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PREPARATION OF SUBMICRON SILICA GEL FROM RICE HUSK ASH USING WATER SHAKER BATH

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Abstract: Rice husk ash (RHA) is an agricultural waste and is rich in silica. Silica gel was extracted from RHA by burning, thoroughly washed and dried rice husk into ash in an enclosed drum, the ash was thereafter conditioned in a muffle furnace at a temperature of 650^{0}C; the conditioned ash was leached with various concentrations (0.5, 1.0, 1.5 and 2.0M) of sodium hydroxide (NaOH) in a water shaker bath at 100^{0}C for 1 hour, the solution was filtered with ashless filter paper number 41 and the filterate was precipitated with concentrated hydrochloric acid (HCl) to obtain solution until pH 7and incubated for 48 hours to promote silica gel formation. The silica gel produced was separated from soluble salt solution by vacuum filteration and washed with deionized water. The silica gel was dried in an air blast oven at 150^{0}C for 48 hours and ground into powder. The presence of silica in the gel was confirmed with Energy Dispersive X-ray Spectrometer (EDX). The chemical composition of the silica increases with increase in the concentration of NaOH. The EDX pattern of silica gel produced at 2.0 M NaOH treatment shows the composition of silica to be 96.28 wt.% . Horiba dynamic light scattering particle size analyser also revealed the particle size of the silica gel to be 427.1 nm.

Keywords: Rice Husk, Rice Husk Ash, Sodium hydroxide, Hydrochloric acid, silica gel

INTRODUCTION

At present, crude silicon known as metallurgical grade silicon, with 98-99% purity is obtained from quartz rocks by carbothermic reduction using electric arc furnace (Prasad and Pandey, 2012). This silicon is further refined into high purity silicon through expensive and complicated purification process such as chemical vapor deposition (CVD) process which is used for production of electronic grade silicon. Due to exponential growth of PV industry, the demand for solar grade silicon (SoG-Si) has increased tremendously over the past decade due to their extraordinary properties and their existing and potential applications in science and technology, silica gel has a wide range of applications such as dessicant, as a preservative tool to control humidity, as an adsorbent, as a catalyst and as a catalyst support (Prasad and Pandey, 2012).

At the beginning of PV-activities in 1980s, the high purity scrap silicon from the micro electronic industry was used by the PV-industry (Muller et al; 2006). However, increased demand that surpassed the limited supply of off-specification electronic grade silicon has created a thrust towards developing a dedicated technology for the production of solar grade silicon (Barry et al; 2008).

One of the approaches towards generation of SoG-Si is utilizing materials of very high purity to produce silicon. As an industrial waste, rice husk (RH) could be one of the potential raw materials for the production of solar grade silicon that can be used to develop siliceous particulate for use in solar panel assembly. Rice husk ash (RHA) is usually obtained by burning rice husk as fuel to generate energy from waste product).

Although various uses for rice husk and RHA has been suggested in the literature, their disposal or utilization remains a major concern. Soluble silicates produced from silica are widely used in glass, ceramics and cement as a major component and in pharmaceuticals, cosmetics and detergents industries as a bonding and adhesives agents (Anon, 1997; Laxamana, 1982). Silica also has been used as a major precursor for variety of inorganic and organometallic materials which have applications in synthetic chemistry as catalysts, and in thin films or coatings for electronic and optical materials (Lender and Ruiter 1990; Brinkerand and Scherer, 1990)

MATERIALS AND METHODS

The main materials that were used for this work are as follows: rice husk, distilled water, sodium hydroxide, hydrochloric acid and ashless filter paper.

Combustion of Rice Husk

The rice husk was collected from a local mill in Ajaokuta Steel City, Nigeria; It was thoroughly washed with water and dried in an oven at 80^{0}C for 24 hours to remove the water content. The dried rice husk was fed into an enclosed drum and burnt into ash, and thereafter conditioned at a temperature of 650 ^{0}C in a muffle furnace. The obtained rice husk ash (RHA) was used in the experimental work.

Extraction of Silica Gel from RHA

The Silica-gel was extracted from the rice husk ash by adding 10 grams of rice husk ash to various volumes and concentrations of sodium hydroxide solution (most solutions were 0.5 M= 660 ml, 1.0 M = 330 ml, 1.5 M = 247.5 ml and 2.0M = 165 ml sodium hydroxide (NaOH) solution. The mixture was then heated in a water shaker bath at 100^{0}C

in a beaker for one hour. The solutions were allowed to cool to room temperature, then, filtered through Whatman No 41 ashless filter paper and the carbon residue was washed with 100 ml of de-ionized water. Concentrated HCl was added to the obtained solution until pH 7.0 and incubated for 48 hours to promote silica gel formation. The silica gel produced was separated from soluble salt solution by vacuum filtration and washed with de-ionized water. Then silica gel was dried in an oven at 150^{0}C for 48 hours. The obtained white gel was pulverized into a powdery form.

