The 4th International Seminar on Sciences

PREFACE

The 4th International Seminar on Sciences (ISS) is an annual meeting organized by the Faculty of Mathematics and Natural Sciences, Bogor Agricultural University (FMIPA IPB). The seminar was held for two days from 19-20 October 2017, at the IPB International Convention Center, Baranangsiang. Bogor. The main theme of this seminar was "Sciences for Green Development" in accordance with the importance of science in maintaining the ecosystem and supporting its sustainability and IPB research expertise. The scope of the seminar comprises these topics:

- Bio-based Functional Materials
- Biophysics, Biomaterials, and Biosensors
- Bioresources, Biosciences, and
- Biotechnology Data Science and Modelling
- Environmental and Climate Change
- Information Technology for Agriculture
- Internet of Things for Sustainable Agriculture
- Life Sciences
- Nanotechnology in Life Sciences
- Renewable Energy
- Actuarial Sciences and Risk Management
- Other related topics

In this year ISS, we were also honoured to have the presence of Canadian Ambassador to Indonesia His Excellency Peter MacArthur who convey his greatest appreciation of the implementation of this activity and the cooperation that exists between FMIPA IPB and University of Waterloo. The 4th ISS this year presents no less than 10 academics / researchers / experts as speakers, both from national and international institutions namely:

- **Prof. Dr. Ir. Kudang Boro Seminar (Institut Pertanian Bogor)**
  Transparency & Trace-ability for Agro-based Products is a Must.
- **Prof. Johnny Li (University of Waterloo)**
  Hedging Crop Yield with Exchange-traded Weather Derivatives.
- **Prof. Sakakibara Masayuki (Ehime University)**
  Environmental Design for Phytotechnology for Sustainable Development in Socialecological Systems.
- **Prof. Santosh Krishna Haram (University of Mumbai)**
  Investigation of Nanomaterial for Energy Harvesting and Storage Systems through Electromechanical Prospective.
- **Dr. Kornsorn Srikunath (Kasetsart University)**
  Contribution of Chromosomics Reveals Diversity of Sex-linked Region and Evolutionary History in Reptiles.
- **Fabio Laurent Lumantau (READI)**
- **Dr. Yaya Rukayadi (Universiti Putra Malaysia)**
  Functional Food and Medicinal Properties of Piper cubeba L.
- **Gasidit Panomsuwan, PhD (Kasetsart University)**
  A Green Route towards Nanomaterials Synthesis.

- **Dr. Agus Salim (La Trobe University)**
  Current Challenges and Opportunities in Statistical Bioinformatics: How Statistics Can Contribute to Medical Advance.
- **Prof. Ali Selamat (Universiti Teknologi Malaysia)**
  Big Data Application in Agriculture.
- **Ika Dewi Ana, drg, PhD (Universitas Gajah Mada)**
  Studies on Development Calcium Phosphate Ceramics for The Application in Dentistry.

We would like to thank all the contributors and participant who made this event a big success. We are also pleased to announce that a record number of 82 papers will be published in the IOP EES Conference Proceeding and hope that you will also consider to join the upcoming ISS conferences.

ISS 2017 Chairperson

Dr. Yessie Widya Sari

ISS 2017 Proceeding Coordinator

Dr. rer. nat. Hendradi Hardhienata

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The Initial Characterization of Nanosilica From Tetraethylorthosilicate (TEOS) with The Addition Polivynil Alcohol by Fourier Transform Infra Red

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The Initial Characterization of Nanosilica From Tetraethylorthosilicate (TEOS) with The Addition Polivynil Alcohol by Fourier Transform Infra Red

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Abstract
Nanosilica (nS) are synthesized using the sol-gel method, with tetraethylorthosilicate (TEOS) as precursors, ammonia (NH_3) as base catalysts, water as hydrolysis and ethanol as solvents. Polyvinyl alcohol is added to the sol solution, serving as a capping agent. The purpose of this research is to know functional group, from TEOS with the addition of variation of PVA concentration and without addition of variation of PVA concentration. Functional groups were analyzed using Fourier Transform Infra Red (FTIR). The nanosilicas of the TEOS precursors using the sol-gel method were successfully synthesized by marked detection of typical peaks of SiO_2 in all samples of Si-O-Si (siloxane) and Si-O.

