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# Utilization of Glass Waste in Silica Gel Production Using Sodium Hydroxide (NaOH) with The Sol-Gel Method

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#### Abstract

The utilization of glass waste as raw material for silica gel using the sol-gel method to produce high-quality products while reducing inorganic waste. Silica is extracted from glass waste using sodium hydroxide (NaOH), then converted into sodium silicate. Silica gel synthesis is carried out by varying two main parameters: the sodium silicate: water ratio (1:1, 1:2, 1:3, 1:4, and 1:5) and sulfuric acid concentration (1.5 M, 2 M, 2.5 M, 3 M, and 3.5 M). Product characterization was performed using XRF for SiO<sub>2</sub> content, XRD for amorphous structure, and BET for specific surface area. The highest SiO<sub>2</sub> content of 86.83% was obtained at a ratio of 1:5 and H<sub>2</sub>SO<sub>4</sub> concentration of 3.5 M. In contrast, the highest specific surface area of 186.82 m<sup>2</sup>/g was achieved at the same ratio and 3 M. Optimization using the Response Surface Methodology (RSM) indicated theoretical optimum conditions at a ratio of 1:10 and H<sub>2</sub>SO<sub>4</sub> concentration of 7.27 M. These conditions highlight the balance between sufficient acid strength to remove metallic and organic impurities and adequate dilution to maintain effective mass transfer, resulting in a SiO<sub>2</sub> purity of up to 99%. These results highlight the high potential of glass waste as an alternative silica source for adsorbent and catalyst support applications.

Keywords: glass waste, sol-gel, sodium silicate, silica gel

## INTRODUCTION

The volume of waste in Indonesia reached 335,707.5 tons in 2023. One type of waste produced is glass waste, which can damage the quality of the environment and has no economic value. Given the large amount of waste generated, more sustainable management efforts are needed, one of which is through the reuse of glass waste. Glass waste has the potential to be recycled into new products because broken glass retains the same properties as new glass, such as transparency, resistance to chemical reactions, and a high melting point at high temperatures. Processing glass waste into a product that highlights the advantages of glass properties can increase the low economic value of glass waste (Abdurrahman & Larasati, 2013).

Because of its high SiO<sub>2</sub> content, glass waste can be a potential precursor for silica gel production. Glass waste contains mainly silicon dioxide (SiO<sub>2</sub>) at 73.76% and several other compounds, namely aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>), and calcium carbonate (CaO) (Nursyamsi et al., 2016). The high SiO<sub>2</sub> content in glass waste can be utilized and processed into silica gel (Rahayu et al., 2022). Silica gel is one of the silica-

based materials with various applications, such as in the pharmaceutical, ceramic, paint, and specialty chemical industries. Silica gel is a high-molecular-weight silicic acid polymer that absorbs a large amount of water, giving it a gel-like consistency (Sanam et al., 2023). Silica gel has several advantages: high inertness, hydrophilicity (ability to bind water), thermal and mechanical stability, and relatively low swelling in organic solvents. These properties make silica gel widely used as a drying agent, catalyst support, absorbent, or adsorbent (Shinde et al., 2021).

Silica gel can be synthesized using two methods, which are the physical (grinding) and the chemical (sol-gel, chemical vapor condensation, and reverse microemulsion) methods (Abro et al., 2025). The solgel method is one of the most successful for preparing nano-scale metal oxide materials. The sol-gel method is one of the "wet methods" because the process involves a solution as its medium. As its name suggests, the sol-gel method undergoes a phase change from sol (a colloid with suspended solids in a solution) to gel (a colloid with a larger solid fraction than sol). The production of silica gel consists of four stages, namely the conversion of silica into sodium silicate by

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adding NaOH, the formation of a hydrogel through the reaction of Na<sub>2</sub>SiO<sub>3</sub> with acid, the production of silica hydrogel, and the formation of xerogel from heated silica hydrogel (Yusuf et al., 2023).

