



Lithium extraction from β -spodumene through chlorination with chlorine gas



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ABSTRACT

The extraction of lithium by means of the chlorination roasting of β -spodumene has been studied in the temperature range from 1000 to 1100 °C for periods of time from 0 to 180 min. The roasting was carried out in a fixed bed reactor using pure gaseous Cl_2 as chlorinating agent. The reactants and products were characterized by X-ray diffraction (XRD), X-ray fluorescence (XRF), atomic absorption spectrometry (AAS), scanning electron microscopy (SEM) and electron probe microanalysis (EPMA). The roasting of β -spodumene with pure Cl_2 at 1100 °C for the period of 150 min led to quantitative extraction of lithium as lithium chloride. The solid products of the reaction of β -spodumene with Cl_2 were found to be $\text{Al}_6\text{Si}_2\text{O}_{13}$ (mullite), and Si_2O (cristobalite).

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1. Introduction

Lithium is a metal highly demanded worldwide mainly because it is known as the lightest metal and it has a fascinating electrochemical reactivity. The global production of lithium increased from 12,370 tons in 2000 to 24,739 tons in 2011. Overall lithium demand increased at a compound annual growth rate (CAGR) of 6.5% from 2000 to 2011, even accounting for the global recession which suppressed overall consumption in 2009 by approximately 13% from the prior year (Baylis, 2012).

Regarding the metallurgy of lithium, it can be found in nature as hard-rock ore and as brine. The current industrial practice for the production of lithium involves conversion to carbonate, then to chloride, followed by molten salt electrolysis. Subsurface brines have become the dominant raw material for lithium carbonate production worldwide because of lower production costs compared with the mining and processing costs for hard-rock ores (Kipouros and Sadoway, 1998). Nevertheless, taking into account the increasing demand for lithium and lithium salts, the current production levels of this metal should be further improved. Thus, lithium ores could be considered as alternative resources once again.

Among the commercially practicable sources of lithium, spodumene is the mineral that attracts the most interest and

various processes for recovering the lithium values from spodumene ore have been proposed. In a number of these processes, only two processes have been practiced: limestone method and sulfuric acid process (Kondás et al., 2006; Shin et al., 2005; Tu et al., 2003; Yang, 2004), and sulfuric acid process has become the main method for production of lithium carbonate from spodumene due to its high efficiency. However, these methods have intrinsic drawbacks, such as high levels of sulfate and heavy metal ions in the product, sophisticated process for recovering sodium sulfate, etc. for the former and too many limestone and high energy consumption for the latter (Chen et al., 2011; Richard and Ulrich, 1997; Yan et al., 2012). There have been reported relevant findings in order to overcome the drawbacks of above processes, for example: the pressure leaching with sodium carbonate process to extract lithium from spodumene concentrate (Chen et al., 2011). Also, chlorination roasting using different chlorinating agents has been developed to recover lithium as lithium chloride from spodumene without preliminary mineral concentration which, in turn, implies an economic advantage over the latter process, but few details of the investigation and evaluation on this method were reported (Davidson, 1981a,b; Medina and El-Naggar, 1984; Dunn et al., 2005).

In this paper, a roasting with pure Cl_2 gas is proposed to recover lithium from β -spodumene and the results of the investigation on this process are presented. The main objective is to study the effect of the temperature and the reaction time on the extraction of lithium as lithium chloride, using a spodumene from the Province of San Luis, Argentina.

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2. Thermodynamic analysis

The thermodynamic calculations for the chlorination of β -spodumene by gaseous chlorine were performed within the temperature ranging from 25 to 1100 °C. Considering that Cl_2 is in excess, the equilibrium composition of the system Si–Al–Li–O–Cl as a function of temperature can be calculated using HSC thermochemical database software (HSC, 2002). This calculation was performed using the system free energy minimization method and the results are presented in Fig. 1.