***Keywords**: nanosilica*, TEOS, PVA, sol-gel method

1. Introduction

Nanotechnology is so rapidly growing in all areas of science and technology such as electronics, aviation, defense, medicine, and health. It relates to the model, synthesis, characterization, and application of materials and equipment on a nanometer scale. The silica nanoparticles represent one of the nanomaterials that have some peculiarities among them: (1) easy in preparation by hydrolysis-condensation reactions of precursors such as tetraethylorthosilicate (TEOS) using acid or base catalysts, (2) allowing modified surfaces with variations of organosilicon compounds [1], (3) silica (SiO_2) has a good chemical, inert, biocompatible stability capable of working in harmony with the body's work system, and forming a single spike [2].

The sol-gel method has been widely used for the manufacture of nanoparticles because it has several advantages such as synthesis can be performed at low temperatures, yielding high purity and also process reaction kinetics can be controlled by varying the composition of the reaction mixture [3]. The preparation of silica gel through a sol-gel process involves the process of hydrolysis and condensation of silicon alkoxy derivatives such as tetraethyl orthosilicate (TEOS) and tetramethyl orthosilicate (TMOS) [4]. Some researchers report that it has successfully synthesized silica

nanoparticles using the sol-gel method, wherein the concentrations of precursors (TEOS) and catalyst (ammonia, NH_3) play an important role in the formation of materials on the nanoscale. Budiharti (2015) synthesizes nanosilica with TEOS as a precursor, NH3 as a catalyst and aquades as solvent, using sol-gel method [5].

The synthesis performed by Budiharti (2015) produces an uneven sample surface and consists of clusters, indicating the presence of fairly large grain sizes with uneven distribution on the surface of the silica sample [5]. This is due to the difference in TEOS concentration that affects the speed of hydrolysis and condensation processes. So that the aging time taken has a difference in each TEOS concentration. This causes the silica grain size to vary and there is a size of clumps greater than 100 nm.

The study of Ardiansyah (2015) in nanosilica with a molar ratio of NH 3 / TEOS 0.20 was performed by adding PVA with a ratio of 100 mL of a mixture of silica: PVA was 20:80, nanosilic acid yielded by particle size of 18.63 nm [1]. In general, the addition of PVA is used to increase the silica binding capacity [6], PVA is an effective stabilizer agent and can prevent the agglomeration of silica nanoparticles because PVAs that tend to negatively charged are adsorbed by silica nano particles, giving rise to repulsive forces among silica particles [7].

Based on the above background of synthesis of nanosilica from TEOS with NH3 catalyst (with various variations) still yielded nanosilica size not yet uniform, hence this research added a substance that can form and control the size of patikel in sol-gel process that is PVA with various variation to determine the ratio of mixed silica solution: PVA which yields a uniform nanosilica size and characterized by fourier transform infrared (FTIR) equipment to know the effect of variation of PVA concentration on nanosilica functional group.

2. Materials

The materials used in this study include Tetraethylorthosilicate (TEOS) (Merck), ammonia (NH_3) (Merck), ethanol (Merck), polyvinyl alcohol (PVA) (aldrich), and aquadest.

3. Methods

3.1 Synthesis of Silica Particles by Sol-gel Method

Synthesis of silica particle by sol-gel method using TEOS solution of 29 mL, 10 mL water and ethanol of 61 mL mixed in erlenmeyer. The mixture is stirred using a stirrer with heating constantly maintained at 50^{o}C for 5 hours. The addition of 0.20 M (36 mL) ammonia is carried out periodically dropwise until it runs out within 5 hours. The liquid was evaporated in the oven at 70^{o}C for 48 hours. The formed crystals are cooled in the desiccator before calcining on the furnace. The crystal is crushed first until smooth, then calcined in the furnace at 600^{o}C for 2 hours to produce a fine powder. The powder that has been formed is stored in the desiccator [1] before it is characterized by FTIR.

3.2 The making of solutions PVA 5%, 10%, and 15% (w/v)

A total of 10 grams of PVA were added to 100 mL of distilled water, then heated to 80°C while stirring until homogeneous ± 15 minutes. The 10% PVA solution is cooled prior to use [8]. The same procedure was performed to make 10% PVA solution (5 grams of PVA in 100 mL of distilled water) and 15% (15 grams of PVA in 100 mL of distilled water).

