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Synthesis of Silica Nanoparticles from Rice Husk by Eco-Friendly Technique

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Abstract

The development of techniques for extracting silica from different waste products is a result of the use of silica nanoparticles (SNPs) in a variety of industrial products. Rice husk is an agricultural waste that contains a lot of amorphous silica and was used for synthesis. However, the color and purity of the result may suffer if metal ion contaminants and unburned carbon are present in the rice husk component. The current work aims to refine the silica extraction process, enhance its purity, and ultimately generate the highest-quality SNPs from agricultural waste using an environmentally benign manner. Hydrochloric acid was used in this work to assess the pre-treatment's impact on rice husk. In an electric furnace set at 700^{0}C, a controlled calcination procedure was used to extract silica. The hydrochloric acid treatment demonstrated the best treatment, according to the results obtained after SEM, FTIR, and XRD investigations. After that, this product was exposed to the sol-gel technique of chemical precipitation to produce silica nanoparticles. The achievement of producing silica nanoparticles with an average size of 200 ± 20 nm, spherical shape, and 98% purity is indicative of successful production.

***Key words*:** Rice husk, Silica nanoparticles, Calcination, SEM, FTIR, XRD

In a wide range of technical applications, including industrial manufacture, packaging, drug delivery, adsorption, biosensing, and catalysis, silica nanoparticles (SNPs) are widely utilized in nanomaterials. Much interest in SNPs stems from their high surface area to volume ratio, low toxicity, high chemical and physical stability, and simple surface chemistry that allows them to be combined or functionalized with a variety of functional species. or molecules. Sodium silicate is used as a source of silicon in the industrial production of silica. However, sodium silicate, which is obtained by fusing quartz sand and sodium carbonate at 1300 °C, not only requires a lot of energy but also additional purification [1] and may also be the cause of widespread environmental pollution. The purpose of using nanomaterials is to increase the efficiency and sustainability of agricultural practices that produce less waste and require fewer inputs than traditional methods and products [2]. The different chemical, optical, mechanical and magnetic properties of nanoparticles compared to bulk materials are due to their larger surface areas [3]. The synthesis of nanoparticles (NPs) by physical and chemical methods is expensive, labour-intensive, time-consuming, and energy-intensive. Stabilizing agents and toxic reduction are harmful to the environment and living beings [4].

Certain plants, such as those in the families *Ekvisetaceae*, *Graminae*, *Cyperaceae* and *Poaceae* are known to have significant concentrations of biogenic silica in the form of hydrated silica (SiO_2. nH_2O) that is deposited in their tissues [5-6]. Plant species with high silica content stored five to twenty percent of their dry weight of tissue. Annually approximately 600 million of rice are produced in the world. India is second with a total production of 120 million tons.

An average of 20% of rice husk ash is produced from 24 million of rice husk, which is an excellent source of silica [7]. Rice husk is so widely available that it is now profitable to use it as a source for other commercial applications. Currently, relative humidity has no commercial value; instead, it is usually burned in open areas, posing a threat to the environment and causing disposal problems [8]. In this work, we concentrated on producing very pure amorphous silica particles from RHA and then employed a delayed gelation approach to produce stable SNPs. The flint was extracted using two distinct techniques in order to accomplish this purpose. Rice husks are first treated with mineral acids (1 N hydrochloric, nitric, and sulfuric acid), and then they are carefully burned. Secondly, elimination with controlled burning without any pretreatment. Freeze drying and delayed gelation were used to create SNPs. FT-IR, SEM, N2 absorption-desorption, XRF, and other techniques were all used to completely characterize the products' structural qualities. The highly reactive surface and volume, chemical and physical stability, low toxicity, and straightforward surface chemistry of silica nanoparticles [9]. It is frequently utilized in medicine administration, cancer therapy, food and agriculture, packaging, industrial production, ceramics, and the synthesis of high molecular-weight composite materials [10]. As a result,

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creating a practical technique by which rice husk produces silica nanoparticles takes 1 hour. The assessment and management of nanoparticle synthesis and its uses depend on nanoparticle characterization. Several methods are employed to do this. The following: techniques aid in the resolution of several factors, including morphology, particle size, shape, crystallinity, and surface area. Utilizing rice husks as a source of SNPs can benefit the environment and the economy because it reduces waste management issues and makes extensive use of low-value agricultural byproducts.

