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Study on The Effect of Para-hydroxybenzoic Acid as A Reduction Agent on Stability, Shape, and Size of Silver Nanoparticles

Fadliah

Petroleum Engineering Department, Faculty of Earth Technology and Energy,
Universitas Trisakti
Jalan Kyai Tapa No.1 Grogol, Jakarta Barat 11440, Indonesia

E-mail: fadliah@trisakti.ac.id

Abstract. Research of the study on the effect of para-hydroxybenzoic acid as a reducing agent on the stability, size, and shape of silver nanoparticles has been done. In this study, pH variations were carried out to examine the effect on silver nanoparticles growth. Besides, various concentrations of AgNO₃ precursors were also performed to see the effect on the number of silver nanoparticles formed. Nanoparticles tested for their stability, shape, and size using UV/Vis spectrophotometer. The Transmission Electron Microscope (TEM) was used to characterize the shape and size of the formed silver nanoparticles. The results showed that the conditions of silver nanoparticles formation was at pH 9 with uniform morphology and the diameter of 34 ± 1.78 nm. Furthermore, from the less decreasing and small shift of the 6 weeks of age silver nanoparticles UV spectrum, it has been proven that the nanoparticles have good stability.

Keywords: Para-hydroxybenzoic, Silver Nanoparticles, Stability, Size, Shape

1. Introduction

Silver is one of the constituent elements of the earth that was abundant and has a flexible nature. This causes silver widely used in various fields such as photography, cosmetics, jewelry, health, industry, etc. Silver is generally used in bulk phase [1, 2]. In this phase, silver has a small surface area which causes the performance of silver less effective. Therefore to improve the performance of silver, the size of silver must be converted into nanoparticles size [3].

Nanoparticles are particles that have size 1-100 nm⁸. Decreasing the size of silver particles into nanoscale causes the effectiveness of silver increase due to the increased surface area of silver. The changes of silver into nano form can generally be done in two ways, chemically and physically [4, 5]. One chemical method was to reduce the size of silver by adding organic compounds containing hydroxy groups as reducing agents [6, 7].

In the process of silver nanoparticles synthesis, agglomeration often occurs and colloidal system damage due to precipitation and flocculation. This problem can be solved by using stabilizers and reducers that are suitable so that they can form a system that protects silver particles and prevents aggregation. Stabilizers were used to protect particles from the aggregation process and reduce the occurrence of collisions between silver particles so that they get stable and monodispersed silver nanoparticles [8, 9].



Previous studies used reducing agents and stabilizers with two different compounds so that they were seen less efficient. Therefore in this study used reducing agents in the form of para-hydroxy benzoic acid which has an OH group so that it is expected to reduce Ag^+ to Ag^0 and with the carboxylic group (-COOH) there can be electrostatic interactions of carboxylic groups with Ag^+ ions in solution. This interaction can prevent the aggregation of Ag^+ ions from settling.

2. Problems

The problem that often arises in the research of silver nanoparticle synthesis was the lack of stability of silver nanoparticles that characterized by the formation of aggregates or agglomeration of silver nanoparticles. Therefore, this study used benzoic acid derivative (para-hydroxybenzoic acid) which is expected to be able to maintain the stability of the formed nanoparticles. This research was carried out by a chemical reduction method using AgNO_3 as the precursor and para hydroxy benzoate acid as a reduction. Observations were made on variations in pH, variations in concentration and effect of storage time on silver nanoparticles stability and size.

3. Materials and Methods

3.1 Determination of potential reduction of para-hydroxybenzoic Acid

Measurement of potential reduction using an electrochemical analyzer with measurement speed 0,01 Vs-1 and the potential is 0.0 to -0.8 V. The data obtained from the electrochemical analyzer in the form of a voltammogram were used to determine the magnitude of the reduction potential energy of the hydroxybenzoic acid. $E_{1/2}$ is the magnitude of the potential energy of the ortho hydroxy benzoate acid reducing agent, E_{pa} was the magnitude of the potential value measured at the cathode and E_{pc} is the potential value at the anode.

3.2 Synthesis of silver nanoparticles

The synthesis of silver nanoparticles was carried out by reducing AgNO_3 with reducing agents at a pH that varied from 1 to 12 by adding a solution of HNO_3 and NaOH . The color of the solution formed was then observed visually and if a yellow color was formed indicates silver nanoparticles have been formed. All solutions obtained were analyzed using a UV / Vis spectrophotometer at a wavelength of 200-800 nm. The maximum absorption of visible light in the wavelength range of 400-500 nm indicates the formation of silver nanoparticles [10].

