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# Treatment of Silica Nanoparticles using waste Agricultural Secondary Products

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## Abstract

The purpose of this paper is to look at the properties and synthesis of silica from agricultural wastes including palm shell ash, corn cubs, and coconut shells. Large amounts of agricultural waste have the potential to be used as renewable energy for silica extraction. In this paper, silica is extracted using an alkaline technique followed by acid precipitation. The Strong acid leaching procedure was applied to three distinct agricultural wastes in order to eliminate metallic impurities and organics. When compared to other methods, leaching treatment is the best way to extract silica since it results in high pure chemical compositions. The goal of this study is to extract silica from agricultural wastes. The obtained silica may then be treated with a basic solution, making it helpful in industrial Column Purification Techniques of Pharmaceutical Industries.

**Key words: coconut shell, palm shell, corn cob, Acid Precipitation and agricultural waste**

## 1.Introduction

Silicon dioxide (SiO2), often known as silica, is a basic material and one of the most useful inorganic multifunctional chemical compounds. Silica occurs naturally as flint, quartz, or sand. It might be amorphous, crystalline, or gel-like. It is the most adrift material in the earth's crust. However, creating pure silica demands a significant amount of energy. The proposed chemical process not only provides a waste disposal solution, but it also recovers a valuable silica product as well as certain useful companion recoveries. Rice husk is a typical agricultural byproduct in rice-growing countries. Every year, India generates around 12 million tons of rice husk. Rice husk is typically not suggested as cow feed due to its poor cellulose and other sugar levels [1]. Furfural and oil are extracted from rice bran using rice husk. Rice husk is used as a fuel in boilers and for industrial power generation. Rice husk has a high ash concentration ranging from 18 to 20%, making it one of the biomass types utilised in gasification. Silica accounts for 85 to 95 percent of rice husk ash. Because of the high silica concentration in rice husk, the extraction of silica from ash, which has a big market and also handles ash disposal, becomes cost-effective [2]. The frequent generation of these wastes leads to their combustion, which pollutes the environment and damages the climate. According to the World Health Organization, the detrimental consequences of climate change cause almost 160,000 fatalities each year, making them a serious worldwide concern. India ranked third in the production of eggs, oranges, coconuts, tomatoes, peas, and beans. It produced agricultural goods across 78% of its area, or about 2,564,065 square kilometers. Coconut trash stands out among India's wastes since it litters our farmlands due to the commercial and social worth of coconut flesh and juice [3,4]. These waste products are made up of carbon, oxygen, and hydrogen, which when burned produce combustible gases such as carbon monoxide, hydrogen, methane, and ash.

Despite its high carbon and silica content, this ash has yet to be used. Coconuts, a fruit of the Aceraceae family, are extensively accessible in India (a member of the palm family). India is third in terms of output, after only the Philippines and Indonesia, with more than 11%. Later, hulls were removed with pestles and a crude equipment known as a rice pounder. Agricultural waste has been used to manufacture a variety of valuable commodities in an effort to alleviate the challenges associated with its disposal [5]. This study attempts to provide a simple procedure for producing precipitated silica from discarded rice husk. A novel technique of producing silica is developed by reacting rice husk ash with caustic soda, followed by leaching treatment with strong acids to generate high pure Silica Crystalline [6].

## 2.Materials and methods

## 2.1 Coconut Shell

They have been utilised to create a number of valuable commodities to make agricultural wastes simpler to dispose of. These wastes are regularly created and burnt, causing pollution and having a harmful impact on the climate. According to the literature, agricultural wastes have been used as construction materials, lignin, composite reinforcement, cellulose, activated carbon, silica, silicon, refectory, ceramic products, livestock feed, inhibitors, and solid fuel briquettes. Pharmaceuticals, archaeology, biology, electronics, and silicon feedstock (the start of the semiconductor revolution) are just a few examples of businesses that have exploited silica [9]. Coconut wastes stand out among wastes produced in India since they litter our farmlands, neighborhoods, and industrial districts due to the economic and social significance of coconut meat and juice. Coconuts, a fruit of the Aceraceae plant, may be found all throughout India (a member of the palm family). After Indonesia and the Philippines, India is third in the world in terms of production, accounting for more over 11% of total output. The fruit is composed of 30% flesh (meat) and juice, 15% shell, and 55% coir. The bulk is made up of meat, from which meals and coconut oil are manufactured. Coir, the fiber component of the plant, has been found to be useful for manufacturing mats, ropes, sacks, door mats, brushes, boat caulks, compost, and mattress fabric. Both shell and coir are used as fuels, with or without carbon enrichment. With all of this use, many shells and coir are tossed carelessly in the home. Both generate ash, which may then be treated to remove the silica, which is abundant in both [10].

