# Green Nanotechnology: Harnessing Rice Husk Ash for Nano-Silica and Characterization Insights


## ABSTRACT

In the form of nanoparticles, silica is a significant inorganic component of rice husk. Consequently, it is feasible to extract high purity amorphous silica nanoparticles by straightforward thermo-chemical processes. So, in this study, an eco-friendly chemical treatment method (Green Synthesis) was used to try and manufacture amorphous silica nanoparticles from rice husk ash. I had done synthesizing silica nanoparticle in Dept. of Soil Science and Agricultural chemistry,TNAU,Coimbatore and the aim of this study is to characterize the silica nano particle and use it on agricultural crops. Selected region from X-ray diffraction analysis and Transmission Electron Microscopy, silica sample exhibited amorphous behaviour as seen in the electron diffraction patterns, whereas the Fourier-transform infrared Spectroscopy spectra primarily contained siloxane and silanol groups. Images obtained in Scanning Electron Microscopy (SEM) revealed the existence of primary nanoparticles with secondary microparticles, possibly as a result of their agglomeration. These silica nanoparticles can therefore be used in the fields of microelectronics, sensors, nano-additives and will be suitable on implication on agricultural crops.



***Keywords: Nanosilica . Rice husk ash . Amorphous silica .Silanol .Siloxane .*ζ(Zeta)**


## 1. INTRODUCTION

Due to its numerous uses in solar panels, concrete, insulators, refractory ceramics, and other products, silica-based nanomaterials have recently attracted attention [1]. Previous publications have described the preparation of silica materials utilizing a variety of extraction procedures, including solution precipitation, sol-gel processing, thermochemical redox reaction, and vapour-phase reaction [2, 3].

Tetraethyl orthosilicate (TEOS), sodium silicate, and silicon alkoxide are typically utilized as precursors of silica for mass manufacture. At high temperature (1300 °C), silicon alkoxide is traditionally derived from raw silica sandvia a multi-step carbo-thermal reaction method [4], while sodium silicate is extracted by heating quartz, an earth substance, with sodium carbonate [5]. Both of the aforementioned methods use a lot of energy and have environmental risks.

Additionally, these technologies produce greenhouse gases like $CO_2$, making them unsuitable for silica production in large quantities [6]. Furthermore, the primary raw material may become scarce if natural resources like quartz are used to produce silica. Therefore, in recent years, there has been an increase in interest among researchers in finding alternative sources of silica and ways to extract it. As a byproduct of agricultural practices, rice husk (RH) is widely accessible in the top rice-producing nations like India.

RH produces a large amount of rice husk ash (RHA) when burned as a fuel and offers extremely efficient thermal energy. It is uncommon for businesses to use RH as fuel, and when it is, a significant amount of RHA (around 20% of RH) is produced. Furthermore, disposal is the key issue for both RH and RHA, particularly for the rice milling sectors. In order to use RH/RHA for the manufacture of nanosilica, which typically resides in the amorphous phase in RH/RHA, numerous efforts have been made. Fernandeset al.[7]andShen[8]provided an overview of numerous techniques for extracting silica from RH/RHA and their long-term use in various industries.

Instead of sodium carbonate ($Na_2CO_3$), sodium hydroxide (NaOH) is preferable for the alkali-$SiO_2$ extraction method for producing silica from RHA because $Na_2CO_3$ releases an enormous amount of $CO_2$ during the extraction process, making the procedure unsustainable for the environment[13].

The recovered silica was then examined using XRD, FTIR, SEM-EDX and TEM to determine its Structural Composition. RHA Silica is the name given to the silica obtained from Rice husk ash.

"X-Ray Diffraction (XRD) has long been used to determine the atomic-scale structure of materials. This technique is based on the fact that the wavelength of X-rays is comparable to the distances between atoms in condensed matter"[21].

"FTIR spectrometry makes it possible to study the averaged properties of molecular species deposited on the surfaces of nanoparticles"[22].

SEM-EDX is used in Identifying the presence and location of nanoparticles in tissue sections[23].

