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EXTRACTION OF POTASSIUM FROM FELDSPAR AND LEUCITE BY TWO DIFFERENT ACTIVATION METHODS: MECHANICAL ACTIVATION (MILLING) AND HIGH TEMPERATURE ACTIVATION (ROASTING)

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ABSTRACT

One of the most important elements in fertilizers is potassium that can be devided from felsdpar and leucite minerals. Both are composed of various minerals and need to be separated from its impurities to get the desired minerals. In this research, test of mineral activation was performed using two different methods, namely mechanical activation (milling) and activation that used high temperature (roasting). The results were followed by potassium extraction process through leaching using sulfuric acid 6 N, 20% solids for 2 hours without heating. The best result was obtained from a 60-hour mechanically, activated leucite in leaching condition and conducted without heating. To evaluate potassium dissolution with soil when applied as fertilizer, the test of solubility in citric acid-the analogy of acid humus within soil-was conducted. The product with the best solubility was obtained from a 60-hour milling process. The results show that leucite, activated by milling process, was more easily extracted its potassium. However, further research is still needed to optimize the leachability of potassium extraction.

Keywords: potassium, feldspar, leucite, extraction, activation method, milling, roasting

INTRODUCTION

Fertilizer is an important commodity in supporting national food security. One of the main elements of fertilizer needed in large quantities by plants as a macro element is potassium (K) but it still relies on imports. Data regarding potassium fertilizer import during 1996-1998 stated that Indonesian averagely 435,000 tons potassium fertilizer a year. It is amounted to USD 61.6 million (Aziz et al, 2001).

Minerals in nature that are potential to be used as potassium source are feldspar and leucite (Kusdarto et al, 2008). The reserves of feldspar are widespread in Indonesia, from Aceh to Flores, West Kalimantan and Central Sulawesi, estimated at 271,693 thousand tons (Mandalawanto, 1997). While leucite reserves occurred only at several places in Indonesia, one of them is Situbondo, East Java, with reserves about 57,060 thousand tons (Kusdarto et al, 2008).

Feldspar is widely use in ceramic and glass industries. As a group of aluminosilicate, it is divided to potassium and plagioclase imported feldspars. Potassium feldspar consists of sanidine, orthoclase and microline. Sanidine has glassy appearance and colorless, while orthoclase and mircroline are usually white or light-gray color (Brittanica Encyclopedia, 2011). The potassium content in feldspar is quite variable, ranging from 4-6%. Therefore, it needs to be upgraded. Flotation by Ardha (1994, 1997) improved the feldspar content as well as increase the K level up to 12%.

Leucite is also a source of potassium mineral. It includes in feldspathoid group that is resemble to feldspar but different in crystal structures and lower

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silica content (Wikipedia, 2011). Leucite has a dominant weathering phase called analcite and it may subordinate alter to halloysite (Giannetti and Masi, 2003). The potassium content in leucite is relatively higher around 6-12% (Kusdarto et al., 2008). Flotation process could be used to obtain a better release of potassium. However, the process requires expensive chemical reagents, therefore it is necessary to find an alternative process that suitable for both minerals.

Some researchers abroad have tried to extract potassium from natural minerals by mechanical activation method (Kleiv, 2007; Alacova et al., 2004). The method aims to destroy the material into very fine size (nanometer scale). The process will damage the structure and results in gaining the desired elements.

METHODOLOGY

Materials

Feldspar rocks were derived from Keling district, Jepara, Central Java. While leucite was obtained from Bungatan district, Situbondo, East Java. The samples were then dried at 105 ^{0}C and crushed into -200 mesh using a jaw crusher and ground by ring mill.

The samples were then divided into two different batches, namely: one for mechanical activation process (milling) and another for high temperature activation (roasting).

Experimental Procedure

(1) Milling Process
It was performed using a planetary ball mill, equipped with alumina balls and steel chamber. The mill was filled by 50 g samples in air condition. The weight ratio of the sample and alumina balls was 1:8. The process was conducted in wet condition using methanol from 0 (zero) minutes to 60 hours at 15 Hz.

(2) High temperature activation process (roasting)
A 100-g sample of -200 # size was placed in a ceramic bowl and the bowl was put in the furnace at variated temperature from 300, 500, 700, 900 and 1100 °C for 2 hours. The roasted samples were removed from the furnace, left in the air and then stored in a closed container.

The product of activation was then leached with the following procedure: 5 g samples were dissolved in 200 ml of sulfuric acid (H_2SO_4) 6 N without and after heating at 85-90 °C. The leaching time was 2 hours. After leaching, 100 ml of distilled water was added to the sample and cooled for 30 minutes. The suspension was filtered and dried at 100 °C overnightly.

