



Synthesis and Characterization of Silica Nanoparticles Derived from Tea Factory Generated Wood Ash

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

India is the second most prolific tea producing nation in the world, which consumes 82% of its tea production, accounting for 19.5% of global tea consumption. Despite the improvement in technology, employing wood to produce heat has been a vital element of tea processing for

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generations. Many tea specialists experience that it adds a particular fragrant touch to the finished tea product. However, the management of wood ash generated by tea factories is a significant concern due to the large amount of wood burned during tea processing. Therefore, transportation and effective disposal of these large quantities of wood ash is a great challenge. Hence, this study aimed to effectively transform wood ash into a valuable product, nano silica particles so as to explore the scope for its better utilization in various applications. A series of experiments were carried out to optimize the parameters in the sol-gel technique for synthesizing silica nanoparticles from wood ash. Further, the synthesized nano silica particles were characterized by employing transmission electron microscopy (TEM) and X-ray diffraction (XRD). The standard operational protocol developed through this study demonstrated that wood ash can be effectively converted to silica nanoparticles in the size range of 20-50 nm, spherical in form with crystalline properties. Overall, the results of this work highlights the possibility of utilizing tea factory generated wood ash into silica nanoparticles with an immense potential for varied applications without environmental hazards.

Keywords: Wood ash; waste management; silica nanoparticles; sol-gel technique.

1. INTRODUCTION

The global energy consumption is projected to rise by around 50 to 55 percent by the year 2040 as per International Energy Agency (IEA) [1]. Biomass is the third most significant energy source, behind coal and oil, in terms of fuel energy. The primary sources of biomass energy are wood (sometimes referred to as woody biomass), agricultural products, solid wastes, biogas and alcohol fuel [2]. Wood constitutes 74% of the total worldwide biomass energy consumption [3], indicating that wood is the primary source of biomass used for energy purposes. In the tea processing factories, wood is the main source of energy for generating heating. Our survey indicated that on an average, 1 kg of wood is utilized for every one kilogram of tea powder that is produced. Despite the fact that electricity based furnace technology is available for heating purpose in tea processing factories, almost majority of the tea industries [4] in South India utilize wood as a heat source. Hence, the growing demand for tea [5] also increases the consumption of wood and in turn huge quantity of wood ash is generated. In India, the tea production for the year 2023 is 1367 M.Kgs as per Tea Board data (2023) and therefore atleast the same quantity of wood biomass would be utilized for tea processing, which eventually would have generated 27,354 tonnes of wood ash. The management of this waste becomes a great challenge due to scarcity of landfill space for its disposal besides a huge cost involved in handling and transportation. Thus, a solution to effectively utilize this resourceful waste is the need of the hour and our study focuses on this environmental issue.

The characterization of ash derived from wood biomass varies with respect to various factors such as wood species, technology used for burning, temperature at which combustion occurs, and also the place where the ash is stored [6]. The key chemicals present in the wood ash are CaO, K₂O, P₂O₅, MgO and SiO₂ [7]. The amount of silica dioxide (SiO₂) also varies with the type of plant species ranging from 2% to 52.4% [8]. In order to promote circular nutrient economy and effective utilization of resources, in this research work, we attempted to convert the wood ash (combination of *Eucalyptus globulus*, *Prosopis julifera*) into nanosilica particles. Though sol-gel method is a popular technique to synthesize nanoparticles like silica or ZnO etc., the chemical composition of wood ash generated in tea factory is unique in nature due to the variety of wood biomass used and hence, optimization of chemical parameters is pivotal to obtain a pure form of nano silica and this study is a maiden attempt to standardize operational protocol (SOP) to transform wood ash into silica nanoparticles. Thus, the SOP for synthesizing silica nanoparticles, followed by its characterization is presented in this paper.

2. MATERIALS AND METHODS

2.1 Reagents Used

All the reagents used were of analytical quality to ensure maximum purity and optimal performance. The solutions were produced using distilled water to minimize any potential contamination. HCl and H₂SO₄ used for its high proton-donating ability, whereas NaOH functioned as a powerful source of counterions. The polyethylene glycol (PEG) was chosen for its

adjustable viscosity and stabilizing characteristics.

2.2 Sources of Wood Ash

The tea factory produces wood ash as a residue during the processing stage (heating, and drying). Wood ash was collected from two factory locations in Kattabedu at Kothagiri taluk of Nilgiris. The primary types of wood used for burning in the tea factory were *Eucalyptus globulus* and *Prosopis julifera*. The wood ash was sieved using a British Standard sieve (BSS) 200 sieve (125 μm) to achieve a consistent distribution of particle sizes. A quantity of 30 grams of weighed wood ash was incorporated into the synthesis process.

2.3 Scanning Electron Microscopy (SEM)

The morphology of wood ash generated by the tea factory was examined using a scanning electron microscope (SEM) from FEI, model Quanta 250. For the analysis, 1 mg wood ash sample was added to 10 ml of distilled water, resulting in the formation of a homogeneous suspension. Followed by, one droplet of the mixture was meticulously applied onto carbon tape attached to a stub using a micropipette.

After the air drying of the sample, it was coated with a layer of gold by a procedure called sputtering. This was done to make the sample conductive and suitable for high-resolution imaging. Once a high vacuum environment was established with a pressure of 3.99e^{-4} Pa, the thermionic emission filament was turned on. Subsequently, other parameters were modified for the purpose of picture capture, such as electron beam energy, beam current, spot size, and accelerating voltage. The electron beam and sample surface contact are influenced by these factors, which ultimately determine the picture resolution, contrast, and depth of field. The wood ash surface characteristics were examined at magnification ranges ranging from 500X to 20000X. A Gaussian curve was fitted in ImageJ software and the average particle size of wood ash was revealed.