"TEM is a technique that uses an electron beam to image a nanoparticle sample, providing much higher resolution than is possible with light-based imaging techniques. TEM is the preferred method to directly measure nanoparticle size, grain size, size distribution, and morphology"[24].

## 2. MATERIALS AND METHODS

### 2.1. Materials

Rice Husk was collected. All chemicals like HCl, NaOH, $H_2SO_4$etc. were obtained from Dept. of Soil Science and Agricultural chemistry, TAMILNADU AGRICULTURAL UNIVERSITY.

### 2.2. Methods

To obtain white color rice husk ash, dried black color rice husk was calcined at 550 °C for five hours. Majority of the inorganic content, silica, is left over when the RHA is calcined, whereas the organic components, lignin and hemicellulose, are destroyed by heat [14]. The husk was acid leached for three hours in a magnetic stirrer with a 1 M HCl (1:10 ratio) solution. After being washed with water to remove extra acid from the surface, the acid-leached husk was dried in an oven for an entire night at 80°C.

To extract the sodium silicate from the rice ash, dried rice husk ash was combined with 2N NaOH solution (1:8) and agitated in a magnetic stirrer for 120 minutes. To test for gel formation, the mixture was titrated against 0.6 M $H_2SO_4$. After gathering the solid material in the centrifuge tube, it was centrifuged at 6000 rpm for 3 minutes while being washed four times with alcohol and twice with water. The solid was calcined at 400°C to 440°C for four hours after being dried in an oven at 80°C overnight.

### 2.3.SILICA RECOVERY

The recovery of silica from RHA was performed in triplicate and calculated using

$$\text{Silica Yield } \% = (\text{Mass of Silica obtained}/\text{Mass of RHA}) * 100$$

### 2.4. CHARACTERIZATION STUDY

RHA-Silica's microstructural, morphological, and compositional analysis were carried out using XRD, FTIR, SEM as material characterization techniques.

## 3. RESULTS AND DISCUSSION

### 3.1. SEM

### Characterization

RHA-Silica's XRD pattern is shown in Fig 1. For the silica sample, a large hump at a 2ϴ angle of 27° was seen, demonstrating the sample's amorphous composition. Ma et al. [6]and Yalc and Sevinc[9]also got similar results when they isolated silica nanoparticles from rice husk and found a large peak centered at a 2ϴ angle of 27°. Other scanning angles from 5° to 80° showed no strong peaks, indicating the absence of any organized crystalline structure. According to An et al.'s[15]analysis on silica's very high disordered particle shape, this also showed that. Additionally, the Bragg diffraction peaks' Gaussian peak fitting was carried out. Broader de-convoluted peaks were produced after the entire Bragg diffraction peak set was fitted with Gaussian functions (Fig. 1). This finding demonstrates that the sample has the normal amorphous components but no organized crystalline phases. Sampath et al.'s [10]use of the Gaussian function for an X-ray diffraction analysis of amorphous and nanocrystalline structure revealed that a larger Gaussian peak denotes the presence of an amorphous component.

