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SYNTHESIS OF SILICA NANOPARTICLES FROM SUGARCANE WASTE: PRECIPITATION-BASED SIZE CONTROL AND CHARACTERIZATION

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ABSTRACT

Sugarcane waste, a byproduct of sugar production, poses significant environmental challenges through releasing harmful gases that contribute to air pollution and greenhouse gas emissions. This work presents the utilization of sugarcane waste to synthesize silica nanoparticles using the extraction-precipitation method. Fourier Transform Infrared (FT-IR) was used to identify the particles, and a Scanning Electron Microscope (SEM) was used to assess their size. By extracting silica from the waste material using sodium hydroxide and lowering the pH to achieve the precipitate, smaller-sized silica nanoparticles were produced. On the other hand, the silicate solution was again treated with sodium hydroxide to increase its size before the pH was lowered and the resulting precipitate was heated to a higher temperature. This also reduced the hydroxyl group content that is present due to silanol vibration. Previous studies have revealed the potential of sodium hydroxide addition for silica particle increment using the Stober method. We reported here an unprecedented increase in silica particle size with the aid of sodium hydroxide as a catalyst through a greener technique, the extraction-precipitation method.

Keywords: Silica Nanoparticles, Sugarcane Waste, Extraction-Precipitation Method

INTRODUCTION

Because they are a promising option for drug delivery, gene therapy, the detection of biomolecules, photodynamic treatment, and bioimaging, silica nanoparticles (SiO_2 NPs) have drawn a lot of attention lately. Researchers have become interested in SiO_2 NPs because they are amenable to modification through well-known organosilane chemistry, which enables the inclusion of functional groups (Purcar et al., 2021). 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_2 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 (Joni et al., 2020). 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 particle size. (Kim et al., 2016). 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 (Mohd et al., 2017). Additionally, the hydrochloric acid processing of the sugarcane waste eliminated the metallic contaminants. After this investigation, high-purity silica particles were 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 waste was chosen as a source of silica particles (Ni'mah et al., 2023).

To create highly monodisperse silica nanoparticles, Nagappan et al. (2021) used the fluorinated surfactant $HOCH_2CH(CF_3)CO_2H$. 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 several different surfactants at a certain concentration and used several characterization methods to examine the size, shape, and morphology. Unfortunately, non-uniform morphology was visible for some surfactants at specific concentrations.

Using a cheap silica source (rice husk) and polyethylene glycol (PEG), Priyan et al. (2023) created spherical silica nanoparticles with adjustable particle size and mesoporous characteristics. According to a FESEM examination, increasing the PEG content from.01 to.005 M resulted in the production of spherical silica nanoparticles with a size range of 100–500 nm. They concluded that this strategy could be a quick and affordable way to make mesoporous silica nanoparticles with adjustable nanoscale properties for useful applications.

Kim et al. (2006) discovered 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, Coelho et al. (2013) 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 Fe^{3+} and Fe^{2+} ions. Additionally, Chaturvedi et al. (2020) reported on the method of replacing ammonium hydroxide with sodium hydroxide as a catalyst

assisting in 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 waste using environmentally friendly extraction and precipitation techniques. 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.

MATERIALS AND METHODS

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 waste was steeped in distilled water for 24 hours. It was once again cleaned with distilled water before drying in the oven at 97 °C.

Silica Extraction

To eliminate the metallic impurities, 1M hydrochloric acid was applied to the sample (figure 1), 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 (Mohd et al., 2017), but our modification to it followed at the precipitation step.

