

IPACT-2013[14<sup>th</sup> – 15<sup>th</sup> March 2013]

National Conference on Industrial Pollution And Control Technology-2013

## Studies On Synthesized Nanosilica Obtained From Bagasse Ash

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**Abstract:** Bagasse is one of the important biomass sources, which is used as a fuel in the sugar industry. As a result, large quantities of ash were generated and create a serious disposal problem. The aim of the present work is to utilize the waste material bagasse ash as a valuable material to obtain nanosilica. The high percentages of pure and nano sized silica was synthesized from bagasse ash by solgel with reflux method. The synthesized nanosilica characteristic parameter were studied using various techniques such as XRD, FTIR and SEM with EDS. X-ray powder diffraction pattern of the nanosilica confirms the amorphous nature of silica. SEM results show that the treated bagasse ash have different morphology with various size while synthesized nanosilica is in spherical with nano level.

**Key Words:** Bagasse ash, Silica, XRD, FTIR, SEM.

### 1. Introduction

India is the second largest sugarcane producer in the world. From the sugarcane industry, bagasse is the residue from the milling process. Burning bagasse as an energy source yields it ash, considered as a waste causing disposal problems. In India about 10 million tons of sugarcane bagasse ash as a un-utilized and hence waste material<sup>1</sup>. Sugarcane bagasse ash is one of bio waste from sugarcane industry. Several studies<sup>2,3</sup> have finalized silica is rich in sugarcane bagasse ash. Fine silica powder is used for the wide ranged applications in the research field including electronic substrates, thermal and electrical insulators and optoelectronic devices, etc<sup>4,5</sup>. However research has focussed mainly on the utilization of industrial waste as a silica source. Hence an attempt has been made to synthesize nanosilica by thermochemical method from the treated bagasse ash. Synthesized nanosilica has been characterized through XRD, FTIR and SEM with EDS. Utilizing either industrial or agricultural waste as a source of raw materials and this waste utilization would not only be economical, but may also result to environmental pollution control.

### 2. Materials And Methods

Sugarcane bagasse ash was collected from uncontrolled burning of Sugarcane bagasse after electric power generation in Chengalvarayan Co-operative sugar industry, Periyasevalai, Thirukovilur Taluk, Tamilnadu, India.

Sugarcane bagasse ash was cleaned, dried and calcined through a heating rate of 300° C/h and then held at 650° C for 2 h. At 500° C the organic compounds decomposed off and at 650° C large amount of ash with high silica (72%) content was obtained<sup>6</sup>.

Nanosilica was synthesized by thermochemical method<sup>7</sup> and slightly modified was adopted. The powder X-ray diffraction pattern of the nanosilica derived from the bagasse ash was carried out by Rigaku made X-ray diffractometer available at NIT, Trichy in the range of  $2\theta = 10 - 80^\circ$ . FTIR spectrum was recorded with Perkin Elmer RX FTIR Spectrometer in the range of  $4000 - 400\text{ cm}^{-1}$ . The microscopic study of the samples was determined by JEOL JSM 5610 LV SEM with by EDS available at Centralised Instrumentation and Service Laboratory [CISL], Annamalai University.

### 3. Results And Discussion

#### 3.1 Synthesis Of Nanosilica

A 10 g of treated bagasse ash (BGA) was dissolved in 80 ml of 2.5 N sodium hydroxide solution under constant stirring with boiling at 100° C for 3 h. After boiling the solution was filtered in a silica crucible. The obtained colourless, transparent, denser sodium silicate solution was allowed to cool at room temperature<sup>8</sup>. After filtration process the sodium silicate solution was titrated with HCl. Under constant stirring, 5N HCl was added drop by drop in a sodium silicate solution, the white silica gel was formed.

The acidic condition pH level of 2 indicates the complete precipitation of silica gel. Constant stirring and maintaining the temperature in the range of 90° - 100° C for 5 h changes the pH to 10, which represents the breaking of silica gel bond and in turn pure silica particles settles down. The silica particles were washed by deionised water for neutrality and then dried at 70° C for 17 h. The pure silica was used to synthesize nanosilica through reflux method.

Puresilica with 6N HCl solution was subjected to continuous refluxing the temperature at 80 - 90° C for 8 h. After refluxing the sample was washed with more amount of warm distilled water to become alkali free and then hot air oven dried. Afterwards 80 ml of 2.5 N sodium hydroxide was added to the silica powder under constant stirring for 9 h. Then con HCl was added to the solution until a white precipitate was formed. The precipitate was washed thoroughly by warm triple distilled water for neutrality. The synthesized nanosilica was dried in a hot air oven at 110° C for 24 h.

