

# Green Synthesis and Formation of Silica Nanoparticles Based on Natural *Spondias mombin* Leaf Extract for Renewable Energy Materials

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## ABSTRACT

Silica is an abundantly available material, with applications including renewable energy materials (fuel cells). Silica has been produced using natural materials (green synthesis) to form smaller and more stable particles, primarily due to the influence of secondary metabolites such as those contained in the leaf of *Spondias mombin*, a species of flowering plants from *Lannea coromandelica* (Houtt.) Merr and the family *Anacardiaceae*. The characteristics of silica nanoparticles obtained by green synthesis using the *Spondias mombin* leaf in a water fraction at 0,6 % were identified using XRD, FTIR, PSA, TGA, SEM, and TEM. Based on the XRD pattern and SEM/TEM images, the nanoparticle is amorphous and has a particle size of 20-30 nm, smaller than that of its precursor, which is 37.6 nm. There are Si-OH and Si-O-Si groups in the FTIR spectrum as an indication of the interaction between silica nanoparticles and secondary metabolites, and the zeta potential based on PSA is -45.9 mV. This indicates that silica nanoparticles have good stability and are supported by high thermal stability based on the results of the TGA analysis. This is correlated with its use as a material composite in renewable energy (fuel cells).

**Keywords:** green synthesis, silica nanoparticles, *Spondias mombin*

## 1. Introduction

Green synthesis is a synthesis method that is more environmentally friendly than chemical synthesis because it involves various inorganic materials like metals or metal oxides that involve natural materials (plants, bacteria, algae, or fungi) and do not cause pollution or hazardous effects. Natural ingredients in the form of extracts are used in the synthesis using the green method. This functioned as a source base (O-H) or an agent reducer because of the presence of metabolites secondary to alkaloids. Besides that, the existence of other secondary metabolites, such as saponins and polyphenols, can also play a role as a capping agent to stabilize the particle structure [1], [2]. Thus, materials particles obtained with the green synthesis method have a smaller, finer, more stable, and homogeneous size particle through controlling parameters such as concentration, pH, time, and

temperature during heating, as well as calcination at the end of synthesis [3]. Several studies related to nanoparticle synthesis use the green synthesis method, including Prabha et al. [4], which used the leaf extract of *Rosa rugose* of gold nanoparticles; Tajbakhsh et al. [5], which used the *Euphorbia heterophylla* of Ag/HZSM5-5 nanocomposite; and Jabeen et al. [6], which used *Azadirachta indica* (Neem) of silica nanoparticles. Green synthesis using leaf extracts in the formation of silica nanoparticles has also been widely conducted by Babu et al. [7], Sethy et al. [8], Durairaj et al. [9] and Azawi et al. [10], who used *Cynodon dactylon*, bamboo, *Bambusa vulgaris*, and *Thuja orientalis*, respectively.

Silica has unique physical and chemical properties, including small size, high surface energy, high scattering performance, and high thermal resistance [11], [12]. Silica has many applications in drug delivery, biosensing, catalysts, and energy storage [13],

including as a composite in proton exchange membranes (PEM), which can help increase proton conductivity in renewable energy (fuel cells). Meanwhile, Njoku et al. [14] stated that *Spondias mombin* leaves contain secondary metabolites. These include tannins, saponins, alkaloids, flavonoids, and phenols in quantities as large as 3.82%, 7.60%, 6.00%, 3%, and 1%, respectively. These plants can be involved in green synthesis methods for materials such as silica.

One study on the PEM performance of a mixed matrix composite comprising oxy-polybenzimidazole and imidazole ionic liquid modified silica nanoparticles showed a proton conductivity value of  $0.219 \text{ S.cm}^{-1}$  [15]. Based on these matters, the characteristics of the silica nanoparticles resulting from the green synthesis based on natural *Spondias mombin* are expected to improve their function as material composites in the PEM of fuel cells.

## 2. Methods

### 2.1. Equipment/Tool/Material

The equipment/tools used in this research include: glassware, hotplate, magnetic stirrer, analytical balance, rotary evaporator, oven, porcelain cup, tanur, desiccator, furnace, Thermo Scientific Nicolet iS50 FTIR, HORIBA SZ 100z Nano Particle Size Analyzer, PAnalytical X'pert Pro Diffractometer, scanning electron microscope Jeol-JSM 6063-LA, thermal gravimetric analysis SHIMADZU DTG-60, and TEM Talos F200X.

