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Article

Tailoring the Adsorptive Efficiency and Pore Characteristics of Silica through Solvothermal Treatment Assisted by Cetyltrimethylammonium Bromide (CTAB)

Fransiska Altrima Key Timu[a], Dwi Ana Anggorowati[b], Harimbi Setyawati[c], Mohammad Istnaeny Hudha[d], Nanik Astuti Rahman[e,*]

ITN Malang Jl. Raya Karanglo, Malang, Indonesia

E-mail: [a]triskey00@gmail.com, [b]anggoro_dwiana@yahoo.com, [c]harimbi@lecturer.itn.ac.id, [d]istnaeny.hudha@gmail.com, [e]nanik.astuti@lecturer.itn.ac.id
*Corresponding author: nanik.atuti@lecture.itn.ac.id | Phone number: +6281230795225

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Abstract
Silica is a porous material widely utilized across various fields. One of the potential sources of silica is sandblasting waste, which contains more than 90% silica. This study aims to investigate the influence of Cetyl Trimethyl Ammonium Bromide (CTAB) concentration and solvothermal duration on producing silica with enhanced adsorption capacity and porosity. The independent variables in this research include CTAB surfactant concentrations of 0.1, 0.2, 0.3, 0.4, and 0.5%, as well as solvothermal processing times of 8, 10, 12, 14, and 16 hours. The optimal result was obtained with the addition of 0.5% CTAB and a solvothermal duration of 16 hours, in which SEM-EDX analysis revealed large visible pores formed by aggregates of fine particles arranged in a homogeneous structure. The adsorption capacity, using a methylene blue concentration of 30 mg, was found to be 27.1822 mg/g. Based on the study on the Enhancement of Adsorption Capacity and Porosity of Silica Using Cetyl Trimethyl Ammonium Bromide (CTAB) via the Solvothermal Method, it can be concluded that both adsorption capacity and porosity are influenced by the addition of CTAB, solvothermal duration, and the concentration of methylene blue used in the adsorption capacity test.

Keywords: Silica, sandblasting waste, CTAB, solvothermal

1. Introduction

Indonesia is one of the countries rich in abundant natural resources, which play a vital role in the national economy and industrial needs. One of the natural resources that has recently gained significant attention is silica sand derived from sandblasting waste, which has been effectively and efficiently developed as an abrasive medium. [1]

Silica is a porous material commonly processed from silica sand, which originates from the weathering of rocks carried by water or wind currents and subsequently deposited in natural environments.[2] It possesses a distinctive characteristic in which its hydroxyl (–OH) functional groups can be replaced by other functional groups. In addition, silica can be applied as a water absorbent when it exhibits high absorption capacity, which can be achieved by increasing its surface area and porosity. [3]

Silica is also capable of maintaining humidity and preventing mold growth caused by unpredictable temperature fluctuations. It can be used as a packaging material incorporated in leather goods, footwear, clothing, food, pharmaceuticals, and household equipment. [4]

At this stage, high-purity sodium silicate is obtained due to the reaction between sodium,

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silica, and oxygen, resulting in sodium silicate that efficiently enhances the adsorption function of silica. The formation of sodium silicate relies on the molar interaction between Na^+ ions and SiO_2, producing a solid material with characteristics that allow it to dissolve in a solvent.[5] In addition, another factor influencing the extraction process is the high temperature, where the presence of heated quartz combined with an alkaline mixture leads to the breakdown of silica mineral bonds, forming crystalline structures that are soluble in ionic compounds. [6]

To achieve high adsorption capacity and porosity, silica is processed using the solvothermal method, which involves silica precursors such as Tetraethyl Orthosilicate (TEOS) or sodium silicate under elevated temperature and pressure. [7] This method also enables modification of the silica surface properties depending on the type of surfactant used. In this study, Cetyl Trimethyl Ammonium Bromide (CTAB), a quaternary cationic surfactant with a long alkyl chain, is employed to produce larger pore diameters within a shorter synthesis time. [8]

