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Extraction of rubidium from Malatya - Kuluncak area complex ore

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Research Article

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Leaching, Roasting, Rubidium.

ABSTRACT

In this study, studies were carried out to take rubidium (Rb), which is in a complex form of Na +K feldspar, pyroxene, and alumina silicate into solution in an environmentally sensitive manner with high recovery. Malatya - Kuluncak complex ore was used in this study. In the first stage of the study, the roasting method was tried to obtain rubidium chloride (RbCl) or rubidium sulphate (Rb_2SO_4) structures, which are the water-soluble forms of rubidium in the sodium-potassium feldspar minerals. Roasting was carried out using different additives and calcium chloride was identified as the most suitable additive material for this ore structure. In studies investigating the effect of roasting temperature and time, the evaporation amount of RbCl was calculated. After deciding on the additives, the additive amount, solid/liquid ratio in the leaching stage, and leach time were optimized. The optimum conditions were determined as a leaching time of 120 minutes at 1/3 solid/liquid ratio after roasting for 60 minutes at the roasting temperature of 900 °C at 70% calcium chloride ($CaCl_2$) addition. the solution efficiency of rubidium was calculated as 96%. Taking Rb into solution from primary sources was first achieved with this study in Türkiye.

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1. Introduction

Rubidium (Rb) was first discovered by German chemists Bunsen, R. W. and Kirchhoff, G. R. in 1861. As a member of the alkali metals, the element Rubidium was described as a having low fusing point (39 °C), high plasticity, soft, and silvery-white in color. (Patnaik, 2003; Zhou et al., 2015). Although it is the most commonly found 16th element in nature, no minerals of rubidium are found. Therefore, the total production of the element varies between 2 to 4 tons annually (Butterman and Reese Jr, 2003). Rb is obtained as a by-product of potassium [(K,Rb) $Li_2AlSi_4O_{10}F_2$] production from the lepidolite mineral and cesium production from ($Cs_2Al_2Si_4O_{12}$) the pollucite mineral. (Patnaik, 2003; Shan et al., 2013; Tavakoli et al., 2015; Xing et al., 2018; Zeng et al., 2019; Liu et al., 2019).

Rubidium and its compounds are popular in space, nuclear, advanced technology and energy industries. Since Rb ionizes easily, it was considered to be used in ion engines, yet cesium is known to be more efficient in this field. Rubidium-87 is used in atomic clocks due to its electron structure. It was used to locate brain tumors since it does not accumulate on normal tissue, and its radioactive properties. (Butterman and Reese Jr, 2003; Wagner, 2011; Heynes et al.,2016; Ertan and Erdoğan, 2017).

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The roasting method is described as processing elements in relatively high temperatures without excessing the melting temperature under suitable atmospheric conditions to induce chemical changes in their structures.Chlorination roasting, on the other hand, is commonly used for taking insoluble metal compounds into the solution by transforming them into soluble metal chlorides. Alkali metals sodium, potassium and Rb are highly soluble in water in their chloride salt forms. Through the process of chlorination roasting of the alkali elements, water soluble alkali metal chloride salts are obtainable. Rubidium chloride (RbCl) can have evaporation behavior above its melting temperature of 718 °C. An alternative method for this process is the sulphation roasting. To achieve the formation of Rb_2SO_4 and its dissolution, insoluble alkali compounds must be heated to the formation heat of the salt, the presence of sulphate. Upon the heating process, highly water-soluble alkali sulphate salts are obtained. Using these methods together may result in more efficient solubility (Gupta, 2003; Yan et al., 2012; Tavakoli et al., 2015; Zheng et al., 2016; Xing et al., 2018; Zeng et al., 2019; Wang et al., 2020).