## 2.2.1Method for Coconut Shell

The main fruit is separated from the coir and shell in this process, and moisture, volatile matter, and carbon are removed to generate ash. The soluble oxides of certain metals are then removed using an acid treatment. The sol-gel method begins with the formation of a silicate solution from ash and the neutralization of an alkaline solution to precipitate silica gel, which is subsequently dispersed in a sodium hydroxide solution.

The silica is separated from the salt solution and centrifuged to remove the NaCl using washing and rinsing with double-distilled water (DDW). Ten coconut fruits were picked, and the coir, shell, and flesh were meticulously separated. The coir and shells were sun-dried for 14 days prior to being fired in a muffle furnace for Calcination. The samples were placed in the furnace and heated to 400-600 degrees Celsius at a rate of 10 degrees Celsius per minute. After retaining the temperature for three hours, the samples were allowed to cool for twelve hours. The samples were then separated and labelled as coconut shell ash (CSA) and shell forming of silica with 3 M an H2SO4, CSA were leached for 60 minutes. After filtering and washing with DDW, residues were collected and dried at 90 degrees Celsius in an oven for 24 hours. TCSA, for CSA, was assigned to the dried samples. Sodium silicate was created by combining 2.5M of CSA and TCSA in NaOH and heating it to 90 degrees Celsius for 60 minutes. After allowing the mixture to cool, it was filtered using Whatman No. 1 filter paper. 3M H2SO4 was titrated into the filtrate to ensure that the initial NaOH was neutralized [6]. After decanting the supernatant, the residual silica gel solution was centrifuged at 4000 rpm for 10 minutes. DDW was added for washing after decanting the supernatant. The resulting silica gel was then oven dried for 24 hours at 90°C [13,14].

## 2.2 Palm shell

Elaeis guineensis is the scientific name for palm shell. Oil palm trees are native to Asia and Nigeria. Palm fruit is solely required for palm oil production. The leftovers, such as palm husk and palm kernel shell, are discarded. Recently, these wastes have been utilised to fill landfills, as fuel for combustion, and so on. These shell wastes constitute a sustainable agricultural waste source. Rather than these, scientists used palm shell as a light weight aggregate to replace typical heavy weight aggregates in crucial parts and motorway building. Palm kernel shell has also been used in a variety of applications ranging from energy storage to biomass and bio-fertilizers to super capacitor electrodes. Palm kernel Shell (PKS), also known in the literature as Oil Palm Kernel Shell (OPKS) and palm Shell (OPS), is a highly lignocellulose biogenic waste produced during the preparation of crude palm oil1. This is the shell portions that were left over after the nut was removed and crushed in the palm oil mill, and it is acquired as residual waste in the extraction of the kernel from the nut. Oil palm is grown in plantations that have been in operation for two to three decades, with rotational farming followed by removal and replanting. It has a low moisture content, which has been found to range between 11-13%. Indonesia and Malaysia generated an estimated value of 3.06 million metric tons in 2001 alone. The PKS is the hard component of the palm kernel fruit that contains the palm kernel seed of the palm tree (Elaeis guineensis).

## 2.3 Corn Cob

Inorganic and organ metallic compounds, as well as thin films or coatings for electrical and optical components, have been produced utilizing silica as a crucial precursor. A low energy approach was used to extract pure silica with a 91% extraction yield from rice husk ash. Techniques such as acid leaching and gasification have also been examined. Maize is an important staple food in Nigeria, with an annual production of 9.4 million tons. Corn cobs are mostly utilized in agricultural operations as manure. Many agricultural products, including rice husk, sugar cane bagasse, and coffee husk, have been effectively utilized to extract silica. The precipitation method was used to produce and analyze nano-$SiO_2$ from corn cob ash [11].