A previous study conducted by Dafnaz et al., titled "The Effect of Adding Cetyl Trimethyl Ammonium Bromide (CTAB) to Silica Derived from Sodium Silicate (Na_2SiO_3)", evaluated the influence of CTAB concentration and aging time on the characteristics of the resulting silica. In that study, CTAB concentrations of 0.1, 0.15, 0.2, 0.25, and 0.3% were tested along with aging durations of 8, 12, 16, 20, and 24 hours.The results showed that a CTAB concentration of 0.2% produced the highest adsorption capacity, reaching 685.314 mg/g. Meanwhile, an aging time of 16 hours provided the optimal water absorption capacity, measured at 586.324 mg/g. [9]

Silica is known as one of the most effective adsorbent materials due to its excellent adsorption capacity. This capability is primarily supported by its well-developed and ordered pore structure, along with a high specific surface area. These characteristics enable silica to capture and retain various types of molecules, both polar and non-polar, through physical and chemical interactions on its surface**.** [10] SEM analysis is highly valuable in evaluating the success of surface characteristic synthesis in silica and can complement other quantitative data such as specific surface area obtained from BET analysis. [11]

2. Material and Method

Materials used in this study included: 20 grams of silica sand, 5N sodium hydroxide (NaOH), 5N hydrochloric acid (HCl), 70% ethanol, and the surfactant cetyltrimethylammonium bromide (CTAB).

CTAB was used as an independent variable at concentrations of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5%, along with solvothermal durations of 8, 10, 12, 14, and 16 hours.

There are several methods commonly employed in the synthesis of silica, namely:
Prepare 1 kg of sandblasting waste, sieve the sandblasting waste using an 80-mesh screen, heat the sieved material at 900℃ in a furnace for 2 hours, allow the heated sand to cool to room temperature, perform magnetic filtration to remove any remaining metallic impurities, weigh 20 grams of sand for each experimental variable, xtract the sand with 120 mL of 5N sodium hydroxide (NaOH) solution for 1 hour at 120 °C using a magnetic stirrer hot plate set at 400 rpm, Separate the resulting sodium silicate solution from the residue using Whatman No. 40 filter paper, slowly add 120 mL of 5N hydrochloric acid (HCl) to the sodium silicate solution until a gel is formed, add Cetyltrimethylammonium Bromide (CTAB) solutions at concentrations of 0.1%, 0.2%, 0.3%, 0.4%, and 0.5% to the formed gel, age the gel at room temperature for 18 hours, filter the gel using Whatman No. 40 filter paper, wash the gel with 70% ethanol three times, subject the gel to a solvothermal process at 100 °C for 8, 10, 12, 14, and 16 hours, cool the product to room temperature.

3. Results and Discussion

The quality of silica derived from sandblasting silica sand waste, treated with Cetyltrimethylammonium Bromide (CTAB) and subjected to various solvothermal durations, can be analyzed through adsorption capacity testing using Methylene Blue and Scanning Electron Microscopy (SEM) analysis. These analytical methods are capable of evaluating the performance of the synthesized silica in terms of its adsorption capability and porosity enhancement, as determined through the following tests:

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3.1. Adsorption Capacity Test

Table 1. Spectrophotometric Absorbance Data

CTAB (%)	Solv	Methylene Blue Concentration	Absorbance Value
		10	1.492
	8	20	1.504
		30	1.863
		10	0.591
	10	20	0.602
		30	1,504
		10	0.602
0.5	12	20	0.843
		30	2.108
		10	0.520
	14	20	0.521
		30	0.600
		10	0.489
	16	20	0.548
		30	0.570

3.1.1. Adsorption Capacity at 0.5% CTAB with 8-Hour Solvothermal Treatment

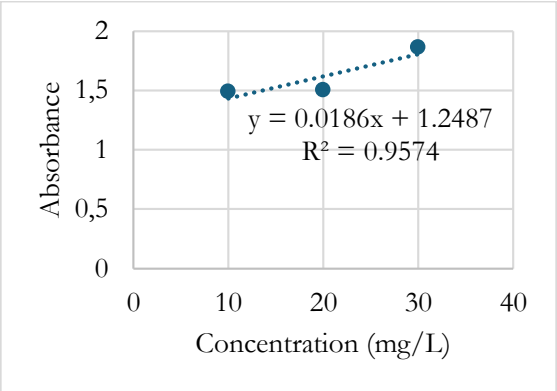

Fig. 1. Calibration Curve Graph for 0.5% CTAB with 8-Hour Solvothermal Treatment

The test results for silica treated with 0.5% CTAB and subjected to an 8-hour solvothermal process showed that the absorbance values increased with higher concentrations of methylene blue. The absorbance measurements were performed using a spectrophotometer by dissolving the adsorbent (silica) with methylene blue according to the concentration variations.