The materials used in this research include: *Spondias mombin* leaf obtained from IPB's Biopharmaca Plantation, n-hexane pa (purchased from Merck), methanol pa (purchased from Merck), distilled water, Whatman 42 filter paper, tetraethyl orthosilicate (TEOS), HCl, Wagner reagent, NaOH, H<sub>2</sub>SO<sub>4</sub>, FeCl<sub>3</sub>, and acetic anhydride.

### 2.2. Experiment

#### 2.2.1. Preparation and Extraction of *Spondias mombin* Leaf

In total, 5-kg *Spondias mombin* leaves were washed and dried. The dried leaves were crushed using a blender to form a fine powder. The extraction process is conducted in two stages, maceration and fractionation. In the maceration step, initially, 50 g of

*Spondias mombin* powdered leaves were placed in a beaker, then macerated with a methanol solvent of 250 mL (ratio of leaf powder and solvent 1:5) for 7 x 24 hours, stirring for 20 minutes daily. Then, the mixture was filtered, and the filtrate was separated from the residue to obtain a methanol filtrate of *Spondias mombin* leaves, which was tested for its phytochemicals. In the next step (fractionation), the methanol filtrate of *Spondias mombin* leaf was partitioned with n-hexane (1:1). The obtained results were in the form of two fractions, namely, the methanol fraction and the n-hexane fraction. Both fractions were subjected to phytochemical tests. Then, the partitioned methanol fraction was evaporated with a rotary vacuum evaporator at 55°C until the solvent was used up. The results are reported as a methanol fraction. A total of 100 mL of water (aquadest) was added. The solution stock extract of the *Spondias mombin* fraction water was also prepared and analyzed by FTIR.

#### 2.2.2. Phytochemicals of *Spondias mombin* leaf extract (SMLE) Test

Qualitative phytochemical tests were performed on SMLE to identify active compounds, including flavonoids, alkaloids, terpenoids, steroids, polyphenols, tannins, and saponins.

##### 2.2.2.1. Alkaloids Test

For alkaloid identification, 1 mL of SMLE was mixed with 4 mL of distilled water, 3 mL of 2 N HCl, and three drops of Wagner's reagent. The formation of a red-brown color indicates the presence of alkaloids.

##### 2.2.2.2. Flavonoids Test

For flavonoid identification, 1.0 mL of SMLE was mixed with 5.0 mL of distilled water, followed by 1.5 mL NaOH and a few drops of 4 M H<sub>2</sub>SO<sub>4</sub>. The formation of a yellow color with NaOH that faded after adding H<sub>2</sub>SO<sub>4</sub> indicated the presence of flavonoids.

##### 2.2.2.3. Polyphenols Test

In the polyphenol identification test: 1.0 mL of SMLE was added to a test tube containing 5.0 mL of distilled water and 3 drops of FeCl<sub>3</sub>. The dark green color indicates the presence of polyphenol compounds.

#### 2.2.2.4. Tannins Test

In the tannin identification test, as much as 0.5 mL of leaf extract of *Spondias mombin*, 1.0 mL of distilled water, and 2 drops of FeCl<sub>3</sub> were added. The formation of a blackish-blue color indicates tannins. Then, 1.0 mL of distilled water and 2 drops of FeCl<sub>3</sub> were added.

#### 2.2.2.5. Saponins Test

In the saponin identification test, 0.5 mL leaf extract of *Spondias mombin* was added to 3.0 mL aquadest and heated in a water bath. Foam formation, which is stable for 30 minutes after shaking, indicates the presence of saponins.

#### 2.2.2.6. Terpenoids and Steroids Test

For steroid and terpenoid identification, 1 mL of *Spondias mombin* leaf extract was mixed with 0.5 mL acetic anhydride, 0.5 mL chloroform, and concentrated sulfuric acid added dropwise. A purplish-red color indicates terpenoids, while a bluish-green color indicates steroids.

#### 2.2.3. Green Synthesis and Formation of Silica Nanoparticles

A total of 5.0 mL of 0.03 M tetraethyl orthosilicate (TEOS) as a precursor was added to the extract of *Spondias mombin* leaf (SMLE) fraction water (0.6%) with a precursor: SMLE volume ratio of 1:5 (SiO<sub>2</sub>.NP1) and 5:1 (SiO<sub>2</sub>.NP5). Treatment with two ratios was carried out to determine the effectiveness of SMLE in stabilizing the formation of silica nanoparticles. The mixture was then stirred at 80 °C for 4 h. The aging process was performed for 24 hours to form a perfect sol. The formed sol was then air-dried in an oven at 120 °C for 6 h until a gel formed. After that, the gel was calcined at 600 °C for 12 h, and then the powder was characterized by FTIR, XRD, PSA, SEM/TEM, and TGA.

