



# Production And Characterization Of Coconut Husk Ash As A Source Of Bio Silica

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## **Abstract:**

The increasing demand for environmentally sustainable materials has encouraged the utilization of agricultural wastes as alternative sources of industrial raw materials. This study focuses on the production and characterization of silica extracted from coconut husk ash (CHA), an abundant agro-waste byproduct. Coconut husks were cleaned, dried and subjected to controlled combustion at varying temperatures (400<sup>o</sup>C, 600<sup>o</sup>C & 800<sup>o</sup>C) to obtain ash. The resulting ash was processed through acid leaching and alkaline extraction to isolate amorphous silica. The silica yield and purity were analyzed using X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). Results were revealed that the optimized process produced silica with purity exceeding 90% exhibiting amorphous characteristics and fine particle morphology suitable for industrial applications. The findings demonstrate that coconut husk ash is a promising and sustainable source of high-purity silica, offering potential use in glass, ceramics and composite materials industries while contributing to waste minimization and value addition.

**Key Words:** Coconut husk ash, Amorphous silica, XRD, Agro waste

## **1. Introduction**

Agricultural wastes have been utilized for the production of several useful materials to reduce their disposal challenges. These wastes are often generated and disposed of indiscriminately or burnt, which results in environmental pollution with adverse effects on climate [1]. The side effects of climate have been a major challenge globally because it leads to the death of about 160,000 people annually, according to the World Health Organization (WHO) [2-5]. India utilized 78% of its landmass, about 2,564,065 square kilometers, to produce several agricultural products and ranked third in eggs, oranges, coconuts, tomatoes, peas, and beans [6]. Literature has shown that agricultural wastes have been used as solid fuel briquettes, adsorbents, cellulose, activated carbon, silica, silicon, refractory, ceramic products, livestock feed, inhibitors, biofuels, construction materials, lignin, and composite reinforcement [7-22]. Silica has been utilized for several applications across industries such as pharmaceuticals, archeology, biomedical, electronics, and silicon feedstock (birth of semiconductor revolution). Silica occurs naturally as quartz with the largest percentage in the earth's crust. It is crystalline in nature with low reactivity. Amorphous silica, on the other hand, finds applications in many areas owing to its advantage of being highly reactive. The non-crystalline type has been synthesized from sodium silicate [35], water glass [23], tetraethoxysilane (TEOS) [24-26]. It has also been produced from agricultural wastes [19] using several methods as reported in reviews [27, 28]. It is usually prepared by reacting sodium carbonate powder and quartz sand at high temperatures to form sodium silicate, which will then react with sulphuric acid to precipitate silica [26]. The methods used

for synthesis from agricultural wastes involve a combination of processes like acid treatment, partial burning (charring), burning, calcination, leaching, enzymatic treatment [31], pyrolysis, hydrolysis, and sol-gel [28 - 30]. Among wastes generated in India, coconut wastes stand out because they litter our farmlands, residential and industrial areas because of coconut flesh/juice's economic and social importance. Coconut is a fruit of an Arecaceae plant (palm family) and can be found everywhere across India. India accounts for more than 11 % of the total world production after Indonesia and the Philippines [32]. The fruit is 55 % coir, 15 % shell, and 30 % flesh (meat) and juice [33]. The main part is the meat, where coconut oil and meals are produced. The coir (fibrous part) has been found to be useful for mats, ropes, sacks, door mats, brushes, boat caulks, compost, and fiber for mattresses [34,36]. The research aims to evaluate the waste natural products to prepare silica powder from coconut shell X-ray diffraction (XRD), Fourier transform spectroscopy (FTIR), Scanning electron microscopy and Energy dispersive spectroscopy (SEM/EDS) was used to analyze the prepared silica.

## 2. Experimental details

### 2.1. Materials

For silica production, in order to lower the cost of material, natural materials are being used. Coconut Husk is used that is one of the natural materials that have limited research area. For this purpose, our country has been supplying the Coconut Husk for analysis, which is grown in seasonal time by separating their shells. For the extraction NaOH was obtained from Sigma Aldrich. All the chemicals and reagents used in this work were of AR grade and solutions were prepared using distilled water.

### 2.1 Preparation of CHA

Initially, Coconut Husks were cut into small pieces and washed thoroughly with water to remove the soluble particles and dust or any other contaminants. Second, dried in sunlight complete evaporation of moisture and also dried in air oven at about 80°C for 48h. Finally, dried shells physically activated at 400°C for 3h in muffle furnace and then dried labelled at CHA. After the different temperature degrees and for different times, it was sintered in order to remove the volatile gases and converted to respective ash powders.

### 2.2. Synthesis of Silica

Silica nanoparticles have been prepared through dissolution and extraction process. The solution of silica was carried out using an acid leaching process 1 M NaOH to partially dissolve carbonaceous materials.

The sintered powder (Ash) was treated with 1 M NaOH to form sodium silicate solution. And then solution was stirred at 80°C for 3hrs, then the solution was filtered using No.1 Whatmann filter paper and adding HCL drop by drop wise and then the solution was dried kept at oven. The product was labelled at GSS.

