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Nanosilica Synthesis from Betung Bamboo Sticks and Leaves by Ultrasonication

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Introductions: Ultrasonication can be used to synthesize nanosilica from silica derived from betung bamboo sticks and leaves. This study aimed to synthesize nanosilica from betung bamboo sticks and leaves by the use of ultrasonication and to characterize the nanosilica produced.

Methods: The main materials used in this study were bamboo sticks and leaves. Betung bamboo sticks and leaves were sun-dried and then burned separately without adding fuel to produce charcoal. Then the produced charcoal was burned at a temperature of 700°C for 6 hours in a furnace to produce ash. Silica was extracted from furnace ash using reflux methods. The production of nanosilica from the silica derived from the betung bamboo sticks and leaves was carried out using ultrasonication.

Results: The yield of silica from sticks and leaves was based on ash dry weight 45.73% and 79.93%, respectively. The nanosilica derived from betung bamboo sticks had a particle size in the range of 169.87–1479.50 nm, with an average size of 502.35 nm and a particle dispersion index value of 0.1420. Nanosilica derived from betung bamboo leaves had a particle size in the range of 234.49–851.36 nm, with an average size of 472.67 nm and a particle dispersion index value of 0.0670. Scanning electron microscopy analysis showed that silica from betung bamboo sticks and leaves still agglomerated. The particle size of silica could minimize through ultrasonication to synthesize nanosilica.

Discussions: X-ray diffraction analysis showed that the structure of nanosilica differed from that of silica, and it appeared to be semicrystalline. The ultrasonication method for the synthesis of nanosilica derived from betung bamboo sticks and leaves ash can produce nanosilica that has a semicrystalline phase. The use of surfactants in the process can make the size of the nanosilica particles more uniform and reduce the size of the nanoparticles produced.

Keywords: betung bamboo, bamboo sticks, bamboo leaves, nanosilica, ultrasonication

Introduction

Bamboo plants are found in tropical regions in Asia, Africa, and the Americas, with some species also being found in Australia. However, Asia contains the largest area of bamboo distribution. From about 75 genera of bamboo comprising 1500 species throughout the world, 10 genera and 125 species occur in Indonesia.[1] Bamboo has many possible uses in Indonesia, and betung bamboo [Dendrocalamus asper (Schult. and Schult. f.) Backer ex. Heyne] in particular is widely used for building materials. Bamboo sticks are the most common parts used to make paper, handicrafts, and medicines, while other plant parts, such as roots, leaves, and branches, have not been fully utilized. Fatriasari and Hermiati[2] investigated the physical and

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chemical characteristics of betung bamboo, and one of their findings was that betung bamboo sticks have a silica content based on dry-weight reaching 3.51%. The high silica content in betung bamboo, especially in the sticks and leaves, makes it an optimal species for many uses. Based on the results of Ding et al[3] from an analysis of different parts of bamboo plants, the silica content ranged from 0.03% in roots to 9.95% in leaves.

Silica has a wide range of applications in various fields, and its utilization can be expanded by manipulating its characteristics. One such manipulation is to synthesize nanosilica from silica. Nanoparticles are solid particles with sizes of about 10–1000 nm.[4] Typical properties of nanoparticles are a large surface area, a large number of atoms on their surfaces, and high surface energy and surface tension. According to Fufa and Hovde[5] (2010), nano-SiO_2 has been used in wood modification either in their pure form or combination for enhancing fire performance, hydrophobicity, biocide, scratch and abrasion resistance. However, utilization of nano oxide increases environmental and health concern concerning nanomaterials. Due to these properties, nanoparticles are in great demand.[6]

One method that can be used to transform silica into nanoparticles (ie, nanosilica) is ultrasonication. The principle underlying ultrasonication is the phenomenon of acoustic cavitation, which occurs due to the propagation of sound vibrations through a medium. Cavitation involves the formation, growth, and rupture of bubbles that form in a liquid medium.[7] Ultrasonication has advantages over methods such as precipitation, including a relatively shorter production time and more homogeneous particle sizes.[8,9] Ismayana[10] showed that nanosilica particles could be obtained using a 2-hour ultrasonication method. Delmifiana and Astuti[10] achieved similar results using ultrasonication methods lasting 3–4 hours to synthesize magnetic nanoparticles that were relatively homogenous in size. Based on such outcomes, this study aimed to use ultrasonication to synthesize silica nanoparticles from betung bamboo sticks and leaves and subsequently characterize the resulting nanosilica.