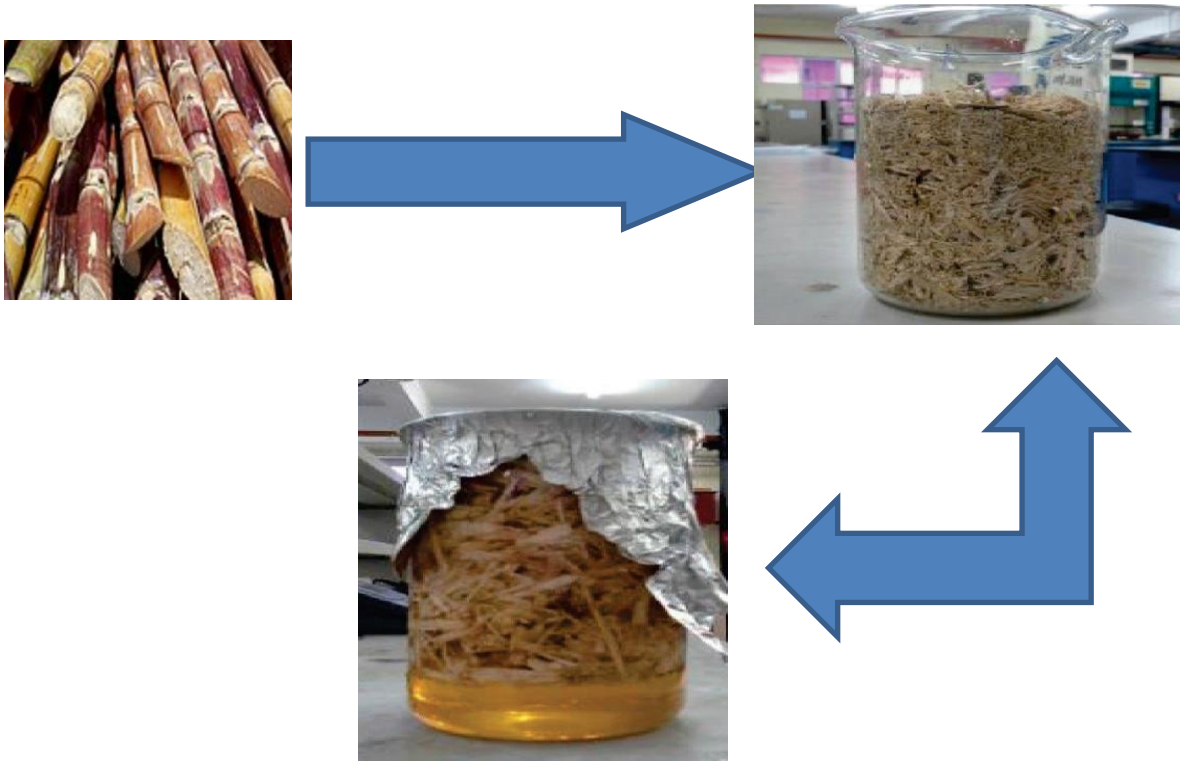

Figure 1: Treatment of Sugarcane Waste with Hcl to Remove Impurities

Precipitation Process

The mixture was filtered to remove the sugarcane waste, the sodium silicate was stirred for an hour. Before precipitation, the solution was divided into two portions. To the first portion, concentrated nitric acid was added in a dropwise manner until the pH was reduced to 8 and 20 ml of ethanol was added. The resulting solution was stirred for 50 minutes then centrifuged at 4000 revolutions per minute. Silica nanoparticles were obtained by heating the precipitate collected at 300℃ for 20 minutes.

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

RESULTS AND DISCUSSION

Fourier Transform Infra-Red (FTIR) Spectroscopy Analysis

The FT-IR spectrum (figure 2) of the first portion of silica particles conformed with the one reported by (Mohd et al., 2017). The vibrational peak of Si-O-Si confirmed the silica characteristics in the sample. The peaks around 450 $cm^{-1,}$ 810 $cm^{-1,}$ and 1080 cm^{-1} assigned the bending vibration, stretching vibration and asymmetric vibration of siloxane (Si-O-Si) respectively (Larkunthod et al., 2022). A broadly intense peak around 3500 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 cm^{-1} was due to hydroxyl bending vibration (Shawky et al., 2016).

