



SYNTHESIS AND CHARACTERIZATION NANOSILICA FROM RICE HUSK ASH USING SOL-GEL METHOD WITH ADDITION OF PEG-6000 AND PVA

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ABSTRACT

Nano-silica (NS) from rice husk ash was successfully synthesized using a sol-gel process with the addition of *polyethylene glycol* (PEG) 6000 and polyvinyl alcohol (PVA) as a template. The purpose of this study is to investigate the properties of NS functional groups and the effect of PEG6000 and PVA concentrations (5%, 10%, and 15% (b / v)) on the size, morphology, and distribution of nanosilica. 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). In contrast the size analyzer characterizes the particle size distribution of particulate matter (PSA). The results indicated that the addition of PEG-6000 and PVA affected the size and morphology of NS. The FTIR spectra showed the presence of silanol (Si-OH) and siloxane (Si-O-Si) groups. According to the SEM results, the morphology of NS -PEG is round and relatively more uniform than the amorphous morphology of NS-Control and NS-PVA. Instead of PEG-6000, Sol-gel PVA can be obtained with a narrow particle size distribution.

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

INTRODUCTION

Indonesia is an agricultural country whose main product is rice. Rice husk production areas include Java, Sumatra, Sulawesi, Kalimantan, Bali, and Nusa Tenggara. The island of Kalimantan is the fourth-largest producer of rice husks after Sulawesi [1]. In 2020, the rice harvest area is estimated at 292,027 hectares with 1.13 million tons of GKG. Converted into rice, rice production is estimated at 667,771 tons in 2020 [2]. Previously, the rice husk waste

was 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 [4] [5] showed that the silica content in rice husk ash is more than 90% so it can be utilized to manufacture the material in the form of nano-silica. A material is called 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]

Nanosilica has been synthesized using rice husk ash as a source of silicon dioxide by the sol-gel method. This study shows that the size of the silica was not uniform, so it is necessary to add a substance that can make the particles and control the size [8]. Substances that can be used to form and control the size and structure of silica pores are polyethylene glycol (PEG) and polyvinyl alcohol (PVA), which can serve as a template and coating for the silica particles to prevent the formation of aggregates. Templates trapped on the surface of the particles mask negative silanol ions, leading to particles with uniform spheres [9].

Jafari and Allahverdi developed a method for producing silica fume become NS with an acid-based precipitation technique [10]. Furthermore, the other methods developed for the production of mesoporous NS from Pumice Rock [11] and also using extraction amorphous nano silica from the solution of olivine in acid [12]. The nanosilica can be made by the ball milling method [13]. Its powder can also be made from Na_2SiO_3 , an inexpensive raw material [8], [14]–[16]. Several researchers reported [8], [17]–[20] 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 determine functional groups, sizes, morphologies, and the distribution of nano-silica.

METHODS

1. Chemicals

The main materials used in this study were rice husk ash from peat's land area, Banjar Regency, South Kalimantan, Indonesia. Rice husk ash sample was prepared in advance by heated in a furnace for 1 hour at 650° into a porcelain dish C.[21] The resulting husk ash is then sieved, passing through the 170 mesh sieve to obtain the ash powder[8].

Merck's pro analyst chemicals used in this study include sodium hydroxide (NaOH), hydrogen chloride (HCl), ammonium hydroxide (NH_4OH), polyethylene glycol (PEG)-6000 and polyvinyl chloride (PVA).

2. Instruments

The preparation equipment used in this study included standard laboratory glass equipment, thermometer, Europe 600 analytical balance, spatula, mortar, and pestle grinder, mesh Fischer sieve, Stuart CB 302 hot plate, DLAB MS H280 Pro magnetic stirrer Memmert oven, Ney Vulcan 3-550 furnace, reflux set, spray, porcelain cup, Whatman filter paper No. 42, and universal indicator pH.

Several analytical instruments used included the Fourier Transform Infrared (FTIR) Spectrophotometer, Scanning Electron Microscope (SEM) (JCM-6000), and Particle Size Analyzer (PSA).