Experimental

Materials

The main materials used in this study were bamboo sticks and leaves. Bamboo sticks and leaves were collected from several areas near the research location. Bamboo sticks are green with dark white spots. Bamboo sticks diameter is 6–9mm. Bamboo sticks thickness is 15–20mm. Bamboo leaves are green with a ribbon shape. The other materials used in this study were polyethylene glycol 6000, NaOH p.a., H_2SO_4 p.a., NH_4OH p.a., and demineral water.

The tools used in this study were furnaces, a reflux system, and an ultrasonicator. The equipment used for analysis included a particle size analyzer is used to characterize the size distribution of silica and nanosilica particles, an x-ray diffractometer (XRD) is used to analyze the structure of crystalline silica and nanosilica materials, and a scanning electron microscope (SEM) is used to study the morphology of the silica and nanosilica from bamboo sticks and leaves.

Methods

Burning Bamboo Sticks and Leaves

The process for burning the bamboo sticks and leaves was adapted from Sa'diyah et al,[12] whose study involved extracting silica from ampel bamboo leaves. Four kilos each of betung bamboo sticks and leaves were sun-dried and then burned separately in an open space without additional fuel for 1 hour. The charcoal produced from the bamboo sticks and leaves was then weighed with an analytical balance.

Furnace Ash Bamboo Sticks and Leaves

Charcoal produced from the bamboo sticks and leaves was placed into a porcelain cup and burned at a temperature of 700°C for 6 hours in a furnace to produce ash.[12] The ash yield was calculated by the following formula:

% Yield ash = [ash weight (g)/estimated oven-dried betung sticks or leaves (g)] × 100

Silica Extraction from Furnace Ash

Every ten grams of furnace bamboo sticks and leaves ash was refluxed in 80 mL of 3 N NaOH for 3 hours. The solution was filtered through filter paper, and the residue was washed using 50 mL of boiling demineral water. The filtrate was cooled to room temperature. Afterward, 5 N H_2SO_4 was added to the filtrate until the pH reached 2, and then 2.5 N NH_4OH was added to reach pH 8.5. This process was carried out on magnetic stirrer. The filtrate solution was left at room temperature for 30 minutes and distilled water was added until the solution reached neutral pH. It was then dried at 105°C for 12 hours.[13] After drying, silica originated from betung bamboo sticks and leaves were produced. And silica yield based on ash dry weight was calculated by equation, as follows:

% Silica yield = [silica weight (g)/ash dry weight (g)] × 100

Synthesis of Nanosilica Using Ultrasonication Method

The production of nanosilica from the silica derived from the betung bamboo sticks and leaves was carried out using ultrasonication. The method was performed using an ultrasonicator with a wavelength of 20 kHz and 130 W of power at 40% amplitude for 120 minutes. This method was adopted from previous study which utilized surfactant to disperse silica. The surfactant used in this study was polyethylene glycol (PEG) 6000 at a ratio of PEG 6000: silica (5:1).[11] The solution underwent an ultrasonication process in which the ultrasonicator was used to break silica particles into nanoparticles. After ultrasonication, the solution was dried at 103±2°C for 24 hours and then calcined in a furnace at 750°C for 3 hours.[9,11]

Characterization of the Nanosilica

Particle size analysis was performed using the particle size analyzer with 0.002 g of nanosilica dispersed in 100 mL of distilled water. The solution was stirred using a magnetic stirrer for 20 minutes, and particle scanning was carried out for 2–10 minutes. Surface morphological analysis of the nanoparticles was performed using SEM with magnification (until ×10,000), which was assisted by energy dispersive x-ray (EDX) analysis to determine the percentage of silica content. Analysis of both silica and nanosilica structures was carried out using XRD.

Results and Discussion

Nanosilica Production

The ash yield based on estimated oven-dried betung sticks or leaves was 10.41% (sticks) and 20.96% (leaves). Bamboo sticks and leaves ash produced silica yields based on ash dry weight were 45.73% and 79.93%, respectively. Silica was then synthesized into nanosilica through the addition of surfactant, followed by ultrasonication.

The synthesis of nanosilica in the current experiment used ultrasonication carried out for 2 hours. Previous studies have indicated that the duration of ultrasonication determines whether nanosilica will be produced. Ismayana[10] showed that ultrasonication needed to be 2 hours long for the formation of nanosilica particles from silica. Similar results were shown by Ismayana et al,[8] who synthesized nanosilica from industrial boiler ash with a sonication time of 2 hours.

