



Processing of Granite Quarry Solid Waste into Industrial High Silica Materials using Leaching Process with HCl Concentration Variation

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ABSTRACT

This study was aimed to increase granite's silica content using the leaching process with HCl concentration variation. The granite used in this study came from Lematang, South Lampung. This study aims to determine the effect of variations in HCl concentration, particle size, and rotational speed on the crystalline phase and chemical elements formed in the silica product produced from granite. The HCl concentration variations were 6.0 M, 7.2 M, 8.4 M, and 9.6 M, the variation in particle size used was 270 and 400 mesh. Variations in rotational speed during leaching were 500 and 750 rpm. Granite powder was calcined at 1000 °C for 2 hours. Characterization was performed using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), and Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP- OES). The results showed that the silica content increased with increasing HCl concentration, the finer the particle size, and the higher the rotational speed. XRF analysis showed that the silica with the highest purity was leached with 9.6 HCl with a particle size of 400 mesh and a rotational speed of 750 rpm, which was 73.49%. Based on the results above, by leaching using HCl, the Si content can increase from before. The XRD diffractogram showed that the granite powder formed the Quartz phase.

1. INTRODUCTION

Lampung Province, with an area of ± 3,528,835 ha, has a very diverse natural resource potential, especially mineral resources. The diversity of mineral resources in Lampung Province includes metal minerals, industrial minerals, energy minerals, and construction minerals. Lampung Province produces industrial excavation of 117,184 m³ andesites, 234,375 m³ feldspars, and 62,232,727 m³ granite (ESDM, 2019). These data show that industrial granite excavation has the potential to be developed. One of the potential granite rocks in Lampung Province is in the Lematang Village area, Tanjung Bintang, South Lampung Regency.

Granite is a deep igneous rock (intrusive). The mineral is coarse-grained to medium light, has many colors, generally white, gray, pink, or red. This color is caused by the color variation of the mineral feldspar. Granite is formed from magma (Judson, Deffeyes & Hargraves, 1978). So far, in Lematang Village, granite is only used as a foundation stone. Silica is refined from granite to increase granite value, which can be used as an advanced material application. So far, small granite quarries measuring 50 mm and below have been discarded, so they are not used because they are used as foundation stones which are larger than 100 mm in size. Therefore mineral silica can be obtained from pumice stone

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(Mourhly, Khachani, Hamidi, Kacimi Halim & Arsalane, 2015), diatomite (Puntharo, Sankram, Chantaramee & Pokmanee, 2013), and quartz sand (Saleh, Ibrahim & Salman, 2015). Apart from quartz sand, granite is a mineral with a high silica content of up to 72.04% (Harvey & Tracy, 1997). Some researchers using several methods to synthesize silica, including combustion (Rozainee Ngo, Salema, Tan, Ariffin & Zainura, 2008), sol-gel (Le, Thuc & Thuc, 2013), leaching acid (Umeda & Kondoh, 2010), the precipitation (Yuvakkumar, Elango, Rajendran & Kannan, 2012). The method leaching and deposition have the advantage that the resulting silica is higher than the silica synthesized by other methods from some of the above methods. The leaching and deposition method is a simple and economical method for silica purification (Ha, Akhtar & Malik, 2014).

Leaching is the extraction of certain materials to remove material impurities by dissolving it (Matori, Haslinawati, Wahab, Sidek, Ban & Ghani, 2009). Inorganic impurities can be removed through a process leaching using an acid solution to obtain high purity silica before the combustion process. Researchers have carried out the process leaching

Table 1. Variations in HCl concentration, particle size and rotational speed.

Sample	HCl (M)	Size (mesh)	Rotation speed (rpm)
1	6	270	500
2	7.2	270	500
3	8.4	270	500
4	9.6	270	500
5	6	270	750
6	7.2	270	750
7	8.4	270	750
8	9.6	270	750
9	6	400	500
10	7.2	400	500
11	8.4	400	500
12	9.6	400	500
13	6	400	750
14	7.2	400	750
15	8.4	400	750
16	9.6	400	750

before conducting the thermal process using HCl, H₂SO₄, HNO₃ (Matori, Haslinawati, Wahab, Sidek, Ban & Ghani, 2009), citric acid (Umeda & Kondoh, 2010), and oxalic acid (Kalapathy, Proctor & Shultz, 2002). The use of HCl because HCl is a strong acid and is more reactive than other acids.