Studies are carried out on the production of rubidium element from both primary and secondary sources. For the production from secondary sources, the wastes of gold, kaolin clay and boron clay were used. Direct leaching and chlorination roasting – water leaching methods were used in these studies. Obtaining Rb from kaolin clay wastes (0.21% Rb) by acid leaching and chlorination roasting – water leaching is studied by Zhou et al. (2015). In both methods, the efficiency of taking Rb into solution was evaluated. The methods were compared based on the metal recoveries. Zhou et al. (2015) determined the chlorination roasting – water leaching method to be more suitable for kaolin clay wastes when compared to acid leaching method. Rb that is contained in the ore was taken into solution with 90.6% recovery. Another study about waste was carried out by Ertan and Erdoğan (2017) for Rb recovery from boron wastes. They used HF in their chlorination roasting and acid extraction process. Although HF was more efficient in the destruction of silicate structures, they decide that this is not the preferred method due to its hazardous nature to humans and the environment. Another study was carried out with waste of Mouteh which is one of the largest gold processing plant. These wastes contain 120 ppm Rb. The mentioned study consists of three main stages: In the first stage, nitric acid leaching is used to remove unwanted elements from the wastes. In the second stage, salt mixtures are used for roasting. In the third and the final stage, Rb is taken into the solution using with water leaching. For the process of roasting, a mixture of sodium sulphate and calcium chloride was used. The Rb conversion rate was calculated as 90.95%. In the water leaching stage 90.95% percent of the Rubidium sulphate and 97.14% of rubidium chloride was taken into solution (Tavakoli et al., 2015; Zhou et al., 2015; Ertan and Erdoğan, 2017).

For the production of Rb from primary sources, rocks and minerals which contain Rb such as granite, biotite and zinnwaldite etc. are generally used in literature. In recent studies, roasting – water leaching, acid leaching and reaction with sulphuric acid methods were used. For production of Rb from biotite, Zeng et al. (2019) used chlorination roasting – water leaching and direct acid leaching as two distinct methods. The recovery of metal from source was calculated as 96.25% with direct acid leaching while it was improved to 97.15% with roasting-water leaching. Ultimately, chlorination roasting – water leaching method provides a higher efficiency in taking Rb into solutions, as compared to sulphuric acid leaching.

Possible reactions of ores containing rubidium in chlorination roasting are presented in Equations 1, 2 and 3 (Zeng et al., 2016, 2019)

$$CaCl_2+2SiO_2+Al_2O_3+Rb_2O = CaAl_2Si_2O_8+2RbCl \quad (1)$$

$$CaCl_2+2SiO_2+Rb_2O = CaSiO_3+2RbCl \quad (2)$$

$$CaCl_2+Al_2O_3+3Rb_2O = Ca_3Al_2O_6+6RbCl \quad (3)$$

However, in chlorination roasting – water leaching method, more K is taken into solution with a higher amount of Rb, as compared to sulphuric acid leaching method. More K in solution together with Rb complicates downstream process to extract Rb. Taking into solution process of Rb-containing granitic rocks with alkaline leaching is studied by Xing et al. (2018). In this study, a sodium hydroxide solution was used at 230 °C, to break the structure that Rb is attached, which allows for the uptake of Rb into the solution with a very high level of efficiency (95%). To remove the silica from the solution, precipitation with calcium oxide technique is utilized. Subsequently, tert-Butyl-2-(α-Methylbenzyl) Pheno (t-BAMBP) is used to separate K and Rb from each other. Finally end-product rubidium chloride and potassium chloride (KCl) were obtained. Another study was carried out by Vu et al. (2013) to produce lithium and Rb from zinnwaldite mineral. In that study, $CaCO_3$ is used for sintering, followed by water leaching and alkaline leaching methods. The efficiency rates of elements taking into solution were determined to be 84% for lithium and 91% for rubidium. Studies on Rb production based on the ore and wastes vary depending on the structure of the ore and the type of waste. (Zeng et al., 2019).

In this study, experiments were conducted to take Rb element that is contained in complex ore structures into solution with a high rate of efficiency in an eco-sensitive motivation. The effects of using different substances in roasting on the Rb conversion and passing into solution rates are examined and the process is optimized. This study is the first of its kind in Türkiye, in taking Rb into solution with a high degree of efficiency from primary sources. Additionally, analysis methods such as SEM, XRF and XRD were used to examine the extraction mechanisms in detail. The ore used within the framework of the study being complex; consists of ~45% Na + K feldspar (Na - K - $AlSi_3O_8$) as main mineral, ~%20 pyroxene (Ca, Na, Fe, Mg, Mn – Si_2O_6), ~11-12% alumina silicate (clay, mica) and 6% calcite minerals. The RbO_2 content of the ore was calculated to be 0.05% by weight.

2. Experimental Studies

2.1. Raw Material and Experiment Equipment

The ore used in the study was provided by Malatya-Kuluncak prospect area belonging to the General Directorate of Mineral Research and Exploration (Öztürk et al., 2019; Pulat et al., 2022). It contains 0.05% RbO_2. Calcium chloride ($CaCl_2.2H_2O$ 99% pure), sodium chloride (NaCl 99.5% pure) and sodium sulphate ($Na_2SO_4.10H_2O$ 97% pure) were provided by ISOLAB.

