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Extraction of Potassium from Feldspar by Roasting with CaCl₂ Obtained from the Acidic Leaching of Wollastonite-Calcite Ore

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Abstract: Potassium, which is included in certain contents in the structure of K-feldspar minerals, has a very important function in the growth of plants. Turkey hosts the largest feldspar reserves in the world and is by far the leader in feldspar mining. The production of potassium salts from local natural sources can provide great contributions both socially and economically in the agriculture industry along with glass production, cleaning materials, paint, bleaching powders, and general laboratory purposes. In this study, potassium extraction from K-feldspar ore with an 8.42% K₂O content was studied using chloridizing (CaCl₂) roasting followed by water leaching. Initially, to produce wollastonite and calcite concentrates, froth flotation tests were conducted on wollastonite-calcite ore after comminution. Thus, wollastonite and calcite concentrates with purities of 99.4% and 91.96% were successfully produced. Then, a calcite concentrate was combined with hydrochloric acid (HCl) under optimal conditions of a 1 mol/L HCl acid concentration, a 60 °C leaching temperature, and a 10 min leaching time to produce CaCl₂. To bring out the importance of roasting before the dissolution process, different parameters such as roasting temperature, duration, and feldspar—CaCl₂ ratios were tested. Under optimal conditions (a 900 °C roasting temperature, a 60 min duration, and a 1:1.5 feldspar—CaCl2 ratio), 98.6% of the potassium was successfully extracted by the water leaching process described in this article.

Keywords: potassium; calcium chloride; K-feldspar; wollastonite; roasting-leaching

1. Introduction

Potassium is an element that occupies an important place in human life and is widely used in different industries as a component of potash. Potassium fertilizer is an essential nutrient, particularly for fruit formation and development. It accelerates plant growth and increases crop yields. In addition to the fertilizer industry, large amounts of potassium salts are used annually to produce glass, cleaning materials, paint, and bleaching powders and for general laboratory purposes [1]. The consumption and cost of potassium salts vary annually and are between 25 and 30 million tons and USD 300 and 500, respectively. In Turkey, potassium salts (potassium chloride (KCl) and potassium sulfate (K₂SO₄)), which are needed by the chemical and fertilizer industries are provided by imports.

Water-soluble potash is used to produce potassium salts; however, the increase in the need for potassium salts and the decrease in water-soluble potash sources have led the producers to find an alternative source such as K-feldspars. Feldspars constitute more than 60% of the earth's crust and contain microcline, albite, and aluminum silicate with varying concentrations (5–12%) of potassium oxide (K₂O) [2–4]. Turkey has approximately 14% of the world's high-quality feldspar reserves. While the most valuable sodium feldspar deposits are located in Western Anatolia (Çine, Milas, Yatağan, and Bozdoğan regions), the majority of K-feldspar deposits are found in the Kırşehir massive [5,6]. In addition to large K-feldspar reserves, there is also a significant amount of wollastonite-calcite deposits in the Kırşehir–Buzlukdağı region. About 4 million tonnes of potash are imported for agricultural



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). and industrial activities each year in Turkey. KCl and CaCl₂ salts, which are the main raw materials of the fertilizer industry, can be obtained from feldspar and wollastonite minerals, respectively. Obtaining these high value-added products from different industrial raw materials in the same geographical region significantly increases resource use and efficiency while reducing costs.

