

Synthesis and characteristics of Silica nanoparticles (SiO_2NPs) from Bamboo Leaves Ash by precipitation method

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ABSTRACT

This study illustrates the low-cost, eco-friendly synthesis of silica nanoparticles using local bamboo leaves collected in August 2024 from the Aqqan Valley in the Al-Masimir District of Lahij Governorate, Yemen. Nanosilica powder synthesis by precipitation method is generated subsequent to thermal treatment of BLA at 750°C followed by characterization using advanced characterization techniques. The absorption peaks close to $\lambda_{\text{max}} = 240 \text{ nm}$ in the UV spectrum showed silica nanoparticles, which were observed using UV-Vis spectroscopy. The presence of a siloxane group (Si-O-Si) in the experimental FT-IR spectral data indicated the high purity of the nanosilica particles. The prepared silica showed a high purity of 97.82% using XRF. The particle size determined by transmission electron microscopy (TEM) was found to be between 10.79 nm and 22.73 nm with minor agglomerations. Analysis of the nanosilica by X-ray diffraction (XRD) confirmed an amorphous structure with a peak intensity at $2\theta=23^\circ$.

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1. INTRODUCTION

Bamboo is one of the fastest-growing and highest-yielding natural resources. Its leaf ash (BLA), which contains over 70% amorphous silica and has a high surface area, serves as a rich silica source suitable for various industrial applications [1]. Bamboo leaves are treated as waste, as countries that have an abundance of bamboo use them mainly for pulp and construction [2].

Amorphous silica nanoparticles have recently become increasingly popular because of their distinctive properties, including high compatibility and extensive surface area, and remarkable chemical and thermal stability, making them suitable for various potential applications, such as drug delivery, biosensing, surface coatings, and abrasives [3]. Silica is widely employed in the manu-

facturing, adsorption, packaging, agriculture, and many other fields. It has wide applications in various industries, including foundries, cement manufacturing, glassmaking, and the chemical industry [4].

Silicon dioxide (SiO_2), commonly known as silica, is an inorganic compound with widespread natural and industrial applications, including its presence as flint, quartz, and sand [5]. Various techniques, such as chemical precipitation, solvent extraction, ion exchange, and electrolytic deposition can be used to extract nanosilica from agricultural waste [6]. Studies on the manufacture of silica from various agricultural wastes, including corn cobs [7], maize husks [8], rice husks [9], sugarcane bagasse [10] and bamboo stalks [11], have been published. Moreover, few studies have been conducted on

the generation of silica from bamboo leaves. Therefore, this study presents the production of nanosilica from bamboo leaves using the precipitation method and the characterization of the as-precipitated nanosilica. This study aids in green waste disposal and contributes to the circular economy's sustainable development goals.

2. MATERIALS AND METHODS

2.1. MATERIALS

Bamboo leaves (Figure 1) tacked from the Aqqan Valley, Al-Masimir District, Lahij Governorate, Yemen, were used as a source of silica for the synthesis of nanosilica particles. The chemical materials used were sodium hydroxide (NaOH) and, hydrochloric acid (37% HCl) from Germany-PanReac AppliChem, and sulfuric acid (98% H₂SO₄) from India Thomas Baker.



Figure 1. Bamboo Tree and its leaves

2.2. METHODS

The collection of bamboo leaves and the synthesis of silica dioxide nanoparticles (SiO₂NPs) were carried out with slight modifications to the methods reported in [9, 12].

2.2.1. Preparation of Bamboo Leaves Ash (BLA)

The collected bamboo leaves have been washed thoroughly with distilled water to remove dust and debris and allowed to dry for 2 days at 60 °C in an oven to remove moisture. The dried leaves were ground in a grinder and the powder was heated in a muffle furnace at 750 °C for 4 hours to obtain ash, which was then collected and milled to obtain fine particles.

2.2.2. Nanosilica Powder Synthesis Method

Bamboo Leaves The (BLA) thus obtained were treated with 7% HCl at 60 °C with continuous stirring for 30 min to remove the metal impurities. Subsequently, the acid was removed from the BLA by filtration through a filter

paper (No.1) and washed with hot distilled water several times. In a beaker, BLA was mixed with sodium hydroxide solution (NaOH 2.5M) and heated at 80 °C for 3 h with magnetic stirring to form sodium silicate Na₂SiO₃. Subsequently, concentrated sulfuric acid H₂SO₄ was added dropwise with continuous stirring to form a silica gel. The silica gel contained water (moisture) and some impurities; therefore, the beaker was placed in an oven at 100 °C for 12 h for drying. After drying, the precipitate was thoroughly washed with hot distilled water to remove residual impurities. The resulting product was collected in crucibles and calcined at 600 °C for 3 h to produce a white silica nanoparticles (SiO₂NPs).

