

BENEFICIATION OF SAMBIROTO SILICA SAND BY CHEMICAL AND BIOLOGICAL LEACHINGS

BENEFISIASI PASIR SILIKA SAMBIROTO DENGAN PELINDIAN SECARA KIMIA DAN BIOLOGI

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ABSTRACT

The commercial value of silica sand significantly affected by the presence and content of iron, aluminium and other metal impurities that can have detrimental effects on the manufactured product. A leaching technology on Sambiroto silica sand had been studied using chemical (HCl and H₂SO₄) and biological (Aspergillus niger's metabolite) methods to remove undesired metal impurities and obtain a high purity silica. The rates and extent of chemical leaching and bioleaching were different depending on the leaching agents used and their concentrations. The results showed that the laboratory experiments improved the silica sand sample to a high grade purity. The major and minor impurities of Fe, Al, Ca, Cr, Ti, Zr and Cu were reduced significantly by acid leaching with HCl and H₂SO₄ 4M at 90°C, 30% pulp density for 4-hours process. The chemical composition of the silica sand improved with the SiO₂ content increased from 97.24 to 98.77%, on the other hand, the sum of impurities decreased from 1.148 to 0.237 %. These changes bring the beneficiated product very close to a feedstock of metallurgical grade silicon for advanced materials. Among the tested methods, chemical leaching is the most efficient in terms of both increasing percentage of SiO₂ and metal impurities removal compared to biological leaching. Nevertheless, bioleaching process is sufficient to produce silica sand with a desired minimum value of Fe and Al contents for high quality glass industries. Both process characteristics could facilitate their industrial applications.

Keywords : beneficiation, silica sand, chemical leaching, bioleaching, metallurgical grade silicon

SARI

Nilai komersial bahan baku pasir silika sangat dipengaruhi oleh adanya kandungan besi, aluminium dan logam-logam pengotor lainnya yang dapat mengganggu produk manufaktur. Teknologi pelindian pasir silika Sambiroto telah diteliti menggunakan metode kimia (menggunakan HCl dan H₂SO₄) dan biologi (menggunakan metabolit Aspergillus niger). Laju pelindian secara kimia dan biologi berbeda bergantung kepada agen pelindi yang digunakan dan konsentrasinya. Hasil percobaan menunjukkan proses benefisiiasi dapat meningkatkan kualitas percontoh pasir silika hingga mencapai kemurnian tinggi. Pengotor utama dan minor Fe, Al, Ca, Cr, Ti, Zr dan Cu berkurang secara nyata dengan pelindian HCl dan H₂SO₄ 4M yang berlangsung pada suhu 90°C dengan 30% padatan selama 4 jam. Komposisi kimia pasir silika membaik dengan meningkatnya SiO₂ dari 97,24 menjadi 98,77%, sedangkan jumlah pengotor menurun dari 1,148 menjadi 0,237 %. Perubahan ini menjadikan produk benefisiiasi sangat mendekati spesifikasi umpan metallurgical grade silicon untuk material maju. Di antara ketiga metode yang diuji, pelindian kimia dengan HCl dan H₂SO₄ paling efisien dalam hal meningkatkan persentasi kandungan SiO₂ dan penghilangan logam pengotor dibandingkan pelindian secara biologi. Namun demikian, pelindian biologi mampu memproduksi pasir silika dengan kandungan minimal Fe dan Al untuk peruntukan industri gelas kualitas tinggi. Kedua karakteristik dari dua metode itu memungkinkan untuk aplikasinya dalam skala industri.

Kata kunci : benefisiiasi, pasir silika, pelindian kimia, pelindian biologi, metallurgical grade silicon

INTRODUCTION

Silica sand is one of the most abundant material that finds many applications in the manufacture of glass, ceramics, refractory materials, paper making and other traditional uses. However, only very few deposits are suitable in both quality and amenability to fit refining methods for speciality high purity applications. The silica sand containing 500 ppm of iron and 800 ppm of aluminium is ready to be used in glass industry (Banza et al., 2006), but in the photovoltaic industry, for instance, the required parameters are more strong (Fe < 100 ppm, Al < 400 ppm). A very high purity silica sand has become one of today's key strategic minerals with applications in high-tech industries including semiconductors, high temperature lamp tubing, telecommunications and optics, microelectronics and solar silicon applications (Haus, 2010, Dal Martello et al., 2012). The International Occultation Timing Association (IOTA) Grade Standards classified silica sand used for electronics and photovoltaic into 3 categories depending on the end uses as shown in Table 1.

