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EXTRACTION OF SILICA FROM RICE HUSK USING HEATING AND CHEMICAL TREATMENT

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ABSTRACT

Rice husk is an abundant agricultural by-product, and its controlled combustion produces rice husk ash (RHA), a rich source of amorphous silica (SiO₂). This study focuses on the extraction of high-purity silica from RHA through optimized chemical and thermal treatments. Raw rice husk was subjected to controlled burning at different temperatures (500–800 °C) to obtain silica-rich ash. Pre-treatment with acid was carried out to remove metallic impurities, followed by alkali leaching using sodium hydroxide (NaOH) or potassium hydroxide (KOH), which converted silica into soluble silicates. The silicate solution was then subjected to acid precipitation with sulfuric acid (H₂SO₄) or hydrochloric acid (HCl), leading to the formation of silica gel. The gel was purified through washing, drying, and calcination to obtain fine silica powder. Characterization was conducted using X- ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA), confirming the amorphous nature, surface

morphology, functional groups, and thermal stability of the extracted silica. The resulting material was tested for potential applications in adsorbents, catalyst supports, cementitious materials, and nanocomposites. The findings demonstrate the industrial significance of RHA-derived silica and highlight its potential as a sustainable raw material within the framework of

waste valorization and green chemistry.

KEYWORDS: Rice husk ash, silica extraction, sodium silicate, acid precipitation, amorphous silica, sustainable materials, waste valorization.

INTRODUCTION

Silica (SiO₂) is a versatile material with applications across industries including construction, electronics, and biomedicine (**Bose et al., 2019**). Conventional methods for silica production rely on quartz and sand, which are energy-intensive and environmentally unsustainable. Rice husk (RH), an agricultural by-product, has emerged as a promising alternative due to its high silicon content and availability (Banerjee et al., 2019). When burned under controlled conditions, RH produces rice husk ash (RHA) containing up to 85–95% amorphous silica (**Haider et al., 2022**). The valorization of RH not only provides a renewable source of silica but also mitigates environmental issues associated with agricultural waste disposal. Furthermore, RHA-derived silica can be used to synthesize advanced materials such as silicon carbide, silicon nitride, and SiO₂ nanoparticles (**Sankar et al., 2018; Aphane et al., 2020; Ismail et al., 2021**).

MATERIALS AND METHODS

SAMPLE COLLECTION AND PREPARATION

Rice husk was obtained from agricultural farms, cleaned thoroughly, dried at 100 °C, and ground into fine powder. The powdered husk was stored at 0–8 °C to prevent degradation.

EXTRACTION PROCESS

- Pre-treatment
- ♦ 10 g of RH was soaked in 0.5 M HCl for 2 h to remove metal oxides. The husk was then washed until neutral pH and dried at 100 °C.
- Combustion
- ❖ The pre-treated husk was combusted in a muffle furnace at 600–700 °C for 4–6 h, producing fine white RHA.
- Alkali Leaching
- ♦ 10 g of RHA was dissolved in 100 mL of 1 M NaOH at 90 °C for 1 h to form sodium silicate (Na₂SiO₃). The solution was filtered.

Acid Precipitation

❖ The sodium silicate solution was titrated with H₂SO₄ or HCl until gelation occurred. The silica gel was washed, dried, and calcined to obtain fine silica powder.

Fourier-Transform Infrared Spectroscopic Analysis

The structural characteristics of the polysaccharide samples were analyzed using a Fourier-transform infrared spectrophotometer (IR Affinity-1, Shimadzu, Japan). Each sample was mixed with spectroscopic-grade KBr powder, ground, and compressed into 1 mm pellets for measurement. FTIR spectra were recorded within the frequency range of 400–4000 cm⁻¹ at a spectral resolution of 0.5 cm⁻¹, averaging 64 scans per spectrum. Both native and pretreated samples were examined. Baseline correction and normalization were performed using the IR Solution software, and absorption bands at 1427 cm⁻¹ and 898 cm⁻¹ were used to calculate the crystallinity index.^[18]

To ensure consistency with microscopy observations, samples were analyzed without homogenization prior to FTIR measurement. The potential limitation of this approach was that surface cells of native rice husk might not represent the bulk material. To verify this, some untreated samples were finely ground and analyzed; negligible differences were observed between homogenized and non-homogenized spectra.

For each sample type, spectra were collected from three different subsamples and corrected using the Standard Normal Variate (SNV) method.^[19] The average of the three corrected spectra is reported for each sample type.

Thermogravimetric Analysis (TGA)

TGA was performed under a nitrogen (N_2) atmosphere at a flow rate of 150 mL/min. Rice husk and coconut pulp samples weighing between 0.5 and 1.0 g were pyrolyzed up to a maximum temperature of 700°C. Initially, the samples were heated to 110°C and maintained at this temperature for 30 minutes to eliminate moisture. Subsequently, heating was carried out at two different heating rates, 50 °C/min and 80 °C/min, until the final temperature was reached. The experiments were repeated for each biomass type using two particle size ranges: $dp_1 < 0.30$ mm and $0.30 \le dp_2 < 0.50$ mm.