3.3 Optimization of AgNO_3 concentration

AgNO_3 with a concentration of 1×10^{-4} to $5 \times 10^{-4} \text{ mol L}^{-1}$ compared to the solution without the addition of AgNO_3 precursors, then in each of the test tubes was added a reducing agent which has been set at maximum pH. All solutions were heated in the water bath. The heated solution was analyzed using a UV/Vis spectrophotometer at a wavelength of 200-800 nm with fast measurement speed.

3.4 Characterization of silver nanoparticle morphology

Silver nanoparticle colloids formed were characterized using TEM to determine their shape and size and to prove the presence of nano-sized particles.

3.5 Study of the stability of silver nanoparticles

Observations were made on changes in absorbance or shifts in absorption of the maximum wavelength of silver nanoparticles during the interval of 0 hours, 1 hour, 2 hours, 3 hours, 4 hours, 5 hours, 1 day, 2 days, 3 days, and 5 days, 1 week, 2 weeks, 3 weeks, 4 weeks, 5 weeks, and 6 weeks.

4. Results and discussion

4.1 Determination of potential reduction of para-hydroxybenzoic acid

The potential reduction of a reducing agent was important because this was related to the ability of the reducing agent that we will use in reducing the precursor of AgNO_3 . The potential reduction of para hydroxy benzoate acid was measured using cyclic voltammetry. The measurement results were shown in Figure 1.

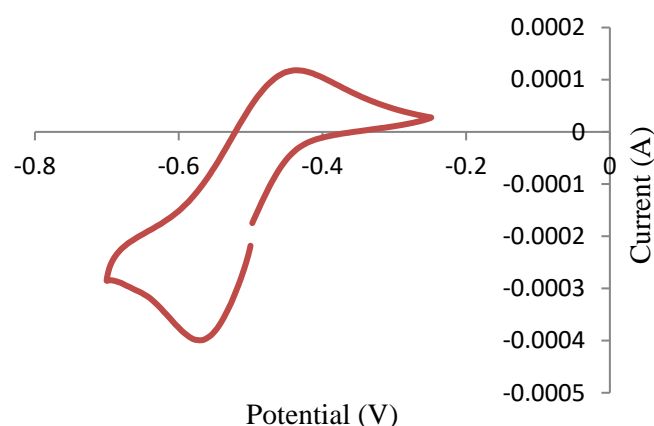


Figure 1. The cyclic voltamogram of para hydroxy benzoate acid.

Figure 1 shows that para hydroxy benzoate acid can reduce silver ions because the reduction potential possessed was lower than silver ions. Besides, the E value of the reaction between para hydroxy benzoate acid and silver ion can be calculated by calculating the potential energy difference. If the difference is positive then the reaction takes place spontaneously, while if the difference is negative then the reaction is not spontaneous[11].

4.2 Effect of pH on the system on the absorbance of silver nanoparticle colloids

PH measurement was done to see the condition of silver nanoparticles formed at acidic or basic pH. The pH variation was carried out on the precursor AgNO_3 by using NaOH to adjust the atmosphere of alkaline and HCl to regulate the acidic atmosphere. Silver nanoparticles were not formed at acidic pH but at alkaline pH and maximum in pH 9 marked by changing the color of the clear solution to yellow as seen in figure 3.

Figure 2 shows that the higher the pH of the solution, the more Ag^+ reduced to Ag^0 at pH 8-9. The pH increases, the color of the solution will change from clear to yellow. This shows in absorbance that measurement using UV/Vis spectrophotometer [12-15].

