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Silica Nanoparticles from Sugarcane Bagasse Fly Ash: Converting Waste to Wealth

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Abstract: ***The Indian cane sugar industry generates significant quantities of bagasse fly ash at boilers as a waste. This fly ash while is of no commercial value at present, also creates environmental issues. Bagasse fly ash is Si-rich and a potential resource to produce silica-based high-value products, such as silica nanoparticles (SiNPs). SiNPs have various applications, such as in crop improvement, diagnostic and therapeutic bio-analysis, membranes for fuel cells, Li-ion batteries, adsorbents, catalysts and so on. This study investigated a method to produce SiNPs from sugarcane bagasse fly ash (SBFA), which also addressed environmental issues. SiNPs have various applications. The structural properties of SiNPs were analysed using different standard techniques, viz. Scanning Electron Spectroscopy, X-Ray Diffraction Analysis and Fourier Transform Infrared Spectroscopy. This techno-economic process developed, as applied to waste material may also provide many benefits to the local agro-industry and increased attention towards silica nanoparticles will help to realize the true potential of silica nanoparticles in agriculture and in other fields.***

Keywords: Sugarcane bagasse fly ash, nanotechnology, silica nanoparticles

1. Introduction

India is expected to produce around 34.0 million metric ton (MMT) of sugar during 2022-2023 after considering diversion to the extent of 4.5 metric million ton for boosting ethanol production. With sugarcane crushing of the order of 350-370 MMT, the bagasse production thus is likely to be about 105-110 MMT per annum.

Innovative and creative ways to reduce waste and the contamination of the environment, minimizing environmental impacts, have been targeted in 21^{st} century. The largest agro-industrial by-products of the world are rice husk and straw, corn cobs, wheat straw and sugarcane bagasse. Among these by-products, sugarcane bagasse, rice husk and straw can be highlighted because of their silicon content, being important renewable sources.

Bagasse at present is majorly used as fuel and steam through boiler and only a limited amount is utilised for other purposes i.e. paper, tableware production etc. For making Indian sugar industry self-sustainable, there is stress on converting sugar factories into bio-refineries producing bio-chemicals and bio-energy. There is also a growing debate on utilizing by-products for producing value-added products so as to enhance revenues and exploit the potential of entire sugarcane value chain. Adding value to agro-industrial solid wastes, produced on a large scale, is a challenge for sustainable and green chemistry.

Sugarcane bagasse fly ash (SBFA) is obtained during the combustion process of the sugarcane bagasse and it is used mainly as landfill. Bagasse fly ash accounts for around 1.5 to 2 wt. % of bagasse and collected. If a 5000 TCD sugar factory crushes about 800, 000 ton of sugarcane in the season, and diverts major parts of bagasse as boiler fuel, then the total fly ash produced is expected to be about 5000 metric ton annually.

Nanomaterials in the dimension area of 1nm-100nm have applications in various fields such as electrochemistry, catalysis, sensors, biomedicines, pharmaceutics, health care, cosmetics, food technology, textile industry, mechanics, optics, electronics, space industry, energy science and optical devices etc. Silica nanoparticles (SiNPs) are a type of nanomaterials with significant industry interests. As mentioned earlier, SiNPs have various applications, such as in crop improvement, diagnostic and therapeutic bio-analysis, membranes for fuel cells, Li-ion batteries, adsorbents, catalysts and so on.

2. Literature Survey

SiNPs have a wide range of applications in crop improvement, paints, biopolymers, membranes for fuel cells, Li-batteries, catalysts, stationary phases for chromatographic columns, diagnostic and therapeutic bio-analysis and adsorbents, among other applications (Norsuraya, S. etal., 2016). For instance, SiNPs showed great adsorption capacity with possibility of reuse up to five cycles, adsorbing more than 90% which indicated it was possible to obtain a good green adsorbent, from a renewable source, at low cost (Rovani, S. et al., 2018). The demand for highly sensitive non-isotopic bio-analysis systems for biotechnology applications, such as those needed in clinical diagnostics, food quality control, and drug delivery, has driven research in the use of nanomaterials for biomedical and biotechnological applications (Lin Wang et al., 2008). Recently SiNPs were applied to crop plants as nano-fertilizers to substantially improve plant growth under multiple stress conditions (Bhat, J. A., et al., 2021).