Observations made on this figure are presented in the following sentences:

- The chlorination of lithium present in the structure of β -spodumene is favoured in the whole temperature range studied. The products of this reaction are lithium chloride, which is a solid or liquid product, depending on the chlorination temperature, and oxygen, which is released as a gaseous product. In the temperature range of this work, the reaction of β -spodumene chlorination would yield lithium chloride in liquid state, remaining in the solid residue. However, it can be seen from Eq. (1), which shows the variation of LiCl(l) vapor pressure with the temperature determined experimentally within the temperature range of this work (Hildenbrand et al., 1964), that lithium chloride has vapor pressures sufficiently high to be volatilized at temperatures ranging from 1000 to 1100 °C. The authors have verified experimentally that pure LiCl contained in a quartz crucible volatilizes quantitatively after a few minutes when it is subjected to a flow of N_2 at 1000 °C. In a flow system, the volatile LiCl may leave the reaction zone entrained by the chlorine stream, favoring the chlorination reaction of lithium, and the formation of more LiCl .

$$\log P(\text{atm}) = (5.031 \pm 0.148) - (8387 \pm 134)/T \quad (1)$$

- Aluminum and silicon also present in the structure of β -spodumene do not form chlorides in appreciable concentrations in the temperature range studied. Part of SiO_2 is segregated in its different crystalline forms (quartz, cristobalite, tridymite, etc) whose composition of equilibrium varies with the temperature. The formation of aluminosilicates such as sillimanite, kyanite, andalusite, and to a lesser extent mullite is favoured at low temperatures. The formation of corundum is favoured at higher temperatures.

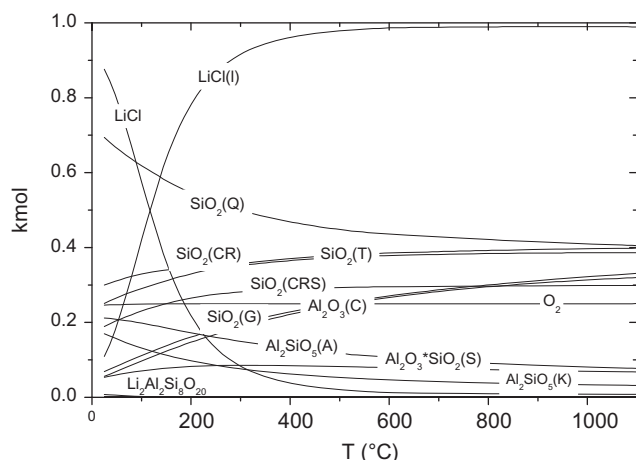


Fig. 1. Equilibrium composition of the system Si–Al–Li–O–Cl, as a function of temperature.

- The formation of compounds such as lithium metasilicates and orthosilicates, and lithium aluminates, which were considered for this calculation, is not favoured in the temperature range in study.

3. Experimental

3.1. Materials

The ore used in this study was spodumene obtained from the deposits situated in the Province of San Luis, Argentina (Angelelli and Rinaldi, 1963). The results of the chemical composition analysis of the ore are given in Table 1. The X-ray diffraction pattern of the raw ore is shown in Fig. 2a. The minerals present in the ore are α -spodumene ($\text{LiAlSi}_2\text{O}_6$) and quartz (Si_2O). According to the quantitative analysis by X-ray diffraction, the mineralogical composition of this sample is: 95.5% α -spodumene and 4.5% quartz. This rock ore was grounded in a ring mill, sieved to a particle size smaller than 50 μm , and then calcined at 1180 °C for two hours so as to produce a change in the crystalline structure; i.e. the polymorphic transformation of α -spodumene (monoclinic) into β -spodumene (tetragonal), which is less dense and more reactive. Fig. 2b shows the X-ray diffraction pattern of the calcined ore. The phases detected from it are β -spodumene and quartz. This indicates that the calcination of the spodumene ore carried out at 1180 °C for two hours is sufficient to produce the polymorphic transformation.

Fig. 3a and b shows the SEM micrographs of α and β -spodumene, respectively. A morphological change due to the polymorphic transformation of α -spodumene into β -spodumene phase can be observed.

The gasses used in the different chlorination assays were chlorine 99.5% (Cofil, Argentina) and nitrogen 99.99% (Air Liquid, Argentina).

The reagents used to prepare the solid samples for the measuring of lithium concentration by AAS were 95–98% H_2SO_4 (Sigma-Aldrich, Argentina) ACS reagent grade, CAS: 7664-93-9, and 40% HF (Merck, Argentina) GR for analysis, CAS: 7664-39-3.

3.2. Equipment

The reactor consisted in a quartz tube (o.d. 16 mm, wall thickness 1 mm, and length 440 mm) placed inside an electric furnace equipped with a temperature controller. The sample was held in a quartz crucible (length 70 mm, width 10 mm, and depth 6 mm) which was contained inside the reactor, as shown in Fig. 4. The temperature was measured with a chromel–alumel thermocouple to within ± 5 K. Chlorine stored in a cylinder was introduced to the inlet of the quartz tube through teflon tubing. Mass flowmeters and metering valves were used to control the flow rate.