In terms of evaluating potassium dissolution within soil, the solubility test was conducted in citric acid as follows: 2 g samples were dissolved in 10 ml of 2% citric acid; shaken for 5 hours and then filtered by Whatman filter paper No. 42. The obtained filtrate was analyzed by AAS. The products of those processes were chemically characterized to anticipate the potassium content. Physical characterization was also conducted by SEM and x-ray diffraction instruments.

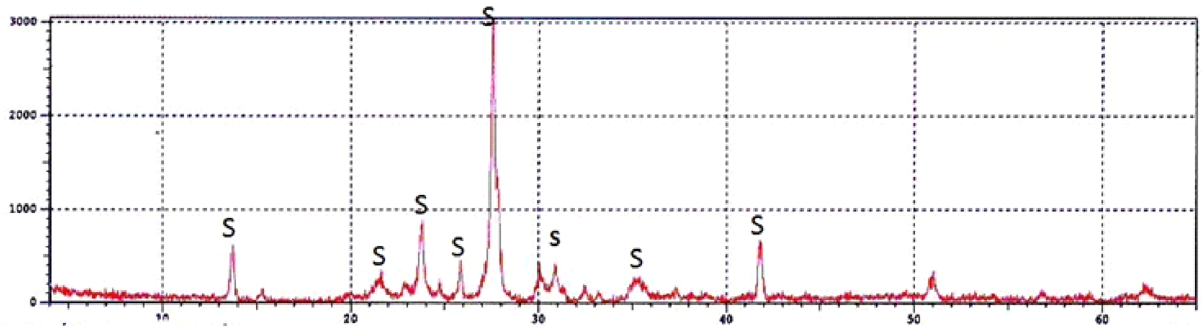
RESULTS AND DISCUSSION

The raw materials for this project were feldspar and leucite. Both were characterized by x-ray diffraction analysis (XRD), SEM and compositions as well.

Based on the XRD data, it indicated that Jepara feldspar was sanidine ($KNaAlSi_3O_8$) with the ratio of K and Na of about 7:5. Situbondo leucite contained orthoclase ($KAlSi_3O_8$) and phlogopite [$KMg_3(AlSi_3O_{10})(OH)_2$] with the ratio of K and Na was 7.86: 0.62 or about 12:1. XRD data and SEM images of Jepara feldspar and Situbondo leucite can be seen in Figure 1, while their chemical composition are shown in Table 1.

These two materials were then activated by roasting process to break up its physical bonds available in feldspar and leucite, so the extraction of the desired potassium compounds would be easier. The roasting process of feldspar and leucite is shown in Figure 3, while the products of roasting are shown in Figure 4.

Figure 4 shows that the feldspar samples were still in the form of powder at 300-900 °C but when the temperature was increased to 1100 °C, the feldspar has sintered, hardened and attached firmly to



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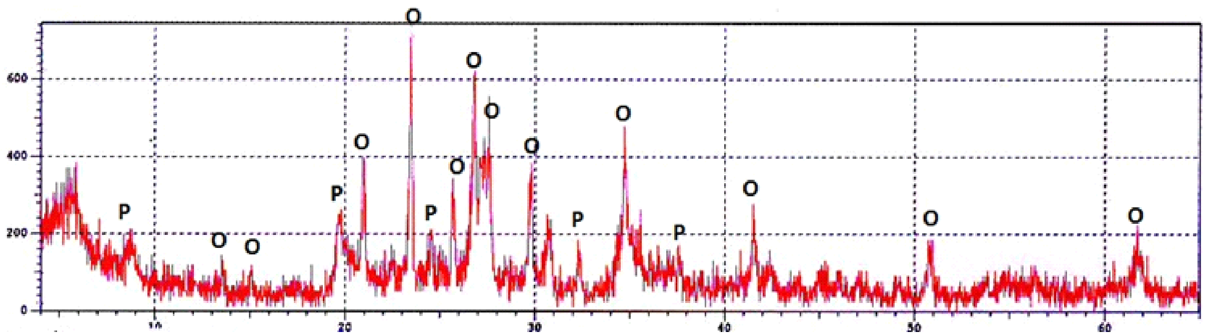

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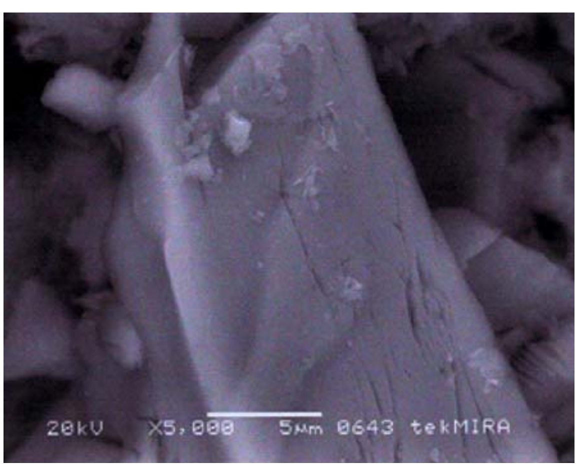
Figure 1 (a). X-ray diffraction pattern of Jepara feldspar indicated sanidin (S) mineral; (b). X-ray diffraction pattern of Situbondo leucite indicated orthoclase (O) and phlogopite (P) minerals

