

Extraction, Characterization and Antifungal Activity in Biosilica Nano-Particle from Rice Husk

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ABSTRACT

This study demonstrates that rice husk as a sustainable, cost-effective source of biosilica, promoting waste reduction and materials sustainability. Biosilica nano-particles were extracted using a simple and environmentally-friendly method, characterized through FTIR and SEM analysis to determine the key chemicals and the morphology respectively and to show antifungal properties against five different fungal species. The results reveal the presence of silanol and siloxane groups. According to the SEM image analysis, the produce has irregular shape and rigid surface which contributed to the Si-O-Si frame work. Antifungal assay exhibited that biosilica inherent antifungal property makes it potential antifungal agent.

Keywords: extraction, characterization, nano-particles, rice husk, and antifungal property.

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I. INTRODUCTION

Globally, Nigeria is the second largest producer of paddy after China in the world. In Nigeria approximately 20 million tons of paddy is produced giving 24 million tons of Rice husk and 4.4 million tons of Rice Husk Ash every year, producing a huge amount of solid waste. Paddy consist of 20-22% of Rice husk [1]. Rice husk has a high calorific value of 2938.86 Kcal/kg. It is the cheapest and the most common renewable energy source, however by burning of rice husk it becomes hazardous for the environment as well our health if it not being disposed properly. Burning of rice husk converts the organic component present into 20% carbon dioxide, air and ash rice husk has a high silica content and it does not burn with an open flame or decompose completely [2, 4]. The concentration of silica in Rice Husk Ash is influenced by the fluctuations of geographical and environmental conditions, including factors such as climate, harvesting season, soil composition and the quantity of fertilizer used in the cultivation. The dry Rice Husk contains of 70-85% organic matter such as lignin, cellulose and hemicellulose which makes it suitable for use in feedstock in bioethanol. From the 70-85%, 21.44% of lignin, 32.24% cellulose, 21.34% of hemicellulose and the inorganic remainder 20-25% which mainly consist of silica, Silicon Dioxide or silica, exists in two forms, amorphous and crystalline. Naturally silica is rice husk ash is amorphous, however when combustion above 650°C silica changes its phase from amorphous to crystalline [3]. Silica amorphous state has a large surface area and is a high purity than crystalline silica which comes under the IARC Group 1 carcinogenic agent [5,6]. Amorphous silica is known for its wide range of industrial application, such as coatings, plastics, rubber, electronics, abrasives, refractories and optics. It also serves as a pivotal precursor for synthesis of wide range of fine compounds. Silica, or silicon dioxide (SiO₂) is a naturally occurring compound widely found in the earth's crust. It plays a crucial role in various industries including, electronics, construction, and pharmaceuticals. The demand for nano-sized silica particle has increased significantly due to their unique properties, which include high surface area, reactivity, and the ability to enhance the mechanical properties of materials[7,8]

Rice husk, a byproduct of rice milling is an abundant agricultural waste that is often discarded or burned, leading to environmental pollution. It is primarily composed of cellulose, hemicellulose, lignin and silica, with silica content ranging from 15% to 25%. The conversion of rice husk into nano-silica not only provides a sustainable solution for waste management but also offer a potential low-cost source of silica for various application[2]. Research has shown that silica extracted from rice husk exhibits unique properties that can be utilized in various fields, such as bioengineering, material science, and nanotechnology[9]. The extraction process involves several steps, which yield silica in nano-particles. The characterization of these nano-form is essential to determine their physical, chemical, and structural properties, creating way for their

application in numerous industries [10]. The study thus aim to extract, characterize and determine the antifungal activity in Biosilica nano-particles from rice husk

II. MATERIALS AND METHODS

2.1 Sample collection

The rice husk used for the extraction of silica was collected from rice mill in Afe Babalola farm (Industrial Parks), Ado-Ekiti, Ekiti state.

2.2 Materials

Rice husk (200 g) was washed and air-dried. The chemical reagents used are sodium hydroxide (AR, Kermel, > 99%), hydrochloric acid (36%, Loba Chemie), ethanol (AR, JHD,99%), N-[3-(trimethoxy silyl)propyl]ethylenediamine (sigma Andrich, > 98%), sulfuric acid (98% purity, J. T. Baker), chlofoam, hydrogen peroxide, potassium iodide, sodium thiosulphate, starch indicator, wij's solution and distilled water.