2.4 Energy Dispersive X-ray Spectroscopy (EDAX)

The elemental composition of a wood ash sample was analyzed using EDAX. A focused electron beam with an energy range of 10-20 keV was directed onto the surface of the

wood ash sample. The strong interaction stimulates the atoms present in the ash, causing them to release distinct x-rays. Every element emits a distinct x-ray frequency, similar to a fingerprint. This technique was used to identify the elements present and determine their relative abundances [9].

2.5 Synthesis of Silica Nanoparticles through Sol-gel Method

The sol-gel technique of synthesis is extensively used and has several industrial applications, particularly in the fabrication of oxide ceramics and polymer ceramic composite materials [10]. Furthermore, the sol-gel technique enables the production of very pure composites with exceptional homogeneity, reaching a purity level of 99.9% [11]. Nano silica was synthesized from wood ash by following the two step process i.e., acid-alkali (sol-gel method) as outlined by Thu et al. [12]; Yadav et al. [13]. The extraction process was modified by varying different ratios of alkali (NaOH) and acids (HCl and H_2SO_4), as well as the temperature (oven drying at 100-200°C and furnace temperature at 500-700°C), and the stabilizer concentration (1-3%).

2.5.1 Optimization of concentrations for synthesis of nano silica

A series of tests were done to examine the effect of different concentrations of HCl on the removal of contaminants from wood ash. Four factors (different ratios of HCl, centrifugation speed and temperatures of hot air oven and muffle furnace) were optimized in this study. Each of the five grams of ash was treated with a ratio of 1M HCl (1:5, 1:10, and 1:15) and then centrifuged (1500, 3000, and 5000 rpm) for 10 minutes. Subsequently, the process of oven drying was carried out at temperatures of 100°C, 150°C, and 200°C for periods of 1, 1.5, and 2 hours, respectively. Ultimately, the ash samples underwent a drying process in a furnace at temperatures of 500°C, 600°C, and 700°C for durations of 2, 4, 6, and 8 hours.

2.5.2 Precipitation of silica nanoparticles from acid treated wood ash

A series of experiments were carried out to optimize the nanosilica recovery from wood ash. In this batch experiment, four factors (different concentrations and ratios of NaOH, varying concentrations of H_2SO_4 and PEG). The acid treated ash resulted from the above said process

(section 2.5.1) was first treated with different concentrations (2M, 2.5M, and 3M) and varying ratios of NaOH (1:2, 1:4, and 1:6). The formed sodium silicate solution was titrated with varying millimolars of H₂SO₄ (150 mM, 300 mM, and 600 mM) in the presence of different quantities of PEG at 1%, 2%, and 3%. This approach was designed to identify the key parameters necessary for attaining the most optimal and controlled silica precipitation. To examine the impact of thermal treatment on the characteristics of the resultant precipitate, several calcination conditions were also studied.

The samples were subjected to thermal exposure at temperatures of 500°C, 600°C, and 700°C for durations of 2, 4, and 6 hours, respectively. This allowed for a comprehensive understanding of

the correlation between temperature and time. Fig. 1. Illustrates a schematic representation of the standard operating procedure for synthesizing silica nanoparticles from wood ash, with the aim of providing a concise summary of the most optimal operating conditions.

2.6 Transmission Electron Microscopy (TEM)

The TEM (Model: Tecnai 12) was used to get a thorough knowledge of the size and structure of the produced nanomaterial in order to explore its potential applications. Measurement of the average particle size of synthesized nanomaterial was achieved through the application of a gaussian fitting curve in the ImageJ software platform.