Chemical Composition Analysis of Silica gels

The silicon content of the samples was estimated using energy dispersive X-ray (EDX) spectroscopy (Kevex Instruments, Valencia, CA).

Particle Size Analysis

The particle size of the powder was analyzed using Horiba dynamic light scattering particle size analyzer. The measurable particle size range of the instrument is 0.05-3000 µm and it is equipped with a small volume sample dispersion unit. A lens range of 300RF, a beam length of 2.4mm, and a presentation of 30AD with polydisperse analysis was used for this measurement. About 0.5 g of the silica powder was dispersed in de-ionized water in the sample dispersion unit of the instrument, vigorously mixed for about two minutes at speed of 2100 rpm, and sonicated for 45 seconds. The ultrasonic waves were used to break or minimize any particle agglomerates that may be present in the suspension. Measurements were taken and the diffraction data and graphs recorded by the instrument software program.

Examination of the surface morphology of Silica gels

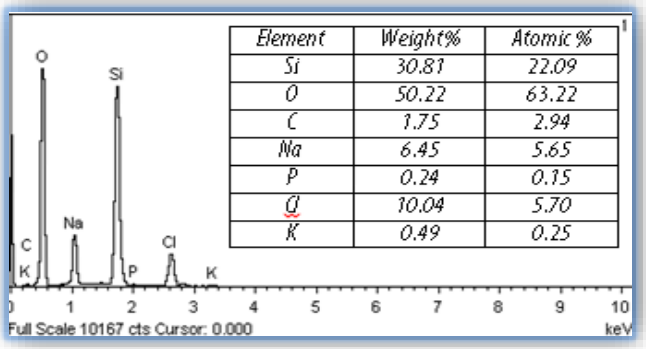
An AURIGA Scanning Electron Microscope (SEM) (Carl Zeiss Germany) with an accelerating voltage of 15KV was used to characterize the particle morphologies of the silica powder. Sample specimens were gold coated in a gold sputter coater for 90 seconds at 15 mA current output. The gold coating was necessary to ensure a conducting surface was obtained for electron bombardment and characterization. The Selected areas of interest were focused and micrographs were taken.

RESULTS AND DISCUSSIONS

Effect of Concentration of Sodium Hydroxide on Pure Silica Powder

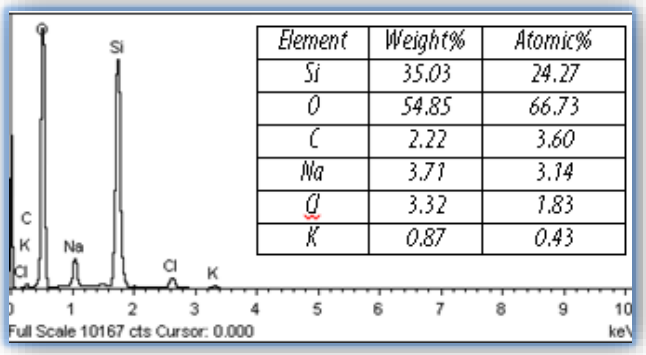
Energy Dispersive X-ray Spectrometric analysis was carried out on the silica powder at different concentrations of sodium hydroxide (0.5, 1, 1.5 and 2.0 M) in other to evaluate the effectiveness of the purification parameter and to confirm the presence of silica.

The results of the chemical composition of RHA and silica gels analyzed with Energy Dispersive X-ray Spectrometer (EDS) are shown in Figures 1- 5. The major elements present are silicon and oxygen along with traces of impurities such as sodium, chlorine, carbon, phosphorus and potassium. The elemental composition of silicon and oxygen increases with increase in the concentration of NaOH whereas the composition of the impurities such as sodium, chlorine and phosphorus decreases. Figure 4 shows the EDX spectra of silica powder leached with 2.0M NaOH which has the highest silicon and oxygen content .



