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Eco Friendly Citric Acid-Assisted Sol-Gel Synthesis of High-Purity Nano Silica from Dieng Geothermal Slag: Characterization and Optimization Method

David C Birawidha[1, 2*], Azwar Manaf[2*], Widi Astuti[1], Amru Daulay[1], Tri Haryono[1], Yuliana Sari[3], Suprihatin[3]

[1]*Research Center for Mining Technology, National Research and Innovation Agency (BRIN), Lampung, Indonesia*
[2]*Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Indonesia, Depok, Indonesia*
[3]*Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Lampung, Bandar Lampung, Indonesia*

Corresponding Author Emails: azwar@sci.ui.ac.id, davi004@brin.go.id

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Abstract

Geothermal slag is a by-product of the geothermal power generation process, but its added value is minimal. With a silica content of up to 70%, geothermal slag has potential as a secondary silicon source for battery silicon anode precursors. Usually, the synthesis of nano-silica was carried out through the sol-gel method, in which HCl is usually used as a modifier to regulate the physical and chemical properties of the material. But in this study chose citric acid for modifier agent because it is more environmentally friendly. The challenge of using citric acid is the formation of carbon-based salts that can cause silica blackening if not washed well. Therefore, optimization was done by adjusting the pH to produce high-purity nano-silica. The sol-gel process was carried out by adding 10% NaOH and 5N Citric Acid, with varying pH base conditions from 8 to 11. XRF analysis results showed the highest purity at pH 8. Impurities were still visible based on XRD data, and the formation of nanoparticles was confirmed through morphological analysis using FESEM and TEM where the average particle size formed is between 55 nm.

Introduction

Geothermal silica is a byproduct of geothermal power plant operations, typically found in the form of silica sludge or crust. This waste material, often called *geothermal slag*, contains a high concentration of amorphous silica—ranging from 90% to 98%—making it a promising secondary source of silica [1]. Slag in the geothermal context refers to solid waste formed due to the heating processes involving hot water or geothermal fluids within the Earth, including in the reservoir, pipelines, equipment, and other geothermal environments. Its formation involves several mechanisms, such as the coagulation of minerals (silica, potash, and soda) due to changes in temperature and pressure, chemical reactions between hot fluids and surrounding materials that produce new precipitated compounds, the formation of scale (intricate layers such as sulfides) on the surfaces of pipes and equipment, and mineral precipitation when geothermal fluids cool down. As a result, slag can cause operational issues such as clogging and corrosion, which makes effective management necessary. Despite its abundance, geothermal silica waste is still underutilized and is commonly discarded without proper processing, potentially leading to environmental issues over time [2], [3].

The amorphous nature of geothermal silica gives it advantageous properties such as high reactivity and ease of modification, which makes it suitable for various high-value applications. One of the most promising uses is as a precursor for the production of nano silica, which is widely recognized for its large surface area, high thermal stability, and chemical inertness [2], [4]–[7]. These properties make nano silica particularly attractive for applications in nanotechnology, including its transformation into nano-silicon for use as an anode material in lithium-ion batteries.

Silicon-based anodes have been extensively studied due to their high theoretical capacity. However, the performance of silicon anodes is highly dependent on particle size. Smaller, nanoscale particles exhibit significantly better electrochemical stability and resistance to mechanical stress caused by volume expansion during charge-discharge cycles. In contrast, larger particles, while achieving high initial capacity, tend to degrade more rapidly [8]–[12]. Therefore, producing high-purity nano silica from geothermal slag presents a sustainable and strategic route to support next-generation battery technologies. To improve the silicon yield obtained from the synthesis process, high purity silica is essential.

