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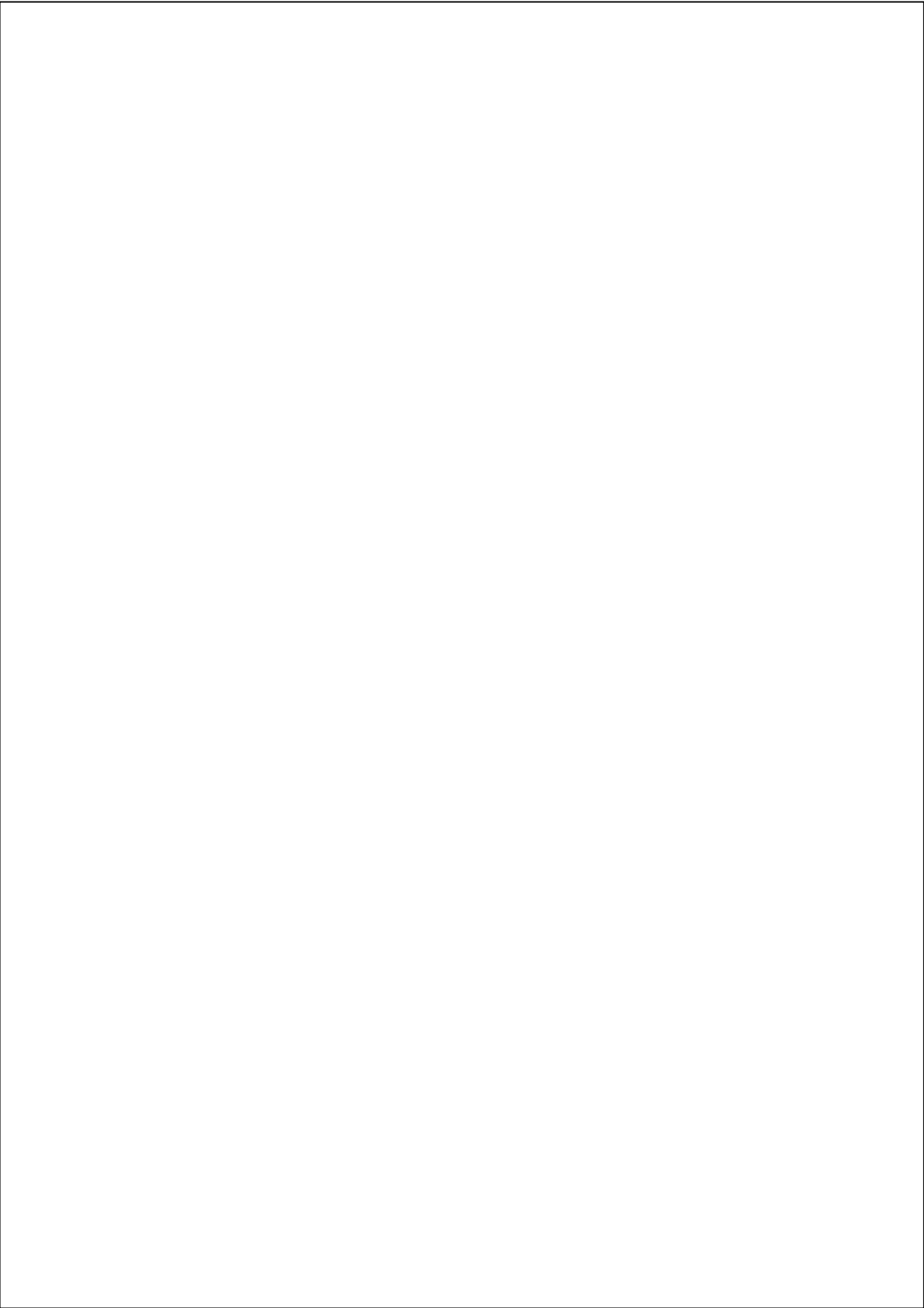
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**Synthesis And Characterization Nanosilica From Rice Husk
Ash Using Sol-Gel Method With Addition Of PEG-6000 And PVA**

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Synthesis And Characterization Nanosilica From Rice Husk Ash Using Sol-Gel Method With Addition Of PEG-6000 And PVA