The XRD analysis of reactants and products was performed on Rigaku D-Max-IIIC equipment with $\text{Cu K}\alpha$, operated at 35 kV and

Table 1
Chemical composition of the raw ore.

Component	% w/w
SiO_2	66.2
Al_2O_3	22.3
Fe_2O_3	0.95
CaO	0.28
MgO	0.25
K_2O	0.03
Na_2O	0.4
Li_2O	7.2
TiO_2	0.06

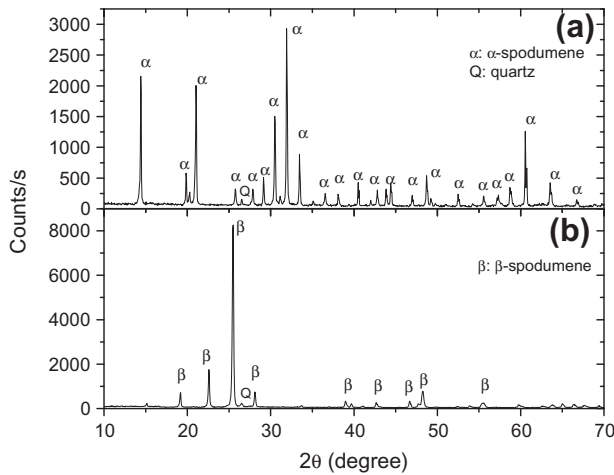


Fig. 2. XRD patterns of (a) spodumene ore and (b) spodumene calcined at 1180 °C for two hours.

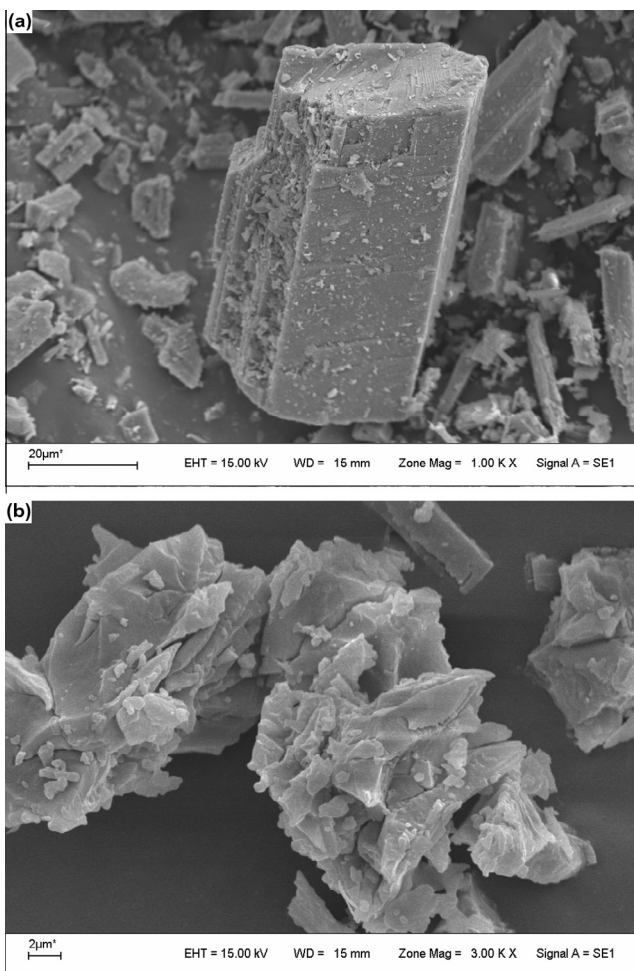


Fig. 3. SEM images: (a) α -spodumene and (b) β -spodumene.

30 mA. SEM and EPMA characterizations were carried out using a LEO 1450VP microscope equipped with an EDS spectrometer (EDAX Genesis 2000). An analytical balance (Mettler Toledo AB204-S/FACT, maximum sensitivity of 0.0001 g) was used for the measurement of the sample mass before and after each experiment.

