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Fabrication and Characterization of Silica Nanoparticles from Beach Sand

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Abstract. Silica nanoparticles (SNPs) have many important applications such as anti-reflection coating, self-cleaning surface and drug carriers. SNPs are usually synthesized from commercial precursor such as tetraethyl orthosilicate (TEOS). On the other hand, silica is found naturally in organic materials such as rice husk and palm shell or in inorganic material such as sand and clay. Extracting silica from natural resources, therefore, has been considered to be very strategic. This motivates the current study which focuses on the fabrication and characterization of SNPs from the natural resource of beach sand. The silica sands were mixed with sodium hydroxide for 2 hours in 90º C temperature, prior to filtering process for gaining the sodium silicate solution (SSS) which was refluxed with hydrochloric acid (HCl) of 3 M and 5 M until it reached pH of 7, and finally dried. The characterizations of the resulting SNPs included UV-Vis and FTIR spectroscopies, XRD and SEM. UV-Vis and FTIR spectra confirmed the progress of SNPs formation due to presence of Si-OH and Si-O-Si bonding. The XRD data were further analyzed using Scherrer equation and obtained the crystallize size of 4.8 nm and 2.9 nm for SNP3M SNP5M, respectively. The analysis of SEM images using Image-J revealed the average area about 107 -112 nm^2 and the average grain size of 11.6-12 nm. On the basis of results, it was confirmed that the desired silica nanoparticles can be successfully synthesized by using the from the natural resource of beach sand.

Introduction

Silica nanoparticles (SNPs) have vast applications in science, engineering, and health. It was used for anti-reflection coating and self-cleaning windows due to the ease of surface modification so it can be hydrophilic, hydrophobic, and superhydrophobic [1]. It also can be used as a carrier for drugs and vaccines because it can be modified as biocompatible material [2]. Since the SNPs can be applied in various applications, it has attracted researcher to elaborate more on basic research, laboratory tests, and prototype.

The synthesis method of nanomaterials can be classified as bottom-up or chemical method (chemical vapor deposition, hydrothermal, and sol-gel) and top-down or physical method (thermal evaporation, laser ablation, and milling) [3]. The sol-gel method is well known due to its simple process and controllable parameters. One of its methods is the Stober method conducted by mixing tetraethyl orthosilicate (TEOS) as precursor and ethanol with NH_4OH as catalyst [4, 5]. However, commercial TEOS which is frequently used is expensive[1].

Silica is one of the most abundant materials on earth that can be stored in many natural resources, such as rice husk, palm shells, clay, and silica sand. However, silica sand has the highest amount of silica percentage, which can reach 99% [6–10]. Indonesia has a large stock of silica sand due to the very long coastline which encourages many researchers to optimize this potential. So in this paper, the fabrication SNPs was synthesized using silica sand from Belitung Island with sodium silicate solution (SSS) route then followed by neutralization using hydrochloric acid (HCl).

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Experimental Details

The experiment is conducted in two steps, they are formation of sodium silicate solution (SSS) from silica sand and synthesis of silica nanoparticles (SNPs) from SSS. Sodium silicate solution were synthesized by mixing milled silica sand and sodium hydroxide 8 M using magnetic stirrer on 90°C temperature for 2 hours in 400 rpm speed. Then, the mixture was cooled in room temperature and filtered by whatman paper to get SSS. hydrochloric acid was dropped to the solution gradually with different concentration of 3 M (SSS-3M) and 5 M (SSS-5M). So, the results from this synthesis were SSS control (SSS-0), SSS-3M and SSS-5M. To confirm the results, Ultraviolet-Visible (UV-Vis) and Fourier Transform Infrared (FTIR) characterization was conducted.

The SSS was settled down for 24 hours until it become gel then followed by washing process using centrifuge method in 10.000 rpm until it reach pH 7. Furthermore, the sample was dried in room temperature to gain SNPs that noted as SNP3M and SNP5M. It was characterized and analyzed by X-Ray Diffraction (XRD) to gain phase information and Scanning Electron Microscope (SEM) to gain its microstructure.

Results and Discussion

The sodium silicate solution was formed by reaction of silica sand and sodium hydroxide as follows.

$$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O \quad (1)$$

The UV-Vis characterization is showed in Figure 1 for three types of sodium silicate solution. The figure shows the absorbance of the solution raise due to increasing of acid concentration[11]. The peak of the spectra is about 260-310 nm that reflect that concentration of dissolved solid particle. It informs that there is increase of solid particle due to addition of HCl to the SSS and significant change is shown after HCl addition to SSS. The absorbance of solution in visible range (about 500 nm) is very high.