Several studies have been conducted to obtain large amounts of silica using the method leaching. For example, Darwis et al. (2017) purified silica from quartz sand using the method leaching with HCl. From XRF results showed that 99.90% of silica obtained from 5 hours-milled sample [14]. Abdellaoui et al. (2013), synthesizing silica with diatomite by method leaching using HNO₃ (Abdellaoui, Islam, Sakurai, Hamzaoui & Akimoto, 2018). Lahsen et al. (2016) also synthesized silica from granite using the method leaching with the solvent HCl. From this experiment, Lahsen et al. (2016) showed the maximum leaching efficiency was 92.4% and about 93.8% the leached can be separated (Lahsen, Mohamed, Cheira, Zaki & Allam, 2016). Therefore this study was aimed to purification of silica from granite quarry solid waste with different HCl concentration into industrial high silica.

Table 2. Result of Granit Stone before leaching

Compounds	Percentage (wt%)
SiO ₂	62.806
Al ₂ O ₃	18.365
P ₂ O ₅	0.764
K ₂ O	10.112
TiO ₂	0.623
Fe ₂ O ₃	6.951

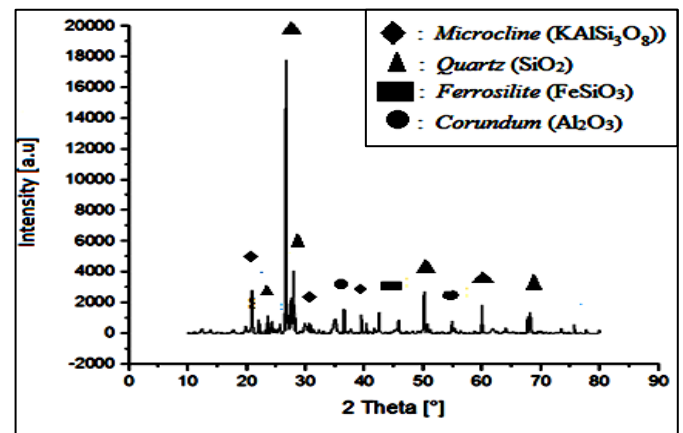


Figure 1. XRD analysis for granite

Table 3. The results of XRF Analysis *Leaching* Granite Stone with Particle Size 270 Mesh and Rotation Speed 750 rpm.

Compounds	% Leaching			
	6.0 M	7.2 M	8.4 M	9.6M
SiO ₂	69.31	71.55	72.229	72.29
Al ₂ O ₃	16.38	15.30	15.068	14.95
Fe ₂ O ₃	-	2.373	2.361	2.413
TiO ₂	0.528	0.515	0.500	0,491
K ₂ O	8.166	9.180	8.95	-
P ₂ O ₅	0.661	0.073	0.711	0.716

2. METHODS

2.1. Material

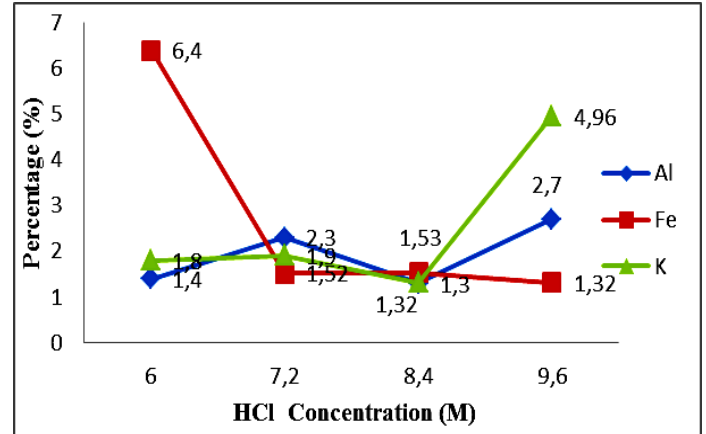
Material used in this study included granite from Lematang-Tanjung South Lampung, 12.06 M HCl, and aquades.