The extent of the applications depends greatly on associated unique physical, physicochemical and chemical properties, which could be tailored by means of various approaches. Beneficiation is a

process whereby solid materials are refined and unwanted impurities are removed using physical, chemical and biological methods. Beneficiation of raw silica sand into refined high-purity products involves several refinement steps, which need to be adapted to effectively minimize the specific impurities of the individual raw mineral feed to comply with stringent end-use specifications (Haus, 2005). Established approaches involve chemical, mechanical and physical methods. Silica sand from Ardhuma of Iraq, for instance, containing 900 ppm Fe was purified by physical and chemical techniques including washing, sieving, magnetic separation, then treated by 5% HCl to produce final sand with 160 ppm Fe (Daykh and Mahdy, 2005).

The preferred beneficiation methods of silica sand depend on the amount and the nature of the mineral impurities associated to it. Although those methods are quite useful in removing impurities, but they are normally costly and complicated environmentally hazardous. Bioleaching processes are usually more economical, ecofriendly and uses less amount of energy compared to other methods (Anjum et al., 2010; Akiril, 2007; Rawling, 2004). *Aspergillus niger* is one of the most widely used fungi in bioleaching, which is used commercially in the production of organic acids

Table 1. The purity of metallurgical-grade silicon and solar-grade silicon (Xakalashe and Tangstad, 2011)

Element	Concentration (ppm)		Solar-grade (Si)
	Metallurgical-grade (Si)		
	98-99%	99,50%	100,00%
Al	1000-4000	50-600	<0.1
Fe	1500-6000	100-1200	<0.1
Ca	250-2200	100-300	< 1
Mg	100-400	50-70	< 1
Mn	100-400	50-100	< 1
Cr	30-300	20-50	< 1
Ti	30-300	10-50	< 1
V	50-250	<10	< 1
Zr	20-40	<10	< 1
Cu	20-40	<10	< 1
B	10-50	10-15	0.1-1.5
P	20-40	10-20	0.1-1
C	1000-3000	50-100	0.5-5

such as citric, oxalic and gluconic acids. Those acids have found application as lixiavants for the leaching of heavy metals from ore materials and kaolin (Anjum et al., 2010; Hosseini et al., 2007; Cameselle et al, 2003), and may reduce the environmental impacts resulting from the mode of treatment (Mulligan et al., 2004). A. Niger has also been found to overproduce organic acids that can serve as leaching agents for the solubilization of Al, Fe, Mn, Ni, Pb, Cd, Cu, Zn from fly ash (Aung and Ting, 2005) and leaching of heavy metals in contaminated soil (Wan-Xia et al., 2009).

Indonesia has a huge reserve of silica sand, but no progressive study on this material deposits, points to increased level of impurities resulting to limited application. To improve the quality of silica sand for enhanced usage, the inherent impurities must be removed or reduced through more economic and ecofriendly techniques. Accordingly, this paper presents a comparative approach (chemical and biological) for beneficiating silica sand from Mojosari, Central Java. Silica sand used for feedstock of metallurgical grade silicon has certain permissible limits of impurities, especially iron and aluminum oxide, which is considered the main limiting factor for the sand. Therefore, a high purity silica sand with total impurity levels less than 1% should be achieved to create a highly valuable raw material and that is the main objective of this study.

METHODOLOGY

Chemicals and Silica Sand Sample

All reagents used in this study were of analytical grade. Sample of silica sand was taken from Sambiroto, Central Java. The sample was washed (desliming), cleaned, filtered and dried prior to crushing, ball milling and sieving. The sample was then subjected to mixing, quartering and dividing to obtain the representative samples. The sand sample used has a particle size distribution within a narrow size range of - 80+120 mesh. The prepared sand was sampled for characterization purposes by X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD).

