

Synthesis and characterization of SiO₂ from Madura Strait sediment with leaching acid treatment

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Abstract

Silicon dioxide (SiO₂) was successfully synthesized from Madura Strait sediment. The production process involved a wet digestion step using a strong acid to decompose the sample. Subsequent calcination and purification with strong acid effectively removed impurities, yielding a white sedimentary material. The synthesized SiO₂ was characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). After impurity removal, the silicon content in the raw sediment increased from 33.5% to 83.3%. XRD analysis confirmed the successful formation of amorphous SiO₂ particles. FTIR spectra indicated the presence of Si–O–Si and Si–OH functional groups, representing silanol and siloxane structures. SEM analysis revealed that the SiO₂ particles formed aggregates with an irregular arrangement, where individual particles appeared loosely associated.

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Keywords

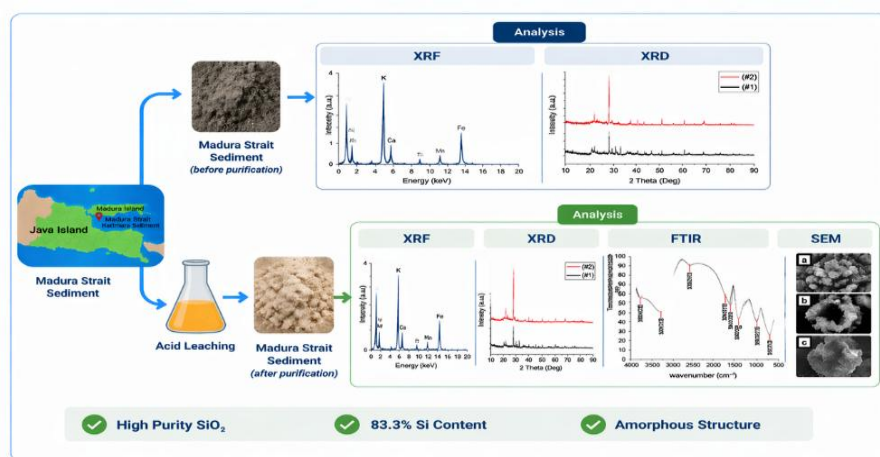
1. amorphous structure
2. Madura Strait sediment;
3. natural source;
4. adsorption properties;
5. impurity removal.

Section Editors

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Highlights

- Successful synthesis and characterization of silica from natural sources.
- The elemental abundance and composition of Madura Strait sediments were determined.
- A novel source to produce amorphous silica from natural sediments is available.
- Silicon content in raw sediment increased from 33.5% to 83.3% after impurity removal.
- FTIR spectra showed the presence of Si–O–Si and Si–OH functional groups.



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1. Introduction

Silicon dioxide (SiO₂) is a semiconductor compound with low toxicity, high thermal stability, and an amorphous and crystalline structure (Griscom, 2000). The properties have been extensively studied in different areas, such as electronics, photonic, chemicals, environmental, electrochemistry, and biomedical fields (Hosseini *et al.*, 2014; Li *et al.*, 2012; Nandanwar *et al.*, 2015). Silica can be found in nature as sand/sediment, quartz, glass, and more. As natural silica, it has a crystalline form, while as a synthesized compound, it is amorphous. Silica compounds can be synthesized from silicate solutions or silane reagents. The process of making silica can be made with the basic ingredients of the alkoxide compound tetraethyl orthosilicate/tetramethyl orthosilicate (TEOS/TMOS) by the sol-gel method (Nikmah *et al.*, 2019). However, due to the high cost of TEOS and TMOS, an alternative approach involves utilizing natural silica sources, offering a more economical perspective.

Some researchers have employed isotope, partial, and total analyses to trace the natural sources of sediment (Wijaya *et al.*, 2018a; 2018b; 2019; Suci *et al.*, 2020). The production of silica from strait sediments has recently emerged as a significant topic in marine chemistry, particularly for evaluating its potential as an alternative to synthetic silica. Given their high silica content, this study explored the feasibility of utilizing natural silica resources from Madura Strait sediments.

This research began with sediment characterization using X-ray fluorescence (XRF) to confirm the presence of abundant silicon (Si) in the Madura Strait sediments. The production of SiO₂ involved a wet digestion process using strong acid to leach the sample, followed by calcination to obtain the solid product. The resulting purified sedimentary material can serve as an alternative source for silica production.