### 2.3.1 Palm shell method

The palm shells were washed and dried in the sun. The dried shell was subjected to combustion in a container at 400 to 600 °C for three hours at a heating rate of 10 °C/minutes in a muffle furnace, and then the furnace was allowed to cool overnight. To extract silica from palm shell ash, the silica extraction process was changed. 250 ml sections of 2.5N NaOH were added to 50g of palm shell ash samples, and the mixture was then heated in a covered 500 ml Erlenmeyer flask for 1 hour while being swirled constantly to dissolve the silica and generate a sodium silicate solution. The solutions were filtered using ash-free filter paper, and the remaining particles were washed away with hot distilled water. The filtrate was brought to room temperature, the pH of the solution was reduced to 11.2 by adding 3N $H_2SO_4$, and an incubation time was utilised to stimulate gel formation. After being created from the sol, the gel was matured for 10 hours. The soft gel was gently broken after ageing, the slurry was centrifuged at 4000 rpm for 5 minutes, the supernatant was discarded, and the gels were placed in a beaker and dried at 90°C for 24 hours to produce xero gels. Several washings with de-ionized water were done on the silica xerogel to successfully remove minerals and contaminants. The crust of the Earth contains the most naturally occurring silica as quartz. Nature is crystalline and has a low reactivity. Amorphous silica, on the other hand, has the advantage of being very reactive and is employed in a variety of sectors. The non-crystalline kind is made from tetraethyl orthosilicate (TEOS), sodium silicate, and water glass. It has also been manufactured from agricultural waste using a number of ways, according to reviews.

### 2.3.2 Method for corn cob

The corn cobs were cleaned and sun dried. The combustion was conducted in a container at 400 to 600 °C for three hours at a heating rate of 10 °C/minutes in muffle furnace, and then let the furnace to cool overnight. The silica extraction method was modified to extract silica from corn cob ash (CCA). To 50g of CCA samples, 250 ml portions of 2.5N NaOH were added, and the mixture was then boiled in a covered 500 ml Erlenmeyer flask for 1 hour while being stirred continuously to dissolve the silica and create a sodium silicate solution. Ash less filter paper was used to filter the solutions, and hot distilled water was used to wash away the leftover particles. The filtrate was brought to room temperature, the pH of the solution was brought down to 11.2 by adding 3N $H_2SO_4$, and an incubation period was used to encourage gel formation. The gel was matured for 18 hours after it was produced from the sol. Following ageing, the soft gel was gently broken, the slurry was centrifuged at 4000 rpm for 5 minutes, the supernatant was discarded, and the gels were put into a beaker and dried at 90°C for 24 hours to generate xero gels. To effectively remove minerals and impurities from the silica, several washings with De-ionized water were performed on the silicagel. The earth's crust has the highest amount of naturally occurring silica as quartz. Low reactivity and crystalline in nature. Contrarily, amorphous silica has the benefit of being extremely reactive and is used in many different industries. Tetraethyl orthosilicate (TEOS), sodium silicate, and water glass have been used to create the non- crystalline kind. According to evaluations, it has also been made from agricultural waste using a variety of techniques. The typical method of making it involves heating up sodium carbonate powder and quartz sand to create sodium silicate, which will then react with sulphuric acid to precipitate silica [15].

## 3.Results and discussion

To extract silica from rice husks, they had to first remove impurities by leaching, then dissolve the silica by adding NaOH solution to generate a sodium silicate solution. The following stage is the deposition of silica in sodium silicate solution by acid treatment with sulphuric acid (H2SO4). The results are as follows: Heated for 3 hours at 600 degrees Celsius. About 11.5%, 3.6%, and 53.3% of the silica recovered from the results of these investigations were effectively extracted. However, the resulting silica still contains 4% sodium impurities and must be washed again to reduce the sodium concentration to roughly 0.10%. As a result, the operation is inefficient and time-consuming. The Silica Production Process Flow Diagram Using Natural Agricultural Resources Sources shown in fig 1.

In this paper, we studied the treatment of natural materials such as coconut shell, palm shell, and corn cob at 400 - 600 °C for a constant period. Further, heated material is neutralised by an alkaline medium of NaOH, and material precipitation is obtained by acid treatment [15,16]. The obtained material is next dried to get a low moisture content and a higher proportion of Silica derived from coconut shell, palm shell, and corn cob, the composition of which is shown below.