Experimental Procedures of Chemical Leaching

All experiments were conducted in a mechanically 4-L stirred chemglass reactor equipped

with thermostatically controlled heating mantle connected with mercury contact thermometer as shown in Figure 1. A 2000 ml of acid solution (HCl or H₂SO₄) with varying concentrations of 2 and 4 M was heated in the glass reactor at 90°C. The solution was agitated at 250 rpm. Then, a pre-weighed amount of dry sand (600 gr) was added to the solution, creating suspension with 30% pulp density. This chemical leaching process was performed for 4 hours. The concentrations of dissolved metals in solution for each test were then measured as a function of time using atomic absorption spectrophotometer (AAS).



Figure 1. Process of chemical leaching

Microorganism, Cultivation and Bioleaching Procedures

The microorganism used in this research was *Aspergillus niger* that had been isolated from Karimun quartz sample. The composition of media for cultivation was as follows (g/L): glucose 35, NaNO₃ 1.5, KH₂PO₄ 0.5, MgSO₄·7H₂O 0.025, KCl 0.025, and yeast extract 1.5. The nutrient solutions were buffered with TRIS at a concentration of 1 M. The pH value was adjusted with NaOH to 6.0 before sterilization process at 121°C for 30 minutes.

The cultivation of *Aspergillus niger* to obtain metabolite as a lixiviant was conducted in a fully controlled fermentor (New Brunswick Bio Flo Celligen 115) that was adopted from previous work by Handayani (2011). The operational parameters of fed batch run were as follows:

- working volume : 1 L
- rpm (day 1) : 100
- rpm (day 2 to 14) : 150
- air flow rate : 0.5 vvm
- temperature : 30°C
- initial glucose : 10 g/L
- addition of glucose : days 4, 5, 6, 7, 8 (5 g/L each until total of 35 g/L)
- dissolved oxygen : 20%
- pH : maintained at 6.0 using NaOH 2N
- fermentation period : 14 days

After 14 days of cultivation, the medium was separated by filtration and the fungi-free supernatant used as a leaching agent for biological beneficiation process. Bioleaching the 180 gr of silica sand samples was carried out using 600 ml metabolite in the 1L-stirred reactor. The leaching process was performed at 250 rpm at room temperature for 8 hours. The concentration of generated metals ions was measured every one hour. All experiments were carried out in duplicates and the reported values were the average values. Maximum deviation from the average value was found to be less than 5%.

Analytical Measurements

XRD and XRF instruments were used for mineral characterization while the Atomic Absorption Spectrophotometer was used to determine the concentration of metal ions in the liquids samples taken during the leaching process. The concentration of oxalic acid produced in the metabolite was determined by Bergemann and Elliot method.

RESULTS AND DISCUSSIONS

Mineralogical and Chemical Composition of Raw Material

The mineralogical analysis using XRD shows that the silica sand sample used in this study consists mainly of quartz (76% SiO₂) and feldspar (24%) as presented in Table 2 and Figure 2.

Table 2. Mineral composition of Sambiroto silica sand

Mineral	Chemical formula	%
Quartz	SiO ₂	74
High grade quartz	SiO ₂	1
Cristobalite	SiO ₂	1
Feldspar/Nepheline	Na ₃ K(Si _{0.56} Al _{0.44}) ₈ O ₁₆	24

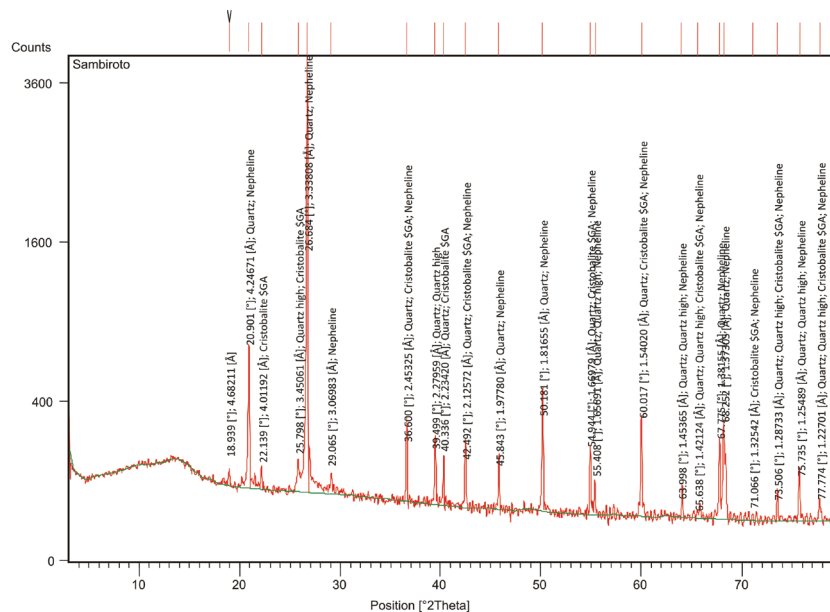


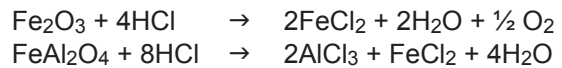
Figure 2. Raw silica sand characterization by XRD