Silica nanoparticles are produced from Si-rich resource and the literature mentions many bio resource precursors for synthesis of SiNPs such as sugarcane bagasse, rice husk, bamboo, maize stalk, corn cob husk etc. SBFA is an abundant source of silica. It is however currently used for low-value applications such as in the concretes, manufacturing of glass and ceramics. SBFA is also used as fertilizer for crop improvement because it also contains minerals such as potassium, which are absorbed by the sugarcane plant from soil and from the chemical fertilizers applied to the crop. The chemical composition of bagasse fly ash depends on the sugarcane growth conditioned, bagasse burning condition i.e. duration, temperature and air intake. Table 1shows the typical composition of SBFA as reported in literature. Bagasse fly ash is an agro-industrial waste material composed of silica dioxide and widely used in the concretes, manufacturing of glass and ceramics, also used as fertilizer for crop improvement. Bagasse ash contains many inorganic salts are the nutrients absorbed by the sugarcane plant from soil and from the chemical fertilizers applied to the crop. Bagasse ash is a rich content of silica dioxide, and other minor metal oxides along with unburned carbon and moisture. The chemical composition of bagasse ash depends on the its burning condition i.e. duration, temperature and air intake. The typical composition of sugarcane bagasse fly ash as reported in literature is as follows:

Table 1: Composition of Sugarcane bagasse fly ash

Composition	Value Percentage
SiO_2	65-70
Al_2O_3	1.7-2.0
Fe_2O_3	7.5-8.0
CaO	3.0-3.5
MgO	0.1-0.3
K_2O	8.0-8.4
Na_2O	3.0-3.4
P_2O_5	2.3-3.2
MnO	0.1-0.2

(Rovani, S. et al., 2018) reported production of silica nanoparticles using sugarcane bagasse fly ash and utilization of product as adsorbent. In the present study the authors worked on developing a technique for producing silica nanoparticles from sugarcane bagasse fly ash. These silica nanoparticles, as discussed earlier have a wide range of applications in crop improvement, paints, biopolymers, membranes for fuel cells, Li-batteries, catalysts, stationary phases for chromatographic columns, diagnostic and therapeutic bio-analysis and adsorbents, among other applications (Norsuraya, S. et al., 2016). Authors have discussed here methodology adopted for obtaining the product techniques considered for characterization of the product, expected yield and quality of silica nanoparticles from sugarcane bagasse fly ash (SBFA).

3. Materials and Methods

Materials

Sugarcane bagasse fly ash sample was collected from various sugar factories of UP, India for the season of 2022-2023. All reagents used were of analytical grade, and their solutions were made up in double distilled deionised water. Sodium hydroxide (NaOH), Chloroacetic acid ($C_2H_3lO_2Cl$), Sulfuric acid (H_2SO_4), n-pentanol were of laboratory grade and Hexadecytrimethyl-ammonium bromide (HCTAB)used as surfactant was also of laboratory grade from Merck (Sigma-Life Science).

Analysis

For determining the efficiency and to characterize of silica nanoparticles, following analysis were conducted.

Microscopic elemental analysis-The Scanning Electron Microscopy (SEM) analysis of prepared silica nanoparticles was done using TM 3000 model tabletop SEM from Hitachi High Technologies, Singapore which is integrated with SwiftED 3000 energy dispersive X-ray spectrometer from Oxford Instruments, England.

Morphology-TheX-Ray Diffraction analysis was done by Atomic Force Microscope (AFM) Model: INNOVAA2-SYS, Bruker, Singapore.

Chemical composition of prepared silica nanoparticles- Fourier Transform Infrared (FTIR) Spectroscopy was observed by Alpha II compact FT-IR spectrometer.

Surface area, total pore volume and average pore size- The Brunauer-Emmett-Teller (BET) analysis of prepared SiNPs was determined using Quanta-chrome Autosorb Automated Gas Sorption System.

Micro-pore volume and pore size distribution- Both were analysed by t-method micro pore analysis and density functional theory (DFT) method, respectively.