2. Experimental

2.1. Synthesis of SiO₂ from Madura Strait sediment

The sediment was dried at 60 °C and ground into a fine powder. The sample was subsequently sieved using a 200-mesh sieve. A 50-gram portion of the sample was weighed and mixed with 50 mL of demineralized water, 350 mL of HCl solution (1.00317.2500 – Hydrochloric acid 37% for analysis EMSURE®ACS, ISO Reag. Ph Eur, Germany industry), and 100 mL of HNO₃ solution (1.00456.2500 – Nitric acid 65% for analysis EMSURE®ACS, ISO Reag. Ph Eur, Germany industry), followed by a 16-hour resting period. Digestion was then performed under reflux at 70 °C for 2 h, after which the sample was cooled to room temperature. The digested solution was filtered, and the residue was neutralized to a pH of 7. The sample was then calcinated in a furnace at 800 °C for 4 h. The resulting ash was moistened with hot distilled water, treated with 100 mL of 37% HCl, and evaporated to dryness, a process repeated three times. Subsequently, 200 mL of distilled water and 100 mL of 37% HCl were added, and the mixture was heated for 30 min before filtration. The residue was washed with hot distilled water and dried using filter paper. Finally, the sample underwent a second calcination at 800 °C for 4 h.

2.2. Characterization of Madura Strait sediment and SiO₂

The composition of the Madura Strait sediment and SiO₂ samples was determined using X-ray fluorescence (XRF). The XRF spectrometer used was PANalytical Type MiniPal 4, equipped with a Rh tube as an X-ray generator (9 W X-ray tube, Max 30 KV, max 1 mA), 5 filter tubes, Silicon detector and 12 position sample tray with sample spinner. X-ray diffraction was used to analyse the samples' structural characteristics (XRD, Merck PANalytical, Model: X'Pert PRO). The diffractograms were obtained in the 2θ range between 10° and 90° with a Cu-Kα radiation wavelength 1.5418 Å until a diffraction pattern was formed which was shown in the form of a diffractogram. The functional groups of SiO₂ were examined using Fourier transform infrared (FTIR) spectroscopy (Shimadzu, Model: IRPrestige 21) within an acquisition range of 400–4000 cm⁻¹. The morphology of SiO₂ was investigated using scanning electron microscopy (SEM, FEI, Model: Inspect-S50). The samples were attached to SEM stubs with a diameter of 10 mm using double-sided adhesive tape. Then the samples were coated with gold and viewed at 1000x, 5000x, and 20000x magnifications with a voltage of 20 kV.

3. Results and discussion

3.1. Synthesis of SiO₂ from Madura Strait sediment

The preparation of sediment samples involved a destruction process using a mixture of HCl and HNO₃/solutions through a reflux method. These acid solutions were used to remove impurities in the form of oxides, such as Na₂O, K₂O, and Ca₂O (Chu and Ko, 2018). Additionally, the reflux process facilitated oxygen enrichment in the sediment, optimizing SiO₂ formation.

Subsequently, the sediment was subjected to calcination at 800 °C. The high-temperature treatment enhanced crystallization in the sample, influenced by the release of water vapor. The bonding between sample particles was strengthened due to the transformation of Si-OH groups into Si-O-Si bonds through a dehydration process at elevated temperatures (Wang *et al.*, 1999). Furthermore, calcination led to pore shrinkage in the sample (Saikumari *et al.*, 2021). An increase in calcination temperature also resulted in a reduction in the specific surface area of SiO₂ (Wang *et al.*, 1999).

The sediment was purified using a strong acid treatment to remove impurities (Munasir *et al.*, 2015). The addition of HCl to the adsorbent facilitated the protonation of siloxyl groups (SiO⁻), converting them into silanol groups (Si-OH) (Ishmah *et al.*, 2020). The increase in proton (H⁺) concentration promoted the transformation of siloxyl groups into silanol groups, which subsequently reacted with other siloxy groups to form siloxane bonds (Si-O-Si). This process occurred repeatedly and rapidly, leading to the formation of amorphous silica. The purification process yielded 23.73 grams of SiO₂ from an initial 50-gram sediment sample.

3.2. Characterization of Madura Strait sediment and SiO₂

Table 1 presents the XRF analysis results comparing the composition of Madura Strait sediment (MSS-1) before and after purification (MSS-2).

As listed in **Table 1**, the sediment obtained from the Madura Strait before purification contained dominant elements, including silicon (33.50%), iron (30.10%), calcium (14.60%), and aluminum (11.00%). XRF analysis also indicated that Si and Fe were present in relatively high concentrations compared to other elements, giving the sediment a light gray appearance (**Fig. 1a**). After purification, the Si content increased significantly to 83.30%, while the Fe content drastically decreased to 5.96%. This increase in Si content led to a color change in the sample, resulting in a wheat-colored appearance (**Fig. 1b**).



Figure 1. Photos of Madura Strait sediment. (a) before purification; (b) after purification.

Source: Elaborated by the authors.

Table 1. XRF data analysis of the Madura Strait sediment.