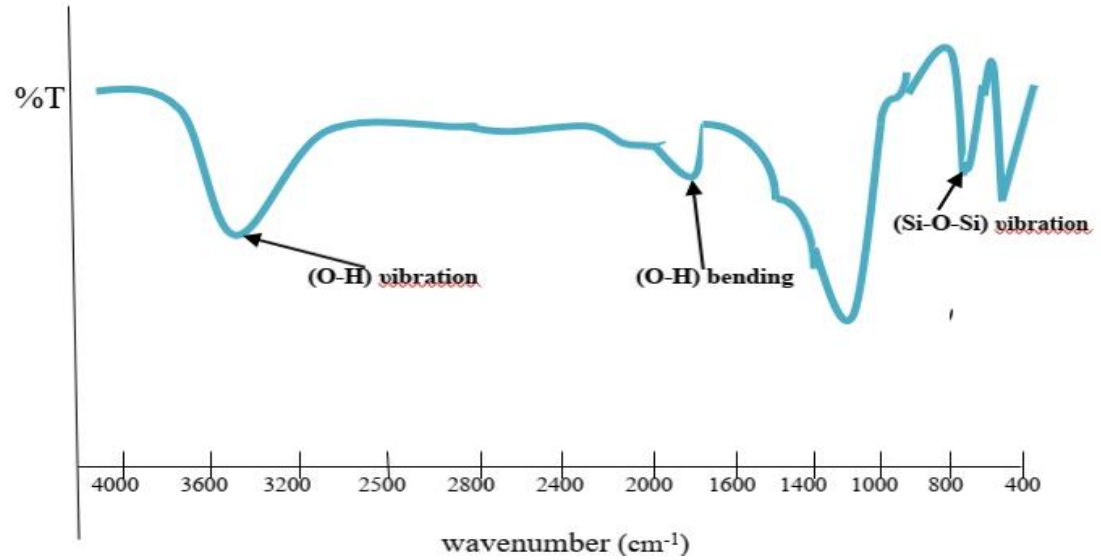

Figure 2: The FTIR spectra of the silica nanoparticles synthesized from sugarcane waste.

The vivid difference between the FTIR spectrum of the silica nanoparticles (figure 2) and that of silica microparticles (figure 3) is the weakness of the hydroxyl (i.e. silanol) peak around 3353 in Figure 3. This is because a higher temperature (700℃) was used in heating the precipitate of silica microparticles. The hydroxyl group content reduces as the temperature increases, due to the promotion of condensation (Kim et al., 2009).

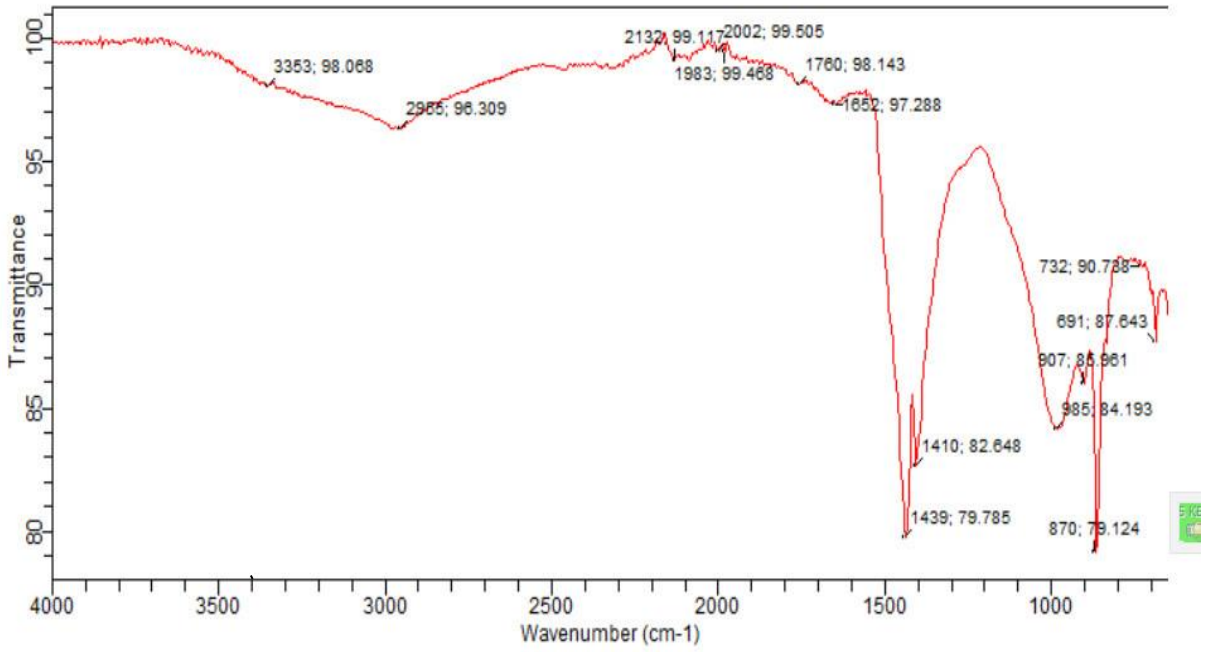

Figure 3: The FTIR spectra of silica microparticles synthesized from sugarcane waste

Scanning Electron Microscope

SEM is a method for capturing images of surface morpholog y by giving details on the size, shape, and morphology of the materials (Mohapatra et al., 2019). The size disparity between the two different particles synthesized was determined with Scanning Electron Microscopes (figure 4&5).

