Potassium chloride recovery from mechanically activated microcline through the chlorination roasting and leaching route

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Abstract: In this study, effects of mechanical activation in the chlorination roasting and water leaching route known as $CaCl_2$ process and developed for the production of potassium chloride (KCl) from potassium feldspar ores were studied. A microcline containing K-feldspar ore with 10.89% K_2O was first intensively dry milled by a planetary ball mill and mixed with calcium chloride ($CaCl_2$) and then roasted at temperatures up to 1000 °C to obtain KCl that will be finally dissolved by the water leaching. Potassium recovery by water leaching increased rapidly up to 800 °C. At higher temperatures, the recovery decreased fast due to the evaporation of KCl. According to the K recovery values per unit energy consumed, the optimum roasting temperature was determined as 750 °C and the milling time was 15 min. It was concluded that intensive milling causes mechanical activation of the microcline to reduce the chlorination roasting temperature, which triggers a rise in the K recovery by the water leaching.

Keywords: KCl, K-feldspar, chlorination roasting, mechanical activation, energy efficiency

1. Introduction

Natural sources of potassium chloride (KCl) around the world are scattered in the unfavourable and limited areas. For agriculture in K-poor areas, it is obligatory to use artificially produced fertilizers in the fields. Mainly depending on the scarcity of the KCl, there are many studies on the artificial KCl production from K-bearing silicate minerals like feldspars (Alyosif, 2021). Although no commercial scale facility has been reported so far, the studies on the artificial K salts production from microcline ($KAlSi_3O_8$) containing K-feldspar ores are generally divided into two classes: (1) direct leaching of the ore with minerals acids like HCl, HNO_3, or H_2SO_4, and (2) chlorination roasting of the ore with a chloride source like $CaCl_2$, $MgCl_2$ or NaCl, followed by the water leaching, known as $CaCl_2$ route (Jena et al., 2014; Samantary et al., 2019; Türk et al., 2021). However, in almost all studies in the literature, research has been done on the $CaCl_2$ route comprised of chlorination roasting, water leaching, and evaporation of pregnant salt solution to recover KCl. The conversion reaction of the K-feldspar at high temperatures is given in Eq. 1.

$$2KAlSi_3O_8 + CaCl_2 \rightarrow CaAl_2Si_2O_8 + 4SiO_2 + 2KCl \qquad (1)$$

The reaction temperature for conversion from $KAlSi_3O_8$ to KCl is in the range of 800-850 °C. At higher temperatures, there is a strong decrease in the K recovery by the water leaching but there is no K recovery from the roasted mixture at temperatures higher than 900 °C (Serdengeçti, 2018; Serdengeçti et al., 2019). The main reason for this is that KCl vaporises at these temperatures and leaves the furnace as flue dust, which was observed in many studies (Uysal, 2018, Alyosif, 2021, Alyosif et al., 2023). That is why the remaining material has no KCl content for recovering by the water leaching.

Unexpected variations in chlorination roasting temperature, thus, will cause KCl deficiency and/or absence in the roasted mixture. Reducing the roasting temperature without reducing the K recovery can be the solution to this problem. Mechanical activation (MA) to be performed before chlorination roasting can reduce the reaction temperatures below the vaporization temperature of KCl as there are many studies on the practice of mechanical activation by intensive milling as a suitable pre-treatment

to decrease the thermal characteristics of the minerals (Uysal et al., 2016; Birinci et al., 2017; Erdemoğlu et al., 2018, Alyosif, 2021, Aydemir, 2021).

Mechanical activation is defined as a process that increases the reaction ability of a solid in which chemical structure and formula stay stable (Smekal, 1942). In mechanical activation with intensive milling, there would be an increase in specific surface area and structural irregularity (Baláž, 1996), an increase in micro tensions (Baláž, 2000), amorphization of mineral crystals (Tkáčová et al., 1993), microtopographic changes (Tromans and Meech, 1999), the formation of new phases that are more prone to dissolution (Welham and Llewellyn, 1998), and a tendency to thermal reduction (Welham, 2002). Thus, these cases show that intensively milled ore is mechanically activated. During mechanical activation, the particle size of the mineral is reduced by milling, so defects in the crystal structure occur depending on the mechanical energy density. This creates fresh, clean surfaces that have not yet been exposed to any effect, and the result is a semi-stable species (Boldyrev, 2000). In addition, milling the ore under energy-intensive conditions causes the crystals of minerals to become amorphous by changing the crystalline properties, so it becomes more unstable. In particular, this instability is considered as a motivation that increases the ability of ore minerals to react to decrease the temperature dependent reactions or increase the speed of the metallurgical process such as calcination (Erdemoğlu et al., 2020) or leaching (Erdemoğlu et al., 2017; Barry et al., 2019).

