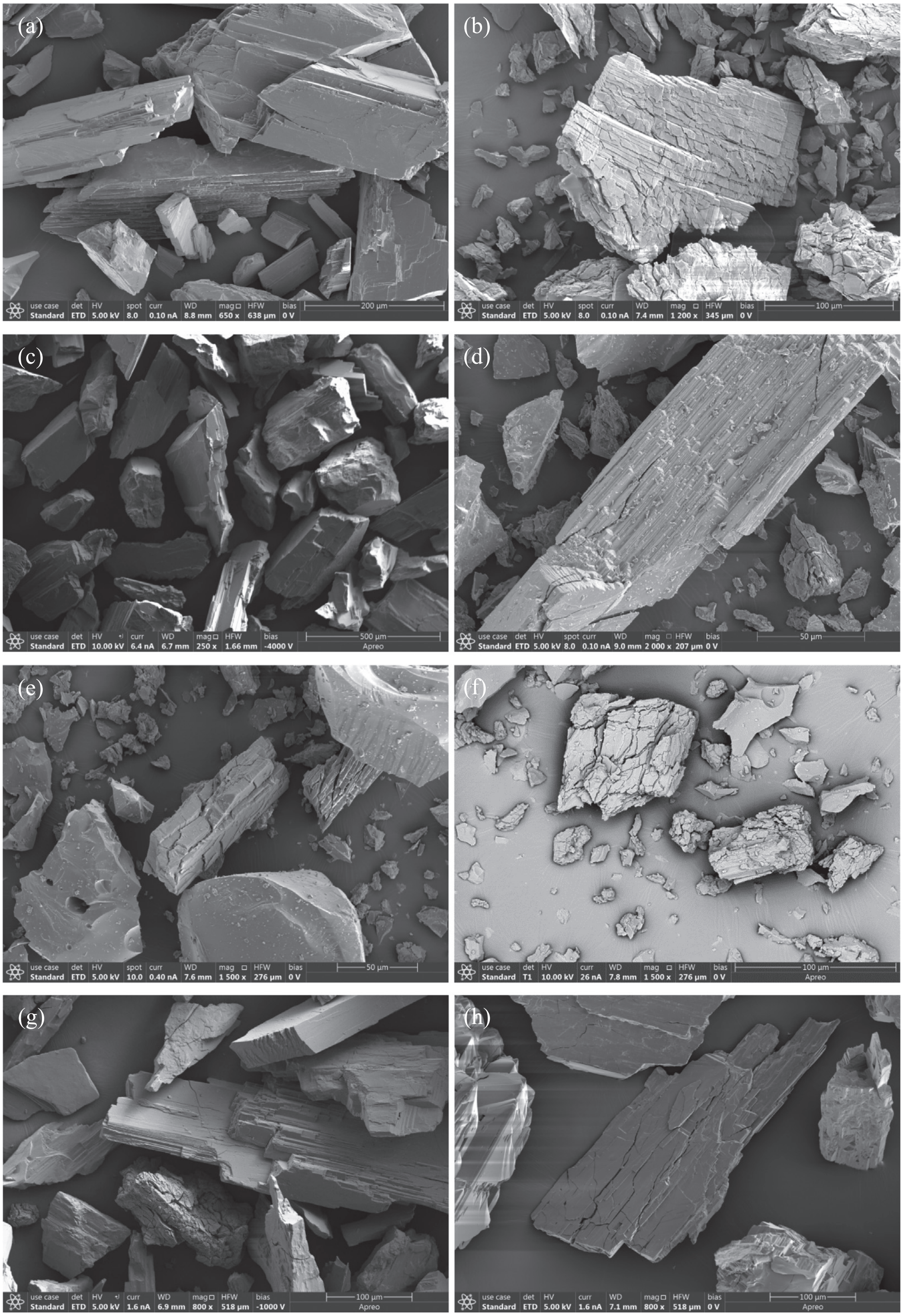

Fig. 5. SEM images of (a) spodumene concentrate, and calcined spodumene via (b) conventional heating at 1100 °C, and microwave heating at (c) 0.5 kW, (d) 1.0 kW, (e) 1.5 kW, (f) 2.0 kW, (g) 2.5 kW, and (h) 3.0 kW powers.

thin particles of spodumene were partially decrepitated, which was not expected as the XRD patterns did not show the conversion of α- to β-spodumene. Such observation that small spodumene particles were prone to convert into β-spodumene even at low sintering time implies that particle size has an effect on the microwave heating of spodumene [32,33,37]. This should be further investigated for the application of microwave heating of spodumene.

