academicJournals

Vol. 9(2), pp. 12-17, February, 2015
DOI: 10.5897/AJPAC2015.0603
Article Number: 06A7E2550929
ISSN 1996 - 0840

http://www.academicjournals.org/AJPAC

African Journal of Pure and Applied Chemistry

Full Length Research Paper

Extraction of silica gel from *Sorghum bicolour* (L.) moench bagasse ash

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Received 3 January, 2015; Accepted 12 February, 2015

Sweet sorghum (*Sorghum bicolor* (L.) moench), a crop that is grown by subsistence farmers in Zimbabwe was used to extract silica gel in order to assess its possible use as a raw material for the production of silica-based products. The gel was prepared from sodium silicate extracted from sweet sorghum bagasse ash by sodium hydroxide leaching. Results show that maximum yield can be obtained at pH 5 and with 3 M sodium concentration. The silica gel prepared at optimum pH 5 had a bulk density of 0.5626 g/cm^3 and anestimated porosity of 71.87%. Silica gel aged over 10 h had improved moisture adsorption properties. X-ray fluorescence (XRF) determinations show that the silica content in the ash is 40.1%. Characterization of sweet sorghum ash and silica gels produced at pH 5, 7 and 8.5 by Fourier Transform Infrared spectroscopy gave absorption bands similar to those reported by other researchers.Transmission electron micrographs show that silica prepared under optimum conditions is amorphous and consisted of irregular particles. Sweet sorghum proved to be a potential low cost raw material for the production of silica gel.

Key words: Silica gel, sweet sorghum, extraction, ash, bagasse, X-ray powder diffraction (XRD).

INTRODUCTION

Silica gel is a non-toxic and inert inorganic polymer composed of amorphous silicon dioxide. It is characterized by a large surface area of up to 800 m^2/g. This property and its ability to adsorb a number of compounds has been exploited in a number of ways especially in catalysis and sorbent technology. Silica has also been used as molecular sieves in petroleum refining, air separation and nuclear waste management, as stationary phase material in liquid chromatography and as reinforcement for rubber and plastic (Devi and Dhanalakshmi, 2012; Tongjian et al., 2009).

Silica gel is prepared commercially through acid precipitation from sodium silicate solution. The sodium silicate is prepared through high temperature reaction of quartz with soda ash. The main disadvantage of this approach is that it is energy intensive requiring temperatures of up to 1300°C and the silica product may contain heavy metal impurities making it unsuitable for certain applications where purity is crucial. Spherical silica gel particles can also be prepared through hydrolysis of Stoiber reagents such as tetraethoxysilane and tetramethoxysilane (Sing et al., 2011). These

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reagents have been reported to be hazardous and are generally very expensive making them unsuitable for large scale production (Dorcheh and Abbasi, 2008).

A number of researches have shown that silica gel can be extracted from plant waste (Worathanakul et al., 2009; Alayande et al., 2012). Extraction from rice husk ashes has been widely reported (Prasad and Pandey, 2012). Research has so far focused on the preparation of silica gels of various morphology e.g. nano particles, meso, macro and microporous particles (Noushad et al., 2012; Thuadaji and Nuntiya, 2008). Another potential silica source is the sugarcane bagasse ash. Ugheoke and Mamat (2012) reported a new approach of preparing ordered and disordered silica gel particles from bagasse ash. A cation exchange method was used to extract silica from sugarcane.

Sorghum bicolor (L.) moench, better known as sweet sorghum is widely grown in sub-Saharan Africa as a subsistence crop under various climatic conditions. A number of researchers have investigated potential applications of sweet sorghum and these include bio-fuels and bio-ethanol production(Almodares and Hadi, 2009; Srinivasa et al., 2009; Nadir et al., 2009). Sweet sorghum can also be a potential raw material for the production of syrups and jaggery (Nimbkar et al., 2006; Gnansounou et al., 2005). The by-product bagasse can be used for firing plants such as in ethanol production. Just like sugarcane and other plants in the grass family, *sorghum bicolor* (L.) *moench*is a silicon accumulator.Venkata et. al., 2012 found that sweet sorghum has a silica content of 5.48%. When produced in large quantities, sweet sorghum can therefore be a potential source of silica.

This research paper reports a new biobased approach of extracting silica from sweet sorghum bagasse ash. Sweet sorghum is a drought resistant crop that grows under various climatic conditions and growing it does not require special skills. Sweet sorghum bagasse ash is renewable since is derived from plant source.