### 3. Results and Discussion

#### 3.1. Properties of the Phytochemicals of *Spondias mombin*

*Spondias mombin* plants (Figure 1) are a species of flowering plants native to tropical America, including the West Indies, and were introduced to

Southeast Asia by the Portuguese in the early 17<sup>th</sup> century. They later expanded to parts of Africa, India, Nepal, Bangladesh, Sri Lanka, and Indonesia. The results show that the *Spondias mombin* leaves used in this research were *Lannea coromandelica* (Houtt.) Merr species and the *Anacardiaceae* family.



Figure 1. *Spondias mombin* Plants

Methanol extract, n-hexane fraction, and water fraction of *Spondias mombin* leaf were subjected to phytochemical tests to determine which secondary metabolites were found in the leaf extract in each fraction obtained. The phytochemicals of all fractions indicated the presence of secondary metabolites, including alkaloids, polyphenols, tannins, saponins, flavonoids, terpenoids, and steroids (Table 1).

Table 1. Phytochemical Test Results for Methanol Fraction, Water Fraction, and n-Hexane Fraction of *Spondias mombin* Leaf Extract

Secondary metabolite content	Identification results
Alkaloids*	Red-brown color (+)
Polyphenols*	Dark green color (+)
Tannins*	Dark blue color (+)
Saponins*	Foam formation (+)
Flavonoids*	Yellow color after the addition of NaOH, and the yellow color fades after the addition of H <sub>2</sub> SO <sub>4</sub> 4 M (+)
Terpenoids*	Red-purple color (+)
Steroids**	Green-blue color (+)

Note:

\*methanol and water fraction,

\*\*n-hexane and methanol fraction

Meanwhile, the results of quantitative analysis of the crude extract of the water fraction of *Spondias mombin* leaf showed total flavonoid, polyphenol, and tannin contents of 0.45%, 28.62%, and 0.95%, respectively.

### 3.2. Characteristics of SMLE and Silica Nanoparticles (SiO<sub>2</sub>.NP) Based on FTIR

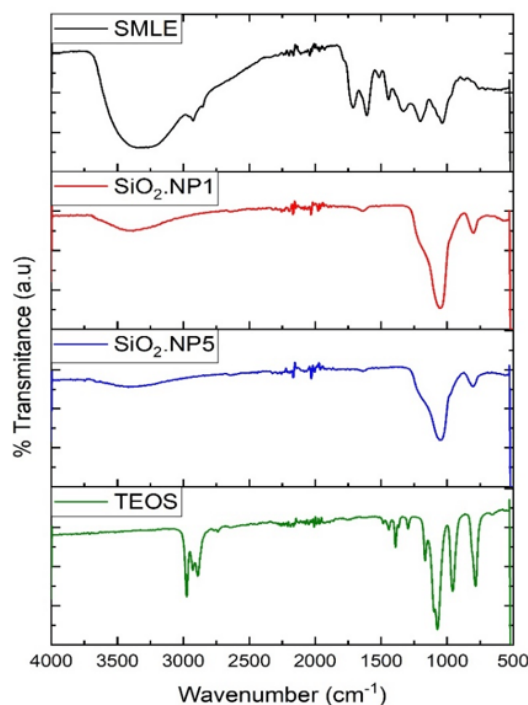
The SMLE water fraction and the green-synthesized silica nanoparticles were characterized by FTIR spectroscopy to analyze the functional groups of the compounds present in SMLE involved in the nanoparticles' synthesis. The results show the presence of functional groups at 3344 cm<sup>-1</sup> (O-H stretching), 2925 and 2854 cm<sup>-1</sup> (C-H stretching), 1713 cm<sup>-1</sup> (C=O stretching), 1443 and 1330 cm<sup>-1</sup> (C-N and C-O), and 1202 and 1039 cm<sup>-1</sup> (C-O) (Table 2). The functional groups found in SMLE are derived from secondary metabolites. After being compared with the results of the phytochemical test and quantitative analysis, it can be said that the group function of O-H stretching, C-H stretching, C=O stretching, C-N stretching, and C-O stretching present in SMLE originates from flavonoids and polyphenols, saponins, alkaloids, and derivatives from tannin compounds. The free electron pair on the O atom of the carbonyl group acts as a capping agent on phenolic compounds, which stabilizes the formation of nanoparticles [2]. Similarly, saponin compounds can also act as capping agents to control nanoparticle growth, while alkaloid compounds are a source of weak bases for the green synthesis of nanoparticles [1].