2.3 Isolation of silica

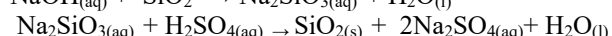
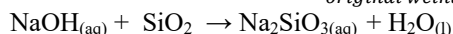
2.3.1 Pre-treatment

One hundred fifty grams of rice husk obtained from ABUAD Industrial Park Mill, Ado-Ekiti, Ekiti State, Nigeria, was washed thoroughly with distilled water and air-dried for 3 days before pretreated with HCl (0.2 M; 500 mL) at 100 °C for 90 min with adequate stirring. Upon cooling to ambient temperature, it was filtered and washed with cold distilled water before air-dried for 24 hours.

2.3.2 Extraction of biosilica

Fifty grams of the pretreated rice husk was ash in a muffle furnace (model SX2 – 4 – 10) at 700 °C for 12 hours using the AOAC method. The ash content (AC) was determined by Eq. 1. Twenty grams of the ash was weighed into a beaker (500 ml) followed by the addition of 100 mL NaOH (1 M) and covered with wash glass. It was heated on a magnet stir- rer at 80 °C for 1 h and 30 min with constant stirring. The solution was filtered, and H₂SO₄ (60 ml; 2 M) was added to the residue dropwise to adjust the reaction medium towards neutral pH and monitor with pH meter while stirred continuously until the biosilica was precipitated. The suspension was centrifuged (model 80 – 2) at 3000 rpm for 15 min. The precipitated native biosilica (NS) was washed with distilled water four times and dried in a thermostat oven (model:DHG – 9101 – OSA) at 80 °C for 6 hours.

$$\% \text{ ash (dry basis)} = \frac{\text{weight after ashing} - \text{tare weight of crucible}}{\text{original weight of sample}} \times 100. [\text{eq.1}]$$



Scheme 1.; Equation for the extraction of biosilica from rice husk.

2.4 Characterization of biosilica

2.4.1 FT-IR Spectroscopy analysis

Fourier transformed infrared (FTIR) spectra of and biosilica nanoparticles are recorded by a Bruker spectrophotometer using the KBr pellet procedure for the sample. Prior to the measurement, the KBr tablets were dried at 102 °C for 3 h to get rid of moisture.

2.4.2 Scanning electron microscopy(sem)

The surface morphology of biosilica nanoparticles was investigated by scanning electron microscope (TESCAN Vega 3LHM) operating at voltage of 200 kV.

2.5 Antifungal activity in the biosilica

Antifungal efficacy of biosilica was evaluated against five different fungal species (*Aspergillus flavus*, *Aspergillus parasiticus*, *Aspergillus scierbtiicarbonari*, *Rhizopus oryzae*.and *Fusarium oxysporum*) at varying concentrations (0.2 to 0.8 g/mL), measured by the diameter of zones of inhibition (mm).

III. RESULTS AND DISCUSSION

3.1 Extraction of biosilica

The pretreatment removes dirty and other impurities such as fat and oil from milling machine that adhered to the rice husk surface for sustainable production of pure biosilica from rice husk [11]. The mercerization of the ash by sodium hydroxide soften the ash particles to transport the biosilica nano-particles component into the extracting solvent in form of sodium silicate. As the PH was adjusted from basic to neutral

with addition of H_2SO_4 , the precipitate of sodium silicate started to form and also the addition of H_2SO_4 removes the traces of metal impurities in rice husk ash. The sodium silicate was washed with distilled water so that the biosilica particles are dispersed uniformly and removes impurities and strengthen the gel. The gel was repeatedly washed and centrifuged to remove residual impurities and then heated at 80°C in the oven to form amorphous biosilica which was white in color that suggested complete removal of carbonaceous compound present in the rice husk ash.



Figure 1: biosilica obtained after extraction process.

