Research Article **Available online www.ijsrr.org** **ISSN: 2279–0543**

International Journal of Scientific Research and Reviews

Synthesis of Silica Nanoparticles from Cocos Nucifera Husk Ash

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ABSTRACT

Silicon dioxide (silica) is one of the valuable chemical compounds. Silica presents an interesting for their various applications in adsorption, catalysts, insulators, fillers in composites and ceramic industries. In this paper, focused the silica synthesized from agricultural waste of coconut husk. An inexpensive chemical method was used to synthesize nanosilica from coconut (cocosnucifera) husk. Burning coconut husk produces the coconut husk ash(CHA). The coconut husk ash reacted with sodium hydroxide the resultant solution was filtered. The filtered solution treated with 50% of sulfuric acid and maintained the pH at 8.5 to 9.5 to precipitate silica dried at 24h. This silica particles refluxed with HCl to form pure nanosilica particles. The results are showed that to obtain silica from coconut husk ash has large surface area, small particle size and high quality. The obtained silica was investigated by Fourier transform infrared (FT-IR) spectroscopy, X- ray diffraction (XRD), Scanning electron microscope (SEM) and Energy dispersive X- ray analysis (EDAX).

KEY WORDS: Coconut Husk Ash, Inexpensive, Combustion, Precipitation, Acidification, Sodium Silicate.

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INTRODUCTION

Cocosnucifera (coconut) belongs to palm family. Every part of the coconut tree can be used for myriads of functions. Coconut is harvest in several countries of the world.[1]World's largest producers of coconuts is the Indonesia, Phillipines, India and Srilanka. Coconut husk is the raw materials in south India like Kerala and Tamil Nadu. Coconut husk is the part of the coconut which found around the outer shell.[2]Fibrous husks have a number of uses and the fibers are separated to produce coir. Coconut husks are used to make all kinds of household items such as rope, mats and brushes etc. In many countries, the coconut husk wastes were burnt as fuel and as a source of activated charcoal which has potential as good adsorbent materials that can solve the waste water treatment. Coconut husk has silica and also has carbon content in their ashes. Silica (SiO_2) is the most abundant in nature as quartz and various living organisms.[3] Silica is the major ingredient of sand. It's preferred in optoelectronic applications and it can exist in gel, crystalline and amorphous state.[4] Extracted silica from agricultural waste material of rice husk ash has been already reported in many authors.[5] Silica is highly applicable in industries because of their high melting point 1710^{o}C. SiO_2 was successfully extracted from coconut husk ash first treated with alkali (NaOH) to form sodium silicate.[6]Sodium silicate was treated with sulfuric acid to precipitate silica which will provide an alternative way of producing nanosilica from waste raw material with low cost of production.[7] Coconut Husk Ash (CHA) is one of the most silica rich raw materials after a complete combustion. The initial step is extraction of silica from ash as sodium silicate using caustic soda.[8]Silica is precipitated from sodium silicate using sulfuric acid.[9,10] Hence, the main aim of this research work to investigate the importance of alternative source in producing silica nanoparticles. Because of the higher demand for silica and silica derivatives thus, the utilization of silica through the modification of extraction process successfully extracted silica nanoparticles from coconut husk ash. The synthesized high quality of pure nanosilica from coconut husk ash and characterized by various sophisticated techniques like FT- IR, XRD, SEM- EDAX.

MATERIALS AND METHODS

Materials

Coconut husk was collected from local area in Kanyakumari district, Tamil Nadu, India. Chemicals were purchased from Merck (AR grade) and were used without purification.

Synthesis of Silica using Chemical Method

Alkali Treatment

Coconut husk was collected, cleaned and dried. It was treated by thermal combustion under controlled condition at 700°C for 6 h. 5 g of coconut husk ash with 4g of NaOH was added and heated at 120^{o}C. Then the mixture was into the beaker added 40ml of deionized water stirring constantly for 6h at 70^{o}C using hotplate magnetic stirrer. After that, the solution was filtered by using ashless filter paper. The residue was washed with 100 ml of distilled water and the filtrate was allowed to cool down to room temperature.

Acid Treatment

The above sodium silicate solution to 50% of H_2SO_4 was added drop wise to reach pH 2 under constant stirring for 4h and then added NH_4OH upto pH 8.5allowed to stand at room temperature. The precipitate was washed several times using deionized water to remove sulphate impurities and dried in a hot air oven at 200^{o}C for 15 h to form pure silica powder.

Preparation of Nanosilica Particles from Coconut Husk Ash

Nanosilica was prepared by reflux method. Pure silica was extracted by refluxing with 6 N HCl for 4 h and then washed repeatedly using deionised water to make it acid free. Then it was dissolved in 2 N NaOH by continuous stirring for 10 h on a magnetic stirrer and then drop by drop added concentrated $H_2SO_4$to adjust the pH in the range of 7.5-8.5.The precipitated silica was washed with warm deionised water after the washing process silica powder was dried at 50°C for 48 h in the oven.