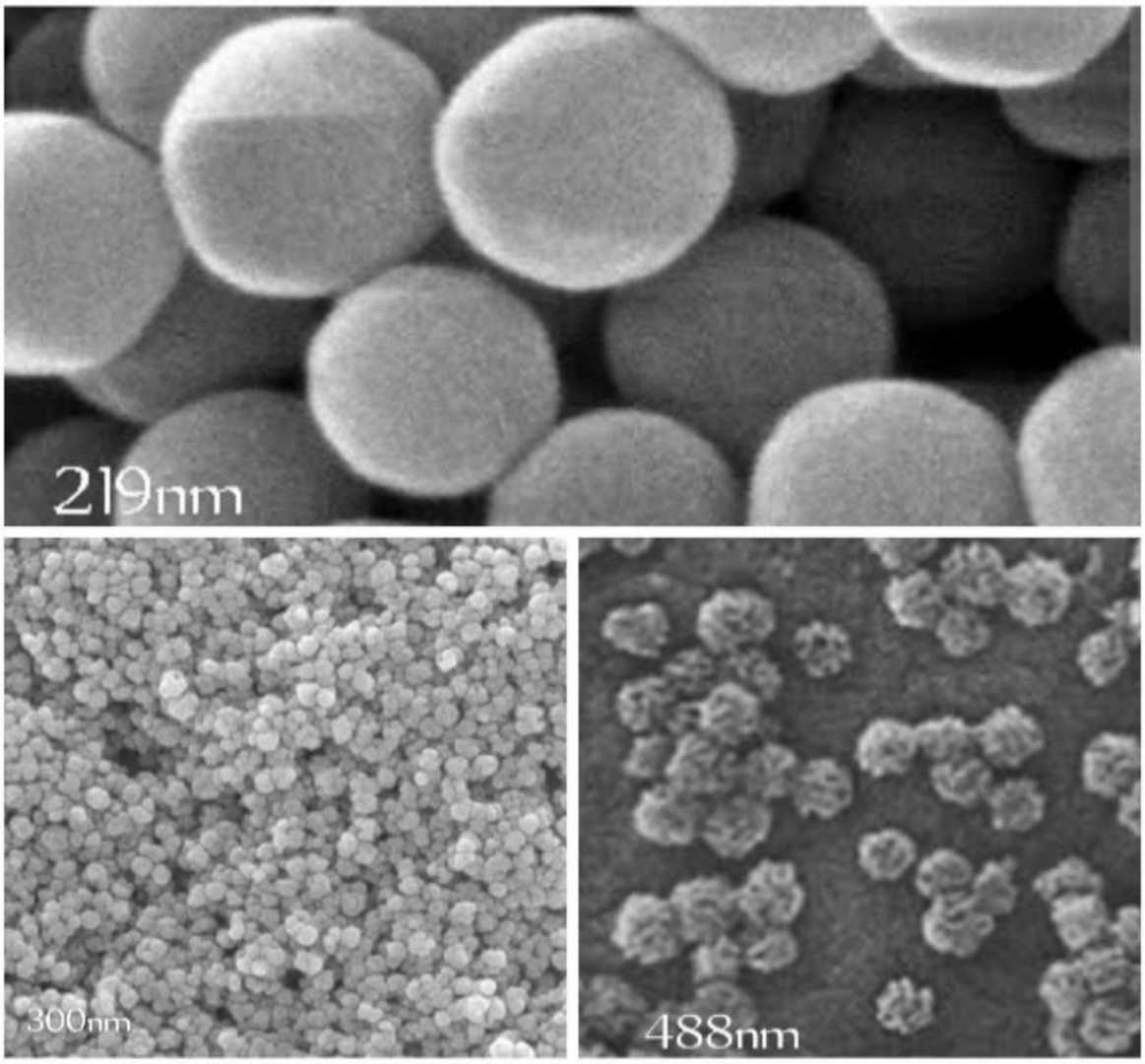

Figure 4: The SEM photograph of silica nanoparticles produced using extraction and precipitation method

