



**Original Research Article**

**Size Controlled Synthesis of Silica Particles Using Extraction Precipitation Method**

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**ABSTRACT**

The extraction-precipitation method has been a desired method of synthesizing silica particles. In this work, silica particles were synthesized from sugarcane bagasse using this method. The identity of the particles was determined with Fourier Transform Infra-Red (FT-IR), and the particle size was measured with a Scanning Electron Microscope (SEM). Smaller-sized silica particles were obtained by extracting silica with sodium hydroxide from the sugarcane bagasse and obtaining the precipitate by bringing the pH down. In order to increase the size, sodium hydroxide was again added to the silicate solution before the pH was brought down, and the obtained precipitate was heated to a higher degree Celsius. Its hydroxyl content was also reduced. Previous studies have revealed the potential of sodium hydroxide addition for silica particle increment using the Stober method. We reported here an increase in silica particle size with the aid of sodium hydroxide as a catalyst through a greener technique, the extraction-precipitation method.

**Keywords:** FT-IR, SEM, Silica Microparticles, Silica Nanoparticles, Sugarcane Bagasse.

## Introduction

Because they are a promising option for drug delivery, gene therapy, the detection of biomolecules, photodynamic treatment, and bioimaging, silica nanoparticles (SiO<sub>2</sub> NPs) have drawn a lot of attention lately. Researchers have become interested in SiO<sub>2</sub> NPs because they are amenable to modification through well-known organosilane chemistry, which enables the inclusion of functional groups [1]. The parameters of its synthesis, such as the synthesis temperature, precipitation time, pH, the use of surfactants, and methods for washing and drying, have a significant impact on the properties of precipitated silica. These elements have an impact on the size, morphology, and aggregation of SiO<sub>2</sub> particles, all of which affect the final particles' size and size distribution. Additionally, the size and size distribution of the silica nanoparticles have a significant impact on the quality of the products made from silica particles [2]. However, there is a barrier to practical use because there is little knowledge among the several recognized approaches on the management of dispersity, reaction time, and size, particularly for creating nanometer-scale particles. Only a small amount of research has been able to successfully manipulate silica particles size down to tens of nanometers [3]. Through the straightforward addition of sodium hydroxide to the silicate solution during the precipitation step, this work claimed a potential size growth of silica particles to microparticles.

The widely used techniques for creating silica nanoparticles, such as the Stober method, sol-gel method, flame synthesis method, and microemulsion modified approach, are less eco-friendly. We used the extraction and precipitation method in this study. This approach has the advantages of using less harmful chemicals, using fewer chemicals overall, being more cost-effective, and requiring less energy-intensive procedures like calcination [4]. Additionally, the hydrochloric acid processing of the sugarcane bagasse eliminated the metallic contaminants. At the conclusion of this investigation, high-purity silica particles will be produced because metallic impurities were not present. Because sugarcane is one of the primary possible sources for silica extraction and subsequent transformation into nanostructured silicon, sugarcane bagasse was chosen as a source of silica particles [5].

In order to create highly monodisperse silica nanoparticles, [6] used the fluorinated surfactant HOCH<sub>2</sub>CH(CF<sub>3</sub>)CO<sub>2</sub>H. By adjusting the surfactant concentration, they were able to control the size of the SiNPs (50–200 nm). Moreover, by raising the surfactant content under the same circumstances, the particles' size, shape, and dispersibility were also adjusted. They also

employed a number of different surfactants at a certain concentration and used a number of characterization methods to examine the size, shape, and morphology. Unfortunately, non-uniform morphology was visible for some surfactants at specific concentrations. They came to the conclusion that MAF-OH appears to behave as a substitute surfactant for the procedures already in use for the uniform and size-controlled synthesis of SiNPs.