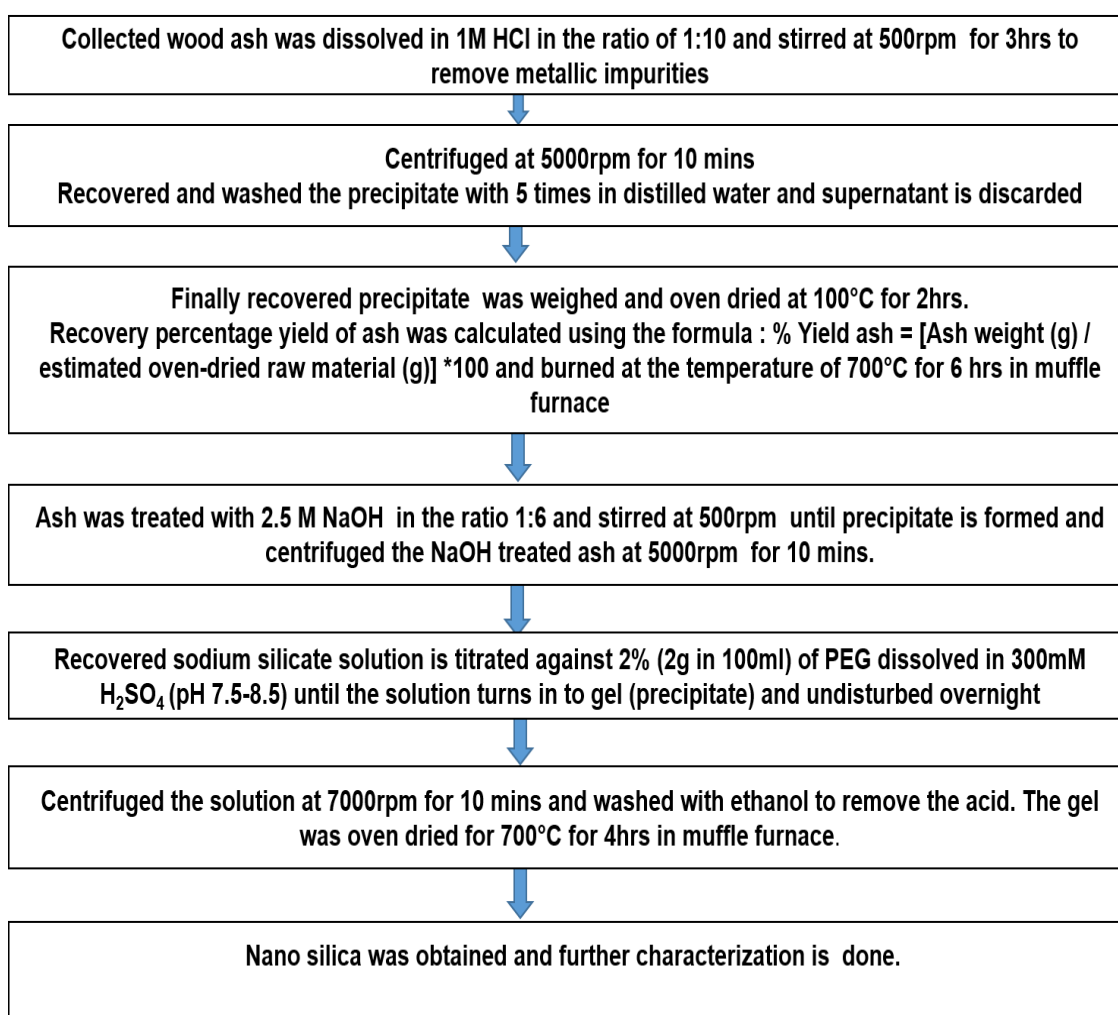


Fig. 1. Flowchart illustrating the process of synthesizing silica nanoparticles using wood ash generated from a tea factory

2.7 X-ray Diffraction (XRD)

The characteristics of the synthesized silica nanoparticles were analyzed using an X-ray diffractometer (Empyrean Series III Diffractometer) that utilizes Copper K α radiation (X-ray energy commonly employed in laboratory settings) with a wavelength of 1.54Å and an energy resolution of 450eV. The cathode ray tube generates x-rays, which are then collimated in order to concentrate them, filtered to create monochromatic radiation, and directed towards the sample. When the circumstances satisfies Bragg's Law ($n\lambda = 2d \sin\theta$), which establishes a connection between the wavelength of the incoming radiation, the diffraction angle, and the spacing of the lattice, the interaction between the incident monochromatic rays, the sample leads to constructive interference (formation of diffracted rays). For our experiment, 0.5 grams of synthesized powder samples, which were positioned on sample holders. The resulting data was then illustrated using Origin version 10.05.

2.8 Fourier Transform Infrared Spectroscopy

The functional group of synthesized nano silica, was recorded by Fourier- Transform Infrared Spectroscopy (FTIR) using Jasco (Model: R-3000-QE). A pinch of the sample was placed on the sample port and infrared radiation of about 10,000–100cm⁻¹ is passed through the sample and part of the radiation is absorbed and some pass through the sample. The sample converts the absorbed radiation to vibrational or rotational

energy. The detector detects the resultant signal generally from 4000 to 400 cm⁻¹ which is the molecular fingerprint of the sample. The data obtained were plotted using ORIGIN Ver. 10.05 [14].

3. RESULTS AND DISCUSSION

3.1 Morphology of Wood Ash

Scanning electron microscopy (SEM) images of wood ash showed varied types of structures such as irregular, spongy and spherical morphology with average particle size of 2.57µm. Our observations closely coincide with the results of wood ash derived from Russian olive which showed coarse, spongy and porous characteristics [15]. In general, wood biomass ashes revealed three distinct morphologies such as spherical, irregular porous conglomerates, smooth and also well-defined edges [16]. Fig. 2(a) is the representation of the structural characteristics of wood ash.

3.2 Elemental Composition of Wood Ash

EDS revealed elemental composition in the ash samples as shown in Fig 2(b), encompassing thirteen elements: C, B, Si, O, Ca, K, Al, Mg, In, Fe, P, S and Na. Similar results were reported in Russian olive wood ash which composed of K, Ca, Mg, P, O, C, S, Al, Si, Na [15]. Other studies also showed that wood biomass ash derived from birch, beech, oak, spruce and alder species contained high percentages of Ca, K, P, Al, Si, and Fe [17].

