Silica extract from Malang beach sand via leaching and sol-gel methods

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Article Info

Article history:

Received Sep 9, 2022
Revised Oct 14, 2022
Accepted Oct 24, 2022

Keywords:

Bajul Mati beach
Extracted silica
Leaching
Sol-gel

ABSTRACT

Many silica minerals are found in beach sands. One of its beach sands is in Bajul Mati beach sand which contains high silica. The silica can be extracted using existing methods, but it is less environmentally friendly. In this research, the extracting process of silica from the sand of Bajul Mati beach (South of Java-Indonesia) has been carried out through a method that is low-cost and more environmentally friendly. The purpose of this study was to characterize extracted silica with instrumentation results and economic analysis. The method of extracting silica from beach sand was via leaching and sol-gel methods: i) leaching with 7 M HCl, ii) the formation of clear sodium silicate solution ($Na_2O.SiO_2$), iii) sol-gel: sodium silicate solution titrated with oxalic acid ($H_2C_2O_4$) 1 M until pH 6 was obtained and silicite $Si(OH)_4$ was formed, and iv) characterization with X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier transform infrared (FTIR), and scanning electron microscopy (SEM) with economic analysis. The result showed that extracted silica has a cristobalite structure based on XRD analysis. The purity of silica (% Wt Si) reached 93.9% based on XRF analysis. FTIR analysis has identified silanol, siloxic, and siloxane groups. The SEM analysis shows that silica (SiO_2) particles tend to form agglomerations about ~1 μm. In addition, economic analysis results showed that this method will support implementation in the future, so silica has been produced by this developed method.

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1. INTRODUCTION

Silica (SiO_2) is silicon dioxide that is often found abundantly in the earth's crust [1]. Silica can be obtained naturally or synthetically. Silica minerals are difficult to obtain in nature, usually from mining. As an alternative, silica is synthesized using template materials such as tetraethoxysilane (TEOS) and Tetramethyl orthosilicate (TMOS) by melting method. Because the process is complicated and synthetic silica is relatively expensive, natural silica has the potential as a source of raw materials, where the silica can be found in sand, rocks [2], plants [3]–[5], and sponges (*Phylum Porifera*) [6].

Silica is widely applied in various applications because silica has unique characteristics such as low density, low thermal conductivity, high surface area, and high specific strength [7]. Some applications of silica are the main ingredients of the glass and ceramic industries [8], raw material for solar cells [9], pozzolanic materials [3], cement mixture [10], silica in nanocomposite bioactive materials was applied to bone tissue repair [11], nano-silica is used by industries related to the production of pigments, pharmaceuticals [12], photocatalyst support [13], catalyst [14], filler material for polymer material modification [15], adsorbent of industrial waste [16], [17], adsorbent of plant nutrients [18], zeolite synthesis material [19], [20], and as a matrix in the immune-affinity column [21]. In addition, silica is used in the coating industry as a corrosion inhibitor [22], in the food industry and anti-fungus agents [23], and used in blood analysis [24]. Thus, a silica extraction needs to be developed with various advantages.

Purification of natural silica has been carried out and has achieved a purity of about 95%. Samsudin has reported that silica from ash waste of the sugar industry has been successfully purified with levels >99% purity [25]. Then, Nuntiya [26] succeeded in synthesizing Nano silica from rice husk ash which reached 98% purity by co-precipitation method. Previous research also managed to extract silica from high-purity rice husks using the sol-gel method with the use of HCl [27] and H_2SO_4 [28]. However, the applied sol-gel method still uses inorganic acid (HCl, H_2SO_4) in the gel formation stage. The use of HCl or H_2SO_4 can be said to have high costs and is less environmentally friendly. The potential use is an organic acid that has a low cost and is more environmentally friendly. Research needs to be developed with the use of organic acids to obtain methods that are more environmentally friendly.

In this study, the extraction of this study will be carried out by combining the leaching method and the organic acid sol-gel method, i.e.: 1 M oxalic acid. Organic acid has more environmentally friendly. The advantages of the sol-gel method compared to other methods are due to low energy consumption (<200 °C) and low cost.