Using a cheap silica source (rice husk) and polyethylene glycol (PEG), [7] created spherical silica nanoparticles with adjustable particle size and mesoporous characteristics. According to a FESEM examination, increasing the PEG content from 0.01 to 0.005 M resulted in the production of spherical silica nanoparticles with a size range of 100–500 nm. They came to the conclusion that this strategy could be a quick and affordable way to make mesoporous silica nanoparticles with adjustable nanoscale properties for useful applications.

[8] discovered that regardless of the solvents employed for the synthesis, the size rises by increasing the amount of the catalyst,  $\text{NH}_4\text{OH}$ , and silica precursor (tetraethylorthosilicate, TEOS), and by reducing the amount of water in the reaction mixtures. Additionally, it was shown that while the composition of the reaction mixture was fixed, the particle size produced by using ethanol as a solvent was substantially bigger than that produced by using methanol as a solvent. By adjusting the iron oxidation state, [9] study further established the silica nanoparticles' pore size dependence. Only spherical SNPs are formed in the absence of any Fe species, but SNPs with rod-like and nanosheet structures are produced in the presence of  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  ions.

[4] Reported on the effective synthesis of silica nanoparticles from sugarcane bagasse using a green approach of extraction and precipitation. Additionally, [10] reported on the method of replacing ammonium hydroxide with sodium hydroxide as a catalyst assisting the control of silica particle size by the Stober method.

The use of sodium hydroxide to regulate particle size has not yet been documented utilizing a green extraction and precipitation technique. In this study, we created silica particles from sugarcane bagasse using environmentally friendly extraction and precipitation technique. By using sodium hydroxide during the precipitation stage, we were able to scale up the size to the microscale. The functional groups present in the particles and their size were determined using Fourier Transform Infra-Red (FTIR) and Scanning Electron Microscope (SEM) respectively.