Another observation when conducting the SEM testing was that the particle size of samples roasted at 1.0–2.0 kW was generally smaller than that roasted at 0.5, 2.5, and 3.0 kW. The size reduction was probably because particles were broken into pieces due to the internal stress and thermal shock during heating, as well as the volumetric expansion of around 30% when α-spodumene is transformed into β-spodumene [38]. In addition, in Fig. 5(e), it is noticeable that there are some particles different from α-spodumene or converted γ- and β-spodumene. Those particles have a smooth surface and do not have cracks. Further investigation under SEM, as shown in Fig. 6 revealed that those particles were common in microwave heated samples at 1.0, 1.5, and 2.0 kW. Fig. 6 (b) and (d) represent a partially converted spodumene particle whose other parts are similar to those particles shown in Fig. 5(e). Although the maximum recorded temperature in this study was 1200 °C and the melting point of spodumene is around 1400 °C or less (depending on impurities), the actual temperature of the roasted spodumene at 1.0–2.0 kW could reach above the melting point since the heating rate at the fourth stage was above 300 °C/min in Fig. 2. At 2.0 kW microwave power, the instantaneous heating rate on the fourth stage could reach 30 °C/s. Based on such evidence, it is reasonable to deduce that those particles are melted spodumene. Previous studies reported similar observations of partially melted particles in their calcined samples via microwave heating [32,37]. Salakjani et al. [37] concluded that the sintered zone in their samples consists primarily of γ-spodumene and/or β-spodumene. Overall, the SEM and XRD results were consistent, and 2 kW microwave heating resulted in maximum phase transfer comparable with the conventional heating.

3.3. Effect of microwave heating on the leaching efficiency

The efficacy of microwave-assisted calcination on the extraction of Li was evaluated. The raw spodumene concentrate (α-spodumene), and the calcined spodumene using microwave and conventional heating were subjected to acid roasting-leaching procedures. Leaching recovery of major elements is shown in Fig. 7. The recovery of lithium increased with the increasing microwave power, up to 97% at 2.0 kW. At 1.0 and 1.5 kW, the leaching efficiency of lithium was only around 60%. Based on the characterization results discussed above, the α-spodumene was not completely transformed into β-spodumene at 1.0 and 1.5 kW, thereby a relatively low leaching efficiency of lithium. Although γ-spodumene was found in the calcined samples via microwave heating but not found in the calcined sample via conventional heating, the recovery of lithium at 2.0 kW via microwave heating was almost as high as that via conventional heating. It can be inferred that the existence of γ-spodumene has little effect on the recovery of lithium as long as there is no α-spodumene in the calcined sample. A previous study by Dessemond et al. [12] also showed that γ-spodumene of less than 10 wt% would have little influence on lithium recovery in the leaching process. Compared with conventional heating, microwave heating can reach the equivalent recovery of lithium. Fig. 7 also presents the recovery of Al, Si, Fe, Na, and Ca. The recovery of Al and Si in the leachate was very low via both microwave and conventional calcination. This is because the mechanism of the acid roasting method is similar to a substitution reaction, with H^+ replacing Li^+ in β-spodumene, producing soluble lithium sulfate, which can be readily dissolved in water [26]:

$$\beta-Li_2O\cdot Al_2O_3\cdot 4SiO_2+H_2SO_4\rightarrow H_2O\cdot Al_2O_3\cdot 4SiO_2+Li_2SO_4 \qquad (2)$$

Through this reaction, the structure of aluminosilicate is not damaged. Thus, Al and Si were not dissolved in the leaching procedures. The recovery results of Fe, Na, and Ca showed that microwave heating had lower recovery of these elements and therefore may have an