MATERIALS AND METHODS

Rice husk washing, acid treatment, and heat treatment

To remove dust, additional impurities and dissolved particles the rice husk has been properly washed in drinking water. In a hot air oven, the cleaned rice husks were dried for four hours at 110 °C. The acidic 1 N HCl solution was refluxed into dry rice husks at 70 degrees Celsius. The sample was stored for approximately 24 hours after cooling to room temperature. After the sample and decantation, it was thoroughly washed in hot distilled water until the wash was acid-free. The rice husk underwent a 24-hour drying process at a temperature of 110 °C in a hot air oven, following its drainage and heating under a reflux. Subsequently, the dried rice husks were subjected to a 4-hour heating process at 700 °C in a muffle furnace, resulting in the production of ash.

Synthesis of silica nanoparticles

The 2.5 grams of silica obtained from the rice husks have been mixed with 250 milliliters of 0.5 M NaOH aqueous solution to produce sodium silicate by dissolution. The mixture was then cooled to 40 °C for 24 hours, where it would be continuously stirred. Subsequently, the resulting solution was filtered to remove any impurities that were not reactive. The concentrated H_2SO_4 was introduced and stirred vigorously until the pH reached 7 as soon as the clear sodium silicate solution filtrate cooled to room temperature. This process allowed for the precipitation of silica by neutralizing the sodium silicate with dilute sulfuric acid. The mixture was aged for 24 hours and continuously stirred for a total of 24 hours to ensure the gradual precipitation of silica gel. After thoroughly rinsing the precipitated silica with distilled water to eliminate any remaining alkali, it underwent a drying process in a hot air oven at a temperature of 105 °C for 18 hours.

Characterization of biosynthesized nanoparticles

Absorbance analysis using UV-visible spectrophotometer

Characterization of nanoparticles using a UV-visible spectrophotometer. To create the sample, silica nanoparticles which are 30mg were mixed with 5 ml of ethanol. A quartz cuvette with a path length of 1 cm was used as a reference for the ethanol. The absorbance of the sample was measured at various wavelengths ranging from 300 to 650 nm, and the corresponding wavelength and absorbed radiation values were recorded as number 039. By plotting the function of wavelength as absorbance, a spectrum was obtained. The wavelength at which the maximum absorption (λmax) occurred was determined from the graph.

Morphology of silica nanoparticles

A research investigation was conducted to examine the morphology of silica nanoparticles. To analyse the morphological properties of these nanoparticles, a specific method was employed. An aluminum tray with a diameter of approximately 1 cm was positioned in the sample after it had been thoroughly cleaned to eliminate any traces of surface oils or dirt. The aluminum tray was affixed with double-coated conductive carbon tape, resembling the adhesive nature of sticky glue. To achieve conductivity, a minute layer of dried substance ~0.2 mg was delicately spread onto the adhesive surface. Following a duration of 90 seconds, a fine mist of palladium was sprayed onto the sample, rendering it conductive. The sample holder is then detached from the sputter cover and carefully positioned within the vacuum chamber of the scanning electron microscope (SEM). To capture a precise depiction of the silica nanoparticles' structure, magnification ranging from one to thirty thousand times was employed, with a sample size of ten mm deemed necessary for optimal results within the working range.

X-Ray diffraction for phase identification

The X-ray diffraction pattern was captured within the high angle 2 theta range (5-70°) while employing a glass sample holder that had been evenly coated with approximately 1 gram of silica nanoparticles. Subsequently, this coated holder was introduced into the scanning chamber. The experimental setup was configured with a predetermined step length of 0.001 seconds and a sweep speed of 0.3 degrees per minute, ensuring accurate data acquisition.

Functional group analysis is made possible by Fourier transform infrared spectroscopy (FTIR)

The potassium bromide pellet method was employed to obtain Fourier transform infrared spectra using a Vertex 70 spectrometer equipped with a digital detector. To prepare the sample, a 0.1% concentration was mixed with 250 mg of powdered potassium bromide. The mixture was then ground using a pestle and mortar. Subsequently, the pulverized sample was pressed into a pellet-forming nozzle to create a 13 mm pellet. This pellet was placed in testing and exposed to transmission mode scanning between 4000 and 450 cm^{-1}, with a resolution of 4 cm^{-1} [11].

RESULTS AND DISCUSSION

Synthesis of silica nanoparticles

To attain the highest possible silica yield from RHA, the synthesis of silicon dioxide has been carried out under ideal conditions. Furthermore, the elimination of undesirable substances such as sand and silt occur during the washing of rice husks with potable water. This process ensures that the relative humidity levels remain free from soluble particles, dust, and other impurities. Subsequently, the dried RH is subjected to reflux with 1 N HCl, resulting in a transformation of the yellow hue of the hydric acid to a brown shade as the digestion process prolongs. To obtain pure silica powder and eliminate impurities, it is necessary to subject the relative humidity to treatment using an optimal solution like 1 N HCl acid. Additionally, an intriguing transformation in color occurs during the digestion process. As the digestion progresses, the initial yellowish hue of rice husks gradually transitions to a brown shade, which can likely be attributed to a chemical reaction between the acid and the metal present.