For the process of chlorination roasting, box furnace (Nevola Reis 120/18) was used. The concentrations of elements in the solution were measured with Agilent 725 Series Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-OES) device. Metal compositions in the input and output samples were determined with Thermo Scientific ARL Perform's X-Ray Fluorescence Spectroscopy device. For the determination of ore structure in the input sample, Panalytical X'pert Pro was used. Electron microscope imaging and EDS analyses were performed with the FEI inspect F50 Scanning Electron Microscope (SEM-EDS) device.

2.2. Method

The entire ore was firstly ground to 53 microns and homogeneously mixed with calcium chloride solid at predetermined proportions. This homogeneous mixture was then placed into alumina-silica crucibles. Structural water was taken into consideration while calculating additives. Crucibles were put into the box furnace at room temperature and were roasted at desired temperature and for certain duration. Upon roasting, the crucibles were taken out of the box furnace and left out to cool down in desiccator for 24 hours at room temperature. In the preliminary roasting experiments, the effects of additives ($CaCl_2$, NaCl and Na_2SO_4) were examined. For the experiments to determine the roasting temperature, the effects of different temperatures (800, 850 and 900 °C) and different additive proportions (10%, 30%, 50%, 70% and 90%) were evaluated.

After the roasting process, samples were ground homogeneously. The ground samples were weighed on Precisa XB 320M. Then, water leaching experiments were performed with 250 mL beakers using magnetic stirrers (Heidolph, MR Hei-Standard). In the water leaching process, the solid sample on the beaker was mixed with pure water at predetermined proportions. Subsequently, as the water leaching process was finalized, solid/liquid separation was performed using blue ribbon filter papers. During the optimization of leaching process, different solid/liquid ratios (1/2, 1/3, 1/4) and leaching durations (60, 120, 180 minutes) were observed. All experiments were conducted with 100 grams of ore. Solids were analyzed with XRF and XRD spectrometers while for the liquids ICP-OES analysis was performed. Flow chart of the method is given in Figure 1.

2.3. Data Processing

In processing of the experimental data, Equations 4-6 were used.

$$\tau_1 = \frac{c_1 \times V_1}{m_0 \times c_0} \tag{4}$$

$$\tau_2 = \frac{m_r \times c_r}{m_0 \times c} \tag{5}$$

$$\tau_3 = 1 - \tau_2 - \tau_1 \tag{6}$$

τ_1, τ_2 and τ_3 represent Rb leaching efficiency, the amount of Rb left in solid after the leaching process, and the ratio of evaporated Rb, respectively. The c_1 in Equation 4 represents the amount of Rb that is inside the solution while the c_0 is the amount of Rb in the solid part. The c_r in Equation 5 is the amount of Rb in solid after the leaching process. The V_1 in Equation 4 represents the volume after liquid leaching.

3. Findings

3.1. Preliminary Roasting Experiments

In the preliminary roasting experiments, the effects of the addition of calcium chloride and sodium sulphate-sodium chloride salts on the efficiency of taking Rb into the solution were examined.

Within the context of preliminary roasting experiments, parameters such as roasting temperature (900 °C), roasting duration (60 min), water leaching (60 min), and the solid/liquid ratio (1/3) were held fixed in order to evaluate only the effect of additives. Reactions were observed when chlorination and sulphatization roasting were done at 800-900 °C and 700 – 900 °C, respectively. (Alonso et al., 2012; Zheng et al., 2016). Therefore, the temperature was selected as 900°C for the preliminary experiments. The effects of the additives on the efficiency of taking Rb into the solution in this study are presented in Table 1.

