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Synthesis and Characterization of Silica from Lapindo Mud Waste with Acid and Base Solvents

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Abstract
Lapindo mud is geological waste produced from mud eruptions in Sidoarjo, East Java, Indonesia, which continues to cause concern in the environmental sector. However, Lapindo mud still has considerable potential due to its high silica content. This study evaluates the effectiveness of acid dissolution and alkali precipitation methods for extracting silica from Lapindo mud and determines the properties of the silica produced. Acid leaching was conducted using HCl (4, 8, and 10 M), while alkaline treatment employed NaOH (2, 6, 8, 10, and 12 M) and KOH (8, 10, and 12 M). The results showed that acid dissolution was effective in removing metal impurities without dissolving the silica framework, resulting in only a limited increase in SiO_2 content from 54% to 57%. On the other hand, silica purity increased significantly with precipitation using a base solvent, with an optimal SiO_2 content of 97.5% obtained with 10M NaOH. Nitrogen adsorption-desorption analysis showed that the precipitated silica had mesoporous properties with type IV isotherm and an average pore size of 20.60 nm. XRD analysis confirmed that there was a conversion process from crystalline silica to amorphous silica. Thus, the most effective method for producing high-quality silica from Lapindo mud waste is alkaline precipitation with 10M NaOH
Keywords: *lapindo mud, silica, precipitation, leaching*

Abstrak
Lumpur Lapindo merupakan limbah geologi yang dihasilkan dari peristiwa semburan lumpur panas di Kabupaten Sidoarjo, Jawa Timur, Indonesia dan hingga kini masih menjadi tantangan pada sektor lingkungan. Di sisi lain, lumpur Lapindo memiliki potensi karena mengandung silika (SiO_2) dalam jumlah yang relatif tinggi. Tujuan dari penelitian ini adalah untuk mengevaluasi efektivitas metode leaching asam dan presipitasi basa dalam mengekstraksi silika dari lumpur Lapindo serta menganalisis sifat silika yang dihasilkan. Ekstraksi menggunakan pelarut asam dilakukan menggunakan HCl (4, 8, dan 10 M), sedangkan pada pelarut basa dilakukan menggunakan NaOH (2, 6, 8, 10 dan 12 M) dan KOH (8, 10, dan 12 M). Penelitian menunjukkan bahwa pelarut asam efektif dalam menghilangkan pengotor logam tanpa melarutkan struktur silika, sehingga hanya meningkatkan kandungan SiO_2 dari 54% menjadi 57%. Sebaliknya, metode presipitasi menggunakan pelarut basa mampu meningkatkan kemurnian silika secara signifikan, dengan kandungan SiO_2 tertinggi 97,5% yang diperoleh menggunakan pelarut NaOH 10 M. Analisis silika menggunakan XRD menunjukkan adanya perubahan silika kristalin menjadi silika amorf. Analisa adsorpsi-desorpsi N_2 menunjukkan bahwa silika hasil presipitasi bersifat mesopori dengan isoterm tipe IV dan rata-rata ukuran pori sebesar 20,60 nm. Metode presipitasi basa menggunakan NaOH pada konsentrasi 10M merupakan metode paling efektif untuk menghasilkan silika berkualitas dari limbah lumpur Lapindo.
Kata Kunci: *lumpur lapindo, silika, presipitasi, leaching*

1. Introduction

The hot mudflow that occurred in Sidoarjo, East Java, Indonesia caused three subdistricts to be submerged, with a total area of approximately 640 hectares. This phenomenon was caused by movement of magma and hydrothermal fluids from within the earth's crust flowing into the subsurface sediments, causing high pressure that was eventually released to the surface due to seismic activity [1]. To date, it is still uncertain when the Lapindo mudflow will stop, causing ongoing environmental impacts and unrest among the surrounding community. However, Lapindo mud has the potential to be utilized because it contains various economically valuable inorganic minerals.