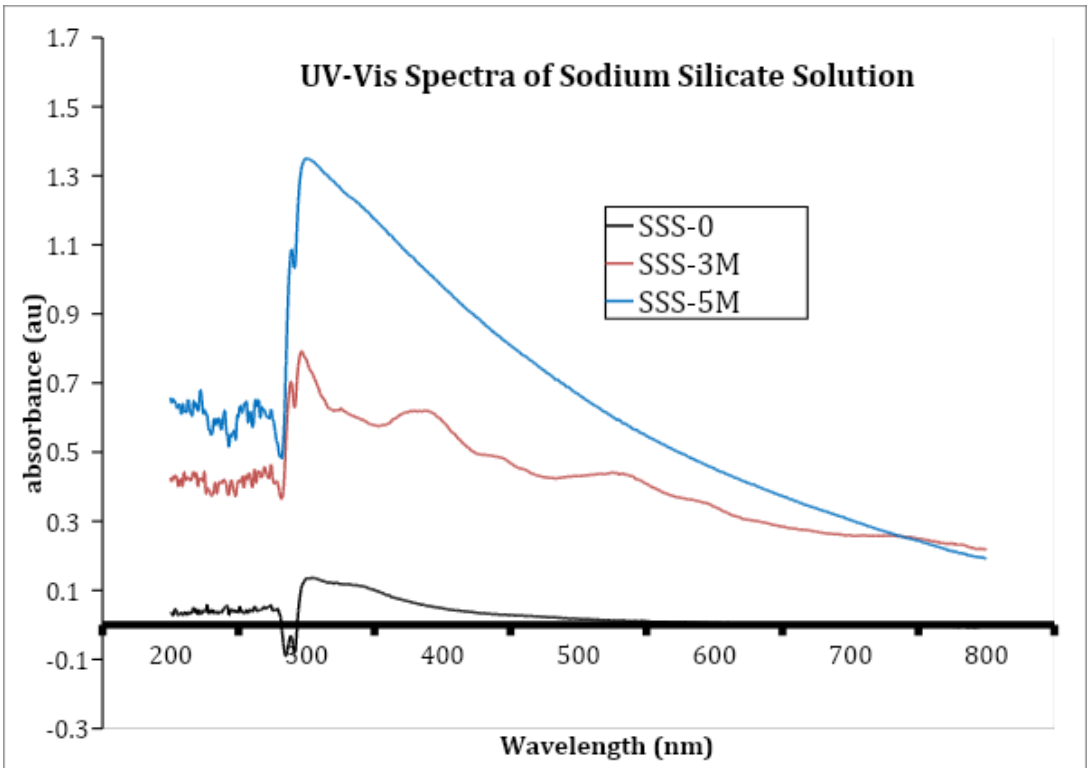

Fig. 1. UV-Vis Spectra of SSS

FTIR result confirms UV-Vis one where the addition of acids increase the peak of spectra as shown in Figure 2. The two peak intensity between 3000-3500 cm^{-1} and between 1500-200 cm^{-1} was attributed to Si-OH where the result of neutralization of SSS. The peak about 800 cm^{-1} and about 400 cm^{-1} were attributed to Si-O-Si bending vibration[8, 12]. It can be stated that formation of silica nanoparticles was on progress. It will be fully formed after washing and drying.

Silicate contain two double bonds between silicone and oxygen that change become silica hydroxide ion with charge -1 where silicone bond with hydroxide ion from sodium hydroxide cause a double bond between silicon and oxygen become single bond. Another double bond and hydrogen from the ion release become silicone trioxide ion that has -2 charges [8]. Sodium ions that positively charge attract the silicon trioxide ion to form sodium silicate. The presence of bonding of Si-OH, Si-O-Si and other bonding that appear in FTIR spectra indicate the reaction and some incomplete proses of change. It is attributed to solution may experience reverse reaction.