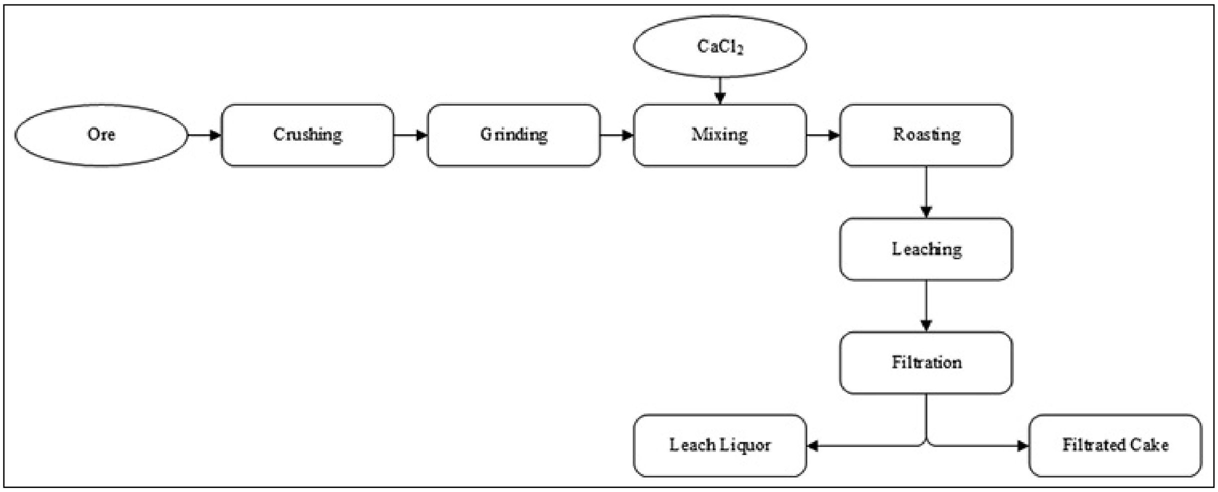

Figure 1- Flow chart of the method.

Table 1- The effects of the ratio of roasting additives on the efficiency of taking Rb into the solution.

Roasting Conditions				Efficiency of Taking Rb into Solution (%)
Unit (Ore)	Unit ($CaCl_2$)	Unit (NaCl)	Unit (Na_2SO_4)	
100	50	-	-	78.3
100	50	20	-	38.5
100	40	30	-	36.6
100	30	40	-	35.2
100	50	-	20	33.4
100	40	-	30	24.5
100	30	-	40	18.2

The experimental results at roasting conditions show that the highest rate of efficiency is obtained when only calcium chloride is used for taking Rb into the solution. In this ore structure, especially the sodium is observed to have a negative effect in taking Rb into the solution. In an instance where sodium chloride is accompanied with calcium chloride, the ratio of the negative effects of sodium chloride on the efficiency seems to be almost the same even if its amount is increased. Calcium chloride seems to be the most effective roasting additive for this ore structure. Thus, only calcium chloride was used for the optimization processes.

3.1.1. Effect of Roasting Time and the Temperature

Firstly, the entire ore size was ground to 53 µm and mixed with a predetermined amount of calcium chloride. Then, roasting was carried out at temperatures of 800, 850, 900 °C. While examining the effects of duration and temperature of roasting; parameters such as leaching duration (60 min), $CaCl_2$ additive amount (50%) and solid/liquid ratio (1/3) were held fixed. The effects of duration for each roasting temperature were also examined. The schematic display of the analyses' results are given in Figure 2. For the 60-minute roasting process, it is seen that changing the roasting temperature from 800°C to 850°C increases the efficiency of taking Rb into the solution by 37% and 5% when the temperature is increased to 900 °C. This increase in the roasting temperature correlates with the experimental result of Zeng et al. (2019) on the roasting temperature. When the temperature approaches 900 °C, solution efficiency decreases.

As the effect of 120-minute roasting time, 1% increase in efficiency is observed when the temperature is increased from 800 °C to 850 °C and 5% increase in efficiency is observed when the temperature is increased from 850 °C to 900°C. While the roasting duration experiments conducted at 800 °C result in a positive effect for the conversion of Rb into RbCl, for the temperatures of 850 °C and 900 °C an increase in roasting duration affected negatively. This is caused by the RbCl vaporization at high temperatures. The effects of roasting duration and temperature on the efficiency of taking Rb into solution and RbCl vaporization is given in Table 2. When the data in Table 2 is examined, the optimum roasting temperature is determined to be 900 °C with 60 minutes of roasting duration.

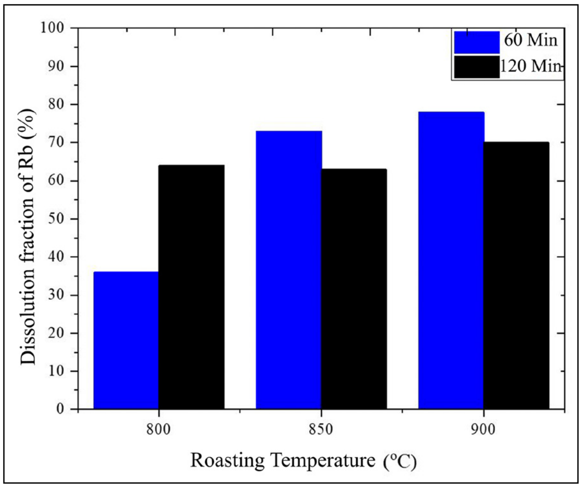

Figure 2- The effects of roasting temperature and duration on Rb dissolution.