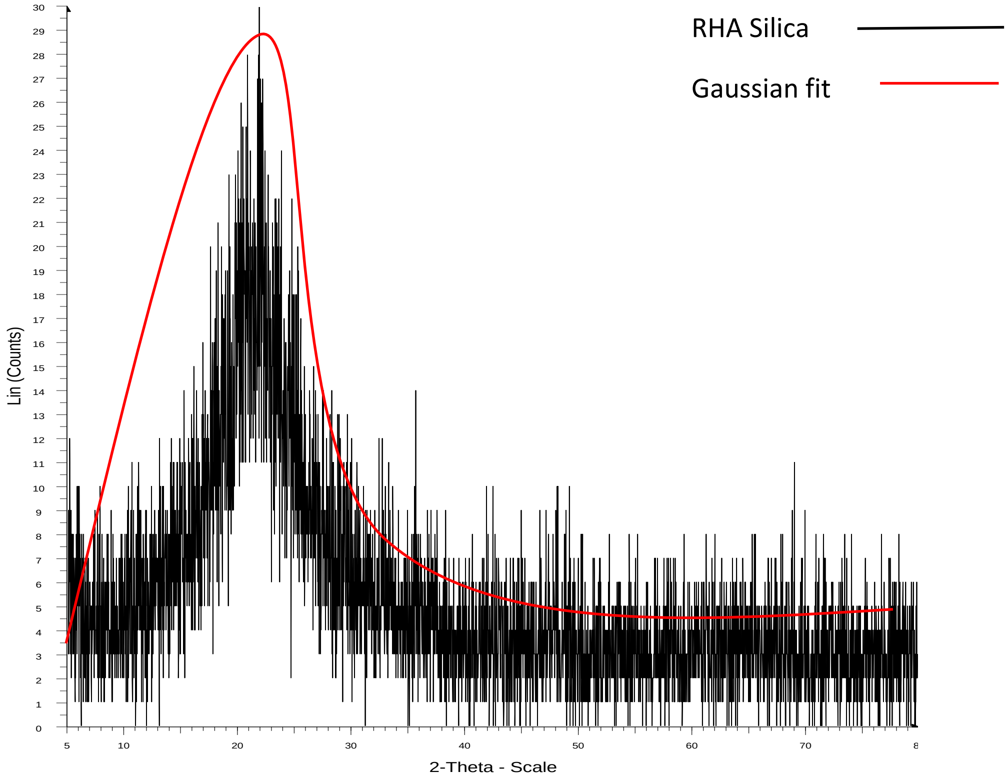


**Fig 1**. XRD of RHA Silica

### 3.2. FTIR Characterization

While bands at No.6 with position at 1640.16 for RHA-Silica sample is due to bending vibration of $H_2O$ molecule in the Si-OH group, bands at No.2 with position 3416.28 of RHA-Silica are attributed to stretching vibration of O-H group, respectively[16]. The Si-O-Si asymmetric stretching vibration was shown by the bands at No.7 with Position 1100.19. The Si-O bond's symmetric stretching vibration caused the wavenumber of No.10 to have band position 616.145. In contrast, the Si-O bending of the siloxane group for RHA-Silica caused the wavenumber of No.11 to have band position 468.617.