Etraction of Silica

RESULTS AND DISCUSSION

Phytochemical screening of rice husk confirmed the presence of several bioactive compounds, including alkaloids, flavonoids, tannins, terpenoids, saponins, steroids, phenols, and glycosides, contributing to its antioxidant and antimicrobial properties (**Rao et al., 2023**). Biochemical estimations also revealed the presence of carbohydrates and proteins, highlighting the nutritional and medicinal value of RH.

The chemical treatment yielded high-purity silica, with XRD patterns confirming its amorphous structure. FTIR analysis identified Si–O–Si stretching vibrations, while SEM showed porous surface morphology suitable for adsorption and nanocomposite applications. TGA demonstrated thermal stability, confirming its suitability for industrial processing.

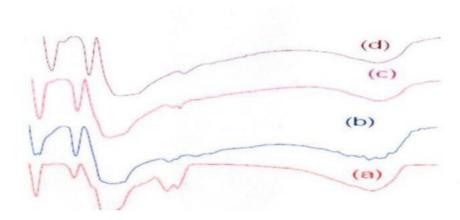


Fig 1: FTIR Analysis.

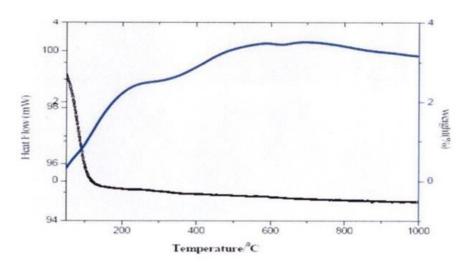


Fig 2: TGA



Fig 3: Test for Carbohydrate.



Fig. 4: Test for Protein.



Fig. 5: Test for Alkaloids.



Fig. 6: Test for Flavonoids.

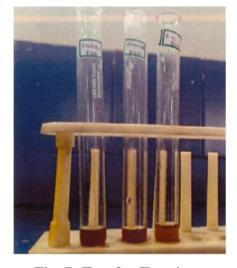


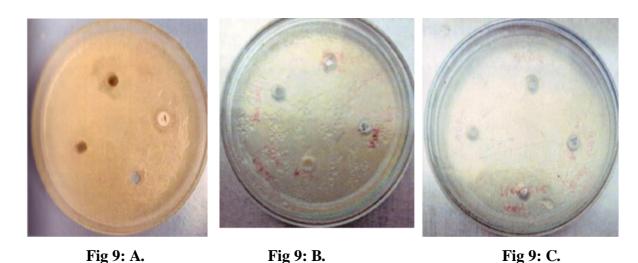
Fig. 7: Test for Tannins.



Fig. 7: Test for Glycosides.



Fig. 8: Test for Tannins.



From the picture number 2 to 8 there was represent the phytochemical estimation of rice husk as well as picture 9(A,B,C) has marked antibacterial active of rice husk.

EXTRACTION SILICA BY HEATING

Rice husk contains a high amount of silica. To extract it, the husk is first cleaned and dried to remove moisture and impurities. The dried husk is then subjected to control heating (pyrolysis) at high temperatures, usually between 500°C and 700°C. During heating, the organic matter (such as cellulose, hemicellulose, and lignin) burns off, leaving behind rice husk ash (RHA), which is rich in silica. The resulting ash mainly consists of amorphous silica, which can later be purified using chemical treatment if required.

EXTRACTION SILICA BY CHEMICAL

In the chemical extraction method, rice husk is first washed thoroughly to remove dirt and soluble impurities, and then dried. The dried husk is treated with acid solutions such as hydrochloric acid (HCl) or sulfuric acid (H₂SO₄) to remove metallic impurities like iron, calcium, and potassium. After acid leaching, the husk is rinsed with distilled water and dried again. The treated husk is then subjected to control burning at 500-700°C, producing rice husk ash rich in silica. To extract pure silica, the ash is further dissolved in sodium hydroxide

(NaOH) solution, forming sodium silicate. On acidifying this solution with sulfuric acid (H₂SO₄) or hydrochloric acid (HCl), silica precipitates out. The precipitated silica is washed, filtered, and dried to obtain high-purity amorphous silica.

CONCLUSIONS

This study demonstrates that rice husk, an agricultural by-product, can be efficiently converted into high-purity amorphous silica using combined thermal and chemical extraction methods. The controlled combustion and alkali-acid treatment produced silica with desirable structural, morphological, and thermal properties, confirmed through XRD, FTIR, SEM, and TGA analyses. In addition to being a sustainable source of silica, rice husk contains bioactive phytochemicals with medicinal potential, further enhancing its value. The extracted silica shows promise for applications in adsorbents, catalyst supports, cementations composites, and nanomaterial's, aligning with green chemistry principles and waste valorization strategies. The findings highlight the dual advantage of environmental waste reduction and material recovery, offering significant potential for industrial and biomedical applications.

Highlights

- Rice husk ash (RHA) is a sustainable source of high-purity amorphous silica.
- Controlled combustion (600–700 °C) yields silica-rich ash with minimal carbon.
- Alkali leaching and acid precipitation enable efficient silica extraction.
- Extracted silica confirmed by XRD, FTIR, SEM, and TGA characterization.
- Silica shows potential in adsorbents, catalyst supports, cement, and Nano composites.
- Rice husk phytochemicals provide additional medicinal and nutraceutical value.
- Process supports green chemistry and agricultural waste valorization.

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