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SYNTHESIS AND CHARACTERISATION OF SILICA NANO PARTICLES FROM COCONUT SHELL

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ABSTRACT

Silica nano particles are produced from coconut shells .Coconut shells are cleaned, dried and made into pieces. Then it is burned in a muffle furnace at 700°C for 5 hrs to get Coconut Shell Ash (CSA) containing silica nanoparticles. The CSA is made into fine powder and stirred with 2.5N sodium hydroxide solution for 4 hours at 100ºC to produce sodium silicate solution. The filtered solution and stirred with 5N sulphuric acid at 100ºC to precipitate silica gel. The gel is washed to remove sulphates impurities and heated in oven at 70ºC for 15 hours to form amorphous silica. The amorphous silica is refluxed at 90ºC with 6N HCl solution for 6 hours and washed with warm water to remove alkali.Then the solution is treated with 2.5 N HCl and H_2SO_4 to form white precipitate. The white precipitate is dried in hot air oven for 30 hours to get silica nano particle. The formed silica nano particles were characterized by FTIR, XRD and SEM.

KEY WORDS: **Silicon nanoparticles, coconut shell, coconut shell ash, chemical treatment**

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INTRODUCTION

Silica nanoparticle has found its application in the biomedical field such as biosensor design, drug delivery, cell labelling and cell separation. In the recent years, silica has been used for gene delivery to obtain more efficient DNA delivery vectors for both basic research and clinical trails[1].Agro-industrial wastes have recently attracted a great deal of attention as potential sources of novel green alternative such as biotransformation for fuels and other materials. Many of these wastes contain amorphous silica that can be transformed into crystalline nanoparticles [2]. At present, nano-silica materials is prepared by several methods such as vapor-phase reaction, sol–gel and thermal decomposition technique[3–6].These techniques are proved to be costly. Silicon is the second most abundant element in soil, which accounts for approximately 32% of the total weight of soil. As a consequence, plants rooting in soil always contain some silicon in their tissues. However, the role of silicon in plant growth has been overlooked for a long period, although silicon is presented in plants in amounts equivalent to macronutrient elements, such as calcium, magnesium and phosphorus[7]. In some plants biosilification process is common. In plants silicic acid is taken up through plant root and transported as a silicon complex through the xylem. This deposit on the stems or leaves in the form of silica nano particles which can be extracted by some methods[8]. Extraction of silica nanoparticles from the plants greatly reduces the cost of production. Amorphous nanosilica can be obtained from burnt and hot acid pre-treated rice hulls and straw[9]. Coconut shell is an agricultural by product whose major constituent is organic material and hydrated silicon. The use of coconut shell husk has the benefit not only of producing valuable silica but also of reducing disposal and pollution problems. The objective of this work is to develop a procedure for obtaining nanoscale silica with a high specific surface area from a coconut shell. This method has advantages like conversion of domestic waste into a useful product and greatly reduces the cost of raw material used for the production of silica nanoparticles. The formed silica nano particles are characterized by FTIR, XRD and SEM.

MATERIALS AND METHODS

Preparation of coconut shell

Around 30 coconut shells are collected from Sathyabama University mess, Chennai. The coconut shells were manually chosen and cleaned. Then coconut shells were washed thoroughly to remove dirt and other impurities. The washed coconut shells are dried to remove unbound moisture content in hot air oven (GUNA, 230 A/C, INDIA) for about 5 minutes at 100^0C. The coconut shell is made into small pieces by using jaw crusher (Lynx, Lawrence &Mayo, India). Proximate analysis was conducted using Thermo gravimetric analyzer (ELTRA's elemental analyzer) to determine the content of fixed carbon, volatiles, moisture and ash of raw coconut shell.

Preparation of coconut shell Ash

50 grams of coconut shell pieces is taken in a silica crucible and heated in muffle furnace (HI-TECH, SUNBIM, INDIA) for 5 hours at 700^0C.The volatile matter is removed and 8 grams of CSA content is left behind. CSA thus obtained is subjected to chemical treatment to recover silica nano particles.