White amorphous rice husk (RHA) is produced through the combustion of dried regurgitated rice husks in a muffle furnace at a temperature of 700 degrees Celsius for four hours. This process results in the formation of a small quantity of residual carbon. Subsequently, sodium hydroxide and silica are combined in the dissolution stage to create sodium silicate. The resulting sodium silicate solution filtrate is characterized by its transparent, colorless, and viscous nature. The utilization of

sulfuric acid as a catalyst resulted in the formation of silica particles. Subsequently, under neutral pH conditions of 7, a thorough precipitation of silica from sodium silicate was observed.

$$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O$$
$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O$$
$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 \text{ (Silica oxide)} + Na_2SiO_3 + H_2O$$

The determination of silica yield was carried out after the extraction of the RHA sample using 0.5 N sodium hydroxide (NaOH). Subsequently, the nano-silica was precipitated by employing H_2SO_4, and any metal contaminants present in the regenerated silica were eliminated through refluxing with 1N HCl and dissolving in 0.5N NaOH under continuous magnetic stirring. Haq *et al.* [12] observed that various processing parameters, such as the solid-to-solution ratio, extraction time, and sodium hydroxide concentration, influenced the amount of precipitated silica. Ultimately, the researchers achieved the production of amorphous pure white nano silica by drying it in a hot air oven at 105 °C for 18 hours.

Fig 1 Biosynthesis of silica nanoparticles

Characterization of synthesized nanoparticles

Absorbance analysis (UV Visible spectrophotometer)

The UV-visible spectra of SNPs revealed a maximum absorption band edge at 310 nm absorption, which was reported to be 1.95. These optical characteristics, associated with the Si-O-Si bond, are consistent with findings from previous studies and suggest the presence of silica nanoparticles [13].

Morphological analysis of biosynthesized nanoparticles

SEM images of the SNP are presented in (Fig 10-10a) the image is magnified over 1500 times, revealing that the silica particles possess a non-spherical and non-uniform architecture. Another, Figure 10b displays an image of silica nanoparticles (SNPs) at a magnification of 100,000x. It is evident that the SNPs exhibit a round shape and have an average particle size of 200 ± 20 nm. Despite particle aggregation caused by gold sputtering, the nonconductive nature of silica facilitated the rapid accumulation of charges on the powder surfaces [14].

X-Ray diffraction for phase identification

The crystalline Structure of the sample was confirmed by the XRD pattern, as indicated by a peak in the absorption region between 20 = 31-45. This peak confirms the presence of silicon oxide particles and their crystalline structure. (Fig 11) illustrates the XRDt, which further supports the confirmation of the crystalline structure of the silica nanoparticles. The pattern is characterized by an absorption peak around 20 = 31-45°.