In this study, it was aimed to mechanically activate the microcline in the ore to enable the chlorination roasting at lower temperatures than the vaporisation temperature of KCl. For this purpose, the specific energy spent and the K recovery were evaluated together to determine the ideal intensive milling time and roasting temperature conditions.

2. Materials and methods

2.1. Materials

K-Feldspar ore containing 65% microcline and 35% albite was obtained from Matel Inc. (Eskişehir, Turkey). All of the solids obtained during the study were characterized primarily by X-ray diffraction (XRD) analysis (İnönü University, IBTAM) using a Rigaku RadB type diffractometer. XRD patterns of microcline ore are given in Fig. 1. The radiation applied was CuKα ($\lambda = 1.5405$ Å) from a long fine focus Cu tube, operating at 40 kV and 40 mA. The samples were measured in the range of 2-80° with 0.02° steps at a rate of 2° per min. ICP-OES analysis (Table 1) as performed by Bureau Veritas (Canada) showed that the ore has 10.89% K_2O, and 2.56% Na_2O content, mainly derived from microcline and albite, respectively. $CaCl_2 \cdot 2H_2O$ as the chlorination agent to be used in the chlorination roasting experiments was provided from Turpil Ltd. Co. (Malatya, Turkey), with a 78% $CaCl_2$ content.
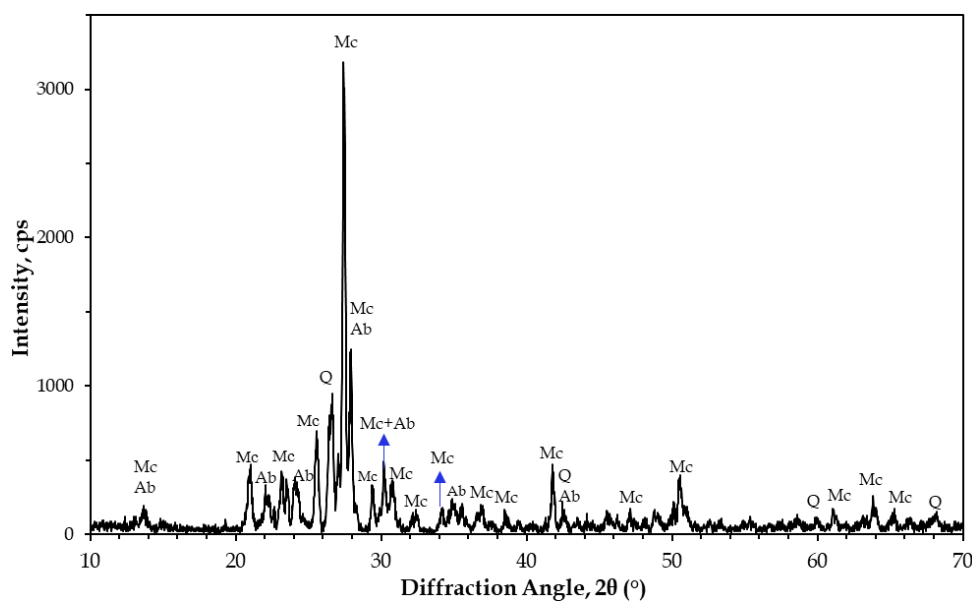

Fig. 1. XRD pattern of the ore (Mc: Microcline, Q: Quartz, Ab: Albite)

According to the XRD analysis of the sample (Fig. 1), the ore consists of mainly microcline (KAlSi$_3$O$_8$; JCPDS PDF No. 89-8572), albite (NaAlSi$_3$O$_8$; JCPDS PDF No. 09-0466), and quartz (SiO$_2$; JCPDS PDF No. 46-1045).

Table 1. Chemical analysis result of the feldspar ore used in the study

Major oxide	SiO$_2$	Al$_2$O$_3$	Fe$_2$O	MgO	CaO	Na$_2$O	K$_2$O	TiO$_2$	P$_2$O$_5$	LOI*
%	68.67	16.51	0.13	0.03	0.26	2.56	10.89	0.03	0.02	0.9

*LOI.: Loss on ignition (1000 °C)

2.2.   Methods

2.2.1. Intensive milling

For intensive milling, Fritsch Pulverisette 6 Mono Mill (Germany) model planetary ball mill designed with high energy efficiency and air cooling, together with a tungsten carbide (WC) bowl (250 cm³) containing WC balls (Φ =10 mm) were used. The use of milling media that will ensure that the chemical content of the original ore does not change is important in terms of product purity. Al$_2$O$_3$ contamination will occur when porcelain or ceramic is selected as the milling medium material, and Fe and/or FeO contamination will occur when steel, etc. ferrous materials are selected. Based on this fact, milling processes were carried out using tungsten carbide (WC) mortars and balls, known as "hard metal" in experimental studies, with a Mohs hardness of approximately 9, in order to avoid the formation of the said impurities. Thus, during the mechanical activation processes, the milling medium was prevented from being eroded and mixed with the ore, and the Al$_2$O$_3$ percentage in the original ore was preserved. The intensive milling was carried out in dry mode at 400 rpm mill speed and ball/ore ratio of 20, up to 120 min of milling time.