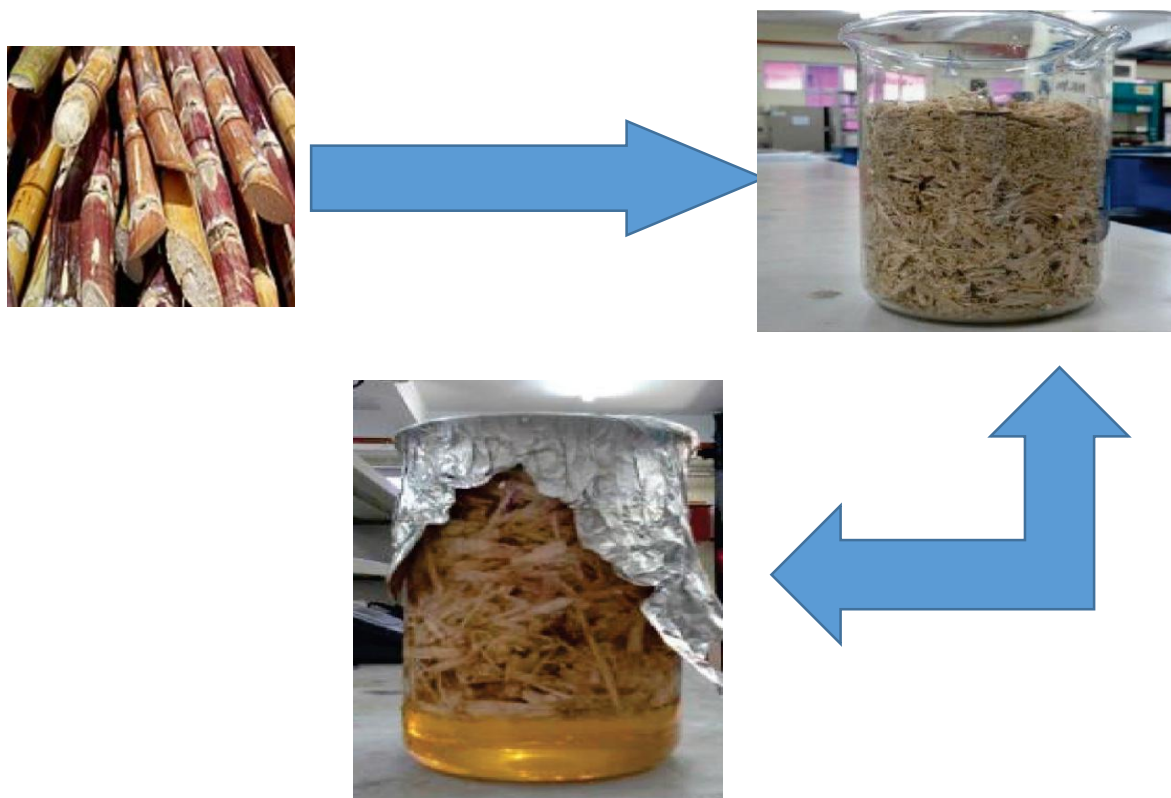
## Experimental

### Sample Collection and Preparation

Sugarcane was purchased from sugarcane vendors. To get the fibrous residue, it was chewed to remove the sugar. After being dried in the absence of sunshine, the sugarcane bagasse was steeped in distilled water for 24 hours. It was once again cleaned with distilled water before drying in the oven for 97 °C.

### Silica Extraction

To eliminate the metallic impurities, 1M hydrochloric acid was applied to the sample, and then it was immersed in a water bath at 70 °C. The mixture was filtered, extensively washed to get rid of the metallic ions, and dried in a 95 °C oven. The material was dissolved in 1M sodium hydroxide and boiled