Preparation of Silica nanoparticles

Pre-treatment of the sugarcane bagasse fly ash (SBFA)

To remove the oxide compounds of minor elements from bagasse fly ash, an acid treatment was performed. Around 100g of SBFA was added to 500 ml of 0.1 M of chloroacetic acid ($C_2H_3lO_2Cl$) and kept under continuous shaking for 2 hrs**.** The suspension was then filtered and washed with double distilled water and pH was maintained to 7. The solid was dried in hot air oven at 110ºC for 24 hrs, then pre-treated fly ash was sieved using 35-mesh size sieve, and the fraction with smaller particles was saved.

Extraction of silica from sugarcane bagasse fly ash (SBFA)

The acid treated and washed SBFA was mixed with NaOH solid (pallets) in a weight ratio of 1: 1. The mixture was heated in a muffle furnace at 400^{O}C for 1 hr, after which, the mixture was left for cooling to room temperature. Then, 100 ml of double distilled water was added to the mixture and refluxed for 4 hrs. The mixture was thereafter filtered to separate the solid residue of sodium silicate (Na_2SiO_3) solution.

Preparation of silica nanoparticles from extracted sodium silicate

Nano-silica was prepared wherein, 5g of HCTAB a surfactant was dissolved in a mixture composed of water and pentanol (1: 1, v/v) solvent in a 500 ml of round bottom flask. 50 ml of SBFA-derived sodium silicate was slowly added into the HCTAB/water/pentanol solution, and the mixture was stirred at 60ºC. Then, 0.4 M sulfuric acid solution was added gradually into the suspension in order to initiate the hydrolysis-condensation reaction. The suspension was adjusted until thepH was4, and the resulting gel was aged 60ºC for 10 hrs. The aged silica gel was dispersed in n-pentanol and washed with warm double distilled water for several times. The final product was obtained and kept in desiccator before further characterisation.

4. Results and Discussion

Sugarcane bagasse fly ash contains a lot of other different impurities especially inorganic salts and carbon species. The pre-treatment procedure was performed to remove excess of inorganic salts, organic compounds, and low solubility elements through washing and to eliminate particles having large size by using sieve.

After this pre-treatment procedure, silicon was extracted from sugarcane bagasse fly ash (SBFA) by performing reaction with sodium hydroxide (NaOH) under heating in muffle furnace. At 400ºC, sodium hydroxide is melted, which increases silicon purity, liberating elements which can be in the structure of fly ash, making them more soluble. The procedure of extraction generates silicon in the form of sodium silicate, which was solubilized in double distilled deionized water by refluxing. Mixture was filtered and saved for the preparation SiNPs.

Nano-silica was obtained from sodium silicate solution using precipitation method as per following reactions:

- $2NaOH + SiO_2 \rightarrow Na_2SiO_3 + H_2O$

SiNPs can be prepared by various methods including hydro-thermal synthesis, flame synthesis, sol-gel method and the reverse microemulsion technique and the functionalization of SiNPs may be performed by grafting or co-condensation methods. In this particular co-condensation, the SiNPs synthesis and functionalization occurs in a single step which involves the hydrolysis-condensation reaction (Wu, S. -H. et al. ., 2013).

In this study, the formation of SiNPs is based on hydrolysis viz. production of silanol groups and condensation viz. production of siloxane, reaction using sulfuric acid in a biphasic medium in the presence of Hexadecytrimethyl-ammonium bromide (HCTAB) (Wang, X. et al., 2004).

HCTAB is a classical micelle maker surfactant and helps to control the size of nanoparticles, by preventing agglomeration and to modify co-condensation procedure using sulfuric acid as a catalyst thus to generates white solid particles with size of 2 to 4 nm (Hoffmann, F. et al., 2006). The result indicates that the HCTAB surfactant has coated uniformly the surface of the material giving it much better dispersion in suspension and easily removed by centrifugation.

The silica particles were generated from the solution by adding sulfuric acid, acidic condition pH 4 ensured almost the complete precipitation of silica from sodium silicate as per following reaction:

- $Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O$

The yield of silica nanoparticles from the screened sugarcane bagasse fly ash was 25%-30%. This result demonstrates significant purification of the sample that generates SiNPs.