2.2.2. Chlorination roasting

CaCl$_2$ used as a chlorine source to obtain KCl, was mixed with unmilled or milled ore samples just before chlorination roasting. The mixture was prepared as the ore to CaCl$_2$ mass ratio is 1/1.1 based on previous studies (Alyosif, 2021). The roasting was carried out in non-isothermal conditions at temperatures between 500 °C and 1000 °C for 1 hour in a muffle furnace (Protherm PLF120/5). The roasting time, which has been used in most studies in the literature, has been preferred (Xie, 2013; Samantary, 2019; Serdengeçti, 2019).

2.2.3. Leaching

KCl in the roasting product was dissolved by leaching only in water at room temperature and stirred with a magnetic stirrer at 400 rpm for 30 min. Required amount of water was calculated to dissolve the maximum amount of KCl and NaCl that can be found in the roasting product. Thus, in the experimental studies, the water/ore ratio was kept constant as 50 cm³ water per 10 g of the chlorination roasting product. After the leaching, filtration was carried out to separate undissolved solids from and to obtain the pregnant leaching solution (PLS). Filtering the PLS was done by vacuum filtration method using filter paper providing slow flow. Potassium content of the PLS was determined by using Thermo Scientific 3000 model flame atomic absorption spectrophotometer (FAAS). K recovery was calculated using Eq. 2.

$$\text{K recovery (\%)} = \frac{\text{Amount of K in the PLS, g}}{\text{Amount of K in the ore, g}} \times 100 \tag{2}$$

The heat capacity of any substance is the amount of heat required to raise the temperature of that substance by one degree. The specific heat energy transferred to the furnace in the roasting process was calculated with Equation 3.

$$Q = m * C_p * \Delta T \tag{3}$$

where m is the mass in kg, Cp is the heat capacity at constant pressure, and ΔT is the difference between the initial medium and the final temperature. The specific heat capacity of microcline at 298.15 K was calculated as 0.74 J/g.K (Openshaw, et al. 1979; Hemingway, et al. 1981).

The specific milling energy (SE, kJ/kg) consumed per unit amount of ore in the intensive milling process was calculated with Equation 4 (Pourghahramani and Forssberg, 2007).

$$SE = \frac{m_B}{m_S} * a * n * t_M * D \tag{4}$$

m_B: mass of the milling medium (balls) (kg), m_S: mass of ore to be milled (kg), a: theoretical acceleration of the balls (26.41 m/s^2 for the planetary mill used), n: mill rotation speed (1/s), t_M: milling time (s) and D: mill diameter (m). In this formula, the specific energy unit is kJ/kg and kWh conversion are made from here (1 kJ: 2.78*10^{-4} kWh).
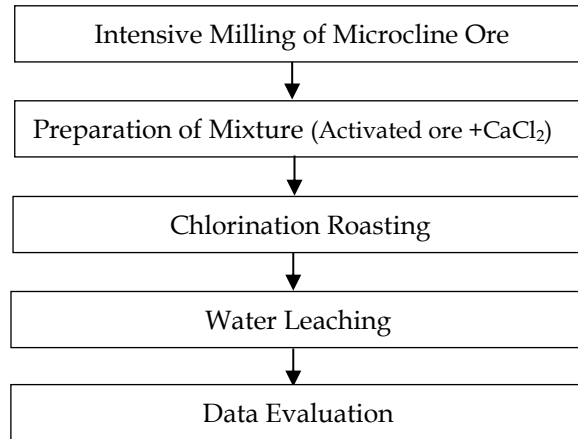
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│       Intensive Milling of Microcline Ore     │
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│  Preparation of Mixture (Activated ore +CaCl₂) │
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│            Chlorination Roasting              │
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│               Water Leaching                  │
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│               Data Evaluation                 │
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Fig. 2. Experimental flow chart for K recovery from microcline ore

