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Sustainable Nano Silica (SiO_2NPs) Production from Bamboo leaves by Green Synthesis Techniques (Sol-Gel Method) and Characterization

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Abstract: *The synthesis of nano silica from bamboo leaves is a sustainable and environmentally friendly and low cost alternative to traditional methods. From different green synthesis technique sol-gel method have been used which is environmentally benign processes, avoiding harmful chemicals and energy-intensive operations and hence comply with green chemistry principles. The bamboo leaves collected from Tadoba Tiger Reserve, District -Chandrapur, Maharashtra, India. Nano silica powder synthesis by solgel method is generated subsequent to thermal treatment of BLA at 650°C for 2 hr followed by characterization using advanced characterization techniques. The absorption peaks 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 Nano silica particles.Analysis of the Nano silica by X-ray diffraction (XRD) and Scanning electron microscope (SEM) confirmed crystalline structure with a peak intensity at 2θ=23°..*

Keywords: low-cost synthesis, silica nanoparticles, solgel method, bamboo leavesash,characterization, XRD, UV-Vis, FTIR, SEM, TEM.

1. Introduction

The Bamboo is one of the most productive and rapidly expanding natural resources. Its leaf ash (BLA), which has a large surface area and more than 70% amorphous silica, is a rich source of silica appropriate for a number of industrial uses. (1)Since nations with an abundance of bamboo primarily use it for construction and pulp, bamboo leaves are typically considered agricultural waste. (2)Due to their unique qualities, which include high compatibility, large surface area, and exceptional chemical and thermal stability, amorphous silica nanoparticles have recently gained popularity. These qualities make them appropriate for a variety of potential uses, including drug delivery, biosensing, surface coatings, and abrasives. (3)Silica is used widely in a variety of industries, including manufacturing, adsorption, packaging, and agriculture. It is widely used in many different industries, including as foundries, the production of cement, glass, and chemicals. (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 nano silica from agricultural waste (6).Research on the production of silica from different agricultural wastes, such as corncobs (7).Recent studies have reportedsilica extractionfrom maize husks [8], rice husks [9], sugarcane bagasse [10], and bamboo stalks [11]. Furthermore, not much research has been done on the production of silica from bamboo leaves.Thus, the precipitation method of producing nano silica from bamboo leaves and the characterisation of the as-precipitated nano silica are presented in this paper. This research supports the sustainable development objectives of the circular economy and promotes the valorization of agricultural waste.

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II. MATERIALS AND METHODS

2.1. MATERIALS

1) Sodium hydroxide (2M NaOH),
2) Hydrochloric acid (1M HCl), and Distilled waterwere procured from the Central Scientific Company in Nagpur, Maharashtra, India.

III. SYNTHESIS OF NANO SILICA PARTICLES FROM BAMBOO LEAVES BY SOLGEL METHOD

Bamboo leaves were collected from the Tadoba forest in Chandrapur, Maharashtra. These leaves (Figure 1) served as the precursor source of silica for the synthesis of nano-silica particles. The chemical reagents employed in the solgel process are as given below

Bamboo leaves were collected from the forest of Tadoba in Chandrapur, Maharashtra. To remove alkali impurities, the leaves were crushed and leached with 1M HCl for 2 hours. Following acid leaching, the leaves were thoroughly washed with distilled water and driedovernight. The treated leaves were then calcined at 650^0 C for 2 hours in a muffle furnace to eliminate organic impurities.The resulting Bamboo Leaf Ash (BLA) was reacted with an alkali solution and subsequently neutralized with acid.

Finally, silica was extracted from the resulting Bamboo Leaf Ash (BLA) using the sol-gel method to produce silica xerogel [12].

In this process, 60 mL of 2M NaOHwas added to 10 g of Bamboo Leaf Ash (BLA). The mixture was boiled for one hour under constant temperature(90^0C) on magnetic stirring for 1 hour to produce a sodium silicate solution. The resulting solution was filtered through Whatman No. 41 filter paper. The filtrate, containing the sodium silicate, was cooled to room temperature, and the pH was adjusted to 7 by the dropwise addition of 1N HCl under continuous stirring to induce gelation. Once the silica gel formed, it was aged for 1 hours. Following the aging period, the soft gel was gently broken by adding 100 mL of distilled water and centrifuged to form a slurry. The slurry was then filtered and washed; the supernatant was discarded, and the resulting gel was dried at 80^0 C 12 hours to produce a fine white powder of silica.