Characterization of Prepared Silica Nanoparticles

- **Scanning Electron Microscopy**

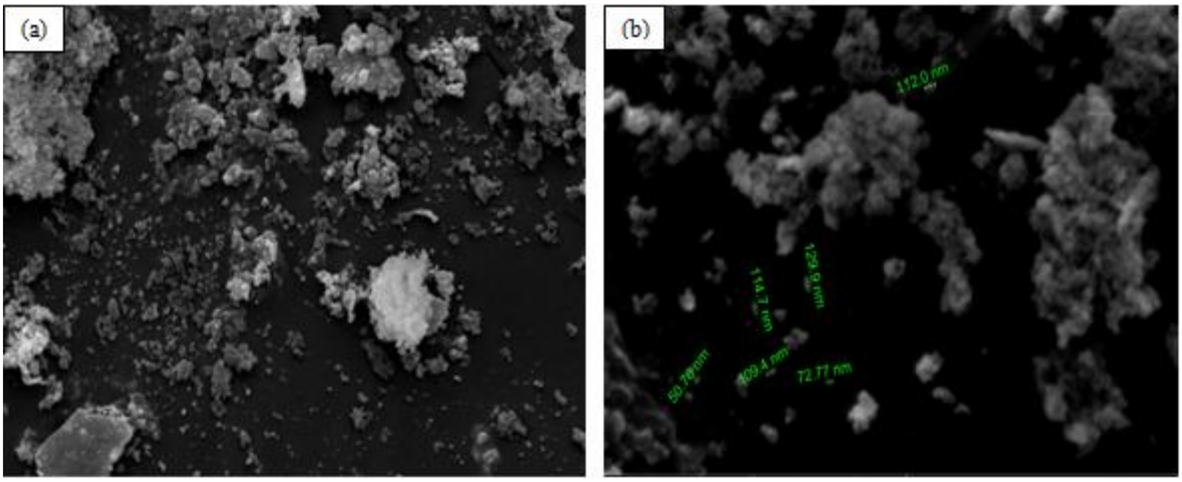

Figure 1: SEM Images of (a) SBFA and (b) Silica nanoparticles.

The SEM image of sugarcane bagasse fly ash shown in Figure 1 (a) reveals a heterogeneous material with irregular shapes and high roughness which are associated with the release of organic matter during burning of bagasse. From figure 1 (b) SEM micrograph of silica nanoparticles obtained from sugarcane bagasse fly ash, drastic change in the product can be observed compared to the original fly ash which is conformity with the findings of other research work (Le, V. H. et al., 2013).

- **X-Ray Diffraction (XRD)**

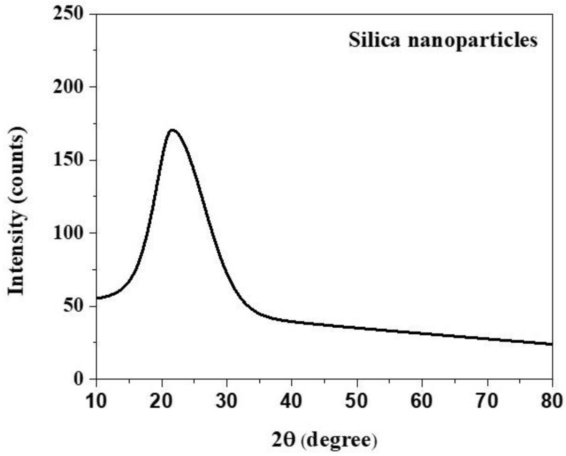

Figure 2: X-Ray diffraction pattern (XRD) of the silica nanoparticles

The characteristic peak corresponding to the silica nanoparticles in diffraction pattern was very similar to amorphous silica SiNPs, as can be seen by a band with a maximum peak at θ = 22° (Chen, X., et al., 2014). The amorphous form of silica is almost inoffensive, being even used for drug delivery, (Chen, F. et al., 2017) whereas the crystalline form of silica is highly toxic, promoting silicosis disease. The bagasse fly ash silica nanoparticles were amorphous, as indicated by the XRD broad peak and the disordered arrangement of the silica network.