3.   Results and discussion

3.1. Intensive milling experiments

The XRD patterns in which the milled ore at different times (0-120 min) were compared with the unmilled ore are given in Fig. 3.
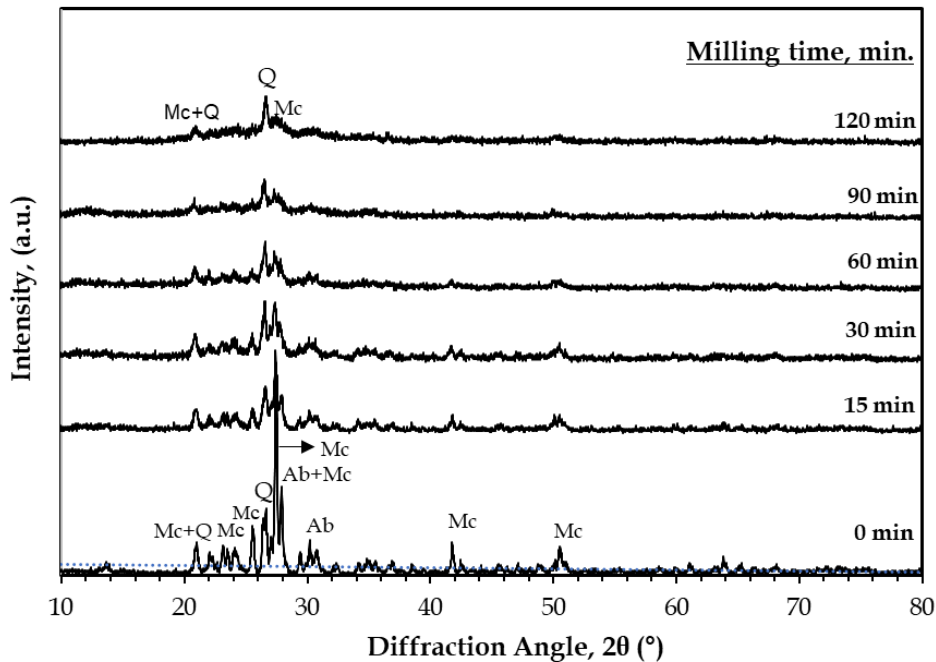

Fig. 3. Comparison of the XRD patterns of the ores milled at different times with unmilled ore (Mc: Microcline, Q: Quartz, Ab: Albite) (Alyosif, 2021)

When the XRD patterns of the unmilled and milled ore samples (Fig. 3) were considered, it was observed that compared to the unmilled ore, the peak intensity of microcline and albite minerals decreased with the increasing milling time. Peaks of albite (2θ: 27.98° and 30.22°) drastically reduced by milling for 90 min. On the other hand, microcline peaks at typical diffraction angles very nearly disappeared by milling for 120 min. The resistance of quartz to intensive milling was observed for a longer time due to its higher Mohs hardness compared to microcline and albite. In contrast to microcline and albite, only quartz in crystalline form was still observed in the ore after 120 min.

3.2. Chlorination roasting experiments

The XRD patterns of the chlorination roasting products of the 15-minute intensively milled ore mixed with $CaCl_2$ are given in Fig. 4. The temperature-dependent mass loss variation of unmilled and milled ore and $CaCl_2$ mixtures is given in Fig. 5.
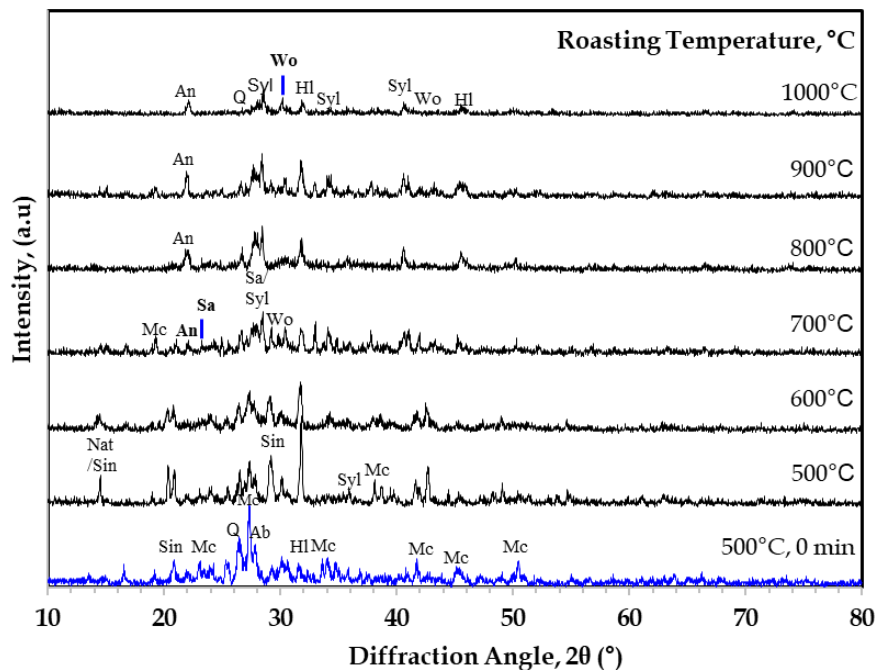
Fig. 4. Comparison of the XRD patterns of chlorination roasting products of 15 min. milled ore (Mc= Microcline, Q= Quartz, An: Anorthite, Hl: Halite, Nat: Natrolite, Sa: Sanidine, Sin: Sinjarite, Syl: Sylvite, Wo: Wollastonite)

