

Extraction and characterization of Amorphous Silica from Corn Cob Ash by Sol-Gel Method

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Abstract

Extraction of amorphous silica from Corn cob Ash (CCA) was carried out in this study. Silica xerogel was produced by dissolving CCA with alkali solution to form sodium silicate solution and lowering the pH to7.0 by adding hydrochloric acid to form silica aquagel followed by drying to form silica xerogel. The silica xerogel was characterized using XRF, XRD and FTIR techniques. Silica and mineral contents of CCA and xerogel were determined by XRF, X-ray diffraction patterns revealed amorphous nature of extracted silica. Fourier transform infrared (FTIR) data indicated the presence of siloxane and silanol groups. Silica yield from CCA was 52.32% while moisture content was 2.89%.

Keywords: corn cob ash; amorphous silica; xerogel; aquagel

1. Introduction

Silica has been widely used in pharmaceutical products, chromatograph column packing, detergents, adhesives, electronics, dental material, and ceramics. (Brinker and Scherer, 1990; Proctor *et al* 1995; Sun and Gong, 2001). Silica has also been used as a major precursor for a 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 (Kalapathy*, et al* 2002).

A low energy method to produce pure silica from rice husk ash with 91% extraction yield, has been developed (*Kalapathy et al*., 2000; Kamath and Proctor, 1998), acid leaching (Chakraverty and Kaleemullah, 1991) and gasification (Luan and Chou, 1990) methods have also been investigated for recovering silica from rice hull.

Corn, is a main staple food in Nigeria, with an annual production of 9.4 million tons, The cobs produced from corn are mainly used as manure for agricultural production. (Latif and Rajoka 2001). Silica has been successfully extracted from different agricultural materials like rice husk (Kamath, and Proctor, 1998, Polska and Radzki, 2008; Yalcin and Sevinc 2001; Sidheswaran and Bhat 1996), sugar cane Bagasse (Affandi *et al* 2009, and Espindola-Gonzalez *et al* 2010) coffee husk (Espindola-Gonzalez *et al* 2010) and wheat husk (javed et al 2011). Mohanraj *et al* (2012), Prepared and characterized nano SiO_2 from corn cob ash by precipitation method.

Hence, objective of this work is to extract amorphous silica from corn cob Ash, using the sol gel technique. This method has dual benefit; valuable silica particle at lower cost can be produce with reducing disposal as well as pollution challenges. The prepared amorphous silica has been characterized using XRF, XRD, and FTIR technique.

2. Materials and Methods

Corn cob (zea rugosa), was obtained from south western Nigeria, after the harvest seasons, the corn cobs was washed and sun dried. The combustion were carried out at 650°C for 3 hours at a heating rate of 10°C/min in a muffle furnace, and left over night to cool in the furnace.

2.1 Silica Extraction

Silica was extracted from Corn Cob Ash, (CCA) adapting the method of Kamath and Proctor (1998), 250 ml portions of 1N NaOH were added to 50g CCA samples and boiled in covered 500ml Erlenmeyer flask for 1 h with constant stirring to dissolve the silica and produce a sodium silicate solution. The solutions were filtered through a whatman No. 41 ashless filter paper, and the residue washed with boiled distilled water. The filtrate was allowed to cool to room temperature and the pH of the solution was reduced with 3N HCl to 7.0 with constant stirring, and incubated to promote gel formation. When the gel formed from the sol, it was aged for 18 hours. After ageing the soft gel was gently broken and the slurry was centrifuged at 4000 rpm for 5min, the supernatant were discarded and the gels transferred into a beaker and dried at 80^{o}C for 24 hours to produce xerogels. The Silica xerogel were subjected to additional washing with deionized water to effectively remove minerals and impurity from the silica. This is because; washing the dried silica (xero-gel) with deionized water is more effective than washing the silica gel (aqua-gel) prior to drying, in removing minerals from the silica. (Prasad and Pandey 2012)

2.2 Moisture content of silica gels

Moisture content of the silica gels was determined using an air oven method (AOAC, 1990). About 1 g of the sample was heated in aluminum moisture pans at 130^{o}C for 1 h. The samples were cooled in a desiccator and weighed. The weight loss (%) was recorded as the moisture content of samples.

2.2.2 Characterization.

X-ray diffraction (XRD) of the corn cob ash and silica extract were monitored using X-ray minidiffractometer MD-10, CuKα radiation (Radicon limited, Russia) using an acceleration voltage of 25 kV and current of 400 µA. The diffraction angle was scanned from 10^{o} to 75^{o} 2θ, at a rate of 3.25^{o} /min. The quantitative analysis of chemical components was done using X-ray fluorescence (XRF). The major chemical groups present in the silica were identified by FTIR.