2. RESEARCH METHOD

The process of silica extraction from the sand of Bajul Mati beach was carried out in four stages. First, beach sand leaching treatment with 7 M HCl. Secondly, the preparation of sodium silicate (Na_2SiO_3) from beach sand with a strong sodium hydroxide (NaOH) base. Third, the preparation of silicic, $Si(OH)_4$, sodium silicate solution reacted with oxalic acid $H_2C_2O_4$ (organic acid) to form precipitation (silica gel) which is still mixed with an alkaline salt. Because $Si(OH)_4$ cannot dissolve in acid. Then the precipitated $Si(OH)_4$ was separated from the solution. fourth, silica preparation, SiO_2, was by drying $Si(OH)_4$ silica gel.

The material used in this study was beach sand taken in Bajul Mati area, Gajahrejo Village, Malang with a content of 31.0 wt% SiO_2 (initial results of X-ray fluorescence (XRF) test), HCl solution 37% pa (Merck), NaOH pa (Merck), $H_2C_2O_4$ anhydrous pa (Merck), aqua demineralization, and distilled water. Instruments used in this study were beaker glass (size: 100, 250, 500, and 1000 ml), pipette, spatula, funnel, mortar, aluminum foil, thermometer, Whatman filter paper number 40, and filter paper. All glass instruments are used from the Pyrex brand. Oven (Memmert UN 55), analytic scales (ATX22), magnetic stirrer, hot plate (SP 131320-33Q digital stirring hotplate cimarec), X-ray diffraction (XRD) PANanalytical X'pert pro, XRF PANalytical Minipal 4, Fourier transform infrared (FTIR) Shimadzu IRPrestige 21, and scanning electron microscopy (SEM) FEI type Inspect-S50.

The experiment began with sample preparation. The beach sand of Bajul Mati Malang Regency was crushed using a mortar and filtered to obtain a homogeneous particle size. After that, 50 grams of dry sand powder was mixed with 50 mL HCl 7 M with a stirrer for 1 hour and a heating temperature of 150 °C. Then, decantation was carried out to separate the impurities in the mixture 15 grams of sand powder from leaching mixed with 15 grams of NaOH solid which was accompanied by a heating temperature of 300 °C for 2 hours. Then, the mixture was added with 150 mL aqua demineralization. The obtained sodium silicate solution was filtered with Whatman paper number 40. Then, it was diluted with 100 mL aqua demineralization and titrated with $H_2C_2O_4$ (1 M) until the pH of the solution approaches neutral (pH 6-7) so a cloudy solution began to form a sol ($Si(OH)_4$) accompanied by heating 200 °C. The precipitated $Si(OH)_4$ (silica gel) was aging for 9 hours later. Next, it was filtered with filter paper and washed with aqua demineralization by several times to clean up the residues of alkaline salts that were still in the gel. Then, it was dried in an oven at 110 °C for 12 hours. SiO_2 was tested for XRD, XRF, FTIR, and SEM.

3. RESULTS AND DISCUSSION

3.1. XRF and XRD analysis

The results of the XRF test performed on silica sand samples as listed in Table 1, elements in the percentage of mass (%). Based on Table 1, the chemical elements found in silica samples from Bajul Mati beach sand have the majority of chemical elements (Si, Ca, and Fe) and the majority of oxide compounds (SiO_2, CaO, and Fe_2O_3) along with the other impurities. It also appears that the results of silica synthesis with this method obtained Si percentage which increased from 31.0 wt% to 93.9% wt.

Table 1. XRF data analysis results of sand samples and extracted silica

Sample	Element (%wt)							
	Si	K	Ca	Fe	P	Sr	Ti	Others
Beach sand	31.0	0.77	45.3	19.0	0.0	1.8	1.41	<0.01
Silica	93.9	0.86	2.0	0.7	2.5	0	0	<0.01

The results of the XRD test on sand samples of Bajul Mati beach, Malang, East Java, Indonesia, showed that the crystal field (peak) was dominated by the quartz phase. The analysis obtained from the software Match! version 3.0. The crystalline phases of SiO_2 can be quartz, tridymite, and cristobalite. The X-ray diffraction pattern from silica synthesis by leaching method and organic acid sol-gel, with titration pH 6-7, is shown in Figure 1, where Figure 1(a) shows the experimental pattern of silica and Figure 1(b) shows the experimental pattern sand of Bajul Mati beach.