BAMBOO LEAVES ASH +SODIUM HYDROXIDE

⬇

SODIUM SILICATE

$NaOH+SiO_2 \longrightarrow Na_2SiO_3 + H_2O$

$Na_2SiO_3 + 2HCl \longrightarrow SiO_2.n\ H_2O$

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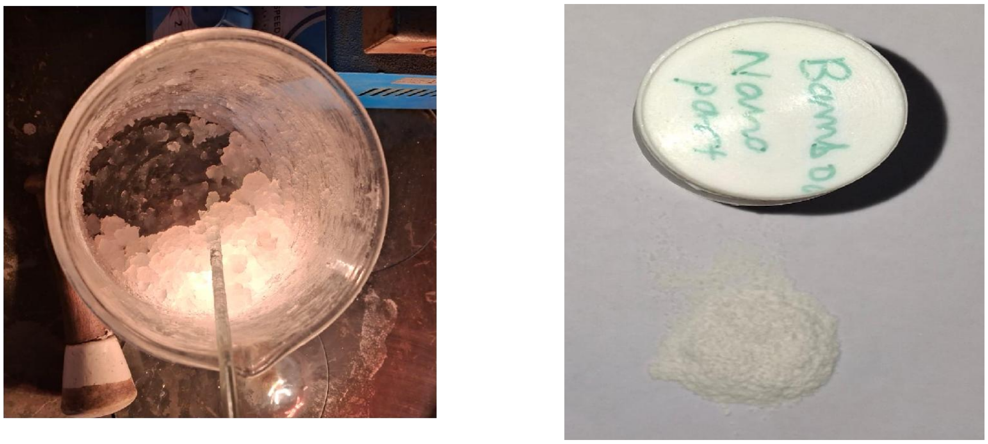

Fig. 1. Photos of synthesized silica nanoparticle from bamboo leaves

IV. SPECTRAL AND PHYSICOCHEMICAL ANALYSIS OF SYNTHESIZED NANOSILICA

The obtained nano silica powder subjected to the phase purity and crystallinity of the nano silica powder were assessed by XRD analysis using Sophisticated analytical instrument Wardha equipped with a Cu Kα (λ =1.54059 Å and 1.54441 A^0) beam radiation source, operated at a voltage of 40 kV and of 15 mA. The diffraction angle (2θ) was scanned from 10° to 90° at a rate of 0.02°min-0.06. The Elemental analysis of the nano silica powder was examined using EDS(STIC). 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 (SAIF-cochin, STIC). A UV-Vis double-beam spectrophotometer (SAIF) was used to determine the optical characteristics of SiO_2 NPs.

4.1. ULTRAVIOLET-VISIBLE (UV-VIS) SPECTROSCOPY

In the UV-Vis molecular spectroscopic analysis, the SiO_2 nanoparticles yielded sharp absorption peak around 190 nm and negligible absorption in the visible range (400–800 nm) this is highly characteristic of SiO□ (silicon dioxide) nanoparticles in their UV spectrum, as shown in Figure 2. 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].

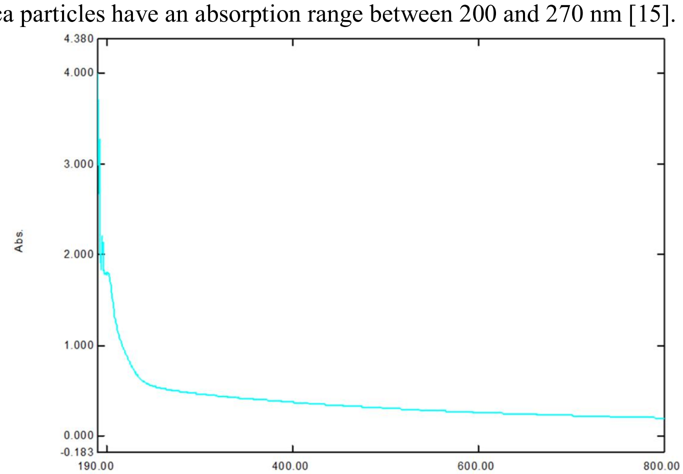

Figure 2. UV-Vis spectroscopy of synthesized nanosilica from bamboo leaves

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4.2. FOURIER-TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Fourier Transform Infrared (FTIR) spectroscopy in the range of 400–4000 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 3, a strong absorption band was observed at 1058.25 cm-1, which corresponds to the asymmetric stretching vibrations of Si–O–Si bonds [16]. The absorption band at 791.85 cm^{-1} is attributed to the symmetric stretching vibrations of Si–O–Si [17]. The adsorption band at 444.10 cm-1 is attributed 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 [6,9,18].The bands at 1636.25 cm^{-1} and 3410.81 cm^{-1} correspond to H-O-H bending and O-H stretching vibration respectively.