Based on the calibration curve above, the obtained equation is y = 0.0816x + 1.2487 with a regression coefficient (R^2) of 0.9574, using a linear graph to calculate the concentration in the sample. The variable y represents the absorbance, while x corresponds to the methylene blue concentration. The regression result indicates a good coefficient of determination, as the R^2 value is close to 1.

The adsorption capacity increased significantly in line with the rise in the equilibrium concentration (Ce), as calculated using the regression equation. Among the three initial concentrations, the highest adsorption capacity was obtained at an initial methylene blue concentration (C_0) of 30 mg/L, with a value of 22.4718 mg/g.

3.1.2. Adsorption Capacity at 0.5% CTAB with 10-Hour Solvothermal Treatment

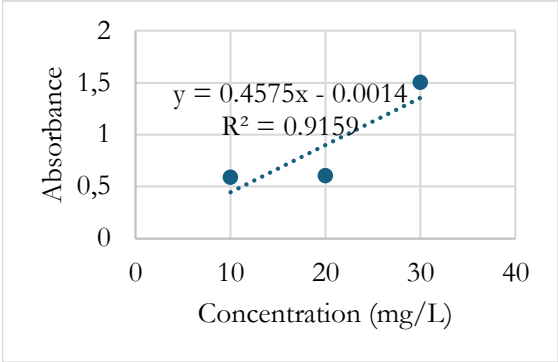

Fig. 2. Calibration Curve Graph for 0.5% CTAB with 10-Hour Solvothermal Treatment

The calculated adsorption capacity data indicate a direct correlation, showing that an increase in the initial equilibrium concentration of methylene blue (C_0) in the solution leads to a corresponding increase in the adsorption capacity (Q_e). The adsorption capacity rose significantly with the increase in final concentration (C_e), as determined using the regression equation. Among the three methylene blue concentrations tested, the highest adsorption capacity was obtained at a solvothermal duration of 10 hours, specifically at an initial methylene blue concentration (C_0) of 30 mg/L, with a value of 26.7156 mg/g.

3.1.3. Adsorption Capacity at 0.5% CTAB with 12-Hour Solvothermal Treatment

The calibration curve yielded the linear equation y = 0.3833x + 0.0037 with a regression coefficient (R^2) of 0.9217. In this equation, y represents the absorbance, while x corresponds to the methylene blue concentration. The R^2 value obtained from the regression plot indicates a good coefficient of determination, as it is close to 1.

The adsorption capacity increased significantly with the rise in the final concentration (Ce), as calculated using the regression equation.

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Among the three methylene blue concentrations tested, the highest adsorption capacity at a solvothermal duration of 12 hours was obtained at an initial methylene blue concentration (C_0) of 30 mg/L, with a value of 24.5100 mg/g.

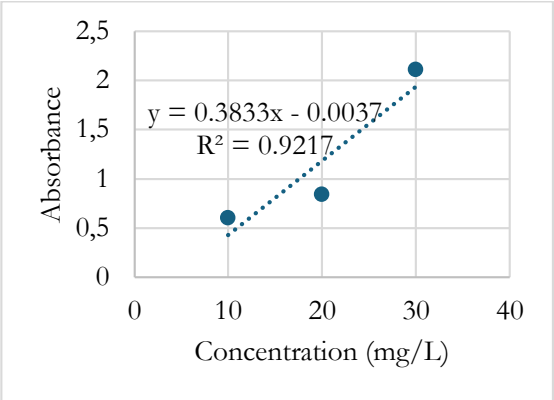

Fig. 3. Calibration Curve Graph for 0.5% CTAB with 12-Hour Solvothermal Treatment