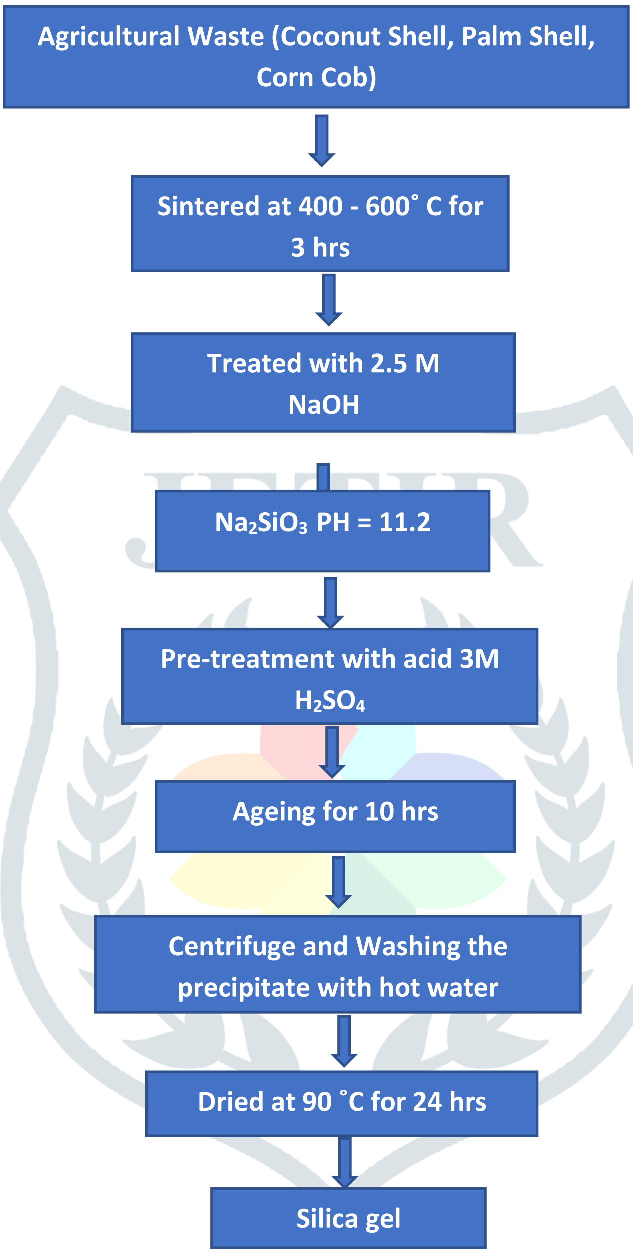


**Fig 1.Schematic representation of silica extraction from the waste materials.**

**Table 1- PERCENTAGE OF SILICA USING VARIOUS TEMPERATURE(RH) by Using coconut shell**

| TEMPERATURE(CELSIUS) | TIME(Hrs) | SILICA( %) |
|---|---|---|
| 400 | 3hrs | 6.4 |
| 450 | 3hrs | 7.54 |
| 500 | 3hrs | 8.1 |
| 550 | 3hrs | 9.26 |
| 600 | 3hrs | 11.75 |

**Table 2 PERCENTAGE OF SILICA USING VARIOUS TEMPERATURE(RH) by Using palm shell**

| ***TEPERATURE(CELSIUS)*** | **TIME(Hrs)** | **SILICA(%)** |
|---|---|---|
| 400 | 3hrs | 1.32 |
| 450 | 3hrs | 2.15 |
| 500 | 3hrs | 2.89 |
| 550 | 3hrs | 3.21 |
| 600 | 3hrs | 3.6 |

***Table 3- PERCENTAGE OF SILICA USING VARIOUS TEMPERATURE*(RH) by Using Corn Cab**

| **TEMPERATURE(CELSIUS)** | **TIME** | **SILICA%** |
|---|---|---|
| 400 | 3hrs | 25.49 |
| 450 | 3hrs | 31.6 |
| 500 | 3hrs | 37.64 |
| 550 | 3hrs | 47.73 |
| 600 | 3hrs | 52.38 |

### 3.1 Effects of temperature on Palmshell, coconut shell and maize cob

The silica content of ash formed from coconut shell, palm shell, and maize cob is determined using different degrees Celsius, as shown in figures 2,3,4. Temperatures were between 400 to 600 degrees Celsius. By increasing the temperature to 400, 450, 500, 550, 600, we got silica content for coconut shell as 6.4, 7.54, 8.1, 9.26, 11.75, for palm shell ash as 1.32, 2.15, 2.89, 3.21, 3.6, and for corn cob ash as 25.49, 31.6, 37.64, 47.73, 52.38. This graph shows that raising the temperature of ash content conversion boosts silica yield percentage.