Table 1. Chemical composition of Jepara feldspar and Situbondo leucite analysed by XRF method

Sample	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	K_2O (%)	CaO (%)	Na_2O (%)	MgO (%)
Jepara feldspar	63.50	18.93	1.80	7.10	0.98	4.99	0.15
Situbondo leucite	49.40	22.80	5.67	7.86	1.29	0.62	0.38



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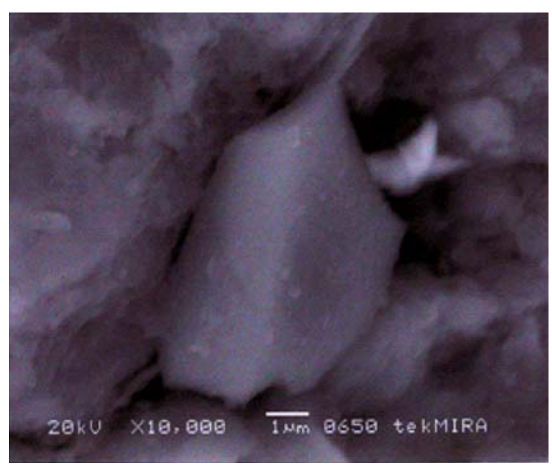

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Figure 2. (a). SEM image of sanidin within feldspar; (b). SEM image of ortholase within leucite

Figure 3. Roasting process of feldspar and leucite

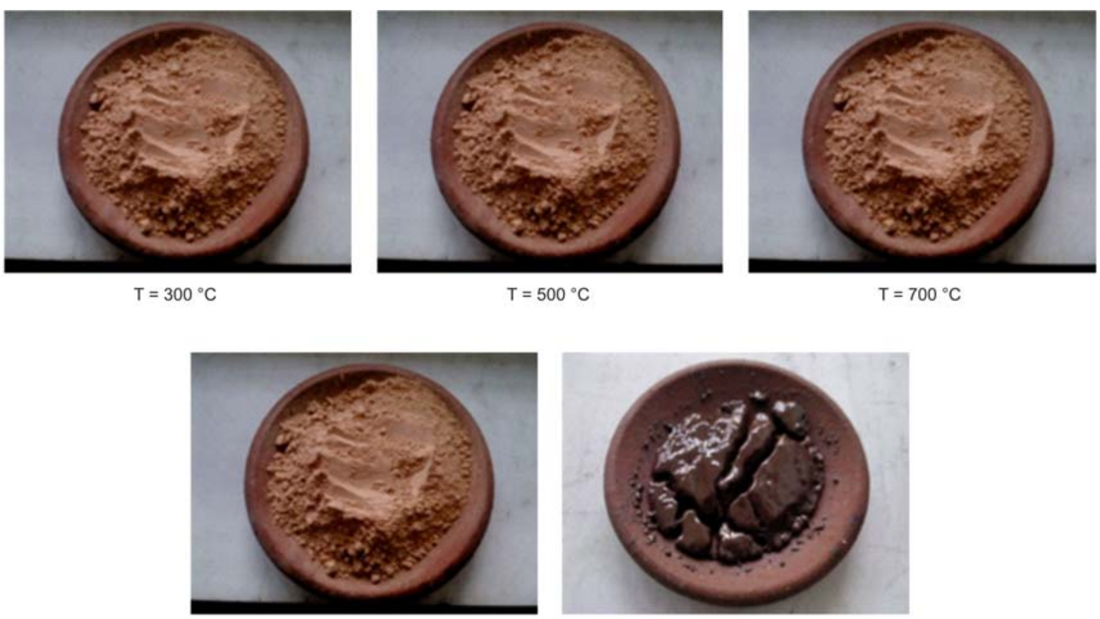
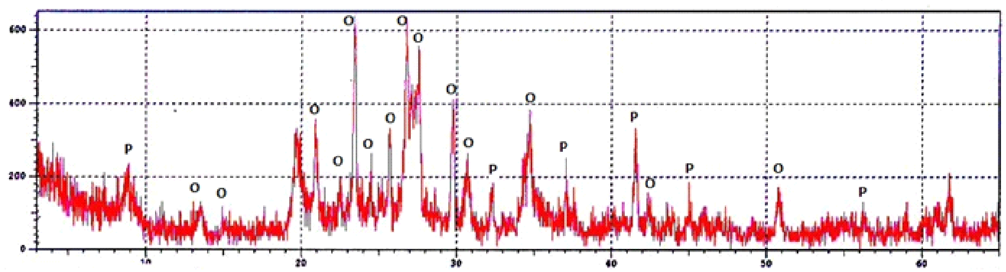