ABSTRACT

Nanosilica (nS) from rice husk ash has been successfully synthesized using a sol-gel method with the addition of polyethylene glycol (PEG)-6000 and polyvinyl alcohol (PVA) as a template. This research aims to know the characteristic of functional groups of nS and the influence of PEG-6000 and PVA concentration (5%, 10%, and 15% (b / v)) on the size, morphology, and distribution of the resulting Nano silica particles. The functional groups of nS are characterized by Fourier Transform Infrared (FTIR), the size and morphology of nS are characterized by Scanning Electron Microscopy (SEM), while the particle size distribution is characterized using Particle Size Analyzer (PSA). The results showed that the addition of PEG-6000 and PVA affected the size and morphology of nS. FTIR spectra showed the presence of silanol (Si-OH) and siloxanes (Si-O-Si) groups. Based on the results of SEM, nS-PEG morphology is round and relatively more uniform compared to the amorphous nS-Control and nS-PVA morphology. In the process, the sol-gel PVA can obtain nS with a narrow particle size distribution rather than PEG-6000.

Keywords: Rice husk ash, Nano silica, sol-gel, polyethylene glycol, polyvinyl alcohol

INTRODUCTION

Indonesia is an agrarian country where one of its main products is rice. Producing areas of rice husks include Java, Sumatra, Sulawesi, Kalimantan, Bali, and Nusa Tenggara. The island of Kalimantan is the fourth-largest producer of rice husk after Sulawesi [1]. In 2020, the rice harvested area is estimated at 292.027 hectares with a production of 1.13 million tons of GKG. If converted into rice, rice production in 2020 is estimated to reach 667,771 tons [2]. Previously, the rice husk waste is still

underutilized. Most rice husk is burned to be mixed on ornamental plant soil. Luh [3] stated that the silica content in rice by-products in the husk was 18-22%.

Rice husk ash has a high enough silica content. Previous researchers that have been done by Nuryono and Narsito [4] and Mujiyanti et.al [5] showed that the silica content in rice husk ash is more than 90% so it can be utilized as the manufacture of material in the form of Nano-silica. A material is said to be nano if it has a size between 1-1000 nm (1 nm = 10⁻⁹m) [6]. Nano-silica has been applied to areas such as ceramics,

rubber, electronics, catalysts, pharmaceuticals, and cosmetics [7]

Mujiyanti, et al [8] has performed nano-silica synthesis using rice husk ash as a source of silica by sol-gel method. The results of this study indicate that the size of silica has not been uniform so it is necessary to add a substance that can form and control the size of the particle. The substances that can be used to form and control the size and structure of the silica pore are polyethylene glycol (PEG) and polyvinyl alcohol (PVA) which can serve as a template and a silica particle wrapper so as not to form aggregates. Templates trapped on the surface of the particles will mask the negative ions of silanol, resulting in particles of uniform spheres [9].

Several parameters that need to be considered in the synthesis of nano-silica using the sol-gel method are the effect of precursor concentration, catalyst concentration, type of solvent used, and aging time [10]. Silica precursors can be used from natural or synthetic materials such as rice husks, quartz sand, sugarcane waste (bagasse), mud, corn cobs, tetramethyl orthosilicate (TMOS), tetraethyl orthosilicate (TEOS), orthosilicic acid, sodium metasilicate. Several researchers reported [8], [11]–[14] that have successfully synthesized NS using the sol-gel method, the greater the concentration of precursor and catalyst concentration, the larger the particle size, due to the faster hydrolysis and condensation reactions that took place.

Based on the above background, this research will synthesize nano-silica from rice husk ash from peat area using a sol-gel

method with a raw material of precursor sodium silicate solution. The influence of PEG and PVA concentration to form the structure of particle become uniform. Characterization of nano-silica is analyzed by Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM), and Particle Size Analyzer (PSA) to determined functional groups, sizes, morphologies, and the distribution of nano-silica.

METHODS

Chemicals

Rice husk ash of South Kalimantan's peatland, solid NaOH (Mr = 40.00 g / mol) (pa Merck), concentrated HCl (37%, ρ = 1.19 g / mL, Mr = (pa Merck), NH_4OH , PEG-6000 (pa Merck), PVA (pa Merck) and aqua dest

Apparatus

The apparatus used in this research are standard laboratory glass equipment, thermometer, analytical balance (Europe 600), oven (Mettler), hot plate (Stuart CB 302), Furnace (Ney Vulcan), magnetic stirrer, reflux set, spray, porcelain cup, Whatman filter paper No. 42, universal indicator pH, Fourier Transform Infrared (FTIR) Spectrophotometer, Scanning Electron Microscope (SEM) (JCM-6000), and Particle Size Analyzer (PSA).