- **Fourier Transform Infrared Spectroscopy (FT-IR)**

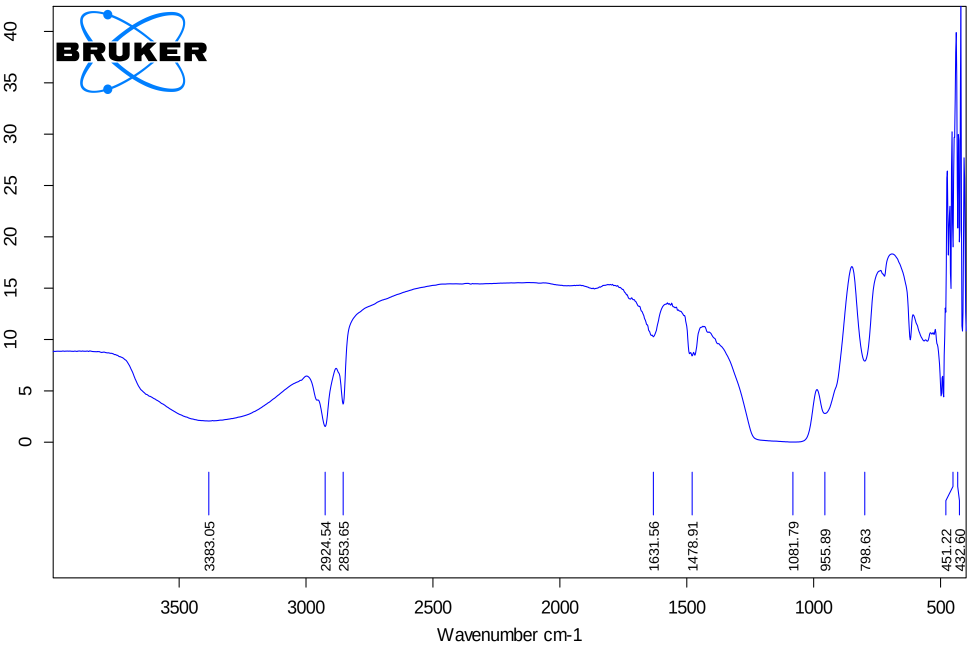

Figure 3: FT-IR total absorbance spectra of silica nanoparticles

The presence of HCTAB on the surface of SiNPs which was dried at 100ºC, can be observed by Fourier Transform Infrared Spectroscopy (FT-IR), as showed in the figure 3 where the IR spectra of SiNPs adsorption are presented. In the FT-IR spectrum of SiNPs, presence of four main bands of silica was observed at 799 cm^{-1} and 451 cm^{-1}, the bands correlate with symmetric stretching of siloxane groups (Si-O-Si), the very strong band at 1062 $cm^{-1}$is referred to (Si-O-Si) asymmetric stretching and band due to OH bending of silanol groups was observed at 956 cm^{-1}which is similar to the other research studies (Boza, A. F., et al., 2016). An absorption band at 1632$cm^{-1}$was found in bagasse silica, showing the presence of a small amount of water with a bending vibration of either free-OH groups or free H_2O molecules.

Bands related to HCTAB dispersed over SiNPs were observed 2925 cm^{-1} and 2854 $cm^{-1}$are due to bending of $CH_2$and $CH_3$of the HCTAB surfactant. The FT-IR spectra show C-H peaks at 2925 cm^{-1} and 2854 cm^{-1}, clearly indicating the organic modification of the nanoparticle surface and the silica nanoparticle obtained in amorphous state, verified by (Hu, S. et al., 2014).

- **The Brunauer-Emmett-Teller (BET)**

The specific surface area was determined using BET method which employs adsorption of adsorbate (nitrogen gas) on the surface of the adsorbent (SiNPs) using BET theory. The resulting BET equation is expressed by the given equation,

$$\frac{P/P_0}{V_a\,(1-P/P_0)} = \frac{1}{V_m C} + \frac{C-1}{V_m C}\,(\frac{P}{P_0})$$

where, P is the partial vapour pressure of adsorbate gas in equilibrium with the surface at-195.85°C (b. p of liquid nitrogen) in pascals, P_0 the saturated pressure of adsorbate gas, in pascal, $V_a$is the volume of gas (Nitrogen gas) at the pressure P, $V_m$is the volume of gas adsorbed when the surface of the solid (SiNPs) is covered completely with the monolayer of the adsorbed molecules of the gas and C is a constant depending upon the nature of the gas which approximately given by,

$$C = e^{\left(\frac{E_1 - E_L}{RT}\right)}$$

where E_1 is the heat of adsorption in the first layer and E_L is the heat of liquification of the gas.