Figure 4. Roasted feldspar at 300-1100 °C

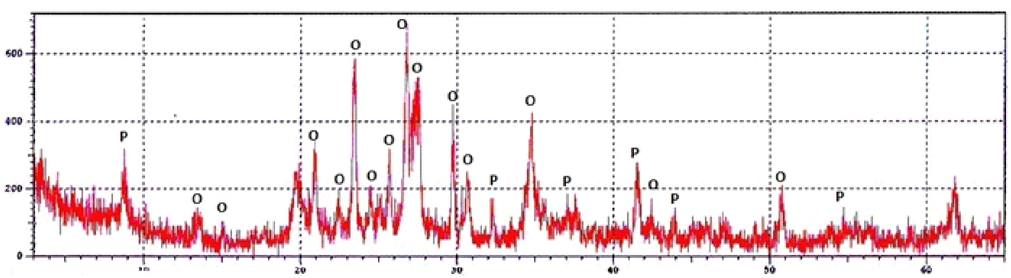
the bowls. Therefore, the leucite was roasted only at 300-900 ^{0}C to avoid sintering condition. The mineral structure of feldspar dan leucite after roasting process is shown in Table 2 and their diffractograms are presented in Figure 5 and 6.

Table 2. The minerals structure of feldspar dan leucite after roasting

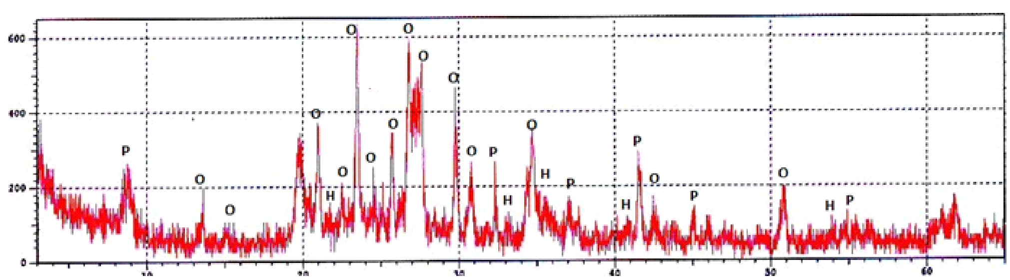
Roasting Temperature	300 °C	500 °C	700 °C	900 °C	1100 °C
Feldspar	Sanidine	Sanidine	Sanidine	Sanidine	Amorf
Leucite	Orthoclase Phlogopite	Orthoclase Phlogopite	Hematite Orthoclase Phlogopite	Orthoclase Phlogopite Hematite	-



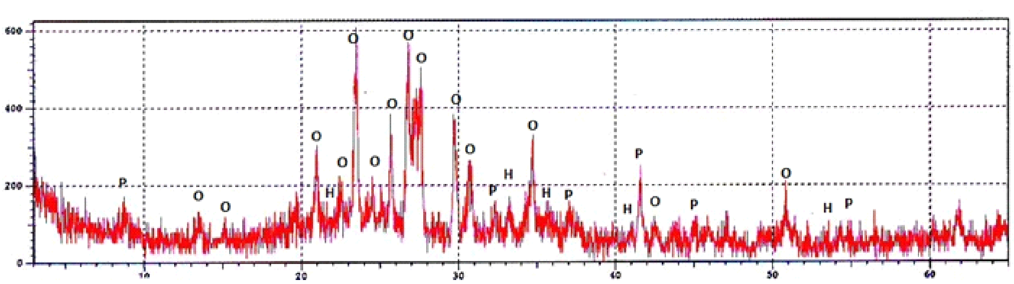
a) 300 °C: Orthoclase, Phlogopite



b) 500 °C: Orthoclase, Phlogopite



c) 700 °C: Hematite, Orthoclase, Phlogopite



d) 900 °C: Orthoclase, Phlogopite, Hematite

Figure 5. X-ray diffraction of leucite after roasting at 300, 500, 700 and 900 °Cs