Synthesis of silicon nanoparticles

10 grams of treated CSA is stirred with 80 ml of 2.5N sodium hydroxide solution prepared using triple distilled water. The solution was heated at 100^0C in a 250 ml beaker for 4 hours with constant stirring to dissolve the silica .The solution is filtered in a silica crucible and the residue is washed with warm distilled water. The ash is made into fine powder by using motor and pestle. Then the powder is stirred with 2.5N sodium hydroxide solution for 4 hours at 100ºC to produce sodium silicate solution. The solution is filtered and stirred with 5N sulphuric acid at 100ºC to precipitate silica gel. The gel is washed with deionised water to remove sulphates impurities and heated in oven at 70ºC for 15 hours to form amorphous silica. The amorphous silica is refluxed at 90ºC with 6N HCl solution for 6 hours and washed with warm water to remove alkali. Then the solution is treated with 2.5 N HCl and H_2SO_4 to form white precipitate. The white precipitate is dried in hot air oven for 30 hours to get silicon nano

particle. The formed silica nano particles were characterized by FTIR, XRD and SEM.

Characterization of Silica nanoparticles

SiO_2 powders were characterized by employing Fourier transform infrared spectrophotometer (FTIR) Bruker Vector 33 using KBr powder. Elemental analysis was carried out using energy dispersion spectroscopy (EDS) on a Leica stereo scan 440 instrument equipped with a phoenix energy dispersive x-ray. The scanning electron microscopy (SEM) characterizations were performed with JOEL JSM-6060 LV Scanning Electron Microscope.

RESULTS AND DISCUSSION

Thermal gravimetric analysis of coconut shell

The proximate analysis of the coconut shell shows high quantity of volatile matter, then fixed carbon, moisture content and least amount of ash content. Proximate analysis results of coconut shell are as shown in the table 1.

Table 1
Proximate analysis results of coconut shell

Fixed carbon (wt. %)	Volatile matter (wt. %)	Sample Moisture (wt. %)	Ash (wt. %)
21.75	71.06	8.53	0.66

The thermal gravimetric analysis of coconut shell shows that the weight loss accelerates from 200 °C, which can be attributed to the release of H_2O and CO_2 resulting from the decomposing of cellulose and lignin in coconut shell. The total weight loss of 84% by weight corresponds to decomposing of in coconut shell such as cellulose, lignin and others that can be removed by heating at 700 °C under a atmospheric condition. The rest of 8% by weight represents silica and metallic impurities in coconut shell.The chemical compositions of CSA obtained after combustion is shown in the tables 2. CSA contain high content of silicon and some impurities like sodium, potassium, calcium, iron, copper, magnesium, lead and manganese.

Table 2
Chemical composition of CSA after combustion

Component expressed as oxides	Weight percentage
SiO_2	88.09±0.02
Na_2O	0.362±0.004
K_2O	1.841±0.01
Fe_2O_3	0.481±0.002
CuO	0.001±0.0003
MgO	0.994±0.017 0
CaO	2.227±0.003
MnO	0.017±0.00010
PbO	0.001±0.0004

Synthesis of silicon nanoparticles

CSA sample is stirred with 80 ml of 2.5N sodium hydroxide solution prepared using triple distilled water. The solution was heated at 100^0C in a 250 ml beaker for 4 hours with constant stirring to dissolve the silica and produce sodium silicate solution. The solution is filtered in a silica crucible and the residue is washed with warm distilled water. The obtained viscous, transparent, colorless sodium silicate solution is allowed to cool to room temperature [10]. The reaction is

$$\underset{(\text{Ash})}{SiO_2} + \underset{(\text{Caustic soda})}{2NaOH} \rightarrow \underset{(\text{Sodium silicate})}{Na_2SiO_3} + \underset{(\text{Water})}{H_2O} \text{ ------(1)}$$

Pure silica can be extracted by adding 5N H_2SO_4 solution under constant stirring at 100°C in normal atmospheric pressure. The acidic condition pH 2 indicates the complete precipitation of silica from sodium silicate by the reaction.

$$\underset{(\text{Sodium silicate})}{Na_2SiO_3} + H_2SO_4 \rightarrow \underset{(\text{Silica})}{SiO_2} + \underset{(\text{Sodium sulphate})}{Na_2SO_4} + H_2O \text{ ------ (2)}$$

The gel formed is washed with warm distilled water to remove sulphate impurities. Then it is kept in a hot air oven at 70ºC for 15 hours to produce 98% pure amorphous silica.Nanosilica particles are obtained by refluxing amorphous silica. About 10 ml of 6N HCl solution is added to amorphous silica and refluxed for 18 hours at 90°C. Then 80 ml of 2.5N NaOH is added and continuous stirred using a magnetic stirrer for 10 hours. Then concentrated H_2SO_4 is added to obtain white color precipitate. It is then kept in hot air oven for a period of 15 hours to get silica gels. The obtained gels are washed with warm distilled water. The silica gels are further dried in hot air oven for 12 hours. Silica nano particle powder is obtained.