Procedures

Preparation of rice husk ash

Rice husk ash is inserted into a porcelain dish then heated in a furnace for 1 hour at 650 ° C. The resulting husk ash is then sieved to obtain the ash powder which passes through the 170 mesh sieve.[8]

Extraction of silica from rice husk ash

A total of 10 grams of rice husk ash was dissolved with 80 mL of 3 M NaOH using hot plate stirring, the mixture was heated at 80 °C in a 250 mL cup for 1 hour with constant stirring. The solution was filtered and the residue was washed with warm water as much as 20 mL. The filtrate obtained is a solution of sodium silicate (Na₂SiO₃). The filtrate is then cooled to room temperature and left overnight.

Preparation of silica gel from sodium silicate solution

The resulting sodium silicate solution was then added with 5 M H₂SO₄ solution dropwise while stirring with a magnetic stirrer until gelatin was formed up to pH 2 and a 2,4 mL NH₄OH solution was added to pH 7. The formed gel was then sterilized overnight at room temperature, then filtered, washed with warm water, and dried in an oven at 100 ° C for 15 hours [8]

Synthesis of nano-silica with PEG-6000 and PVA

The silica powder formed was refluxed with 80 mL of HCl 6 M for 4 h at 95 °C. The sample was then washed with warm aqua dest until the silica was released from the acid and dried again in an oven at 110°C for 3 hours. As much as the reflux silica powder was reconstituted in 50 mL of 3 M NaOH in a 400 mL beaker while stirring using a magnetic stirrer. After 1 hour, PEG-6000 was added with a concentration of 5% (w / v) to the silica sol solution at a 2: 1 volume ratio (PEG: silica). Stirring is carried out for 10 hours, then added with H₂SO₄ 9 M dropwise until pH 7. The nano-silica gel is washed repeatedly with warm aqua dest until the

filtrate is completely free of salt, and then the gel is dried at 50 °C for 48 hours in the oven [15]. Then the silica powder is calcined at 600 °C for 2 hours to remove the template. The same way is done for the manufacture of nano-silica with PEG-6000 concentration of 10%, 15%, and addition of PVA (5%, 10%, and 15% (w / v)).

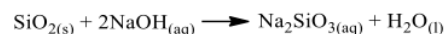
RESULTS AND DISCUSSION

Preparation of rice husk ash

The peatland husk ash is inserted into a porcelain dish to be consumed in a furnace for 1 hour at 650 °C. Burning is carried out at temperatures < 800 °C to prevent the transformation of amorphous silica into crystalline. In addition, combustion at high temperatures serves to remove the organic components contained in the husk, leaving only inorganic components and the expected rice husk ash is SiO₂.

Extraction of silica from rice husk ash

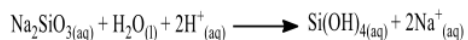
The extraction process in this method is based on large amorphous silica solubility in alkaline or alkaline solutions such as potassium hydroxide (KOH), sodium hydroxide (NaOH), or sodium carbonate (Na₂CO₃). The extraction of rice husk ash was conducted using a 3 M NaOH solution at 80 ° C while stirring with a magnetic stirrer for 1 hour. The extraction of silica from rice husk ash with NaOH solution will produce sodium silicate (Na₂SiO₃).



Silica gel from sodium silicate solution.

The silica compounds readily dissolve in an alkaline atmosphere and will settle in an acidic atmosphere. According to Nuryono & Narsito [4], the gel formation process depends on the pH or the concentration of

protons in the solution. Reactions that occur in the acidification process



The addition of acids to the precursors leads to protonation resulting in higher concentrations of the proton (H^+) in sodium silicate solution and partly siloxy (Si-O-) groups forming silanol (Si-OH) groups. The silanol group formed is then further attacked by the siloxy (Si-O-) group with the aid of an acid catalyst to form a siloxane (Si-O-Si) bond [5]

Synthesis of Nanosilica

This study used polyethylene glycol (PEG) which has a molecular weight of 6000 grams/mol and PVA. Based on the results of the study, the higher the template concentration, the less H_2SO_4 9 M volume required for the condensation and gel (aggregate) stages because PEG and PVA have solubility at the acidic pH so that condensation reactions take place rapidly.