METHODS

Materials and reagents

Sweet sorghum was harvested from Hippo Valley High School Garden near Chiredzi, in south eastern Zimbabwe. Chemical reagents used in this research were of analytical grade and these were sodium hydroxide (NaOH), hydrochloric acid (HCl) and ammonia (NH_3) obtained from Associated Chemical Enterprises (PTY) Ltd, Republic of South Africa.

Sampling and sample preparation

Mature sweet sorghum (SS) stalks were cut into smaller pieces and dried overnight at 110°C. The dried samples were ignited in a muffle furnace for three hours. The ash content of the whole plant was determined atignition temperatures of 200, 400, 600 and 800°C.

Preparation of silica gel

Silica gel extraction was adapted from methods reported in the literature with minor changes (Gnansounou et al., 2005; Venkata et al., 2012). Five grams of sweet sorghum bagasse ash was leached for 30 min in 100 ml of 1 M HCl solution to remove heavy metals. The leached ash was washed with copious amounts of distilled water. This was followed by refluxing the ash suspended in 100 ml of 3 M NaOH for three hours. The resultant solution which predominantly contained sodium silicate was filtered off to remove any suspended solids. The clear solution was titrated with 3 M HCl until an optimum pH 5 whereby then silica gel would have started precipitating. The gel was allowed to age for 10 h. Silica gel was separated by centrifugation and washed until the aqueous part reacted negative to a chloride test. The experiments were repeated for pH 7 and 9 and aging times of 10, 18 and 48.

Characterization of silica gels

The bulk density and porosity were determined using reported methods by Kalapathy et al. (2002) and Adam et al. (2011) and calculated as follows:

$$\rho = \frac{M}{V} \quad (1)$$

Where ρ is the bulk density, M and V the mass and volume of silica gel respectively.

The porosity of the gel was estimated using the following equation:

$$p = (1 - \frac{\rho_{Si}}{\rho_s}) \times 100 \quad (2)$$

Where ρ_{Si} is the bulk density of silica gel and ρ_s the specific density of amorphous silica assumed to be 2.0 g/cm^3 (Adam et al., 2011).

FTIR spectra of samples were recorded on a Thermo-Fisher Nicolet 600 FTIR instrument.The samples were prepared as KBr pellets. The composition of the ash and purity of extracted silica was determined using an XRF-spectrometer, Oxford X-Supreme 8000 with a tungsten X-ray tube and powder sample holder.The results are shown in Table 1. A BrukerD2 phase XRD instruments equipped with Cu-Kα, powder sample holder, powder sample holder and LynxEye detector in the range 2 theta 10° to 65° was used to record diffractograms of the silica samples and the results are shown in Figure 5. SEM images were recorded on a Jeol JSM 6510 instrument. Samples were prepared on a powder sample holder.

RESULTS AND DISCUSSION

The effect of temperature on ash content was investigated for the temperature range 200 to 800°C. At temperatures above 600°C, no significant changes in ash content were observed and 600°C was hence selected as the optimum ignition temperature. The effect of ignition temperature on ash content is shown on Figure 1. The average ash content for the whole plant was found to be 5.6%. A similar value 5.48% was reported by other researchers (Alayande et al., 2012).

Results of XRF analysis on the ash and silica prepared under optimum conditions are shown in Table 1. The ash

Table 1. Percentagecomposition of sweet sorghum bagasse ash and extracted silica.

Component	Ash	Extracted silica gel
SiO_2	40.16	96.36
K_2O	26.46	0.922
Na_2O	-	2.146
Al_2O_3	15.23	0.467
CaO	6.91	-
P_2O_5	5.92	-
SO_3	0.55	-
Fe_2O_3	0.41	-
Cl^-	4.25	-
MnO	0.03	-
TiO_2	0.03	-