In addition to natural sources of silica, several agricultural wastes have also been studied. Silica gel synthesis has so far been carried out from various sources, such as rice husk ash and corn cob ash. One form of utilizing rice husk waste as raw material for silica gel has been conducted by Febryzha et al. (2025), which states that the synthesis of silica gel from rice husk ash activated using a 0.2 M NaOH solution produces a silica content of 53.09%. Fathurrahman et al. (2020) conducted a study on the characteristics of silica gel synthesized from corn cob ash with variations in acid usage (acetic acid and hydrochloric acid), resulting in a silica content of 64.12% in the silica gel.

Based on previous studies, various wastes such as rice husk ash and corn cob ash have been used as raw materials for silica gel production. However, glass waste has not yet been optimally utilized. Therefore, this study was conducted to utilize glass waste as an alternative raw material in silica gel synthesis. This study also aims to investigate the effect of the sodium silicate-to-water ratio and sulfuric acid concentration on the silica gel formation process. It is hoped that this research will help reduce the amount of glass waste and provide an alternative solution to meet the demand for silica gel.

#### METHODOLOGY

## **Materials and Instrumentals**

The materials used in this study were glass waste obtained from the Asahi glass store, sodium hydroxide (NaOH) as a solvent in SiO2 extraction, sulfuric acid as a silica gel former, and distilled water as a universal solvent, which were obtained from the Tidar Kimia store in Surabaya, East Java. The equipment used included a magnetic stirrer with a heating plate, porcelain plates, mortars, pestles, beaker glasses, filter paper, glass funnels, pipettes, racks and clamps, analytical scales, sieves, furnaces, ovens, and spatulas. Several tests were conducted, including testing the initial content of materials and products using an X-ray fluorescence spectrometer (PANalytical type Minipal 4) and then Design Expert 13 software to optimize the silica results. A device was used to test the material's crystal structure using an X-ray diffractometer (PANalytical type X'pert Pro), and surface area analysis was performed using a surface area analyzer (Micromeritics Tristar II Plus 3020).

## Methods

#### **Preparation of Materials**

Prepare the necessary materials, namely glass waste, sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), sodium hydroxide (NaOH), and distilled water. The glass waste obtained is washed to remove any impurities, then dried. The glass waste is crushed into fine particles and sieved using an 80-100 mesh sieve. The glass powder used is the powder that passes through the 80-mesh sieve and is retained by the 100-mesh sieve.

## Production of Sodium Silicate Solution (Na<sub>2</sub>SiO<sub>3</sub>)

Fine glass powder obtained in 50 grams was added to 300 mL of 3M NaOH solution. The mixture was then heated and stirred constantly at 250 rpm using a magnetic hot stirrer for 2 hours. The mixture was then calcined in a furnace at 400°C for 4 hours to produce a sodium silicate solid. The reaction that occurred was as follows:

$$SiO_{2(s)} + 2 NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)}$$
 (1)

#### Silica gel production

The sodium silicate solid is then dissolved in 500 mL of H<sub>2</sub>O and stirred with a magnetic stirrer for approximately 2 hours at a temperature of 100°C. The solution is then filtered using filter paper. The filtrate is used for the subsequent process, and the residue from the filtration is discarded. The sodium silicate solution is then diluted according to the conditions being tested (1:1, 1:2, 1:3, 1:4, and 1:5). 100 ml of sodium silicate solution was added with H<sub>2</sub>SO<sub>4</sub> at a concentration corresponding to the experimental conditions (1.5, 2, 2.5, 3, and 3.5 M). The mixture was stirred until the pH reached 7, which was analyzed using pH indicator paper, and a gel (hydrogel) was formed. The reaction that occurs is as follows:

$$Na_2SiO_{3(aq)} + H_2SO_{4(aq)} \rightarrow SiO_{2(S)} + Na_2SO_{4(aq)} + H_2O_{(l)}$$
 (2)

The hydrogel that is formed is left for 24 hours at room temperature. The hydrogel is washed with H2O and then filtered. The filtered hydrogel is used for the next process and the filtrate is discarded. The hydrogel is dried in an oven at 100°C for 6 hours until silica gel is formed.