Reaction Mechanism

The first step is the extraction of silica from coconut husk ash. The reaction of the initial step was coconut husk ash react with alkaline sodium hydroxide. Coconut husk ash contains silica react with sodium hydroxide to form sodium silicate.

Ash contain silica (SiO_2) +Alkali (NaOH) $\longrightarrow$ Sodium silicate (Na_2SiO_3)

The sodium silicate reacted with 50% sulfuric acid to precipitate nanosilica and impurities. The impurities are removed by washing with boiling water.

Sodium silicate (Na_2SiO_3) + Sulfuric acid (H_2SO_4) $\longrightarrow$ Silica (SiO_2) + Na_2SO_4 +H_2O

Flow Diagram of Silica Synthesis

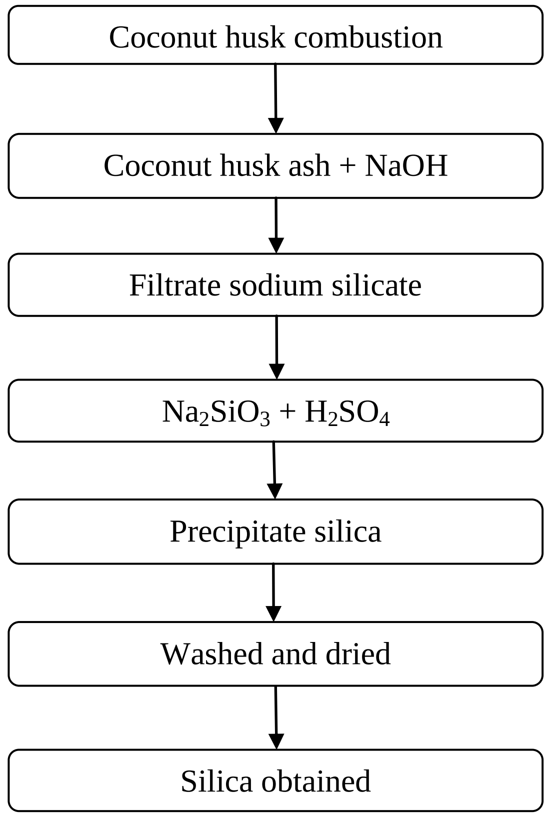

Figure.1: Flow Diagram of Silica Synthesis

RESULTS AND DISCUSSION

The present work is aimed for the synthesis of pure nanosilica from the ashes of coconut husk and characterized by using sophisticated analytical techniques FT- IR, XRD, SEM- EDAX.

Fourier- Transform Infrared Spectroscopy (FT-IR)

Fig. 2.Shows that the broad band corresponds to the O-H stretching vibration of the Si-OH the peak around 3433.98 $cm^{-1}$and the band around 1635.78 cm^{-1} corresponding to the bending vibration of the Si-OH.[11-13] The absorption peaks of the Si-O-Si asymmetric stretching vibration were observed at 1095.74 cm^{-1} and the symmetric stretching vibration peak observed at 801.6cm^{-1}.[14,15] The transmission peak around 488.73cm^{-1} corresponding to the symmetric bond vibrations of the Si-Si bond and the corresponding peak at 801.6 cm^{-1} presence of Si-O bond.[16,17]