2.2. Method

The powder preparation process refers to the research conducted by Lahsen et al. (2016), which started with washing granite stones using aquades until they were cleaned, then drying them for 2 hours at 120°C using an oven. Furthermore, the granites was refined using ball milling for 5 hours to produce granite powder, which was still in a rough state. Then the granite powder was dried using an oven for 2 hour at temperature of calcination. After drying, the granite powder is sieved with a 270 mesh and 400 mesh sieves to obtain a fine granite powder. Furthermore, granite's fine powder is weighed as much as 5 grams as a sample to be tested using XRD and XRF.

This refining process is a process carried out to obtain pure silica from granite with various treatments, as shown in Table 1.

This granite process leaching referred to research that has been conducted by Lahsen et al. (2016). First, the preparation of 100 mL of HCl, with concentration variations of 6.0 M, 7.2 M, 8.4 M, 9, 6 M. After the solution has been prepared, 10 grams of granite powder was dissolved into each of the HCl solutions that have been prepared. Each solution was then stirred for 7 hours while stirring at a rotational speed as in Table 1 and heated to purify the silica contained in granite powder. The next stage was the result stirrer used filtered with filter paper Whatman No. 41. The filtered residue was then oven-dried for 2 hours at a temperature of calcination. Then it was calcined with furnace at for 2 hours, then characterized by XRD and XRF.

**Figure 2.** The graph of the relationship between HCl concentration and the solubility of Al, Fe, and K, the graph of a particle size of 270 mesh and a rotating speed of 750 rpm.

3. RESULT AND DISCUSSION

Granite structures were analyzed using XRF Analysis. Leaching process were conducted using HCl 6.0 M; 7.2 M; 8.4 M and 9.6 M for 7 hours at 110 °C. After that it was filtered with Whatmann filter paper no. 2, then calcined for 2 hours at a temperature of 1000 °C. Last tested with XRD and XRF. The results of the XRF analysis of granite are as in Table 2.

The results of XRD analysis for granite as shown in Figure 1. The phases formed are Quartz (SiO₂), Microcline (KAlSi₃O₈), Corundum (Al₂O₃), and Ferrosilite (FeSiO₃). The highest peak is at $2\theta = 26.6251^\circ$ Quartz (SiO₂).

The analysis results of XRF leaching granite with 270 mesh particle size and 750 rpm rotation speed are as in Table 3.

Based on Table 3, XRF analysis results showed that the percentage of SiO₂ compound after leaching was higher than before leaching. The SiO₂ highest produced was found in samples leached with a concentration of 9.6 M. In this study, the silica obtained continued to increase the concentration of HCl was used. The concentration of HCl affects the hydrolysis's speed and condensation reactions of the material, which affects the silica gain and the crystallinity of the material (Hilmy, 2007).

Leaching using HCl can dissolve other metals, including Fe, Al, and K, so that after leaching, the levels of other metals such as Fe, Al, and K decreased, and the silica content in the sample increased, as the concentration of HCl is used (Hasbi, Sigit, Indah, Septian & Efendi Bintang, 2016). Analysis the solution sample was leaching analyzed using ICP-

OES to detect chemical elements dissolved during the process leaching. Graph of analysis results from ICP-OES leaching granite with a particle size of 270 mesh, and a rotating speed of 750 rpm is presented as in Figure 2. Dissolving with HCl on elements Al, Fe, and K does not have trend the same due to HCl's ability, which is not completely dissociated so that it cannot dissolve the metal completely (Fitri, 2013).

The XRD analysis results for leaching granite stone with a particle size of 270 mesh and a rotating speed of 750 rpm are as shown in Figure 3.

Table 4. The results of XRF Analysis for Leaching Granite with Particle Size 400 Mesh and Spin Speed 750 rpm.