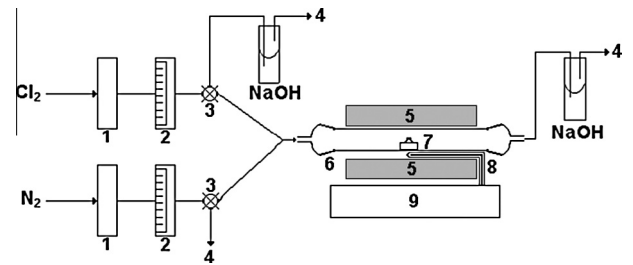


Fig. 4. Fixed-bed reactor. (1) H_2SO_4 dryers, (2) flowmeters, (3) three-way valves, (4) venting, (5) electric furnace, (6) quartz tubular reactor, (7) quartz crucible, (8) thermocouple, and (9) temperature controller.

The composition of the spodumene ore and the chlorination residues was determined by XRF with Philips PW 1400 equipment. The concentration of lithium present in β -spodumene and in chlorination residues was determined by AAS using a Varian SpectraAA 55 spectrometer with a hollow-cathode lamp. Previously, the samples were dissolved using concentrated sulfuric acid and hydrofluoric acid according to the method of Brumbaugh and Fanus, 1954.

3.3. Procedure

The experimental protocol was the following:

1. The reaction temperature was set, the furnace switched on, and N_2 fed into the reactor at 100 ml/min. The heating rate was 10 °C/min.
2. The sample holder was filled with 400 mg approximately of β -spodumene, this mass was registered as m° .
3. Upon attainment of the working temperature, the quartz crucible containing the sample was gently slid into the centre of the reactor.
4. This charge was kept in nitrogen atmosphere for 15 min.
5. The nitrogen supply was cut off and 100 ml/min of pure Cl_2 was fed into the reactor, keeping the sample in Cl_2 atmosphere. The time recording starts at this point.
6. Upon attainment of the reaction time, Cl_2 was replaced by nitrogen again to purge the system for 15 min.
7. The sample was taken out from the reactor, cooled down and then, the mass of the resultant solid residue was determined and registered as m^t .
8. The sample was slid again into the reactor and the procedure just described was repeated from step 4, until all points of each isothermal experiment are obtained.

4. Results and discussion

4.1. Chlorination of β -spodumene

The chlorination extent was determined by observing the mass changes undergone by the samples of β -spodumene. The mass change was expressed as mass change percentage, $\Delta m\%$, according to the following equation:

$$\Delta m\% = (m^t - m^\circ) * 100/m^\circ \quad (2)$$

where m° and m^t are the initial mass and the mass at time t , respectively.

Fig. 5 shows the evolution of the mass change percentage, as a function of time during isothermal roasting of β -spodumene in pure Cl_2 at 1000, 1050, and 1100 °C. For the construction of these curves, a single sample was used for each temperature during constant reaction periods. The process conditions were as follow:

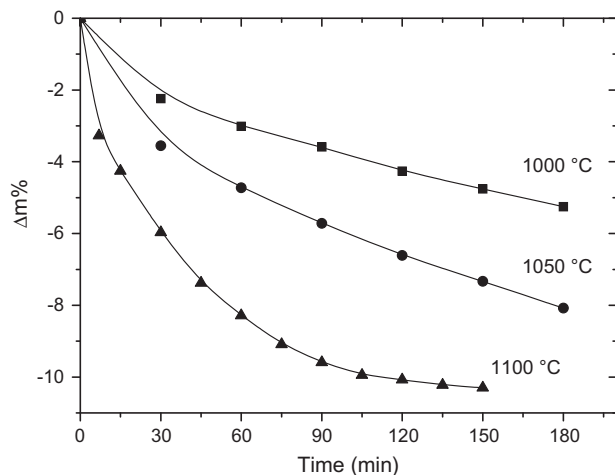


Fig. 5. Mass change percentage, as a function of time during isothermal roasting of β -spodumene in pure Cl_2 .

chlorine flow rate 100 ml/min; chlorine molar fraction, 1; mass of the sample, 400 mg ca.

Data from Fig. 5 show that the mass changes correspond to mass losses, which in turn increase markedly with the increase of temperature to a given reaction period. These mass losses may correspond to the volatilization of lithium chloride, according to the observations in the thermodynamic analysis section. In order to verify this, further isothermal chlorinations were performed. They were carried out under the same process conditions described in the procedure section, using a different sample for each chlorination period. The results of these isothermal experiments are presented in Table 2. The concentration of Li_2O in the residual samples was measured by AAS.