#### 3.2 Studies On Nanosilica

The powder X-ray diffraction pattern of nanosilica is shown in Figure 1. A strong broad peak centered at  $22^\circ (2\theta)$  confirms the amorphous nature of silica which is supposed to be the characteristic of amorphous  $\text{SiO}_2$ . Due to thermochemical process, the crystalline particles transforms into amorphous silica which is indicated by broad peak<sup>9</sup>.

Fourier Transform Infra Red (FTIR) spectrum of nanosilica is presented in Figure 2. A large broad band at  $3440\text{ cm}^{-1}$  is assigned to the presence of the OH stretching frequency of silanol groups<sup>10</sup>. A strong intense band at  $1100\text{ cm}^{-1}$  is associated to the siloxane Si-O-Si vibration of the molecules. Amorphous silica exhibits a relatively strong peak around at  $804\text{ cm}^{-1}$ . The peak at  $475\text{ cm}^{-1}$  is due to Si-O bending mode of vibration.

Scanning Electron Microscope (SEM) in combination with EDS (Energy Dispersive Spectroscopy) was used to investigate the morphological property of individual particles and the percentage of the elements present. SEM image and EDS of treated BGA are given in Figure 3. BGA particles with various sizes and geometry like prismatic, spherical and fibrous were recognized. The results of SEM with EDS suggested prismatic particles consist mainly of Silica only. The spherical ones contain Si as well as Na, K, Al, Fe, Mg and  $\text{Ca}^3$ . The percentage of silica in the ash is 72% with some metallic impurities.

The nanosilica analysis provided micrograph with EDS of nanosilica particle is shown in Figure 4. The morphology of synthesized nanosilica is observed as spherical in shape<sup>11</sup> and it exhibits the agglomerated particle with uniform size in the range of 90 nm. The presence of the nano-sized spherical particles leads to a highly porous structure. Nanosilica's EDS peaks predominantly with element of Si and silica of 99% purity.

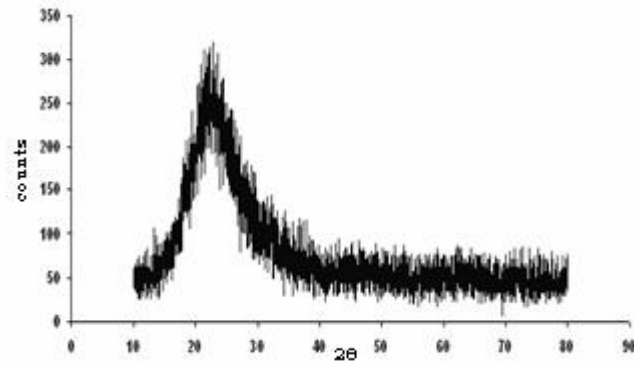


Figure 1: XRD spectrum of nanosilica

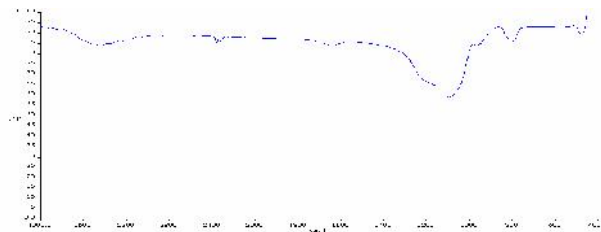
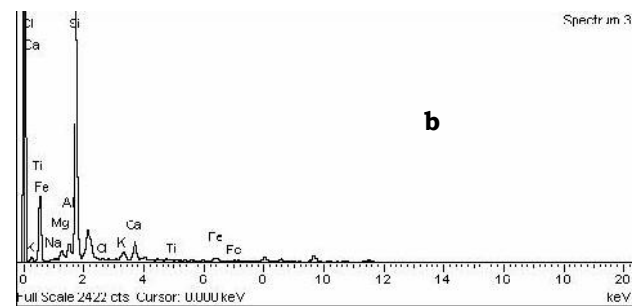