The sol-gel method is a practical and widely used approach to synthesizing nano-silica. It involves the dissolution of silica in a strong base such as sodium hydroxide (NaOH), followed by a condensation process using acid to form a silica gel [13]–[16]. However, a significant limitation of this process lies in the environmental impact of using strong acids such as hydrochloric acid (HCl)[17]–[19], which can generate hazardous waste and pose handling risks[20]. Strong acids (with a pH of 1–3) pose serious health risks due to their highly corrosive nature. Direct contact with skin can cause chemical burns, ranging from irritation to third-degree burns that may leave permanent scars. If exposed to the eyes, strong acids can lead to severe irritation, corneal damage, and even blindness. Inhalation of acid vapors or mist can irritate the nose, throat, and lungs and, in severe cases, may cause pulmonary edema (fluid buildup in the lungs). Ingesting strong acids can result in injuries to the mouth, esophagus, and stomach[21]. Also, prolonged acid fumes exposure can erode tooth enamel, leading to dental erosion. Long-term effects include permanent lung damage, dental deterioration, and other chronic health issues.

To address this challenge, our study introduces a novel approach using citric acid, a biodegradable weak organic acid and considerably more environmentally friendly than conventional acids[22]–[24]. The use of citric acid not only reduces the environmental footprint of the sol-gel process but also aligns with green chemistry principles. Weak acids offer various environmental advantages due to their relatively safe, less corrosive, and biodegradable nature. Compared to strong acids, weak acids release fewer hydrogen ions, thereby reducing the risk of water, soil, and air pollution. In addition, natural microorganisms can more readily break down weak acids, minimizing the potential accumulation of harmful chemical substances in the environment. However, the main challenge of using citric acid is the formation of residual citrate salts during the gelation process. These byproducts are notoriously difficult to remove and can lead to burned or degraded silica morphology, thus affecting the final quality of the nano-silica product [14].

The importance of this research lies in the successful application of citric acid in the sol-gel process for synthesizing nano silica (purity 94.8%) from geothermal slag while overcoming the typical issues of salt residue and morphological degradation. In this study, optimized process conditions, especially pH variation and microwave-assisted heating, enabled the effective removal of residual salts and prevented the formation of "burned" morphology. Microwave heating, known for its rapid and uniform energy transfer, facilitated homogeneous particle formation and improved the morphological integrity and thermal stability of the resulting nano silica; so, based on research from Freitas, microwaves were used to maintain the morphology of the silica produced [25]. Therefore, this research provides a sustainable and innovative method for valorizing geothermal waste into high-performance nano-silica, with the potential to be further developed as a precursor for battery-grade nano-silicon. The integration of eco-friendly chemistry, waste valorization, and simple material synthesis highlights the importance and relevance of this study in the context of circular economy and green energy materials.

Experimental Method

Material

This study used raw material such as geothermal silica slag from PT Geo Dipa Dieng, Central Java, Indonesia where samples were taken from geothermal slag collection and storage areas. NaOH with CAS number 1310-73-2-106498 was purchased from Merck and Citric Acid with CAS number 77-92-9-818707 was purchased from Merck. Methanol was purchased from Merck with CAS number 67-56-1-107018. For the synthesis process until washing using distilled water.

Characterisation test

X-ray diffraction (XRD: Panalytical Xpert 3 Powder XRD) with a Cu-Kα as a source of X-ray operating at 40 kV and 30 mA was used to determine the crystallization and phase of the starting material and nano-silica. At the same time, to determine the chemical composition of the material used, X-ray fluorescence (XRF Epsilon 4 XRF Spectrometer from Malvern Panalytical) operating at 50 kV and 3 mA. In addition, to determine the morphology of nano-silica are used FESEM (Quatro Thermo Scientific). To determine the particle size of nano-silica using TEM Talos F200X G2 then from the data, we used Image J software to calculate the particle distribution chart.