# **Data Analysis**

In this research, three main analytical methods were used to characterize the results of silica gel synthesis, namely X-ray Fluorescence (XRF), X-ray Diffraction (XRD), and Brunauer-Emmett-Teller Surface Area Analysis (BET SAA). The XRF method was used to determine the chemical composition of the resulting material, particularly to measure the silica (SiO<sub>2</sub>) content formed during the synthesis process.

This technique enables the accurate identification and quantification of constituent elements. Next, the XRD method is applied to identify the crystal structure and calculate the crystallite size of the produced silica gel. This analysis is important to determine whether the product is amorphous or crystalline, as well as to understand the mineral phases formed.

Meanwhile, the BET method is used to determine the specific surface area of the silica gel by measuring the adsorption volume of nitrogen gas on the material's surface. This surface area is an important parameter in assessing the quality of silica as an absorbent or catalyst. These three methods are used in conjunction to obtain a comprehensive understanding of the physicochemical properties of the synthesized silica gel.

#### RESULTS AND DISCUSSION

### Analysis of silica gel raw materials

The characterization of raw materials in the form of glass waste was carried out using the X-Ray Fluorescence (XRF) method. The results of the XRF analysis of glass waste samples are shown in Table 1.

Table 1. Results of Glass Waste Content Analysis

Compound	Concentration (%)		
SiO <sub>2</sub>	75.10		
$K_2O$	0.60		
CaO	22.50		
$TiO_2$	0.10		
$Cr_2O_3$	0.05		
MnO	0.03		
$Fe_2O_3$	1.49		
CuO	0.05		
BaO	0.07		

The X-Ray Fluorescence (XRF) analysis showed that the glass waste contained 75.1% SiO<sub>2</sub> and 22.5% CaO as the main components, with small amounts of other elements such as K<sub>2</sub>O, Fe<sub>2</sub>O<sub>3</sub>, and TiO<sub>2</sub>. This composition indicates that the glass waste is rich in silica and has good chemical stability. This makes it highly suitable for reuse as a secondary raw material for glass recycling and other applications such as silica gel.

Figure 1 shows the silica diffractogram of glass waste, indicating an amorphous structure. This is characterized by broad diffraction peaks in the range of  $2\theta = 18^{\circ}-27^{\circ}$ , with the highest intensity at  $2\theta = 23.23^{\circ}$ , indicating very small (nano) crystal sizes. This amorphous structure is formed due to rapid cooling

after high heating, which prevents silica atoms from arranging themselves in a crystalline manner.

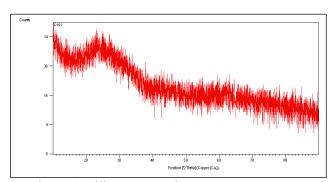


Figure 1. Silica Waste Glass Factogram Pattern

The cooling rate affects the degree of regularity of the silica structure, with rapid cooling resulting in a more irregular structure, similar to glass (Shekunov, 2020).

## Silica Gel Product Analysis

Table 2 presents the results of SiO<sub>2</sub> purity measurements (%) from various combinations of sodium silicate: water ratios and H<sub>2</sub>SO<sub>4</sub> concentrations based on analysis using the X-Ray Fluorescence (XRF) method.