Silica (SiO<sub>2</sub>) obtained was characterized using FTIR to analyze the functional groups of secondary metabolites in the SMLE water fraction, which play a role in the synthesis of SiO<sub>2</sub>. Furthermore, the FTIR spectrum of SiO<sub>2</sub> at a ratio of 1:5 (SiO<sub>2</sub>.NP1) and 5:1 (SiO<sub>2</sub>.NP5) was compared with the FTIR spectrum of the SMLE water fraction. This was done to determine the interaction between functional groups of secondary metabolites and precursor solutions (TEOS) in the synthesis of SiO<sub>2</sub> through a shift in the wavenumber of the functional groups in the SMLE water fraction (Figure 2). The results of the analysis show that there is a shift in the wavenumber of the water fraction SMLE before and after it is used in the synthesis of SiO<sub>2</sub> in both ratios. The shift that happens on the -OH group stretching at wavenumbers 3344 cm<sup>-1</sup> to 3383 cm<sup>-1</sup> (SiO<sub>2</sub>.NP1) and 3414, 3643, 3746 cm<sup>-1</sup> (SiO<sub>2</sub>.NP5) indicates the effect of the presence of Si-OH and Si-O-Si at wavenumber 1050-1051 cm<sup>-1</sup>. Silica nanoparticles are formed through the hydrolysis of TEOS, then condensation between silanol groups or between

silanol groups and ethoxy groups produces siloxane groups [16]. These two functional groups or chemical compounds are also found in silica nanoparticles using Coconut Husk Ash [17].

**Table 2.** Wavenumber FTIR Spectrum of Literature and SMLE

Functional groups	Wavenumber (cm <sup>-1</sup> )	
	Literature [18]	SMLE
O-H	3550-3200	3344
C-H	3000-2850	2925, 2854
C=O	1780-1630	1713
C-N	1450-1280	1443, 1330
C-O	1200-1020	1202, 1039



**Figure 2.** FTIR Spectrum of SMLE, SiO<sub>2</sub>.NP1, SiO<sub>2</sub>.NP5, and TEOS

### 3.3. Pattern of Silica Nanoparticles Based on XRD

Characterization of SiO<sub>2</sub> by XRD was carried out to determine its crystallinity by comparing the 2θ diffraction angle of the observed peak with JCPDS data. Based on the results of the analysis, no sharp peaks were found in the presence of crystals, but a wide

peak with a  $2\theta$  angle of  $22.65^\circ$  and  $23.07^\circ$  (ICSD collection code: 164161) was observed for  $\text{SiO}_2\text{.NP1}$  and  $\text{SiO}_2\text{.NP5}$ , respectively. This indicates that the  $\text{SiO}_2$  obtained has an amorphous structure (Figure 3) [19]. A wide peak with a  $2\theta$  angle of  $22.01^\circ$  was also produced by silica nanoparticles using *Bryophyllum Pinnatum* leaf extract [20].

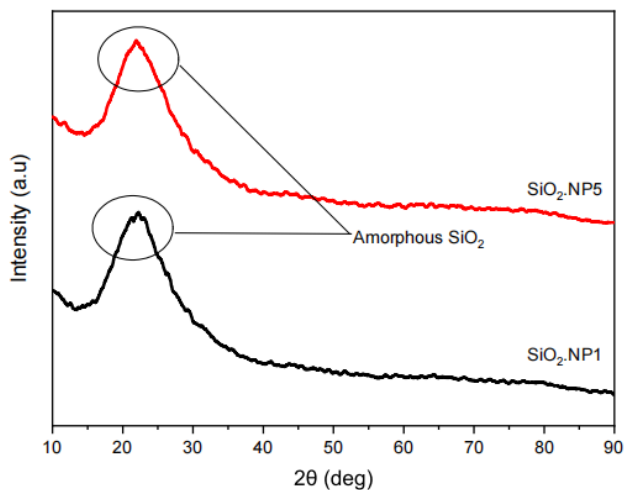


Figure 3. XRD Diffraction Pattern of  $\text{SiO}_2\text{.NP1}$  and  $\text{SiO}_2\text{.NP5}$