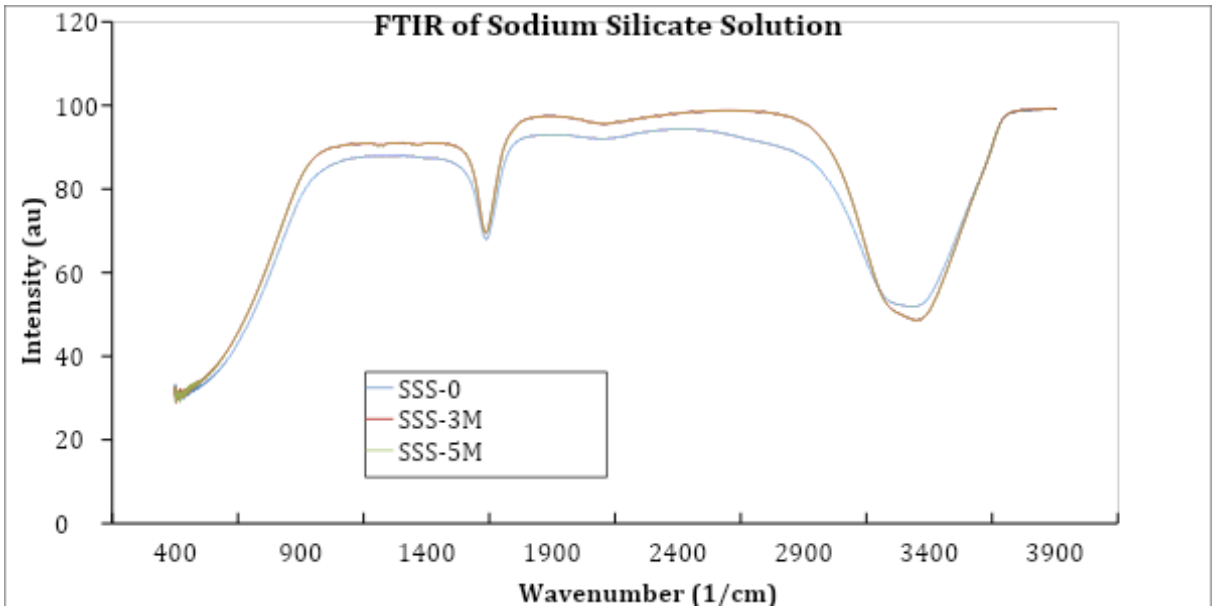

Fig. 2. FTIR Spectra of SSS

The dried silica nanoparticles were identified using XRD to find out if it was formed. The XRD spectrum shows for different acids concentration of 3 M and 5 M are shown in Figure 3. Both are amorphous as only gave a peak around 23º and SNP5M is more amorphous rather than another one. It means the crystal orientation of SNPs is random. The XRD spectrum is confirmed as the work by other researcher [6]. Quantitative analysis was applied to get crystallite size using Scherrer equation[13]. The computations result 4.8 nm for SNP3M and 2.9 nm for SNP5M where the higher hydrochloric acid concentration cause smaller crystallite size. The width of the XRD spectra shows the trend which wider peak represents the smaller crystal.

The formation of SNPs as following equation (2) where sodium silicate react with hydrochloric acid and produce silica, sodium chloride and water. The washing process using centrifuges remove the sodium chloride where the drying process removes the water.

$$Na_2SiO_3 + HCl \rightarrow SiO_2 + NaCl + H_2O \quad (2)$$

It can be confirmed from SEM image that show small SNPs were agglomerated as shown in Figure 4. Agglomeration is a characteristic of nanoparticles due to the natural state of it is unstable. It is then confirmed that silica nanoparticles was formed and the size in nanometer. The agglomeration can be avoided by change the functional group on SNPs surface. It result the similar charge that exert repulsion among the particles.