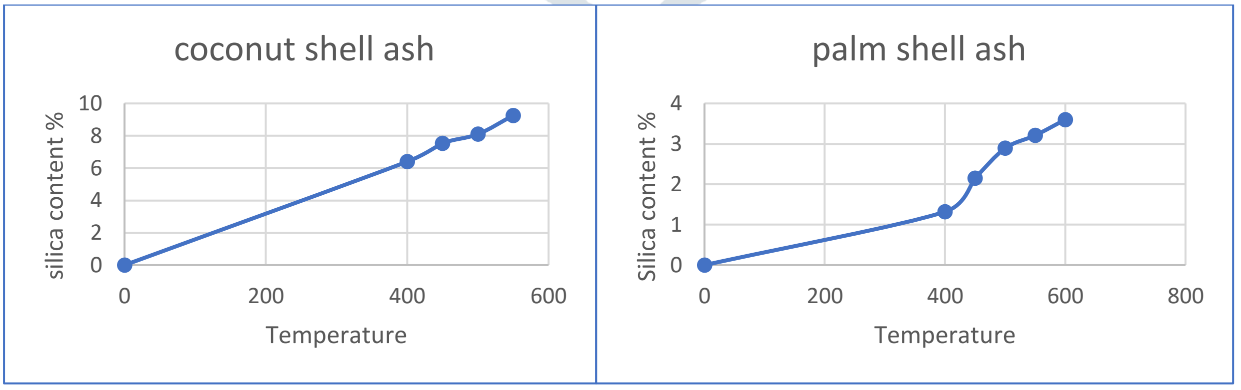


**Fig 2: Effect of temperature on coconut shell** **Fig 3: Effect of temperature on Palm shell**

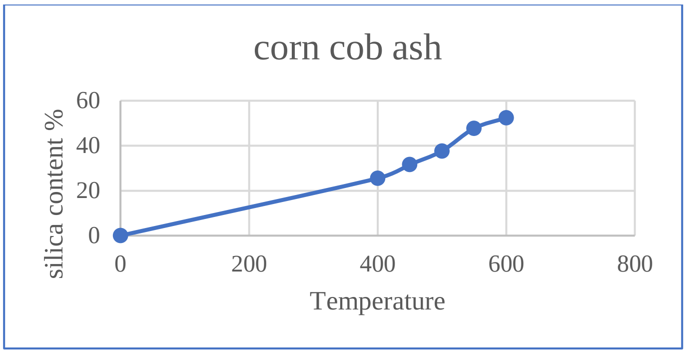


**Fig 4: Effect of temperature on corn cob**

At 600 degrees Celsius, the greatest percentage rate of silica was reached for three distinct materials such as coconut shell, palm shell, and maize cob. When comparing the three agricultural wastes stated above, maize cob has the greatest purity rate of silica compared to coconut, as shown in fig 5. At 600 degrees Celsius, the maximum purity rate of silica generated in the maize cob was 52.38%.

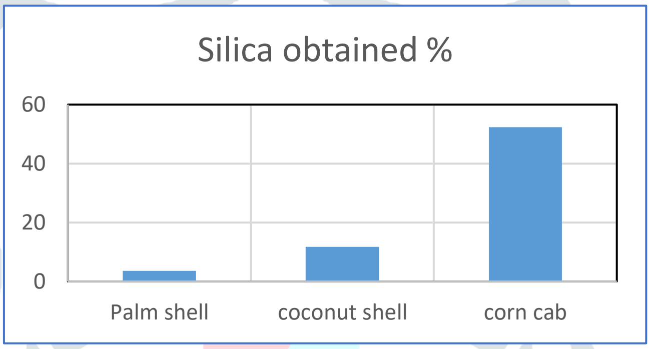


**Fig 5: comparison of silica obtained percentage of three different material**

## 3.2 Functional group and phase development of the powder sample determination

The chemical bond in the generated powder was studied using the SHIMADZU Fourier infrared spectroscopy apparatus. Waves ranging from 400 to 4000 cm-1 were used in the study. The IR spectra of sodium silicate gels generated by alkali treatment are shown in Fig. The absorption of silanol groups resulted in a volume increase of 3700 cm-1. H2O molecules' stretching and bending vibrations correspond to a band at 3437 cm-1 and a band at 1632 cm-1, respectively.The TO and LO modes of the Si-O-Si asymmetric stretching vibrations are responsible for the strong and broad band at 1111 cm-1, with a weak shoulder at 1188 cm-1. The strong bands at 800 cm-1 and 474 cm-1 are created by Si-O-Si vibrations caused by symmetric stretching and bending. All of the alkali-treated materials' FT-IR spectra reveal absorption bands at 1390 cm-1 and 890 cm-1, indicating that sodium silicate may be produced.

## 4.Conclusion

Secondary agricultural products include silica, and some have a significant amount of silica in their matrix. By burning the material and then exposing it to chemical or acid-base treatment, a high silica range with good purity is achieved. Silica nanoparticles may also be extracted using ionic solutions treated with corn cob. Secondary agricultural resources such as maize cob, wheat straw, grasses, and sugar cane waste are the most suitable Renewable Energy Sources for silica extraction. The use of silica nanoparticles benefits the development of polymer nanocomposites, biosensors, catalysts, agricultural, environmental, and coating applications. In agricultural secondary products, silica may be found in huge amounts and from renewable sources, and its use will assist to improve purifying procedures in many sectors for Cost saving on Raw materials [17].

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