Samples without the addition of PEG and 5% PEG addition formed white gel overall, while samples with 10% PEG and 15% yielded the product in the form of a solution with a small gel dispersed throughout the solution and when allowed to form 2 phases. This is probably because the acid solution is too saturated with the template present in the precursor solution. This is also influenced by the concentration of PEG, where the higher the concentration of PEG the more gel is trapped in the PEG template so that no further aggregate is formed and produces gel with a small size. The interaction between PEG and silica particles is presented in. Figure 1. The Si-OH group on the silica surface will interact with the hydroxyl group from the end of the PEG chain through the hydrogen bond. The $-\text{OH}$ group of PEG is more polar compared to $-\text{OH}$ silanol so that $-\text{OH}$ of PEG replaces $-\text{OH}$ silanol in a silicate solution thereby PEG will cover the silica surface and prevent further agglomeration.

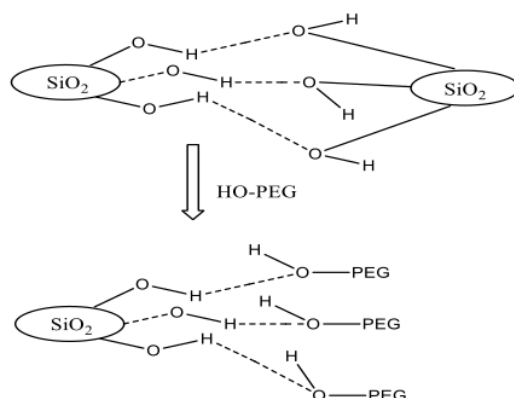


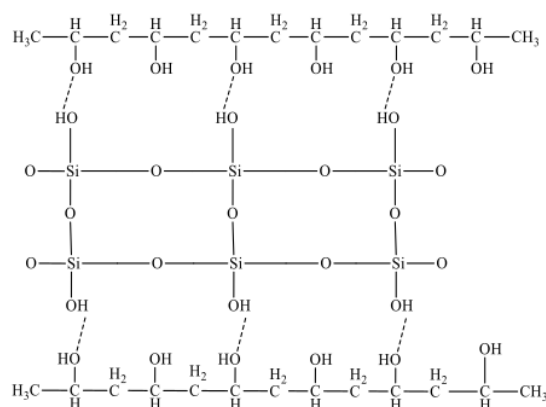
Figure 1. The interaction between SiO_2 and PEG [16]

While the process of synthesis of nS with the addition of PVA produces a clear

yellow solution and there is a swelling blob (swelling). However, gel formation occurs

thoroughly such as in the nS-Control sample. PVA has the very nature of swelling when it interacts with water. This is because the PVA chain is longer than the PEG and PVC hydroxyl groups (-OH) more so that the -OH group interactions on the PVA are stronger. The hydroxyl group present in the polymer chain causes PVA to be polar [17] The

interaction between the PVA and the silica particle is presented in Figure 2. The silanol group (Si-OH) on the silica surface will interact with the -OH group of PVA through the hydrogen bond. Since the hydroxyl-OH group of PVA is more polar than the -OH group of silanol then -OH of PVA replaces -OH silanol in silicate solution.



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Figure 2. The interaction between SiO₂ and PVA [17]

The removal of PEG and PVA can be done by the calcination method. In this study, calcination was carried out at a temperature of 600oC for 2 hours. From the results of the study, the removal of the template by calcination method leads to the carbonization of silica powder, where the silica powder with the addition of PEG before calcination is white after calcination is grayish-white which only occurs in the sample. While the silica powder with the addition of PVA changed color from white to gray. This is likely PVA is still trapped in the pore in greater quantities than the PEG template [13]

The characterization of Nanosilica

Identify functional groups on nano-silica using FTIR

The identification of functional groups present in nano-silica was performed using the FTIR spectrophotometer method. Each functional group present in nano-silica (nS) has an absorption at a certain wavelength so that it can be qualitatively identified. Characterization with FTIR was performed in the range of 500-4000 cm⁻¹ as shown in Figures 3 and 4.