In Fig. 4, it is seen that mineralogical changes begin to occur in the mixture of ore and $CaCl_2$ after roasting at 500 °C. The presence of KCl (sylvite) and NaCl (halite) at 500 °C indicates that the transformation of microcline and albite has begun, while the presence of microcline and sinjarite ($CaCl_2$) has not yet ended. The XRD peaks of albite disappeared at 700 °C due to the formation of sodium sanidine ($(Na,K)AlSi_3O_8$) at this temperature (Freeman et al., 2008) and the XRD peaks of anorthite also became more visible. Moreover, peaks of anorthite, wollastonite, and sylvite started to be very-well observed, and the peaks intensities increased with the increasing roasting temperature. Apart from anorthite, quartz, and sylvite, which are expected to occur according to Eq. 1, the occurrence of wollastonite in the roasting product of intensive milled ore, for instance, at 700 °C, can be explained as follows: The free quartz in the ore or was slightly mechanically activated during the intensive milling and reacted with calcium species in the system to form wollastonite. This temperature is quite low for the formation of wollastonite (Göktaş, 2012). Likewise, the XRD traces of wollastonite was not found in the roasting product of the unmilled ore at these temperatures.

In Fig. 5, the highest mass loss at 500 °C was at 120 min (~9%) and the lowest mass loss was at 0 min (~7%). The minor mass loss measured at 500 and 600 °C is thought to be due to dehydration of $CaCl_2 \cdot 2H_2O$ mixed with the ore. Because, according to the TG analysis by Essen et al., (2009), $CaCl_2 \cdot 2H_2O$ loses 28% of its mass up to 150 °C and turns into $CaCl_2$, and after this temperature, no further mass loss

occurs up to 300 °C. When the temperature was 650 °C, the mass loss in the mixtures reached 10% on average. After this temperature, the amount of mass loss increased rapidly. It has been evaluated that KCl and NaCl evaporate away from the environment as the main reason for the mass loss from 750 °C. Because KCl starts to melt at 771.4 °C and NaCl at 802.1 °C (Schindler and Schöneich, 2021). On the other hand, the mass loss profiles of the mixtures with 60 min. and longer times milled ores differ significantly from the previous ones, beginning from 650 °C. However, the continuance of the upward trend in their mass loss profiles may be another sign that mechanical activation also lowers the removal temperature of KCl by evaporation.
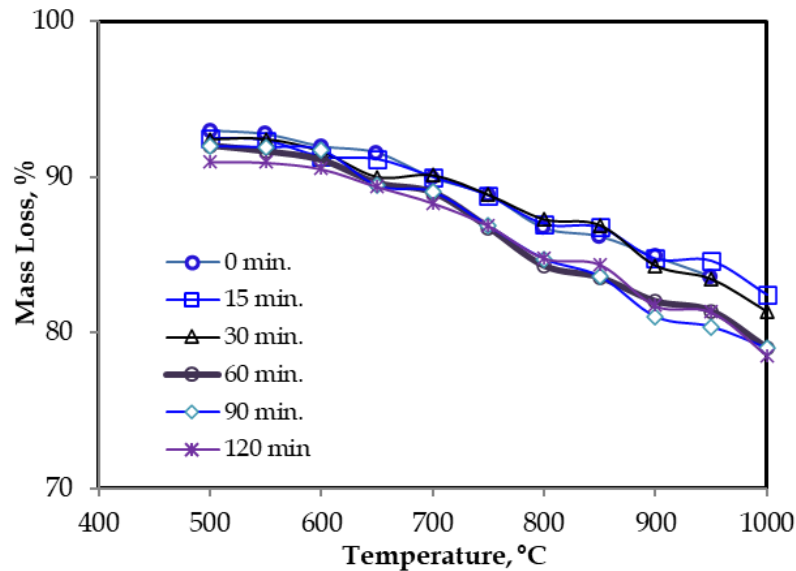
Fig. 5. Temperature dependent mass loss variation of milled ore and $CaCl_2$ mixtures

3.3. Water leaching experiments

The products of the chlorination roasting were leached with water to dissolve KCl formed by the roasting to reveal the effects of mechanical activation and roasting temperature, as seen in Fig. 6 which illustrates the variation of K recovery by water leaching with respect to mechanical activation time and chlorination roasting temperature.