in a water bath for an hour to extract the sodium silicate. This is the procedure of [4], but our modification to it followed at the precipitation step.



### Precipitation Process

The mixture was filtered to remove the sugarcane bagasse, the sodium silicate was stirred for an hour. Prior to precipitation, the solution was divided into two portions. To the first portion,

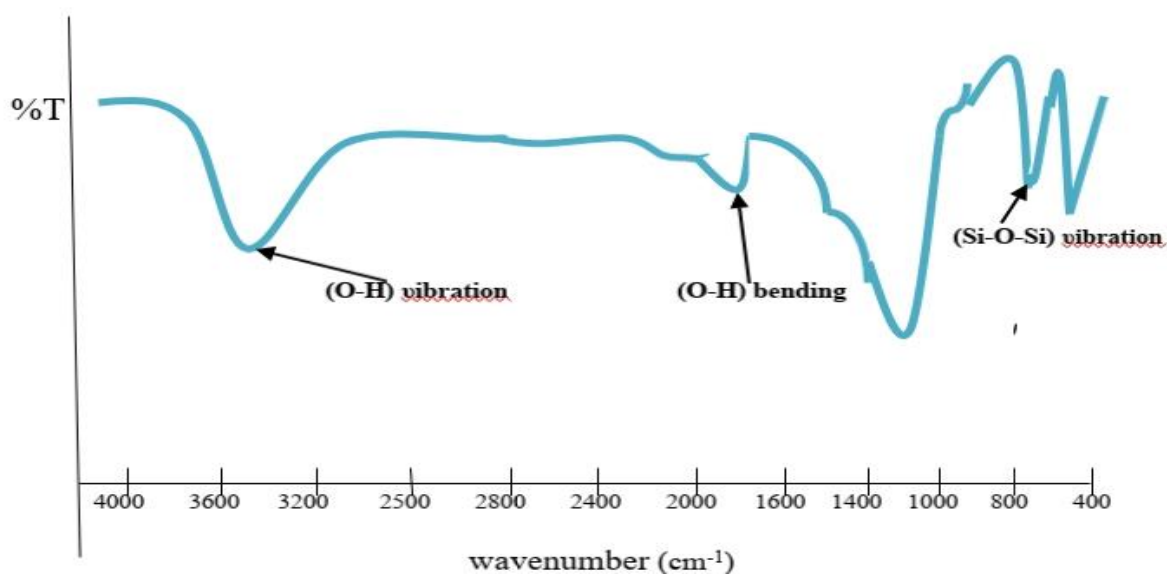
concentrated nitric acid was added in a drop wise manner until the pH reduced to 8 and 20ml of ethanol was added. The resulting solution was stirred for 50 minutes then centrifuged at 4000 revolution per minute. Silica nanoparticles were obtained by heating the precipitate collected at 300°C for 20 minutes.