The content of Li_2O present in the residues decreases with the increase of temperature and reaction time as shown in Table 2. Thus, the chlorination roasting of β -spodumene in the temperature range studied results in different levels of extraction of lithium as lithium chloride, depending on the temperature and reaction time. For the experiment at 1100 °C and a reaction time of 150 min, lithium is not detected in the residue, which may indicate a quantitative extraction of Li_2O . Thus, the mass loss should be 7.2% (the initial concentration of Li_2O in the sample of β -spodumene). However, the mass loss observed in this experiment was 10.3%, which may indicate that further compounds are being produced and volatilized by the chlorination roasting. Bearing in mind the impurities present in the sample of β -spodumene, such compounds would correspond mainly to FeCl_3 , CaCl_2 , and MgCl_2 since these

Table 3

Chemical composition of the residual sample obtained at 1050 °C for 180 min.

Component	%w/w
SiO_2	69.5
Al_2O_3	25.9
Fe_2O_3	0.33
CaO	0.31
MgO	0.28
K_2O	0.03
Na_2O	0.4
Li_2O	0.79
TiO_2	0.05

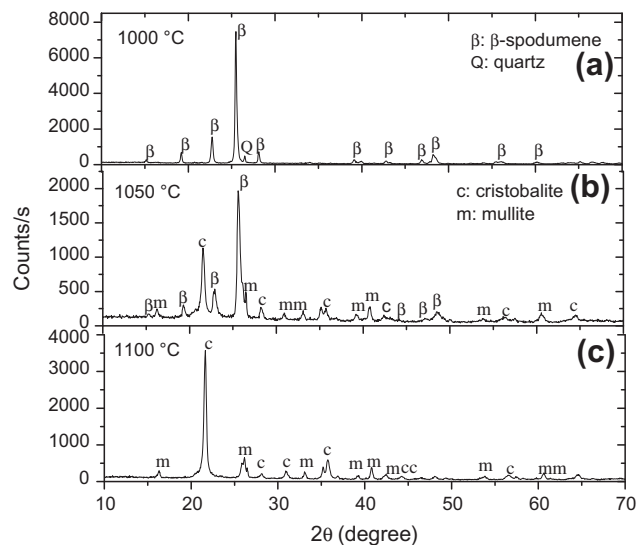


Fig. 6. XRD patterns of the residual samples of the isothermal chlorination.

impurities corresponding to such chlorides are feasible to be chlorinated at the operating temperatures of this work (Orosco et al., 2011, 2013). Analysis by XRF conducted on the chlorination residue obtained at 1050 °C and a reaction time of 180 min shows that the concentration of Fe_2O_3 decreases (Table 3), which indicates that Fe_2O_3 is partially removed by the chlorination roasting as volatile FeCl_3 ; this chloride has vapor pressures values of 1.51×10^{-3} , 1.77×10^{-3} , and 2.048×10^{-3} atm at 1000, 1050, and 1100 °C, respectively (HSC, 2002). By contrast, the concentrations of CaO and MgO increase; this is because at 1050 °C, almost all of the CaCl_2 and MgCl_2 remain in the chlorination residue as liquid chlorides.

Table 2

Residual sample mass and lithium content as a function of reaction time and temperature.

Time (min)	Initial mass of the sample (mg)	Initial content of Li_2O (mg)	Residual mass of the sample (mg)	Mass loss ($\Delta m\%$)	Residual concentration of Li_2O w/w	Residual content of Li_2O (mg)
<i>T = 1000 °C</i>						
30	401.1	28.8	391.8	2.3	6.6	26.0
90	403.7	29.1	388.6	3.7	5.8	22.6
180	401.6	28.9	380.5	5.2	4.0	15.4
<i>T = 1050 °C</i>						
30	401.4	28.9	388.2	3.3	5.1	19.9
90	400.0	28.8	375.6	6.1	2.8	10.7
180	402.4	28.9	369.9	8.1	0.7	2.7
<i>T = 1100 °C</i>						
15	400.3	28.8	382.0	4.6	3.8	14.6
45	400.3	28.8	368.5	7.9	1.8	6.8
150	397.2	28.6	356.3	10.3	0.0	0.0