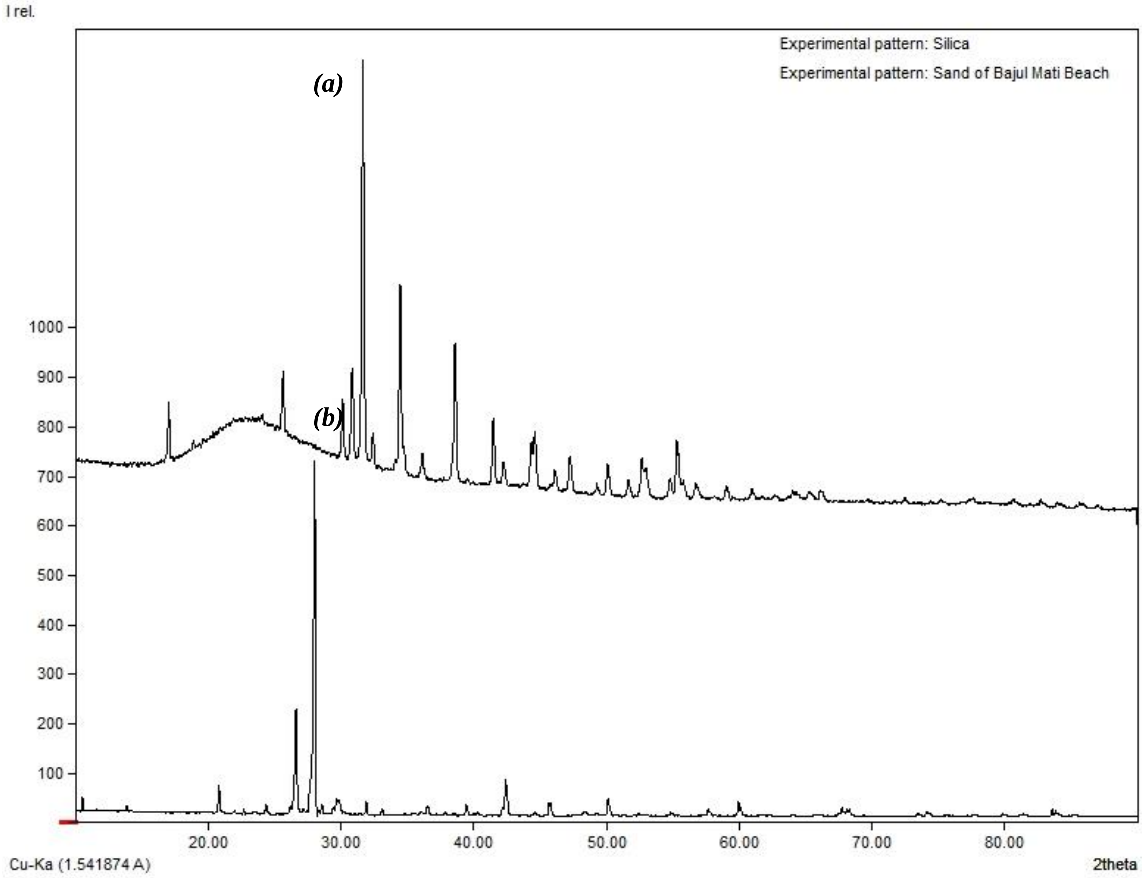


Figure 1. The XRD pattern of extracted (a) silica and (b) Bajul Mati sand sample

From the search results of Match! version 3.0 software, crystalline cristobalite phase peaks are formed. In addition, there is another peak in silica, namely the natroxalate (oxalate salt) phase. This is similar to the Kalapathy research [29]. However, it can be seen that NaOH is successful in the process of forming sodium silicate (Na_2SiO_3) to produce silica (SiO_2). Furthermore, the SiO_2 phase formed changes from the quartz phase to the cristobalite phase. This is due to the influence of temperature used in gel formation where changes in the crystal phase are affected by temperature. Based on Figure 1, the results of this study allow it to be studied, that theoretically, oxalic acid can turn into CO_2 gas when it is put in an oven. In contrast to the results of this study, the oxalate group still binds to the silica matrix, so the peak of the oxalate group was detected on XRD.

3.2. FTIR analysis

The FTIR shows a formed absorption patterns and it is to determine the functional groups of the sample. The sample test results are shown in Figure 2. FTIR interpretation is to explain the formed functional groups can be seen in Table 2.