Previous study reported the major composition of Lapindo mud is SiO_2 about 50.00%, in addition to other oxides like Fe_2O_3 (27.7%), CaO (8.59%), and Al_2O_3 (6%) [2]. The relatively high silica content makes

Lapindo mud one of the potential sources of silica in Indonesia. Research conducted by [3] confirmed that silica from Lapindo mud has been used in the manufacture of silica gel for adsorption applications. However, research [4] explains that the synthesis of silica-based material, such as zeolite Y, still produces a fairly high level of impurities due to an optimization of the purification process. Therefore, a more effective extraction method is needed to reduce the impurity content and increase the purity of silica in Lapindo mud.

Methods commonly used in purification of silica from mineral-based materials into waste are leaching and precipitation. The acid leaching process aims to dissolve or reduce the composition of the metal impurities such as Al, Fe, Ca, and Mg, which will increase the silica content without dissolving the SiO_2 structure. In this process, hydrochloric acid is often because it is strong, selective towards metal oxides, and does not form complex precipitates with silica. Research by [5] obtained an increase in SiO_2 content from 80% to 90.2% after acid leaching treatment on diatomite, indicating that this process is effective in removing metal impurities, especially Fe and Ca, which are generally in the form of oxides or carbonates that are easily soluble in acid. Research by [6] has shown similar results on coal fly ash material from Lampung, from a concentration of 50.2% to 97.61% with amorphous phase products. Meanwhile, research conducted by [7] also confirmed that the acid leaching process using higher concentrations of HCl can accelerate the dissolution of metal oxides such as Fe and Al. This process increases the SiO_2 content from 68.31% to 72.29% without removing the crystalline structure.

Conversely, precipitation methods using base solvents, such as NaOH and KOH, are carried out through a mechanism of silica extraction via dissolution into soluble silicates, which are then re-precipitated into amorphous silica through the addition of acid or pH adjustment. This basic method has the potential to produce high-purity silica, although the yield obtained is greatly influenced by the solvent concentration and process conditions. Research by [8] confirmed that agricultural waste in the form of amorphous rice husks can be extracted using a 3N NaOH solution through a precipitation method with a silica purity of up to 99.7%. Meanwhile, research by [9] shows that the process of dissolving silica from quartz sand under atmospheric conditions is quite difficult due to the high stability of the crystalline SiO_2 phase. However, the reaction time can be extended to obtain optimal conditions. From this study, amorphous silica with a purity level of 96% was obtained using a 7.5M NaOH solution, indicating that the use of a strong base with the right operating conditions can convert crystalline silica into amorphous silica with high purity.

Based on the description, the methods of base precipitation and acid leaching have different mechanism and benefits for the purification of silica. While the base precipitation method can directly extract silica with a high degree of purity, it is heavily influenced by the material's initial phase properties and operating conditions. In contrast, acid leaching is effective at removing metal impurities without dissolving the silica structure. The usefulness of these two approaches on materials with high mineral complexity, especially Lapindo mud, hasn't been thoroughly compared in research up to this point. In order to obtain silica of the highest quality that may be utilized as a precursor in the synthesis of advanced materials, this study compared the effects of acid and base treatments on the properties of the resulting silica.

2. Materials and Methods

Materials

The materials used in this study were Lapindo mud taken within a 600 m radius of the eruption center, hydrochloric acid (HCl 37% Merck), sodium hydroxide (NaOH 99% Merck), potassium hydroxide (KOH 99% Merck), and distilled water.

Pre-treatment of Lapindo Mud

The Lapindo mud to be used was first washed with distilled water to remove impurities for 24 hours, followed by a filtration process. The resulting sediment was then dried at 100°C for 24 hours to achieve maximum drying due to the relatively high moisture content of the raw material and its considerable mass.

Silica Leaching Using Hydrochloric Acid

100 mL of 4M, 8M, or 10M HCl solution is combined with 10 grams of pre-treated Lapindo mud that has been weighed. A magnetic stirrer is used to agitate this mixture for 24 hours at 300 rpm and 90°C. After this process, the mixture is separated through a filtration process using a vacuum pump, so that a precipitate is formed from the filtrate. The precipitate obtained is neutralized with distilled water to a pH 7 and dried at temperature of 100°C for 12 hours.