Micro-pore analysis and pore size distribution were determined by "*t*-method" of de-Boer micro-pore analysis method and density functional theory (DFT) method respectively.

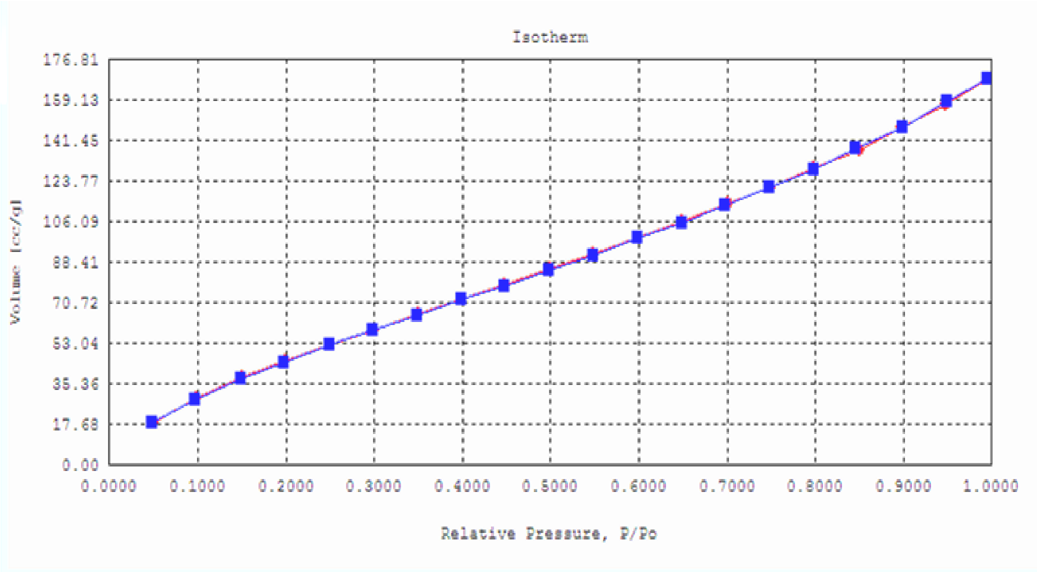

Figure 4: Adsorption-desorption isotherm of N_2 of the prepared silica nanoparticles

Figure 4 shows N_2 adsorption-desorption isotherms of sugarcane bagasse fly ash based SiNPs prepared. Both the isotherms are type I, this means SiNPs contained mesopores and are intended for various uses as adsorbent due to their small pore size. The BET surface area of the prepared SiNPs was 222 m^2/g which is higher than as reported by other research workers (Rovani, S. et al., 2018). The other characteristics indicated inTable 2.

Table 2: Silica nanoparticles characteristics

Characteristics	Value
Surface Area	222 m^2/g
Average pore size	3.58 nm
Pore width	2.64 nm
Total pore volume	0.26 cc/g
Micro-pore volume	0.17 cc/g
Micro-pore area	63.9 m^2/g

Properties of Silica

Silanol groups are formed on the silica surface during the condensation-polymerization of silicate Si $(OH)_4$. In amorphous silica, the bulk structure is a random packing of a Si $(OH)_4^{-4}$unit. Silanol groups are generally found on the surface and within the structure of silica particles. The silanol surface OH groups are the main centre of water molecule absorption. The water molecule can be associated by hydrogen bonds to surface silanol and sometimes to internal silanol groups. There are two types of physically adsorbed water (physiosorbed water) on the silica surface, i.e., low activation energy and high-low activation energy (Zhuravlev, L. T. 1993).

Figure 5: Images of (a) bagasse ash (b) silica nanoparticles

Effect of surfactant on the of silica nanoparticles

The results show that the cationic surface-active substances coat uniformly the particle surface. In addition, due to the high surface energy and free OH groups on the silica surface which produce the hydrogen bond with water molecules, when the dispersed silica was isolated from the solvent, this hydrogen bond was also removed, resulting in much better dispersion and formation of smaller particles and preventing it from agglomeration.