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Characterization of silica nanoparticles

FTIR analysis

Figure 1 shows the FTIR analysis to the samples obtained from the coconut shell in the range of 500-4000 cm^{-1} with a resolution of $4cm^{-1}$

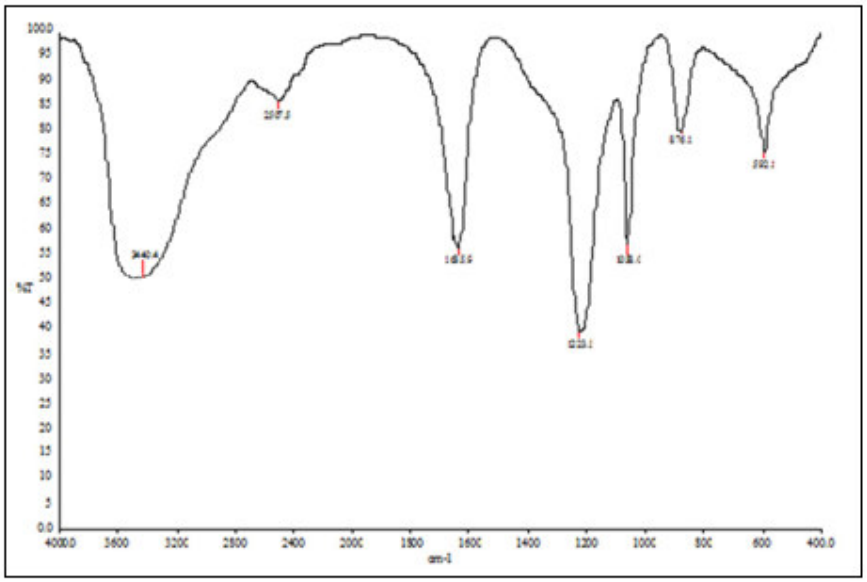

Figure 1

FTIR of silica nanoparticles from coconut shell ash

The spectra shows five important band for identification of the SiO_2 nanoparticles. A band at 3440.4 is in the range of O-H stretching vibration that is contributed by the O-H group of anti-symmetric vibration. Weak bands at 2507, 1635.9 correspond to the Si-H stretching vibration. A band at 1058.0 represent a band assigned to the anti symmetric stretching mode of Si-O-Si group and the deformation of the Si-H group. Band at 876.1 correspond to absorption peak of Si-O group. Another band at 592.1 corresponds to the bending vibration of Si-O-Si band. The bands corresponding to O–H vibration mode and H–O–H twisting vibration mode indicate that particles synthesized remain hydrate.SEM

measurements of the silica nano particle was performed on a Leica stereo scan 440 instrument equipped with a phoenix energy dispersive x-ray (EDS instrument).The table-3 shows the high percentage of silica. The value is calculated by considering mean low curve area in the peaks from XRD.The EDS results Figure 2 shows the presence of high silica content. Trace amount of sodium, sulphur, potassium and oxygen was also present in sample.