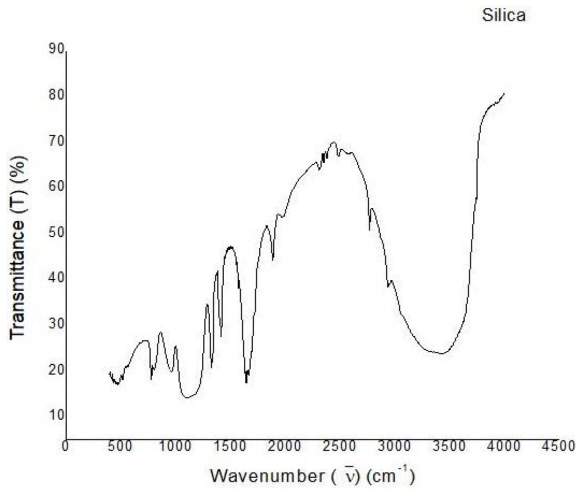


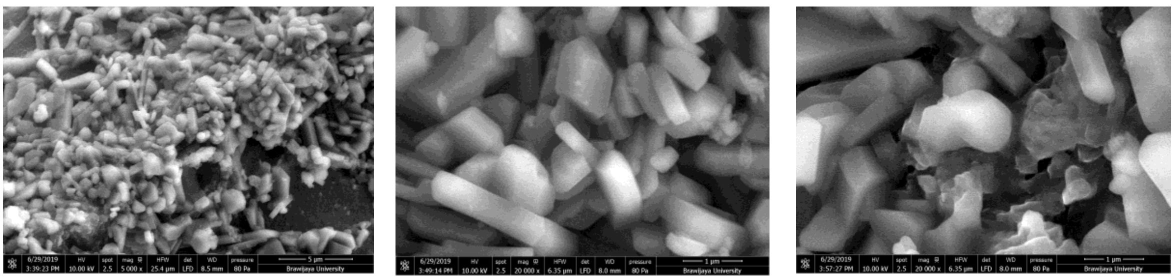
Figure 2. FTIR pattern of extracted silica

Table 2. FTIR graph interpretation of extracted silica based on literature

Wavenumber (cm^{-1})	Function Group	Literature
3400	The stretching vibration of the OH group from Si-OH and H_2O	[21], [30]–[32]
2930	The stretching vibration of Si-O from siloxane (Si-O-Si)	[33]
1883	overtone mode of silica	[30]
1640	The buckling vibration of –OH of the H_2O molecule which is adsorbed on the surface of the solid	
1325	The Stretching vibration of Si-O from siloxane (Si-O-Si)	
1190	The asymmetric stretching vibration of Si-O from Si-O-Si	[21], [31], [32]
963	The asymmetric stretching vibration of Si-O from silanol (Si-OH)	[34]–[36]
785	The symmetric stretching vibration of Si-O from Si-O-Si	[31], [37]
479	buckling vibration of Si-O-Si	[21], [31], [32]

3.3. SEM analysis

The results of the SEM analysis for extracted silica are shown in Figure 3, Figure 3(a) shows a magnification 5 μm, and Figures 3(b)-(c) show a magnification 1 μm. Based on SEM observations, it can be seen that SiO_2 particles of ~1 μm size can coalesce to form large particles (agglomeration). This can be predicted by looking at the results of XRD where cristobalite crystals are formed in silica and the presence of oxalate salts. Brighter color intensity indicates the presence of oxalate salts. Then, it was observed that extracted silica has been a mixture, i.e. amorphous and crystalline phases. It can be seen by the differently brightness, shape, and size of extracted silica.



(a) (b) (c)

Figure 3. Microstructure (SEM results) of extracted silica with magnification (a) 5 μm, (b) 1 μm, and (c) 1 μm

3.4. Chemical and economic analysis

In the initial treatment, leaching method was carried out on the sand of Bajul Mati beach, Malang. This process aims to dissolve heavy metals and other impurities in the sand of Bajul Mati beach [38], [39]. In this process, silica (SiO_2) is insoluble in a 7 M acidic (HCl) solution. The results were obtained after 1 hour, in the form of white sand powder precipitation. The precipitation is separated by decantation techniques accompanied by purification with distilled water until clean. The treatment of HCl leaching is better than the treatment of oxalic acid. On the other hand, the treatment of oxalic acid can remove iron metal through the formation of complex compounds with oxalate ligands [40]. In addition, silica extraction from rice husk by leaching method of 0.5 M citric acid ($C_6H_8O_7$) obtained 91.7% purity [41], and silica extraction from palm ash by leaching citric acid obtained purity of more than 90% [42]. The study differs in terms of the source of silica (biomass) which is relatively easier for silica extraction from sand and has fewer impurities compared to silica sources from beach sand.