3.2. The Effect of Calcium Chloride Amount

After the optimization of roasting temperature and duration, the effects of $CaCl_2$ additive amount on the recovery of taking Rb into the solution were examined. Parameters of the leaching duration (60 minutes) and the leaching solid/liquid ratio as (1/3) were kept fixed. Experiments were conducted by using the additives of $CaCl_2$ at 10 - 30 - 50 - 70 and 90% ratios.

In Figure 3, the efficiency of $CaCl_2$ on taking Rb into solution is given. In this study, a change in the

Table 2- Effects of roasting temperature and duration on efficiency.

Roasting Temperature (°C)	Roasting Duration (min.)	τ_1(%)	τ_2 (%)	τ_3(%)
800	60	35.8	63.2	0.0
800	120	63.8	36.0	0.0
850	60	73.0	24.2	2.8
850	120	64.2	21.4	14.4
900	60	82.5	13.3	4.2
900	120	69.7	12.2	18.1

amount of $CaCl_2$ from 10% to 30% results in a 39% increase in efficiency of taking Rb into solution. A change in $CaCl_2$ amount from 30% to 50% yields 29% increase in efficiency whereas a respective change in $CaCl_2$ amount from 50% to 70% results in a 5% increase in efficiency. A change in the calcium chloride by more than 70% does not affect the conversion rate. This phenomenon is a result of the reaction ending as all Rb atoms found in feldspar convert into RbCl. The lack of increase in the efficiency of taking Rb into the solution despite the end of reaction is related with the solid/liquid and duration parameters during the leaching process. When the effects of $CaCl_2$ additive on the efficiency of taking Rb into the solution are examined, similar results are obtained as compared to the results of the studies of Zheng et al. (2016) carried out with kaolin. Their studies, as similar to our study, show an increase in efficiency as the calcium chloride amount is increased until a point is reached where the efficiency remains constant.

3.3. The Effect of Solid/Liquid Ratio

Leaching duration (60 min.) in the solid/liquid ratio optimization stage, and the amount of $CaCl_2$ additive (70%) were kept fixed. For the examination of the effects of solid/liquid, ratios of 1/2 – 1/3 and 1/4 were used. The effects of parameters on the efficiency of taking Rb into solution is given in Figure 4.

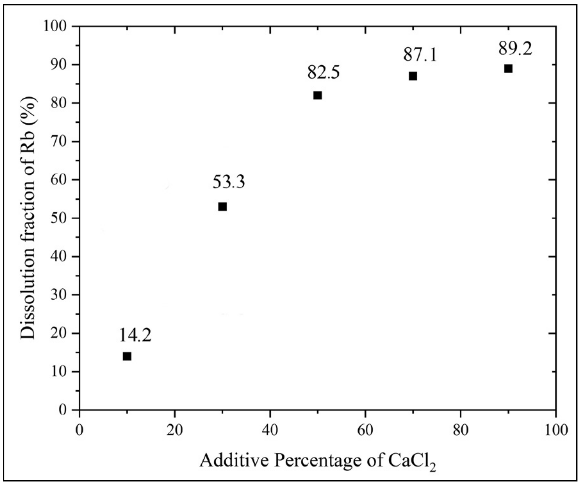

Figure 3- The effects of the amount of $CaCl_2$ Additive on the efficiency of taking Rb into solution.

In this study, a change in solid/liquid ratio from 1/2 to 1/3 results in 9% increase in efficiency of taking Rb into solution while the respective efficiency remains constant at a solid/liquid ratio change from 1/3 to 1/4. This is attributed to taking the entire Rb that is converted into solution. Therefore, 1/3 solid/liquid ratio is specified as the optimum parameter.

3.4. The Effect of Leaching Duration

In this study, the durations of 60, 120 and 180 minutes were tested while the parameters of solid/liquid (1/3) and $CaCl_2$ (70%) were kept fixed. The respective results are given in Figure 5. The results show that a change in the duration of leaching from 60 to 120 minutes results in a 9% increase in efficiency of taking Rb into solution. On the other hand, a change in the duration of leaching to 180 minutes lowers the efficiency by 1%. Hence, a duration longer than 120 minutes does not affect the efficiency of taking Rb into solution.