3.3 Synthesis of Nanosilica with Addition of PVA

The synthesis of nanosilica was carried out by addition of PVA at the time the solvent formed solution with the ratio between PVA and silica sol is 80:20, the mixture was stirred using a magnetic stirrer. The PVA solution used has concentration variations ie 5%, 10% and 15% w/v. The solution mixture was then evaporated in an oven at 70^{o}C for 48 hours. The formed crystals are cooled in the desiccator before calcining on the furnace. The crystal is smoothed first, then calcined at 600^{o}C for 2 hours to produce a fine powder. The powder that has been formed is stored in the desiccator [1] before it is characterized by FTIR.

4. Results and Discussion

4.1. Synthesis of Silica Particles by Sol-gel Method

The method used in synthesizing nS is the sol-gel method. Sol-gel method there are four stages, namely hydrolysis, condensation, maturation, and drying. The first step is hydrolysis, which in the process of hydrolysis of precursor metal (alkoxide) dissolved in alcohol and hydrolyzed by addition of water at acidic, alkaline or neutral conditions produce colloid sol [9]. TEOS solution is used as a precursor in synthesizing nS. Water serves as a hydrolysis, ethanol acts as a solvent, and ammonia acts as an alkaline catalyst. Meanwhile, the reaction that occurs in the process of hydrolysis are:

$Si(OC_2H_5)_4 + xH_2O + NH_3 \longrightarrow Si(OH)_x(OC_2H_5)_{4-x} + xC_2H_5OH + NH_4^+$ [10].

At the time of the hydrolysis reaction, the ethoxy group ($-OC_2H_5$) in TEOS will be replaced by hydroxyl groups (-OH). The speed of hydrolysis depends on the concentrations of TEOS, H_2O and NH_3. Increasing the concentration of NH3 in the system, then the H_2O molecule will dissociate, so the OH-ions easily attack the Si atom (Beganskiene et al., 2004). The OH-ion (from H_2O) will replace the ethoxy group in TEOS, whereas the H + ion will form a coordinate covalent bond with NH_3, so NH_3 becomes NH^{4+}.

The second stage is the condensation process. The reactions that occur in the condensation process are:

$(HO)_3$-Si-O-H + H-O-Si-$(OH)_3 \longrightarrow (HO)_3$-Si-O-Si-$(OH)_3 + H_2O$ [11].

The hydroxyl group (-OH) of the product will react to form a Si-O-Si bridge.

The next process is gel maturation that is formed. In the process of maturation the gel tissue reaction process is more rigid, strong, and shrinking in solution. The last stage is the process of evaporation of unwanted liquids and liquids such as ethanol, to obtain a sol-gel structure that has a high surface area. The gel maturation and evaporation process of the solution occurs when the sole solution mixture is evaporated in an oven at 70^{o}C for 48 hours, after which it is cooled in a desiccator prior to calcination on the furnace.

The powder that has been formed is crushed first, then calcined in the furnace at a temperature of 600^{o}C for 2 hours to produce a fine powder. The powder that has been formed is stored in the desiccator before it is characterized by FTIR.

Figure 1. The results of nanosilica synthesis powder

4.2 Result of Nanosilica Synthesis (nS) with Addition of PVA Variation Using Sol-Gel Method

The synthesis of nanosilica with the addition of PVA is carried out when the solution becomes sol. The comparison used between PVA: silica sol is 80:20 in 100 mL mixture. A total of 80 mL of the sol solution was inserted into the erlenmeyer, then 20 mL of PVA solution was added, stirring to make the solution homogeneous. The PVA solution used has concentration variations ie 5%, 10% and 15% w/v. After the silica sol and PVA solution is mixed, it is evaporated in an oven at 70^{o}C for 48 hours, after which it is cooled in a desiccator before it is calcined on the furnace. The powder is smoothed, then calcined at 600^{o}C for 2 hours. PVA solution serves as a capping agent, avoiding the occurrence of clumping between particles one with other particles Interactions that occur between silica and PVA:

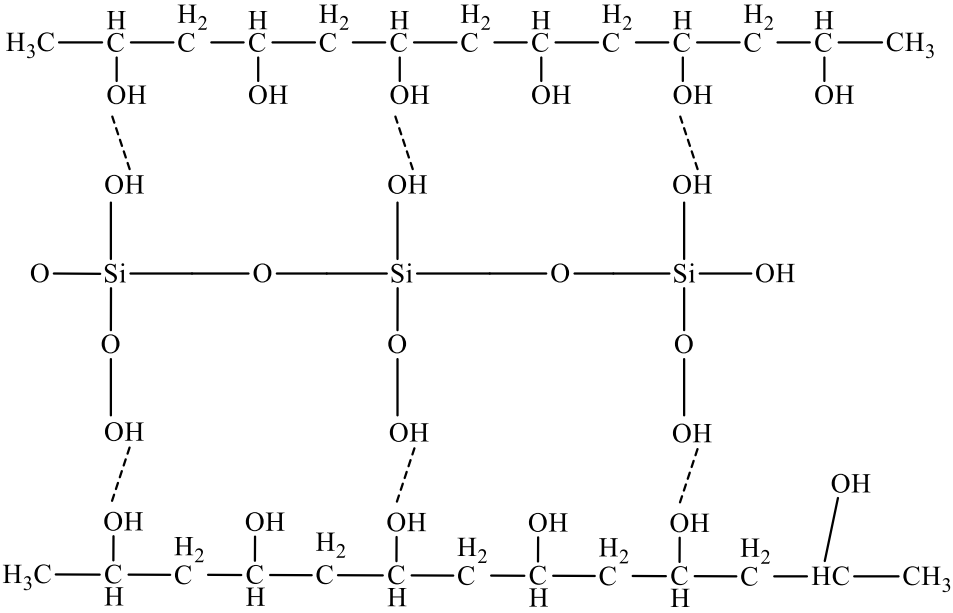

Figure 2. The interaction between silica and PVA

The calcination process is intended to remove any organic compounds that are still present in nanosilica powders, such as PVA. The powder that has been formed is stored in the desiccator before it is characterized by FTIR. Figure 3 is a powder result of the calcination process.

(a) (b) (c) (d)

Figure 3. Powder after calcination (a). nS control, (b). nS+PVA 5%, (c). nS+PVA 10%, (d). nS+PVA 15%

Based on Figure (3a) the resulting powder is white. Figure (3b) of the sample with the addition of 5% PVA produces gray powder, but the gray color intensity is still low, the image (3c) with the addition of 10% PVA of the resulting powder color is gray, while the image (3d) the addition of PVA 15% gray color increasingly thick. According to Sukmawati (2015), an increase in PVA concentration can increase the amount of PVA attached to the particle's surface [12].

4.3 Analysis the functional groups

Functional group analysis using Fourier Transform Infrared (FT-IR) spectrophotometer. Characterization was performed on nanosilica control (nS) and nanosilica (nS) samples with PVA addition, while PVA used variations of 5%, 10%, and 15% w/v. Fig.4 is a composite spectra of all four samples.