Synthesis process of geothermal silica slag

Based on the experimental illustration in Figure 1, 5.62 g of slag was mixed with 75 ml of 10% NaOH to form Sodium silicate (Na_2SiO_3). The mixture was stirred using a magnetic stirrer at

100°C for 3 hours. After that, sodium silicate was filtered to separate the solids from the liquid using filter paper (whattman no. 42). After that, the filtered liquid was titrated with 5N citric acid for pH variations from 8 to 11. After the gel precipitates, the mixture is centrifuged to separate the solids and liquid using centrifuge machine at 4000 rpm. Then washed to neutral pH using a mixture of 10% methanol and distilled water repeteadly. After the gel was pH neutral, it was dried in the oven for 3 hours at 100°C, and for final procedure the sample put into the microwave with multistage heating at 100 W-300W-700W and 900W for 5 minutes for each heating condition.

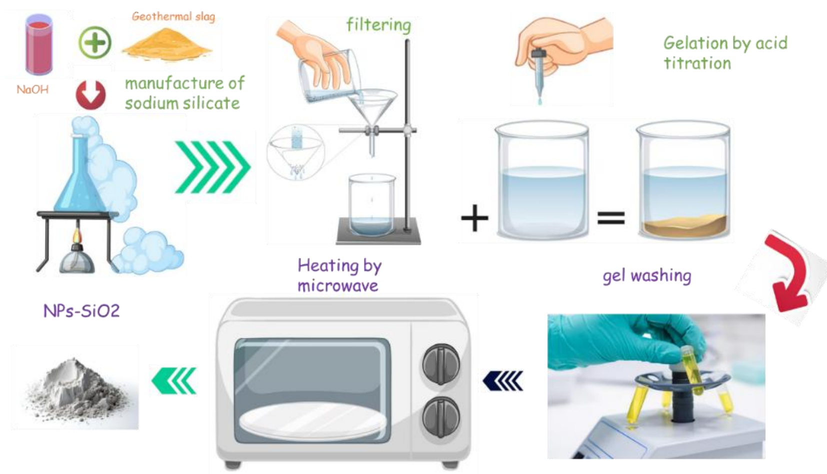

Figure 1. Illustration of the Preparation of Nano Silica from Geothermal Slag by Sol-Gel Process

Result and Discussion

Based on the results of composition testing using XRF seen in table 1, the highest SiO_2 content is obtained when conditioning condensation solution at pH 8 with SiO_2 content reaching 94.846%. It can be seen that the more alkaline environmental conditions are, the more impurities are trapped, thereby reducing the purity of SiO_2 obtained. The recovery or percent recovery is being calculated using equation 1 and be represented in table 2. Based on Table 2, the most optimal recovery when in pH 8 conditions is 79.41%.

$$\%\,Recovery = \left[\frac{M1 \times \%m1}{M2 \times \%m2}\right] \times 100\% \quad (1)$$

Where :
% Recovery = the percentage of recovery of the target element
M_1 = initial mass of the sample containing the target element
% m_1 = initial percentage content of the target element
M_2 = final mass of the sample containing the target element
% m_2 = final percentage content of the target element

Table 1. Chemical composition of geothermal slag before and after synthesis.

Oxide	Raw % weight	Citric pH 8 % weight	Citric pH 9 % weight	Citric pH 10 % weight	Citric pH 11 % weight
Na_2O		0	0	0	0
MgO		0.646	0.328	0.27	0.279
Al_2O_3		1.233	1.415	1.358	1.309
SiO_2	73.24	94.846	94.281	94.5	93.766
P_2O_5	0.683	0.639	0.573	0.562	0.598
K_2O	3.031		0.32	0.306	0.319
CaO	1.336	0.249	0.268	0.26	0.267
Fe_2O_3	1.589	0.698	0.836	0.749	0.959
ZnO	0.126	1.507	1.802	1.78	1.864
PbO	0.137	0.117	0.11	0.17	0.556
Cl	19.078				

In some literature, in utilizing citric acid in the silica sol-gel synthesis process, the optimal conditions for solution pH range from 10 to 11. The nano-silica obtained has high purity [25], [26]. Therefore, the study adopted alkaline conditions as a variable for gel formation.

Table 2. SiO_2 compound recovery at various pH variations.