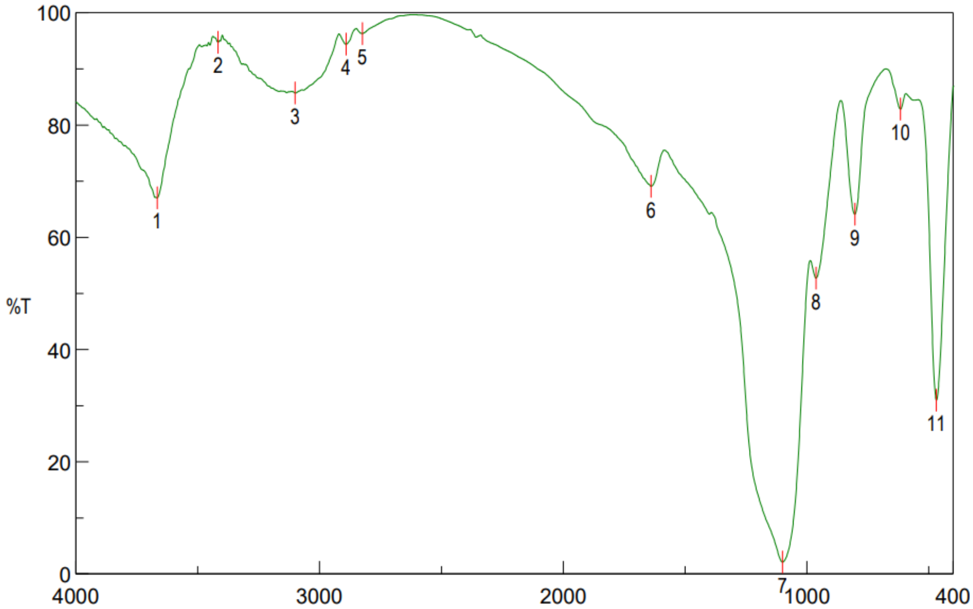


**Fig 2.** FTIR Spectra of RHA Silica

| No | Position | Intensity |
|---|---|---|
| 1 | 3666.02 | 66.985 |
| 2 | 3416.28 | 94.7105 |
| 3 | 3100.01 | 85.6836 |
| 4 | 2890.77 | 94.3866 |
| 5 | 2824.24 | 96.281 |
| 6 | 1640.16 | 69.0455 |
| 7 | 1100.19 | 2.0944 |
| 8 | 962.305 | 52.7116 |
| 9 | 803.206 | 64.0635 |
| 10 | 616.145 | 82.8119 |
| 11 | 468.617 | 30.9392 |

**Table 1.** Absorption bands of silica sample

| Band origin | Position/cm$^{-1}$ | RHA Silica |
|---|---|---|
| Bending vibration of Si-O | Siloxane group | 468.617 |
| Symmetric stretching vibration of Si-O | (Si-O-Si) | 616.145 |
| Asymmetric stretching vibration of Si-O-Si | | 1100.19 |
| Absorption of single broad band | Silanol group | - |
| Bending vibration of H-O-H | (Si-OH) | 1640.16 |
| Stretching vibration of O-H | | 3416.28 |

### 3.3. SEM, EDX and Particle size characterization

The SEM is a powerful tool used for taking SEM images. The SEM analysis helps to analyze the size of the suspended silica nanoparticles. The SEM creates the magnified images by using electrons instant light waves[17].SEM micrographs **Fig 3a**, particle size histograms **Fig 3b** and EDX peaks **Fig 3c** are showed. “Spherical $SiO_2$-NPs were observed with uniform surface morphology in RHA-Silica along with agglomeration. The agglomeration effect may be due to the dominance of strong cohesive intramolecular forces instead of gravitational force”[11]. The elemental composition analysis was performed through EDX study and the results are presented in Table 2. It was observed that only two strong peaks of O Kα and Si Kα were present in both RHA-Silica. The results obtained are in accordance with the results of Mourhly et al. [12], who worked “on mesoporousnano-silica. From particle size distribution histograms, it was noticed that average particle size of the $SiO_2$-NPs extracted from RHA was 17.80 ± 10.10 nm. However, it is worth mentioning here that a few secondary micro-particles were also observed may be due to the agglomeration effect”.