Particle Size Analysis of Nanosilica

Silica derived from the bamboo sticks and leaves ash had an average particle size of ≥6000 nm. This size is quite large compared with the size of nanosilica. The nanosilica resulting from sticks ash had a particle size in the range of 169.87–1479.50 nm, with an average size of 502.35 nm, and a particle dispersion index (PDI) value of 0.1420. Furthermore, the nanosilica derived from bamboo leaves had a particle size in the range of 234.49–851.36 nm, with an average size of 472.67 nm, and a PDI value of 0.0670. The nanosilica from both sticks and leaves can be categorized as nanoparticles, which are particles under 1 μm in size.[5,14]

PDI values are indicators of both the level of dispersion and the particle size. Homogeneous dispersions have PDI values close to zero. With regard to particle size distribution, PDI values from 0.01 to 0.5–0.7 indicate homogeneous particles, while PDI values ≥0.7 indicate a wide and non-uniform (heterogeneous) size distribution. A low PDI value indicates the amount of mono dispersion in the solution. PDI can be measured by using dispersing media, and in the case of nanosilica, the dispersing medium is water.[15]

A nanosilica PDI value of 0.067 indicates that both the particle dispersion and size distribution are homogeneous. The uniformity in the nanosilica particle size is caused by the addition of a surfactant in the ultrasonication process. Our results are consistent with research conducted by Ismayana et al,[9] who reported that the lower the PDI value in the sample, the more uniform the particle distribution will be.

Analysis of SEM-EDX Silica and Nanosilica

SEM analysis at a magnification of until ×10,000 was conducted to obtain morphological data on the silica and nanosilica derived from betung bamboo sticks and leaves. Figure 1A shows the morphology of the silica from betung bamboo sticks, which appears dense and relatively large in size. In addition, silica particles are also more closely distributed resulting in agglomeration.

Figure 1B shows the morphology of nanosilica from betung bamboo sticks, with a more scattered distribution as evidenced by the low PDI value of 0.1420. This value indicates that the agglomeration process has been minimized by the addition of surfactants.[9] Similar results are shown for silica and nanosilica from betung bamboo leaves in Figure 2.

Figure 2A shows that the morphology of silica derived from betung bamboo leaves has denser appearance and the particle size is classified as large, which indicates agglomeration. In Figure 2B, nanosilica produced from the silica in