Compounds	% Leaching			
	6.0 M	7.2 M	8.4 M	9.6M
SiO ₂	71.25	71.49	72.81	73.49
Al ₂ O ₃	15.1	14.38	14.29	13.1
Fe ₂ O ₃	2.89	2.64	2.53	267
TiO ₂	0.50	0.44	0.42	0.48
K ₂ O	9.29	9.08	8.90	9.22
P ₂ O ₅	0.73	0.73	0.74	0.78

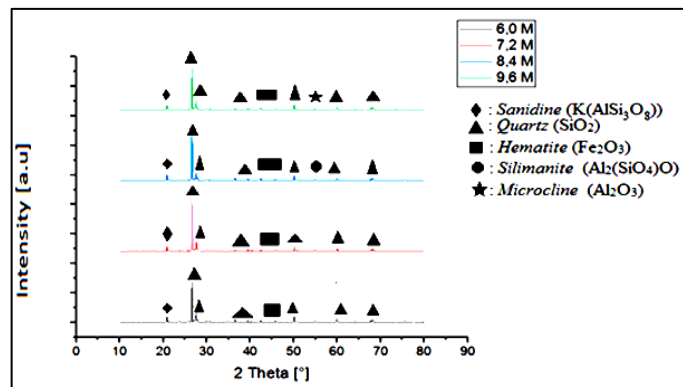


Figure 3. The XRD analysis results for granite with a particle size of 270 mesh and a rotational speed when leaching 750 rpm.

Based on Figure 3, it can be seen that the results of XRD characterization on variations samples leaching of granite with variations in HCl concentrations of 6.0 M, 7.2 M, 8.4 M, and 9.6 M indicate that the phase that dominates the diffraction peaks is Quartz (SiO₂), Sanidine (K(AlSi₃O₈)), Hematite (Fe₂O₃), Sillimanite (Al₂(SiO₄)O) and Microcline (Al₂O₃) consecutively. Phase Quartz (SiO₂) is the phase that dominates the diffraction peaks because SiO₂ is calcined at

temperatures of 800 and 1000 °C (Wibawa, Eko & Anggoro, 2015). The XRF analysis results for leaching granite with a particle size of 400 mesh and a rotation speed of 750 rpm are as in Table 4.

Based on Table 3. and Table 4. it can be seen that the resulting silica content in leaching granite with a particle size of 400 mesh is higher than the silica produced in leaching granite with particle size 270 mesh. This result agreed with the theory of particle size, namely the smaller the particle size, the larger its surface area. Hence, reaction will be faster, and the resulting product will be more and more (Bentz, Garboezi, Haecker & Jensen, 1999).

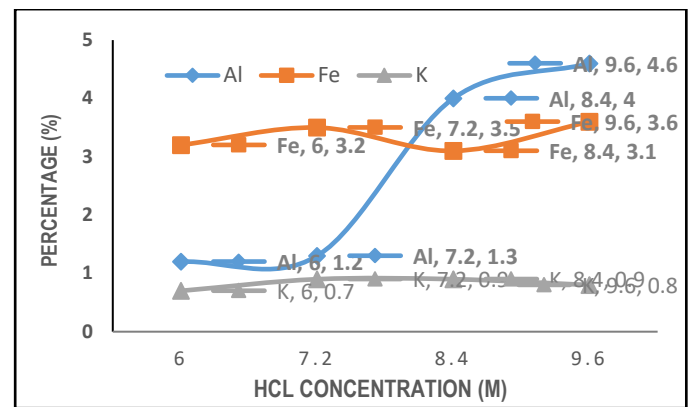


Figure 4. The relationship between HCl concentration and the solubility of Al, Fe, and K, at a particle size of 400 mesh and a rotational speed of 750 rpm.

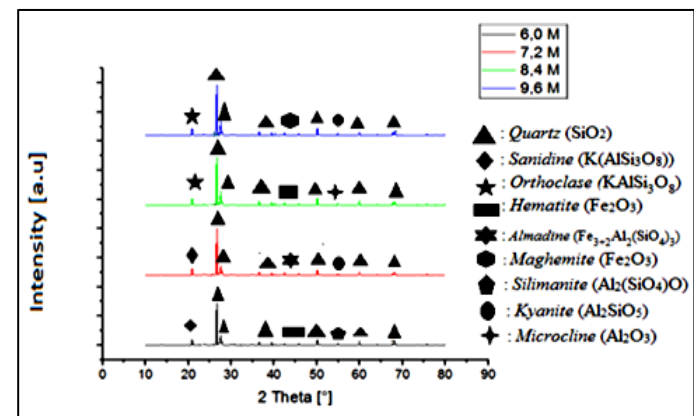


Figure 5. The XRD analysis of granite with a particle size of 400 mesh and rotational speed leaching of 750 rpm.