### 2.3. Characterization

The chemical bond in the prepared sodium silicate solutions was studied by using Fourier infrared spectroscopy. The study was carried out in the wave number of 400 – 4000cm<sup>-1</sup>. The XRD measurement was taken at the step size of 0.02deg/min by using CuK $\alpha$  and Ni filtered radiation. The surface morphology was studied by scanning electron microscope (SEM).

## 3. Results And Discussion

All extraction process involved the HCl treatment, calcinations at a temperature around 500 to 600°C and NaOH treatment. We observed a change in the color of the ash from black to white, with the final result being bio silica.

### 3.1.Phase Investigation Of Bio Silica

The structure and phase purity of the nanocomposites were analyzed by the X - ray powder diffractometer with a Cu target radiation source. The XRD pattern for phase analyses was recorded in

the  $2\theta$  range  $10-80^\circ$ . The X-ray spectra and characteristic absorption peak of Coconut husk silica have been acquired at varying temperatures  $400^\circ\text{C}$ ,  $600^\circ\text{C}$ ,  $800^\circ\text{C}$  and displayed in Fig.3.1 (a -c). The sample is amorphous as pointed out by the featureless diffractograms and the appearance of a scatter maximum at  $2\theta = 23^\circ$  characteristic of amorphous silica with hexagonal system according to the JCPDS 79-1915 [40]. The structure of silica present in the selected as optimum temperature for bio silica synthesis. This pattern exhibits a very broad line and no defined peaks due to crystalline are encountered. Figure 3.1(a) also describes the theoretical postures of the main reflexion of the phase cristobalite ( $\text{SiO}_2$ ) and graphite (C) and no peaks are discovered in these postures.

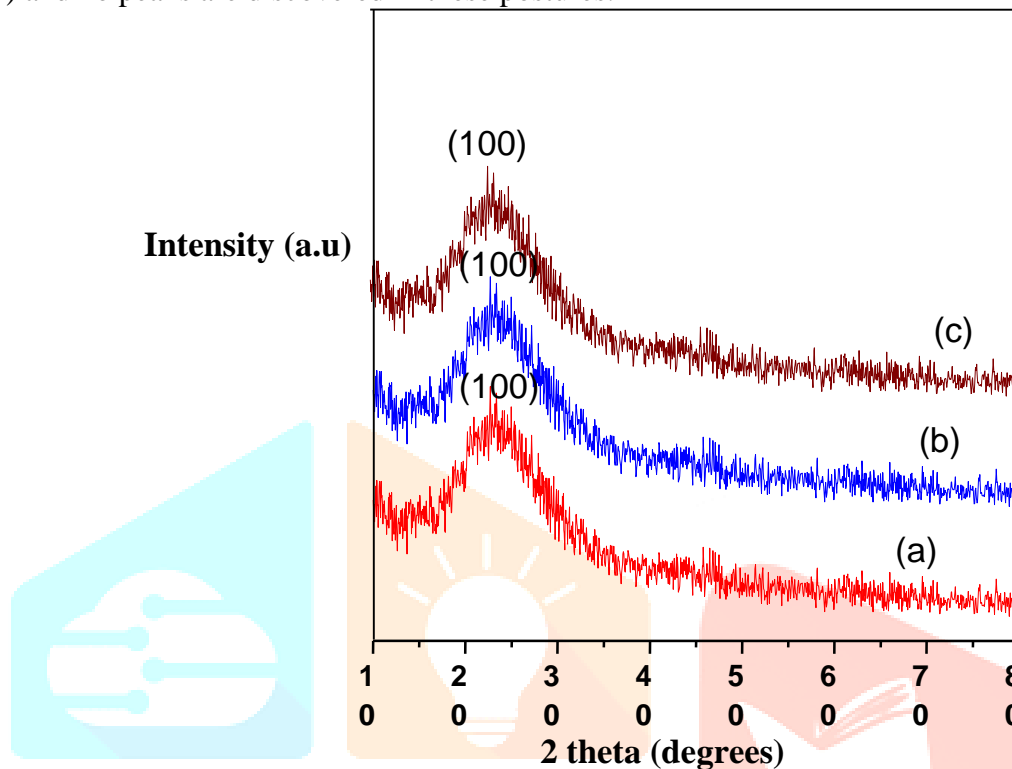


Fig. 4.1 XRD Spectra of CHA (a)  $400^\circ\text{C}$  (b)  $600^\circ\text{C}$  (c)  $800^\circ\text{C}$

### 3.2. Spectral Analysis of Bio Silica

Fig.3.2. (a - c) shows the FTIR spectrum of CHA. A broad band in the range of  $3375\text{ cm}^{-1}$  is due to the stretching vibration of the O – H band. This band is due to silanol groups ( $\text{Si} - \text{OH}$ ) and the adsorbed water bound to the surface. The weak band  $1627\text{ cm}^{-1}$  is assigned to H – O – H bending vibration mode. The predominant peak at  $1350\text{ cm}^{-1}$  is due to siloxane bonds ( $\text{Si} - \text{O} - \text{Si}$ ).