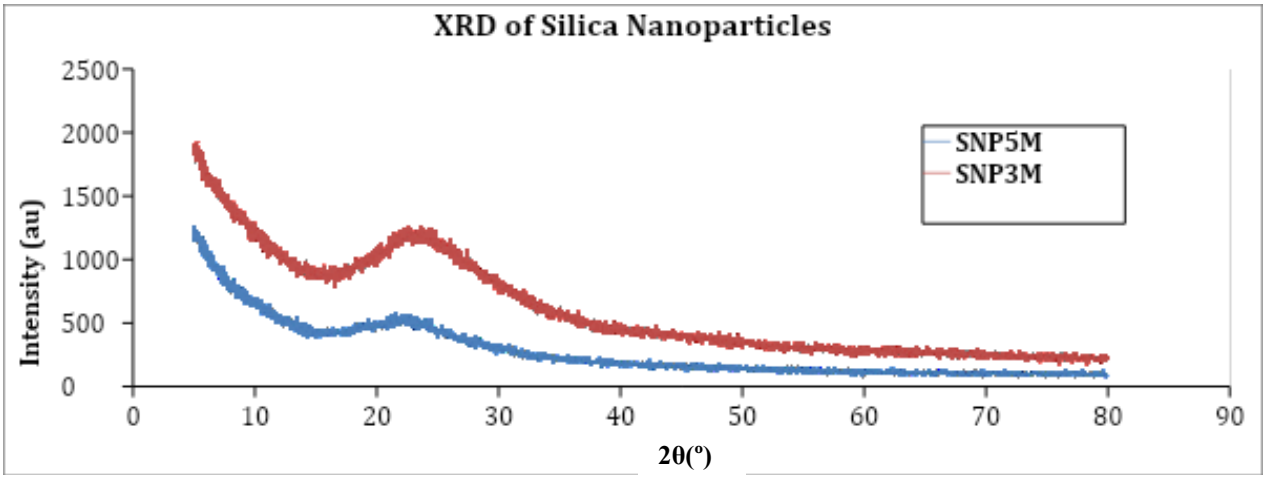

Fig. 3. XRD Spectra of SNPs

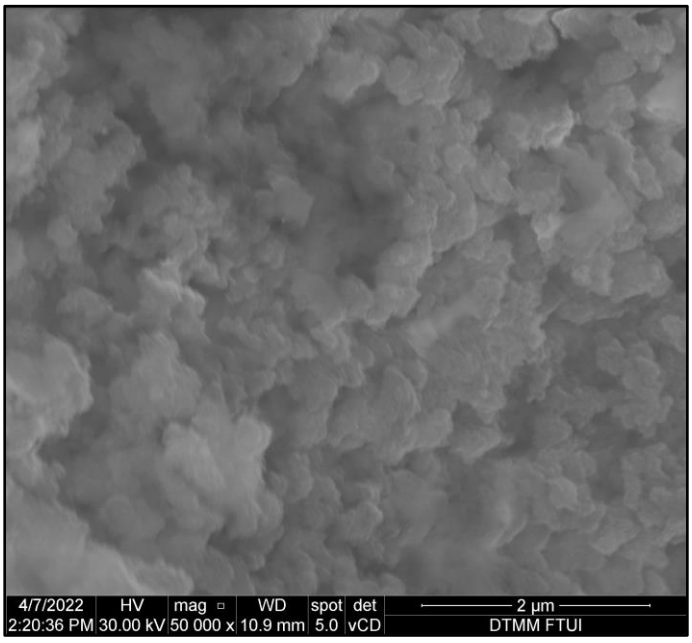

Fig. 4. SEM image of SNP3M

Quantitave analysis of the SEM image was conducted to gain the grain size of the SNPs. The data were gotten from 5 magnifications of SEM image using bar scale as standard [14] using Image-J software. The software read the image and measures the area of the particles. The diameters of the particles were gained by assumption that shape of the particles is circles. The results of image analysis were presented in Table 1.

The average area for all image of SNPs about 107 -112 nm^2 that correlates to diameters of 11.6-12 nm. The minimum area of all analyzed particles was 56 nm^2 and the maximum one is 195 nm^2. It makes the diameter of SNPs spread from 8.4 nm to 15.8 nm where this size is nanomaterial size. So, the resulted SNPs were in nanometer size that confirmed by SEM image analysis.

Table 1. Average diameter Calculation of SNPs using Image-J

Magnification of Image	Average Area (nm^2)	Average Diameter (nm)	Minimum Area(nm^2)	Maximum Area(nm^2)	Scale
5000X	109.76	11.82162309	56	195	688px: 20000 nm
10000X	107.066	11.6756441	56	184	692px: 10000 nm
25000X	106.33	11.63544416	56	172	852px: 5000 nm
50000X	109.426	11.80362277	64	168	638px: 2000 nm
100000X	111.95	11.9389768	69	160	688px: 1000 nm

Summary

The silica nanoparticles from silica sand have been successfully fabricated through sodium silicate solution. The UV-Vis and FTIR spectra show the progress of SNPs formation. XRD analysis using Scherrer equation result 4.8 nm and 2.9 nm for SNP3M and SNP5M. The quantitative SEM analysis result average diameter was 11.6-12 nm. Therefore, XRD spectra and SEM image confirmed that SNPs was successfully formed.