Raw material	Atomic wight (%)								
	Al	Si	K	Ca	Mn	Fe	Cu	Mo	Others
(#1)	11.00	33.50	3.030	14.60	0.540	30.10	0.150	3.800	<0.01
(#2)	00.00	83.30	3.030	2.960	0.100	5.960	0.100	0.000	<0.01

Note: (#1) MSS-1 and (#2) MSS after purification.

Source: Elaborated by the authors.

Table 2. Semi-quantitative phase analysis results of XRD for MSS (**Fig. 2**).

Samples	Ref. Code	Compound name	Chemical formula	Quantification (based on RIR method)
MSS-1	01-078-1253	Quartz	SiO ₂	22%
	01-075-0650	Labradorite	Na ₄₈ Ca ₅₂ Al _{1.52} Si _{2.48} O ₈	61%
	00-005-0586	Calcium carbonate	CaCO ₃	5%
	01-087-1920	Iron aluminum silicon	Fe ₃ Al ₂ Si ₃	12%
MSS-2	01-078-1253	Quartz	SiO ₂	32%
	01-075-0650	Labradorite	Na ₄₈ Ca ₅₂ Al _{1.52} Si _{2.48} O ₈	50%
	00-005-0586	Calcium carbonate	CaCO ₃	9%
	01-087-1920	Iron aluminum silicon	Fe ₃ Al ₂ Si ₃	9%

Source: Elaborated by the authors.

Figure 2 presents the XRD diffractogram of MSS-1 and MSS-2. Further semi-quantitative analysis was performed using the Reference Intensity Ratio (RIR) method (Davis *et al.*, 1990), which determined the composition of the compounds present in MSS-1 and MSS-2, as summarized in **Table 2**.

A quantitative analysis was performed using the external standard method to quantify the degree of crystallinity. The results indicated that the crystallinity of MSS-2 was 70.3%. Additionally, the estimated crystallite size of MSS-2 was determined using the well-known Scherrer formula (Fatimah *et al.*, 2021), yielding a value of 42.9 nm.

FTIR spectral analysis confirmed the successful extraction of SiO₂ from Madura Strait sediment (**Fig. 3**). The characterization was performed within the wavenumber range of 400–4000 cm⁻¹. The observed silica absorption patterns primarily correspond to silanol (≡Si-OH) and siloxane (≡Si-O-Si≡) functional groups. The absorption band at 800.56 cm⁻¹ is attributed to the bending vibration of Si-O in siloxane (Dubey *et al.*, 2015). Peaks at 1049.28 and 1157.29 cm⁻¹ correspond to the stretching vibrations of Si-O in siloxane (Trivana *et al.*, 2015). Strong peaks at 1614.41 and 1631.77 cm⁻¹ indicate the stretching vibration of O-H in silanol (Trivana *et al.*, 2015). A broad peak in the range of 3000–3700 cm⁻¹ suggests the presence of O-H groups (Nandiyanto *et al.*, 2019), while the band around 3435.22 cm⁻¹ is associated with the hydrogen-bonded stretching vibration of Si-OH (Dubey *et al.*, 2015).

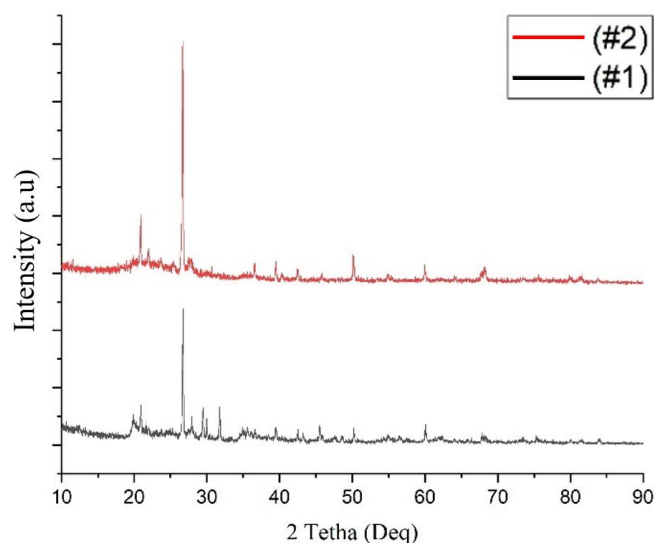


Figure 2. (#1) MSS-1 and (#2) MSS-2.

Source: Elaborated by the authors.