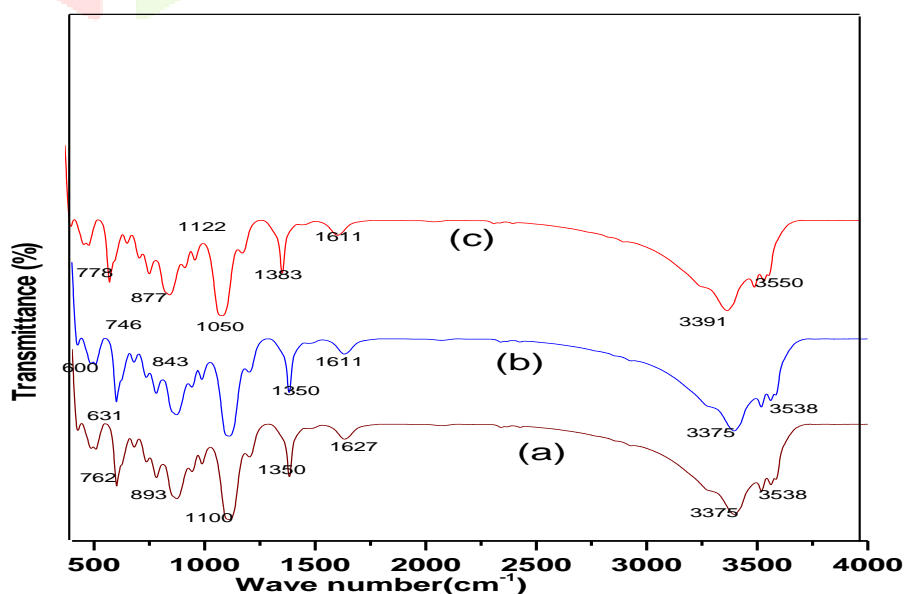


Fig. 3.2 FTIR Spectra of CHA (a)  $400^\circ\text{C}$  (b)  $600^\circ\text{C}$  (c)  $800^\circ\text{C}$

The adsorption band at  $893\text{ cm}^{-1}$  is because of silica structures and other peaks observed at  $1627\text{ cm}^{-1}$  are because of impurities such as carbonate and sodium groups. The most intense bands at  $1100\text{ cm}^{-1}$  and a peak  $762\text{ cm}^{-1}$  are due to asymmetric and symmetric stretching mode at Si – O – Si. The vibration of oxygen atoms joined with the adjacent atoms in the asymmetric stretching vibrations of Si – OH bond which appeared at  $1100\text{ cm}^{-1}$ [41].

### 3.3. Morphological Analysis of Bio Silica

The SEM image of Coconut husk silica is shown in figure 3.3. It is seen that the CHS has irregular shape that are loosely packed structure with holes.

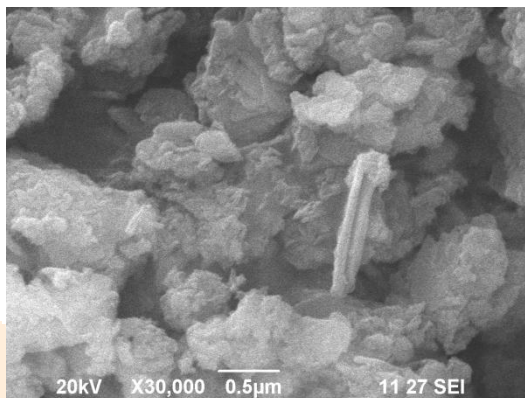


Fig.3.3. SEM image of CHS

### 3.4. Elemental analysis Bio Silica

Energy dispersive spectroscopy or EDX analysis of silica prepared is demonstrated in Figure 3.4 indicates silica ( $\text{SiO}_2$ ) as the major compound in the coconut husk silica. In addition, the impurities of the precipitated silica obtained. There are two elements of impurities found in the precipitated silica: K and O.

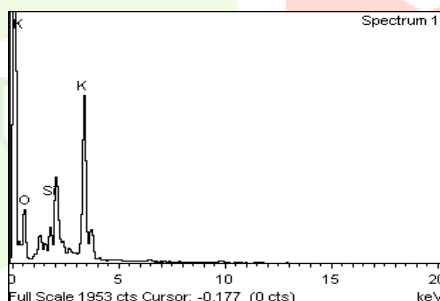


Fig.3.4. EDX spectra of CHS

### Conclusion:

This study confirmed that we successfully extracted agricultural waste particularly Coconut husk derived bio silica. Acid treatment of the ash is effective for the removal of metallic impurities. Impurities observed in the silica from EDX are K and O. The silica particles are produced and highly agglomerated due to high attraction between the Si-O and Si- OH bonds. XRD results show the silica from shell of coconut is amorphous.

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