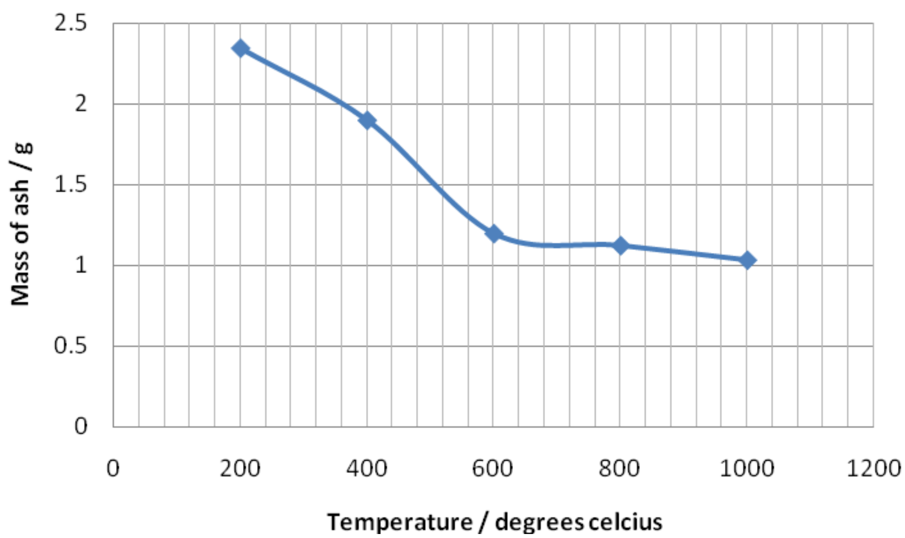

Figure 1. Effect of ignition temperature on ash content.

was found to have an average silica content of 40.16%. This shows that silica can commercially be extracted from sweet sorghum bagasse ash in processes where it is produced in large quantities as a waste product and adds value to the crop that is a potential raw material for bio-ethanol and syrups.

An XRF spectrum of sweet sorghum ash is shown in Figure 2. A small peak at 1.74 keV is typical of Si-k_α and hence confirms the presence of Si in the ash. The spectrum shows that the ash contains fewer heavy metals or very low concentrations of heavy metals that can be leached out prior to extraction.

Silica gel yield was dependent upon the pH and decreased from pH 5 to 8.5. At pH outside this range no meaningful precipitation was observed. The silica gel prepared at optimum pH 5 had a bulk density of 0.5626 g·$cm^3$and an estimated porosity of 71.87%.

Water adsorption properties of silica gel prepared at pH 5 but at different aging times were determined. Silica gel that was aged for 10 hours had better moisture adsorption properties than any gel aged over longer periods of time.The results of selected aging times are shown in Table 2. It can be assumed that the decrease in adsorption capacities is due to a decrease in surface area for gels aged over longer periods.

FTIR spectra of sweet sorghum bagasse ash and dried silica gel samples prepared at different pH are shown in Figure 3. The strong absorption bands at 1044 cm^{-1} was observed in all the samples and was expectedly much stronger in silica gel samples than the ash. The absorption band is associated with asymmetric vibrations of the siloxane, Si-O-Si. The spectra also show absorption bands at about 800 cm^{-1} which corresponds to a stretching vibration of Si-O-Si. The weak absorption

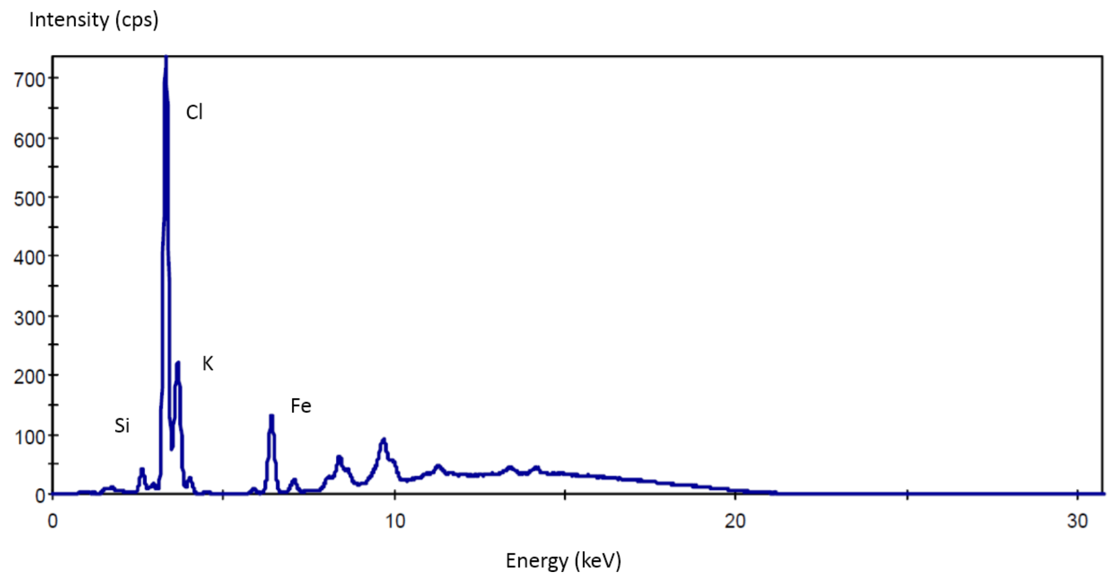

Figure 2. XRF spectrum of sweet sorghum ash.