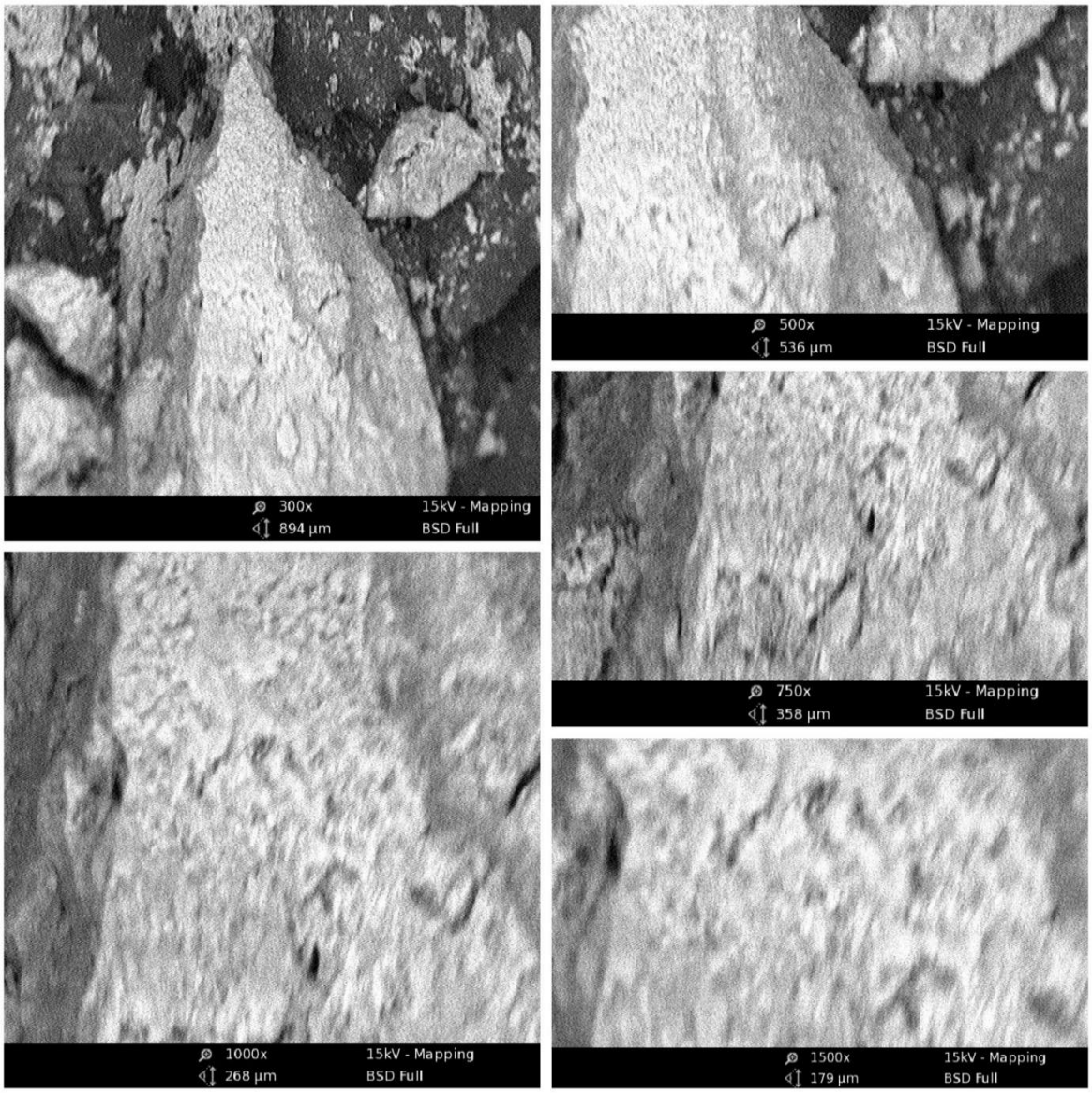

Figure 5: The SEM photograph of silica microparticles synthesized using extraction and precipitation method

The size of the first precipitate was found within the range of 200 nm to approximately 400 nm. For the second precipitate, it was in the range of 170 µm to approximately 900 µm. The increase in size was because 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. 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 (Lim et al., 1970).

CONCLUSION

Silica particles were synthesized using a safer approach named the 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 200 nm to 500 nm, while that of microparticles was 100 µm to 800 µm.