Table 2. Purity of SiO<sub>2</sub> (%) at Various Ratios of Sodium Silicate: Water and H<sub>2</sub>SO<sub>4</sub> Concentration

H <sub>2</sub> SO <sub>4</sub> concentration (M)	Ratio Na <sub>2</sub> SiO <sub>3</sub> :H <sub>2</sub> O	Purity of SiO <sub>2</sub> (%)
	1:1	70.50
	1:2	70.05
1.5	1:3	69.60
	1:4	72.15
	1:5	74.70
	1:1	71.23
	1:2	71.72
2	1:3	72.20
	1:4	74.97
	1:5	77.73
	1:1	71.97
	1:2	73.38
2.5	1:3	74.80
	1:4	77.78
	1:5	80.77
	1:1	72.70
	1:2	75.05
3	1:3	77.40
	1:4	80.60
	1:5	83.80
	1:1	73.43
	1:2	76.72
3.5	1:3	80.00

H <sub>2</sub> SO <sub>4</sub> concentration (M)	Ratio Na <sub>2</sub> SiO <sub>3</sub> :H <sub>2</sub> O	Purity of SiO <sub>2</sub> (%)	
	1:4	83.42	
	1:5	86.83	

Increases in sulfuric acid concentration and the solubility ratio of sodium silicate to water significantly affect the purity of SiO<sub>2</sub> in silica gel. At a sulfuric acid concentration of 1.5 M, the purity of SiO<sub>2</sub> ranges from 69.60% to 74.70%. However, at a concentration of 3.5 M, the purity of SiO<sub>2</sub> increases to 86.83% at a solubility ratio of 1:5. This indicates that increasing both variables can produce silica gel with higher purity. Higher acid concentrations accelerate the silica precipitation process, while an optimal dissolution ratio supports the formation of a more stable gel. Combining both provides the best conditions for achieving the highest SiO<sub>2</sub> purity.

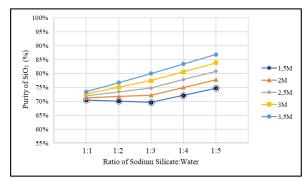


Figure 2. Effect of sodium silicate:water dilution ratio on SiO<sub>2</sub> purity with various variations in sulfuric acid concentration

Increasing the dilution ratio of sodium silicate solution: water has been shown to have a positive effect on the purity of SiO2 in silica gel. The higher the dilution ratio (e.g., 1:5), the purer the silica produced, as there are fewer impurities in the solution, making the precipitation process more selective. Conversely, at higher sodium silicate concentrations, more impurities precipitate, thereby reducing silica purity (Lee et al., 2021). Optimal conditions are achieved at a ratio of 1:5 with an acid concentration of 3.5 M, resulting in a SiO<sub>2</sub> purity of 86.83%. Although relatively high, the silica purity is still quite low compared to the study by Fabiani (2018), which achieved 99.76%. However, not all high ratios result in high purity, as other factors, such as washing efficiency, also play a role. For example, at a ratio of 1:3 and a concentration of 1.5 M, the purity of SiO<sub>2</sub> was only 69.60%, which was thought to be due to suboptimal washing (Yuliatun et al., 2019). Therefore, in addition to dilution, the washing process

is also very important for producing silica gel with high SiO<sub>2</sub> purity.

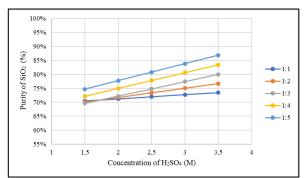


Figure 3. The Effect of Sulfuric Acid Concentration on SiO2 Content with Various Variations in Sodium Silicate: Water Dilution Ratios

The silica purification process is influenced by acid concentration, especially in methods such as solgel or leaching. The addition of H2SO4 serves to dissolve impurity metals such as Fe, Ca, and Mg through reactions with H+ ions, forming soluble salts that are easy to separate. As the acid concentration increases, the efficiency of impurity metal dissolution improves, resulting in purer silica. The purity of SiO<sub>2</sub> increases with increasing H<sub>2</sub>SO<sub>4</sub> concentration, with the highest purity of 86.83% at a concentration of 3.5 M. These results indicate that increasing the concentration of H2SO4 enhances the removal of metal elements such as CaO, Fe<sub>2</sub>O<sub>3</sub>, CuO, and Cr<sub>2</sub>O<sub>3</sub>, which can reduce the purity of silica. Silica purification can be achieved by dissolving impurity elements using an acidic solvent, such as Fe and Ca (Harimu et al., 2019).