References

[1] Sharma, K., Hooda, A., Goyat, M.S., Rai, R., Mittal, A.: A review on challenges, recent progress and applications of silica nanoparticles based superhydrophobic coatings. Ceram. Int. 48, 5922–5938 (2022). https://doi.org/https://doi.org/10.1016/j.ceramint.2021.11.239

[2] Li, T., Shi, S., Goel, S., Shen, X., Xie, X., Chen, Z., Zhang, H., Li, S., Qin, X., Yang, H., Wu, C., Liu, Y.: Recent advancements in mesoporous silica nanoparticles towards therapeutic applications for cancer. Acta Biomater. 89, 1–13 (2019). https://doi.org/https://doi.org/10.1016/j.actbio.2019.02.031

[3] Abid, N., Khan, A.M., Shujait, S., Chaudhary, K., Ikram, M., Imran, M., Haider, J., Khan, M., Khan, Q., Maqbool, M.: Synthesis of nanomaterials using various top-down and bottom-up approaches, influencing factors, advantages, and disadvantages: A review. Adv. Colloid Interface Sci. 300, 102597 (2022). https://doi.org/10.1016/j.cis.2021.102597

[4] Chi, F., Liu, D., Wu, H., Lei, J.: Mechanically robust and self-cleaning antireflection coatings from nanoscale binding of hydrophobic silica nanoparticles. Sol. Energy Mater. Sol. Cells. 200, 109939 (2019). https://doi.org/https://doi.org/10.1016/j.solmat.2019.109939

[5] Li, X., Zheng, Y., Xu, X., Xue, C., Han, Z., Yang, H., Zhang, X.: Fabrication of single-layer antireflective coating with environmental stability by modified SiO2 mixed sol. Colloids Surfaces A Physicochem. Eng. Asp. 630, 127553 (2021). https://doi.org/https://doi.org/10.1016/j.colsurfa.2021.127553

[6] Ismail, A., Saputri, L.N.M.Z., Dwiatmoko, A.A., Susanto, B.H., Nasikin, M.: A facile approach to synthesis of silica nanoparticles from silica sand and their application as superhydrophobic material. J. Asian Ceram. Soc. 9, 665–672 (2021). https://doi.org/10.1080/21870764.2021.1911057

[7] Zulfiqar, U., Subhani, T., Husain, S.W.: Synthesis and characterization of silica nanoparticles from clay. J. Asian Ceram. Soc. 4, 91–96 (2016). https://doi.org/10.1016/j.jascer.2015.12.001

[8] Trivana, L., Sugiarti, S., Rohaeti, E.: Sintesis Dan Karakterisasi Natrium Silikat (Na2SiO3) Dari Sekam Padi. J. Sains &Teknologi Lingkung. 7, 66–75 (2015). https://doi.org/10.20885/jstl.vol7.iss2.art1

[9] Novita, L., Idris, I.: Effectiveness of silica gel from palm kernel shell ash as a moisture absorber of bottle packaging medicine. IOP Conf. Ser. Earth Environ. Sci. 1041, 12044 (2022). https://doi.org/10.1088/1755-1315/1041/1/012044

[10] Ismail, A., Akbar Alamsyah, I., Kholil, M., Susanto, B.H., Nasikin, M.: The Effect of Milling Time on the Size of Silica Particles from Silica Sand. Mater. Sci. Forum. 917, 162–166 (2018). https://doi.org/10.4028/www.scientific.net/MSF.917.162

[11] Donanta Dhaneswara Frans Wensten Situmorang,Alfina Nurul Haqoh, J.F.F.: Synthesis of Amorphous Silica from Rice Husk Ash: Comparing HCl and CH3COOH Acidification Methods and Various Alkaline Concentrations. Int. J. Technol. 11, 291–319 (2020). https://doi.org/https://doi.org/10.14716/ijtech.v11i1.3335

[12] Zahid, M.A., Cho, Y.H., Yi, J.: Improvement in optical and electrical performance of hydrophobic and antireflective silica nanoparticles coating on PMMA for lightweight PV module. Opt. Mater. (Amst). 119, 111371 (2021). https://doi.org/https://doi.org/10.1016/j.optmat.2021.111371

[13] Monshi, A., Foroughi, M.R., Monshi, M.R.: Modified Scherrer Equation to Estimate More Accurately Nano-Crystallite Size Using XRD. World J. Nano Sci. Eng. 02, 154–160 (2012). https://doi.org/10.4236/wjnse.2012.23020

[14] Kurniawan, C., Waluyo, T.B., Perdamean Sebayang: Analisis Ukuran Partikel Menggunakan Free Software Image-J. Semin. Nas. Fis. 1–9 (2011)