Table 2. Effect of aging time on moisture absorption for silica gel prepared at pH 5.

Aging time (hours)	Maximum moisture absorption (%)
10	167.0
18	126.7
48	122.3

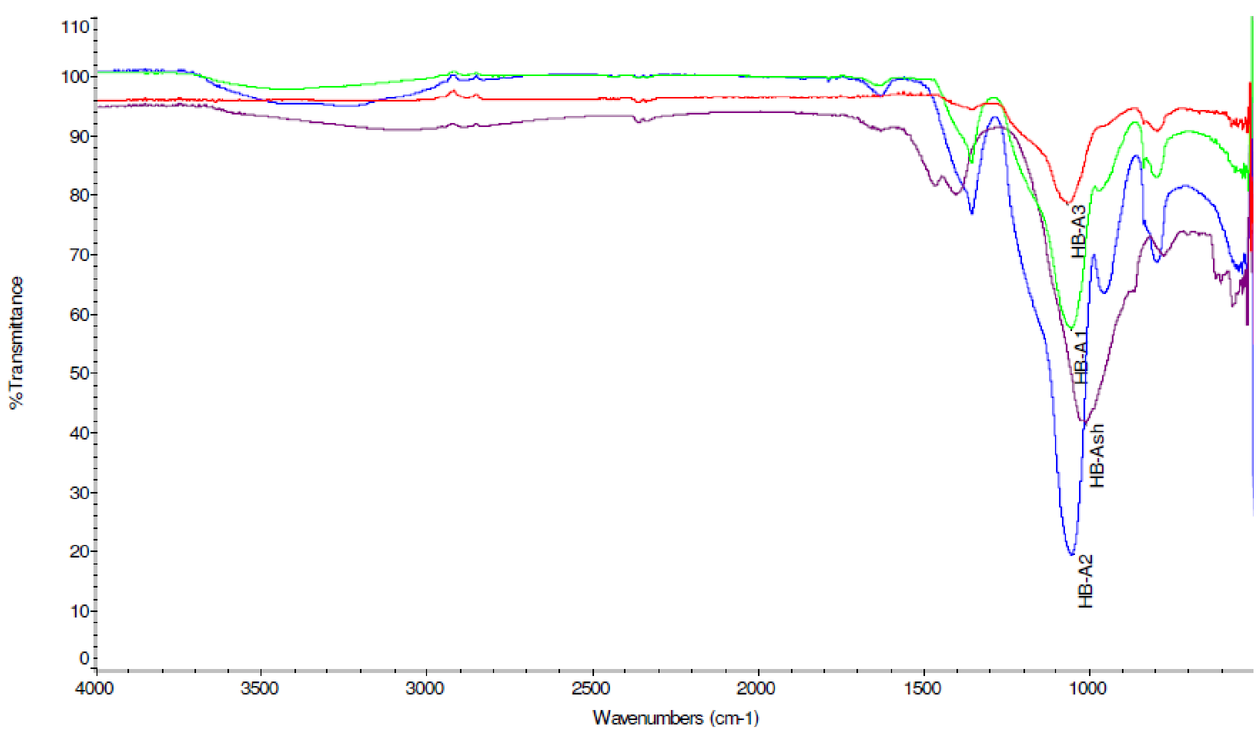

Figure 3. FTIR Spectra of sweet sorghum ash, silica gels prepared at pH 5 (HB-A1), pH 7 (HB-A2) and pH 9 (HB-A3).