The XRF results showed that the chemical composition of sample has total SiO₂ of 97.24% along with some undesirable impurities as shown in Table 3. The mineral also contains high level aluminum and iron, which are 6600 and 3000 ppm respectively. Fortunately, several impurities as prerequisite for metallurgical and solar-grade silicon such as Mg, Mn and V are undetectable.

Chemical Leaching with HCl

The experiments were carried out by varying HCl concentrations of 2 and 4M at temperature of 90°C and pulp density of 30% (w/v). In this study, the release of Fe, Al and other metal impurities was used as indicator of mineral dissolution and beneficiation of silica sand quality. The dissolution of iron and aluminum as a function of time for the above experiment is presented in Figure 3. It can be seen that under such experimental condition, the maximum iron and aluminum dissolutions are approximately 91.3 and 86.1 % respectively. This extraction is obtained by leaching the sand using HCl at concentration of 4M. The iron and aluminum dissolution is significantly accelerated as HCl concentration increases from 2 to 4M as can be

seen in Table 3. The kinetics profile of leaching shown in Figure 3 suggest that when HCl is used as the leaching reagent for solubilizing Fe and Al from the sand after a very effective solubilization in the first two hours of exposure to the acid solution, there is a stagnant condition in the amount of metal extracted. By using lower concentration of HCl (2M), the solubilization of Fe and Al was drastically reduced compared to HCl 4M (Table 4). The chemical reaction between HCl and the iron and aluminum can be represented by the generic expression as follows (Alafara et al., 2005):



From the results presented in Table 4, it is observed that HCl strength of 4M is sufficient for the optimal leaching of Sambiroto silica sand. Al, Fe, Ca, Cr and Cu reduce progressively while Cr and Ti remain almost unchanged especially at lower acid strength. Simultaneously, SiO₂ increase with the increase in reaction time, associated with leaching of Fe, Al and some other metal ions, from 97.24% SiO₂ in raw material to 98.77% in the treated silica sand after leaching with 4M HCl.

Table 3. Chemical composition of silica sand sample with impurities quantification

Location	Concentration of SiO ₂ (%) and impurities (ppm)										
	SiO ₂	Al	Fe	Ca	Mg	Mn	Cr	Ti	V	Zr	Cu
Sambiroto	97.24	6600	3000	689	ud	ud	120	371	ud	148	551