3.2 Fourier transform infra-red ray spectroscopy

FT-IR analysis was employed to identify surface functional groups on extracted silica. Results are shown in Figure 2. An absorption band between $3200\text{--}3500\text{ cm}^{-1}$ represents O-H groups of Si-OH and absorbed water molecules [11]. The absorption band at $1519\text{--}1665\text{ cm}^{-1}$ was associated with absorbed water molecules [14]. Strong absorption bands at $1039\text{--}1062\text{ cm}^{-1}$ and 790 cm^{-1} corresponded to asymmetric stretching vibrations of siloxane (Si-O-Si) groups and Si-O, respectively. Peaks at $447\text{--}565\text{ cm}^{-1}$ were linked to Si-O-Si structure bending vibrations [4,11]. The absorption band at around $1397\text{--}1454\text{ cm}^{-1}$ Si-O stretching vibration. The absorption peak at 952 cm^{-1} in MS spectrum was ascribed to Si-C bond stretching vibration resulting from modification. The absorption peak at 1327 cm^{-1} was associated with C-O stretching [14].

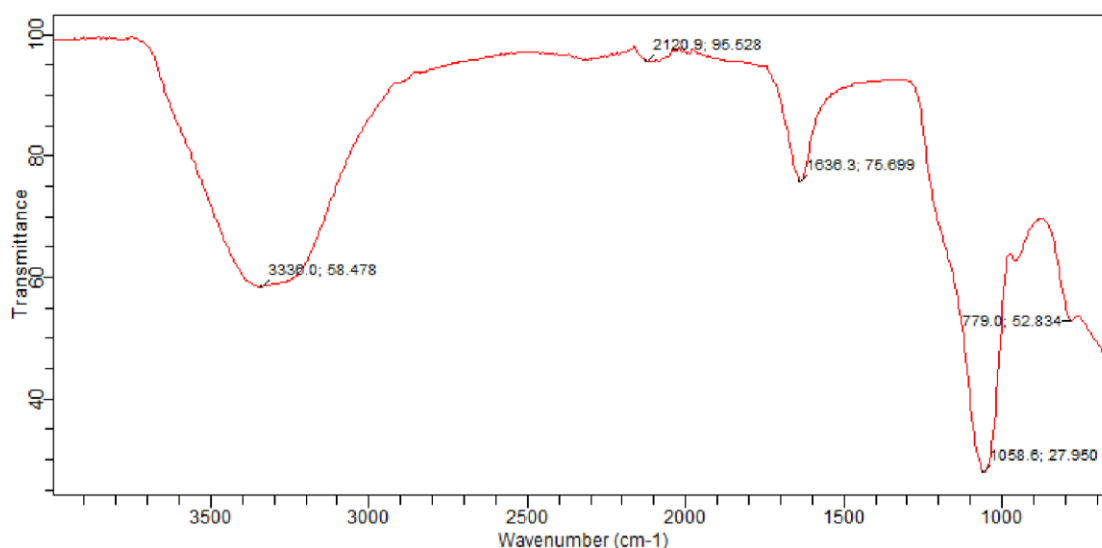


Figure 2: Fourier transform infrared spectroscopy (FT-IR) spectra of the extracted biosilica,

3.3 Scanning electron microscopy

SEM analysis is a vital tool in investigation of samples surface morphology. The SEM images recorded for the sample are shown in Figure 3 showed that extracted silica has irregular shape, rigid and rough rocky surface. The surface rigidity of extracted silica might be attributed to Si-O-Si framework which agreed with literature report [16].