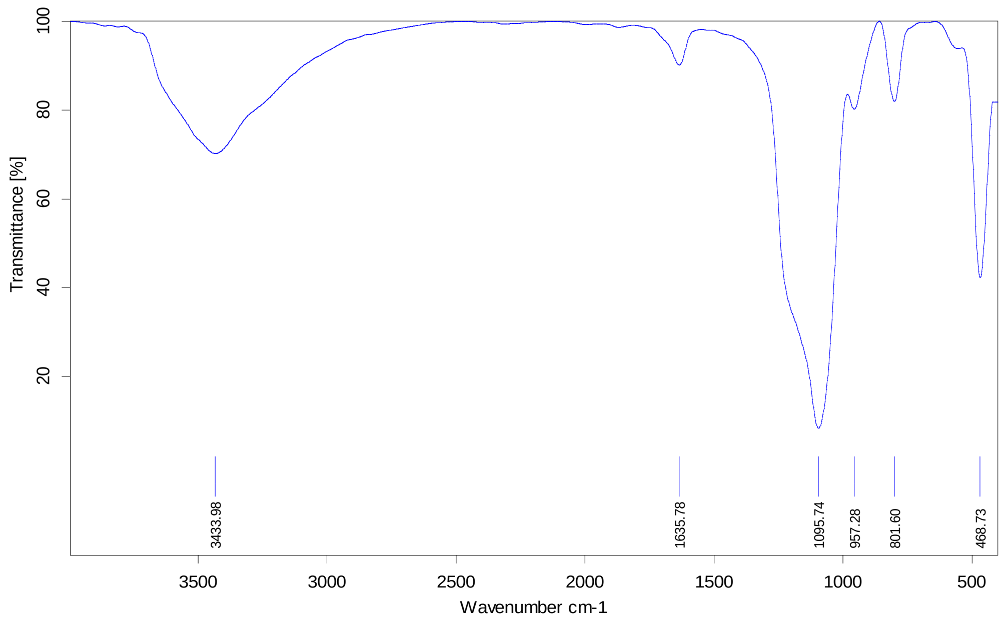

Figure.2: FT- IR Spectrum of Silica

X –Ray Diffraction (XRD)

The crystallinity of particles was determined by X-ray diffraction (XRD) pattern.Fig.3.shows the XRD pattern of the synthesized nanosilica powder from coconut husk ash (CHA). The average crystallite size estimated using the Scherrer's equation. The X-ray diffraction analysis was used to determine the crystallinity and the phase of the synthesized compound.[18,19]The presence of sharp peaks in the XRD pattern of mesoporous silica powders indicated the crystalline nature of the material.[20,21]

X- ray diffraction (XRD) patterns of the prepared silica sample shows a crystalline sharp peak at $2\theta = 33.82^{o}$ with a number of less intense peaks at $2\theta = 19.07^{o}$, 28.07^{o}, 32.15^{o}, 33.82^{o}, 38.63^{o}, 48.78^{o} and 55.40^{o}.The average crystallite size of these silica nanoparticles was calculated using the Debye-Scherrer diffraction form $D = K\lambda/ \beta\cos\theta$

Where,

D = crystallites size (nm)
K = 0.9 (Scherrer constant)
λ = 0.15406 nm (wavelength of the x- ray sources)
β = FWHM (radians)
θ = peak position (radians)

Here K = 0.9, λ = 0.1540 nm, is the X- ray wave length and β is the full width at half peak heigh. $\beta = 0.1506$, $2\theta = 33.8236$, $\theta = 16.9118$, D=53nm.

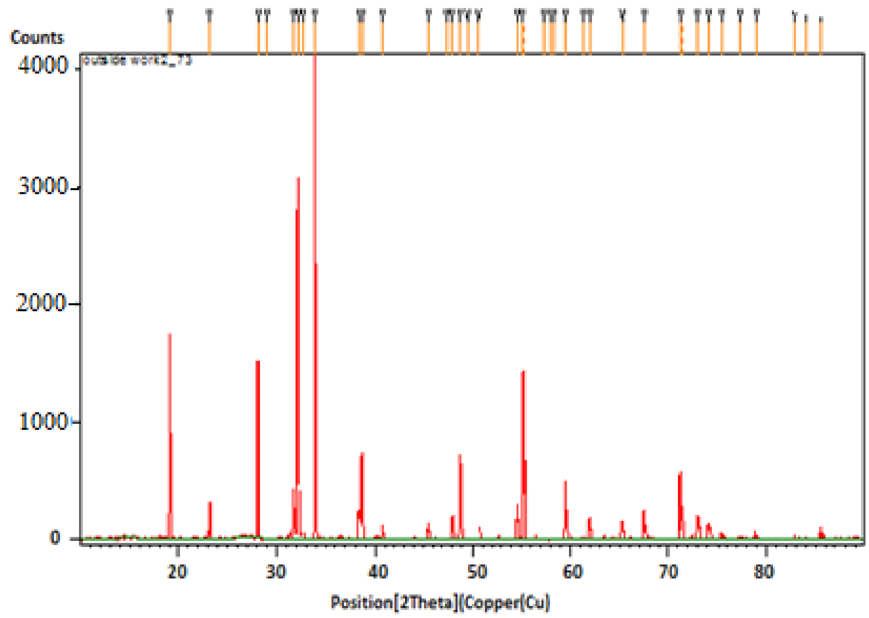

Figure.3: XRD Image of Silica

Scanning Electron Microscope (SEM)

Scanning electron microscopy was used to determine the surface morphology of the sample. The SEM image of silica nanoparticles is shown in fig 4. The size of microstructure in the sample of nanosilica particles consists of very fine particles with size less than 5 micrometer. The particles are present in spherical shape which is good agreement with the crystalline size.