Note : ud = undetectable

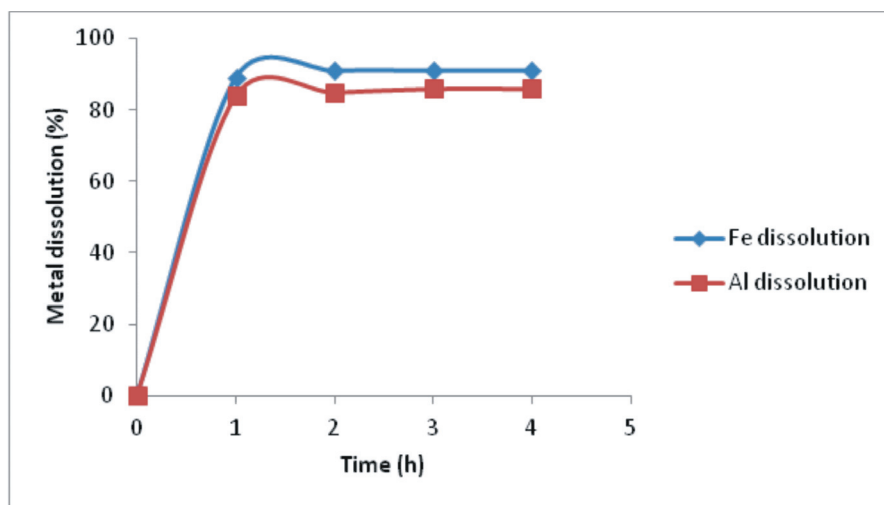


Figure 3. Kinetics profile of iron and aluminium removal by HCl (4M) leaching

Table 4. Chemical composition of silica sand sample after leaching with HCl

Treatment	Concentration of SiO ₂ (%) and impurities (ppm)										
	SiO ₂	Al	Fe	Ca	Mg	Mn	Cr	Ti	V	Zr	Cu
Raw Material	97.24	6600	3000	689	ud	ud	120	371	ud	148	551
Leaching-HCl 2M	98.08	3800	1650	464	ud	ud	118	361	ud	116	322
Leaching HCl 4M	98.77	920	960	98	ud	ud	110	261	ud	35	21

Note : ud = undetectable

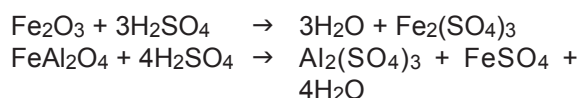
The sum of unwanted metal impurities decreases from 1.148 to 0.237%. This product has met the specification of feedstock for metallurgical grade silicon.

Chemical Leaching with H₂SO₄

One comparative test was also carried out at the same experimental conditions using H₂SO₄ as the leaching agent. The dissolution curve of Fe and Al from the silica sand sample in H₂SO₄ solution is shown in Figure 4. The Fe and Al extractions increase rapidly during the first hour and remained nearly constant after 2 hours of leaching process, reaching 81.9 and 91.1% extracted Fe and Al respectively. It can be seen that the shape of the kinetics curve is similar to that of leaching with HCl and also the removals of Fe and Al increase with the increase of H₂SO₄ concentration from 2 to 4M (Table 5).

The leaching run with H₂SO₄ 4M displays the highest rate of impurities reduction reaching the SiO₂ purity of 98.65% after 4-hour process. The data of increasing SiO₂ simultaneously with the removal of Al, Fe and other metal impurities from the silica sand sample are summarized in Table 5.

The reaction equations of Fe and Al with H₂SO₄ have been reported as follows (Ledgerwood and van der Westhuyzen, 2011) :



Extraction of Al is more rapid in comparison with Fe extraction suggesting that H₂SO₄ has a good ability to dissolve Al. In this leaching, the Cr and Ti removal is only 8.3 and 21.8% after 4-hours leach-