Silica Precipitation Using Sodium Hydroxide and Potassium Hydroxide

100 mL of NaOH solutions with concentrations of 2, 6, 8, 10, and 12M and KOH solutions with concentrations of 8, 10, and 12 M were combined with 10 grams of pre-treated Lapindo mud, which were stirred using a magnetic stirrer for 24 hours at 300 rpm and 90℃. After the reaction process, a mixture of precipitate and solution was obtained for filtration. The filtrate obtained was then titrated using HCl solution until reached at pH 4 and a white precipitate appeared. The precipitate was aged for 24 hours and filtered tp separate the solution. The precipitate obtained was washed repeatedly with distilled water until reached neutral pH (pH 7) and dried at 100℃ for 12 hours to obtain dry silica.

Characterization

Silica characterization was performed using wide-angle X-ray Diffraction (XRD) to evaluate the success of the extraction process through analysis of the resulting crystal structure and comparison with silica standards. Wide-angle XRD measurements were performed using a Binary XPert diffractometer (Philips MPD Cu Kα) at a diffraction angle range of 2θ = 5–50°, with a step size of 0.01670°, a current of 30 mA, and an operating voltage of 40 kV. X-ray Fluorescence (XRF) analysis was used to determine the elemental composition and oxide compounds in Lapindo mud, both before and after the leaching and precipitation processes. In addition, nitrogen (N_2) adsorption–desorption characterization was performed to determine the specific surface area and pore volume of silica using a Quantachrome Touchwin v1.11 instrument. The yield of the silica obtained was calculated using the following equation:

$$yield\ (\%) = \frac{final\ mass\ SiO_2\ (gr)}{initial\ mass\ SiO_2\ (gr)} \times 100\%$$

3. Results and Discussion

XRF analysis

The XRF analysis results show transformation in the composition of the mud after treatment using HCl, NaOH, and KOH solvents at various concentrations, as shown in **Table 1**. Based on **Table 1**, Lapindo mud as raw material, contains 54% SiO_2 with impurity oxides such as 20.5% Fe_2O_3, 9.5% Al_2O_3, 4.1% TiO_2, and 3.26% CaO. When treatment was carried out using acid leaching with HCl concentrations of 4,8, and 10M, the Fe_2O_3 content decreased from 20.5% to 4.24%, as did the concentration of TiO_2 and K_2O. However, acid leaching did not increase the concentration of SiO_2 as its concentration remained at 57%. It can be concluded that acid leaching is capable of dissolving metal impurities without altering or dissolving SiO_2 structure. According to [5], silica stays relatively inert under strong acidic leaching conditions, but metal oxides like Fe, Al, Ca, and Mg, which are frequently present as oxides or carbonates easily soluble in acidic media, are preferentially dissolved by HCl. Meanwhile, when the HCl concentration was raised to 10 M, the SiO_2 content decreased to 47% while the Fe_2O_3 content rose to 26.9%.

This implies that a relative enrichment of Fe_2O_3 results from the selective dissolution of alkali and alkaline earth oxides, such as CaO and K_2O, under extremely acidic conditions. Additionally, the majority of the iron in Lapindo mud is found in stable, well-crystallized iron oxide phases, which are less vulnerable to acid leaching because they are more resistant to strong acid dissolution than amorphous or poorly ordered iron species [10]. Therefore, it can be concluded that low-concentration HCl leaching is more effective in reducing Fe and other metallic impurities while maintaining silica content, whereas the use of highly concentrated HCl is less favorable for enhancing SiO_2 purity.

In contrast, alkaline precipitation treatment using NaOH and KOH resulted in a significant increase in SiO2 content. At a NaOH concentration of 2M, the SiO_2 content increased to 93.5%, reaching a maximum value of 97.5% at 10M NaOH. This improvement was accompanied by a sharp reduction in impurity oxides, particularly Fe_2O_3, which decreased to as low as 0.031%, while CaO and Al_2O_3 were nearly eliminated at higher alkaline concentrations. These results indicate that NaOH effectively dissolves SiO_2 to form soluble sodium silicate, whereas most metallic oxides remain in the solid phase and can be separated by filtration [11]. Nevertheless, at NaOH concentrations of 6 M and 8 M, although the SiO_2 content remained high (96.6% and 93.7%, respectively), increasing alkaline concentration did not consistently lead to higher silica purity. This phenomenon may be attributed to the partial re-precipitation of impurity oxides such as V_2O_5, CuO, and ZnO due to the accumulation of metal ions in highly alkaline solutions [12].