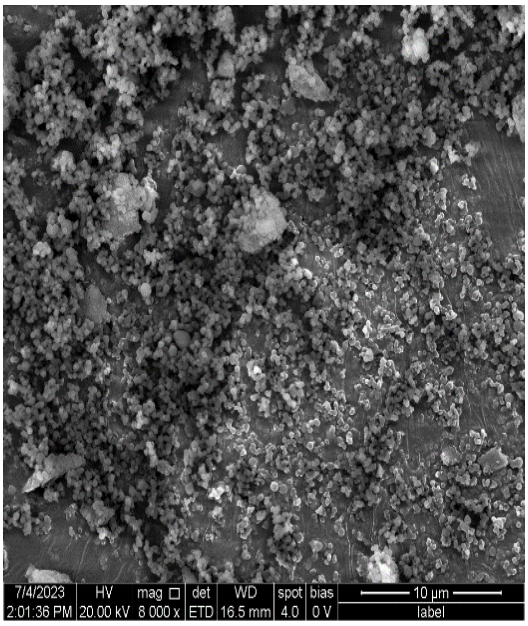


**Fig 3a.** SEM image

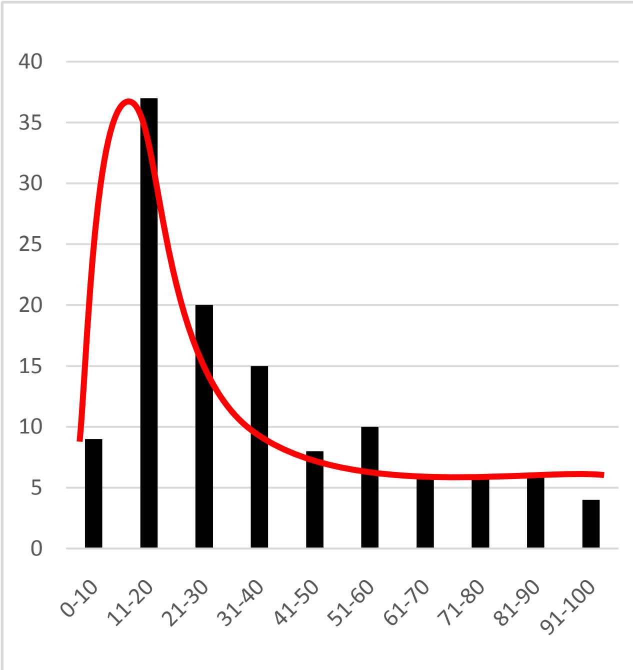


**Fig 3b.** Particle size

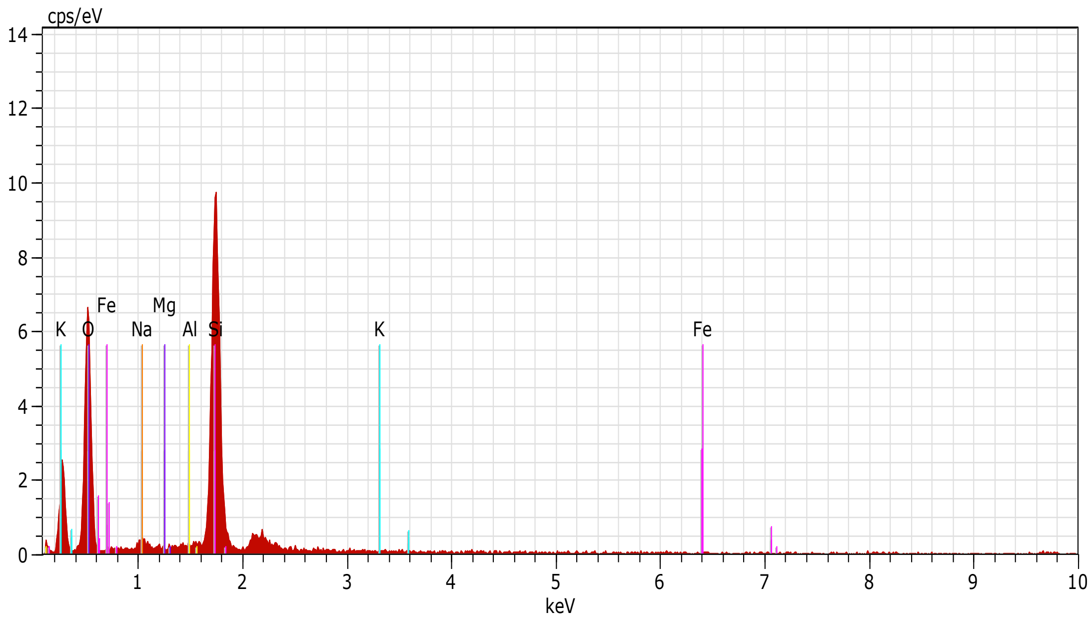


**Fig 3c**. EDX

**Table 2.**Elemental composition of silica obtained from EDX analysis

| Elements | RHA Silica | |
|---|---|---|
| | Weight % | Atomic % |
| O k | 63.7775.35 | |
| Si k | 32.77 | 22.05 |
| Na k | 2.04 | 2.04 |
| Al k | 0.46 | 0.32 |
| Fe k | 0.34 | 0.11 |
| Mg k | 0.12 | 0.09 |
| K k | 0.06 | 0.03 |
| Total | 100 | 100 |