Wollastonite and calcite minerals, which are used in ceramics, plastics, metallurgy, paint, and other industries are usually found in the same deposits. The most common method for separating these two industrial minerals is flotation. As wollastonite has a needle-like structure, the flotation process can be performed in the presence of collectors such as amines or fatty acids. In a study by Kangal et al., the best result was obtained at pH 6 and 1500 g/t K-oleate dosage. Under these conditions, a calcite concentrate assaying 55.89% CaO, 0.35% SiO₂, 0.03% Fe₂O₃, and 42.30% loss on ignition (LOI) was produced. At the same time, a wollastonite concentrate containing 52.71% SiO₂, 44.65% CaO, 0.44% Fe₂O₃, and 0.60% LOI was obtained [7]. Several investigations have been carried out to separate wollastonite from calcite-rich wollastonite ore [8–10]. Çinku and co-authors indicated that a marketable wollastonite concentrate could be produced by flotation following magnetic separation from wollastonite ore containing 40% CaO and 48% SiO₂. As a result of the flotation tests at pH 9, the optimal flotation conditions were determined as 1000 g/t of collector dosage, 1.7 kg/t of sodium silicate, and 500 g/t of caustic soda. Using oleic acid, a concentrate containing 54.43% SiO₂, 41.44% CaO, 0.59% Fe₂O₃, and 0.97% loss on ignition was obtained. In addition, when oleic acid is used, a concentrate assaying 57.10% SiO₂, 40.88% CaO, 0.30% Fe₂O₃, and 0.13% loss on ignition was produced [9]. In another important study by Ravi et al., a 97% purity wollastonite concentrate was obtained from low-content wollastonite ore through the flotation and magnetic separation methods. In the flotation experiments, a particle size of $100 \ \mu m$ and a sodium oleate concentration of 1 kg/t were found to be optimal, and a wollastonite concentrate (about 95% purity) with 2.64% iron content and 0.4% ignition loss was produced [9]. In the study by Bulut and co-authors, a wollastonite (about 87.9% purity) concentrate containing 0.44% Fe₂O₃ and 52.71% SiO₂ with 0.60% loss on ignition was obtained using 1500 g/t of potassium oleate. In addition, a calcite concentrate containing 99.8% CaCO₃ was recovered as a by-product with 85.4% recovery [10].

CaCl₂ is an essential component for obtaining potassium salts from feldspar. Although it is not conventional to treat wollastonite with acid to produce CaCl₂, acidic digestion of wollastonite can be used as a step in the carbonation pathway for CO₂ sequestration. Zhang et al. [11] studied the leaching of wollastonite at various hydrochloric acid (HCl) concentrations and temperatures. It was reported that 81.9% and 96.1% of calcium were dissolved using 1 and 4 mol/L HCl, respectively. By maintaining a 4 mol/L HCl concentration, constant leaching tests were performed at different temperatures (40, 60, 80, and 90 °C) and the calcium dissolutions values were achieved as 91%, 96.9%, 97.1%, and 97.5%, respectively.

In the literature, many studies have been carried out to produce KCl from run-of-themine feldspar ore using pure CaCl₂ salt. Using K-feldspar ore and pure CaCl₂, roasting tests were carried out in a high-temperature furnace at various temperatures for 1 h. After the roasting was completed, the leaching process was performed under the constant conditions of a 10% solid-in-pulp ratio, a temperature of 60 °C, and a 500 rpm mixing speed [12]. As a result, a 98.3% potassium extraction was obtained with the dissolution process after roasting at 850 °C. In another study by Kangal et al. [13], a 90% dissolution efficiency was achieved at a 15% solid-in-pulp ratio and at a 40 °C temperature.

Yuan et al. [14] reported a 91% potassium dissolution (under the leaching condition of 70 °C, 30 min, and a solid–liquid ratio of 1:50) from a 50–75 μ m potassium feldspar fraction with a 13.25% K₂O content by roasting at 900 °C for 40 min with a 1:1.15 CaCl₂– potassium feldspar ratio. Serdengeçti et al. [15] investigated the production of KCl from a potassium feldspar ore containing 9.69% K₂O and succeeded in dissolving 99.8% of potassium through water leaching (a 60 °C temperature and a 120 min leaching time) following the roasting process (1:1.5 potassium feldspar—CaCl₂ ratio, 850 °C of temperature, and 60 min of roasting time). Samantray et al. [16] investigated the effects of the CaCl₂ amount, roasting temperature, leaching time, and temperature on KCl production from feldspar ore containing 11.64% K₂O. After roasting at 900 °C and with a ratio of 1:1 CaCl₂—feldspar, more than 80% of potassium was dissolved after 30 min. Tanvar and Dhawan [17] investigated the effects of additives, roasting temperature, and time on the recovery of potash from feldspar containing 9.67% K₂O. Roasting experiments were carried out at 950 °C for 60 min, and CaCl₂ was used as the slag material. At the end of citric acid leaching at room temperature, a 1:10 solid–liquid ratio, and 60 min, a 95% potassium dissolution efficiency was achieved. Samantray et al. [18] investigated the extraction of potassium from feldspar ore containing 11.64% K₂O using eggshell powder as a calcium source. Feldspar and eggshells were ground together below 45 µm and roasted at 900 °C for 30 min with a 1:1.8 eggshell—feldspar ratio. Then, this mixture was leached with HCl for 30 min at room temperature and a 99% potassium recovery was obtained.