The analysis sample of the solution was leaching analyzed using ICP-OES to detect chemical elements dissolved during the process leaching. Graph of analysis results

from ICP-OES leaching granite with a particle size of 400 mesh and a rotating speed of 750 rpm is presented as in Figure 4.

The XRD analysis results leaching stone granite with a particle size of 400 mesh and rotation speed 750 rpm are shown in Figure 5.

Based on Figure 5, it can be seen that the phase that dominates the diffraction peaks is Quartz (SiO₂), Sanidine (K(AlSi₃O₈)), Orthoclase (KAlSi₃O₈), Hematite and Maghemite (Fe₂O₃), Almandine (Fe₃₊₂Al₂(SiO₄)₃), Kyanite (Al₂SiO₅), Mullite (Al_{2,35}Si_{1,64}O_{4,82}), Sillimanite (Al₂(SiO₄)O) and Microcline (Al₂O₃). Phase Quartz (SiO₂) is the phase that dominates the diffraction peaks.

The results of XRF of leaching granite with 270 mesh particle size and 500 rpm rotation speed are as in Table 5.

Table 5. The results of XRF Analysis for Leaching Granite with 270 Mesh Particle Size and 500 rpm Rotation Speed.

Compounds	% Leaching			
	6.0 M	7.2 M	8.4 M	9.6M
SiO ₂	68.33	69.05	69.05	69.14
Al ₂ O ₃	16.76	16.57	16.59	16.36
Fe ₂ O ₃	3.08	2.66	2.77	2.73
TiO ₂	0.71	0.70	0.70	0.72
K ₂ O	10.06	9.96	9.84	9.99
P ₂ O ₅	0.79	0.8	0.78	0.8

Table 6. Results of XRF analysis for Leaching Granite with Particle Size 400 Mesh and Rotation Speed 500 rpm.

Compounds	% Leaching			
	6.0 M	7.2 M	8.4 M	9.6M
SiO ₂	68.57	68.92	70.17	70.24
Al ₂ O ₃	17.55	17.73	16.6	16.20
Fe ₂ O ₃	2.44	2.23	2.22	2.35
TiO ₂	0.65	0.62	0.65	0.66
K ₂ O	9.87	9.60	9.44	9.37
P ₂ O ₅	0.71	0.7	0.71	0.77

Based on Table 2 and Table 5, it can be seen that the silica produced in leaching granite with a particle size of 270 mesh with a rotating speed of 750 and 500 rpm, the greater the silica made at speed turn 750 rpm. This is following the stirring speed's effect, the faster the stirring of a particle. When the faster stirred, the contact between the granite powder and the HCl will occur, and the more product is produced at the highest stirring speed (Fitri, 2013).The solution's analysis

sample was leaching analyzed using ICP-OES to detect chemical elements dissolved during leaching. Graph of analysis results ICP-OES leaching granite with a particle size of 270 mesh, and a rotating speed of 500 rpm is presented as in Figure 5.

The results of XRD analysis leaching stone granite with a particle size of 400 mesh and rotation speed 750 rpm are shown in Figure 6.

Based on Figure 6, it found that the phase dominates the diffraction peaks such as Quartz (SiO₂), Sanidine (K(AlSi₃O₈)), Hematite (Fe₂O₃), Ferrosilite and Clinoferrosilite (Fe₂SiO₃), Kyanite (Al₂SiO₅), Mullite (Al_{4,75}Si_{1,25}O_{9,63}), Corundum (Al₂O₃) and microcline (Al₂O₃.K₂O.6SiO₂). Phase Quartz (SiO₂) is the phase that dominates the diffraction peaks.

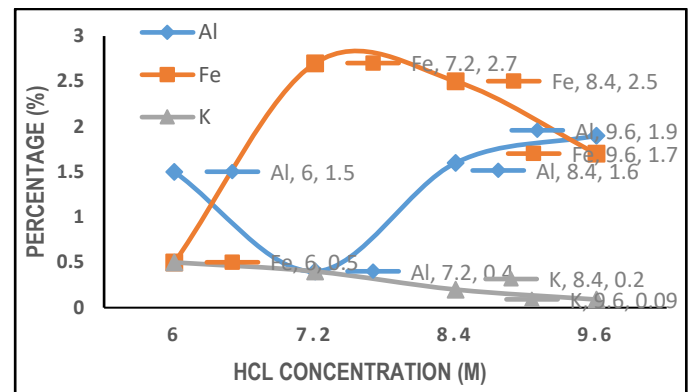


Figure 5. The graph of the relationship between HCl concentration and the solubility of Al, Fe and K, a particle size of 400 mesh and a rotational speed of 750 rpm.