2.2.3. Characterization of Nanosilica

The obtained nanosilica powder subjected to various characterization studies to identify its structure and morphology. The samples were analyzed using characterization devices at Al-Mansoura University in Egypt. X-ray fluorescence spectroscopy (XRF) (EDX-720, Shimadzu, Japan) was used to determine the elemental composition and purity of the oxide-based nanosilica powder. The phase purity and crystallinity of the nanosilica powder were assessed by XRD analysis using a PANalytical X'Pert Pto MPD instrument equipped with a Cu K α (λ = 1.5406 Å) beam radiation source, operated at an acceleration voltage of 40 kV and an output current of 30 mA. The diffraction angle (2θ) was scanned from 10° to 80° at a rate of 1 °min⁻¹. The shape and size of the nanosilica powder were examined using TEM (JEOL, JEM-2100, Japan) with a 200 keV electron beam and a sample mounted on a carbon-coated copper grid. FTIR spectroscopy was used to determine the potential vibration of functional groups found in powdered silica nanoparticles, and was performed in the wavenumber range of 400–4000 cm⁻¹ using a Bruker FT-IR spectrometer (Invenio S, Germany). A UV-Vis double-beam spectrophotometer (Model: UH5300, Hitachi High Technologies, Tokyo, Japan) was used to determine the optical characteristics of SiO₂ NPs.

3. RESULTS AND DISCUSSION

3.1. SYNTHESIS OF SILICA DIOXIDE NANOPARTICLES

The detailed synthesis method for the SiO₂ NPs is shown in Figure 2. In this method, the following chemical reactions occur and SiO₂NPs are formed:

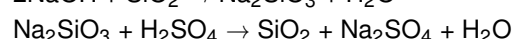
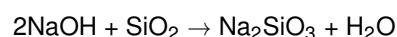
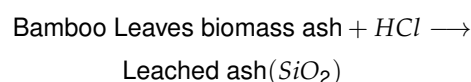




Figure 2. Green Synthesis of Silica dioxide Nanoparticles from Bamboo leaves ash.

3.2. ULTRAVIOLET-VISIBLE (UV-Vis) SPECTROSCOPY

In the UV-Vis molecular spectroscopic analysis, the SiO_2 nanoparticles yielded an absorption peak at approximately 240 nm in their UV spectrum, as shown in Figure 3. This may be attributed to electronic changes within the chemical bond and interference of light with the nanoparticles [13]. This finding matches that of [14] who showed that the synthesized nano-silica particles have an absorption range between 200 and 270 nm., [15] reported that the maximum absorption band for nanosilica produced from extract of *Gracilaria crassa* was at 250 nm. and [16] reported that the maximum absorption band for nanosilica produced from the leaf extract of *Punica granatum* was 310 nm.