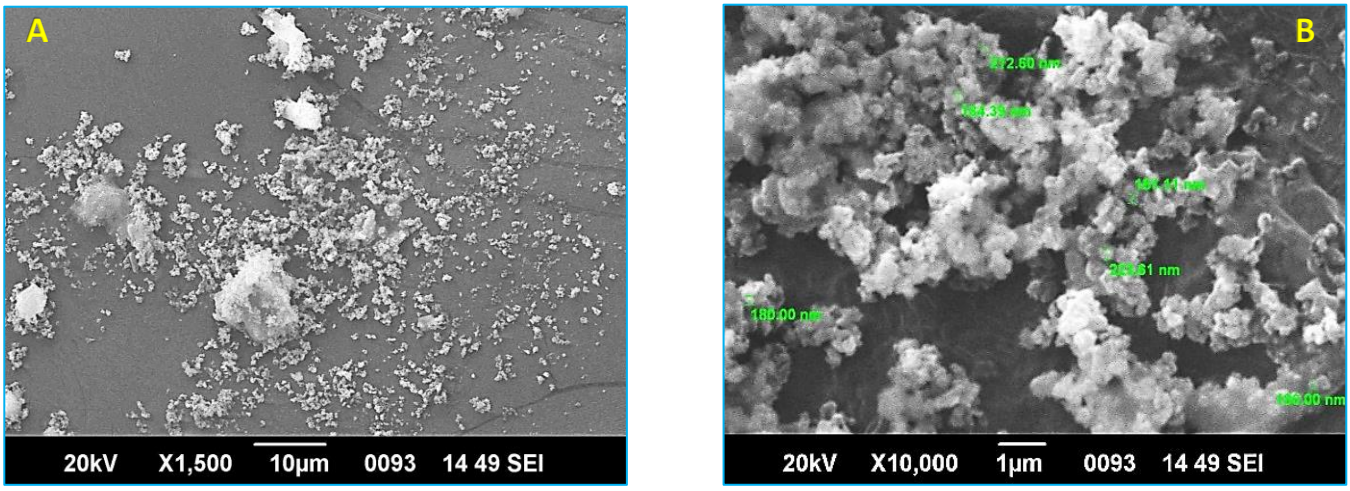

Fig 2 Scanning electron microscopy of (a) Magnification at 1500 (b) magnification at 10,000

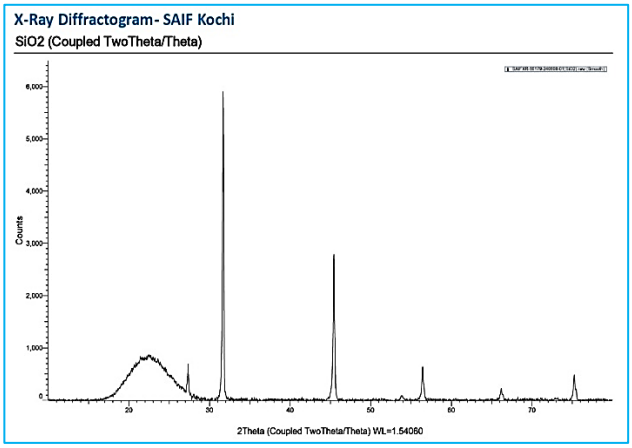

Fig 3 X-ray diffraction pattern of silica recovered from rice husk treated in 1 N HCl acid and burned at 700 °C

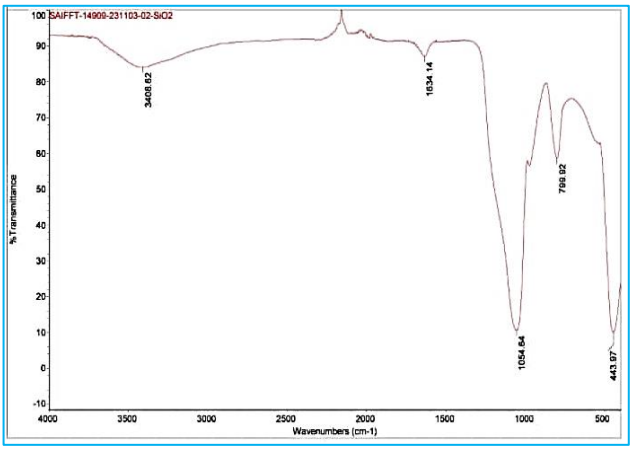

Fig 4 FTIR spectrum of silica nanoparticles

FTIR spectroscopy enables the examination of functional groups through the use of Fourier transform infrared analysis

FTIR spectroscopy was employed to identify the presence of authoritative clusters within the SNPs, as depicted in (Fig 12). According to Ghorbani *et al.* [5], the vibration signals observed at approximately 1054.64, 799.92, and 443.97 cm^{-1} can be attributed to Si-O-Si groups and are indicative of deviated stretching, symmetric stretching, and twisting, respectively. These three peaks represent the fundamental characteristics of the silica materials, which signify the successful synthesis of SNPs. The H-O-H bending vibrational absorption band in water is located around 1634 cm^{-1}, as reported by An *et al.* [15]. Within the spectra, the broad band between 2700 and 3700 cm^{-1} can be attributed to the presence of adsorbed water molecules, as observed by Kalapathy *et al.* [16]. The pure silica's IR spectrum was readily visible.

CONCLUSION

The synthesizing Process of silica nanoparticles from rice husk can be considered an environmentally friendly combination strategy. Utilizing rice husk, a readily available and inexpensive agricultural by-product, as a derivative of silica nanoparticles has significant positive effects on both the environment and the economy. These biosynthesized silica nanoparticles find various applications in the fields of agriculture and industry. The silica extract obtained through the slow gelation process yielded SNPs that were highly pure, exhibiting a round shape and an average size of 200 ± 20 nm. Additionally, these SNPs possessed a significantly large surface area, measuring up to 409 m2. The average pore size was approximately 10.89 nm, and the total pore volume was 0.95 cm3.g-1, indicating successful nanoparticle production. By utilizing agricultural by-products instead of quartz, the production of SNPs not only resulted in a value-added product that was both technically sound and aesthetically pleasing but also environmentally friendly.

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