Today, the main approach of beneficiation and production is based on a circular economy and environment. In this study, potassium recovery from K-feldspar ore was achieved using CaCl₂ produced from wollastonite-calcite ore in the same region. In the first part of this study, wollastonite-calcite ore was subjected to flotation tests to produce wollastonite and calcite concentrates. High-grade CaCl₂ was produced through a HCl acid treatment using a calcite concentrate obtained as a result of flotation. Second, roasting followed by the water leaching process was applied to K-feldspar with CaCl₂. With this original and innovative work, the extraction of potassium using wollastonite—calcite and K-feldspar ores located in a similar mineralization zone will be possible, and these local natural resources can be brought into the economy.

2. Materials and Methods

2.1. Material and Characterization

The wollastonite-calcite and potassium feldspar ores were obtained from the Buzlukdagi Region, Kırşehir, Turkey. The representative ore samples were crushed below 2 mm using jaw, cone, and roller crushers, and wet sieving was performed to determine the particle size distribution. According to the results, the d_{80} size of wollastonite and feldspar samples were found to be 1.2 and 0.5 mm, respectively. The BSE (Back-Scattered Electron) images of samples are given in Figure 1. The chemical contents of the samples (Table 1) were determined by inductively coupled plasma (ICP) at Activation Laboratories Ltd., Hamilton, ON, Canada. To determine the loss on ignition of the samples, the samples were placed in crucibles and weighed. After, they were kept in an oven at 100 °C overnight to remove moisture. Then, the temperature was raised to 550 °C, and the organic materials were completely removed after 4 h. The carbonate structure turned into oxides at 1000 °C after 2 h, and the ratio of the initial and final weight difference to the feed represented the ignition loss.



Figure 1. BSE images of wollastonite-calcite and feldspar samples.

Commound	Content, %				
Compound	Wollastonite-Calcite	K-Feldspar			
SiO ₂	28.00	62.30			
Al_2O_3	1.91	18.70			
Fe_2O_3	0.45	1.89			
TiO ₂	0.04	0.20			
Na ₂ O	0.47	4.32			
K ₂ O	0.40	8.40			
CaO	48.20	2.16			
MgO	1.15	0.26			
P_2O_5	0.03	0.09			
SrO	0.04	0.14			
MnO	0.03	0.06			
LOI	20.80	1.83			

Table 1. Chemical analyses of the representative samples.

LOI: Loss on ignition.

An X-ray diffraction (XRD) analysis was conducted with a copper X-ray-sourced Panalytical X'Pert Pro diffractometer (Malvern Panalytical Ltd., Malvern, UK). PDF-4/Minerals International Centre of Diffraction Data (ICDD) software (PDF-4/Minerals, ICDD, Delaware County, PA, USA) was used for mineral characterization. The crystals' mineral phase ratio was determined through the Rietveld method. XRD patterns of wollastonite-calcite and feldspar samples are shown in Figures 2 and 3, respectively. According to the results, the secondary mineral was calcite in the wollastonite-calcite sample and the feldspar sample mostly consisted of potassium feldspar.



Figure 2. X-ray diffraction patterns of the wollastonite-calcite sample.

The chemical and mineralogical properties of samples were defined with a differential thermal analysis (DTA) and the thermogravimetric analysis (TGA) equipment's STA 449 F3 Jupiter[®] thermal analyzer (NETZSCH, Selb, Germany). DTA and TGA curves of wollastonite-calcite, calcite concentrate (from flotation), and feldspar samples are illustrated in Figures 4–6, respectively.



Figure 3. X-ray diffraction patterns of the potassium feldspar sample.