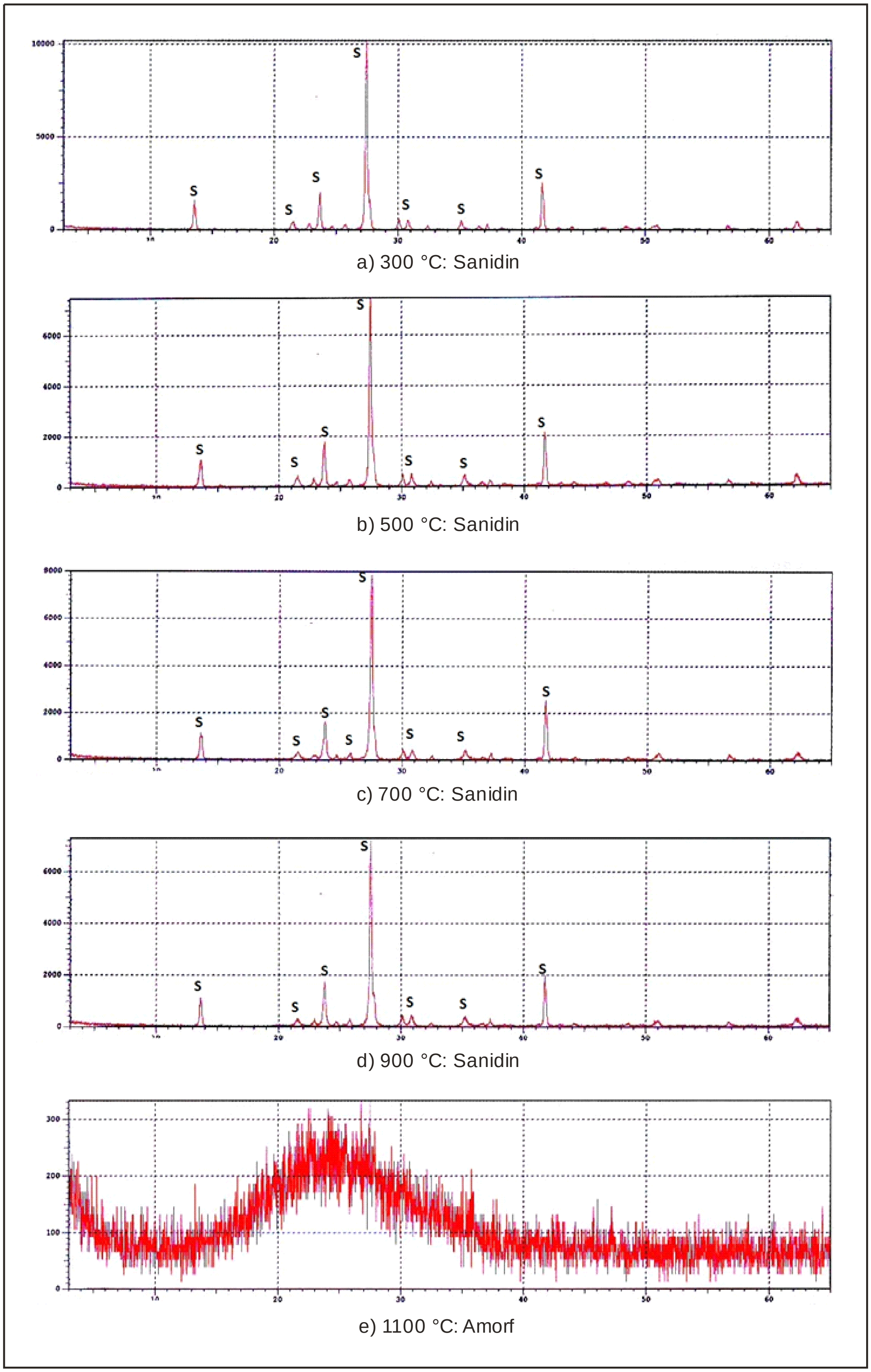


Figure 6. X-ray diffraction of feldspar after roasting at 300, 500, 700, 900 and 1100 °Cs

The mineral composition at each roasting temperature represented the changes in mineral structures. The alteration mineral at 700 and 900°C was shown by the presence of hematite because water crystal and volatile matters began to vapor at 700°C, resulted in rising the amount of hematite. Hematite was not detected in the raw material as its content may be less than 2%.

Chemical compositions of roasted feldspar and leucite are shown in Table 3. Figure 7 and 8 present the potassium content and LOI during roasting at different operating temperatures. Conclusion regarding what the best temperature is in gaining the highest K_2O content can not be determined yet as all K_2O contents for all roasting temperature are similar (Figure 8). Meanwhile for leucite, higher roasting temperature increased K_2O content. The XRD results in Table 2 show that hematite appeares at 700 °C, but was not detected in the raw material. This may be explained that the K_2O was initially bound with hematite but due to roasting process, they separated resulted in increasing the K_2O. Moreover, leucite containing phlogopite that has crystalline water began to evaporate at 700°C and increased the K_2O content.

Mineral activation process was also carried out mechanically by milling process. A-200 # sample was milled by PBM machine in varying milling time of 10, 30, and 60 hours. The particle size of feldspar and leucite after milling was measured by PSA. The result are shown in Figure 9, stated that the milling process for feldspar could reach 81 nm while for leucite, the particle size increases. Feldspar contains sanidine ($KNaAlSi_3O_8$), while leucite contains orthoclase ($KAlSi_3O_8$). Based on their chemical formulas, sanidine is more stable than orthoclase which is lack of one charge. To get its stability, the orthoclase will bind one another to form a larger particle while the Sanidin is more easily reduced in size because of its stability.