3.1.4. Adsorption Capacity at 0.5% CTAB with 14-Hour Solvothermal Treatmen

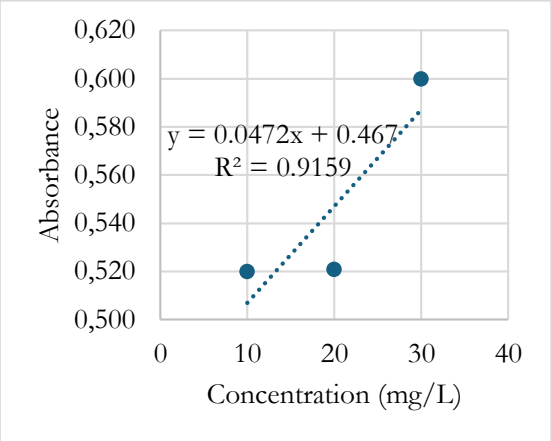

Fig. 4. Calibration Curve Graph for 0.5% CTAB with 14-Hour Solvothermal Treatment

The calculated adsorption capacity results indicate that an increase in the initial equilibrium concentration of methylene blue (C_0) in the solution leads to a corresponding increase in the adsorption capacity (Q_e). This capacity rises concurrently with the increase in final concentration (C_e), as determined using the regression equation. Among the three methylene blue concentrations tested, the highest adsorption capacity at a solvothermal duration of 14 hours was obtained at an initial concentration (C_0) of 30 mg/L, with a value of 27.1822 mg/g.

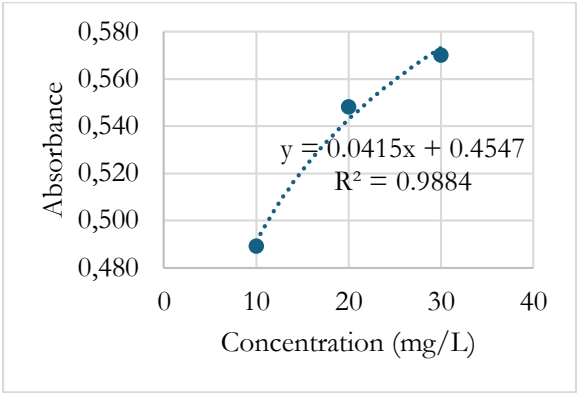

Fig. 5. Calibration Curve Graph for 0.5% CTAB with 16-Hour Solvothermal Treatment

3.1.5. Adsorption Capacity at 0.5% CTAB with 16-Hour Solvothermal

The test results for silica treated with 0.5% CTAB and subjected to a 16-hour solvothermal process showed that higher variations in methylene blue concentration led to an increase in absorbance values.

The calibration curve produced the logarithmic equation y = 0.0415x + 0.4547 with a regression coefficient (R^2) of 0.9884. In this equation, y represents the absorbance and x represents the methylene blue concentration. The high R^2 value indicates a strong coefficient of determination, as it is close to 1. This result confirms that the use of the logarithmic graph is appropriate for the analysis.

The calculated adsorption capacity results indicate that an increase in the initial equilibrium concentration of methylene blue (C_0) in the solution leads to a corresponding increase in the adsorption capacity (Q_e). This increase in adsorption capacity occurred simultaneously with the rise in the final concentration (C_e), as determined using the regression equation. Among the three methylene blue concentrations tested, the highest adsorption capacity at a solvothermal duration of 14 hours was obtained at an initial concentration (C_0) of 30 mg/L, with a value of 27.2217 mg/g.

3.2. Scanning Electron Microscopy (SEM) Analysis

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Table 2. SEM Data

CTAB (%)	Solv	SEM-EDX Result
0,5	8	fairly ordered pores, dense material surface, rough texture
	10	homogeneous material surface, porous aggregate structure
	12	aggregates of fine particles stacked together, larger pores, crystalline structure
	14	the particles form micron-sized agglomerate structures, high surface area
	16	the material surface exhibits a pore structure with more visibly enlarged pores, particles form aggregates, porous and amorphous

