SYNTHESIS AND CHARACTERIZATION OF AMORPHOUS NANO-SILICA FROM BIOMASS ASH

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ABSTRACT

An attempt was made to synthesize nano-sized silica from cow dung ash, which has good potential source of silica. The silica from the cow dung was converted into sodium silicate and then the SiO_2 Precipitated in a controlled manner using surfactant. The nano sized amorphous silica was characterized using SEM, EDX and XRD data. The XRD powder pattern confirms the amorphous nature of SiO_2. An FTIR study was also carried out to evaluate the purity and surface functionalities present in nano silica. FTIR spectra recorded for the n-SiO_2 and Commercial SiO_2. Both the samples show almost similar IR spectra confirms the purity of n-SiO_2.

Keywords: Amorphous, Cow Dung, EDX, Nano-Silica, Sodium Silicate , XRD.

I. INTRODUCTION

Biologically diverse countries like India have lot of potential by-products from agro and animal based wastes, many of which are not explored for their further utility. Exploration of useful materials will make any process economically beneficial and also minimizes the environmental pollution created by waste products. Cattle dung is one of the most abundant waste products generated in large quantities from the developing countries and also developed countries. Cow dung has been used as manure for agricultural field for better crop yields, as a renewable energy resource to produce energy in biogas plant etc., In rural areas of tropical countries the cow dung cake was as such used as a fuel produces ash which creates some environmental problems. Since all the nations are looking for waste reduction or wealth from waste, research was carried out for making waste into wealth in sustainable manner. At present, nano scale silica materials are prepared using several methods including vapour phase reduction, sol-gel and thermal decomposition technique and the powder silica production is highly energy intensive process. Extraction of nano silica from cattle dung can be beneficial in terms of economy and environmental aspects.The cattle dung ash contains nearly 60 % of Silica along with other materials in the form of oxides [1]. The composition of cow dung ash also varies from animal to animal, food habits and geographical areas. Silica of high purity, small size was synthesized from cow dung ash by sol gel method [1] and can be used as an adsorbent or catalytic support in the chemical synthesis [2]. The amorphous nano silica powder was extracted from rice husk ash by precipitation method has been reported [3] and also used as green corrosion inhibitor [4]. Many studies have reported the applications of silica fume as a constituent in cement [2, 5-11] which was used as a filler for polypropylene composite that had good mechanical properties [2 ,12]. Similarly silica in micro and nano size forms finds

applications in the field of semiconductor, industries, synthesis of highly active silica gel, pharmaceutical products, precursor in ceramic industries etc. The objective of the present work is to prepare and characterize amorphous nano silica using ash that is generated from cow dung

II. MATERIALS AND METHODS

2.1 Raw Material

The cow dung cake was dried in sunlight. And the ash was produced by burning the dried cow dung cake in atmospheric oxygen. The obtained cow dung ash was used as raw material for the extraction and synthesis of n- SiO_2. All reagents used for this synthesis are commercially available.

2.2 Synthesis Of Amorphous Silica From Cow Dung Ash

10 g of cow dung ash sample was taken in 250 ml Erlenmeyer flask. Exactly 80 ml of 2.5 N aqueous solution of sodium hydroxide solution was added and stirred continuously for about 4 hours at 95° - 100° C. The formed sodium silicate was filtered and the residue was washed with warm double distilled water. The filtrate of sodium silicate at 60° - 70° C was acidified with 5N H_2SO_4 until the silica gel was precipitated. The white silica gel precipitate obtained was washed repeatedly with double distilled water until the filtrate is completely free from alkali. Then the silica was dried at a temperature of 110°C for 24 h in a hot air oven.