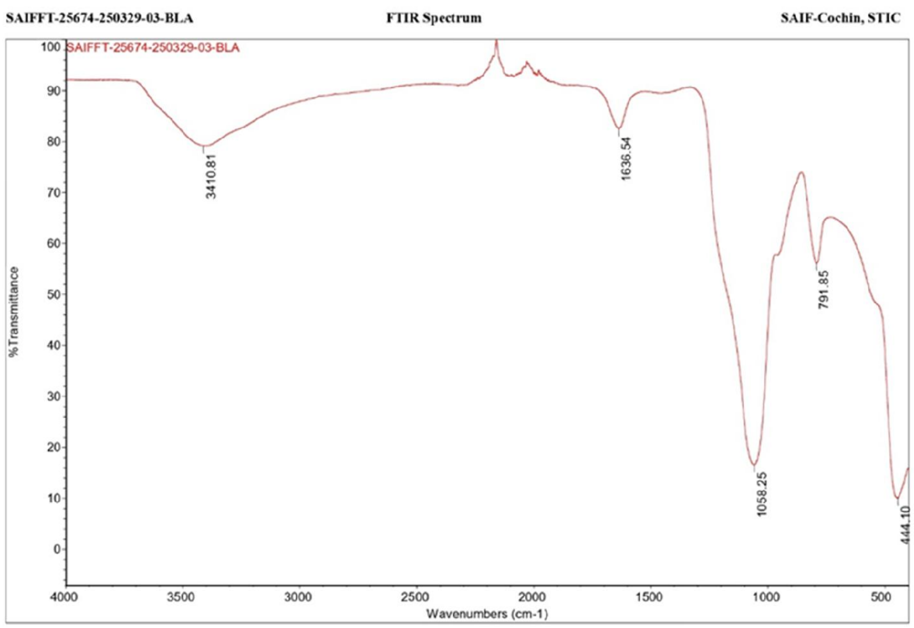

Figure 3. FT-IR spectral of synthesized SiO2 NPs

4.3. SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

The surface morphology and particle distribution of the silica biofiller were visualized using SEM at various magnifications is given in figure 4. The micrographs reveal an irregular, granular morphology with a broad particle size distribution. At higher magnification ($X7,000$), the particles show a rough surface texture, which is advantageous for mechanical reinforcement in rubber composites as it increases the effective surface area for polymer-filler contact. While some agglomeration is visible, the primary particles remain in the sub-micron to low-micrometre.