Meanwhile, treatment with 8M KOH produced a SiO_2 content of 93.7%, which showed high purification efficiency despite being somewhat lower than that obtained with 10 M NaOH. The residual potassium incorporation into the silica structure, which frequently happens during KOH-based precipitation processes and may affect the surface characteristics of the resulting silica, is indicated by the presence of

K_2O (0.57%) [13]. These findings suggest that the best techniques for increasing the purity of silica from Lapindo mud are alkaline extraction techniques, especially when high-concentration NaOH is used. On the other hand, HCl leaching is not very effective because it mostly dissolves alkali and alkaline earth oxides, whereas metal oxides like Fe_2O_3, which results in impurity fractions that persist or even rise in extremely acidic environments.

Table 1. Lapindo Mud Before and After Treatment

Lapindo Mud		HCl (M)			NaOH (M)				KOH (M)
		4	8	10	2	6	8	10	8
Comp.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.
Al_2O_3	9.5	13	9.2	8	-	-	3.4	-	3.4
SiO_2	54	57	57	47	93.5	96.6	93.7	97.5	93.7
P_2O_5	0.7	10	0.7	0.9	4,6	2.5	1.2	1.7	1.2
SO_3	3.2	3.2	3.5	2.2	-	-	-	-	-
K_2O	4.26	2.9	4.31	2.88	-	-	0.57	-	0.57
CaO	3.26	6.8	3.31	8.53	1.7	0.82	0.48	0.58	0.48
T_iO_2	4.1	1.7	4.11	2.18	-	-	0.093	-	0.093
V_2O_5	0.14	0.2	0.13	0.1	-	-	0.2	0.1	0.2
Fe_2O_3	20.5	4.24	17.2	26.9	0.13	0.02	0.27	0.031	0.27
CuO	0.21	0.22	0.19	0.14	0.082	0.046	0.065	0.054	0.065
ZnO	0.04	0.04	0.03	0.05	0.01	0.004	-	0.003	-

Mass and yield analysis of silica

The yield calculation results shown in **Table 2** indicate the mass variation and extraction efficiency of Lapindo mud after treatment with HCl, NaOH and KOH solution with different concentration. According to the results shown in **Table 2**, it can be seen that the silica yield varies greatly with various solvent and its concentration. In the HCl leaching process, the recovery of is still high (from 63.33% to 73.88%). The maximum yield of 73.88% was achieved using an HCl concentration of 8M, whereas yields of 63.33 and 69.62% were achieved at concentrations of 4M and 10 M, respectively. The acid leach yield is relatively high, which means a large portion of non-silica components or mineral impurities remained undissolved as did solid residual product. This indicates that acid leaching preferentially dissolves certain elements, while the rest of the minerals, such as relatively resistant metal oxides, concentrated in the residue [7]. Thus, even though a large amount of residual mass remained, HCl leaching did not effectively strip down the impurities.

NaOH treatment, however, was considerably less effective than HCl and the values varied from 9.86% to 48.38%. The minimum yield was 9.86% at 2 M NaOH and subsequently increased with increasing concentration of NaOH to the maximum value 48.38% at 10 M NaOH, decreasing slightly to 37.47% upon further increase in molarity of NaOH (12 M). The low yield obtained when submitting MWI to alkaline treatment, suggests that during the process most of the impurities were dissolved and the solid material that remained was mostly silica re-precipitated in a fresh stage of precipitation. Upon KOH treatment, the yield was quite stable in the 22–23% range between 8 and 12 M KOH, these figures are lower than those obtained from NaOH at its optimal concentration, indicating that silica precipitation efficiency is less effective using KOH as the alkaline solution. This behaviour could be due to the differences in the ionic radius of K^+ with respect to Na^+ affecting the nucleation processes for silicate species in aqueous solutions and kinetics of silica precipitation. KOH-based extraction was observed to render silica with relatively lower ratios but unique surface characteristics associated with the possible inclusion of K^+ ions in the silica network [13].