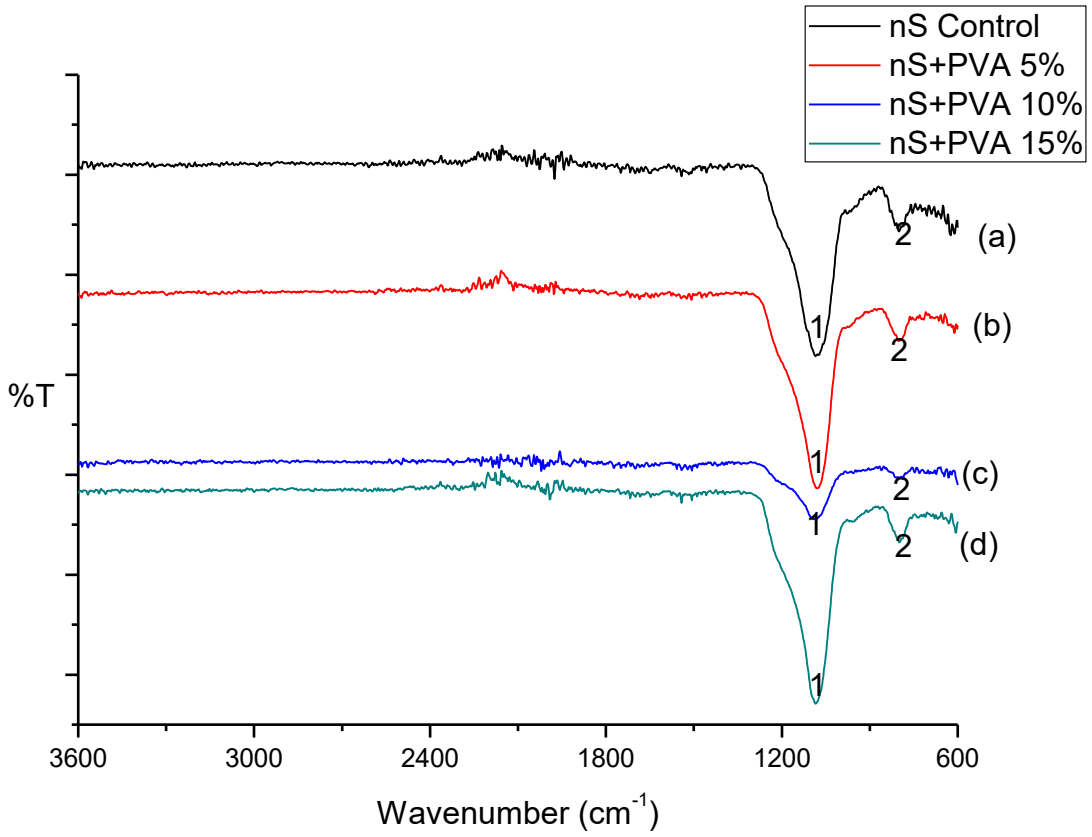

Figure 4. FT-IR spectra for (a) nS Control, (b) nS+PVA 5%s, (c) nS+PVA 10%, (d) nS+PVA 15%

The TEOS precursor detected five typical functional groups of Si-O stretching, alkyl stretching, stretching of CO vibration, CH stretching (CH_2), and CH bending (CH_3), while PVA as capping agent had three distinctive functional groups -OH stretching, alkyl stretching, and stretching of CO vibration. This corresponds to the chemical structure of TEOS and PVA. In the four samples (nS control, nS + PVA 5%, nS + PVA 10%, and nS + PVA 15%) each sample there were only two distinct functional groups namely Si-O-Si (siloxane) and Si -O stretch.

The Si-O-Si functional groups (siloxanes) are detected at wave numbers 1080-1200 cm^{-1}. The Si-O-Si group is the result of a condensation reaction. At the condensation stage, the hydroxyl group of the intermediate product, $[(OH)_xSi(OR)_{4-x}]$ will react with the ethoxy group of another TEOS (alcohol condensation) or with the hydroxyl group of the other intermediate product (water condensation) to form Si-O-Si bridge [9].

The calcination process succeeded in removing the organic compound PVA as a capping agent, the success is characterized by the absence of a typical functional group of PVA in all four samples. Typical functional groups of TEOS are also undetectable in all four samples except the Si-O functional group. TEOS functional groups are alkyl stretching, stretching of C-O vibration, C-H stretching (CH_2), and C-H bending (CH_3) at synthesis will be replaced by OH (hydrolysis process). When the hydrolysis reaction takes place, the ethoxy group ($-OC_2H_5$) in TEOS will be replaced by hydroxyl groups (-OH), then in the second stage the condensation reaction of the hydroxyl group (-OH) of the product (the hydrolysis process) will react to form Si bridge -O-Si.

The emerging functional groups are Si-O-Si (siloxane), Si-O which is the reaction product of hydrolysis and condensation. The sharpness of a spectrum varies from the four samples, the 5% nS + PVA spectrum sample is sharper than the control nS spectrum and the other PVA variations. The

typical spectrum of SiO_2 is detected in all samples. So it can be concluded that nanosilica successfully synthesized from TEOS precursors by using sol-gel method.

5. Conclusion

Based on the results of research and discussion it can be drawn some conclusions as follows:

1. Functional groups in nanosilica samples without addition of PVA and addition of PVA are the same, no detection of PVA functional groups due to the calcination process.
2. The synthesis of nanosilica with TEOS precursors using the sol-gel method was successful with the detection of typical peaks of SiO_2 in all samples of Si-O-Si (siloxane) and Si-O stretch

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