As can be seen in Figure 4, a loss of 21% of weight occurs in the wollastonite ore as the temperature rises from 700 °C to 950 °C. The main reason for this loss is calcite, which constitutes more than 50% of the structure of the ore. The DTA and TGA results shown in Figure 5 indicate that a significant amount of structural deterioration occurred at 900 °C. Only 0.6% of weight loss was determined in the feldspar ore sample between 400 and 800 °C (Figure 6). Partial structural deterioration was encountered at 700–750 °C. This small loss might be explained by probable carbonate components in the ore.



Figure 5. DTA–TGA curves of the calcite concentrate.



Figure 6. DTA-TGA curves of the potassium feldspar sample.

2.2. Methods

Calcite must be treated with hydrochloric acid to produce $CaCl_2$. Calcite reacts with HCl to form $CaCl_2$ and releases CO_2 and H_2O as by-products (Equation (1)) [19].

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + CO_2 + H_2O \tag{1}$$

The potassium dissolution process can be finished by mixing the CaCl₂ obtained in the first equation with K-feldspar ore in certain proportions and by subjecting them to

roasting and leaching processes. The mixture of feldspar and $CaCl_2$, which is exposed to a temperature higher than the decomposition temperature during the roasting process, reacts to form KCl salt. On the other hand, quartz and anorthite minerals are produced as by-products (Equation (2)) [14].

Since the KCl salt obtained as a result of this process is easily soluble, it can be taken into a solution by leaching with water.

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

In the first step, wollastonite-calcite ore was subjected to comminution (below 75 μ m) and then froth flotation was used to separate calcite and wollastonite. An amount of 300 gr of wollastonite-calcite ore was subjected to self-aerated Denver flotation equipment with a 1.5 L cell volume and a 1200 rpm mixing rate. The conditions of the flotation tests are given in Table 2. While calcite particles were floated with the help of a collector, wollastonite particles were not activated and taken as the sinking product. Afterward, the calcite concentrate was subjected to HCl acid leaching (Figure 7). Different HCl concentrations (0.25, 0.5, 2, 3, 4, and 6 mol/L), temperatures (25, 40, 60, and 80 °C), and leaching times (5, 10, 15, 30, 60, 90, and 120 min) were investigated to determine the optimal treatment conditions with a constant 1:2 solid–liquid ratio. After obtaining the pregnant solution, CaCl₂ was precipitated by evaporation. Second, the CaCl₂ sample obtained from the calcite concentrate was ground and mixed in an agate mortar with potassium feldspar in a 1:1.5 feldspar–CaCl₂ ratio until it had a homogeneous appearance. Afterward, 7.5 g of the mixture was spread in a thin layer on four porcelain crucibles (diameter: 8 cm) and the roasting tests were performed in a Protherm brand PLF 130/6 furnace at elevated temperatures of 800, 850, 900, and 950 °C for 1 h. After roasting, water leaching was performed for 2 h under constant conditions, namely a 10% solid-in-pulp ratio, a 60 °C temperature, and a 500 rpm mixing speed.

Table 2. The conditions of the flotation experiment.

Particle Size	-74 μm
рН	6.0
KOI Dosage	300 + 300 + 300 + 300 + 300 g/t
Conditioning Time	$10 + 5 + 5 + 5 + 5 \min$
Flotation Time	2 + 2 + 1 + 1 + 1 min

After acid leaching, titration was used to determine HCl consumption. A 0.01 mol/L NaOH solution was prepared and transferred into a 10 mL burette. A beaker containing 30 mL of the test solution was placed under the burette. Four drops of phenolphthalein (a color-change indicator) were added to the solution, and the burette faucet was opened enough to allow a single droplet to pass. A 0.01 mol/L NaOH solution was mixed into the solution until it turned pink, at which point the faucet was closed, and the spent NaOH was detected. Lab-scale vacuum equipment with fine filter paper (Sartorius brand 391 code, blue label) was used for liquid–solid separation and the filtered cake was dried at 70 °C for approximately 24 h. The elemental analysis of the liquid was provided by the atomic absorption spectrometer (Varian brand AA240FS, Palo Alto, CA, USA).



Figure 7. General flowchart of the chemical processing following flotation.