To the second portion of the silicate solution, concentrated nitric acid was slowly poured until the pH reduced to 2. The pH was then increased to 8 by another addition of 1.5M NaOH and 20ml of ethanol was added. The resulting solution was stirred for 50 minutes then centrifuged at 4000 revolution per minute. Silica microparticles were obtained by heating the precipitate collected at 750°C for 25 minutes.

## Results

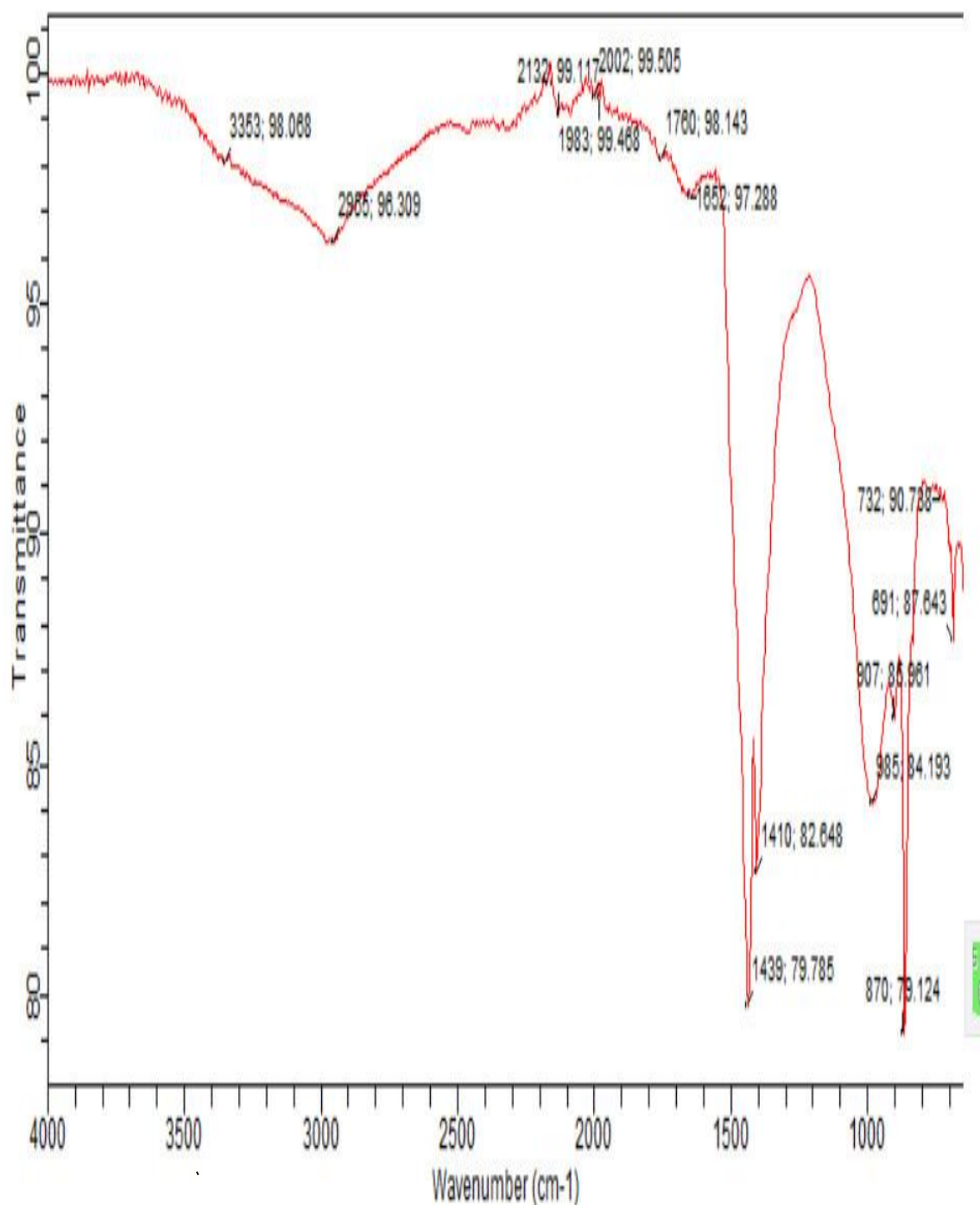
### Fourier Transform Infra-Red (FTIR) Spectroscopy Analysis