It is seen from Fig. 6 that regardless of the time applied, the intensive milling gave rise to shift the roasting temperature to temperatures as low as 750 °C to achieve the K recovery almost identical to that of the unmilled (0 min.) ore. It also appears from Fig. 6 that the K recovery values from the chlorination
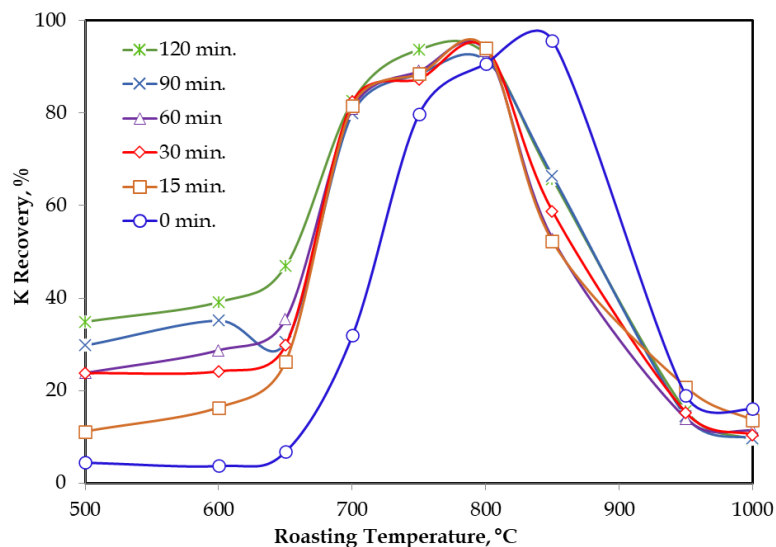

Fig. 6. Variation of K recovery with respect to milling time and chlorination roasting temperature

roasting products of the intensively milled ores at different times starting from the roasting temperature of 700 °C are almost identical. However, the effect of intensive milling time on the K recovery was evident at lower roasting temperatures. Mechanical activation obtained by intensive milling for 120 min. resulted in about 38% K recovery even with roasting at 500 °C. Whereas, for the same temperature, the K recovery from the unmilled ore is about 4%.

Besides, a sharp increase at the K recovery up to 800 °C and a sharp decrease after this temperature were observed. The highest K recovery in the unmilled ore sample was obtained at 850 °C, while it was at 800 °C for all of the intensively milled ore samples. The K recovery increased significantly with the increase in the milling time. The K recovery of unmilled ore roasted with $CaCl_2$ at 700 °C was 31.95%, while it increased dramatically to 81.57% for 15 min milled ore roasted at identical conditions. The K recovery values decreased after 800 °C as the milling time increased. The K recovery from the unmilled ore's chlorination roasting product which 95% at 850 °C decreased to about 10% at 950 °C. This can be attributed to the evaporation of KCl and NaCl at 771.4 °C, 802.1 °C respectively, as reported in the literature (Uysal, 2018; Schindler and Schöneich, 2021). On the other hand, all of the early formed KCl and NaCl can be easily leached by water because mechanical activation promotes the formation of KCl and NaCl at lower roasting temperatures, at about 800 °C. The K recovery was at the level of 50-60% for the chlorination roasting products of the milled samples. This decrease in the K recovery suggests that mechanical activation also promotes the evaporation of KCl at early temperatures, even for the intensively milled ore samples.