Table 2. Silica Yield Data

HCl			NaOH					KOH		
4M	8M	10M	2M	6M	8M	10M	12M	8M	10M	12M
Mass (g)										
8.27	7.81	5.26	0.57	1.74	1.73	2.68	2.13	1.28	1.3	1.29
Yield (%)										
63.33	73.88	69.62	9.86	31.12	30.01	48.38	37.47	22.21	22.55	22.38

XRD analysis

The X-ray diffraction (XRD) patterns of Lapindo mud and the treated silica samples are presented in **Figure 1**. As shown in **Figure 1**, untreated Lapindo mud consists of a mixture of several crystalline phases, including gibbsite (2θ = 18.28°, 20.31°, 20.54°, 36.59°, and 37.64°; JCPDS 00-021-1272), boehmite (2θ = 38.38°, 49.21°, and 49.93°; JCPDS 00-021-1307), hematite (2θ = 24.14°, 33.15°, 35.61°, 40.86°, and 49.48°; JCPDS 00-033-0664), and quartz (2θ = 20.86°, 26.64°, 36.54°, and 39.47°; JCPDS 00-046-1045).

When treated with alkaline extracts using NaOH and KOH for the removal of iron-containing phases, it can be observed after XRD analysis that Lapindo mud experienced phase transition to form the sodium aluminosilicate ($NaAlSiO_4$) at around 37° and sodium silicate (Na_2SiO_3) peaks at 2θ values of 17°, 19°, and 23° which was in agreement with previous researches [14], [15]. The formation of an amorphous silica is rightly evidenced by no evident signals on sharp crystalline peaks, and a very intense pattern of diffraction halo, demonstrating its high reactivity and precursor solubilization that are interesting for the future synthesis work [16]. However, if strong alkaline medium are employed, such as NaOH 12 M, we can see the appearance of a diffraction peak at 2θ ≈ 31.6°. The peak of this impurity phase is attributed to the accumulation of oxides under highly alkaline conditions, where impurity oxides (pure forms) CaO and ZnO reappear during the reaction [2]. Thus, it is not advisable to perform silica syntheses at very high alkaline concentrations since they favour non-silica phase formation and decrease the structural purity of the final silica obtained.

