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Recovery of silica from various low cost precursors for the synthesis of silica gel

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ABSTRACT

The objective of this study was to (1) use sugar cane ash, Bagasse ash and silica fumes as raw materials for silica recovery, due to its low price and availability and (2) compare the recovery from the raw materials and produce silica gel.The methods used to recover silica were alkali-solubility based method and sonication. The ash from the low cost precursors were treated with NaOH solution and sonicated for a period of time. The silica recovered as sodium silicate, by filtration, from each raw material is compared and analyzed. In general, when sodium silicate is treated with acid, different amorphous and mesoporous silica are produced. The silica gel was synthesized using the acidification of sodium silicate solution.Silica recovery was promising based on the comparison of the silica content before and after the process, with silica fumes having the highest recovery of silica. Thus the sodium silicate from the raw material silica fumes was used in the preparation of silica gel using acidification method and then analyzed. The modified recovery process proves to be a promising method for the recovery of silica from natural sources.

Key words: Silica recovery, sugarcane ash, silica gel preparation, bagasse ash, silica fumes.

INTRODUCTION

Sustainable development depends on the usage of renewable form of energies. Recovery of silica from sugarcane leaf ash [3,4], bagasse ash [1,5] and silica fumes, in the form of sodium silicate has many industrial applications. Silica, also known as silicon dioxide, is a chemical compound with a chemical formula of SiO_2. It has got industrial importance over a long period of time. The most common form of silica occurring in nature is quartz. Silica is one of the most complex material. It exists both naturally as well as being synthesized. Silica gel and aerogel [1,2] are some of the examples. One of the major applications of silica is glasses and bottles. It is also used in the optical fibers used for telecommunication [6] and sensors. In food industry, its used as additive and in the pharmaceutical industry, It aids in the flow of tablets. Silica is generally non-toxic when consumed orally. It has a lethal dose of 5g/kg. On the contrary, inhalation of silica dust causes bronchitis, cancer, etc., Silica has been recovered from many natural sources like Rice hull ash [7], sugarcane leaf ash [3] etc.,

Sodium silicate, readily soluble in water, is obtained when silica is treated with excess NaOH [3]. It is also referred to as Water glass and has a general formula of $Na_2(SiO_2)_nO$ pure form is white or colorless. It can be in both hydrated and anhydrous forms. In acidic solutions, sodium silicate reacts with the hydrogen ions and forms silicic acid, which upon heating forms silica gel. The major uses of sodium silicate is water treatment, adhesives, food preservation etc., Agricultural waste such as leaves, are rich in essential compounds. Sugarcane, one of the major crop, has a high amount of silica in the leaves as well as a sugarcane industry waste called as bagasse. The silica content [1] in these resources is an essential raw material for silica gel production. Generally, the bagasses are used as a fuel in boilers. Burning of bagasse produces ash, which is rich in silica content. Sugar cane leaves are another important agricultural waste that is rich in silica content and economically viable source. The usage of these wastes provides a better solution for waste disposal from sugarcane industries. The smelting process, that is part of the silicon and ferrosilicon industry results in a by-product called Silica Fumes. Highly pure quartz when reduced to silicon at a temperature range of 2,000 °C produces vapors of SiO_2. These vapors oxidize and condense when exposed to lower temperature zone and results in tiny particles made up of non-crystalline silica.

Silica fume is also referred to as condensed silica fume [8], micro silica or silica dust. The size of silica fume particle is extremely small. Silica fume has a very high content of amorphous silicon dioxide and consists of very fine spherical particles. The major component of Silica fume is SiO2, that forms around 90%. Minor quantities of iron, magnesium, and alkali oxides are also found.

MATERIALS AND METHODS

Silica was extracted from three different sources, namely Sugarcane leaf ash, Bagasse ash and Silica fumes. The chemical composition of each of the raw materials is shown in the table 1. The sugarcane leaf ash was collected from a nearby sugar cane field farm. The dried leaves were collected and used for the present study. Bagasse ash was collected from Ariyur Sugar industries, where bagasse are a waste product of sugar industries. The silica fumes were also collected from a nearby industry. . Chemicals such as Sodium Hydroxide, methanol, hydrochloric acid were purchased from Merck Chemicals Pvt Ltd.

Silica Recovery

The silica recovery protocol is shown in the Figure 1. The Sugarcane leaf was cut into pieces. The sugarcane leaves and bagasse were burnt in a furnace. The ashes were obtained. All of the sources were calcinated at a temperature of 300° C. The silica was extracted from the sources [4] by alkali treatment. The alkali solution was added in the ratio of 1.8:1 in terms of weight and water in the ratio of 4:1 are added and the solution is kept in a hot water bath for about 3 hours. The solution is then kept for sonication [3]. After the process, the solution is filtered using a filter paper to remove the ash residue. The filtrate consists of sodium silicate which was subsequently used for silica gel preparation.