3. Results and Discussion

3.1. Flotation of Wollastonite-Calcite Ore

To produce $CaCl_2$ for the roasting and leaching experiments, previously, wollastonitecalcite ore was subjected to flotation experiments after comminution, and a calcite concentrate with low iron content was obtained in the first two flotation stages. In the following steps, the augite mineral was activated and started to float. On the other hand, wollastonite was not activated and remained in the cell despite the increasing potassium oleate concentration. As seen in Table 3, Float-1 and Float-2 products contained high grades of CaCO₃, 91.55% and 92.77%, respectively. Float-3, Float-4, and Float-5 were characterized as tailings, since their Fe₂O₃ contents were high. A marketable wollastonite concentrate having a 99.4% purity was produced with 0.44% Fe₂O₃, 0.95% CaCO₃, and 49.70% SiO₂ contents using a 1500 g/t potassium oleate. Calcite concentrates obtained in the first two flotation stages were combined and a final calcite concentrate (91.96% CaCO₃) with 0.11% Fe₂O₃ and 2.14% SiO₂ was produced.

Products	Weight (%) –	Fe ₂ O ₃		SiO ₂		Loss on Ignition		Calcite		Wollastonite	
		С	D	С	D	С	D	Р	D	Р	D
Float-1 *	31.1	0.09	6.2	1.55	1.8	40.28	59.3	91.55	59.3	3.10	1.8
Float-2 *	15.8	0.15	5.3	3.30	2.0	40.82	30.6	92.77	30.6	6.60	2.0
Float-3	2.9	1.31	8.5	24.20	2.7	36.47	5.1	82.89	5.1	48.40	2.7
Float-4	3.3	2.17	16.0	40.80	5.2	17.52	2.8	39.82	2.8	81.60	5.2
Float-5	3.1	3.09	21.4	45.60	5.4	8.83	1.3	20.07	1.3	91.20	5.4
Sink	43.8	0.44	42.6	49.70	82.9	0.42	0.9	0.95	0.9	99.40	82.9
Total	100.0	0.45	100.0	26.24	100.0	21.08	100.0	47.90	100.0	52.47	100.0

 Table 3. Results of flotation tests.

C: Content; D: Distribution; P: Purity. * Combined flotation products.

3.2. CaCl₂ Production by Acid Leaching

The final calcite concentrate obtained after the flotation process was treated with HCl to produce CaCl₂. The effects of the concentration, temperature, and time on acid leaching were investigated. After the determination of the most suitable condition, CaCl₂ was mixed with potassium feldspar in certain proportions, the roasting and dissolution processes were completed, and finally, potassium was successfully extracted.

3.2.1. Effect of HCl Concentration

Based on the formula in Equation (1), the acid treatment was applied to the calcite concentrate. HCl was used as a solvent to investigate the effect of various acid concentrations (0.25, 0.5, 1, 2, 3, 4, and 6 mol/L) on CaCl₂ production. The leaching temperature and time were chosen as 60 °C and 10 min, respectively. The HCl-Calcite ratio was adjusted to 2:1 by weight. Calcium dissolution efficiencies versus acid concentration are illustrated in Figure 8.



Figure 8. Calcium dissolution curve as a function of acid concentration.

Figure 8 clearly shows that 2 mol/L HCl provides an optimal calcium extraction rate of 93.7%. The 2 mol/L HCl concentration was kept constant in the following experiments, as there was no more than a 3% increase in dissolution efficiency at higher acid concentra-

tions. Similar results were obtained in the study of Zhang et al. [11], who performed the dissolution experiments to produce $CaCl_2$. The highest calcium dissolution rate (96.1%) was obtained at a 4 mol/L HCl concentration, while 1 mol/L HCl led to only 81.9% calcium dissolution. To determine the amount of HCl consumption for our case, the titration method was chosen and the acid consumption was found to be 165 kg/ton for this study.

3.2.2. Effect of Temperature

After a suitable acid concentration was obtained, the effect of temperature on calcium dissolution efficiency was investigated at different temperatures (25, 40, 60, and 80 °C). The HCl–Calcite ratio was kept constant at 2:1, and tests were performed with a 10 min leaching time, a mixing speed of 350 rpm, and 2 different HCl concentrations (1 and 2 mol/L). The calcium dissolution efficiencies are presented in Figure 9.