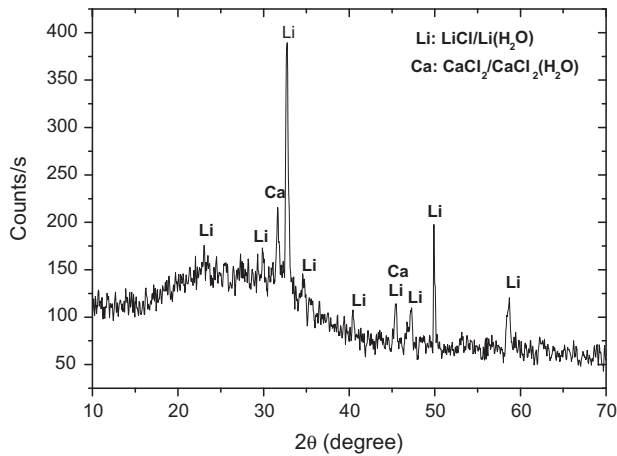


Fig. 7. XRD pattern of the chlorination products collected from the reactor.

Fig. 6 shows the results of the XRD analysis of the isothermal chlorination residues. The XRD data for the sample chlorinated at 1000 °C for 180 min (Fig. 6a) indicates that the main constituent in the sample is β -spodumene because after the chlorination roasting, 4% Li_2O remains in the solid residue. Quartz is also present in this sample but as a minor compound, which comes from the original spodumene ore. The XRD pattern for the sample chlorinated at 1050 °C for 180 min (Fig. 6b) shows a decrease in the intensity of the crystalline peaks corresponding to the β -spodumene phase; this is because the concentration of Li_2O in the residue dropped to a value of 0.7%. From this figure, one can also observe the appearance of the diffraction peaks corresponding to the phases of mullite (JCPDS 15-776) and cristobalite (JCPDS 76-941). The diffraction data of the sample chlorinated at 1100 °C for 150 min (Fig. 6c) led to identification of the phases of mullite and cristobalite, noticing the absence of the β -spodumene phase. This could be due to the completion of the lithium chlorination reaction. It is

worth highlighting that, in none of the chlorination residues, the crystalline phases corresponding to lithium chloride was identified by the XRD analysis.

At the end of the series of the isothermal chlorinations, the condensate accumulated in the cold areas of the tubular reactor was collected and analyzed by XRD (Fig. 7). These data show the presence of both anhydrous and hydrated LiCl , and other lower-intensity peaks, which agree with the major peaks of CaCl_2 . The presence of hydrated crystalline phases is due to the hydration process that occurs during the X-ray diffraction analysis since the chlorides generated by the chlorination of the mineral are very hygroscopic compounds. These results are consistent with the observations made in the thermodynamic section, i.e. lithium chloride is entrained by the gaseous stream as a volatile product. The presence of CaCl_2 in the product indicates that part of it is also volatilized but the amount of volatilization should be very low because the vapor pressure values of CaCl_2 at 1000, 1050, and 1100 °C are 2.25×10^{-5} , 5.92×10^{-5} , and 1.71×10^{-4} atm, respectively (HSC, 2002).

The SEM micrographs of the residual samples obtained from the chlorinations at 1000 and 1050 °C are shown in Fig. 8a and b, respectively. The comparison between these SEM images and that corresponding to the raw sample (Fig. 3b) indicates that the β -spodumene particles have been attacked and coated with smaller particles, which may correspond to the products of the chlorination reaction. Fig. 8c and d shows the micrographs corresponding to the residual sample obtained from the chlorination at 1100 °C. These micrographs show sintered particles with lengthened morphology (Fig. 8c) and other smaller particles with from spherical to amorphous shape (Fig. 8d).

The results of EPMA analysis on different points of the particle shown in Fig. 8a, indicate that the atomic concentration is similar in all of them, with an atomic ratio Si:Al close to 2:1. These data agree with the chemical composition of β -spodumene. EPMA analysis on particles from Fig. 8c and d indicates that these particles are different in their chemical composition. On one hand, the analysis on the lengthened particles indicates that they have an atomic