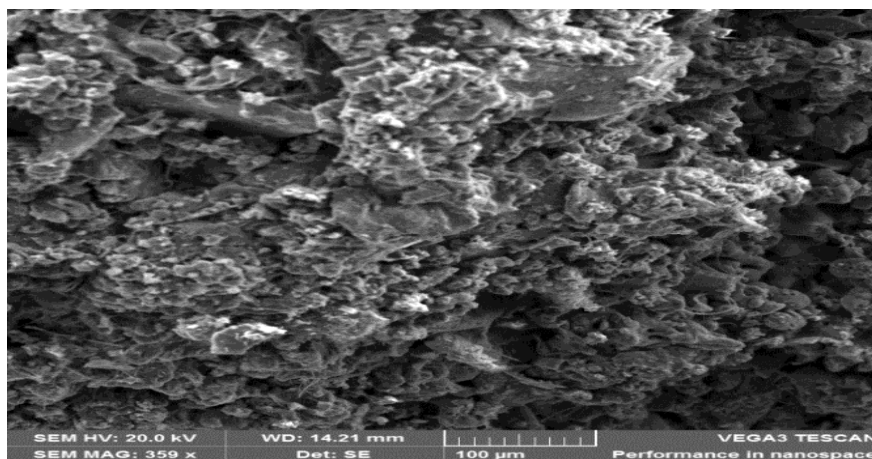


Figure 3: Scanning electron microscopy (SEM) of extracted biosilica

3.4 Antifungal activity

Table 1: Antifungal Activity in Biosilica

Test Organisms	0.2 g/mL	0.4 g/mL	0.6 g/ML	0.8 g/ML
<i>Aspergillus flavus</i>	21.00	9.00	8.00	9.00
<i>Aspergillus parasiticus</i>	–	–	–	–
<i>Aspergillus scirbtiicarbonari</i>	8.00	8.00	9.00	10.00
<i>Rhizopus oryzae</i>	10.00	–	–	–
<i>Fusarium oxysporum</i>	8.00	10.00	11.00	12.00

Fungal infections pose significant threats to human health and agriculture, necessitating the development of novel antifungal agents. Biosilica, a naturally derived form of silicon dioxide often sourced from organisms like diatoms, has garnered interest due to its unique properties and potential antimicrobial activity. Table 1 presents data evaluating the antifungal efficacy of biosilica against five different fungal species at varying concentrations (0.2 to 0.8 g/mL), measured by the diameter of zones of inhibition (mm). The findings demonstrate that biosilica exhibits selective antifungal activity depending on fungal species and concentration. *Aspergillus flavus* showed the highest susceptibility at the lowest concentration (0.2 g/mL) with a 21.00 mm zone of inhibition, followed by reduced activity at higher concentrations. This inverse trend may suggest that biosilica's effectiveness against *A. flavus* is concentration-sensitive or due to possible aggregation at higher concentrations limiting bioavailability, as reported in similar studies [18]. In contrast, *Aspergillus parasiticus* showed complete resistance across all concentrations, indicating possible innate resistance or inadequate penetration by biosilica, aligning with findings by Sharma *et al.* (2020), who observed species-specific resistance mechanisms to silica-based antimicrobials. *Aspergillus scirbtiicarbonari* responded modestly, with inhibition zones ranging from 8.0 to 10.0 mm. The gradual increase in inhibition with concentration suggests a dose-dependent antifungal effect. *Rhizopus oryzae* showed inhibition only at 0.2 g/mL (10 mm), indicating either a narrow window of susceptibility or instability of biosilica efficacy over higher concentrations. Lastly, *Fusarium oxysporum* demonstrated increasing sensitivity with higher concentrations, from 8.0 mm at 0.2 g/mL to 12.00 mm at 0.8 g/mL, reflecting a clear concentration-dependent response. This is consistent with reports by Duraipandiyar *et al.* (2011), which highlight the sensitivity of *Fusarium* species to silica-based biocontrol agents. Overall, the antifungal effect of biosilica varies across fungi and concentrations, suggesting its potential as a selective antifungal agent. Further studies are needed to elucidate mechanisms and optimize its application [19].

III. CONCLUSION

This work illustrates that rice husk can be successfully prepared highly pure amorphous biosilica nanoparticles and just not only agricultural waste product, but a valuable by product and composed of antifungal property. The chemical group in biosilica are comprises of silanol and siloxane as confirmed by FTIR analysis. The SEM analysis confirmed irregular shape, rigid and rough rocky surface. The surface rigidity of extracted biosilica might be attributed to Si–O–Si framework. The antifungal screening of biosilica against selected fungal pathogens reveals its selective and concentration-dependent efficacy. *Fusarium oxysporum* and *Aspergillus scirbtiicarbonari* demonstrated progressive sensitivity with increasing concentrations, indicating the potential of biosilica as a dose-responsive antifungal agent. *Aspergillus flavus* showed high susceptibility at lower

concentrations, though the efficacy declined at higher doses suggesting a possible saturation or aggregation effect. *Rhizopus oryzae* responded only at the lowest dose, while *Aspergillus parasiticus* exhibited complete resistance, underscoring the organism-specific nature of biosilica activity. Overall, biosilica demonstrates promising antifungal potential, especially for managing fungal species like *Fusarium*, which are agriculturally and clinically significant can be used in food application and various other fields where the particle size of amorphous biosilica is required and to be generated at reduced cost and used as high-tech applications.

Conflict of interest

There is no conflict to disclose.

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