3. Procedures

a. 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, and 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 (Na_2SiO_3). The filtrate is then cooled to room temperature and left overnight.

b. Preparation of silica gel from sodium silicate solution

Then, the resulting sodium silicate solution was added dropwise with a 5M H_2SO_4 solution while stirring with a magnetic stirrer until gelatin formed to pH 2 and a 2.4 ml NH_4OH solution was added to pH 7. The gel formed was then kept overnight and sterilized at room temperature, then filtered, washed with warm water and dried in the oven at 100 °C for 15 hours [8]

c. Synthesis of nano-silica with PEG-6000 and PVA

The formed silicon dioxide powder was refluxed with 80 ml of 6M HCl for 4 h at 95 °C. The sample was then washed with warm aquadest until the silica was freed from the acid and dried again in an oven. at 110 °C for 3 hours. Refluxed silica powder was reconstituted in 50 ml 3M NaOH in a 400 ml

beaker with stirring using a magnetic stirrer. After 1 hour, PEG-6000 at a concentration of 5% (w / v) was added to the silica sol solution in a volume ratio of 2: 1 (PEG:silica). The mixture is stirred for 10 hours, then 9M H_2SO_4 is added dropwise at pH 7. The nanosilica gel is repeatedly washed with warm distilled water until the filtrate is entirely free of salt, and then the gel is dried in the oven at 50 °C for 48 hours [15]. The silica powder is then calcined at 600 °C for 2 hours to remove the template. The exact route is carried out for the production of nanosilica with a PEG-6000 concentration of 10%, 15% (w / v)).

RESULTS AND DISCUSSION

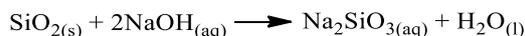
1. Preparation of rice husk ash

The ashes of the peat are placed in a porcelain plate, which is consumed in the oven at 650 °C for 1 hour. The fire takes place at temperatures of 800 °C to prevent the amorphous silicon dioxide from turning crystalline. In addition, high-temperature combustion serves to remove the organic components contained in the husk, so that only inorganic components remain and the expected rice husk ash is SiO_2 .

2. Extraction of silica from rice husk ash

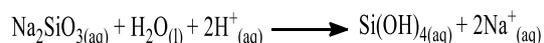
The extraction process in this process is based on the high solubility of amorphous silicon dioxide in alkaline or alkaline solutions such as potassium hydroxide (KOH), sodium hydroxide (NaOH) or sodium carbonate (Na_2CO_3). The rice husk ash was extracted with a 3M NaOH solution at 80 °C while stirring with a magnetic stirrer for 1 hour.

Extraction of silica from rice husk ash with a NaOH solution produces sodium silicate (Na_2SiO_3).



3. Silica gel from sodium silicate solution.

Silica compounds dissolve easily in an alkaline atmosphere and settle in an acid atmosphere. According to Nuryono & Narsito [4], the gel formation process depends on the pH value or the concentration of protons in the solution. Reactions that occur when acidifying



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]

4. Synthesis of Nanosilica

For the synthesis of nanosilica, polyethylene glycol (PEG) with a molecular weight of 6000 grams / mol and PVA were used. According to the results of the study, the higher the concentration of the template, the less volume of H_2SO_4 9 M is required for the condensation and gel (aggregate) stages, since PEG and PVA are soluble at acidic pH, therefore condensation reactions expire rapidly. Samples without the addition of PEG and with the addition of 5% PEG formed an overall white gel, while samples with 10% PEG and 15% gave the product as a solution with a small gel that was dispersed in the solution

and caused 2 phases to form. The phenomenon of nanosilica formation is probably due to the fact that the acidic solution is too saturated with the template present in the precursor solution. Nanosilica gelation is also affected by the PEG concentration, the higher of PEG concentration, the more gel is trapped in the PEG template, so that no more aggregate is formed and a gel with a small size is produced. The interaction between PEG and silica particles is shown in Figure 1. The Si-OH group on the surface of silica interacts with the hydroxyl group at of the PEG chain through hydrogen bonding. The -OH group of PEG is more polar compared to OH -silanol, so that the OH of PEG replaces OH-silanol in a silicate solution and the PEG covers the surface of the silicon dioxide and prevents a more significant accumulation.