Figure 6a shows the SEM image of the input sample whereas Figure 6b shows the SEM image of the sample after roasting with 70% $CaCl_2$, a duration of 60 minutes at 900°C. As observed in Figure 6a, feldspar crystal is clearly visible. Figure 6b shows that the crystal structure of feldspar is alterated and new structures are formed. These new structures

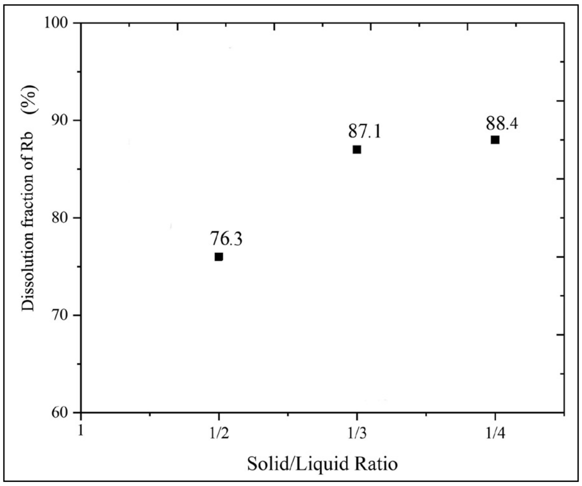

Figure 4- The effects of solid/liquid ratio on the efficiency of taking Rb into solution.

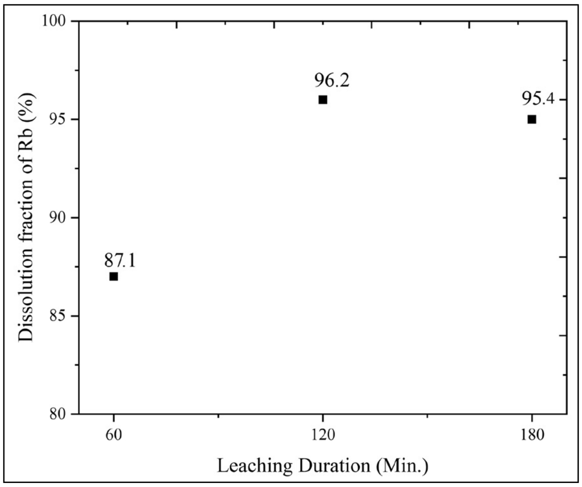

Figure 5- The effects of leaching duration on the efficiency of taking Rb into solution.

were analyzed with XRD analyses. Pre-roasting chlorination, post-roasting and post-extraction structures are given in Figure 7.

XRF analyses' results of the output solid from the roasting – water leaching on the input ore are given in Table 3. When the analyses' results are examined, it is seen that 96% of Rb is taken into solution with 4% evaporation. Additionally, the efficiency of potassium and sodium elements in taking into solution are determined as 92.6% and 59.0% respectively.

According to the Table 3, calcium oxide amount in the input ore increases from 1.41% to 13.13% after roasting – leaching process. The increase in the calcium oxide is the result of reactions given in Equation 1 – 3. In the reaction between calcium fluoride and feldspar, chloride ion bonds with rubidium, sodium and potassium, which creates rubidium chloride. Meanwhile, calcium, aluminum and silicon create a triple bond, resulting in a calcium alumina silicate structure. Therefore, in the XRF analysis, an increase in calcium oxide at the solid is observed after the leaching.

XRD analyses of the input sample (Figure 7a), chlorination roasting (Figure 7b) and leaching (Figure 7c) were performed for this study. When Figure 7a is examined, feldspar and albite are determined in the input sample. After roasting with calcium chloride (Figure 7b), an alterated of K-feldspar to rubidium chloride, sodium chloride, potassium chloride, silica (SiO_2) and andesine $[(Ca,Na)(Al,Si)_4O_8]$ mineral is observed. Also, the salts from alkali K, Na and Rb metals contained in K-feldspar are obtained. When the next step, the extraction process (Figure 7c), is examined, alkali metal salts are dissolved and the minerals such as albite [(Na,Ca)AlSi3O8] and hibschite [Ca3Al2(SiO4)2(OH)4] remain undissolved. Reactions during roasting are given in Equations 1 – 3.