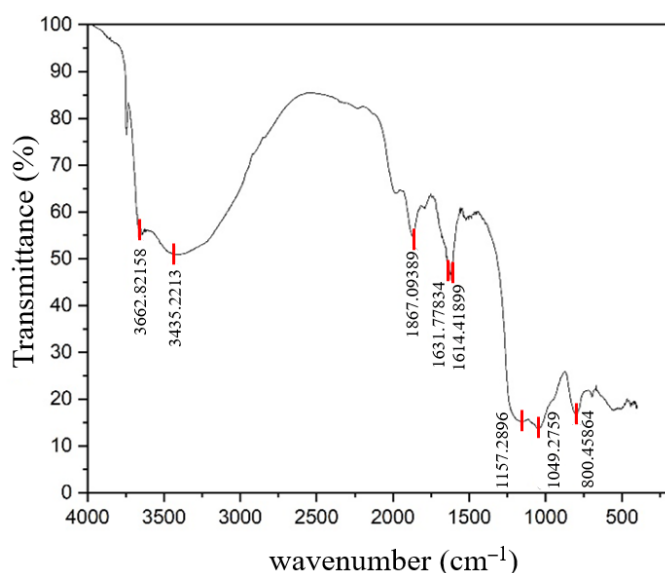


Figure 3. FTIR spectra of MMS after purification.

Source: Elaborated by the authors.

Field emission scanning electron microscopy (FE-SEM) was employed to analyze the morphology of the silica samples. The SEM results in **Fig. 4** reveal that the SiO₂ morphology consists of particle clumps, with individual particles appearing separated yet forming aggregates, leading to an irregular arrangement. Prolonged calcination at high temperatures contributes to an increase in particle size (Azlina *et al.*, 2016). When silica is calcined at an optimal temperature, the material undergoes diffusion, resulting in well-defined boundaries (Nandanwar *et al.*, 2015).

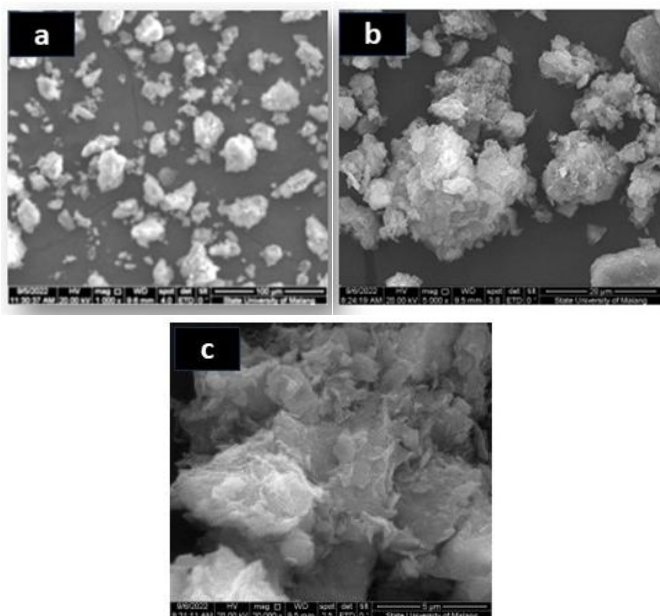


Figure 4. SEM images of SiO₂ powder, (a) 1.000x magnification; (b) 5.000x magnification and (c) 20.000x magnification.

Source: Elaborated by the authors.

4. Conclusions

SiO₂ has been successfully synthesized from Madura Strait sediment. The prepared sample was characterized by using XRF and FT-IR. Si content in the raw sediment increased from 33.5% to 83.3% after the removal of impurities. The XRD data analysis showed that the SiO₂ particles had a particle size of 42.94 nm and

crystallinity of 70.28%. FT-IR tests observed Si-O-Si and Si-OH groups in silanol and siloxane groups. The morphology of SiO₂ using SEM shows particle clumps; the particles appear separated, forming particle aggregates, which produce an irregular arrangement.

Authors' contribution

Conceptualization: Arif Hidayat; Anugrah Ricky Wijaya; Irma Kartika Kusumaningrum; **Data curation:** Arif Hidayat; **Formal Analysis:** Arif Hidayat; Anugrah Ricky Wijaya; **Funding acquisition:** Universitas Negeri Malang, Thesis Research [Grant No. 19.5.835/UN32.20.1/LT/2022]. **Investigation:** Arif Hidayat; **Methodology:** Arif Hidayat; Anugrah Ricky Wijaya; Irma Kartika Kusumaningrum; **Project administration:** Arif Hidayat; **Resources:** Anugrah Ricky Wijaya; Irma Kartika Kusumaningrum; **Software:** Arif Hidayat; **Supervision:** Anugrah Ricky Wijaya; Irma Kartika Kusumaningrum; **Validation:** Anugrah Ricky Wijaya; Irma Kartika Kusumaningrum; **Visualization:** Arif Hidayat; **Writing – original draft:** Arif Hidayat; **Writing – review & editing:** Anugrah Ricky Wijaya; Irma Kartika Kusumaningrum.

Conflict of interest

The authors declare that there is no conflict of interest.

Data availability statement

The data will be available upon request.

Artificial Intelligence usage statement

The authors declare that they did not use Artificial Intelligence tools at any stage of the preparation, correction, or evaluation of this work.

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