Silica gel preparation

Silica gel can be produced from [1,9] the sodium silicate solution.The sodium silicate solution obtained was made to undergo acidification [1,10] to produce silica gel. Silica gel has a variety of applications such as in cosmetics, pharmaceutical products, paints and coatings [11], can be used as an adsorbent etc., and has been produced from various natural resources. The obtained sodium silicate solutions were titrated against 1N HCl and the pH were monitored. The pH was maintained between 7 and 10 during silica gel formation process. After the titration, the solution was kept for aging up to 24 hours to form a gel like substance. The gel was further washed with distilled water and dried silica gel was obtained.

Characterization

The content and composition of the ashes and silica fume, before and after the recovery process was analyzed using X-ray Fluorescent (XRF) method. Fourier Transform InfraRed Spectroscopy(FTIR) data was utilized to analyze the chemical bonds and molecular structure of the gel. The spectra were collected in the range of 4000-400 cm^{-1}

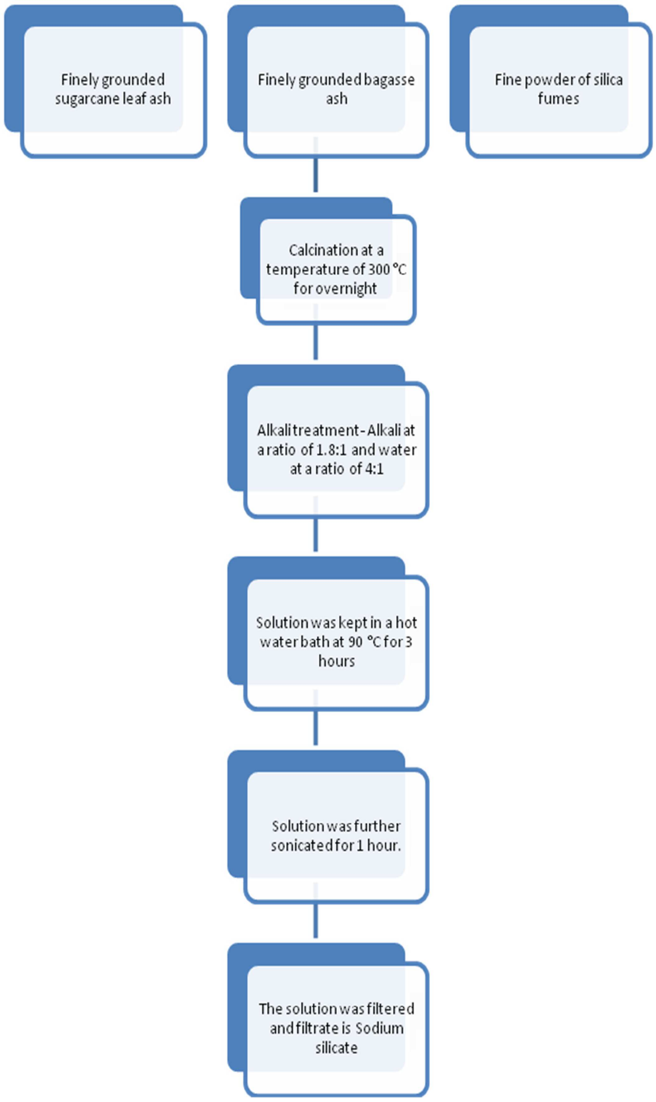

Figure i: The Silica recovery process-protocol

RESULTS AND DISCUSSION

Prior to the use of raw materials for silica recovery, the composition of each of the raw materials was obtained using XRF analysis and presented in table 1. Each of the raw materials has major component as silica. Each of the raw materials [3] was subjected to improved method of recovery of silica. In the recovery process, the first step of the reaction was between sodium hydroxide and silicon dioxide, forming sodium silicate also known as water glass. The second stage involved the acidification of the sodium silicate solution, recovered from each raw material [1], producing silica gels. U. Kalapathy et al [4], 2000 successfully recovered silica from rice hull ash. The extraction was done using alkali and it was followed by acid precipitation to obtain pure silica gels. Subsequently, the gels were heated at 80ºC to obtain xerogels. Energy dispersive X-ray (EDX) and inductively-coupled plasma (ICP) emission spectrometers were used to determine the contents of the xerogels. Xerogels, so obtained was found to contain 93% silica and 2.6% moisture. Some of the impurities were Na, K, and Ca. The yield was found to be 91%. Acid washing was carried out before extraction in order to reduce the concentration of Ca.