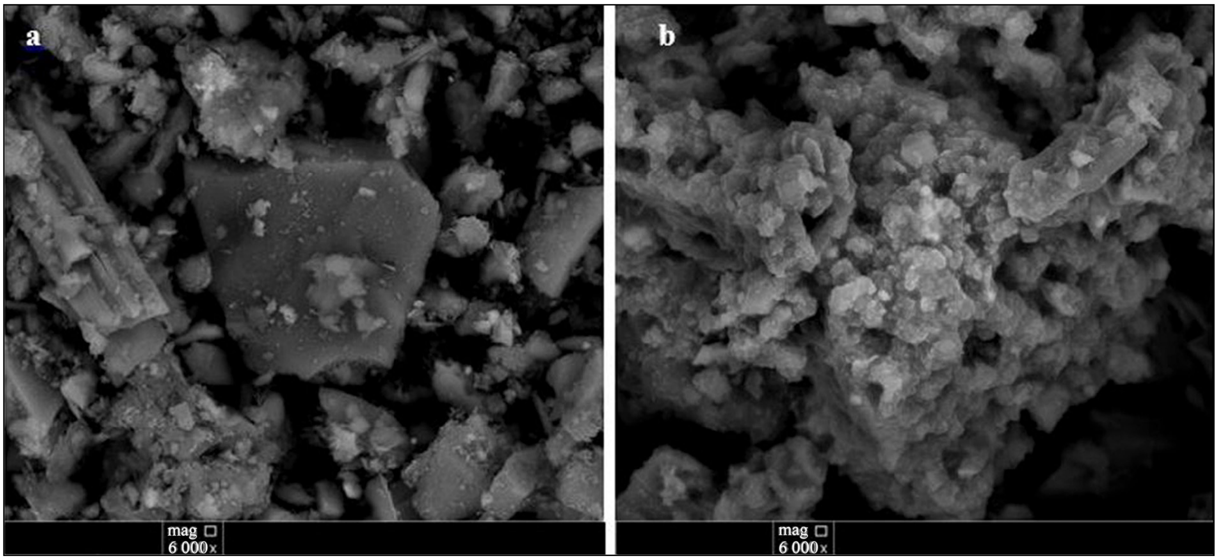

Figure 6- a) Input sample and, b) SEM imaging after roasting.

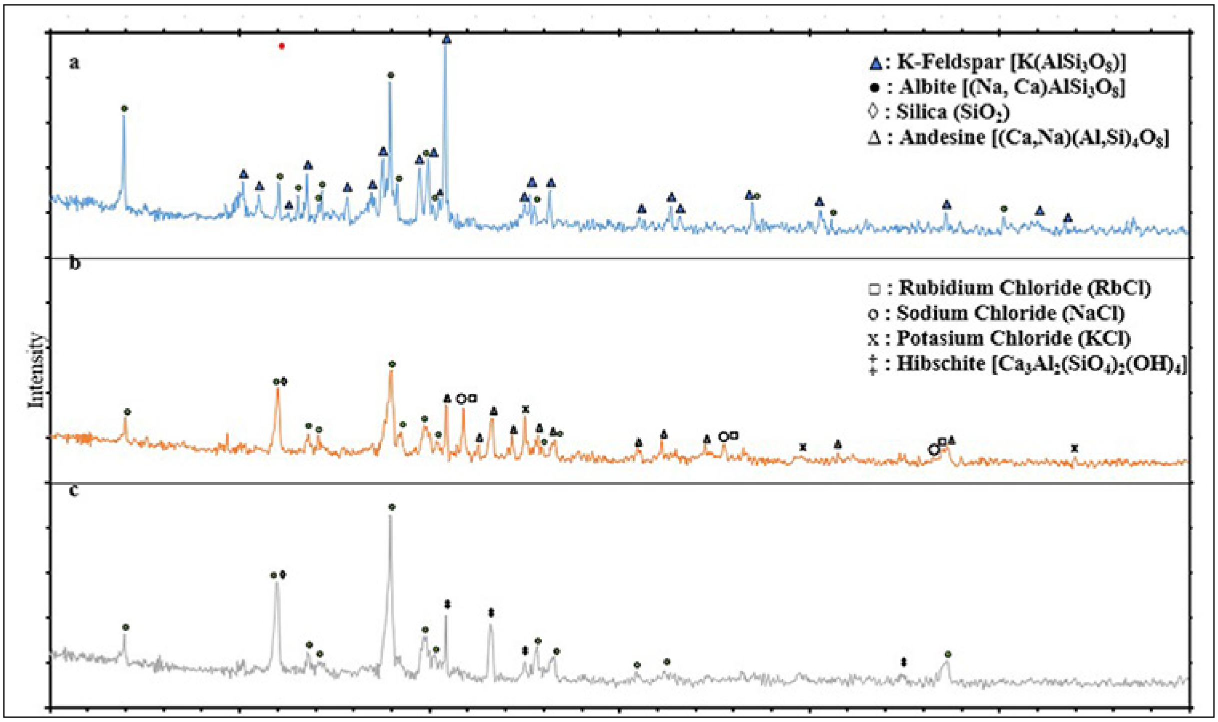

Figure 7- XRD powder sample results in order; a) before roasting, b) after chlorination roasting and c) after leaching.