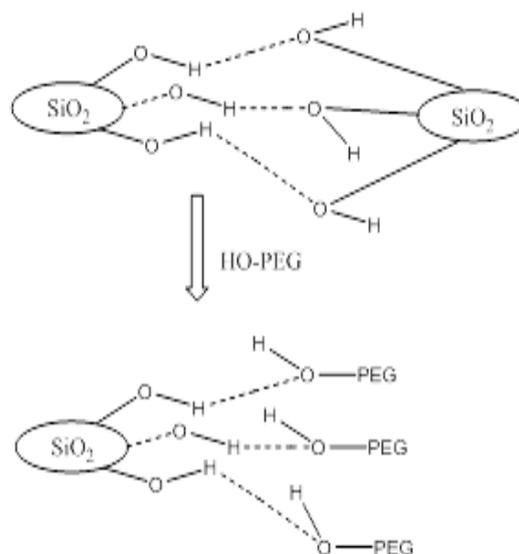


Figure 1. The interaction between SiO_2 and PEG [16]

While the nS synthesis process with the addition of PVA creates a light yellow solution and there is a bulging spot (swelling). However, gel formation takes place as thoroughly as with the nS Control. PVA has

vibration of each nS-PEG sample of 608 cm^{-1} , 605 cm^{-1} , 619 cm^{-1} , 612 cm^{-1} [23]. The symmetric Si-O Si vibration of Si-O-Si is at $720\text{--}808\text{ cm}^{-1}$, 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} [24]. 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 CH_2 PEG [25].

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 [26]. The presence of Si-O-Si groups is due to the reaction of

condensation wherein anionic silicate species will replace -OH in silanol (Si-OH) to form siloxane (Si-O-Si) [22], [27]. 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 [23]. The wave number $3450\text{--}3640\text{ cm}^{-1}$ 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 [19].