The purity of the extracted silica reached a maximum value of 86.83%, indicating success in removing most of the impurities from the initial raw material. The initial CaO content of 22.5% was successfully reduced to 0.59% after treatment with 3 M H<sub>2</sub>SO<sub>4</sub> and a sodium silicate ratio of 1:5, demonstrating the effectiveness of the acid process. However, impurities such as SO<sub>3</sub> were still found in the final product. Unlike SiO2 and CaO, which are indeed the main components of glass waste, SO3 is not part of the original structure of the material. The presence of SO<sub>3</sub> is most likely due to suboptimal washing during the filtration stage, so the acid residue was not completely removed and caused further contamination (Nuraini et al., 2019). As the results are based on single measurements, future work incorporating replicate experiments and error analysis is recommended to improve their reliability and reproducibility.

The sodium silicate:water dilution ratio is a key factor in the synthesis of silica gel using the sol-gel method. The higher the dilution ratio (more water), the greater the surface area of the resulting silica.

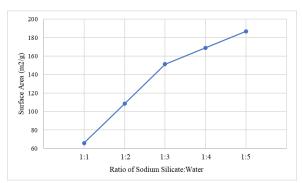


Figure 4. Effect of Sodium Silicate: Water Ratio on Surface Area (m<sup>2</sup>/g)

Concentrated solutions accelerate the condensation reaction and form a solid structure with small pores. At the same time, high dilution reduces the silica concentration, increases the distance between particles, and produces smaller particles with a larger surface area (Lee et al., 2021). Although pore size and volume

were not measured in this study, they are generally influenced by the same conditions that control surface area. According to Minju et al. (2021), lower silicate concentrations tend to produce larger pores and higher pore volumes, while higher concentrations form denser networks with smaller pores, suggesting that adjusting the synthesis conditions can affect both surface area and pore structure.

In this study, the results show that silica gel with a sodium silicate:water ratio of 1:5 and an H<sub>2</sub>SO<sub>4</sub> concentration of 3 M has the highest surface area, namely 186.82 m<sup>2</sup>/g. This value far exceeds the minimum standard of 30 m<sup>2</sup>/g for catalyst applications (Dreibelbis, 1986). A surface area that is too low (e.g., fused silica 0.5–3 m<sup>2</sup>/g) reduces catalyst effectiveness, while a very high surface area (>300 m<sup>2</sup>/g) can hinder reactant diffusion due to excessively small pores. Thus, a value of 186.82 m<sup>2</sup>/g is considered ideal because it provides a large active surface while maintaining sufficient pore size for efficient reactant diffusion, supporting optimal catalytic performance.

Table 3. ANOVA results on SiO<sub>2</sub> purity response: Quadratic Model

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	246.69	5	49.34	355.44	< 0.0001
A-Concentration of H <sub>2</sub> SO <sub>4</sub>	73.36	1	73.36	528.51	< 0.0001
B-Sodium silicate:water	116.16	1	116.16	836.86	< 0.0001
dilution ratio					
<u>Residual</u>	0.9716	7	0.1388		
Lack of Fit	0.8716	3	0.2905	11.62	0.0192
Pure Error	0.1000	4	0.0250		
Cor Total	247.66	12			
$\mathbb{R}^2$	0.9961				
Adj R <sup>2</sup> Pred R <sup>2</sup>	0.9933				
$Pred R^2$	0.9637				
Adec precision	67.2148				

## Optimizing SiO<sub>2</sub> purity with Design Expert 13

Based on Table 3, analysis of variance (ANOVA) was obtained to interpret the relationship between the H2SO4 concentration variable and the sodium silicate:water dilution ratio with the SiO<sub>2</sub> purity response. The model used had a significant effect on the response because it had a p-value of <0.0001. The adjusted R<sup>2</sup> and predicted R<sup>2</sup> values are 0.9933 and 0.9637, respectively, which are reasonable since the difference is less than 0.2. The coefficient of correlation (R) reflects the strength of the relationship between predicted and actual values in a statistical experiment, indicating prediction accuracy.