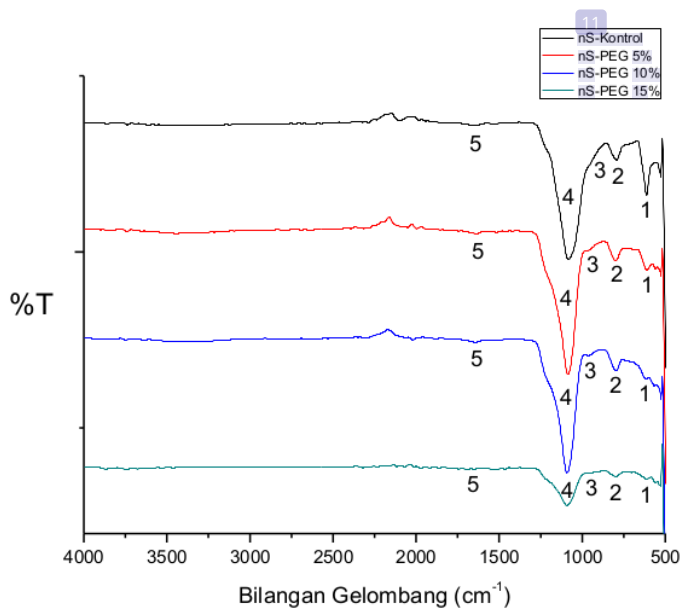


Figure 3. The FTIR spectra for the various samples of nS-PEG

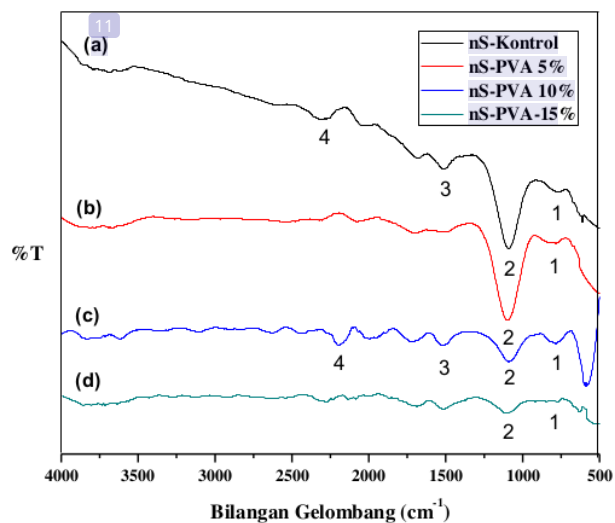


Figure 4. The FTIR spectra for the various samples of nS-PVA

The spectra show several peaks depicting the presence of functional groups in the sample nS. In the wavelength number

602-670 cm^{-1} indicates the presence of asymmetric siloxane (Si-O-Si) asphalt vibration of each nS-PEG sample of 608

cm⁻¹, 605 cm⁻¹, 619 cm⁻¹, 612 cm⁻¹ [18]. The symmetric Si-O Si vibration of Si-O-Si is at 720-808 cm⁻¹, the absorption result of each of the ns-PEG samples is 794 cm⁻¹, 797 cm⁻¹, 796 cm⁻¹, 795 cm⁻¹, while Ns-PVA is 781 cm⁻¹, 792 cm⁻¹, 787 cm⁻¹ and 776 cm⁻¹ [19]. In the 5% nS, PEG sample, nS-PEG 10% and nS-PEG 15% appeared 950 cm⁻¹, 956 cm⁻¹ and 953 cm⁻¹ uptake showing CH of CH₂ PEG [20].

The appearance of absorption at wavenumbers 1083 cm⁻¹, 1085 cm⁻¹, 1090 cm⁻¹, and 1092 cm⁻¹ for samples ns-PEG and 1090 cm⁻¹, 1095 cm⁻¹, 1090 cm⁻¹, and 1089 cm⁻¹ for the nS-PVA samples from each sample showed the presence of asymmetric Si-O straining of Si-O-Si [21]. The presence of Si-O-Si groups is due to the reaction of

Based on the FTIR spectra of Figs 4 and 5, some changes occur in the nS-PEG spectra and the ns-PVA spectra. Changes that occur include the shift of wavenumbers, emerging, and the loss of absorption at certain wavenumbers. The generally emerging silica-absorption patterns are the silanol (Si-OH) and siloxane (Si-O-Si) groups. The FTIR spectra data above shows that the smaller the concentration of the sharper the peak template. But basically, all nS gives

condensation wherein anionic silicate species will replace -OH in silanol (Si-OH) to form siloxane (Si-O-Si) [17], [22]. For bending -OH vibrations from Si-OH on nS-PEG appeared at wavenumbers 1641 cm⁻¹, 1637 cm⁻¹, 1641 cm⁻¹, and 1660 cm⁻¹, whereas in nS- PVAs appear 1505 cm⁻¹ and 1525 cm⁻¹. In the nS-PVA samples appear absorption at 2304 cm⁻¹ and 2440 cm⁻¹ regions indicating the presence of Si-O bending of Si-O-Si [18]. According to Dominic et al. [23], wave number 3450-3640 cm⁻¹ is a typical absorption for the vibration of the -OH (hydroxyl) group of Si-OH. But in the sample nS, there is no absorption in the area. No absorption may be due to the intensity at which the absorption is so weak that it is not readable on the IR spectrum [13].