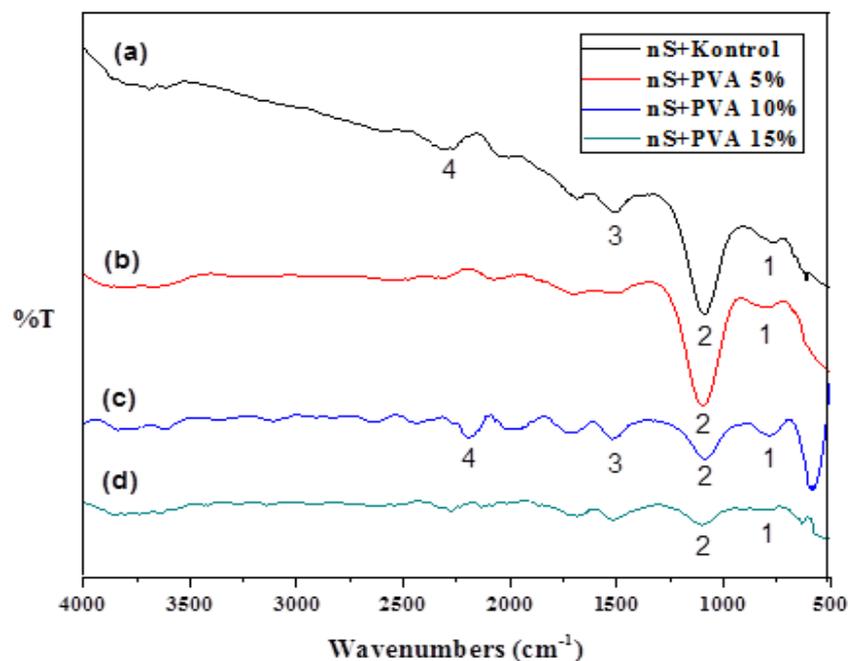


Figure 3. The FTIR spectra for the various samples of nS-PEG

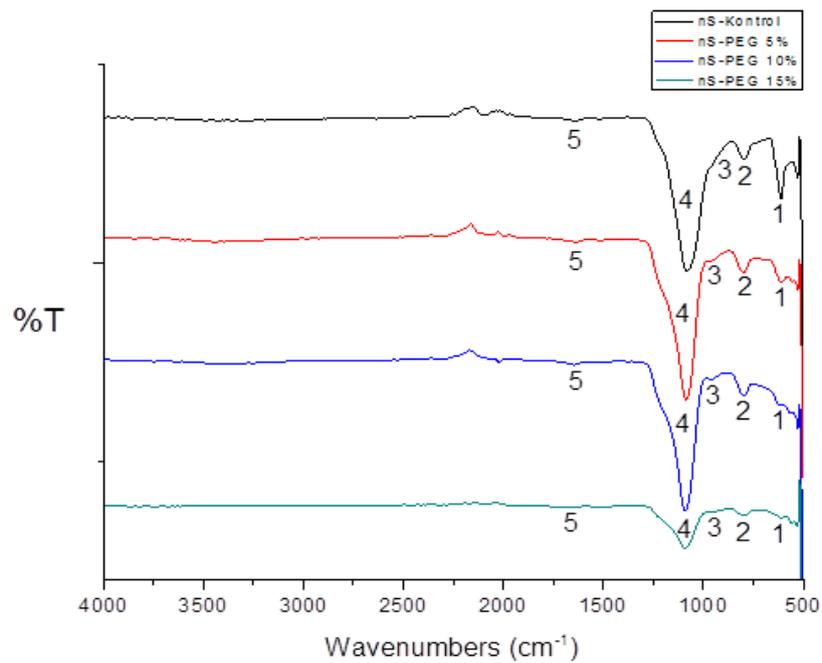


Figure 4. The FTIR spectra for the various samples of nS-PVA

Based on the FTIR spectra of Figure 3 and 4, some changes occur in the nS-PEG spectra and the ns-PVA spectra. Changes that occur include the shift of wavenumbers, emerging, and absorption loss at certain wavenumbers. The generally emerging silica-absorption patterns are the silanol (Si-OH) and siloxane (Si-O-Si) groups. In addition, the FTIR spectra data above shows that the smaller the concentration of the sharper the peak template. But basically, all

nS gives similar characteristic absorption even in different wave numbers, so it can be concluded that nano-silica has been successfully made from rice husk ash.

b. 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.