3. Results and Discussions

The silica and moisture content of the washed xerogel were 52.32% and 2.89% respectively.The The X-ray diffractogram of amorphous silica extracted from corn cob ash is shown in Figure 1. The broad X-ray diffraction pattern which is typical of amorphous solids (Kamath and Proctor, 1998) shows that the extracted silica is predominantly amorphous. Diffraction peak at theta = 22 degree confirms the formation of amorphous silica, in general, it has been reported that Diffraction broad peak at theta = 22 degree indicates amorphous silica along with some crystalline silica (Javed *et al* 2011).

The major chemical groups present in the extracted silica were identified by FTIR spectral as shown in figure 2. The broad band at 3410cm^{-1}to 3470 cm^{-1} is due to the stretching vibration of the O-H bond from the silanol groups (Si-OH) and is due to the adsorbed water molecules on the silica surface.(Javed *et al* 2011, Nayak 2010). The band at 1070 cm^{-1} to 1090cm^{-1} is due to the Si-O-Si asymmetric stretching vibration, while the band at 791cm^{-1} to 806 cm^{-1} has been assigned to the network Si-O-Si symmetric bond stretching vibration. (Nayak 2010) The band at 461cm^{-1} to 476 cm-$^{-1}$ is associated with a network O-Si-O bending vibration (javed *et al* 2011) modes. The chemical composition of CCA and silica extract are shown in table 1

4. Conclusion

This study revealed that silica xerogels with 52.32% silica content and minimal mineral contaminants can be produced from CCA using the sol gel method. The silica extracted yield from CCA was 50% while moisture content was 2.89%. XRD analysis reveals the amorphous nature of the silica; Fourier transform infrared (FTIR) data indicated the presence of siloxane and silanol groups

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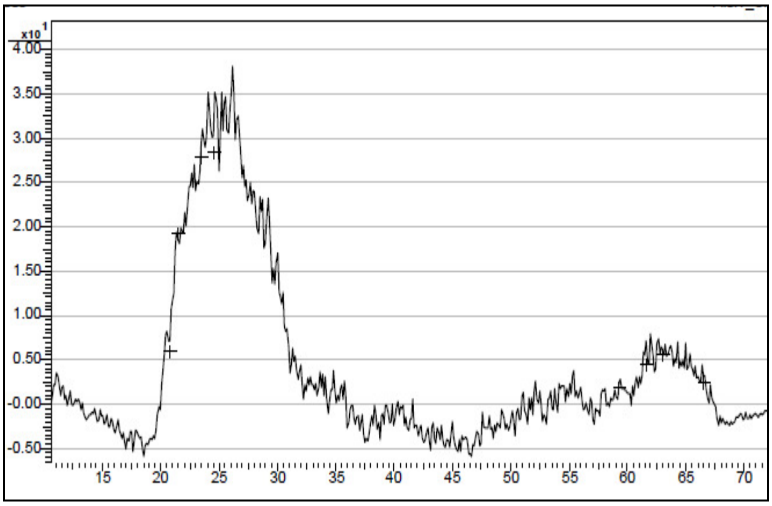

Figure 1. XRD of extracted amorphous silica

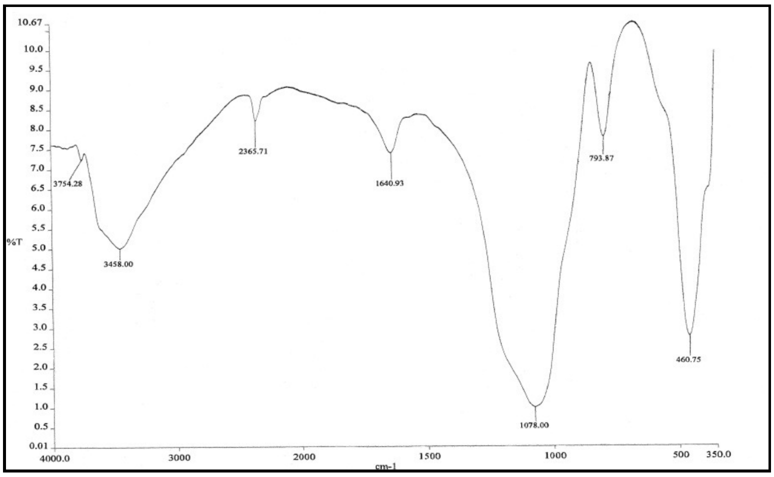

Figure 2. Fourier transform infrared spectra of silica produced from CCA

Table 1. Chemical composition of corn cob ash and silica extracted

Constituent	Amount present in corn cob ash (wt %)	After extraction (wt %)
SiO_2	47.66	97.13
Al_2O_3	8.50	0.00
FeO_2	7.90	0.48
CaO	17.70	0.89
MgO	7.20	0.92
SO_3	0.70	0.00
MnO_2	2.20	0.00
K_2O	4.80	0.58
NaO_2	1.67	0.00