### 3.4. Morphology and Stability of Silica Nanoparticles

Silica nanoparticles were characterized using SEM and TEM to determine their morphology, particle size, and distribution of elemental composition. The STEM bright field (BF) image shows an agglomeration of the silica nanoparticles in some spots, likely due to the hygroscopic properties of silica. This condition is indicated by the polydispersity index (PI or PDI) of  $\text{SiO}_2$  nanoparticles, which is higher than that of its precursor (TEOS), which are 0.609 and 0.347, respectively. The polydispersity index is a measure of the particle size distribution in a solution. A solution is classified as homogeneous if it has a PI value close to zero, while a solution is categorized as non-homogeneous if the PI is close to 1 [21]. Based on the PI, the  $\text{SiO}_2$  size distribution is non-homogeneous in the SMLE produced in this research. Nevertheless, the results indicate that the silica nanoparticles have a particle size of 20-30 nm. The particle size of  $\text{SiO}_2$  produced by green synthesis is smaller than the particle size of its precursor ( $\text{SiO}_2$  without SMLE) at 37.6 nm.

In previous research, silica nanoparticles mediated in *Bryophyllum Pinnatum* leaf extract showed a particle size of 24 nm.

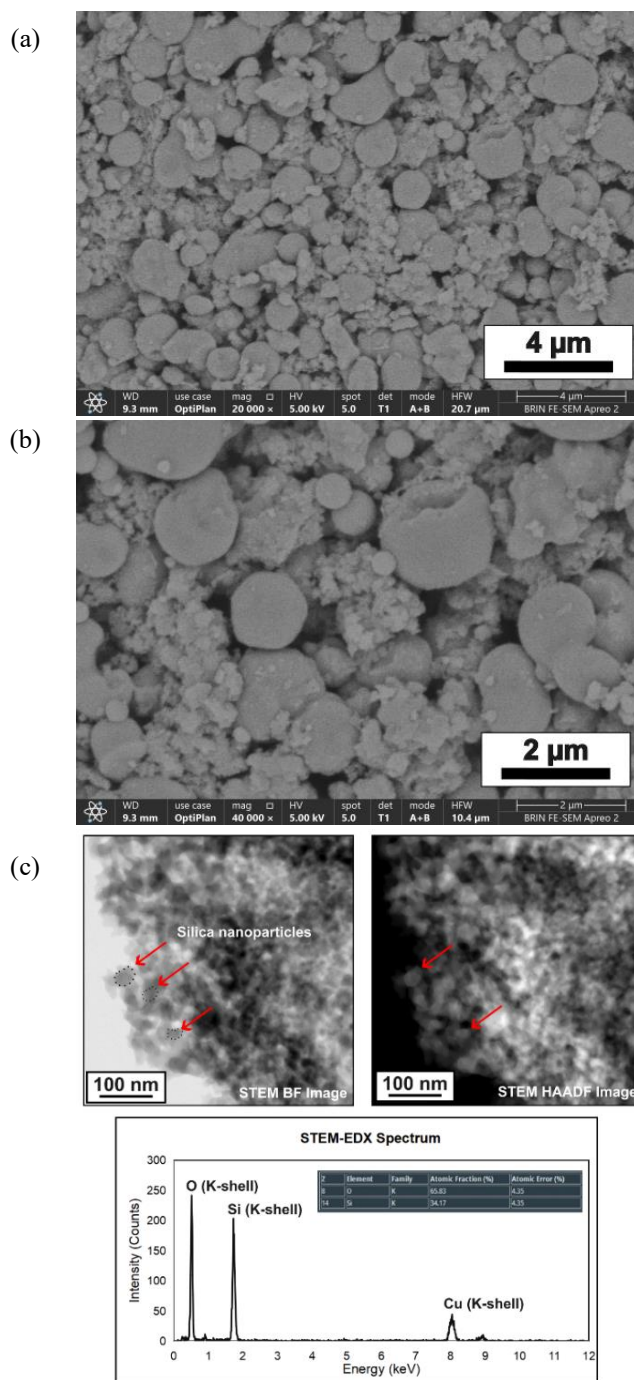


Figure 4. SEM image of the surface (a) x 20000 and (b) x 40000; (c) TEM image and EDX spectrum of  $\text{SiO}_2\text{.NP1}$

For elemental analysis, a combination of STEM-HAADF imaging and EDX analysis was performed. STEM-HAADF and semiquantitative EDX analysis

revealed that the nanoparticles contain silicon (65.83 at%) and oxygen (34.17 at%), indicating a silica compound. Copper (Cu) was detected from the copper TEM grid.