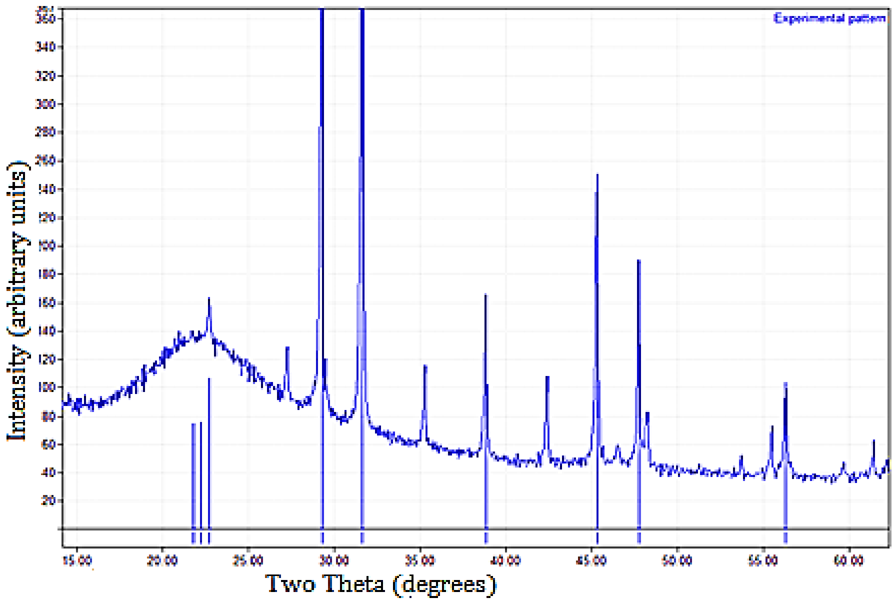

Figure 4. XRD diffractogram of sweet sorghum silica.

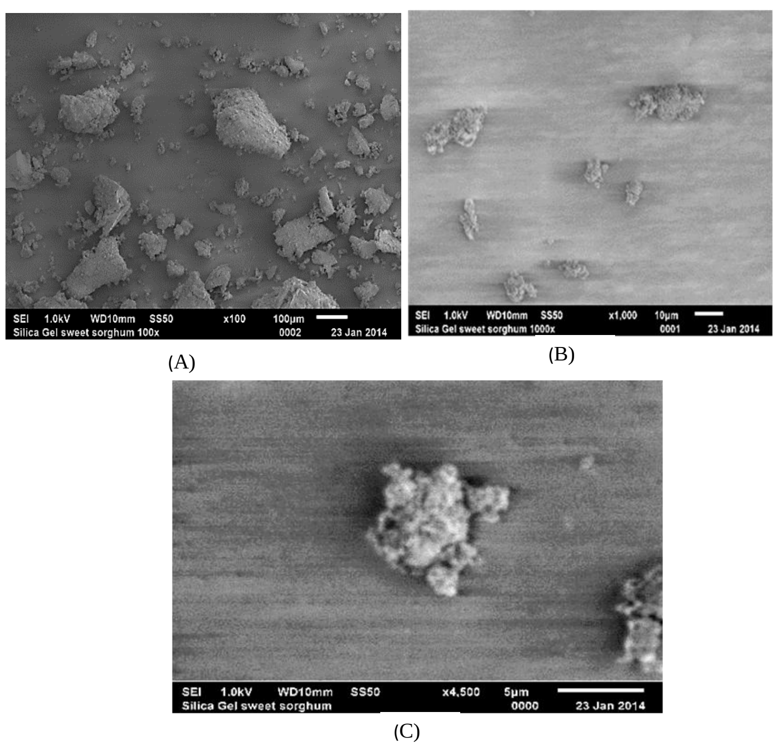

Figure 5. Micrographs of sweet sorghum silica at (A)100x, (B) 1000x and (C) 4500x magnification.

band at about 480 cm^{-1} is due to bending vibration of Si-O-Si. These Si-O-Si absorption bands are typical of silica gels and have also been observed by various researchers (Kalapathy et al., 2002; Essien et al., 2012; Nayak and Bera, 2009; Uzma et al., 2008).

An XRD diffractogram of sweet sorghum silica is shown in Figure 4. This shows that silica gel particles werehighly amorphous and the other phase was to sodium salts trapped in the gel. Figure. 5 shows micrographs of sweet sorghum silica at magnification of 100x, 1000x and

4500x. The silica gel consist of irregular particles of non-uniform size ranging from 5 μm to more than 100 μm.

Conclusion

Sweet sorghum is an important agricultural crop from which a number of products can be derived. The main advantage of this crop is its ability to grow under different climatic conditions. This research was able to demonstrate that sweet sorghum bagasse ash, a waste product of heat energy generation in the production of bio-ethanol can be used for extraction of silica gel for various applications.

Conflict of Interest

The authors have not declared any conflict of interest.

ACKNOWLEDGEMENT

The researchers would like to thank the Research Borad of the Bindura University of Science Education for providing funds under the Research Grant No 123/2013 and the International Office, Bonn-Rhein-Sieg University of Applied Sciences for having facilitated my research visit to the institution.

REFERENCES

Adam F, Chew TS, Andas J (2011). A simple template-free sol-gel synthesis of spherical nanosilica from agricultural biomass. J. Sol-Gel Sci. Technol. 59:580-583.