3.4. Energy analysis

Specific Milling Energy: The K recovery and specific milling energy variation as a result of the leaching of the intensive milled product at different times and roasted at 750 °C are given in Fig. 7. In the same conditions (at 750 °C), the K recovery per unit specific energy (J/kg) consumed relative to the roasted product is given in Fig. 8. The K recovery per unit specific energy expended (J/kg) was calculated by dividing the percentage of K recovery by the specific energy expended for each milling time.
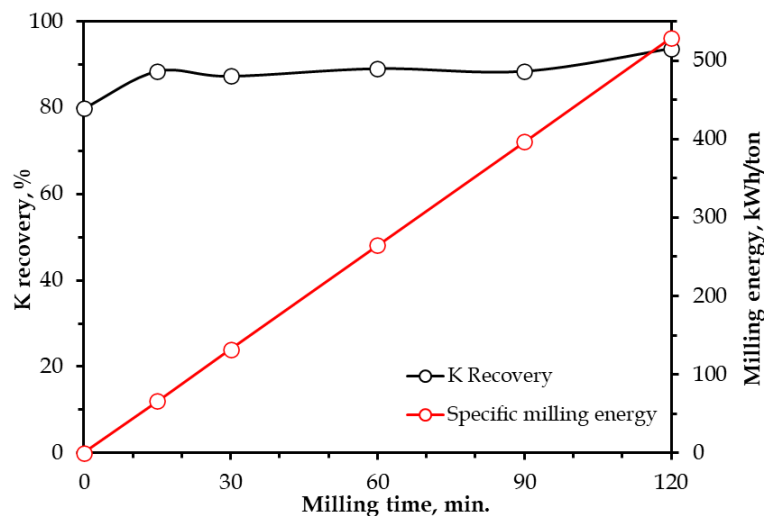

Fig. 7. Variation of K recovery and specific milling energy depending on milling time

According to Fig. 7, the specific milling energy increased linearly as the milling time increased. In the leaching of the product roasted at 750 °C, the K recovery increased up to 15 min., and after 15 min, no significant increase was observed in the K recovery value. As a result of milling for 15 minutes, 88.43% K recovery is consumed versus 66.11 kWh, while after 60 minutes of milling, 264.44 kWh energy is consumed for 89.04% K recovery. According to these results, 198.33 kWh more energy is required for 0.61% more K recovery.

In determining the ideal milling time, the specific milling energy value spent per unit K recovery was evaluated together. Accordingly (Fig. 8) it is thought that 15 min of milling is more ideal since K recovery per unit energy is higher.

Specific Heat Energy: The specific heat energy transferred to the furnace in the roasting process is calculated with the help of Eq. 1. K recovery was evaluated together with thermal energy to determine the ideal roasting temperature. The K recovery per unit specific energy expended (J/kg) was calculated by dividing the percentage of K recovery by the specific energy expended for each milling time (Fig. 9). The K recovery per unit specific energy (J/kg) consumed is given in Fig. 10.
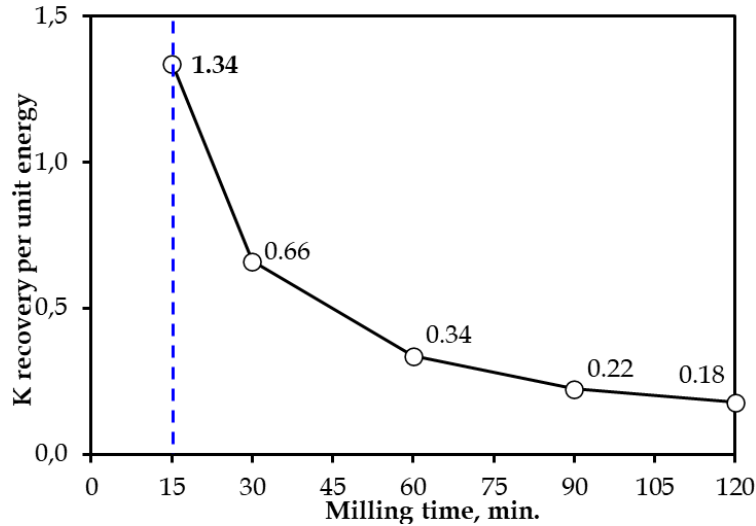

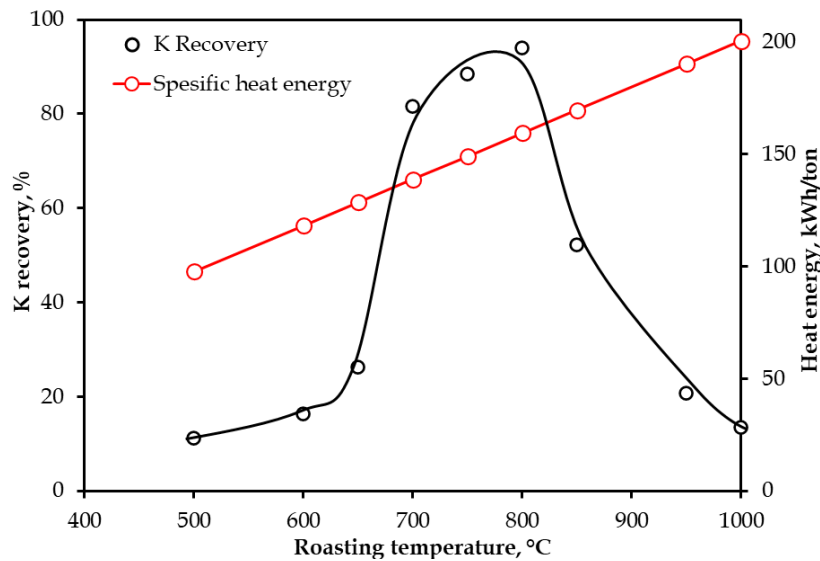Fig. 8. K recovery per unit specific energy consumed