Table 3. Chemical composition of roasted feldspar and leucite

Composition	SiO_2 (%)		Al_2O_3 (%)		Fe_2O_3 (%)		K_2O (%)		CaO (%)		Na_2O (%)		LOI (%)	
Temperature (°C)	F	L	F	L	F	L	F	L	F	L	F	L	F	L
300	64.23	53.87	19.39	22.52	2.21	6.72	7.24	8.45	1.13	1.30	4.09	0.16	1.49	4.80
500	64.57	55.04	19.68	23.20	2.21	6.92	7.26	8.70	1.13	1.31	4.13	0.19	0.80	2.41
700	64.65	55.19	19.85	23.40	2.30	6.88	7.26	8.72	1.14	1.30	4.16	0.20	0.42	2.05
900	64.87	56.11	20.05	23.85	2.21	7.07	7.26	8.87	1.14	1.33	4.09	0.20	0.15	0.30
1100	64.39	-	20.06	-	2.41	-	7.20	-	1.13	-	4.05	-	0.51	-

Note : F = Feldspar; L = Leucite

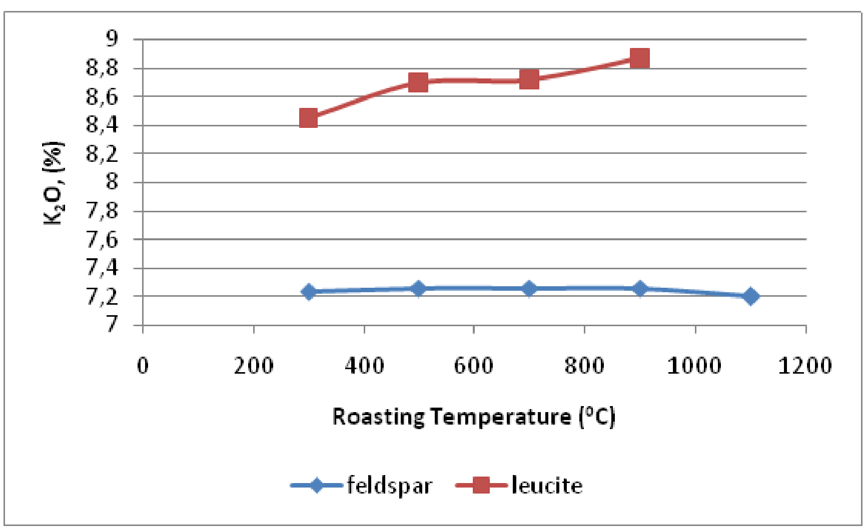


Figure 7. K_2O level in roasted feldspar and leucite

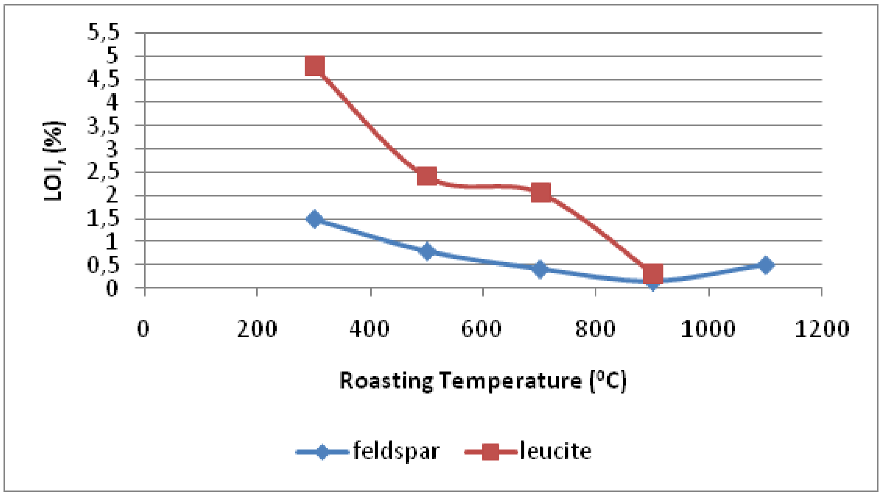


Figure 8. LOI level in roasted feldspar and leucite

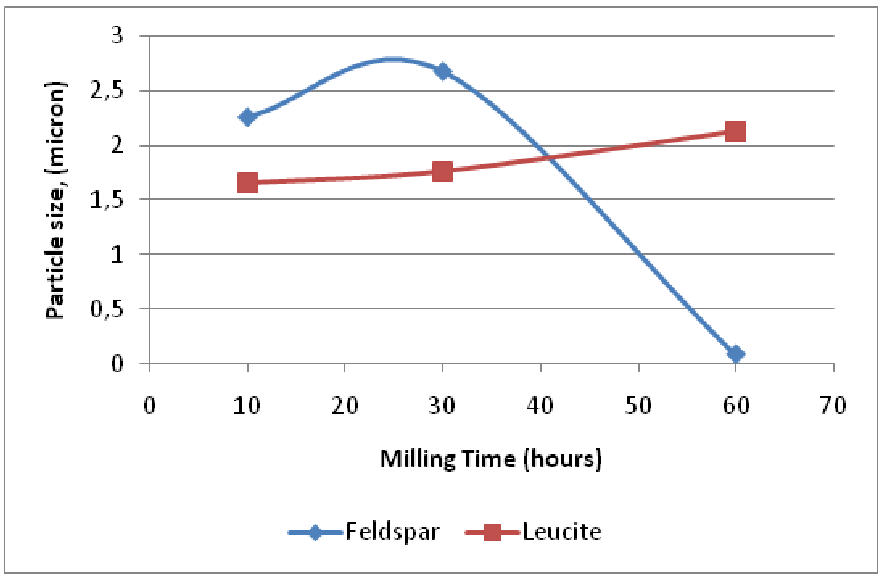


Figure 9. Curve of particle size of feldspar and leucite resulted vs milling time

Samples resulted from milling and roasting were the leached materials using 20% sulfuric acid for 2 hours with and without heating. The heating temperature was at 85-90 °C. After filtration, the filtrates were analyzed to determine the potassium content. The results are shown in Table 4 and 5.

Based on the data above, the K_2O content are presented in Figure 10 for both milled and roasted samples.

Development of extraction process with and without heating can be observed by evaluating their extraction percentages in Table 7. It can be seen that the highest extraction result was reached by 60 hours milling process and used feldspar as a raw material. The leucite and feldspar resulted from the milling and roasting process were then tested in citric acid to see how much the potassium element can be absorbed by soil (Table 8).