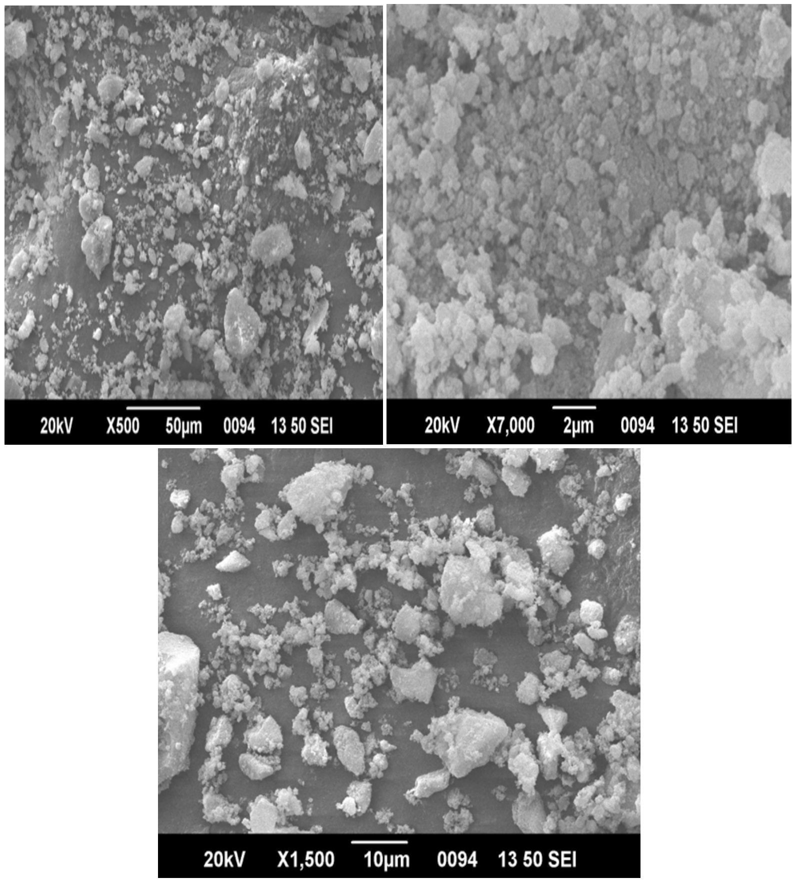

Figure 4. SEM of nanosilica from bamboo leaves

4.4. EDX (ENERGY DISPERSIVE X-RAY SPECTROSCOPY)

The elemental composition of the SiO2 nanoparticles was analysed using Energy Dispersive X-ray (EDX) spectroscopy. The spectrum (Figure 5) exhibits high-intensity signals for Silicon (Si) and Oxygen (O), providing definitive evidence for the formation of a silica framework. The presence of trace amounts of Sodium (Na) and Chlorine (Cl) was also detected. These minor peaks are attributed to residual salt byproducts from the green synthesis reagents. The overall spectrum confirms a high degree of elemental purity, consistent with the siloxane network identified in the FTIR analysis.

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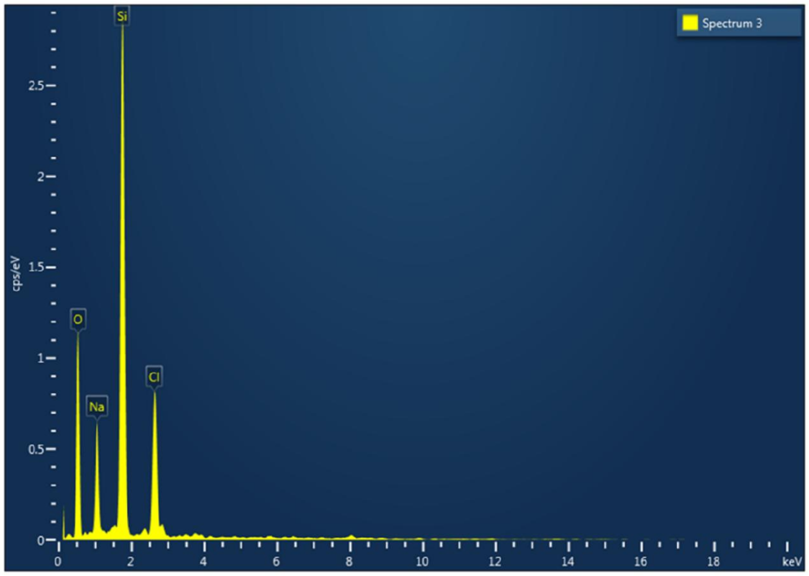

Figure 5. EDX of Synthesized nanosilica from bamboo leaves.

4.5. XRD (X-RAY DIFFRACTION)

The XRD pattern of the BLA sample (Figure 7) exhibits a combination of sharp diffraction reflections and a broad amorphous halo. The prominent sharp peaks observed at2θ values of approximately 31.7°, 45.5°, 56.4°, and 75.3° correspond to the (100), (200), (220), and (311) planes of a face-centered cubic (FCC) lattice, consistent with crystalline NaCl. Additionally, a broad diffraction peak centered at 2θ=23^{0}within the range of20^0-30^{0}which is indicating the presence of an amorphous phase within the sample.

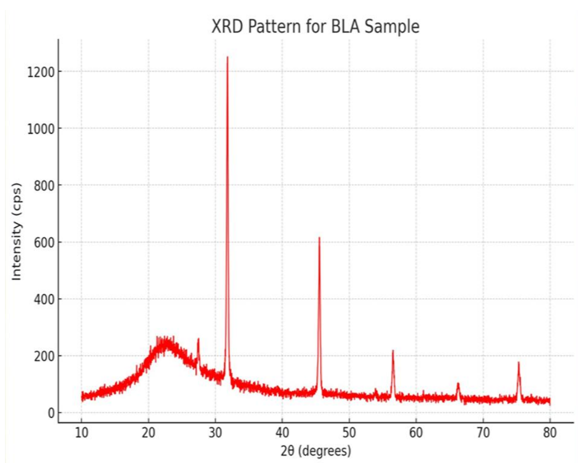

Figure 6. XRD Spectral of synthesized nanosilica from bamboo leaves

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4.6. TEM (TRANSMISSION ELECTRON MICROSCOPY)