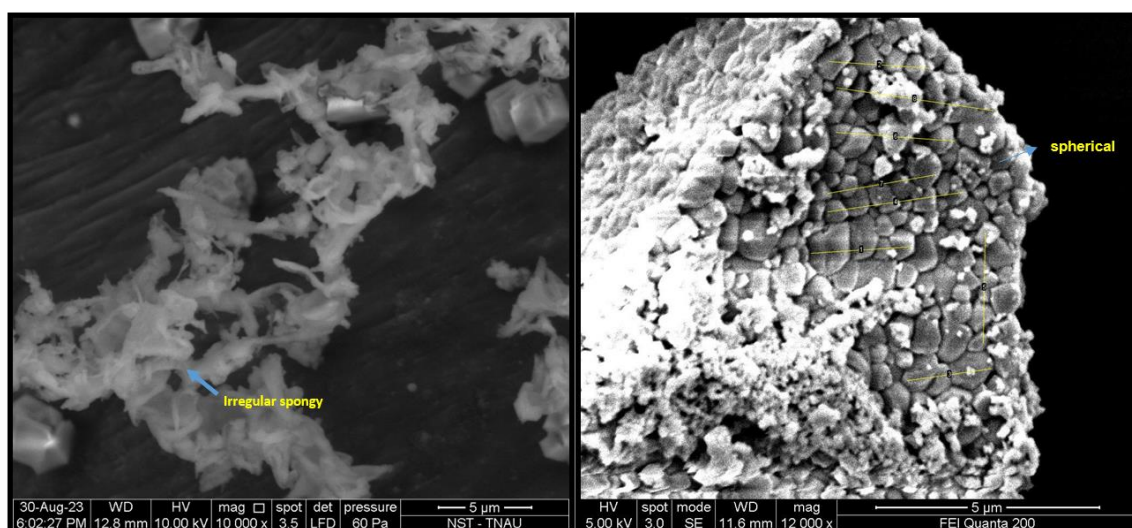


Fig. 2. (a) SEM image of wood ash at 10000X and 12000X with average particle size (b) EDS of Wood ash

Table 1 showed the chemical composition of ash derived from the wood species such as *Eucalyptus globulus* and *Prosopis julifera*. Silicon was found to be the fifth most prevalent element in wood ash, with a share of 1.9%. Our results are in line with the earlier reports which has registered 2.51% SiO₂ in eucalyptus ash and 3.57% SiO₂ in avocado ash [18]. Similarly, in the experiments performed with *Prosopis juliflora*, the SiO₂ percentage was 4.14% and 4.43%, respectively in its wood and charcoal [18].

3.3 Mechanism of Nanosilica Synthesis from Wood Ash

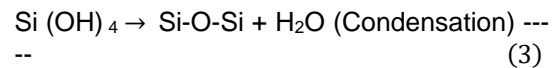
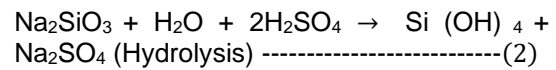
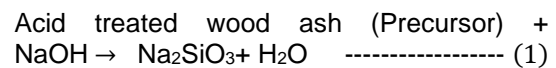
In the sol-gel technique for synthesis of nano silica particles, two key stages are involved. Firstly, the wood ash was subjected to acid treatment, and then the nano silica was formed and precipitated. A series of experiments were carried out to optimize the chemical concentrations involved in these processes and the most optimal conditions as indicated in the Fig. 1 was adopted and the results are presented hereunder.

3.3.1 Acid-assisted recovery of nanosilica from wood ash

The wood ash generated from tea factory was subjected to treatment with 1M HCl, with the concentration optimized at a ratio of 1:10, and underwent acid treatment in order to purify it by eliminating the metallic impurities. Followed by oven drying at 100°C for 2 hours, the wood ash was then subjected to a muffle furnace at 700°C for 6 hours. The Fig. 3(a) illustrates key stages of these process.

3.3.2 Laboratory preparation of sodium silicate solution

The acid - treated wood ash was stirred with alkali (NaOH) at 2.5M concentrations in 1:6 ratio. After thorough stirring, the mixture was centrifuged at 5000 rpm for 10 minutes. Centrifugation is a technique that uses centrifugal force to separate different components in a mixture based on their densities. In this case, the heavier undissolved particles in the mixture (impurities and unreacted ash) settle at the bottom of the centrifuge tube, while the lighter sodium silicate solution remains suspended on top, as illustrated in Fig. 3(b).



3.3.3 Transforming dissolved silica into nanosilica particles

The precipitation process continued when the sodium silicate solution was titrated. In the sol-gel, the hydroxyl group (OH) is substituted by the alkoxide group (OR) with the addition of water. The subsequent reaction is condensation, in which (R-OH) forms oxane bonds with the water as byproduct. As the quantity of oxane groups rises, the nanoparticles connect with one another to create a gel network.

Table 1. Elemental composition of tea factory generated wood ash (EDAX)

Elements	Wt% (weight percent)	At% (atomic percent)
Boron (B) (K)	51.2 %	57.5%
Carbon(C)(K)	33.3 %	33.6%
Silicon (Si) (K)	1.90 %	0.82%
Oxygen(O)(K)	6.80%	5.16%
Calcium (Ca)(K)	2.75%	0.83%
Potassium(K)(K)	1.08%	0.34%
Nitrogen(N) (K)	1.32%	1.34%
Aluminium (Al) (K)	0.51%	0.23%
Magnesium (Mg) (K)	0.27%	0.13%
Phosphorus(P) (K)	0.13%	0.05%
Sulphur(S) (K)	0.11%	0.04%
Indium (In) (L)	0.31%	0.03%
Iron (Fe) (K)	0.25%	0.06%
Total	100%	100%