The results show that the potassium from leucite milling process was more easily soluble in citric acid compared to that of feldspar. In addition, the milling process provided a better result than that of roasting because the smaller size will be more

Table 4. Chemical composition of filtrate from milled sample after leaching process without heating

Sample	Filtrate				
	Fe_2O_3	Na_2O	K_2O	SiO_2	Al_2O_3
	(%)	(%)	(%)	(%)	g/L
FO 20%	0,366	0,008	0,027	0,010	0,08
LO 20%	1,523	0,004	0,282	0,064	0,33
FM 10H 20%	0,909	0,163	0,533	0,035	0,34
FM 30H 20%	2,126	0,322	1,026	0,083	0,68
FM 60H 20%	3,862	0,552	1,738	0,208	1,06
LM 10H 20%	2,526	0,016	0,891	0,042	0,61
LM 30H 20%	3,554	0,029	1,797	0,224	1,12
LM 60H 20%	4,970	0,029	2,062	0	1,23

Note: FO (original feldspar, particle size -200#);
LO (original leucite, particle size -200#);
FM (milled feldspar);
LM (milled leucite)

Table 5. Chemical composition of filtrate from roasted sample after leaching process without heating

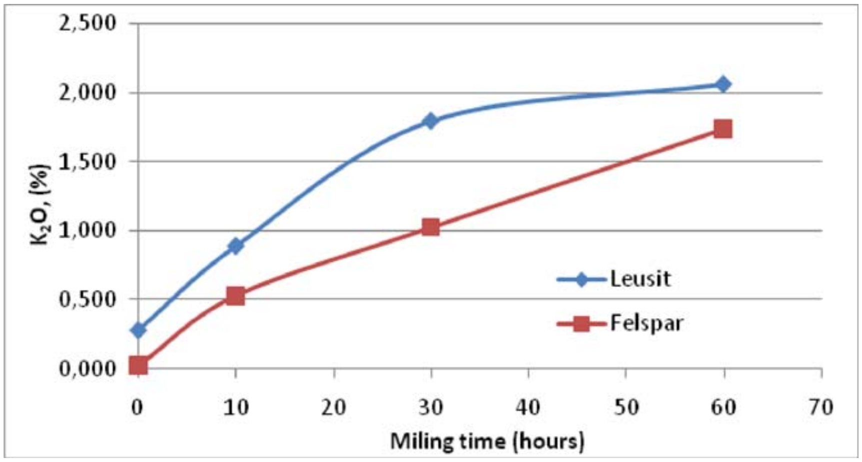
Sample	Filtrate				
	Fe_2O_3	Na_2O	K_2O	SiO_2	Al_2O_3
	(%)	(%)	(%)	(%)	g/L
FO 20%	0,200	0,010	0,021	0,010	0,080
LO 20%	0,840	0,005	0,220	0,064	0,330
FR 300C 20%	2,130	0,010	0,024	0	0,067
FR 500C 20%	0,290	0,020	0,040	0,013	0,280
FR 700C 20%	0,420	0,040	0,050	0,045	0,350
FR 900C 20%	0,018	0,011	0,012	0,003	0,015
LR 300C 20%	0,740	0,006	0,210	0,010	0,270
LR 500C 20%	0,460	0,010	0,380	0,045	0,680
LR 700C 20%	0,340	0,010	0,400	0	0,810
LR 900C 20%	0,053	0,003	0,072	0,042	0,053