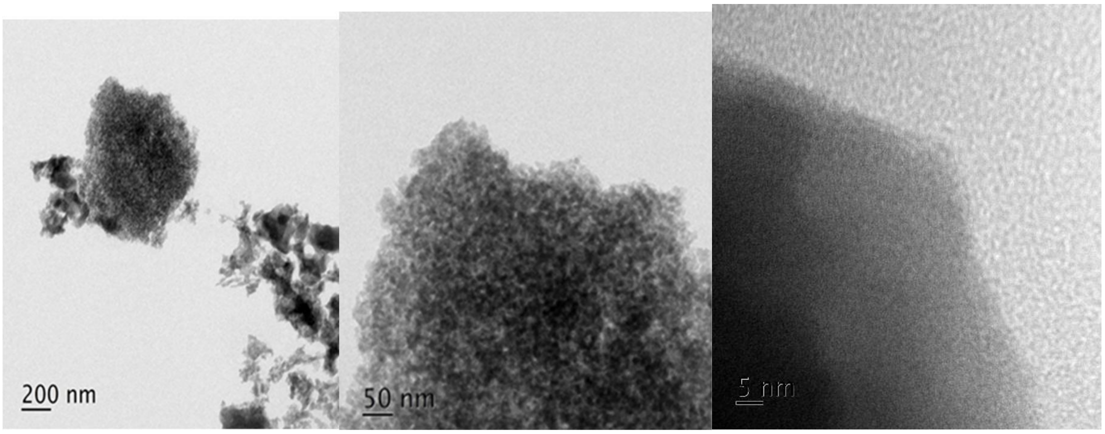

Figure 7. TEM of nanosilica from bamboo leaves at 200 nm, 50 nm, 5 nm

The morphology and microstructural features of the synthesized nanoparticles were investigated using transmission electron microscopy (TEM). The TEM images obtained at various magnifications are shown in Figure 7. The primary aim of this analysis was to evaluate the size and shape of the SiO_2 nanoparticle aggregates. The silica nanoparticles displayed a range of geometries such as spherical, triangular, and pentagonal forms, with spherical shapes being predominant and only slight agglomeration observed. Comparable observations of morphology and reduced aggregation, attributed to silicon–oxygen bridging interactions, have been reported in earlier studies [18].

TEM analysis of SiO_2 nanoparticles prepared via the sol–gel method indicated particle sizes in the range of 5–20 nm. Similar dimensions have been reported in previous works, where spherical SiO_2 nanoparticles synthesized using RHA extract exhibited sizes between 5–20 nm [18], while another study reported average particle diameters of 10–15 nm [20]. In contrast, larger particle sizes have also been documented; for instance, nanosilica derived from *Rhus coriaria* L. extract showed an average size of 55 nm [18], while precipitation-based methods yielded particles of about 66 nm [21] and 60 nm [22].

The low-magnification TEM image (Figure 8, 200 nm scale) illustrates the presence of irregular aggregates extending to several hundred nanometers. These clusters are composed of numerous ultrafine primary particles densely packed together. Such aggregation behavior is characteristic of nanoparticles synthesized via precipitation techniques, where high surface energy leads to strong interparticle interactions during drying and sample preparation.

At intermediate magnification (Figure 8, 50 nm scale), the internal arrangement of these aggregates becomes more evident. The image reveals that the secondary structures consist of nearly spherical primary nanoparticles forming an interconnected network. The uniform contrast across the particles indicates a consistent composition throughout the sample. Based on the scale bar, the average particle size remains within the nanometer range, typically below 20 nm. The lack of elongated or anisotropic structures suggests that the particles underwent isotropic growth during nucleation.

The high-resolution TEM image (Figure 8, 5 nm scale) provides deeper insight into the structural characteristics of the nanoparticles. The image shows diffuse contrast without clear lattice fringes, indicating that the particles are predominantly amorphous or possess low crystallinity. This type of structure is commonly associated with materials synthesized through precipitation methods under controlled thermal conditions, where rapid nucleation restricts the development of long-range atomic order.

V. CONCLUSION

Silica nanoparticles were produced usingas source of the bamboo leaves ashby sol-gel method. Silica yields as filler with high chemical purity and favourable morphological characteristics. The combination of a well-defined siloxane network (FTIR), high silicon content (EDX), granular nano-scale morphology (SEM), microstructural and morphology suggest synthesizednanoparticles (TEM) Sand X-ray diffraction indicates amorphous as well as crystalline nanoparticles confirms that this bio-silica is a viable, sustainable alternative to synthetically derived fillers. Its unique optical properties and surface silanol density make it a promising candidate for various industrial reinforcement applications.

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