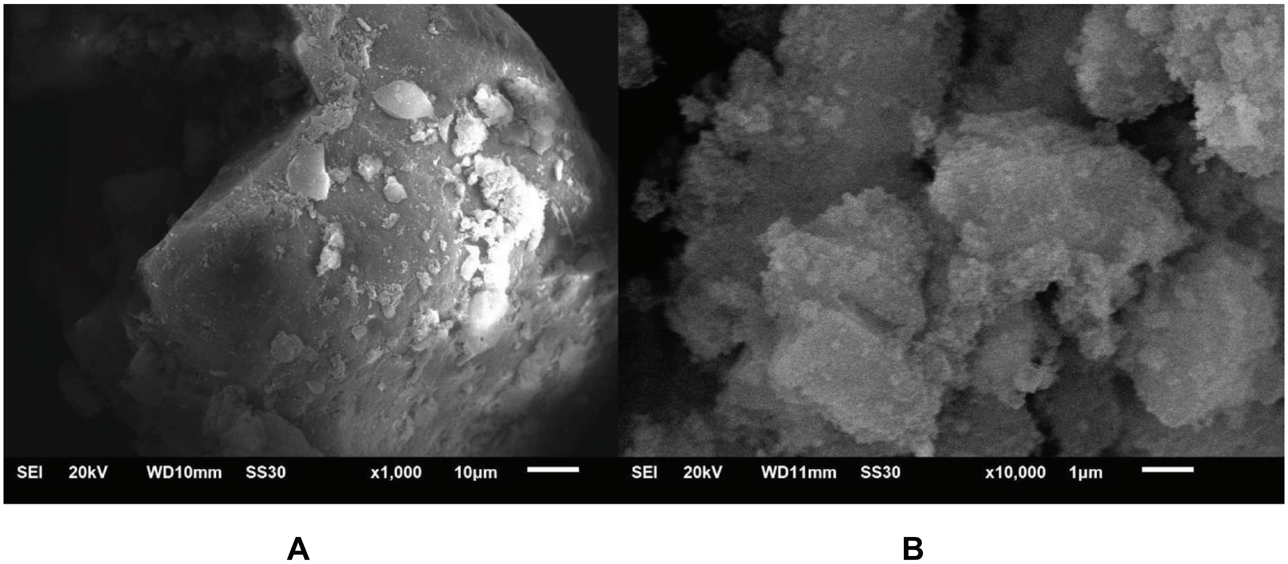

Figure 1 Morphology of (**A**) silica derived from betung bamboo sticks magnification x1000 and (**B**) nanosilica derived from betung bamboo sticks magnification ×10,000.
Abbreviations: SEI, secondary electron imaging; WD, work distance; SS30, spot size 30.

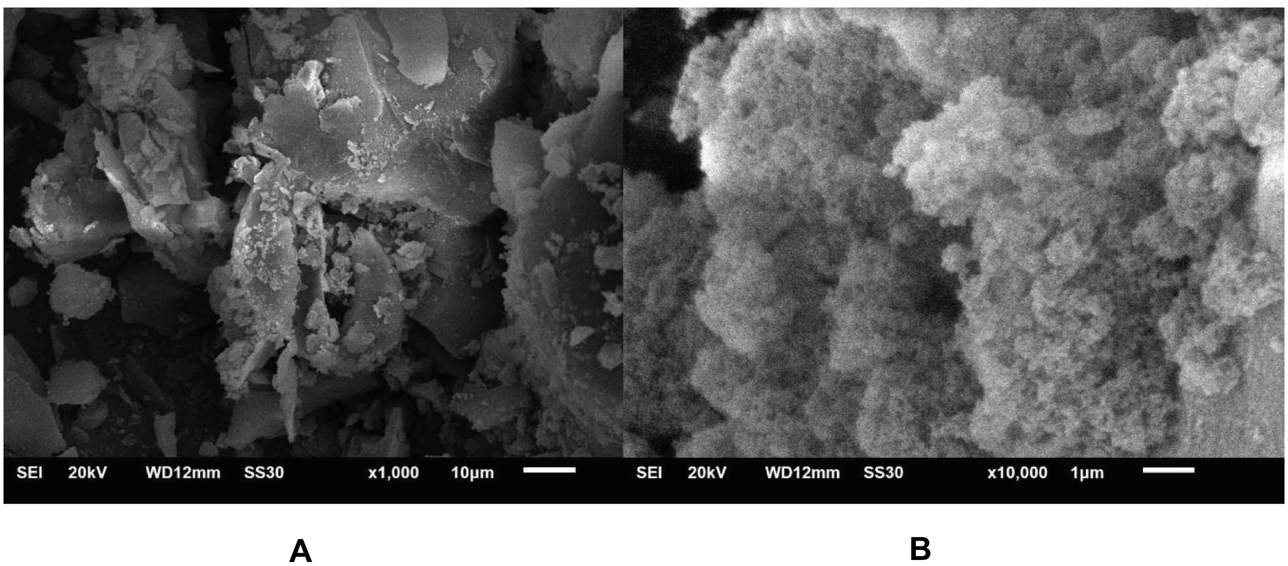

Figure 2 Morphology of (**A**) silica derived from betung bamboo leaves magnification x1000 and (**B**) nanosilica derived from betung bamboo leaves magnification ×10,000.
Abbreviations: SEI, secondary electron imaging; WD, work distance; SS30, spot size 30.

betung bamboo leaves shows the morphological changes compared with the silica. The nanosilica is distributed more widely as evidenced by the low PDI value of 0.067.

This result was likely influenced by the ultrasonication treatment used on the silica from bamboo sticks and leaves. According to Ismayana,[10] the process of nanosilica synthesis through ultrasonication results in a more orderly arrangement of crystals because the method can create heat. This heat can encourage the nanosilica crystals to become more organized.

Additional information obtained from the SEM analysis was the EDX data, which revealed the elements contained in the sample. The results indicate the presence of various elements in the sample, including oxygen, silicon, and gold. Oxygen and silicon are the elements that constitute silica, while gold was the coating material for the silica and nanosilica samples. The results of the EDX analysis are presented in Table 1.