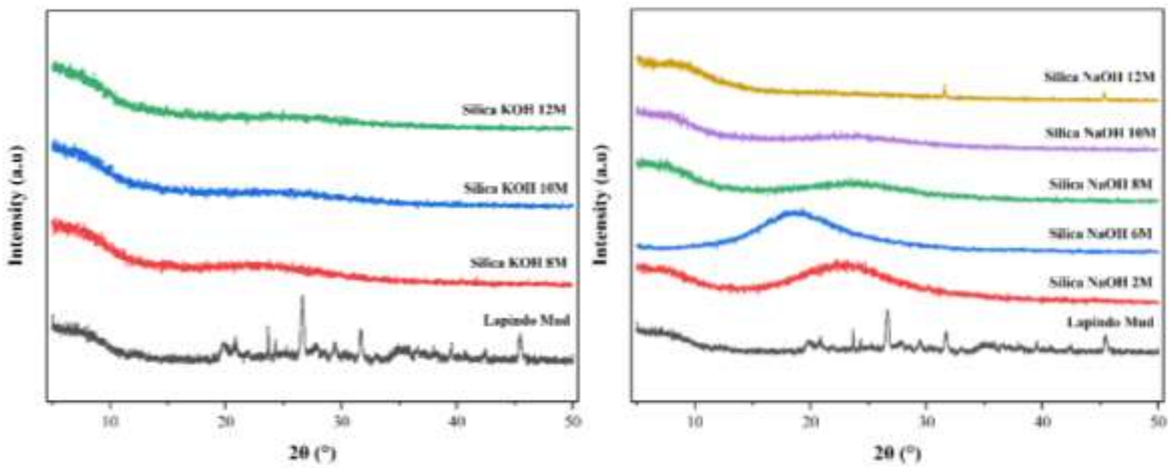

Fig. 1 XRD of Lapindo Mud and Treated Silica

Nitrogen adsorption-desorption analysis

N_2 adsorption–desorption was used to measure the specific surface area, pore size distribution, total pore volume and average pore diameter of Lapindo mud and unmodified and modified silica samples (as presented in **Fig. 2**). Nitrogen adsorption–desorption isotherms have shown that both Lapindo mud and silica extracted with 10 M NaOH showed the type IV, which indicates mesoporous material due to the capillary condensation at high and medium relative pressures [17]. This conduct indicates that these two materials feature active surface sites available to N_2 molecules adsorption. For untreated Lapindo mud, at intermediate to high relative pressures ($P/P_0 > 0.8$), the increase in adsorption capacity tends to be relatively constant and it reaches an adsorption volume of about 22 cm^3/g, indicating that its isotherm pattern shows the existence of mesopores with a less open structure and a non-homogeneous distribution, probably due to some mineral oxides as well as inorganic impurities. The precipitation of silica with 10 M NaOH also has less adsorption capacity than raw Lapindo mud. The lower adsorption volume indicates partial pore structure collapse has occurred from the use of too high alkaline concentrations in the extraction step. High

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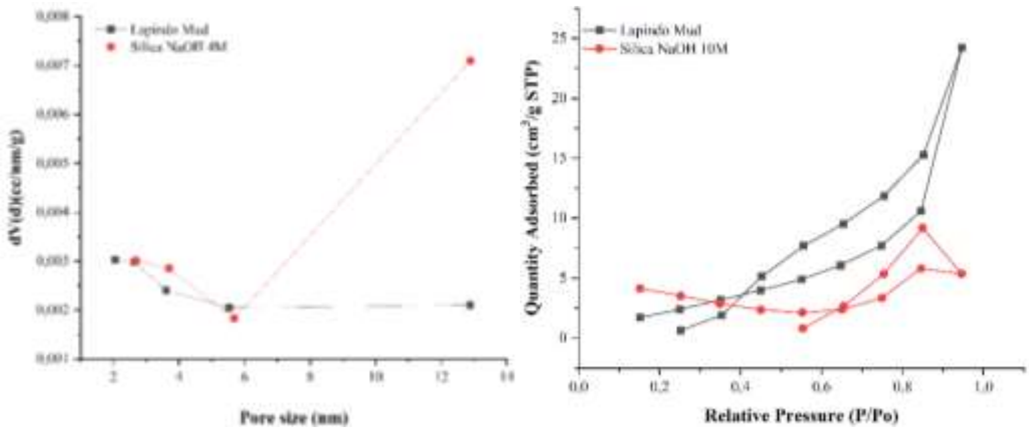
alkaline reaction conditions can cause over etching of the silica (over-etching) and may lead to softened or slumped pore walls, and consequently reduced effective surface area [18].



(a) (b)

Fig. 2 Pore size distribution (a) and N_2 adsorption-desorption isotherms (b)

Table 3, Lapindo mud has a specific surface area of 11.33 m^2/g, total pore volume of 0.03753 cm^3/g, and average pore diameter of 6.63 nm. The surface area of 10 M NaOH extracted silica is reduced to 8.08 m^2/g whereas the pore volume increases significantly to 0.83171 cm^3/g with an average pore diameter of ~20.60 nm prevalent in this case. his trend suggests that pore widening and mesopore formation begin during the alkaline treatment, along with damage of the pore walls by high alkaline level, leading to decrease of material filling in [19].

Table 3. Surface Area and Pore Structure Analysis of Samples

Sample	Surface Area (m^2/g)	Pore Volume (cm^3/g)	Average Pore Size (nm)
Lapindo Mud	11.328	0.03753	6.6263
Silica NaOH 10M	8.07523	0.83171	20.5991

4. Conclusion

The precipitate of silica from the Lapindo mud waste is better with alkaline, rather than acid solvent. Acid leaching achieves an increase in SiO_2 content of only from 54% to 57%, due to the lower dissolution ratio of crystalline silica under acid environment. On the other hand, precipitation with NaOH and KOH in an alkaline media significantly improves the silica purity level where SiO_2 can be up to 97.5% with 10 M NaOH based on XRF results. XRD results reveal that the crystalline silica is changed to amorphous silica with mesoporous property as proved by BET analysis. It is therefore concluded that the alkaline precipitation using an optimum base concentration is a highly effective alternative for fabricating high-quality silica material from the Lapindo mud.

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