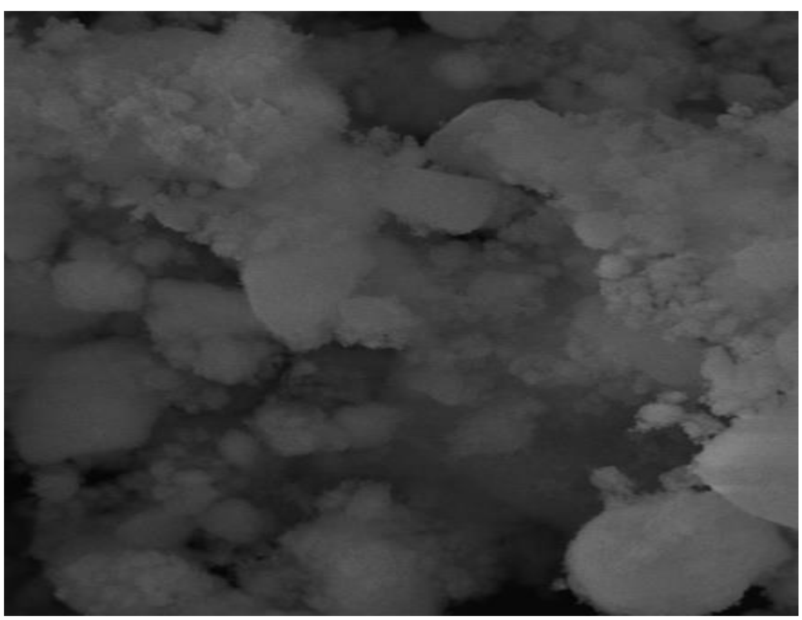

Figure.4: SEM Image of Silica

Energy- Dispersive X- ray Spectroscopy (EDX)

An energy dispersive X- ray analysis is used to provide elemental identification and quantitative compositional information. EDX image fig. 5.shows that the silica contains the elements such as silicon (Si) and oxygen (O).

Table 1.EDX Data of silica from CHA

SL. NO	Element	Weight%	Atomic %
1	O	68.02	78.87
2	Si	31.98	21.13

A weight percentage of oxygen and silicon ratio is 68.02: 31.98 and the atomic percentage of oxygen and silicon ratio is 78.87: 21.13. The weight percentage and atomic percentage of Oxygen possess higher percentage than Silicon. The data confirmed the presence of silicon and oxygen in the sample of synthesis nanosilica particles.

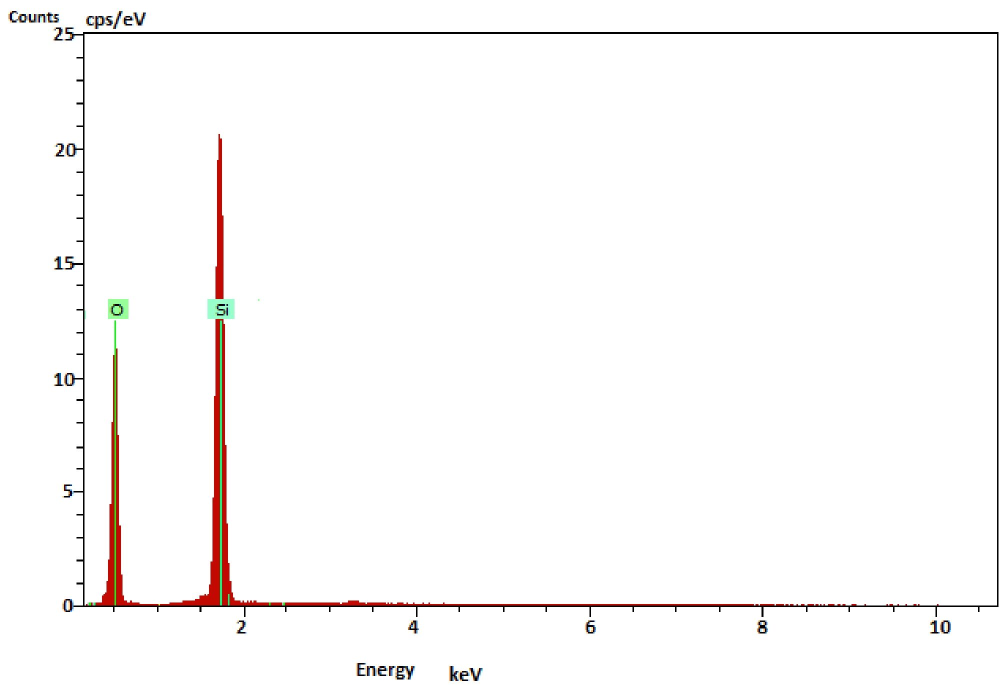

Figure.5: EDX Image of Silica

CONCLUSION

Silica was successfully synthesized from coconut husk ash. Silica is a useful material for industrial applications. The sharp peaks of XRD pattern confirm the nature of the silica possess crystalline nature. EDX spectrum demonstrated the pure silica nanoparticles are obtained.This method was used to synthesis the nanosilica is inexpensive, sustainable and also suitable for large scale production.

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