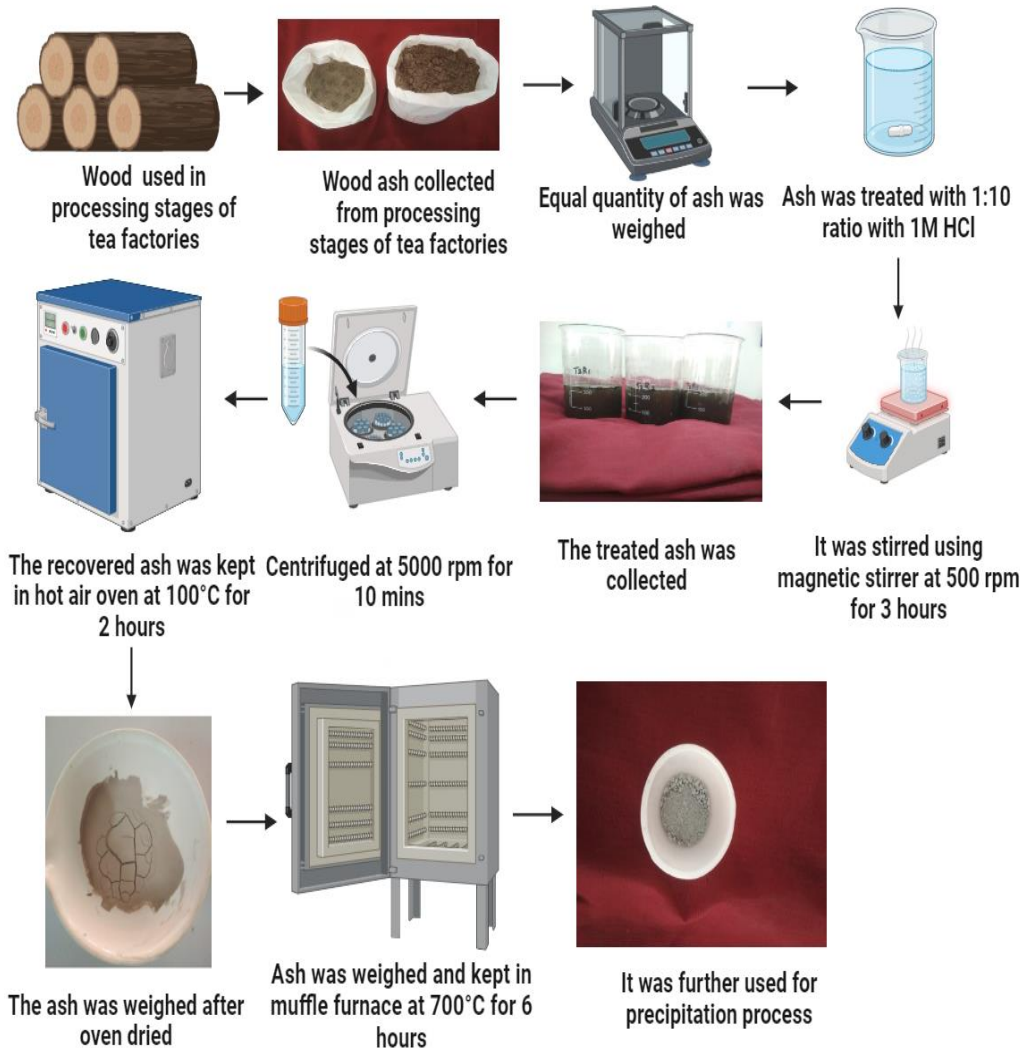


Fig. 3(a). Leaching of metal impurities from wood ash

The wet gel undergoes age and dehydration, resulting in the release of the solutes that were previously confined inside the gel's structure. The application of high-temperature treatment eliminates the organic waste inside the structure, resulting in the destruction of interconnected empty spaces and the formation of nano-silica. Bokov et al. [11]. The formation of nanosilica is visualized in Fig. 3(c).

To elaborate the steps involved in the above said two processes, equations 1, 2, and 3 illustrate the processes of sodium silicate formation and acidification, respectively. Precipitation of silica occurs when the pH drops below 10 during the acidification process. Controlling the water-based conditions is crucial to the production of silica nanoparticles. When the silicate concentrations

are low and the pH is below 8, the solution mostly generates individual $\text{Si}(\text{OH})_4$ molecules. However, when the concentration is increased, there is a shift towards alkalinity with a pH greater than 8, an intriguing transition occurs. When $\text{Si}(\text{OH})_4$ groups initiate a self-assembly process, they will spontaneously connect via disiloxo linkages to create more complex oligomers, which ultimately lead to the production of nanoparticles. The pH value indirectly regulates the concentration of protons (H^+) in the solution, which has a substantial influence on the gel formation process as reported during the synthesis of nanosilica from rice husk ash using this sol-gel technique [19]. The interdependence arises from the interaction between protons and different silica precursors, which affects their condensation and polymerization processes,

eventually determining the structure and characteristics of the resulting gel. Overall, the sol-gel process is the most preferred technique owing to its sustainability, cost-effectiveness, ecological compatibility, and extensive industrial applicability compared to other approaches [20].

3.4 Size and Shape of Synthesized Nano Silica

The TEM examination of silica nanoparticles exhibited a predominant spherical morphology (Fig. 4a). Although there was some slight aggregation, the particles mostly retained their distinct spherical shape. The size of nano silica particles ranged from 25 to 50 nm. The average particle size was 33 nm calculated by Gaussian fitting curve with image J software (Fig. 4b). TEM examination of SiO_2 nanoparticles produced from South African coal fly ash showed a primarily spherical shape with an average diameter of 98 nm. Nevertheless, a slight agglomeration of particles was seen and it necessitates high level of dispersion of nanoparticles [21]. TEM images of silica nanoparticles derived from sugarcane bagasse ash exhibited a little variation in size, with no

significant presence of large agglomerates over 50nm [22]. Similarly, studies with silica particles generated from rice husk ash were mostly in the size of 25 nm [23].