### 3.4. TEM Characterization

In **Fig 4.**The microstructures of silica samples were also verified through FETEM and the TEM image clearly showed spherically shaped nanoparticles (size <200nm) with high agglomeration.“TEM micrographs revealed spherical morphologies of SiNPs with particle aggregation; this aggregation is possibly due to electrostatic interactions between particles and the imaging grid (mesh copper Formvar-coated grid) during drying process for TEM analyses. It has been established that size, ζ, and morphology may play an important role within the biological performance of different NM, especially with cellular interaction and cellular internalization”[18].“Cellular internalization of NM is strongly size dependent; nanoparticles with ≤ 80 nm exhibit high cellular uptakes, unlike particles with ≥ 85 nm, where the internalization is signicantly reduced. Additionally, these size features may have a signicant impact on the toxicity of the particles”[19]. Other studies have determined that “ζ possess important implications in the interactions between NM and cells. Findings of Chen et al.Finally, computational simulations have demonstrated that morphology and shape of NM potentiate cellular interactions/internalization, where spheroidal particles internalize 60% faster than other nanoparticles with different shapes”[20] .

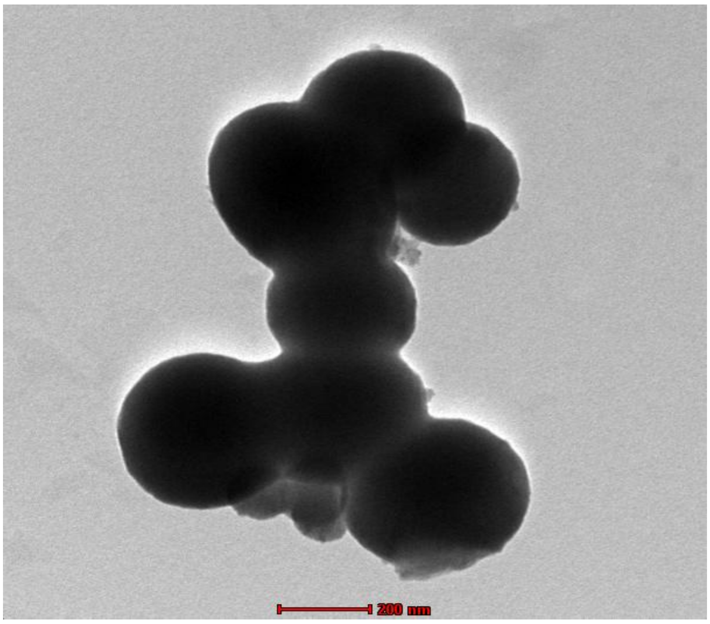


**Fig 4.** TEM Image of RHA Silica

## 4. Conclusion

This study demonstrated that spherical, nano-sized, thermally stable $SiO_2$ NPs could be synthesized using a green method. silica extraction ($SiO_2$) utilizing the acid-leaching and sol-gel methods with rice husk base material was accomplished satisfactorily.It may be said that rice husk ash, which was used to extract silica which has amorphous crystal structure, is a potential and healthier green resource.The suggested process for making nano silica from rice husk is straightforward, suitable for large production, and reduces waste disposal issues. This method is eco-friendly, one-pot, cheap, productive, and leads traditional synthesizing methods.

Several The shape, purity, crystal structure, stability, thermal, and optical characteristics of biosynthesized $SiO_2$ were investigated using characterization methods. Considering all of these aspects, the green synthesized $SiO_2$ NPs were compared with the chemically synthesized $SiO_2$ NPs.

1. FTIR analysis for RHA $SiO_2$ NPs demonstrated two dominant peaks at roughly 616.145 $cm^{-1}$ and 468.617 $cm^{-1}$, which are associated with the asymmetric stretching vibration of the Si-O bond.
2. According to the XRD measurements, RHA $SiO_2$ NPs had the peak at $2\theta = 27°$, this was due to phytochemical presence in green synthesis.
3. Due to the fact that in the green synthesis method, different phytochemicals are participating in reducing capping and stabilizing the process. Besides, the FESEM images revealed that RHA $SiO_2$ NPs are spherical in shape with a minimum degree of agglomeration, and the NP sizes were between 17.80 ± 10.10 nm, respectively.


## ACKNOWLEDGEMENTS

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## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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