Note: FO (original feldspar, without roasting);
LO (original leucite, without roasting);
FR (roasted felsdpar);
LR (roasted leucite)

Table 6. Chemical composition of filtrate leaching after heating (sample from milling process)

Sample	Filtrate				
	Fe_2O_3	Na_2O	K_2O	SiO_2	Al_2O_3
	(%)	(%)	(%)	(%)	g/L
FO 20%	0,366	0,008	0,027	0,010	0,08
LO 20%	1,523	0,004	0,282	0,064	0,33
HLM 10H 20%	4,56	0,03	1,25	0,07	0,48
HLM 30H 20%	5,28	0,04	1,88	0,05	0,71
HLM 60H 20%	4,25	0,05	1,87	0,42	0,12

Note: FO (original feldspar, particle size -200#);
LO (original leucite, particle size -200#)



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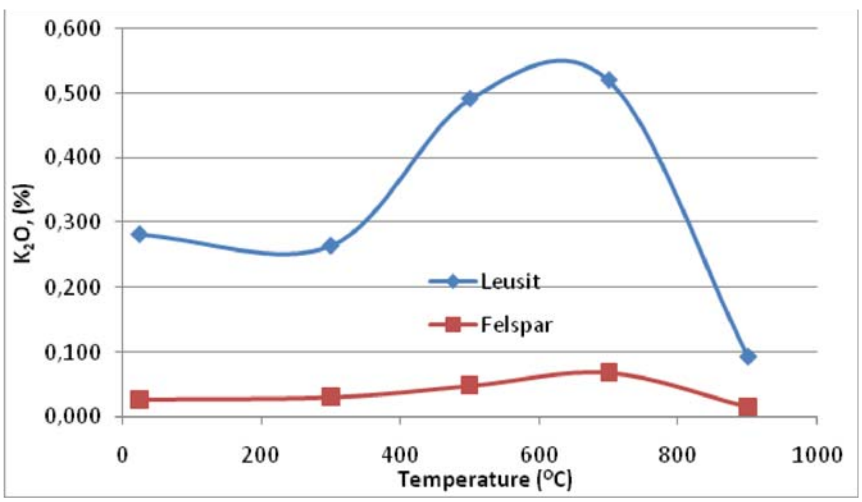

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Figure 10. (a). K_2O content of the filtrate resulted from leaching of feldspar and leucite leaching that had been milled in varying milling times;
(b) K_2O content of the filtrate resulted from leaching of feldspar and leucite leaching that had been roasted in varying temperatures

Table 7. Extraction percentages of K_2O

Code	K_2O (%)		% extraction	
	Feldspar	Leucite	Feldspar	Leucite
	Extraction without heating			
MO 20%	0,027	0,282	3,97	3,59
RO 20%	0,021	0,22	3,10	2,80
M 10H 20%	0,533	0,891	12,55	11,34
M 30H 20%	1,026	1,797	25,31	22,86
M 60H 20%	1,738	2,062	29,04	26,23
R 300C 20%	0,024	0,21	2,96	2,67
R 500C 20%	0,04	0,38	5,35	4,83
R 700C 20%	0,05	0,40	5,63	5,09
R 900C 20%	0,012	0,072	1,01	0,92
Code	Extraction with heating			
LO 20%	-	0,282	-	3,59
LM 10H 20%	-	1,25	-	15,90
LM 30H 20%	-	1,88	-	23,92
LM 60H 20%	-	1,87	-	23,79

Notes: MO = Milling original sample; RO = roasting original sample
M = milling; R = roasting; LM = Leucite milling

Table 8. Total potassium solubility in 2% citric acid

Code	K_2O (%)	
	Leucite	Feldspar
LO	0,051	-
FO	-	tt
M 10 H	0,45	0,30
M 30 H	0,87	0,51
M 60 H	1,02	0,81
R 300°C	0,084	0,003
R 500°C	0,12	tt
R 700°C	0,12	0,004
R 900°C	0,022	tt

Note: LO (original leucite);
FO (original feldspar);
M (Milling); (Feldspar)

soluble easily in a solution. The chemical bonding in leucite was easier to be destroyed than that of feldspar.

CONCLUSION

The K_2O content in leucite was higher than that of feldspar and the ratio of K and Na in leucite (12:1) was higher than that of feldspar (7:5). To obtain a good results in extracting potassium element, the mineral activation needs to be conducted through milling and roasting processes.

Roasting process produced in significant results, whereas the milling process improved the potassium extraction by leaching process. The optimum extraction result was obtained from the feldspar milling because its physical bonding was easy to break down by the acid. Smaller particle size allowed the solubility of potassium much better.

RECOMMENDATION

Quenching process can be performed after roasting feldspar and leucite. This will avoid the mineral structure from return to the initial structure. As a result, the process of extraction will be better. In addition, to increase feldspar and leucite contents can be carried out by separating the impurities.

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