Shaikh et al, 2013 used Wheat husk ash for the preparation mesoporous silica. The MCM-41 samples synthesized using commercially available silica and wheat husk ash silica at room temperature. The characteristics of MCM-41 were compared between (i) MCM-41 synthesized from the commercially available silica source; (ii). MCM-41 synthesized using wheat husk ash as silica source, and (iii). the MCM-41 reported earlier. The recovery from rice husk ash was reported to be over 90% by alkali digestion with aqueous sodium hydroxide and precipitation of silica by treatment of acid with the sodium silicate solution so obtained. Shamle et al [6] , 2013 isolated amorphous silica from acha husk ash (AHA), wheat husk ash (WHA) and rice husk ash (RHA). The various different husk ashes were obtained by a process called calcination of the husks in a muffle furnace at 500°C for five hours. The sodium silicate solution was obtained from the respective ashes using NaOH solution. Acid treatment using HCl was done to the sodium silicate to lower the pH to 7.0. The percentage of silica in the AHA, WHA and RHA were (57.50)%, (59.68)% and (68.50)% respectively. Correspondingly, the silica content of the respective xerogels was (92.03)%, (88.43)% and (88.55)%. These results lead to a conclusion that AHA is an alternative source of silica, having a very high yield and minimum mineral contaminants.

In the present work the X-ray Fluorescence analysis technique was utilized to analyze the ash composition before and after the improved recovery process. The composition of ash before the process showed a major amount of silica in silica fumes and sugar cane ash, and a moderate amount of silica in Bagasse ash respectively. The XRF analysis of the ash after the improved recovery process showed a varied recovery in each of the raw materials. Up to 67% recovery was obtained in silica fumes and 56% recovery was obtained in sugarcane leaf ash and 38% recovery in Bagasse ash. The silica gels prepared from the filtrates of each of the raw material had very less impurities and high silica content. The improved process proved efficient in the recovery of silica in each of the raw materials.

The sodium silicate solution obtained after the recovery process from each of the raw material was used and was subsequently used in the preparation of silica gel. Silica gel has various commercial uses, such as absorbent, raw material for ceramics, in cement industries etc., The silica gel obtained from the above procedure was analyzed using the Fourier Transform InfraRed Spectroscopy. The result is shown in the Fig 2. The FTIR spectra of the silica gel showed various bands corresponding to various structural units present in the molecule. The intense and broad band at around 1082 cm^{-1} attributes to the asymmetric vibration mode of Si-O-Si. A broad band at 3432 cm^{-1} belongs to the O-H stretching bands of H_2O molecule.

Table i: Table shows the Chemical Composition of the raw materials

FORMULA	Sugarcane ash (wt%)	Bagasse ash (wt%)	Silica fumes (Wt%)
SiO_2	85.9	52.3	93.27
CaO	4.32	7.64	0.40
MgO	3.67	----	0.25
SO_3	2.02	----	0.17
Al_2O_3	0.89	----	0.44
Cl	0.52	----	----
Fe_2O_3	0.35	19.70	1.52
Na_2O	0.92	----	0.32
MnO	1.41	0.68	----
K_2O	----	18.22	0.59
TiO_2	----	0.28	----
V_2O_5	----	0.45	----
CuO	----	0.40	----
ZnO	----	0.33	----
LOI	----	----	2.68

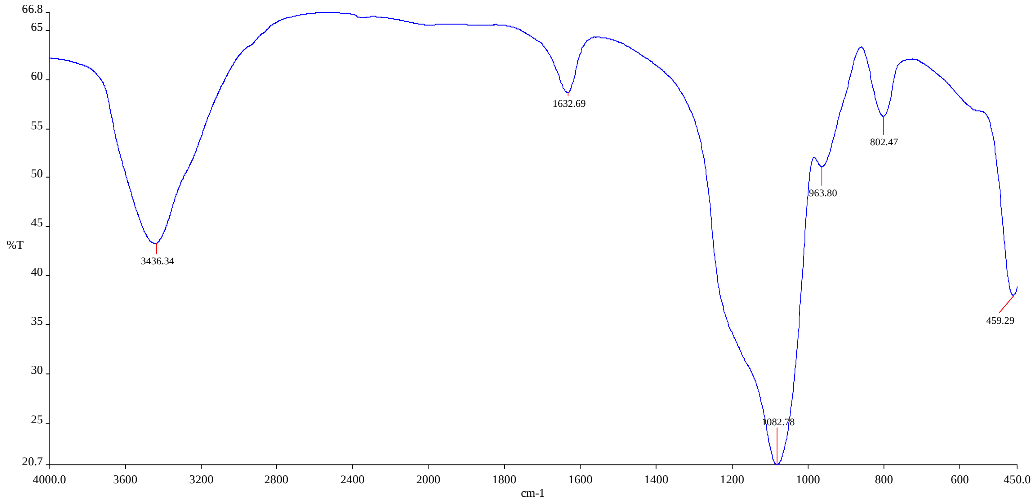

Figure II: The Fourier Transform Infrared Spectroscopy image of the silica gel

The SEM analysis image of the silica gel prepared from the silica fume source is shown in the Fig 3. Scanning Electron Microscopy has a very high resolution and it is used to study the structure of the sample molecule. The silica gel was produced from the sodium silicate solution by acidification at a pH of 8 and the structure was studied using SEM.