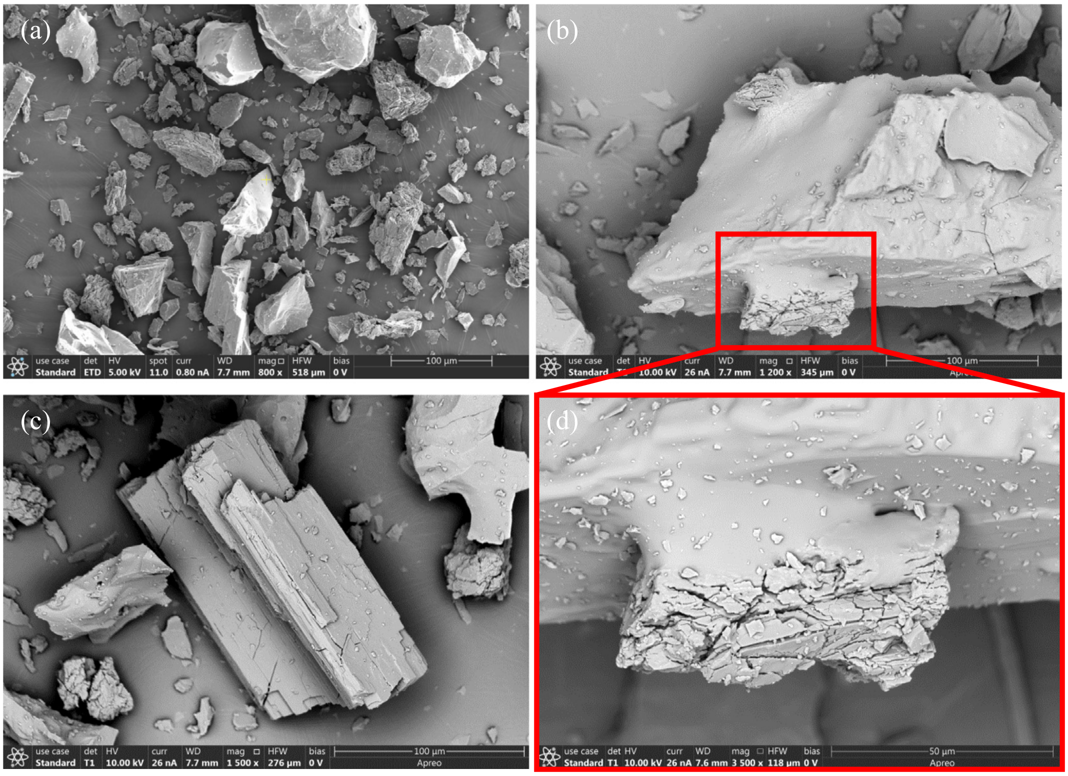

Fig. 6. SEM images of microwave-assisted calcined spodumene particles with melted parts on the surface at the microwave powers of (a) 1.0 kW, (b) 1.5 kW, (c) 2.0 kW, and (d) inset of (b) at a higher magnification.

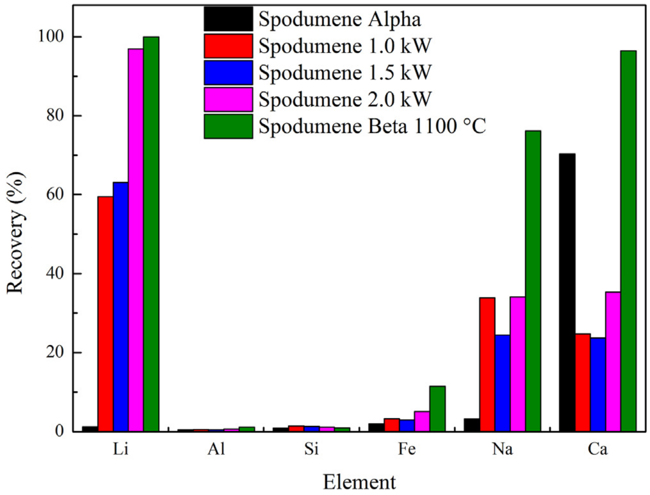

Fig. 7. Comparison of leaching recovery of major elements from the spodumene calcined via microwave and conventional heating methods.

advantage over the conventional heating in downstream purification processes. This could be due to the selective and volumetric heating characteristics of microwave heating, generating different temperatures for different minerals during calcination which are important to the potential reactions and could change the calcination products. However, it should be mentioned that the concentration of each of these elements in the sample was less than 1%.

4. Conclusion

This work studied the effect of microwave power on phase transformation of spodumene ($\alpha \rightarrow \gamma + \beta$) and its leachability, and compared the results with those of conventional heating. The microwave heating profiles of spodumene implied that the critical temperature for α-spodumene was around 570 °C, but the accurate critical temperature needs further study. When the temperature increases up to around 900 °C, the heating profile suddenly becomes almost vertical, reaching 1200 °C in about 1 min from 900 °C. Microwave power was found to have a significant influence on the temperature profile of spodumene, phase transformation, and corresponding leachability of the calcined products. The optimum microwave power was found to be 2 kW, above which the sintering time was not enough for phase transformation of the spodumene. Below this value, also, the α-spodumene was only partially converted to $\gamma + \beta$ phases. At this optimum power, a lithium recovery of 97% was obtained, which was comparable with that of calcined sample by conventional heating. Furthermore, the microwave-assisted calcination resulted in a lower amount of impurities (i.e., Fe, Na, and Ca) in the leachate, which is an advantage in downstream purification processes. The results also showed that the formation of γ-spodumene in the microwave heating had little effect on the leaching efficiency of lithium as long as there is no α-spodumene in the calcined sample.

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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