2.3 PREPARATION OF N-SIO_2

Pure silica extracted from cow dung ash (CDA) was refluxed with 6N HCl for about 6 hours. Then the residual mass was washed repeatedly in a round bottom flask by using double distilled water until the filtrate was completely free from acid. The washed sample was treated with 2.5 N aqueous sodium hydroxide solution and 1ml of surfactant. And the whole mixture was stirred continuously on magnetic stirrer for about 10 hours at 95° - 100° C. The formed sodium silicate was acidified by Con. H_2SO_4 yield the precipitated silica. The precipitated silica was washed repeatedly with double distilled water until the filtrate was completely free from alkali. The following equation (1) and (2) are the chemical reactions involved in the synthesis of nano-silica from CDA.

$$\underset{\text{CDA}}{SiO_2} + 2NaOH \longrightarrow \underset{\text{Sodium silicate}}{Na_2SiO_3} + H_2O \longrightarrow \quad (1)$$

$$Na_2SiO_3 + H_2SO_4 \longrightarrow SiO_2 + Na_2SO_4 + H_2O \longrightarrow \quad (2)$$

Sodium silicate nano-Silica

III. RESULTS AND DISCUSSION

3.1 Characterization

The strong and broad XRD pattern from 18° to 31° (2θ) of nano silica shows that the prepared nano SiO_2 is amorphous [13] which is advantageous for catalytic and other applications. The XRD pattern of cow dung ash

is shown in Fig.1. The sharp and intensive peaks substantiate that all the constituents (predominantly the oxides) were present in the form of crystalline. It is also clear from the XRD pattern of ash and n-SiO_2 that the sequential sol-gel process completely eliminates all other constituents and only amorphous n-SiO_2 is synthesized. In order to confirm the purity and nature of surface functionalities present on the surface of n-SiO_2, the FTIR spectra is recorded for the n-SiO_2 and Commercial SiO_2 and is shown in the Fig 2 and Fig. 3. Both the samples show almost similar IR spectra that confirms the purity of n-SiO_2. A small peak around 1867 cm^{-1} for the commercial silica (C-SiO_2) is due to the presence of –C=O stretching which is caused by the impurities or adsorbed atmospheric CO_2. The same peak is totally absent in the case of n-SiO_2. A broad band at 3200 – 3700 cm^{-1} is observed for both C-SiO_2 and n-SiO_2 which is due to the silanol –OH groups and adsorbed water. Absence of peaks around 2350 to 2400 cm-1 indicates the absence of Si-C stretching which confirms that no carbon impurities mixed to SiO_2. A sharp peak at 1635 cm^{-1} assigned for –C-O stretching is caused due to the atmospheric CO_2 adsorbed by the porous silica and C-SiO_2. A strong band at 1052 – 1228 cm^{-1} is caused by the presence of Silaoxane (Si-O-Si) stretching. A slight change in the structure of a strong band at 1052 – 1228 cm^{-1} suggests a change in the local bonding structures of Si and O atoms at smaller particle size [14, 15]. A sharp and intense peak at 799 – 801 cm^{-1} is due to the presence of Si-O bending vibrations. A peak corresponding to Si-OH str is observed at 957 – 972 cm^{-1} for both the samples. The peaks at 442 – 485 cm^{-1} is caused by the Si-O out of plane bending vibrations. The FTIR spectrum shows no significant changes in the peak position for commercial silica and nano-silica. The percentage composition of cow dung ash and n-SiO_2 are analyzed using EDX studies. The percentage composition of various elements present in the ash and n-SiO_2 are given in **Table 1**. The EDX plot of the ash sample (Fig. 4 and Fig. 5) shows many peaks for various elements, whereas the EDX plot of n-SiO_2 gives only two peaks for Silicon and Oxygen respectively as per the ratio given in **Table 1**.