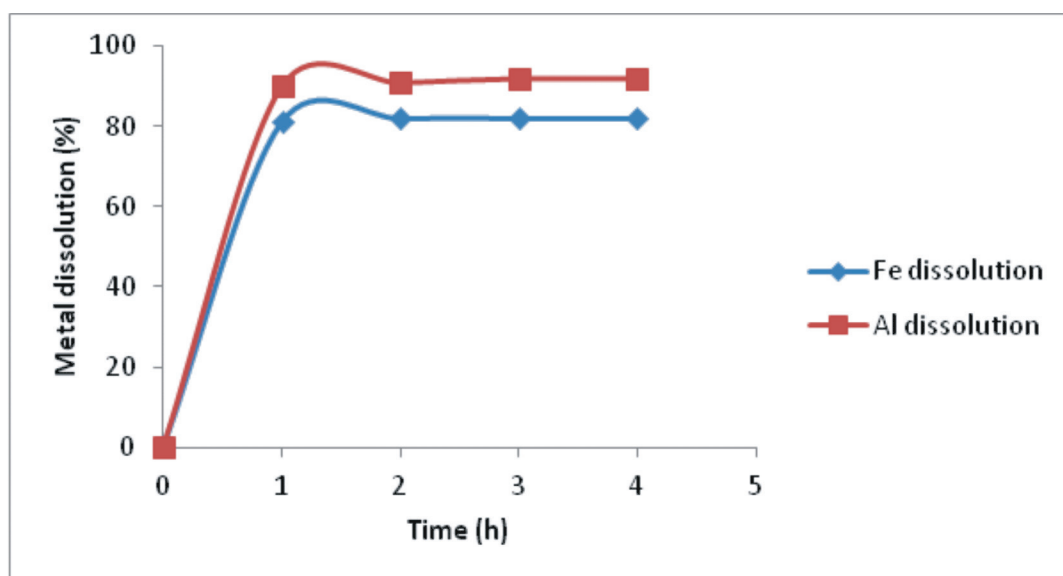


Figure 4. Kinetics profile of iron and aluminium removal by H₂SO₄ leaching

Table 5. Chemical composition of silica sand after leaching by H₂SO₄

Treatment	Concentration of SiO ₂ (%) and impurities (ppm)										
	SiO ₂	Al	Fe	Ca	Mg	Mn	Cr	Ti	V	Zr	Cu
Raw Material	97.24	6600	3000	689	ud	ud	120	371	ud	148	551
Leaching-H ₂ SO ₄ 2M	97.52	3560	2140	330	ud	ud	110	298	ud	140	210
Leaching-H ₂ SO ₄ 4M	98.65	587	543	61	ud	ud	110	290	ud	38	32

Note : ud = undetectable

ing. This low acidic solubility of Cr and Ti probably because the metals bound in silica structure.

Bioleaching

The bioleaching operations, performed with metabolite produced by *A. niger* as the leaching agent was carried out in a reactor which was keep stirred at 250 rpm at room temperature for 8 hours. The removal of iron and aluminum from the silica sand by the metabolite alone is presented in Figure 5 and Table 6. The removal rate was high between

hour 2-7 of process and after that it remains constant. There was a strong relationship between the rate of iron and aluminum removal and oxalic acid concentration. The metabolite contained loose slime and organic acids (included oxalic acids) which are solely responsible for metals removal. The chemical analyses showed that the metabolite contained 25 g/L (0.2M) of maximum oxalic acid production and it was used up for the bioleaching process. After the first 7 hours, the oxalic acid concentration remained stable and so was the removal rate.

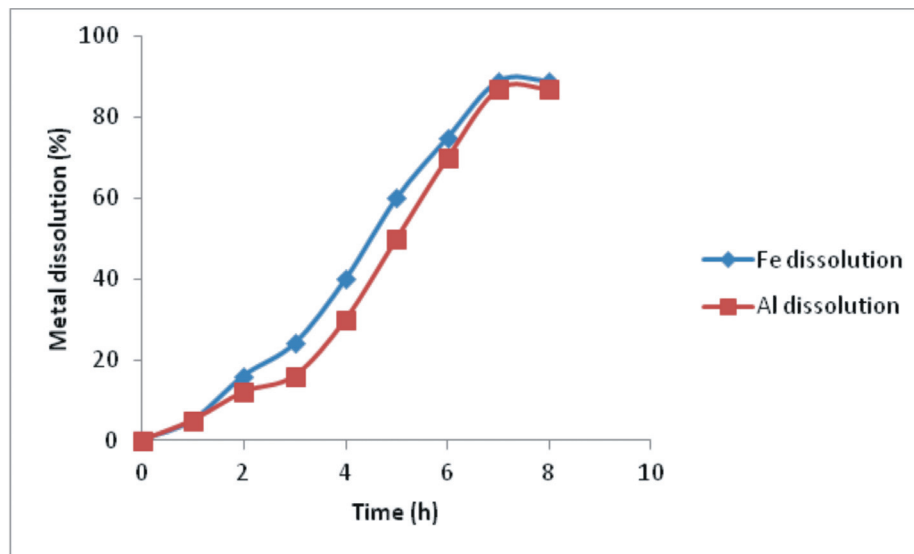


Figure 5. Iron and aluminium dissolution by metabolite of *Aspergillus niger*

Table 6. Chemical composition of silica sand sample after bioleaching

Treatment	Concentration of SiO ₂ (%) and impurities (ppm)										
	SiO ₂	Al	Fe	Ca	Mg	Mn	Cr	Ti	V	Zr	Cu
Raw Material	97.24	6600	3000	689	ud	ud	120	371	ud	148	551
Bioleaching	98.38	800	336	624	ud	ud	ud	320	ud	130	ud