Alayande OS, Dare OE, Ayinde WB, Bamigbose J, Ayedun PA, Osinkolu GA (2012). Development of Ordered and Disordered Macroporous Silica from Bagasse Ash. Afr. J. Pure Appl. Chem. 6(1):10–14.

Almodares A, Hadi MR (2009). Production of Bio-ethanol from Sweet Sorghum: A review. Afr. J. Agric. Res. 4(9):772-780.

Devi PR, Dhanalakshmi KG (2012). Application of Mesoporous silica nanomaterial: An Overview. Int. J. Adv. Life Sci. 4:1-9.

Dorcheh AS, Abbasi MH (2008). Silica aerogel: Synthesis, properties and characterization. J. Mater. Process. Technol.199:10-26.

Essien ER, Olaniyi OA, Adams LA, Shaibu RO (2012). Sol-gel derived porous silica: Economic Synthesis and Characterization. J. Minerals Mater. Charact. Eng.11:976–981.

Gnansounou E, Dauriat A, Wyman CE (2005). Refining sweet sorghum to ethanol and sugar: Economic Trade-offs in the context of North China. Bioresource Technol. 96(6):985–1002.

Kalapathy U, Proctor A, Schultz J (2002). An improved method for the production of silica from rice hull ash. Bioresource Technol. 85:285-289.

Nadir N, Miel M, Karim MI, Yunus RM (2009). Comparison of sweet sorghum and cassava for ethanol production by using *Saccharomyces cerevisiae*. J. Appl. Sci. 9(17):3068-3073.

Nayak JP, Bera J (2009). Preparation of Silica aerogel by ambient pressure drying process using rice husk ash as raw material. Trns. Ind. Ceram. Soc. 68(2):1-4.

Nimbkar N, Akande NMJM, Rajavanshi AK (2006). Syrup production from sweet Sorghum. National Agricultural Research Institute (NARI).

Noushad M, Rahman IA, Huein A, Mohamad D, Ismail AR (2012). A Simple Method for obtaining Spherical Nanosilica from Rice Husk, Int. J. Adv. Sci. Eng. Infor. Technol. 2(2):28–30.

Prasad R, Pandey M (2012). Rice Husk Ash as a renewable source for the production of value added silica gel and its application: An Overview. Bull. Chem. Reaction Eng. Catalysis. 7(1):1-25.

Sing LP, Agrawal SK, Bhattacharyya SK, Sharma U, Ahalawat S (2011). Preparation of silica nanoparticles and its beneficiation role in cementitious materials. Nanomater. Nanotechnol.1:44-51.

Srinivasa RP, Rao SS, Seetharama N, Umakath AV, Reddy S, Reddy BVS, Gowda CLL (2009). Sweet Sorghum for biofuels and strategies for its improvement. Information Bulletin No. 77, Patancheru 502 324, Andra Pradeshi, India: International Crops Research Institute for the Semi-Arid Tropics, 80 pp, ISBN: 978-92-9066-518-2.

Thuadaij N, Nuntiya A (2008). Synthesis and characterization of Nanosilica from Rice Husk Ash Prepared by Precipitation Method, CMU. J. Nat. Sci. Nanotechnol. 7(1):59–65.

Tongjian Z, Yun X, Jiang W, Xin F (2009). Effect of application Rates of Assistant on the Properties of Nano SiO_2/NR Composites. J. Wuhan Univ. Technol. Mater. Scie. Ed. 24(3):387–392.

Ugheoke IB, Mamat O (2012). A critical assessment and new research directions of rice husk silica processing methods and properties. Maejo Int. J. Sci. Technol. 6(03):430–448.

Uzma KH, Bangi A, Venkateswara R, Parvathy RA (2008). A new method of sodium silicate-based hydrophobic silica aerogels via ambient-pressure drying. Sci. Technol. Adv. Mater. 9:035006(10 pp).

Venkata SCh, Ramana YR, Nagalashmi D, Jagadeeswara RS (2012). Evaluation of Sweet Sorghum (*Sorghum bicolour* (L.) moench) bagasse by Chemical, In sacco and *In vivo* Techniques in Graded Murrah Buffallo. Bulls. J. Veterinary Adv. 2(8):418–423.

Worathanakul P, Payubnop W, Muangpet A (2009). Characterization for post-treatment effect of Bagasse Ash for Silica extraction. World Acad. Sci. Eng. Technol. 32:360–262.