Table 3- Before and after leaching XRF results of the solid.

Compound	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	Rb_2O	SiO_2	A.ZA	Other
Ore input (%)	15.8	1.4	8.4	4.7	1.8	4.1	0.05	56.7	4.1	3.7
After leaching (%)	14.4	13.2	7.8	0.6	1.2	1.7	0.00	56.4	1.4	3.3

As the amount of Rb in the sample after roasting is very low, the determination of the element is not expected at the XRD analysis. However, it is possible to determine the salts, such as NaCl and RbCl, in solid as these compounds peak at the same angles (2θ: 31.8, 45.7, 56.7).

4. Discussion

In the literature, maximum efficiency was achieved by taking Rb into solution when calcium chloride is added only. Sodium sulphate affects negatively whereas sodium chloride has no negative or positive effect. Therefore, calcium chloride was selected as additive material for the experiments.

After the decision of using calcium chloride as a roasting additive, experiments were conducted at different roasting temperatures. In this context, RbCl vaporization was also taken into account for the calculations. The effects of vaporization were presented with analyses and calculations. Especially, 120-minute experiments were conducting at increasing roasting temperatures and durations. Within this experimental framework, optimum parameters were determined.

Subsequently, the effects of additives were examined after the effects of roasting temperatures. During this examination, the effects of additive increase at taking Rb into solution were calculated and it is given in the Table 1. Certain amount of chloride ions are necessary in the environment to take the entire Rb in feldspar to solution. Therefore, an increase in the efficiency is observed when the amount of calcium chloride increases. In contrast, no increase in efficiency is observed for $CaCl_2$ level above 70% due to the fact that conversion is finalized. Lack of higher increase in efficiency after the end of reaction is related to solid/liquid ratio and duration parameters during the leaching process.

An overlook of the solid/liquid ratio parameter points out the requirement of sufficient amount of water present in the environment to take Rb fully into solution. An increase in the solid/liquid ratio to 1/3 ratio results in a positive effect while the efficiency remains constant at higher ratios. This is due to $CaCl_2$, NaCl and KCl salts accompanying RbCl in solid. As these salts have a high solubility rate, an increase in efficiency is observed until the environment reaches a sufficient level of water.

Another important parameter during the leaching process is the duration. An increase in the duration of leaching process causes an increase in the efficiency of taking Rb into solution. However, the efficiency remains stable even if the duration increases. This is speculated to be caused by the entire structure of RbCl being taken into solution with water. It is not possible to achieve higher levels of efficiency due to RbCl vaporization, as previously mentioned.

5. Results

In this study, roasting process is used as the beginning stage of taking Rb element into the solution containing feldspar mineral within the complex ore structure. RbCl is obtained in the roasting stage, and taken into solution with water leaching.

Calcium chloride was observed to be the most effective additive compound. Subsequently, the effects of roasting temperature and duration were examined.

In the stages of examining the effects of different roasting temperatures and durations, the evaporation amount of RbCl was determined. It is observed that an increase in roasting temperature and duration results in more evaporation. Evaporation factor was taken into account in determining temperature and duration of roasting. In this study, the optimum roasting temperature and duration were determined as 900 °C and 60 minutes, respectively.

Upon determining the roasting duration, the temperature and the additive, experiments were conducted on the additive amount. The optimum additive amount needed to achieve the highest efficiency was determined as 70% in experiments using $CaCl_2$ at different proportions ranging from 10% to 90%.

After the optimum roasting parameters were determined, solid/liquid ratio and leaching durations were examined. The highest efficiency was achieved at 1/3 solid/liquid ratio.

Following that, the effects of leaching duration on the efficiency of taking Rb into solution were examined. In these examinations, the optimum duration was determined as 120 minutes. However, longer durations do not increase the efficiency.

In the studies carried out, optimum parameters were determined as 70% $CaCl_2$ additive amount, 900 °C roasting temperature, 60 minutes of roasting duration, 1/3 solid/liquid ratio, 120 minutes of water leaching. The efficiency of taking Rb into solution was determined to be 96.2% in these conditions.

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