similar characteristic absorption even in different wave numbers, so it can be concluded that nano-silica has been successfully made from rice husk ash *The size and morphology of nano-silica particles using SEM*

The characterization results using SEM show the nS morphological form with the addition of PEG and PVA at each concentration of 5%, 10% and 15% (w / v) with 20,000x magnification

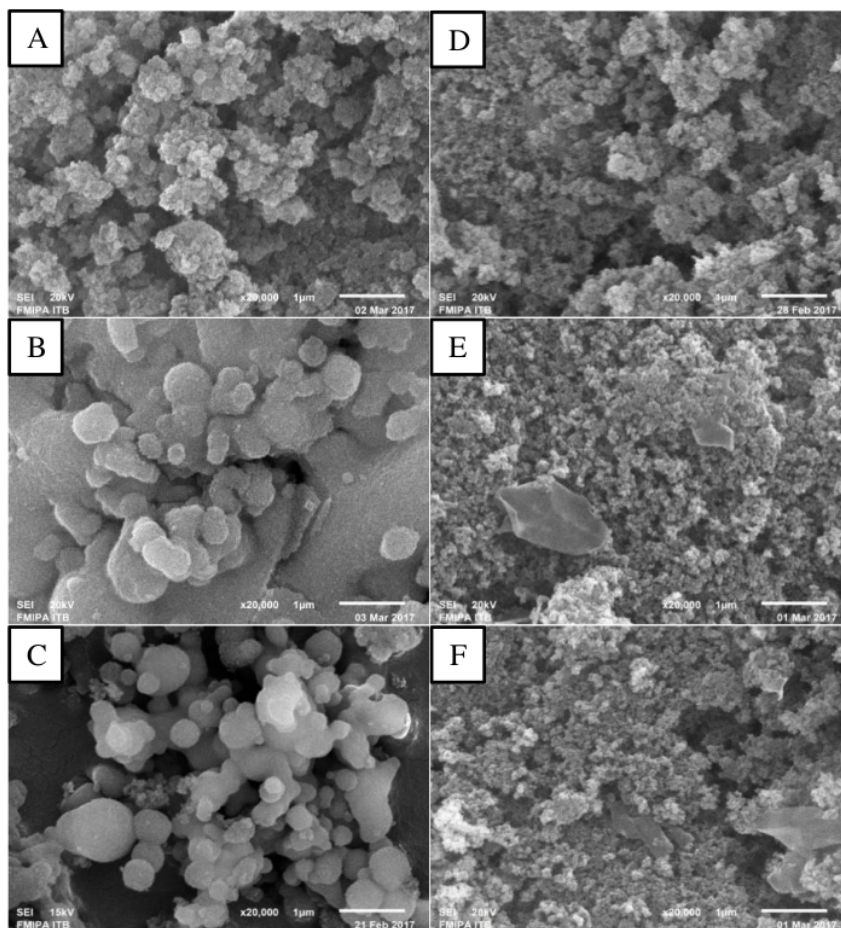


Figure 5. The morphology SEM of various samples (A) nS-PEG 5%, (B) nS-PEG 10%, (C) nS-PEG 15%, (D) nS-PVA 5%, (E) nS-PVA 10% dan (F) nS-PVA 15%

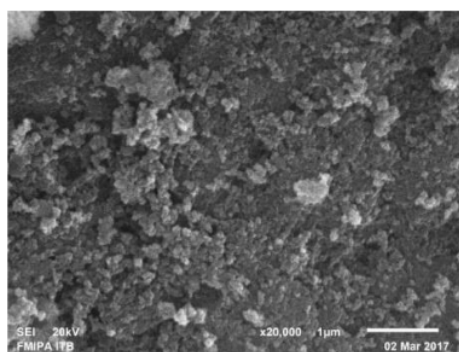


Figure 6. The morphology SEM of nS-Control

Table 1. Effect of PEG-6000 and PVA concentration on nS particle size

No.	Sample	The average of Particle Size (nm)
1.	nS-Control	82.68
2.	nS-PEG 5%	106.49
3.	nS-PEG 10%	170.23
4.	nS-PEG 15%	97.19
5.	nS-PVA 5%	120.30
6.	nS-PVA 10%	131.60
7.	nS-PVA 15%	100.22