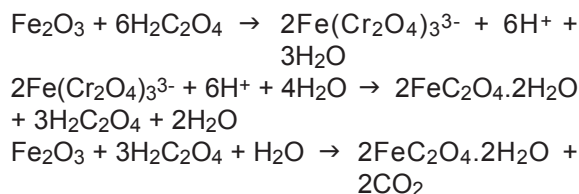
Note : ud = undetectable

Analyzed altogether, the results expressed in Table 6 and Figure 5 show that bioleaching gave more higher Fe removal and reach the maximum removal of iron and aluminium 88.8 and 87.9% respectively, but 320 and 130 ppm of Ti and Zr are still remained in the treated sand. The metal dissolution rates seem to be slower and need longer time (7 hours) compared to chemical leaching (2 hours). However, the advantage of this bioleaching process is that the metabolite could remove all the Cr and Cu completely from the sand. This observation clearly indicates that the bioleaching has positive effects in removing metal impurities from the sand but is not as effective as HCl and H₂SO₄ because the result of bioleaching does not meet the metallurgical grade silicon specification for electronics or other advance applications yet. Nevertheless, this experimental condition is sufficient to produce silica sand with a desired minimum value of Fe and Al contents for high quality glass industry.

By comparing the effectiveness of extracting Fe and Al from the silica sand expressed in Table 3, 4 and 5; it is observed that the metals dissolution was more effective when chemical leachings were performed using 4M HCl and H₂SO₄ than using the metabolite of *A. niger*. The results of this study seems to be disagreement with those reported by Mustafa et al. (2011) who showed that bioleaching was very effective in extracting metals from silica sand which reached 85 ppm of Fe in the final product. The different results suggest that the type of minerals, the metal impurities and their concentrations present a different susceptibility to the leaching process.

The present bioleaching experiment was run in a medium at ambient temperature (around 25°C) without vitamins and other supplements such as oligoelement. Probably by increasing temperature to 30°C and with addition of vitamins would likely optimize the rate and extent of the bioleaching process and may allow reaching values similar to those reported in the literature for bacterial reduction of Fe (Jaisi et al, 2007). However, the use of higher temperature and additives would increase the overall cost of the refinement procedure. It has been known that *Aspergillus niger* produced enzymes and oxalic acid specifically to degrade and solubilize minerals. They break down mineral structures and extract the elements required for metabolism or structural purposes (mineralases). This is especially important for ions such as Fe and Al. The probable chemical reactions of oxalic

acid with the iron can be drawn as follows (Salmimies et.al., 2011; Lee et al., 2007) :



Fungi interact with minerals and synthesize an array of organic compounds that have been shown to affect the mobility of metal ions. Their biological activity is high and dependent on production of organic acid such as oxalic acid. They play an important role in the iron and other metal removal from silica sand. From a practical point of view, the high yield of rate of extracting metals from silica sand by the liquor of *A.niger* culture is noteworthy. This biological process present two advantages as compared to chemical leaching processes: (i) the very low concentrations of organic compounds present in this process represent a lower ecological risk and (ii) even with a lower final yield, the economical cost of this process may be considered as it is performed at ambient temperature. Both advantages could facilitate its industrial applications.

CONCLUSION AND SUGGESTION

The Sambiroto silica sand deposit is economically interesting raw material but its properties should be improved by removing undesirable metal impurities. It is evident that both chemical and biological leachings have an important role in the beneficiation process of Sambiroto silica sand samples. The choice and the optimal concentrations of the selected reagents and lixiviant allow to obtain high purity silica sand. From a raw sand containing 3000 ppm Fe and 6600 ppm Al, a product containing 260 ppm Fe and 920 ppm Al was obtained after 4-hours by HCl attack and 543 ppm Fe and 587 ppm Al by H₂SO₄ attack. Both results have met the specification of metallurgical grade silicon. Meanwhile, bioleaching product still contains 800 ppm Al, 336 ppm Fe, 320 ppm Ti and 130 ppm Zr. Those results suggest that chemical leaching using HCl and H₂SO₄ is more effective compared to metabolite of *A. niger* in the dissolution of metal impurities from the silica sand. Chemical leaching is a suitable way for the improvement of qualitative properties of silica sand used in the advanced industry and bioleaching

route for high quality glass industry. Bioleaching seems to be less effective but very advantageous with the respect to ecology and might be economical costs although the processing time is longer. Further research and development should focus on optimizing the rate and extent of these processes before their application at a larger pilot or industrial scale. In particular, further studies should evaluate the environmental and economical benefits comparing those approaches.

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