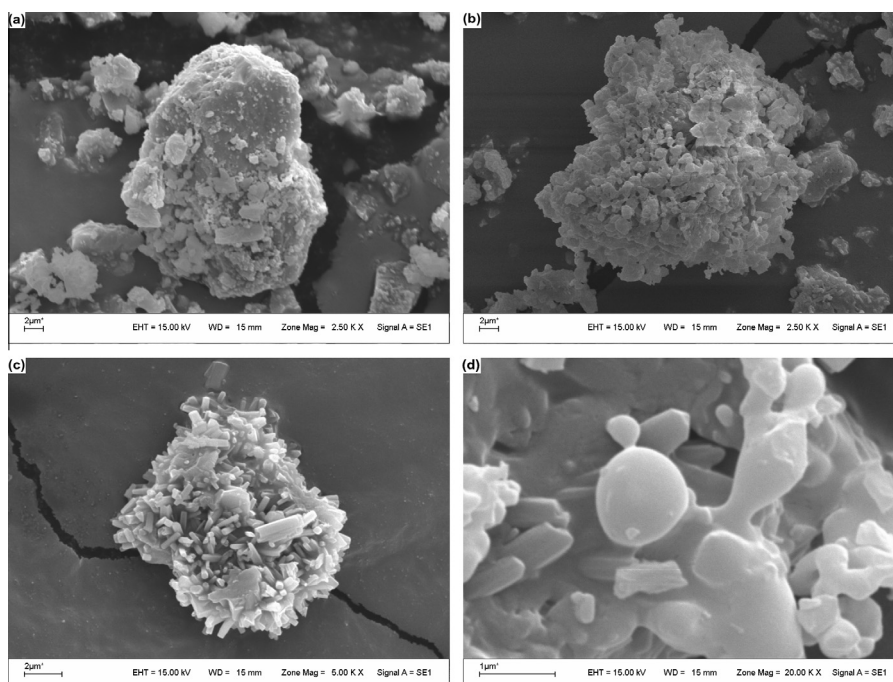


Fig. 8. SEM micrographs of the residual samples obtained from the chlorinations at: (a) 1000 °C, (b) 1050 °C, and (c and d) 1100 °C.

ratio Si:Al close to 1:3, which is coincident with that of mullite. On the other hand, the analysis on the in-between sintering of the lengthened particles and on the particles from amorphous to spherical shape showed a composition with high concentration of silicon and oxygen and low concentration of aluminum; these results agree with the chemical composition of SiO_2 , which may correspond to cristobalite according to the XRD results.

In light of these results, silicon and aluminum remain in the residue of the β -spodumene chlorination roasting, forming the crystalline phases of mullite and cristobalite, whereas lithium is extracted quantitatively as lithium chloride. Chlorine, which could come from lithium chloride produced by the chlorination reaction, was not detected in none of the solid residues by EPMA or by XRF. Therefore, while the reaction of β -spodumene with chlorine progresses, the lithium chloride generated is volatilized quantitatively.

The elimination of impurities from the spodumene, using the process of chlorination with chlorine gas, has been applied in industrial processes to purify the mineral through an adequate control of temperature (Garrett, 2004). In an actual process, the chlorination of β -spodumene to extract lithium as LiCl may consist in roasting the mineral with chlorine at temperatures lower than 1000 °C to eliminate the impurity of Fe_2O_3 first, followed by the chlorination roasting at temperatures higher than 1000 °C to extract lithium as LiCl. This product may be contaminated with the chlorides of the other impurities contained in the ore; to eliminate such chlorides, the next step would be the purification by distillation in a fractioning column (Habashi, 1986).

A previous stage of decrepitation at 1180 °C is required, so as to perform the extraction treatment. Lithium extraction through chlorination could be applied immediately after the decrepitation stage with an adequate control of the temperature in static beds, fluidized beds, or rotary kilns (Habashi, 1986). Also, it would be necessary to recycle Cl_2 . For such a purpose, the gas stream must be cooled down and condensed in a condenser. Thus, the residual Cl_2 is separated from the chlorides and can be recirculated to the reactor.

5. Conclusions

The results of the isothermal chlorination roasting of β -spodumene showed that the sample undergoes mass losses which increased markedly with the increase of the chlorination temperature.

According to the AAS and XRF analysis of the residual samples, and the XRD results of the chlorination product, the mass losses during the chlorination roasting is due to the volatilization of both lithium chloride and the chlorides formed by the impurities present in the spodumene ore. The extraction of lithium as lithium chloride is complete at 1100 °C and a reaction time of 150 min. This product should be further treated to eliminate the chlorides of the impurities that could accompany the ore by distillation.

The XRD, SEM and EPMA results of the residual samples showed that Si and Al are not chlorinated through this procedure, remaining in the solid residue in the phases of mullite and cristobalite.

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