3.5 Crystallinity Nature of Nanosilica

The crystallinity of the synthesized silica nanoparticles was determined using XRD examination. The silica nanoparticle diffractogram (Fig. 5) showed that the greatest peak was located at $2\theta = 22^\circ$ without any hill range. This phenomenon is attributed to the presence of silica in an organized crystalline form. This is attributed to the fact that elevated temperature during the synthesis process leads to crystalline nature. The investigation of silica extraction from rice husk ash using the sol-gel technique with the assistance of ultrasound revealed the correlation between the temperature of the thermochemical process and the crystallinity of silica. Higher temperatures proved to be a key factor in facilitating the transformation from the amorphous to the crystalline form, offering valuable information for controlling the material's structure [24].

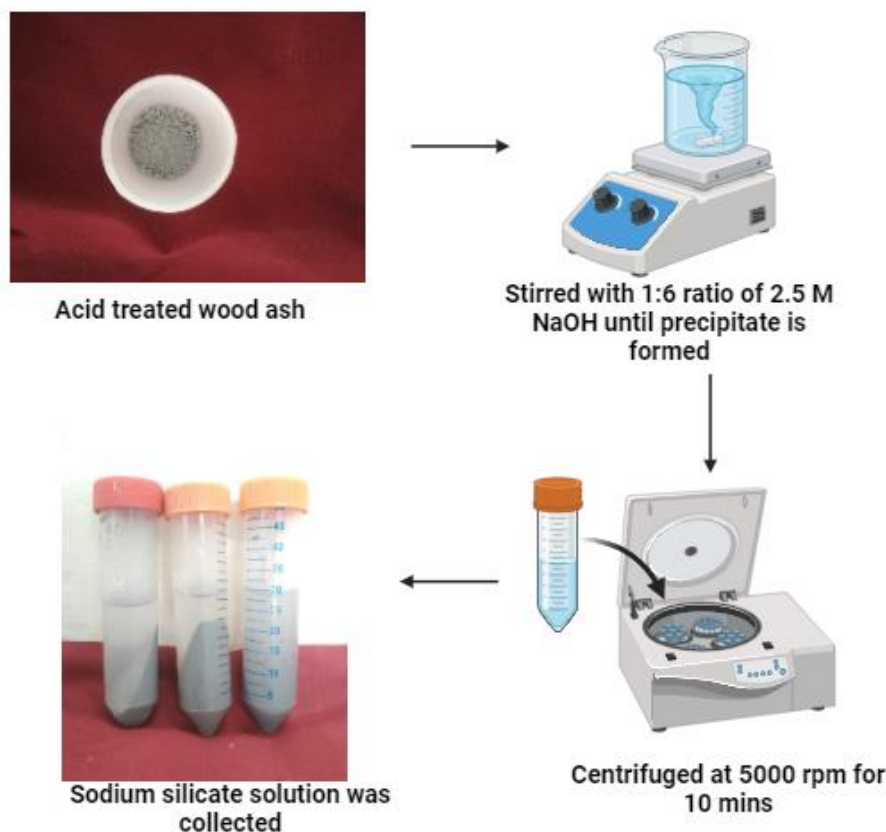


Fig. 3(b). Fusion of silica and NaOH for Na_2SiO_3 solution preparation

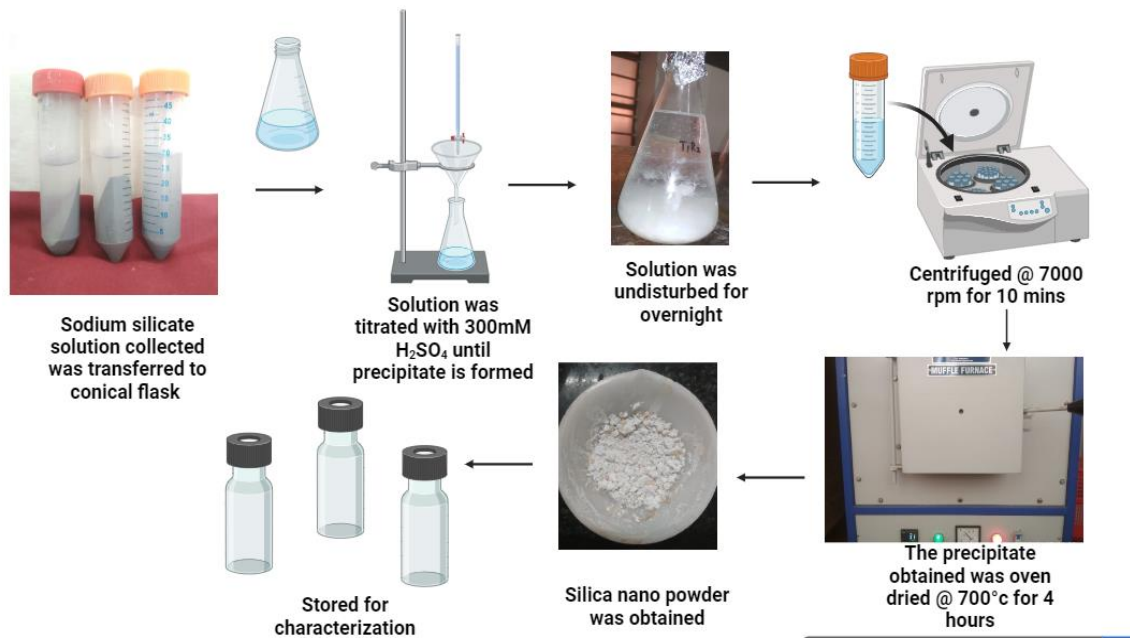
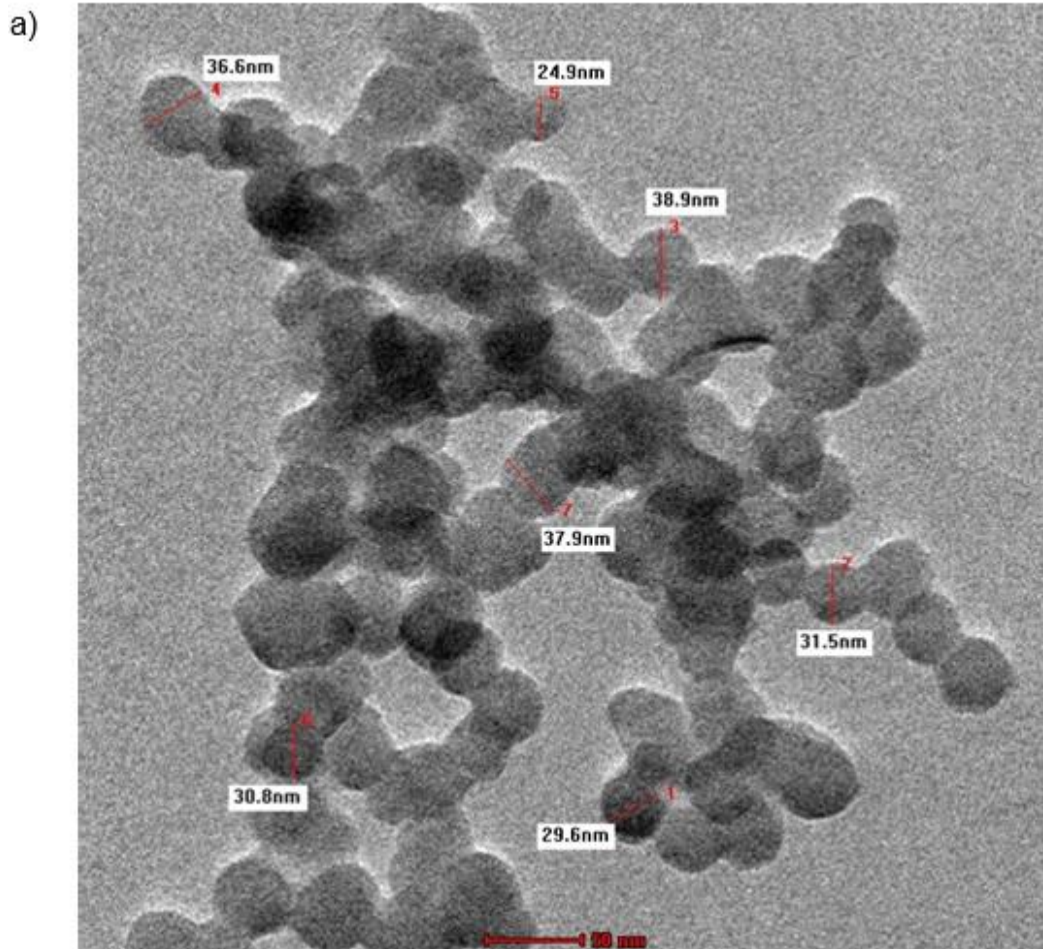