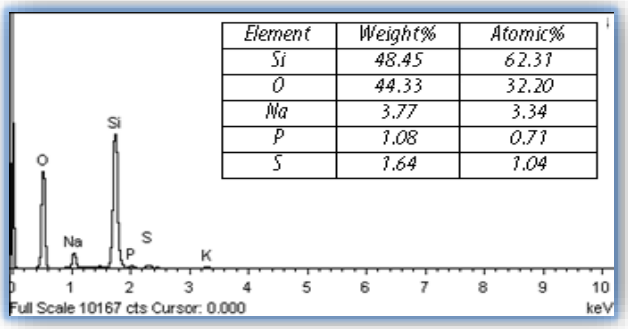
Element	Weight%	Atomic %
Si	30.81	22.09
O	50.22	63.22
C	1.75	2.94
Na	6.45	5.65
P	0.24	0.15
Cl	10.04	5.70
K	0.49	0.25

Figure 1: EDX spectrometric data of silica extracted from RHA with 0.5M NaOH



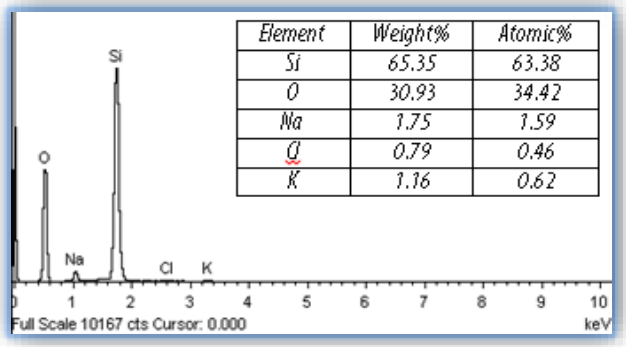
Element	Weight%	Atomic%
Si	35.03	24.27
O	54.85	66.73
C	2.22	3.60
Na	3.71	3.14
Cl	3.32	1.83
K	0.87	0.43

Figure 2: EDX spectrometric data of silica extracted from RHA with 1.0M NaOH



Element	Weight%	Atomic%
Si	48.45	62.31
O	44.33	32.20
Na	3.77	3.34
P	1.08	0.71
S	1.64	1.04

Figure 3: EDX spectrometric data of silica extracted from RHA with 1.5M NaOH



Element	Weight%	Atomic%
Si	65.35	63.38
O	30.93	34.42
Na	1.75	1.59
Cl	0.79	0.46
K	1.16	0.62

Figure 4: EDX spectrometric data of silica extracted from RHA with 2.0M NaOH

Particle Size Analysis

The chemical composition of silica powder extracted from RHA has been shown to be mainly silica (SiO_2). Since it is a known fact that particle size of filler materials has influence on the properties of composite, therefore, the cumulative particle size distribution of powder with the highest silica content was analyzed with Horiba dynamic light scattering particle size analyzer as show in Figue 3. The particle size distribution of the powder is approximately 427.1nm. The result of the average particle size of the silica gel is shown in Figure 5.

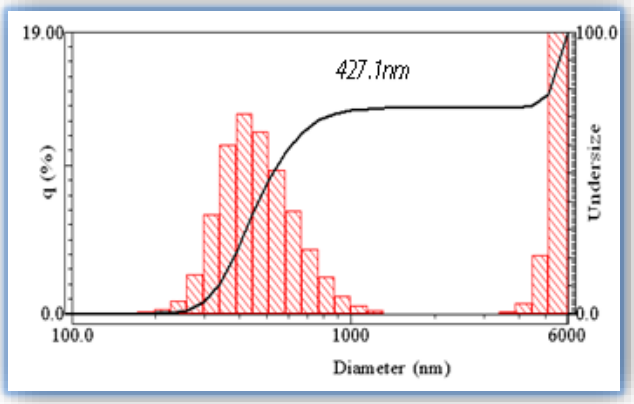
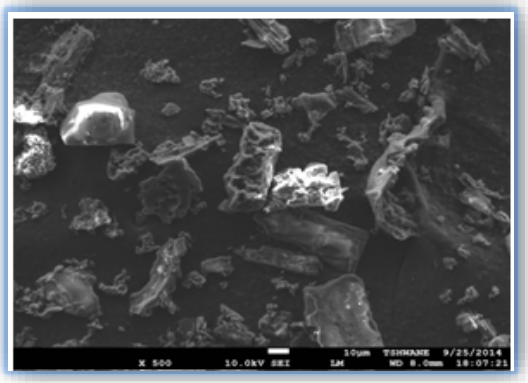


Figure 5: Particle size distribution by intensity produced by Horiba Dynamic Light Scattering Particle Size Analyzer. The average Particle size is 427.1nm