3.2.1. Test Results for 0.5% CTAB at 8-Hour Solvothermal Duration

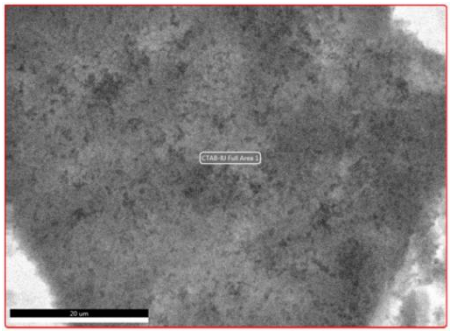

Fig. 6. Scanning Electron Microscopy (SEM) of 0.5% CTAB with 8-Hour Solvothermal Treatment

Based on the analysis using Scanning Electron Microscopy (SEM), the surface morphology of the sample treated with 0.5% CTAB and a solvothermal duration of 8 hours exhibited a relatively uniform structure with dispersed particles forming aggregates. The image at 20.0k magnification revealed that the material surface consisted of fine clusters with an amorphous form that appeared to be geometrically defined. The surface appeared dense yet exhibited a rough texture, indicating potential porosity or interparticle voids that may play a significant role in adsorption applications or as a catalyst support material.

The analysis was performed using Energy Dispersive X-ray Spectroscopy (EDX), integrated within the SEM system. Quantitative results indicated that the dominant elements in the sample were oxygen (O) at 40.69 wt% and silicon (Si) at 23.30 wt%, suggesting that the material is silica-based (SiO_2). A relatively high carbon (C) content of 17.12 wt% indicates the presence of organic compounds, likely originating from the surfactant CTAB (Cetyl Trimethyl Ammonium Bromide) used during the synthesis process. Other elements detected in smaller amounts include sodium (Na), aluminum (Al), chlorine (Cl), and iodine (I), which are presumed to be residual reactants or surface modification by-products.

3.2.2. Test Results for 0.5% CTAB at 10-Hour Solvothermal Duration

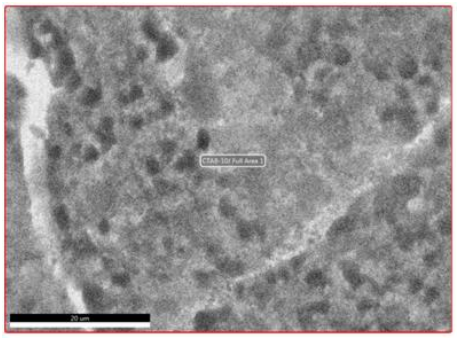

Fig. 7. Scanning Electron Microscopy (SEM) of 0.5% CTAB with 10-Hour Solvothermal Treatment

The SEM image of the sample treated with 0.5% CTAB and subjected to a 10-hour solvothermal process shows a material surface with a homogeneous morphology and a rough, dense texture. The surface appears to consist of microscopic clusters forming a porous aggregate structure. At the displayed magnification (20.0k), these clusters are clearly visible, indicating a well-developed porous network. Compared to the 0.5% CTAB sample at 8-hour solvothermal treatment, this image reveals larger pore distributions. The presence of bright and dark regions suggests possible differences in material density or composition. A faint curved line observed near the center of the field of view may indicate the boundary between agglomerates or a pore formation path.

3.2.3. Test Results for 0.5% CTAB at 12-Hour Solvothermal Duration

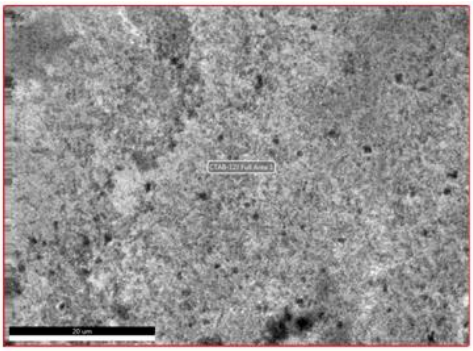

Fig. 8. Scanning Electron Microscopy (SEM) of 0.5% CTAB with 12-Hour Solvothermal Treatment

The Scanning Electron Microscopy (SEM) image of the sample treated with 0.5% CTAB and subjected to a 12-hour solvothermal process at 20.0k magnification reveals a surface structure composed of stacked fine particle aggregates. The observed pores are interpreted to be larger than those seen in previous tests at 8 and 10 hours under the same CTAB concentration. The material's surface exhibits randomly distributed micro-clusters forming a crystalline-like structure. At the displayed magnification, the surface texture appears relatively rough and porous, which is a typical indication of surfactant-templated synthesized materials such as those using Cetyltrimethylammonium Bromide (CTAB).