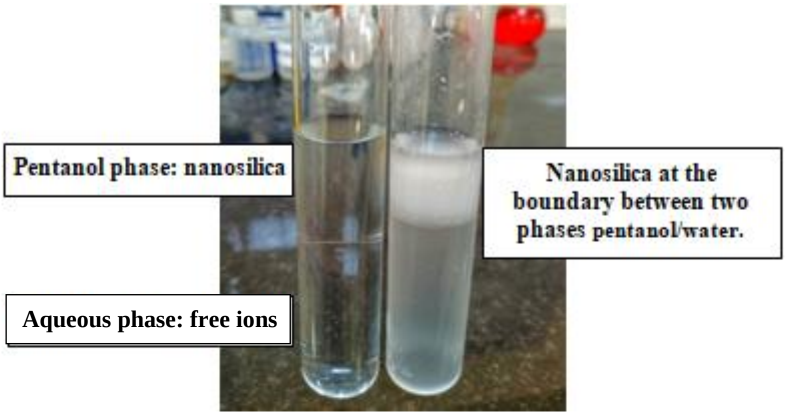

Figure 6: Silica nanoparticles dispersed in pentanol/water

Effect of aging temperature and time on the particle size of silica nanoparticles

Achieving the particle size of the silica nanoparticles depends on the stability of silica sol. It mainly influenced by aggregative stability, the most characteristic for colloidal systems, Colloidal stability means that the particles do not aggregate at a significant rate. Aggregation is used to describe the structure formed by the cohesion of colloidal particles. So, in this investigation, we observed the effect of aging temperature and time on the stability of silica nanoparticles.

To optimize the formation condition of silica nanoparticles, the effect of aging temperature plays a vital role. When the aging temperature changes the dispersion states and sizes of silica nanoparticles also changes and the best results of silica nanoparticles were achieved at 60ºC which is validated by similar research work (Van Hai Le et al., 2013). Increase in the temperature leads to increase interaction between the hydroxyl groups on the silica surface with HCTAB-a cationic surfactant. However, when the aging temperature is increased up to 80ºC-100ºC, the surfactant molecules adsorbed on the surface of silica tends to desorption, leading to reduction of dispersion of the silica nanoparticles forming a Si-O-Si linkage and resulting to larger size particles by which agglomeration occurs (Van Hai Le et al., 2013). In the instant studies also, temperature to the extent of 60ºC was therefore kept.

5. Conclusion

Authors could successfully synthesize silica nanoparticles were from the sugarcane bagasse fly ash obtained from the boilers of various sugar factories. A new green synthetic method for producing silica nanoparticles using SBFA as the silica source and HCTAB as surfactant via sol-gel technique in water/pentanol was investigated. The method issimpler and provides an effective route aiming at production of fine powders on a nano-meter scale with a homogeneous particle size distribution. This study leads to the development of technology for low-cost production of silica nanoparticles for various practical applications such as nano-fertilizer, pollution treatment, nano-composite material etc. The results indicate that it was possible to obtain highly pure silica in a nanosize from the waste material and produce an adsorbent with good adsorption capacity with possibility of reuse.

It is envisaged that utilization of SBFA in such a manner will help in resolving environmental issues in the sugar factories with development of a value-added product.

6. Future Scope

The study shall further be taken up to analyse ash from boilers working on other biomasses or from multi-fuel boilers to explore possibilities of deriving silica nanoparticles.

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Author Profile

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Dr. Vishnu Prabhakar Srivastava after completing his doctorate in Chemistry, joined National Sugar Institute, Kanpur as Assistant Professor in Organic Chemistry. Dr. V. P Srivastava is a research worker par excellence who developed many technologies for producing value-added products from by-products and waste from sugar industry. At present he is working as Associate Professor in Department of Chemistry, University of Allahabad, (Prayagraj).

Prof. Narendra Mohan, an inspiring teacher, admirable research worker and an esteemed government official having a long and distinguished career of working in sugar industry and at the institute. An author of 6 books and more than 150 papers been published in various reputed journals which reflect his passion for innovative work to covert "Waste to Resource". He is a visiting speaker and expert who travelled to many countries.