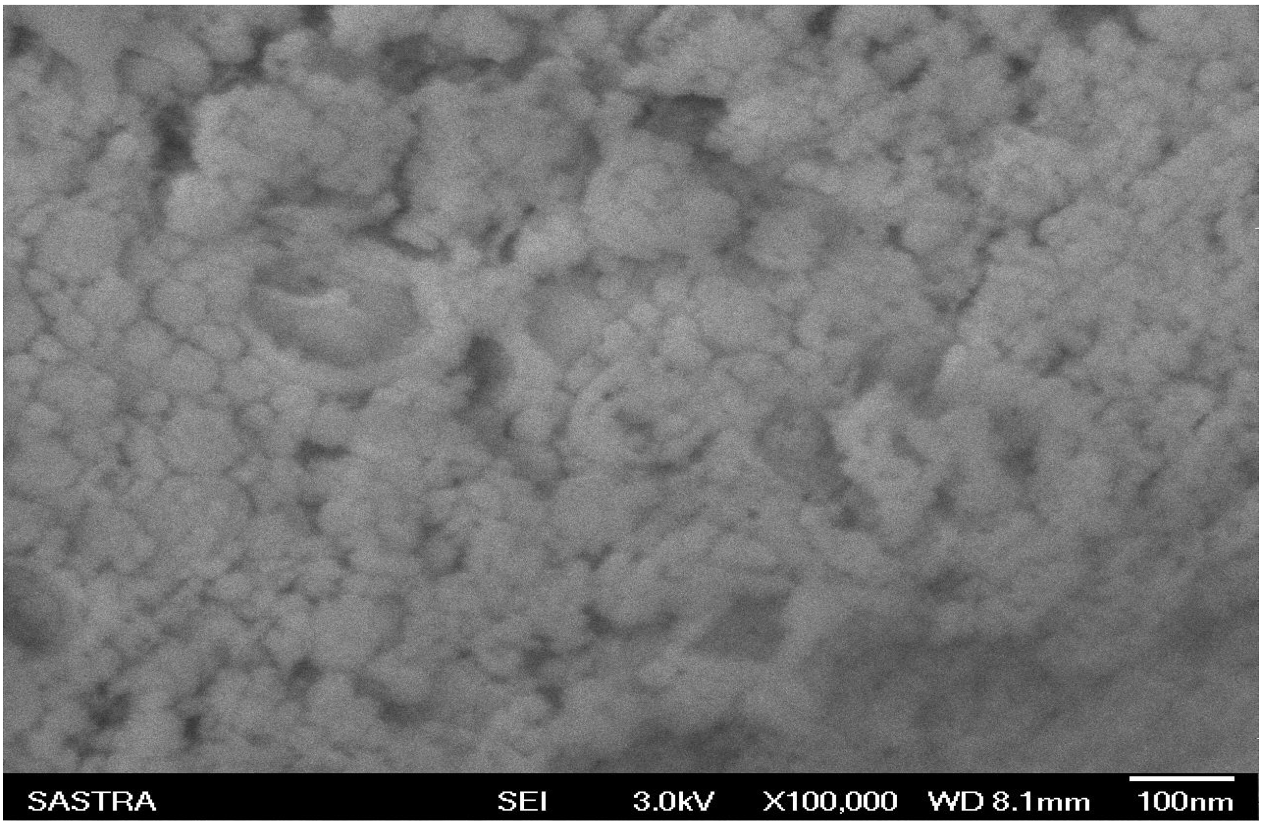

Figure iii: The SEM analysis image of the silica gel

CONCLUSION

Recovery of silica was carried out using three different sources, namely sugarcane leaf ash, Bagasse ash and silica fumes. The recovery was done using an improved method comprising of alkali treatment and sonication. The X-ray fluorescence was done for all the raw materials before and after the recovery process and it was understood that silica fumes showed maximum recovery of 67%, while sugarcane ash showed recovery of 56% and Bagasee ash showed 38%. The sodium silicate obtained from the raw material silica fumes was used further for the synthesis of silica gel, by acidification process. The silica gel was characterized and visualized using SEM analysis and also using FTIR spectroscopy.

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