This image suggests the potential presence of mesoporous or macroporous structures, which are commonly desired in applications such as adsorbents, catalyst supports, or functional materials. The porous and incompletely dense morphology also implies that the material may possess a high specific surface area—an essential property for molecular transport systems or surface-driven reactions.

3.2.4. Test Results for 0.5% CTAB at 14-Hour Solvothermal Duration

The SEM image of the sample treated with 0.5% CTAB at a solvothermal duration of 14 hours reveals a surface morphology composed of randomly distributed fine particle aggregates forming a homogeneous layer. The surface texture appears complex, featuring a combination of bright and dark regions that represent variations in topography and material density. At a magnification of 20.0k, micron-sized agglomerate structures are observed, indicating potential porosity and diffusion pathways between the cavities.

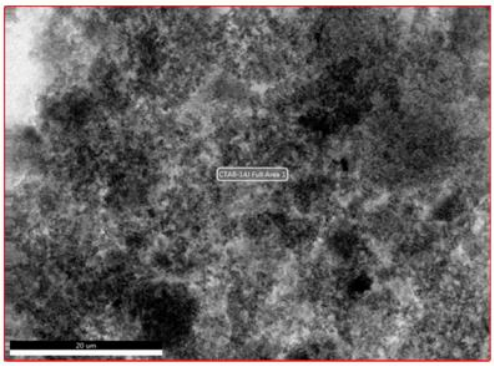

Fig. 9. Scanning Electron Microscopy (SEM) of 0.5% CTAB with 14-Hour Solvothermal Treatment

The surface also exhibits well-defined crystalline shapes, suggesting that the structure has developed larger porosity compared to the samples treated with 0.5% CTAB at 8–12 hours, likely due to an increase in specific surface area. The dense yet uneven distribution of aggregates is a typical characteristic of materials synthesized using structure-directing surfactants such as CTAB. This surfactant facilitates the formation of mesoporous materials with high surface areas, making them suitable for applications such as adsorbents, catalytic materials, or carriers of active molecules.

3.2.5. Test Results for 0.5% CTAB at 16-Hour Solvothermal Duration

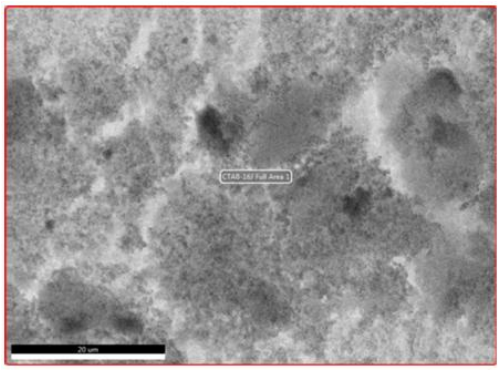

Fig. 10. Scanning Electron Microscopy (SEM) of 0.5% CTAB with 16-Hour Solvothermal Treatment

The SEM image of the sample treated with 0.5% CTAB and a solvothermal duration of 16 hours shows a material surface with visibly larger pore structures compared to previous observations. The surface consists of clusters of

particles forming aggregates with a homogeneous distribution. The bright and dark areas in the image reflect topographical variations and potential differences in material density, which may be associated with the presence of pores or voids between particles. This morphological form suggests a porous and amorphous structure, a common characteristic of materials synthesized using surfactants such as CTAB as a structure-directing agent.

To support the morphological observations, elemental composition analysis was performed using EDX. The results revealed that the sample surface was predominantly composed of carbon (C) at 29.69 wt% and oxygen (O) at 33.42 wt%, indicating the presence of both organic compounds and inorganic oxides. A significant amount of silicon (Si) was also detected at 17.35 wt%, confirming that the material is modified silica.