The results in Figure 9 indicated that the leaching temperature had a considerable effect on the dissolution of calcium. Especially, no significant change was observed after the 2 mol/L HCl concentration at 40 °C. On the other hand, calcium dissolution yields increased continuously with increasing temperature while the dissolution of calcium started to stabilized after the 1 mol/L HCl concentration at 60 °C. Considering the gelation problem caused by the use of a vacuum after the experiment using a 2 mol/L HCl concentration, a 1 mol/L HCl concentration, and a 60 °C temperature were found to be more suitable for the dissolution process. Zhang et al. [11] indicated the importance of temperature on leaching in their study. After 20 min of dissolution at a 4 mol/L HCl concentration, 91.0%, 96.9%, 97.1%, and 97.5% calcium dissolution efficiencies were achieved at 40, 60, 80, and 90 °C, respectively.



Figure 9. Calcium dissolution efficiency curve in different hydrochloric acid concentrations depending on temperature.

3.2.3. Effect of Leaching Time

The effect of leaching time on the calcium dissolution was investigated using various durations (5, 10, 15, 30, 60, 90, and 120 min). The HCl-Calcite ratio was kept constant at 2:1, and tests were performed at a 1 mol/L HCl concentration, a 60 °C temperature, and a mixing speed of 350 rpm. The results are shown in Figure 10. As seen in Figure 10, although there is not much change in recovery, the recovery decreases as the leaching time increases. A 5 min leaching time is not considered sufficient for the system to balance; therefore, a 10 min leaching time was preferred.



Figure 10. Calcium dissolution curve at different leaching times.

3.3. The Roasting Followed by Leaching

Roasting followed by water leaching was investigated by adopting Equation (2). CaCl₂ produced in the previous process was treated with K-feldspar. The aim was to replace calcium ions with potassium at temperatures above the melting point of the feldspar sample. The feldspar—CaCl₂ ratio was kept constant at 1:1.5, and the mixture was roasted at different roasting temperatures (800, 850, 900, and 950 °C) for an hour. Water was used as a solvent to dissolve the potassium from the roasted sample. Under constant conditions, a pregnant solution containing potassium ions was obtained. Table 4 shows the results of the roasting process followed by leaching.

Table 4. Potassium dissolution recoveries after roasting with calcium chloride.

Roasting Temperature , °C	Potassium Recovery, %
800	97.1
850	97.1
900	98.6
950	97.3

According to the results, the highest potassium extraction was achieved at a 900 °C roasting time. Since CaCl₂ is an expensive additive, producing CaCl₂ from local reserves can be helpful in improving both the usage of natural resources and in producing high-value-added products. In light of the data obtained in this study, it is possible to design a process that allows for the recovery of potassium from wollastonite-calcite and K-feldspar ores in the same region.

4. Conclusions

As it is known, mineral processing is very significant in terms of both technological and economic evaluation of raw materials before chemical beneficiation. In this study, using K-feldspar and CaCl₂ roasting followed by leaching tests were performed to extract potassium from the natural resources that were located in the same district. For that purpose, the comminuted wollastonite-calcite ore was subjected to flotation tests, and a marketable wollastonite concentrate and a high purity CaCO₃ were produced. Calcite concentrate from the flotation test was treated with HCl to produce CaCl₂. Under the

optimal conditions of a 1 mol/L HCl acid concentration, a 60 °C leaching temperature, and a 10 min leaching time, 92.7% of calcium was extracted. The CaCl₂ produced by the evaporation method was mixed with K-feldspar in certain proportions and then subjected to roasting experiments followed by dissolving experiments. The optimal feldspar–CaCl₂ ratio was found to be 1:1.5, and 98.6% of the potassium was successfully extracted from the potassium feldspar ore. It is inevitable for economic development to produce KCl, which is very important for the fertilizer industry, from wollastonite-calcite and K-feldspar ores in the same region. Obtaining high-value-added products from different industrial raw materials increases the added value of the raw material considerably and will enable Turkey to use its natural resources more efficiently.

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