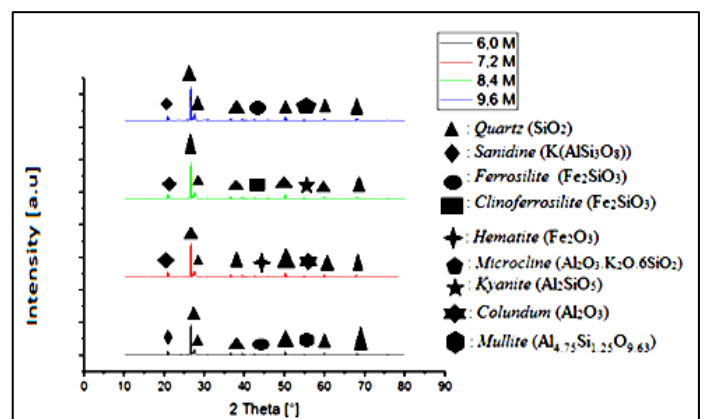


Figure 6. The results of XRD analysis of granite with a particle size of 400 mesh and rotational speed leaching of 750 rpm.

The XRF analysis results of Leaching Granitewith a Particle Size of 400 Mesh and Rotation Speed of 500 rpm are as in Table 6.

Based on Table 5 and Table 6, it can be seen that the silica produced in leaching granite with a particle size of 270 mesh with particle size of 400 is greater than the resulting silica when the particle size is 400 mesh. The silica produced when the particle size is 270 mesh with a rotating speed of 750 rpm as in Table 3 bigger than the silica when particle size is 270 mesh with a rotating speed of 500 rpm as shown in Table 6. So that, the highest silica results at the particle size 400 mesh and leaching at 750 rpm rotational speed. The solution sample was leaching analyzed using ICP-OES to detect chemical elements dissolved during the process leaching. Graph of analysis results ICP-OES leaching granite with a particle size of 400 mesh, and a rotating speed of 500 rpm is presented as in Figure 7.

The results of XRD analysis leaching stone granite with a particle size of 400 mesh and rotation speed 750 rpm are shown in Figure 8.

Based on Figures 9, it can be seen that the silica produced in leaching linear granite rises upward along with the large concentration of HCl used and the fine particle size. Silica with a particle size of 400 mesh is larger than the silica produced in leaching granite with a particle size of 270 mesh. Based on rotation speed, it can be seen that the silica produced in leaching linear granite rises upward along with the amount of HCl concentration used and the rotational speed used. The silica produced in leaching at a rotational speed of 750 rpm is greater than the silica produced in leaching granite at a rotational speed of 500 rpm.

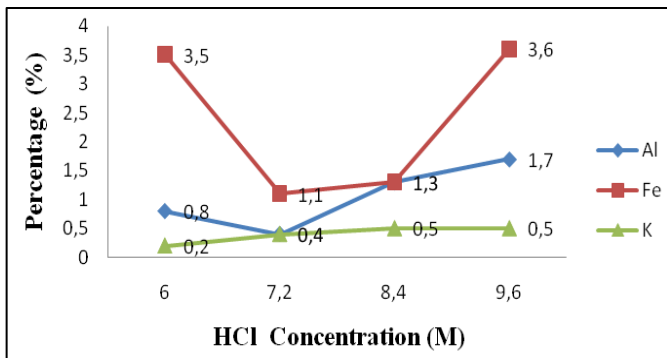


Figure 7. The Graph of the relationship between HCl concentration and the solubility of Al, Fe and K, at a particle size of 400 mesh and a rotational speed of 500 rpm.

Table 7. Phases and types of minerals formed.