Future research can also be directed toward developing more efficient methods for removing CTAB, such as solvent extraction or gradual heating techniques, to obtain silica with a clean surface and fully open pore structure. Further characterization using techniques such as XRD, BET, FTIR, TEM, and zeta potential analysis is also essential to understand the relationship between synthesis conditions and the physicochemical properties of the resulting material.

This study has several limitations to maintain a clear research focus and allow for in-depth analysis of the results. First, the silica synthesis process was carried out on a laboratory scale using the solvothermal method with predetermined parameter variations; therefore, the findings are only applicable under the experimental conditions employed. The surfactant used was limited to Cetyltrimethylammonium Bromide (CTAB) as the pore-forming agent, and the effects of other surfactants beyond CTAB were not examined in this study. Second, the types of silica precursors and solvents were restricted to a single selected type (such as tetraethyl orthosilicate or sodium silicate) to maintain result consistency and to facilitate the analysis of the solvothermal treatment's influence on pore structure.

The higher carbon content compared to other CTAB-treated samples suggests the presence of residual CTAB surfactant within the structure or that the purification process did not completely remove organic compounds.

4. Conclusions

Based on the study on the Enhancement of Adsorption Capacity and Porosity of Silica Using Cetyl Trimethyl Ammonium Bromide (CTAB) via the Solvothermal Method, it can be concluded that both adsorption capacity and porosity are influenced by the addition of CTAB, solvothermal duration, and the concentration of methylene blue used in the adsorption capacity test. The optimal results for both analyses were obtained with the addition of 0.5% CTAB and a solvothermal duration of 16 hours. SEM-EDX analysis revealed large pore structures composed of uniformly distributed particle aggregates, indicating suitability for adsorption and catalytic applications. The highest adsorption capacity, achieved at a methylene blue concentration of 30 mg, was 27.1822 mg/g.

Acknowledgement

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References

[1] **Abdillah, N., 2019. Utilization of Sandblasting Silica Sand Waste as a Substitute Material for Fine Aggregate in Concrete Mixtures. Journal of Civil Engineering, pp. 10–16.**

[2] Bakri, S., 2023. Study on SiO_2 Separation from Silica Sand Using Shaking Table. Journal of Mining and Environmental Innovation, pp. 85–90.

[3] **Al'amiin, A. B., 2020. Characterization of Cellulose-Silica Fiber Composites Using Ethanol as Solvent. Journal of Chemical and Process Engineering, pp. 6–11.**

[4] Vazquez, N. I., 2017. Synthesis of Mesoporous Silica Nanoparticles By Sol–Gel As Nanocontainer for Future Drug Delivery Applications. Mesoporous Journal, pp. 140-144.

[5] Nuryono, 2005. Effect of Acid Concentration on the Characteristics of Silica Gel Synthesized from Sodium Silicate. Engineering Journal, pp. 23–30.

[6] Sukandar, 2010. Preliminary Study of Sandblasting Waste Utilization As A Coagulant. Jurnal Teknik Lingkungan , pp. 93-102.

[7] Efiyanti, L., 2021. Synthesis of Mesoporous Silica from Beach Sand Using Variations of Cetyl Trimethyl Ammonium Bromide (CTAB). Biological Sciences Research, pp. 374–381.

[8] Iqbal, M., 2024. Effect of pH Variation and CTAB (Cetyl Trimethyl Ammonium Bromide) on Silica Synthesis from Mount Kelud Volcanic Ash. Journal of Physics, pp. 433–438.

[9] Dafnaz, H. R., 2022. Effect of Cetyltrimethylammonium Bromide (CTAB) Addition on Silica Derived from Sodium Silicate (Na_2SiO_3). Chemistry Journal, pp. 102–105.

[10] Rizky, M., 2022. The Manufacturing of Silica Gel from Cane Pulp Cinders (Saccharum Officinarum) and Application for Adsorption Cu (Li) Ion. Jurnal Kimia, pp. 1-8.

[11] Marsyahyo, E., 2009. Brunauer–Emmett–Teller (BET) Analysis of the Surface Topography of Ramie Fibers (*Boehmeria nivea*) as a Reinforcing Medium in Composite Materials. Flywheel Journal, pp. 1–9