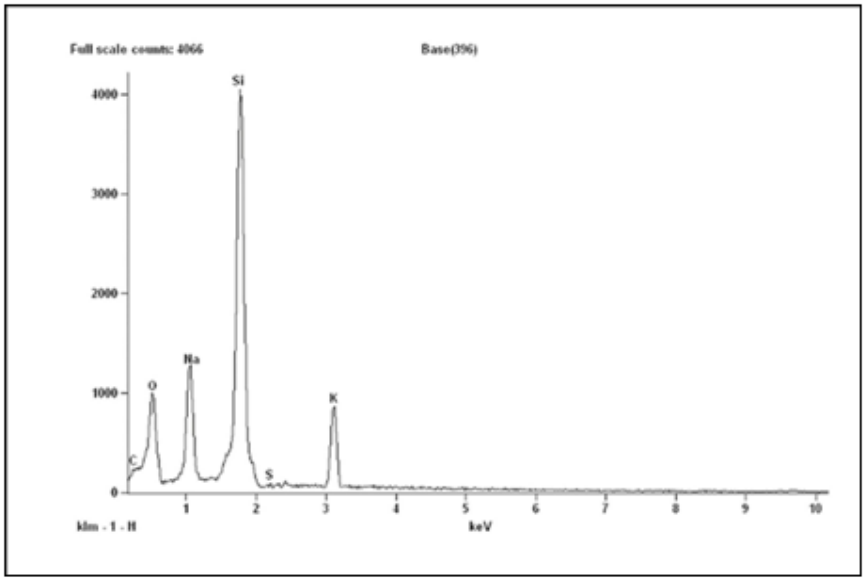

Figure 2
EDS of Nanosilica obtained from coconut shell ash

Table 3
Elemental composition of silica nanoparticles by EDAX

Element	Percentage by weight
C	0.301
O	0.867
Na	0.76
Si	98.01
S	0.062
Total	100

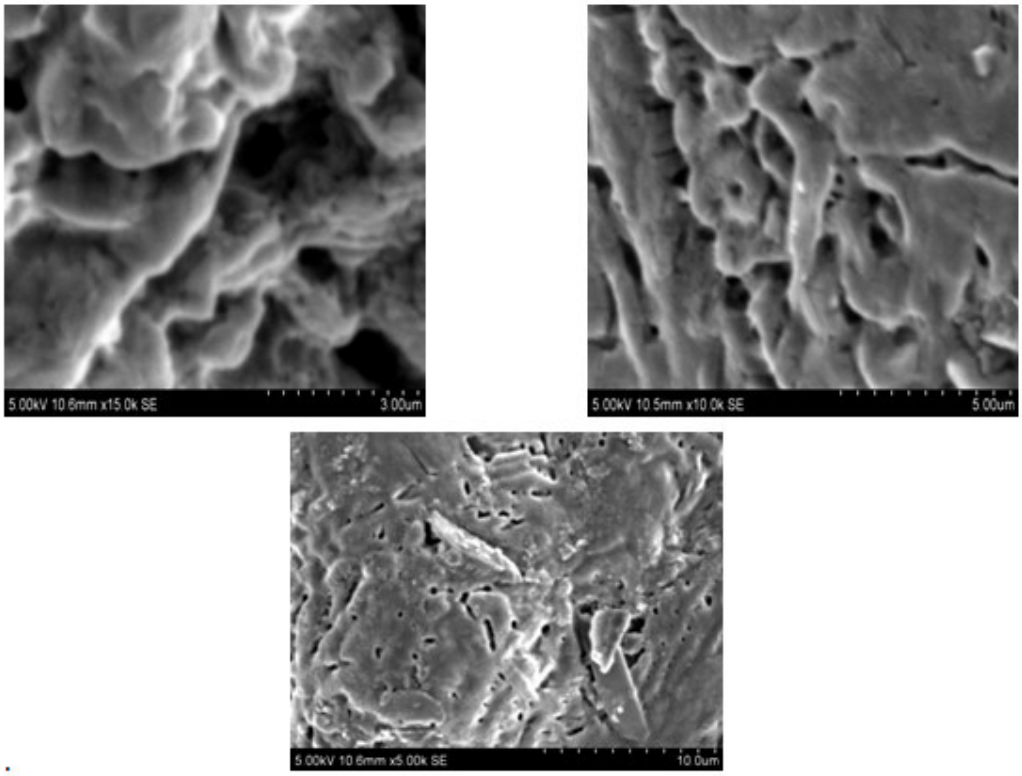

Figure 5
SEM of nanosilica obtain from chemically treated CSA at 3µm, 5µm and 10µm.

The SEM of nanosilica provides further insight of the morphology and size details of the silica nano particles. The silica nanoparticles particles were non uniform as shown in Figure-5. This might may be due to the to the fact that, in coconut shell the silica form rigid caves that surround the organic matter.

CONCLUSION

The need for well-defined SiO_2 nano particles is becoming increasing now a days. Coconut shell a common household waste burnt at controlled temperature produces large amount CSA. The CSA can be subjected to chemical treatment to recover SiO_2 nano particles. The use of domestic waste as a raw material reduces the environmental risk and cost of production silica nano particles.

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