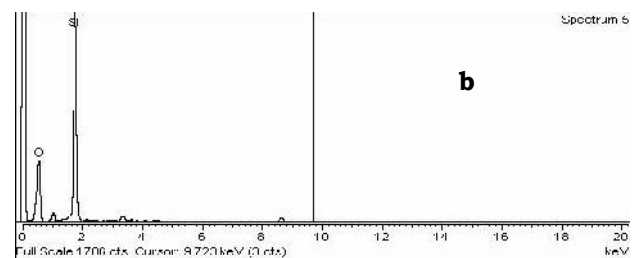
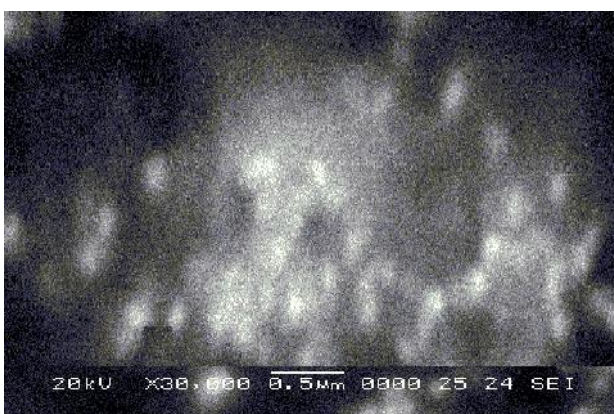
Figure 2: FTIR spectrum of nanosilica



a

b

Figure 3: (a) SEM and (b) EDS of bagasse ash



a

b

Figure 4: (a) SEM and (b) EDS of nanosilica

## Conclusion

By controlled burning of bagasse ash at 650° C for 2 h, obtained a reactive material relatively free from organic matter. Metallic impurities with trace elements in bagasse ash got destroyed in the formation of nanosilica. XRD spectrum shows that the silica is in amorphous nature. FTIR peak confirmed that the nanosilica belongs to the siloxane and silanol groups. SEM micrograph of nanosilica shows that the particles are almost spherical, homogeneous and agglomerated form. SEM-EDS report the silica particle is 90 nm in size and of 99% purity. The study reveals that the industrial waste material sugarcane bagasse ash acts as an alternative source for the production of silica.

## References

1. Ajay Goyal, Hattori kunio, Ogata and Mandula, "Properties and Reactivity of sugarcane Bagasse Ash," 12<sup>th</sup> International colloquim on structural and geotechnical engineering, 2007, vol 1, 1-5.
2. Faria K.C.P., Gurgel R.F., Holanda J.N.F., " Recycling of sugarcane bagasse ash waste in the production of clay bricks" Journal of Environmental Mangement, 2012 101, pp. 7 -12.
3. Aigbodin V.S., Hassan S.B., Ause T. and Nyior G.B., "Potential Utilization of solid Waste (Bagasse ash)" Journal of Minerals and Materials Characterization and Engineering, 2010 vol 9 [1], pp. 67 – 77, 2010.
4. Kalapathy U and Proctor A, Shultz J, " A Simple method for production of pure silica from rice hull ash", Bioresour. Technol., 2000, vol 73, pp. 257 – 262, 200.
5. Kamath S R., Proctor A., "Silica gel from rice hull ash: preparation and characterization", Ceral Chem, 1998, vol 75, pp. 484 – 487.
6. Amutha K., Ravibaskar R. and Sivakumar G., 2010, "Extraction, Synthesis and Characterization of Nanosilica from Rice husk ash," International Journal of Nanotechnology and Applications vol 4 (1), 61-66.
7. Nittaya Thuadaji, Apinon Nuntiya, "Preparation of Nanosilica powder from Rice husk Ash by precipitation method", Chjang Mai,J,Sci., 2008, 35 (1), 206-211.
8. Davinder mittal, " Silica from Ash a valuable product from waste material, Resoanance, July 1997.
9. Ajay Goyal, Hattori Kunio Ogata Hidehiko and Mandula, "*Properties and Reactivity of Sugarcane Bagasse ash*", <http://soil.en.a.u-tokyo.ac.jp/jsidre/search/PDFs/07/07006-35.pdf>, 2007.
10. Yuttapong Saporn, Sirilukchaiarakorn, Pojjanee Khummongkol and Nurak Grisdanurak, "Synthesis of Mesoporous Molecular Sieve MCM-41 by using Silica Obtained from Rice Husk Ash", Tungkrue Bangkok, Thailand, 2001.
11. G. Sivakumar and K. Amutha, "Studies on Silica obtained from Cow dung ash", Advanced Materials Research, 2012, 584, pp. 470-473.

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