Sample	After synthesis (g)	% wt SiO_2 taken	Innitial weight (g)	%wt SiO_2 raw	% recovery
pH 8	3.45	94.846	5.62	73.324	79.41
pH 9	1.62	94.281	5.62	73.324	37.06
pH 10	1.95	94.5	5.62	73.324	44.72
pH 11	1.85	93.766	5.62	73.324	42.10

To determine the crystal formation and phase of the NPs-SiO_2 sample, based on the XRD test results graph in Figure 2, it can be seen that the geothermal slag before and after the synthesis process is amorphous silica. Amorphous silica does not exhibit distinct diffraction patterns in the 2-theta diagram. Typically, the 2-theta diagram of amorphous silica shows a broad peak around 2θ = 20–22 degrees, indicating its amorphous nature[27]–[30]. In the initial material, geothermal silica slag is mixed with salt impurities that are influenced by the composition of the brine flow during the geothermal power plant operation process, where salt content such as NaCl is the dominant composition in the brine flow [31]–[35]. Where based on figure 2, NaCl impurities in geothermal slag samples were detected at 2theta 27.3; 31.7; 45.4; 56.4; 66.2; 75.2 and 83.9 respectively, and based on COD 96-900-6375 is the impurity category of NaCl. In addition, after the sol-gal process is carried out to obtain nano-silica, the impurities that are still trapped are metal compounds. At 2theta 44.6, based on COD data 96-901-3473 is an indication of metal elements (Fe) trapped in silica particles and align with Table 1 shows that metal composition is relatively constant in all pH variations.

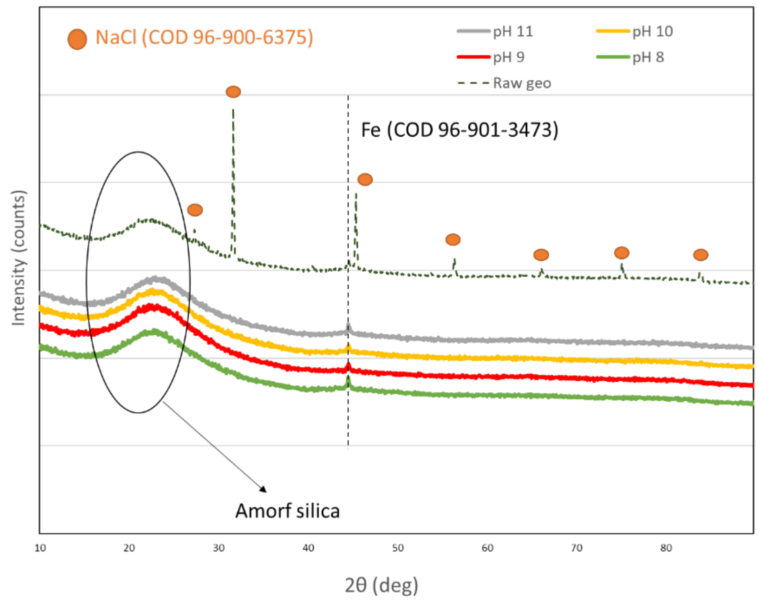

Figure 2. XRD Diffraction Results of Geothermal Silica Slag Before and After the Synthesis Process.

For the morphological shape of nano-silica, a sample of pH 8 was taken to be tested using FESEM. As seen in Figure 3a, for pH 8, nano silica grains have a spherical morphology where impurities are seen to collect on the surface with a brighter appearance than the surrounding appearance. This impurity is likely based on Table 1 as a metal compound like Fe, Al_2O_3 or ZnO, which is usually spherical [36]–[38]. TEM testing was used for the pH 8 sample with the highest purity presented in Figure 3b to prove that the silica obtained is nano-dimensional. It can be seen that the silica particles have a size between 20 - 100 nm with a generally literally spherical shape. The spherical shape of silica particles is generally preferred in various industrial applications due to several key advantages. Spherical silica particles exhibit better packing density and smoother flow characteristics, facilitating production processes such as mixing and packaging. Additionally, the uniform spherical shape reduces interparticle friction, making the particles less prone to mechanical degradation like breakage or wear during further processing. This is particularly beneficial in composite materials, cosmetics, and electronics applications, where consistent particle shape and size are crucial for optimal performance[39], [40]. Figure 3d presents a particle distribution graph that gives information from data analysis using Image J; the mean for the size distribution of silica particles is around 55 nm, whereas COD values in the graph are almost near 1, which indicates the graph is much more reliable.