The FT-IR spectrum (figure 1) of the first portion of silica particles was in conformity with the one reported by [4]. The vibrational peak of Si-O-Si confirmed the silica characteristics in the sample. The peaks around 450  $\text{cm}^{-1}$ , 810  $\text{cm}^{-1}$ , 1080  $\text{cm}^{-1}$  assigned the bending vibration, stretching vibration and asymmetric vibration of siloxane (Si-O-Si) respectively [11]. A broadly intense peak around 3500  $\text{cm}^{-1}$  indicates the presence of hydroxyl stretching vibration of the silanol group (Si-O-H) on the silica surface. The slightly broad and intense peak around 1690  $\text{cm}^{-1}$  was due to hydroxyl bending vibration [12].



**Figure 1:** The FTIR spectra of the silica nanoparticles synthesized from sugarcane bagasse.

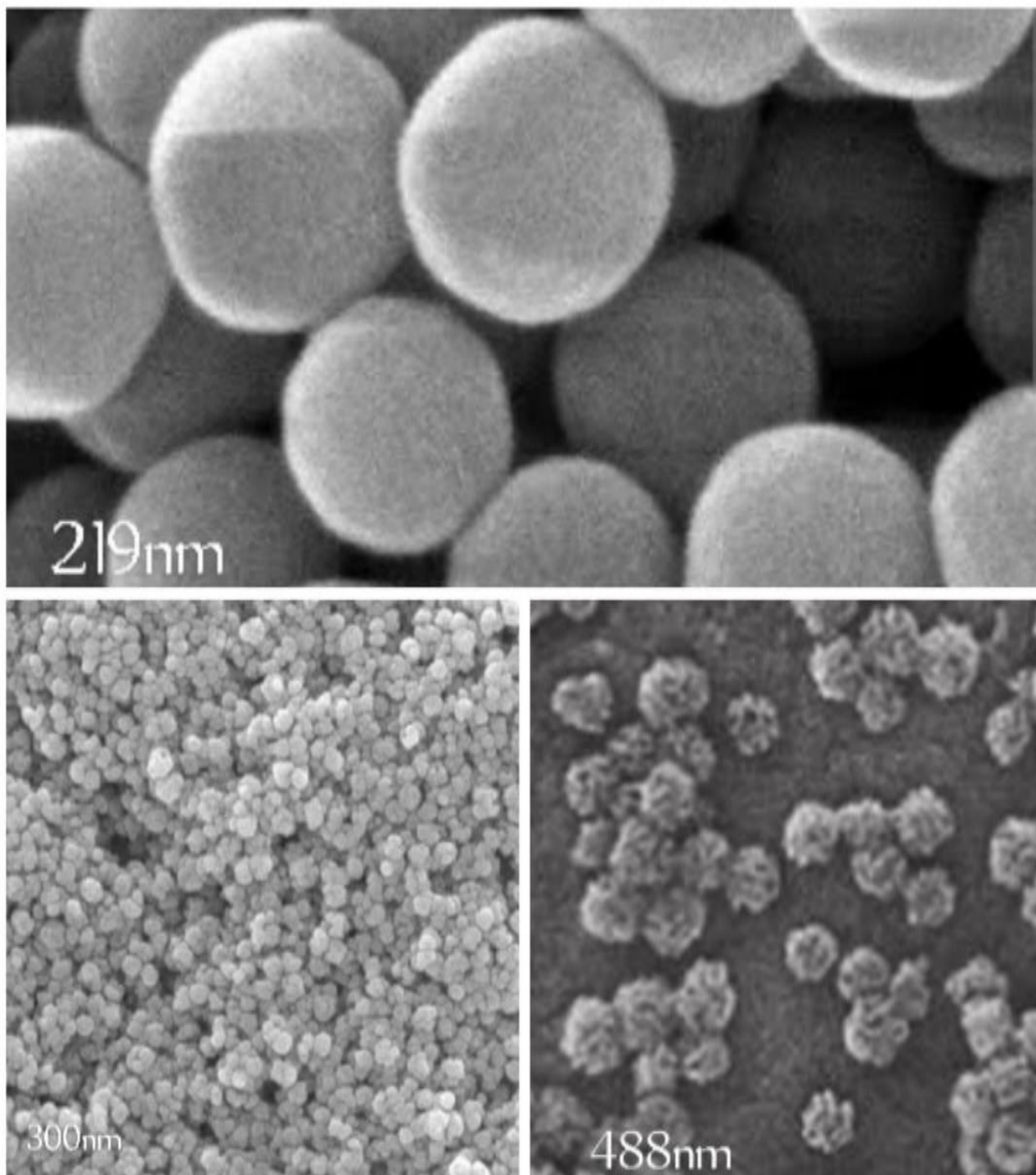
The vivid difference between the FTIR spectrum of the silica nanoparticles (figure 1) and that of silica microparticles (figure 2) is the weakness of the hydroxyl (i.e silanol) peak around 3353 in figure 2. This is because higher temperature (700°C) was used in heating the precipitate of silica microparticles. The hydroxyl group content reduces as the temperature increases, due to the promotion of condensation [13].