(Djimtoingar et al., 2022). Generally, a high R<sup>2</sup> value demonstrates good agreement between predicted and experimental data (Chicco et al., 2021).

Based on the model, lack of fit, the difference between adj  $R^2$  and pred  $R^2$ , and adequate precision, this RSM model can be applied. The equation obtained from the selected model for the  $SiO_2$  content response is as follows:

$$SiO_2 = 73,01 + 3,50A - 4,40B - 2,30AB + 0,0493A^2 + 3,32B^2$$
 (3)

Explanation:  $A = Concentration of H_2SO_4(M)$ 

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B= Dilution ratio of sodium silicate:water

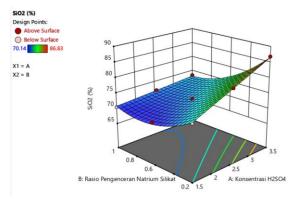


Figure 5. Response Surface of SiO<sub>2</sub> Purity

The response surface model for SiO<sub>2</sub> purity with the variables of H<sub>2</sub>SO<sub>4</sub> concentration and the appropriate sodium silicate dilution ratio is a quadratic curve model. The response surface model shows that SiO<sub>2</sub> purity increases with increasing H<sub>2</sub>SO<sub>4</sub> concentration and decreasing dilution ratio. The optimization in this study helps determine the most appropriate process conditions and facilitates the exploration of factors quickly achieve to the optimal range (Fadhilah et al., 2025). After obtaining the mathematical model from Design Expert, further optimization was performed using the Solver feature in Microsoft Excel. Although Design Expert provides built-in optimization tools, Excel Solver was employed to provide greater flexibility in applying variable constraints and to validate the regression model outside the statistical software environment. Previous work by Khalifa et al. (2020) demonstrated that Response Surface Methodology (RSM) can be effectively implemented and optimized using Excel and its Solver function, showing comparable accuracy to specialized statistical software while being more accessible and versatile. By setting constraints on the variables and the objective to achieve maximum SiO<sub>2</sub> purity, the optimal combination was obtained at an H<sub>2</sub>SO<sub>4</sub> concentration of 7.27 M and a sodium silicate dilution ratio of 0.1, resulting in a predicted SiO<sub>2</sub> purity of 99%. This optimum represents a theoretical outcome from the response surface model and Excel Solver optimization, and no experimental validation was performed under these exact conditions.

Although the response optimized here is purity, this parameter is directly relevant to catalytic applications because impurities can block pores, alter acidity, and reduce catalyst stability. Thus, purity optimization represents a crucial first step, while further characterizations such as full nitrogen adsorption—desorption isotherm analysis (BET surface

area, pore size distribution, and pore volume) and XRD on the optimized sample are needed to confirm its suitability for catalytic use.

#### **CONCLUSION**

Glass waste shows great potential as a raw material for silica gel synthesis via the sol-gel method. Unlike agricultural wastes such as rice husk and corn cob, which have been widely studied and produce silica gel with silica contents of 53.09% and 64.12%, respectively, glass waste remains underutilized yet can provide higher silica content, making it a promising alternative. In this study, variations in the ratio of sodium silicate to water and H2SO4 concentration significantly affect the purity and characteristics of the resulting silica gel. A dilution ratio of 1:5 with a H<sub>2</sub>SO<sub>4</sub> concentration of 3 M produces silica gel with the highest specific surface area of 186.8174 m<sup>2</sup>/g and SiO<sub>2</sub> purity reaching 86.83%. This value exceeds the minimum surface area standard of 30 m<sup>2</sup>/g for catalyst applications, making the synthesized silica gel highly promising as an adsorbent and catalyst support material. Additionally, optimization using Design Expert indicated optimal conditions at an H<sub>2</sub>SO<sub>4</sub> concentration of 7.27 M and a ratio of 1:10, with theoretical SiO<sub>2</sub> purity reaching 99%.

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