Mixing the leached sample with NaOH solids results in a mixture in the initial reaction. The mixture will dry out. In mixing, NaOH compounds reacted perfectly by forming Na^+ ions and OH^- ions. Meanwhile, SiO_2 in Bajul Mati beach sand powder will form an intermediate ion $(SiO_2OH)^-$ which is not stable. It is known in SiO_2, the electronegativity of O (Oxygen) atoms is higher, so it is certain that Silicon (Si) atoms become more electropositive. Then, a hydrogenation process takes place where the hydroxyl ions that bind to hydrogen form a water molecule, and two Na^+ ions which will neutralize the negative charge formed, interact with SiO_3^{2-} ions to form a sodium silicate (Na_2SiO_3) [21], [35], [43]–[45]. In this mixing process, sodium silicate is in the form of dry solids. With the addition of aqua demineralization (H_2O), a solution of sodium silicate (Na_2SiO_3 (*aq*)) is obtained. The sodium silicate solution serves as a precursor in the formation of silica gel (SiO_2) in the next stage.

In the reaction in sodium silicate with acid, the sodium silicate solution (Na_2SiO_3) is stirred by dropping 1 M oxalic acid ($H_2C_2O_4$) to get pH 6-7. In this reaction, the formation of siloxy groups (Si-O-) and silanol (Si-OH) groups occur. The silanol group will form a bond with the siloxy group to form a siloxane group (Si-O-Si). This process occurs very quickly until silica gel is formed with a by-product of sodium oxalate ($Na_2C_2O_4$) [29]. Next is the process of aging and neutralization to remove $Na_2C_2O_4$ with distilled water (H_2O). Based on the results of XRD, it can be shown the presence of oxalate salts is absorbed on the surface of the silica matrix. This is because the natroxalate salt ($Na_2C_2O_4$) is trapped in the silica matrix during heating in the oven. This oxalate salt interacts with silanol (Si-OH) groups on the matrix surface through the interaction of van der walls and hydrogen bonds. Furthermore, the size of the oxalate salt is greater so that it is easily absorbed into the matrix. The heating of the gel is proven to accelerate the reaction but the interaction of the oxalate salt and the silica matrix becomes stronger, so the oxalate salt is difficult to separate from the silica matrix during washing.

Thus, when compared with other methods commonly used to extract sand or glass waste into silica, this method is far more economical, as is the case with the formation of the crystalline phase (cristobalite). When viewed from the morphology of SEM, particle size was observed on a scale of less than 1 μm for samples with pH 6-7, so it can be concluded that this sample is a SiO_2 crystal. For the sol-gel method, previous studies have carried out the extraction of silica from rice husk (biomass), sol-gel using acetic acid (CH_3COOH) [46], use oxalic acid, and citric acid [29]. Kalapathy's study using sol-gel oxalic acid for silica extraction from rice husk ash obtained purity of 91%. Of course, this research with 93.9% purity is the development of beach sand extraction methods that emphasize environmentally friendly aspects.

With this method, an economic analysis through a laboratory approach that comparison of using chloride acid and oxalic acid in the sol-gel method. Its analysis is shown in Table 3. Based on Table 3, using oxalic acid as a reactant in the sol-gel process can reduce material costs. In addition, oxalic acid is an environmentally friendly or non-corrosive material like hydrochloric acid. Furthermore, the making process of oxalic acid solution from solids is longer than that of hydrochloric acid, so this would not be a problem if the process of making oxalic acid used a specially designed reactor. However, the use of oxalic acid still supports environmental sustainability.

Table 3. Economic analysis in this study

Method	Price (IDR)	Remark
Oxalic acid	40,000	100 grams
Hydrochloric acid	130,000	100 mL
aquades	1,000	1 liter
pH indicator	5,000	10-40 cutting sheet
Whatman paper 40	10,000	1 sheet
NaOH (solid)	15,000	15 grams
Aqua demineralization	5,000	1 liter

4. CONCLUSION

Some conclusions can be drawn from the results of this study, i.e.: it has been able to synthesize silica powder from natural sources (Bajul Mati beach sand) which achieved 93.9% purity with cristobalite phase through the combined method of leaching and sol-gel. The leaching method is a separation of silica from heavy metals and other impurities. The sol-gel method includes the stage of precursor formation of sodium silicate ($Na_2O.xSiO_2$), and the stage of formation of $Si(OH)_4$. Silica extraction requires very economical costs, so it can be mass-produced and increase the selling value of beach sand.

ACKNOWLEDGEMENTS

The author thanks LPPM Universitas Negeri Malang for financial support in this study.

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