Meanwhile, the resulting zeta potential for SiO<sub>2</sub>.NP1 and SiO<sub>2</sub>.NP5 was -45.9 mV and -24.3 mV, respectively, indicating an increase in the nanoparticles' stability relative to TEOS (-15.1 mV). Zeta potential analysis is a traditional method for determining the stability of nanomaterials [20]. This result indicates that the greater the SMLE in the mixture with the precursor (TEOS), the more stable the formation of silica nanoparticles. In the word, formation of SiO<sub>2</sub> nanoparticles with a precursor: SMLE ratio of 1:5 (SiO<sub>2</sub>.NP1) is more stable than 5:1 (SiO<sub>2</sub>.NP5). The zeta potential of SiO<sub>2</sub>.NP1 is higher than the silica nanoparticles from *Bryophyllum Pinnatum* leaf extract at -32 mV. A good zeta potential is in the range of ± 31-60 mV. The more positive or negative the charge, the more stable the particles/nanoparticles obtained [22] As a result of increasing electrostatic repulsion between particles and vice versa, a low zeta potential indicates aggregation of nanomaterials [12]. Thus, it can be said that green synthesis using *Spondias mombin* leaf extract (SMLE) increased the stability of silica nanoparticle formation at a precursor: SMLE ratio of 1:5 in a 0.6% water fraction (SiO<sub>2</sub>.NP1).

### 3.5. Thermal Stability of Silica Nanoparticles

The thermal stability of a material can be tested by TGA. Based on observations, silica nanoparticles, both SiO<sub>2</sub>.NP1 and SiO<sub>2</sub>.NP5, respectively, decreased in total weight by 6.43% (145.04 °C) and 4.11% (151.66 °C) due to a loss of surface water. silica nanoparticles and the values of the two were not significantly different, inversely proportional to the volume of the extract (Figure 5).

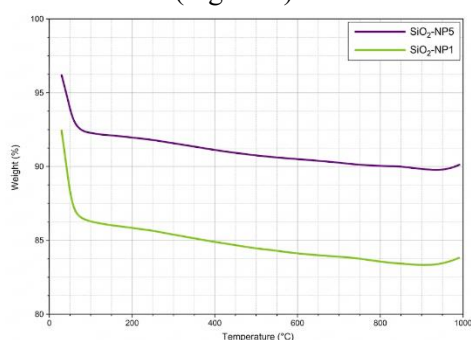


Figure 5. Thermogram of SiO<sub>2</sub>.NP1 and SiO<sub>2</sub>.NP5

This indicates that the low volume effect of extracts of natural materials in the synthesis of silica nanoparticles decreases the thermal stability of silica because silica is composed at temperatures above 600 °C [23]. In other words, the thermal stability of silica nanoparticles synthesized via the green method using SMLE is high.

## 4. Conclusion

Silica Nanoparticles based on natural *Spondias mombin* leaf extract with secondary metabolites, which include tannins, saponins, alkaloids, flavonoids, terpenoids, and polyphenols in fraction water 0.6%, were successfully synthesized through the green synthesis method at a ratio of 1:5 of tetraethyl orthosilicate precursors: *Spondias mombin* leaf extract. This was identified by characterization using XRD, FTIR, PSA, SEM/TEM and TGA which showed formation silica nanoparticles with an amorphous structure and the particle size of 20-30 nm, the presence of silanol and siloxane groups, and high particle stability (-45.9 mV) and good thermal stability, so they are suitable for use in applications such as material composite of renewable energy (fuel cells).

## CRedit Authorship Contribution Statement

Armi Wulanawati: Data curation, Conceptualization, Software, Visualization, Investigation, Data analysis and/or interpretation, Characterization, Writing-Original Draft Preparation, and Funding acquisition. Yoki Yulizar: Supervision, Conceptualization, Validation, Methodology, Critical Revision, Approval of Final Version of Manuscript. Sri Mulijani: Supervision, Validation, Methodology, Resources, Writing-Reviewing, and Editing.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available upon reasonable request.

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## Declaration of Generative AI and AI-Assisted Technologies in the Writing Process

During the preparation of this manuscript, the author used generative AI tools to assist in language editing and improving the clarity of the text. The author reviewed and edited the content as necessary and takes full responsibility for the accuracy, integrity, and originality of the manuscript.

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