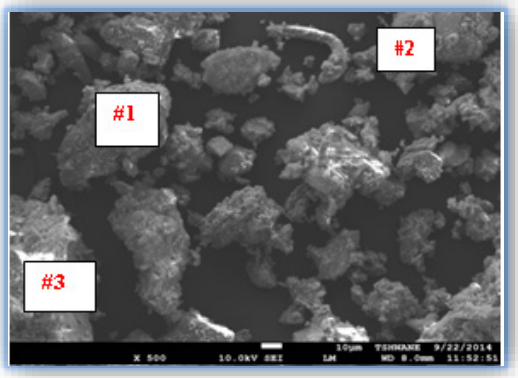
Scanning Electron Microscope (SEM) Images of RHA and Silica gel

The morphological features of the RHA and silica powder observed by scanning electron microscope (SEM) are shown in Plates (a-e). The SEM images were taken at a magnification of 500X, as from Plate (a), the as received RHA shows a porous and multifaceted particle shape and size. The honeycomb and porous morphology seen in Plate(a) can be attributed to burning out of organic component in the rice husk during combustion. The hydrated silica subsequently polymerizes to form a skeletal silica network which may explain the flaky and honeycomb-like structure in the SEM image of Plate (a).

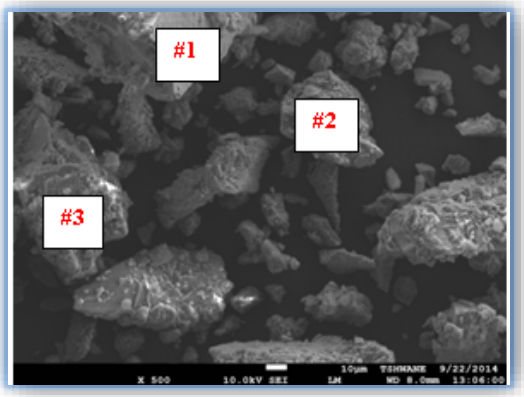
The EDX analysis at points #1, #2 and #3 in Plates (b-e) shows the crusty and fibrous surface to be silicon-rich and was mainly SiO_2 . The average elemental composition for each silica powder produced was calculated by adding the elemental composition of point #1, #2 and #3 and the average was calculated as shown in Figures 1-4.



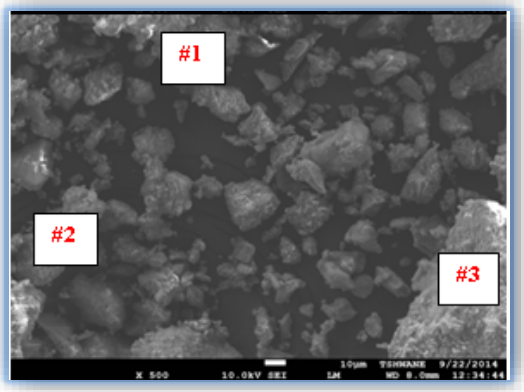
a)



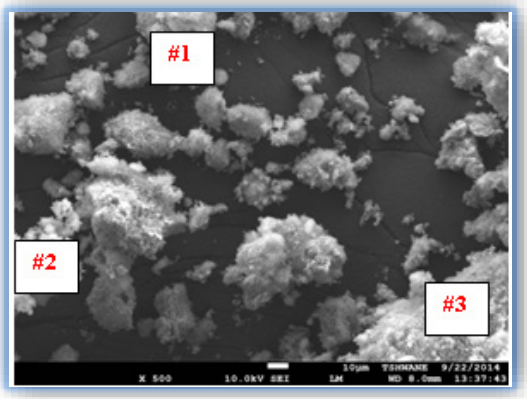
b)



c)



d)



e)

***Plates (a- e)**: SEM Images of RHA and silica gel extracted from RHA by Sol-gel process with NaOH (a) RHA (b) 0.5M NaOH (c) 1.0M NaOH (d) 1.5M NaOH (e) 2.0M NaOH*

CONCLUSION

This study revealed that silica gel and powder can be extracted from RHA, which is regarded as agricultural waste which can be used for several applications in industry. It has been confirmed that the concentration of NaOH when leached with RHA influences the purity of silica gel; The EDX pattern of submicron silica powder at various concentrations (0.5,1.0,1.5 and 2.0M) has shown that the elemental composition of silicon and oxygen increased, whereas impurities such as carbon, sodium, phosphorus,, chlorine and potassium decreased at 2.0M NaOH treatment. The silica extraction yield from RHA was 96.28 wt.% at 2.0M NaOH treatment. The average particle size of the silica powder as determined by particle size analyzer was 427.1nm which is regarded as submicron particle.

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