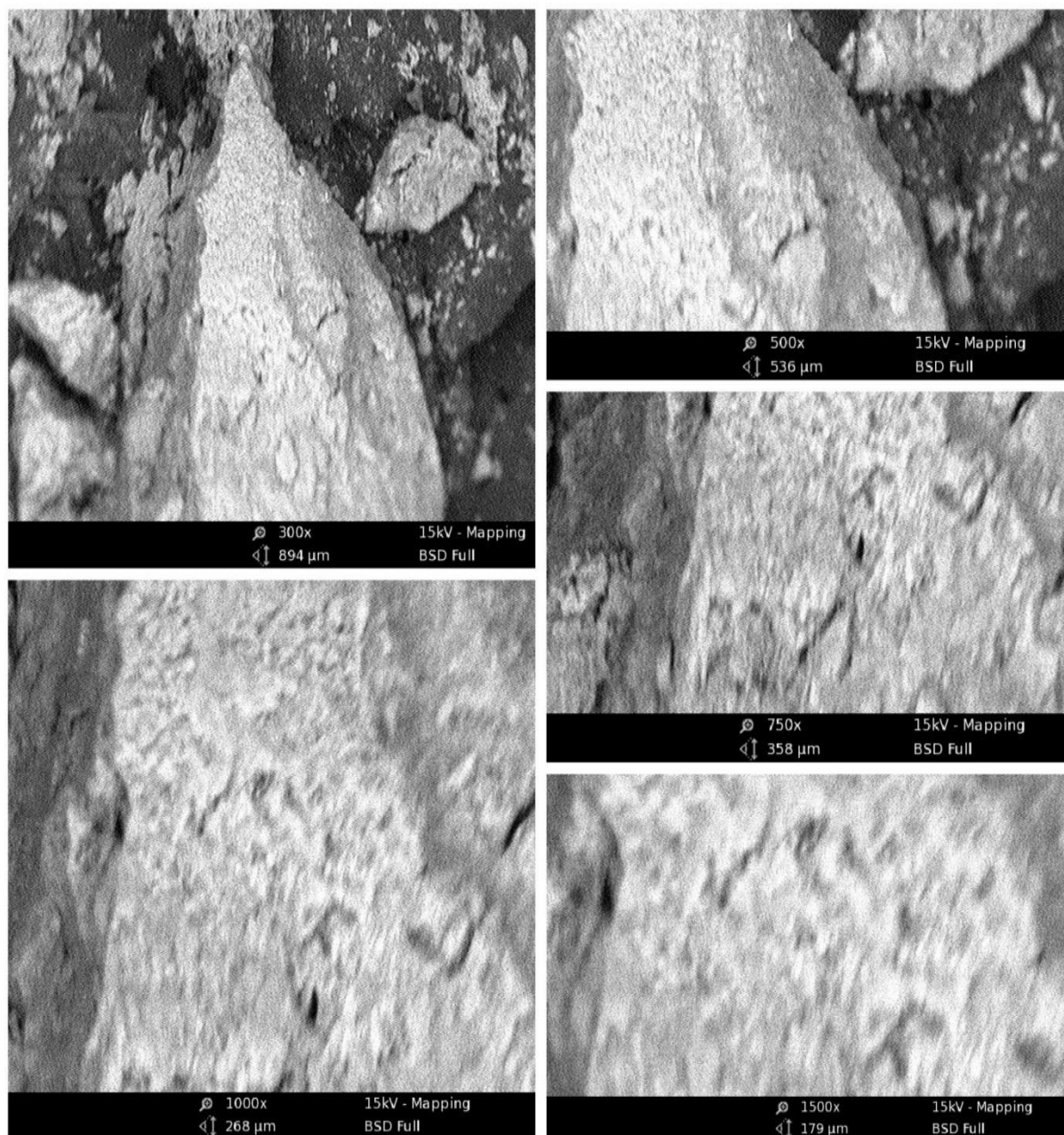
**Figure 2:** The FTIR spectra of silica microparticles synthesized from sugarcane bagasse

### Scanning Electron Microscope

SEM is a method for capturing images of surface morphology by giving details on the size, shape, and morphology of the materials. Size disparity between the two different particles synthesized was determined with Scanning Electron Microscopes [14].



**Figure 3:** The SEM photograph of silica nanoparticles produced using extraction and precipitation method



**Figure 4:** The SEM photograph of silica microparticles synthesized using extraction and precipitation method

The size of the first precipitate was found within the range of 200nm to approximately 400nm. For the second precipitate, it was in the range of 170μm to approximately 900μm. The increase in size was due to the fact that 1.5M NaOH was employed as a catalyst in the precipitation step and a higher temperature was used. The addition of sodium hydroxide allows for a decrease in the size of silica nanoparticles [10]. The higher temperature engineered in heating its precipitate has also contributed to the size increase. The size of silica nanoparticles also increases with increasing temperature [15].

## Conclusion

Silica particles was synthesized using safer approach named extraction-precipitation method. The catalytic activity of sodium hydroxide in increasing the particles' size using a green method was reported. The FT-IR result of the microparticles obtained indicated less hydroxyl group content. The SEM result of the nanoparticles revealed a size range of 200nm to 500nm, while that of microparticles was 100µm to 800 µm.

## Recommendations

For the goal of structural and surface area analysis, we hereby advocate additional characterization of the produced silica particles with X-ray diffraction (XRD) and a BET surface analyzer.

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## Conflict of Interest

The authors declare that they have no conflict of interest.

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