RECOMMENDATION

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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REFERENCES

Chaturvedi, K. R., Trivedi, J., & Sharma, T. (2020). Single-step silica nanofluid for improved carbon dioxide flow and reduced formation damage in porous media for carbon utilization. *Energy*, *197*, 117276. https://doi.org/10.1016/j.energy.2020.117276

Coelho, J. V., Guedes, M. S., Barrera, D., Sapag, K., Pereira, M. C., & De Oliveira, L. C. A. (2013). Controlling the morphology and pore size of mesostructured silica

nanoparticles: the role of the iron oxidation state. Dalton Transactions, 42(31), 11271. https://doi.org/10.1039/c3dt50794g

Joni, I. M., Rukiah, N., & Panatarani, C. (2020). Synthesis of silica particles by precipitation method of sodium silicate: Effect of temperature, pH and mixing technique. *AIP Conference Proceedings*. https://doi.org/10.1063/5.0003074

Kim, H. M., Lee, C. H., & Kim, B. (2016). Sonochemical synthesis of silica particles and their size control. Applied Surface Science, 380, 305–308. https://doi.org/10.1016/j.apsusc.2015.12.048

Kim, J. M., Chang, S. M., Kong, S. M., Kim, K. S., Kim, J., & Kim, W. S. (2009). Control of hydroxyl group content in silica particle synthesized by the sol-precipitation process. *Ceramics International*, *35*(3), 1015–1019. https://doi.org/10.1016/j.ceramint.2008.04.011

Kim, J. W., Kim, L. U., & Kim, C. K. (2006). Size Control of Silica Nanoparticles and Their Surface Treatment for Fabrication of Dental Nanocomposites. Biomacromolecules, 8(1), 215–222. https://doi.org/10.1021/bm060560b

Larkunthod, P., Boonlakhorn, J., Pansarakham, P., Pongdontri, P., Thongbai, P., & Theerakulpisut, P. (2022). Synthesis and characterization of silica nanoparticles from rice husk and their effects on physiology of rice under salt stress. Chilean Journal of Agricultural Research, 82(3), 412–425. https://doi.org/10.4067/s0718-58392022000300412

Lim, Y. H., Kim, D. K., & Jeong, Y. K. (1970). Effect of Reaction Parameters on Silica Nanoparticles Synthesized by Sol-gel Method. *Hanguk Bunmal Jaeryo Hakoeji*, *23*(6), 442–446. https://doi.org/10.4150/kpmi.2016.23.6.442

Mohapatra, S. S., Ranjan, S., Dasgupta, N., Mishra, R. K., & Thomas, S. (2019). Characterization and Biology of Nanomaterials for Drug Delivery. Elsevier.

Mohd, N. K., Wee, N. N. a. N., & Azmi, A. A. (2017). Green synthesis of silica nanoparticles using sugarcane bagasse. AIP Conference Proceedings. https://doi.org/10.1063/1.5002317

Nagappan, S., Mohan, A., Thomas, A. M., Yoo, J. M., Eid, N., Chung, I., Ameduri, B., & Ha, C. S. (2021). Synthesis of size-controlled and highly monodispersed silica nanoparticles using a short alkyl-chain fluorinated surfactant. RSC Advances, 11(4), 2194–2201. https://doi.org/10.1039/d0ra08114k

Ni'mah, Y. L., Muhaiminah, Z. H., & Suprapto, N. (2023). Synthesis of silica nanoparticles from sugarcane bagasse by sol-gel method. AIP Conference Proceedings. https://doi.org/10.1063/5.0107310

Priyan, S. R., Kumar, G. S., Surendhiran, S., & Shkir, M. (2023). Size-controlled synthesis of mesoporous silica nanoparticles using rice husk by microwave-assisted sol–gel method. *International Journal of Applied Ceramic Technology*, *20*(5), 2807–2816. https://doi.org/10.1111/ijac.14444

Purcar, V., Rădiţoiu, V., Nichita, C., Bălan, A., Rădiţoiu, A., Căprărescu, S., Raduly, F. M., Manea, R., Şomoghi, R., Nicolae, C. A., Raut, I., & Jecu, L. (2021). Preparation and Characterization of Silica Nanoparticles and of Silica-Gentamicin Nanostructured Solution Obtained by Microwave-Assisted Synthesis. *Materials*, *14*(8), 2086. https://doi.org/10.3390/ma14082086

Shawky, S. M., Abo-AlHassan, A. A., Lill, H., Bald, D., & EL-Khamisy, S. F. (2016). Efficient loading and encapsulation of anti-tuberculosis drugs using multifunctional mesoporous silicate nanoparticles [Running Title: Mesoporous Silicate Nanoparticles as Smart Drug Delivery System]. Journal of Nanoscience and Current Research, 1, 103. https://doi.org/10.4172/jncr.1000103