Analysis of XRD Silica and Nanosilica

The XRD patterns from silica and nanosilica derived from bamboo sticks and leaves are characterized by diffraction peaks between angles of $2\theta = 5°–40°$. As shown in Figure 3,

Table 1 Chemical Composition of Silica and Nanosilica Samples

Element	Atomic Percentage (%)			
	Betung Bamboo Sticks		Betung Bamboo Leaves	
	Silica	Nanosilica	Silica	Nanosilica
Oxygen (O)	55.11	49.04	33.71	41.51
Silicon (Si)	42.84	49.15	60.14	52.05
Gold (Au)	2.05	1.81	6.15	6.44

the diffraction patterns for silica from bamboo sticks did not show sharp peaks. This result indicates that the silica produced has an amorphous phase. The XRD pattern of silica from sticks has a greater intensity at an angle of 2θ = 21.96°, which corresponds to the peak of silica in the JCPDS (Joint Committee of Powder Diffraction Standard) database 44–1934 (ie, angle 2θ = 21.93°). The nanosilica diffraction patterns appeared to have a high intensity at an angle of 2θ = 22.58°, which corresponds to the peak of silica in the JCPDS database 44–1394 (ie, angle 2θ = 22.49°). These silica and nanosilica peaks widen between 2θ = 20°–25° angles. In the nanosilica diffraction pattern, a new peak with high intensity appears at an angle of 2θ = 26.58°, which indicates that the nanosilica produced has a semicrystalline phase.

Figure 4 shows the diffraction pattern of silica with no visible sharp peaks. This shows that the silica produced has an amorphous phase. The XRD patterns of silica and nanosilica derived from bamboo leaves are, respectively, characterized by the appearance of high-intensity diffraction peaks at an angle of 2θ = 20.78°, 20.86°. This peak widens between angles 2θ = 20°–25°. In the nanosilica diffraction pattern, a new peak appears at an angle of 2θ = 25.00°, which shows that the nanosilica produced has a semicrystalline phase.

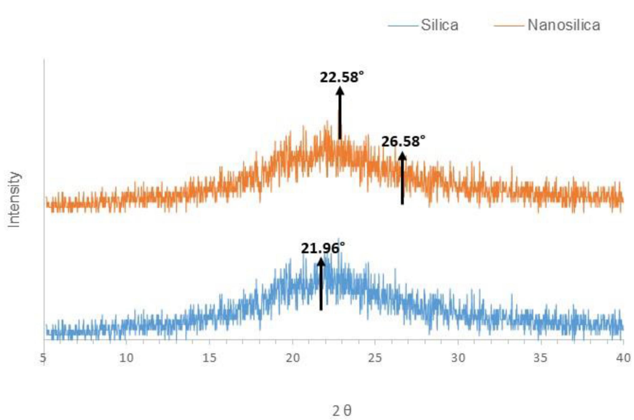

Figure 3 The structural properties of silica and nanosilica derived from betung bamboo sticks.

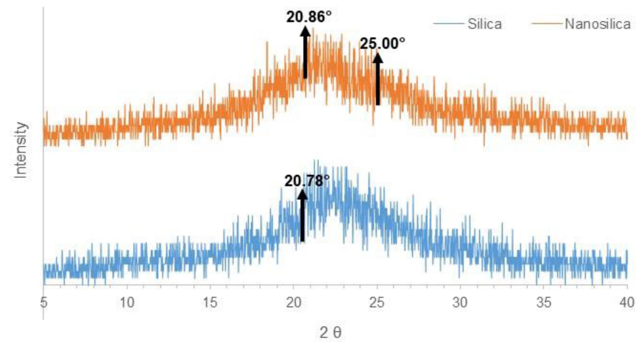

Figure 4 The structural properties of silica and nanosilica derived from betung bamboo leaves.

The semicrystalline phase of nanosilica derived from sticks and leaves is thought to have been influenced by the ultrasonication treatment. The ultrasonication process results in the silica particles becoming more crystalline because the process only cuts large crystals without breaking the structure of their chemical bonds.

Conclusions

The ultrasonication method for the synthesis of nanosilica derived from betung bamboo sticks and leaves ash can produce nanosilica that has a semicrystalline phase. The use of surfactants in the process can make the size of the nanosilica particles more uniform because it can prevent the agglomeration of particles. In addition, the addition of surfactants reduces the size of the nanoparticles produced.

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Disclosure

The authors report no conflicts of interest for this work.

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