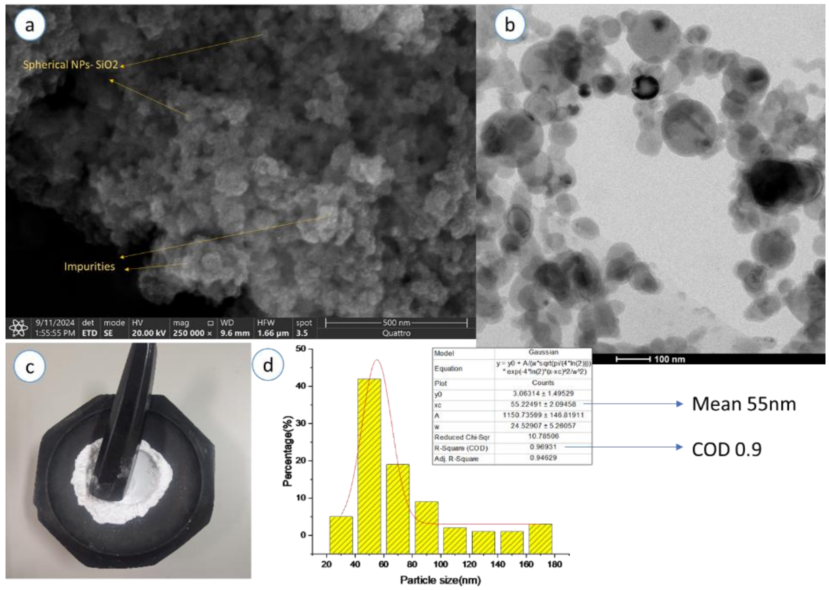

Figure 3. Morphology of pH 8 Sample Using FESEM at 250,000 Times Magnification. a). SEM image, b). TEM image, c). Photo sampel pH 8, d). Distribution Particle graph of Figure 3b.

The synthesis process using the sol-gel method to obtain nano-sized silica was successfully carried out by utilizing citric acid, which is more environmentally friendly and easy to handling. The problem of salt remaining during the combustion process causing scorching can be avoided with the appearance of the sample with pH 8, which is pure white, as shown in Figure 3c. The combination of washing using methanol to break down organic compounds and remaining salts optimized the washing stage of the silica gel. According to Table 1, the remaining impurities was metal compound which need further treatment if the sample want to get a higher purity. The microwave combustion process can maintain the spherical shape of nano-silica, although there is still a visible appearance of aggregated grains. The selection of using a microwave with a gradual power supply rather than an oven as the final stage of sample drying, based on Figure 3, shows the method's effectiveness in Freitas' research, which maintains a round shape during the sample drying process.

Conclusion

This study successfully obtained silica nanoparticles from geothermal silica slag through the sol-gel method under alkaline conditions with pH 8 can produce silica with purity reaching 94.846% and recovery rate of 79.41%. Although there are still some impurities such as metals that cannot be removed, with a purity reaching 95% it is hoped that it can sufficiently increase the potential for silicon yield when used as a precursor for silicon synthesis. The sol-gel process using citric acid gave the nano-silica a pure white appearance that could remove the citric salt which usually can caused the black scorched color when entering the sample drying stage. The nano-silica obtained has size dimensions of 20-100 nm and have an average particle size around 55 nm with a spherical shape. For future research, optimization and added characterization data is indispensable for more clearer result such as surface area for silica product.

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