Fig. 9. Variation of K recovery and specific heat energy depending on roasting temperature

Fig. 9 shows the K recovery and specific energy values as a result of roasting the intensive milled ore at different temperatures for 15 min. According to this Fig., although the temperature with the highest K recovery is 800 °C, the specific energy spent and the K recovery were evaluated together. Accordingly, 159.43 kWh energy is required to obtain 94.01% K recovery at 800 °C, while 149.15 kWh energy is required for 88.43% K recovery at 750 °C. According to these results, although the K recovery is high at 800 °C, it is predicted that 750 °C is more appropriate when evaluated together with the energy consumption. To support this view, the K recovery value per unit energy has been calculated.

According to Fig. 10, it is seen that the temperature with the highest K recovery per unit specific energy consumed is 750 °C. Thus, it has been determined that 750 °C is the ideal roasting temperature.

The unit energy costs in the determined ideal conditions (15 min MA, 750 °C roasting) are given in Table 2. Other operating costs (such as mineral processing, leaching) are not given here. This study is laboratory-scale and serves as an example for pilot studies.
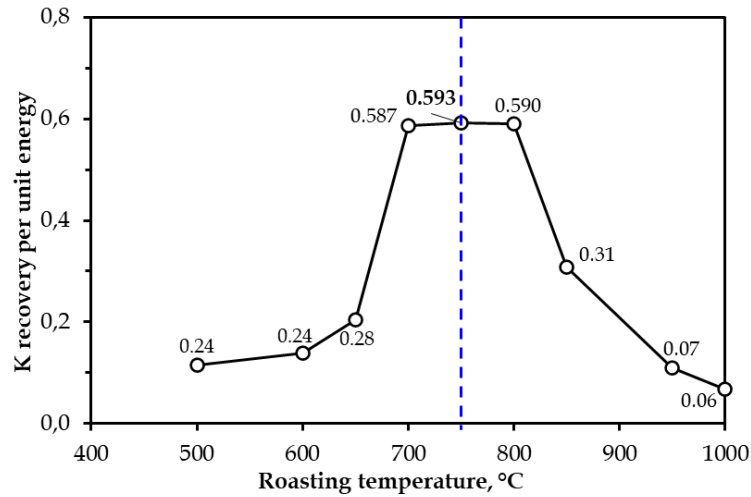
Fig. 10. K recovery per unit specific energy consumed depending on the roasting temperature

Table 2. Energy consumption, cost and K recovery values depending on processes

Cost	Roasting	MA-Roasting
Energy consumption, kWh/ton	149.15	215,16 (66.11+149.15)
K recovery, %	79.83	88.43
Energy Cost*, $	22.37	32.27

*The average electricity tariff per kWh of US business for March 2023 is 15 cents (BLS, 2023).

According to Table 2, although the energy cost of the roasting process is more suitable, the K recovery efficiency is 8.6% lower than the MA-Roasting process. Accordingly, it would be more accurate to determine the appropriate method by making a detailed economic analysis.

4. Conclusions

In this study, effect of mechanical activation applied before the chlorination roasting step on K recovery from microcline using $CaCl_2$ route was investigated. Optimum intensive milling time and roasting temperature were determined by evaluating both the K recovery and energy consumption. Accordingly, the following conclusions were reached.

1. With the increase of roasting temperature, microcline and albite were transformed into water-soluble KCl and NaCl, and water-insoluble silicate species as anorthite.
2. Mechanical activation promoted the formation of KCl, NaCl and anorthite at low temperatures, which in turn promoted its reaction with SiO_2 found in the ore or produced during chlorination roasting, resulting in the formation of wollastonite.
3. K recovery significantly increased with intensive milling. While the K recovery was 31.95% by roasting the unmilled ore at 700 °C, the K recovery was 81.57% as a result of intensive milling for only 15 minutes and roasting at 700 °C. In addition, it was observed that similar K recovery values were achieved in the 50 °C lower temperature region with intensive milling.
4. According to the K recovery values per unit energy consumed, the optimum roasting temperature was determined as 750 °C and 15 min of intensive milling time is enough.
5. KCl was formed at lower roasting temperatures with the effect of mechanical activation obtained by intensive milling. Thus, high K recoveries were obtained from chlorination roasting products obtained at lower temperatures.

Acknowledgments

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