Fig. 3(c). Chemical precipitation of nanosilica from alkoxide precursors



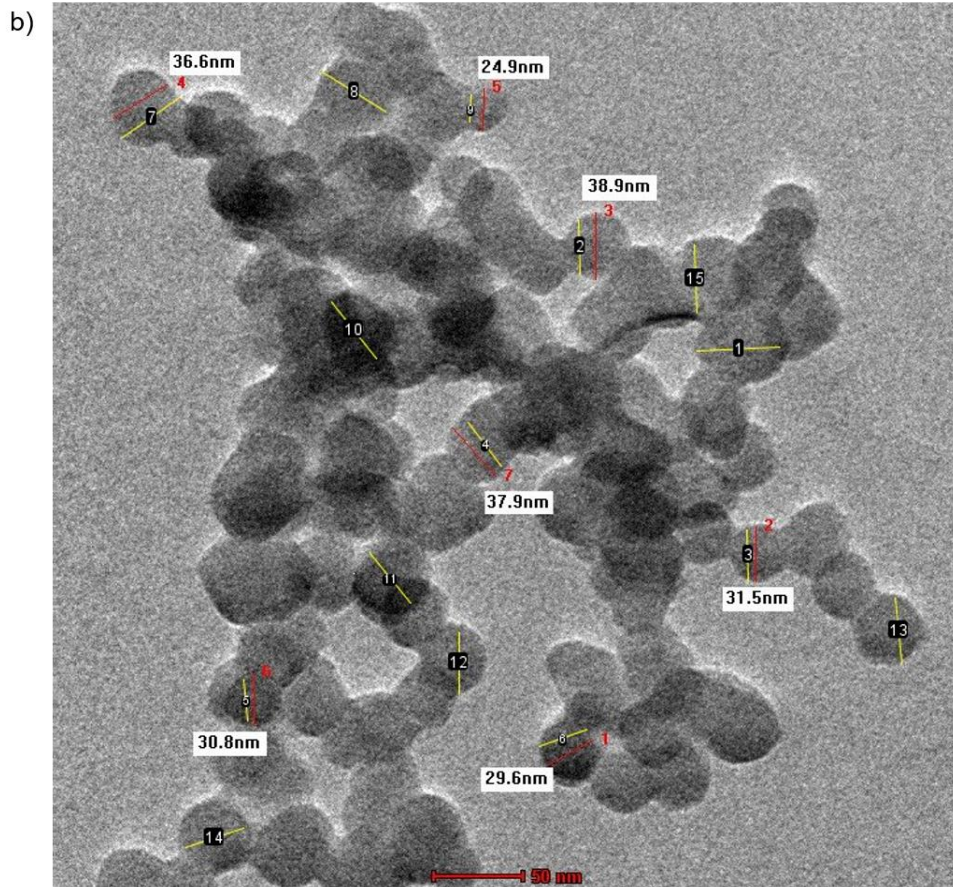


Fig. 4. (a) TEM image of Silica Nanoparticle synthesized from tea factory generated wood ash, (b) Average particle size of with Gaussian fitting curve on image J

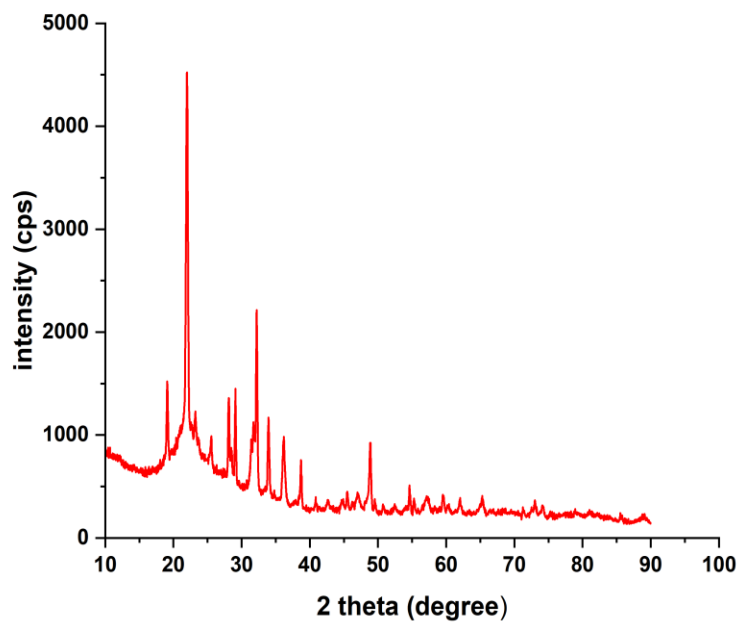


Fig. 5. X-ray spectra showing crystalline nature of Silica nanoparticles

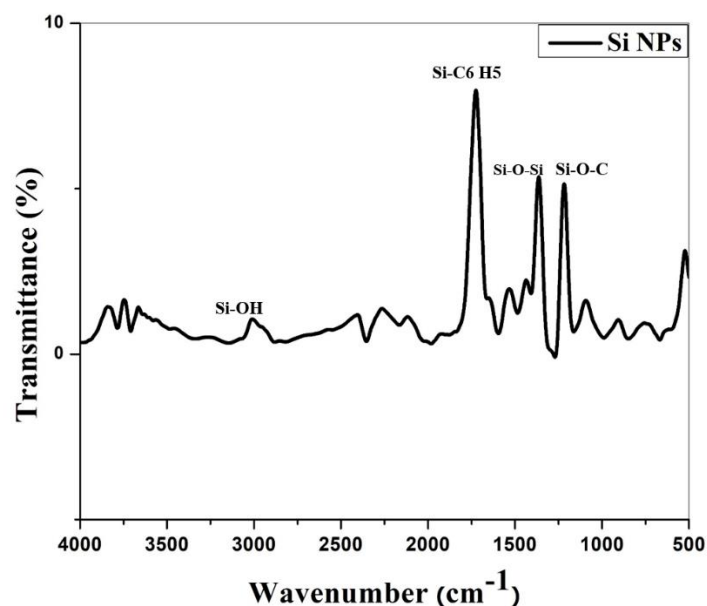


Fig. 6. FT-IR image showing functional group of nanosilica particles

3.6 FT-IR (Fourier Transform infrared spectroscopy)

The FTIR examines the functional group of synthesized nanosilica. In our current study, the peaks at 1110-1050 cm^{-1} represents the Si-O-C stretching, 1130-1000 cm^{-1} represents the Si-O-Si asymmetric stretching, at 1500-1430 cm^{-1} Si-C₆-H₅ stretching, 2250-2100 cm^{-1} Si-H stretching and 3700 – 3200 cm^{-1} Si-OH stretching. The values of these silica nanoparticles in FTIR spectra were in good agreement with the previous study [25].

4. CONCLUSION

This study aimed to provide a practical solution to one of the growing solid waste ie, wood ash generated in a large quantity by tea factories. In this research work, through a series of experiments, optimal conditions in the sol-gel method were evolved to effectively synthesize nanosilica from wood ash. The characterization results indicate that nanosilica is in the size range of 25 to 50 nm, spherical in shape and crystalline in nature. Overall, this study demonstrates that sol-gel method is an effective technique to transform wood ash generated from tea factories into high value nano silica particles with a potential of varied applications in agriculture and environmental remediation.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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