Particle measurements from SEM results are done using Image-J software through Threshold and Outline processes, then grouped into groups. The measurement results are presented in Table 1. From the data, the particle size is still relatively uniform. Figures 5 and 6 show SEM images of nS. From the figure, it can be seen that nS-Control has a uniform particle shape and looks a lot of agglomeration. While nS with the addition of PEG has a rounded morphology and its form is relatively more uniform. Preparation of nS by the addition of PVA produces clumps (clusters) that are stacked with a less uniform and morphological size. shaped amorphous.

Variations of PEG and PVA concentrations affect the size of the resulting particles. These results are inversely proportional to the theory that increasing the number of templates can decrease the particle size. Based on the data in Table 1, the nS particles synthesized with the addition of PEG-6000 and PVA cause the particle size

to increase and agglomeration decreases as the template concentration increases. This is because during the gel-forming process most of the templates are absorbed and dispersed into silica gel tissue thereby promoting particle growth and producing particles of uniform spheres. Research conducted by [20] also showed similar results, where the particle size increased with the addition of PEG. From the data, it can be concluded that the addition of templates affect the distribution of particle size distribution and the uniformity of nano-silica particle shape

The distribution of nano-silica particle size using PSA

Particle Size Analyzer (PSA) is an instrument used to analyze the size distribution of a particle measuring 0.1 nm-10 µm based on the Dynamic Light Scattering method that utilizes infrared scattering fired into the sample to produce Brownian motion (random motion of very small particles in fluids from collisions with molecules present in liquids).

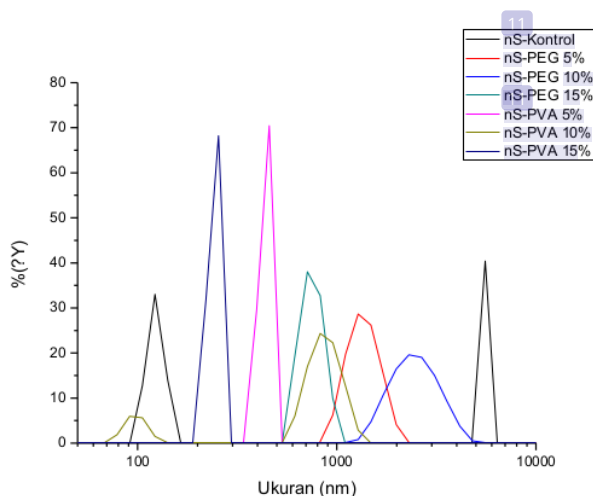


Figure 7. The PSA characterization for nS with variations concentration in PVA and PEG .

Figures 7 show that the size distribution of nS-PEG and PVA of each concentration (5%, 10%, 15% (w / v)) has size > 100 nm. From the picture, there is a shift in particle size, the wider the curve and the larger the particle size as the template concentration increases. The PSA results illustrate the size and uniformity of the particles. nS-Control has 2 peaks on the curve indicating uneven size distribution. The nS sample with the addition of PEG has a better size distribution when compared to nS-Control. While nS-PVA has a narrow size distribution compared to nS-Control and nS-PEG. This indicates that nS-PVA has a better size distribution when compared to nS-Control and nS-PEG.

Based on these data the use of PEG-6000 and PVA as a template affects the uniformity of nS particles, but see the average particle size distribution generated in the micrometer range. The use of templates in a sol-gel process is capable of

obtaining nS with a better particle size distribution than without adding templates.

CONCLUSION

The generally emerging silica-absorption patterns are the silanol (Si-OH) and siloxane (Si-O-Si) groups. The use of PEG-6000 and PVA influences the particle size distribution and the uniformity of nano-silica particle shape. Based on the results of SEM, nS-PEG morphology is round and relatively more uniform compared to the amorphous nS-Control and nS-PVA morphology. In the process, the sol-gel PVA can obtain nS with a narrow particle size distribution rather than PEG-6000.

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