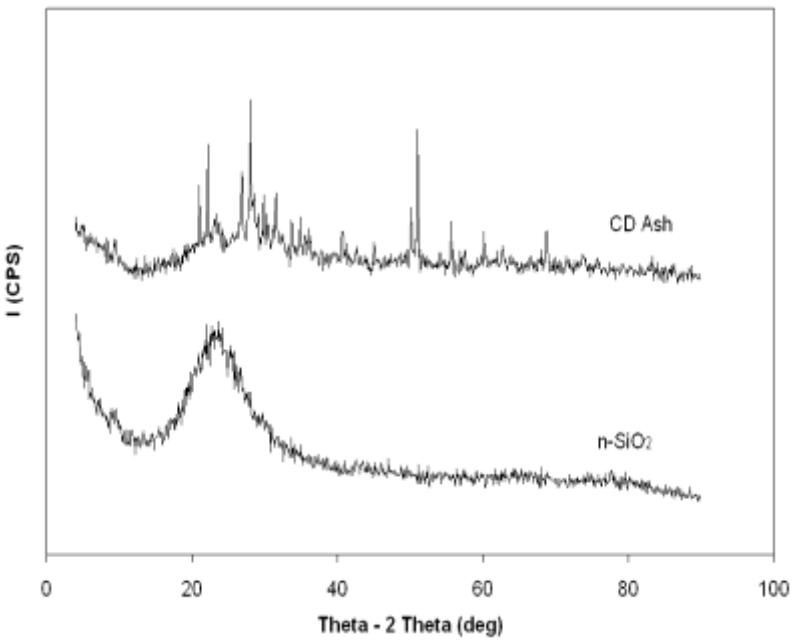

Figure 1. XRD Plot Of CD Ash And N-SIO_2

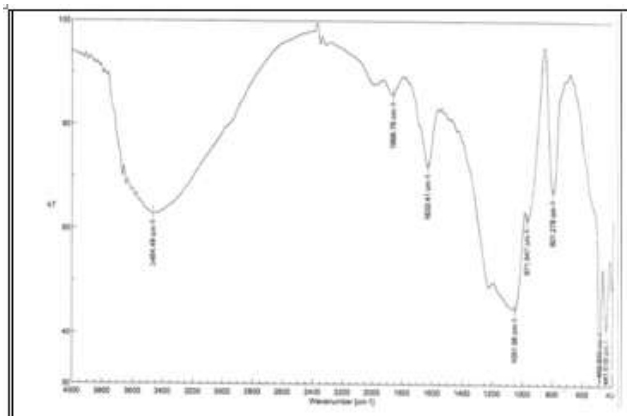

Figure 2. FTIR image of commercial SiO_2

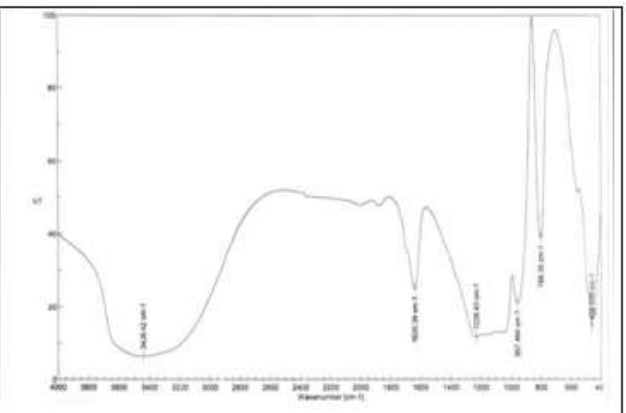

Figure 3. FTIR image of n-SiO_2

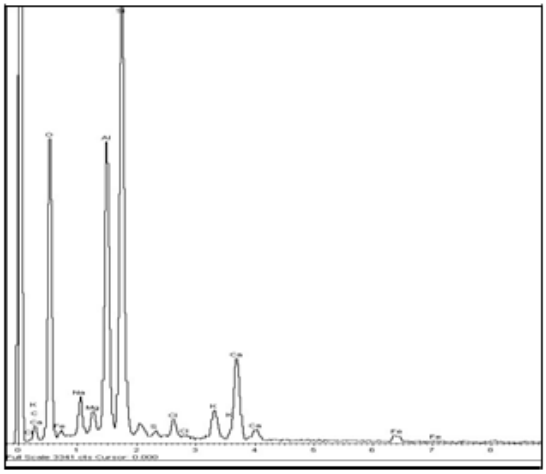

Figure 4. EDX Plot of cow dung ash

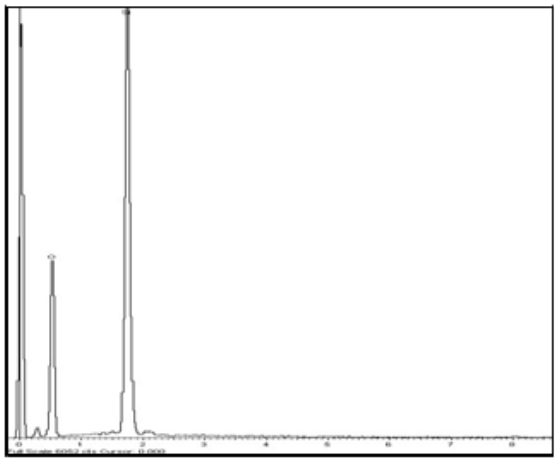

Figure 5. EDX plot of n-SiO2

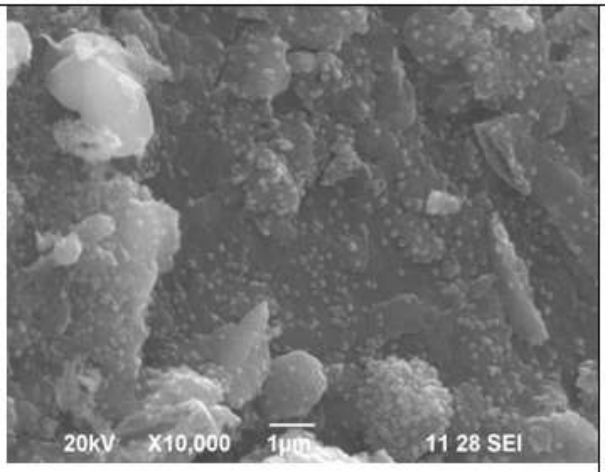

Figure 6. SEM image of cow dung ash

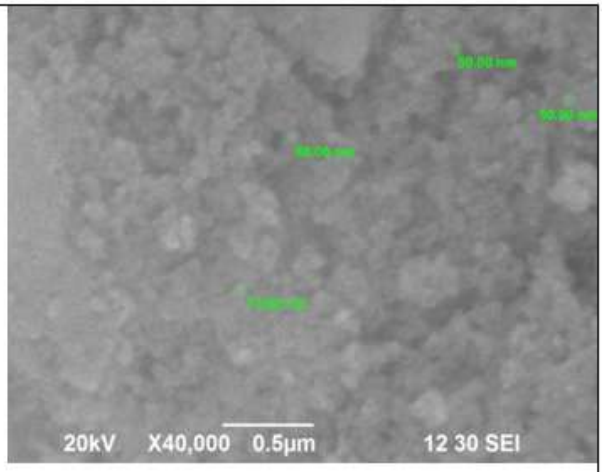

Figure 7. SEM image of n-SiO_2

Table 1 Elemental Composition of Ash and n-SiO_2

Cow dung ash			n-SiO_2	
Element	Approximate Concentration	Weight %	Approximate Concentration	Weight %
C	4.48	7.65	–	–
O	76.63	50.07	83.36	71.54
Na	4.12	2.47	–	–
Mg	1.56	1.04	–	–
Al	18.72	11.02	–	–
Si	29.76	18.50	54.16	28.46
S	0.54	0.34	–	–
Cl	1.40	0.96	–	–
K	3.44	1.71	–	–
Ca	9.82	5.12	–	–
Fe	1.86	1.13	–	–
Total		100.00		100.00

The SEM image of cow dung ash and n-silica are shown in the fig. 6 and 7. The crystalline form of metal oxide constituents present in cow dung ash is confirmed by sharp XRD peaks. The nano-size silica synthesized from cow dung ash is in agglomerated form and its amorphous nature is confirmed by a broad peak of XRD pattern. The particle size of the prepared n-SiO_2 was calculated by using Scherrer's formula ($D=K\lambda/(\beta \cos \theta)$) for 2θ values 23.3750 and corresponding FWHM value of 6.75000 is 1.25nm and hence the prepared silica is in nanosize.

IV. CONCLUSIONS

Amorphous form of n-SiO_2 is successfully prepared from cow dung with good and comparable yield. The prepared n-SiO_2 exhibits similar peaks like commercial silica, which confirms the purity of synthesized n-SiO_2. The FTIR analysis shows that the prepared n-SiO_2 is highly pure. The percentage composition of cow dung ash and n- SiO_2 are analyzed using EDX studies. The value of Si is 28.46 % (by weight) and the value of Oxygen is 71.54 % (by weight) and hence the synthesized n-silica is 100 % pure and is confirmed by EDX spectrum. The strong and broad XRD pattern from 18° to 31° (2□) of nano-silica shows that the prepared nano-SiO_2 is amorphous in nature. The research substantiates that cow dung ash is a promising and economically beneficial source for the manufacture of n-SiO_2. For further research, the obtained n-silica can be used as an adsorbent or catalytic support for environmental applications.

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