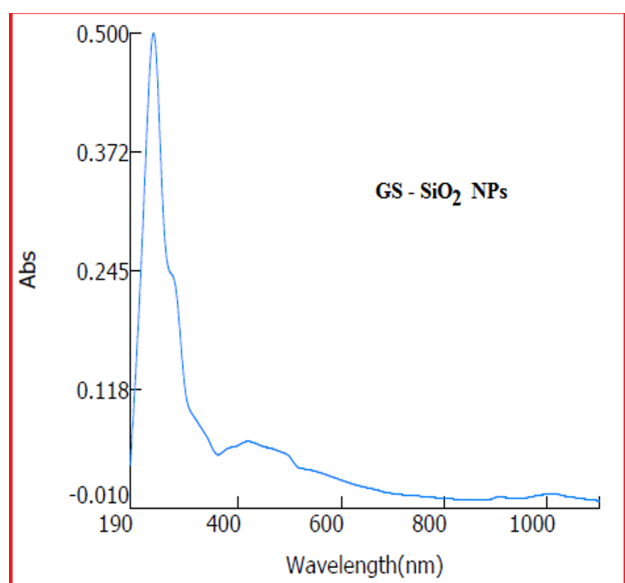


Figure 3. UV-Vis spectroscopy of synthesized SiO_2 NPs

3.3. FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Fourier Transform Infrared (FTIR) spectroscopy in the range of $400\text{--}4000\text{ cm}^{-1}$ was extensively employed to identify the bonding characteristics and functional group modifications of the synthesized nanomaterials. Analysis of the FTIR spectra provided valuable insights into the chemical composition, structural features, and purity of the nanoparticles. As illustrated in Figure 4, a strong absorption band was observed at 1092 cm^{-1} , which corresponds to the asymmetric stretching vibrations of Si–O–Si bonds, consistent with the findings of [17]. The absorption band at 791 cm^{-1} is attributed to the symmetric stretching vibrations of Si–O–Si, as reported by [18]. The adsorption band at 955 cm^{-1} and 464 cm^{-1} are due to the Si–O stretching vibrations of silanol groups and bending vibrations of Si–O–Si bonds, respectively [14]. These FTIR results confirmed the successful synthesis of the silica nanoparticles and revealed the presence of characteristic bonding patterns. Furthermore, these findings are in line with those of previous studies [6, 9, 19].

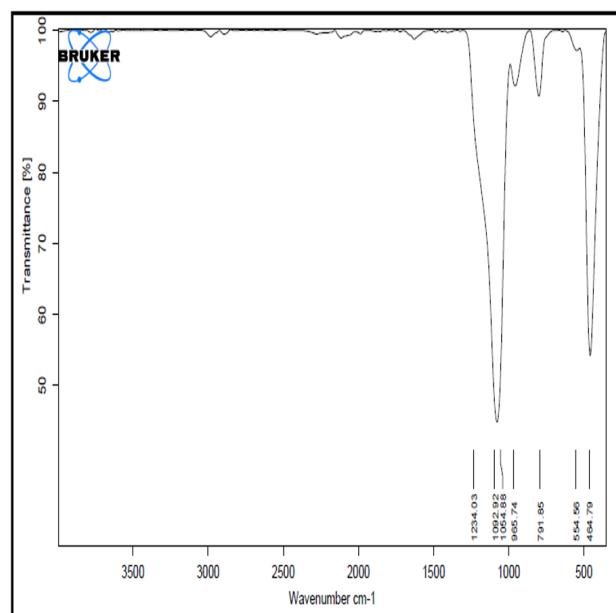


Figure 4. FT-IR spectral of synthesized SiO_2 NPs

3.4. X-RAY FLUORESCENCE SPECTROSCOPY (XRF)

The purity and elemental composition of the synthesized nanosilica powder in the form of oxides (SiO_2NPs) were analyzed using X-ray Fluorescence (XRF) spectroscopy. As shown in Table 1, the XRF results indicated that the nanosilica had a high purity of 97.82%, with minimal contamination from sodium (Na), aluminum (Al), and sulfur (S). These elements are commonly reported as residual impurities in the synthesized nanosilica [9]. The

presence of sulfur in the sample can likely be attributed to the use of sulfuric acid (H_2SO_3) during the synthesis process, which is consistent with the observations made by [20]. These findings are consistent with those of previous studies. For instance, [6] reported the highest level of nano-silica purity, reaching 94.6% from Bamboo Leaves, whereas [9] noted a range of 69% to 98% purity for nanosilica synthesized from rice husks. Similarly, [21] reported that the silica content of the xerogels produced from washed rice husks reached 99.5%.

Table 1. Elemental Component of SiO_2 NPs using X-ray Fluorescence Spectroscopy (XRF)

Elemental Component	Value (wt.%)
SiO_2	97.82
Fe_2O_3	0.058
Al_2O_3	0.563
CaO	0.065
Na_2O	0.749
K_2O	0.015
SO_3	0.567
MgO	0.019
TiO_2	0.096

3.5. TRANSMISSION ELECTRON MICROSCOPE (TEM) ANALYSIS

The primary objective of TEM analysis was to determine the size and morphology of the SiO_2 nanoparticle clusters. The silica particles exhibited various shapes, including spherical, triangular, and pentagonal, with the majority being spherical and exhibiting minimal agglomeration (Figure 5). These observations are consistent with [22], who reported similar morphologies and low agglomeration levels attributed to silicon-oxygen bridge bonding. Figure (3,4) clearly demonstrates that the synthesized SiO_2 nanoparticles exhibit an amorphous structure, as indicated by the absence of any crystalline features. This observation was further supported by the XRD analysis. TEM images of SiO_2 NPs synthesized using the green method showed an NPs size range 10.79–22.73 nm. A similar size was obtained by [23] utilizing the RHA extract to form spherical SiO_2 nanoparticles with diameters in the range of 5–20 nm, and [22] the average diameter of the SiO_2 NPs was found to vary from 10–15 nanometers. A larger size was obtained by [19] who reported that nanosilica from *Rhus coriaria* L. extract has average dimensions of 55 nm, [24] established that nanosilica fabricated using the precipitation method was 66 nm, and [25] indicated that nanosilica size was 60 nm.