Type of mineral	Phase	Percentage (%)
<i>Quartz</i>	SiO ₂	67
<i>Sanidine</i>	K(AlSi ₃ O ₈)	8
<i>Orthoclase</i>	KAlSi ₃ O ₈	1.13
<i>Hematite</i>	Fe ₂ O ₃	8
<i>Maghemite</i>	Fe ₂ O ₃	1.13
<i>Almadine</i>	Fe ₃₊₂ Al ₂ (SiO ₄) ₃	1.13
<i>Ferrosilite</i>	Fe ₂ SiO ₃	3.4
<i>Clinoferrosilite</i>	Fe ₂ SiO ₃	1.13
<i>Silimanite</i>	Al ₂ (SiO ₄)O	1.7
<i>Microcline</i>	Al ₂ O ₃	1.13
<i>Kyanite</i>	Al ₂ SiO ₅	1.7
<i>Mullite</i>	Al _{2,35} Si _{1,64} O _{4,82}	0.56
<i>Colundum</i>	Al ₂ O ₃	0.56
<i>Mullite</i>	Al _{4,75} Si _{1,25} O _{9,63}	0.56
<i>Microcline</i>	Al ₂ O ₃ .K ₂ O.6SiO ₂	0.56
<i>Andalusite</i>	Al ₂ (SiO ₄)O	0.56

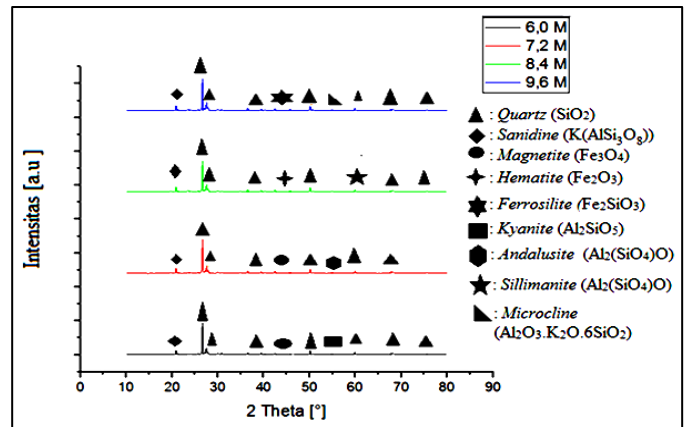


Figure 8. The results of XRD analysis of granite leaching with a particle size of 400 mesh and rotational speed of 500 rpm.

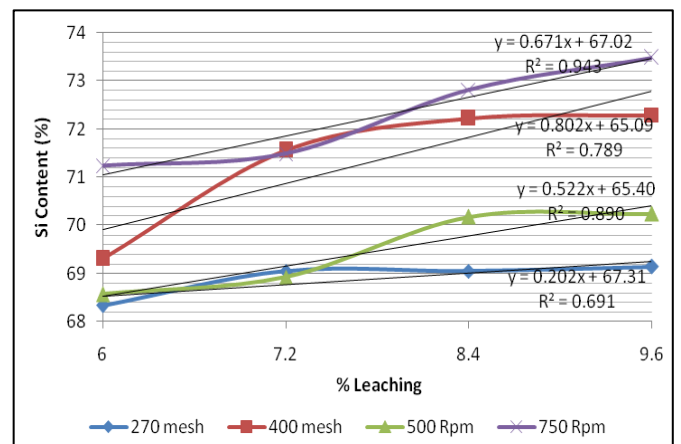


Figure 9. The relationship between silica concentration and various parameters

Several phases and minerals formed in the diffractogram pattern in the XRD analysis are presented in Table 7.

4. CONCLUSION

The highest silica obtained at a concentration of 9.6 M. Thus, the higher the concentration of HCl is used, then the higher the SiO₂ obtained. So that the concentration of the HCl solution affects the solubility of impurity oxides other than SiO₂. The silica obtained at a particle size of 400 mesh is higher than that of 270 mesh. This shows that the finer the particle size, the higher the silica is obtained. This is due to the wider the surface area of the particles, the faster the HCl solution will reduce impurity oxides other than SiO₂. Silica obtained during leaching at a rotational speed of 750 rpm is higher than at 500 rpm. This shows that the higher the rotational speed when leaching, the higher the silica will be obtained. This is because the rotational speed affects the friction that occurs, so the higher the rotational speed or, the faster the stirring, the faster the HCl solution will dissolve other impurity particles besides SiO₂.

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