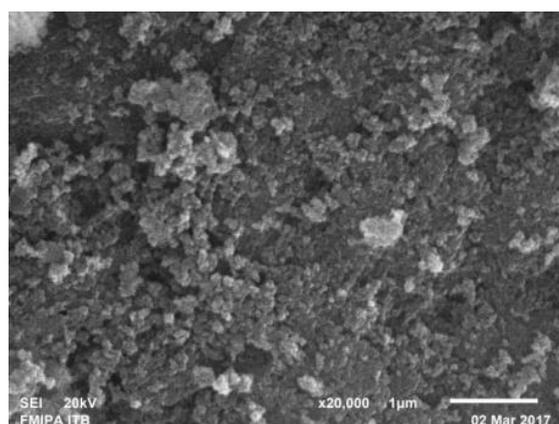


Figure 5. The morphology SEM of nS-Control

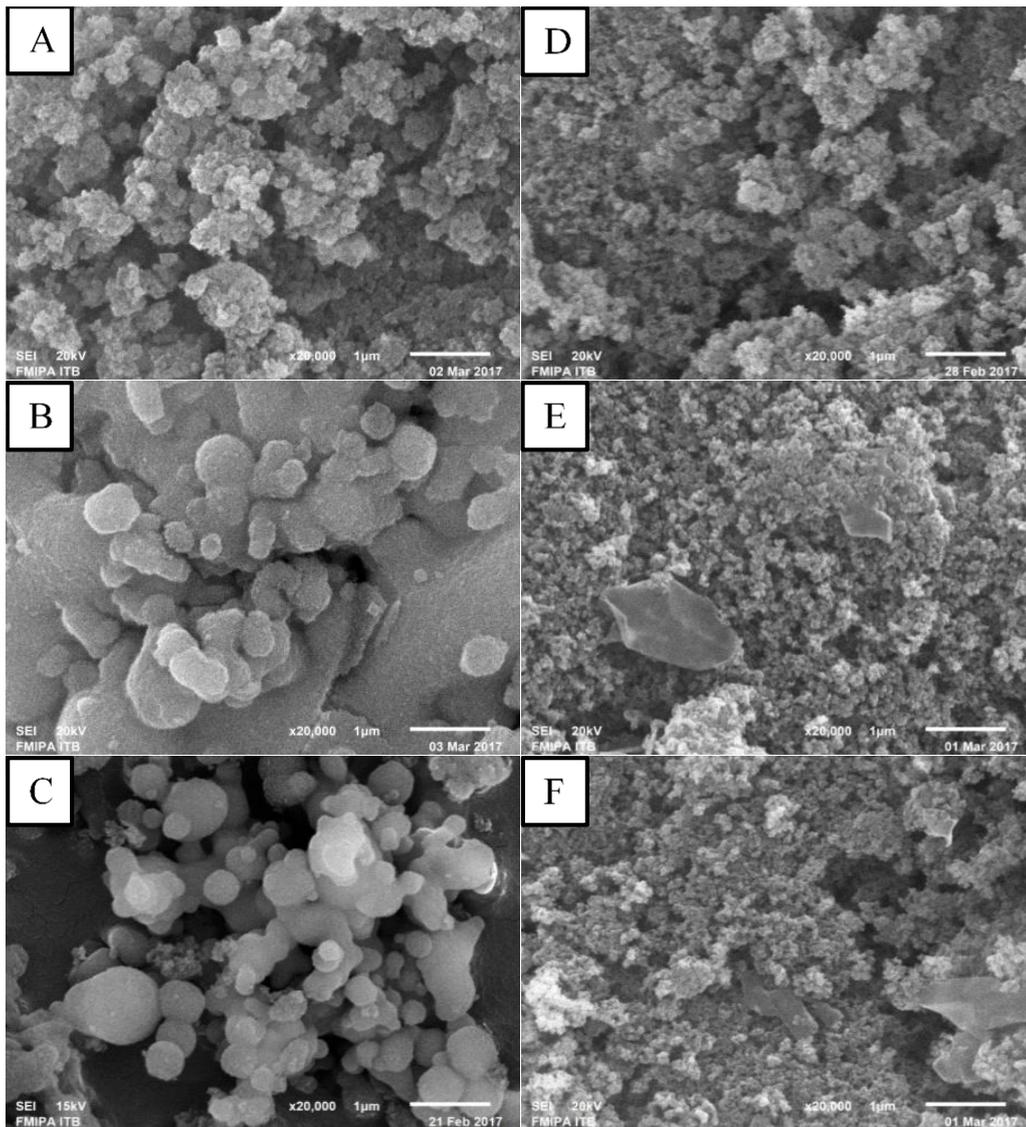


Figure 6. The morphology SEM of various samples (A) nS-PEG 5%, (B) nS-PEG 10%, (C) nS-PEG 15%, (D) nS-PVA 5%, (E) nS-PVA 10% dan (F) nS-PVA 15%

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

No.	Sample	The average 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 accumulation. While nS with the addition of PEG has a rounded morphology and its form is relatively more uniform. On the other hand, preparation of nS by the addition of PVA produces clumps (clusters) 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 most of the templates are absorbed and dispersed into silica gel tissue during the gel-forming process, thereby promoting particle growth and producing particles of uniform spheres. Research conducted by [25] also showed similar results, where the particle size increased with 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

c. The distribution of nano-silica particle size using PSA

The Particle Size Analyzer (PSA) produces a size distribution of particles with a size of 0.1 nm 10 μ m based on a dynamic light scattering method that detects Brownian motion (very small random motion) using infrared scattering applied to the sample. It is a device for analysis. Particles in a liquid) Generated by collisions with molecules present in a liquid).

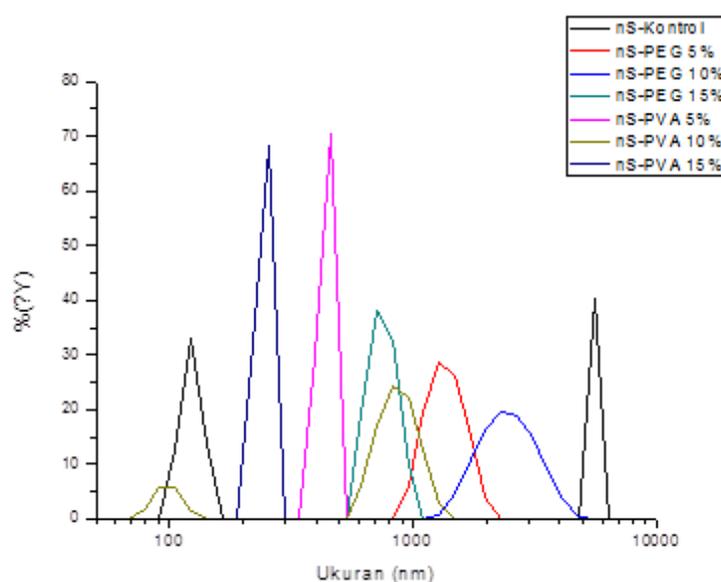


Figure 7. The PSA characterization for nS with variations concentration in PVA and PEG

Figures 7 show that the size distribution of nS-PEG and PVA of each concentration (5%, 10%, 15% (w / v)) has size > 100 nm. The picture shows 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. In contrast, 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 micrometre range. Using templates in a sol-gel process can obtain 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 than 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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