3.6. X-RAY DIFFRACTION (XRD)

The synthesized SiO_2 nanoparticles (SiO_2 NPs) were analyzed using XRD to determine whether they were crystalline or amorphous. A broad diffraction peak ap-

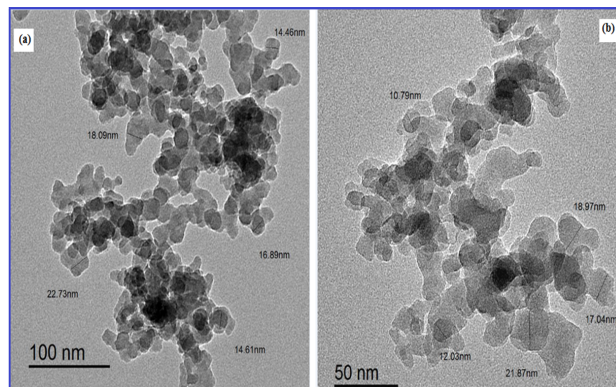


Figure 5. TEM analysis of SiO_2 NPs

peared at $2\theta = 23^\circ$ ($\text{hkl} = 100^\circ$), which is characteristic of amorphous silica, as shown in Figure 6. This broad peak results from the scattering of X-rays by a disordered atomic arrangement, indicating a predominantly amorphous structure. The absence of sharp diffraction peaks further confirms the lack of long-range crystalline order in the synthesized material [26]. These findings are consistent with earlier studies, such as those by [12, 25, 27], which also reported similar broad amorphous patterns for synthesized silica. Additionally, the diffraction pattern matched the standard SiO_2 reference (JCPDS file No. 89-0510), confirming the purity of the material, as no impurity peaks were observed [28].

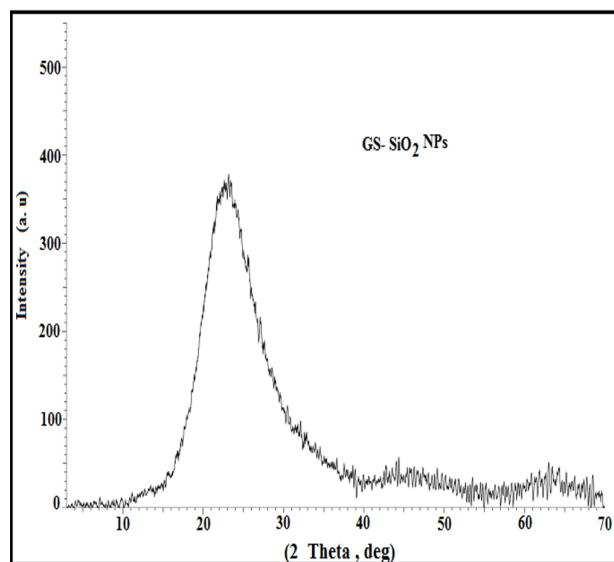


Figure 6. XRD spectral of synthesized SiO_2 NPs

The current study contributes to the ongoing national research efforts focused on the valorization of local biological resources for material and health-related applications. Previous investigations, including those of Al Maqtari and Al Maqtari [29], demonstrated the antibacterial potential of *Lawsonia inermis* leaf extracts, whereas more recent studies have explored the phytochemical



and antimicrobial properties of *Mirabilis jalapa* [30] and the industrial significance of *Boswellia sacra* in the South Arabian Peninsula [31]. Additionally, microbiological assessments of locally produced food items, such as ice cream, have highlighted quality and safety concerns that may benefit from the application of antimicrobial nanomaterials, such as silica nanoparticles [32]. Collectively, these studies support the importance of developing functional nanomaterials such as silica NPs synthesized from bamboo leaves from indigenous and agricultural waste sources for sustainable biomedical and environmental innovations.

4. CONCLUSION

Amorphous silica nanoparticles were produced using bamboo leaves as the source of silica. The precipitation method successfully produced nanoparticles from the bamboo leaf ash. The physical properties of nanoparticles end up being amorphous silica produced after being heated at 750 °C, uniform particle size distribution, and a large surface area. This workable approach proved to be cost-effective, easily scalable, and environmentally friendly for the mass production of silica nanoparticles with a high surface area suitable for different applications. its separation properties," J. Polym. Eng. **39**, 679–687 (2019).

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