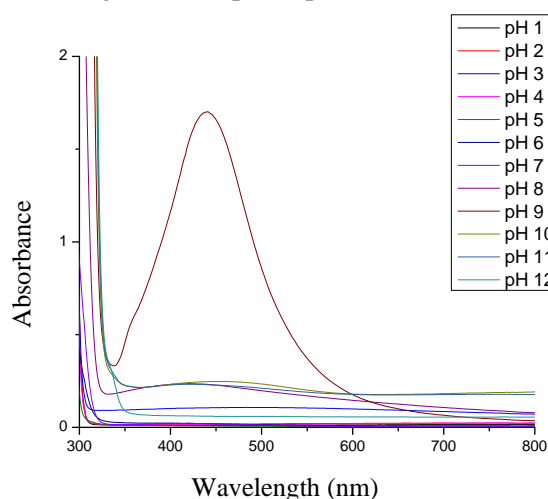


Figure 2. The spectrum of silver nanoparticles synthesized using AgNO_3 $3 \times 10^{-4} \text{ mol L}^{-1}$ and para-hydroxybenzoic acid pH 1 to pH 12.



Figure 3. Colors of silver nanoparticles synthesized using AgNO_3 $3 \times 10^{-4} \text{ mol L}^{-1}$ and para-hydroxybenzoic acid pH 1 to pH 12.

The formation of silver nanoparticles was characterized by the presence of maximum absorption of visible light in a wavelength range of 400-500 nm followed by a change in color to pale yellow [16].

4.3 The effect of the concentration of AgNO_3 on the absorbance of silver nanoparticle colloids

The effect of the concentration of AgNO_3 can be seen from the absorbance of the measured solution using a UV/Vis spectrophotometer when the concentration of AgNO_3 used increases, the absorbance value at the maximum absorption wavelength that is almost close or equal will increase, because more silver nanoparticles are produced [17].

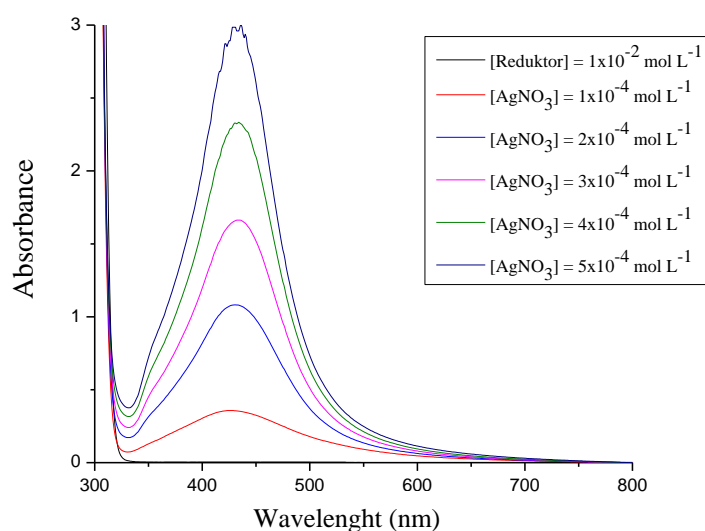


Figure 4. The spectrum of silver nanoparticles synthesized using AgNO_3 1×10^{-4} to $5 \times 10^{-4} \text{ mol L}^{-1}$.

4.4 Characterization of silver nanoparticles morphology

The size of the silver nanoparticles produced was $34 \pm 1.78 \text{ nm}$.

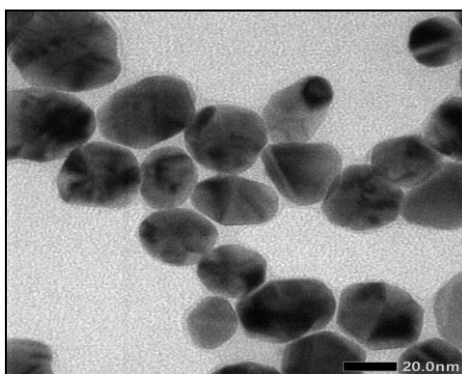


Figure 5. Shape of silver nanoparticles formed.

Characterization results of nanoparticles shape and size with TEM showed that the silver nanoparticles produced had a uniform morphological shape with a uniform size. Besides, from TEM images, no aggregate was formed which indicates the para-hydroxy benzoate acid reducing agent besides having the ability to reduce silver ions also can maintain the stability of silver nanoparticles as indicated by the absence of agglomeration in the silver nanoparticles.

4.5 Study of the stability of silver nanoparticles

The stability of silver nanoparticles was observed by looking at the absorbance of nanoparticles formed at alkaline pH and the shift in their wavelengths. The results of absorbance measurements showed that the silver nanoparticles produced were stable for 6 weeks of storage. This can be seen from the shift in wavelength that was not too significant, which is only 4 nm from the wavelength of 427 nm to 431 nm.

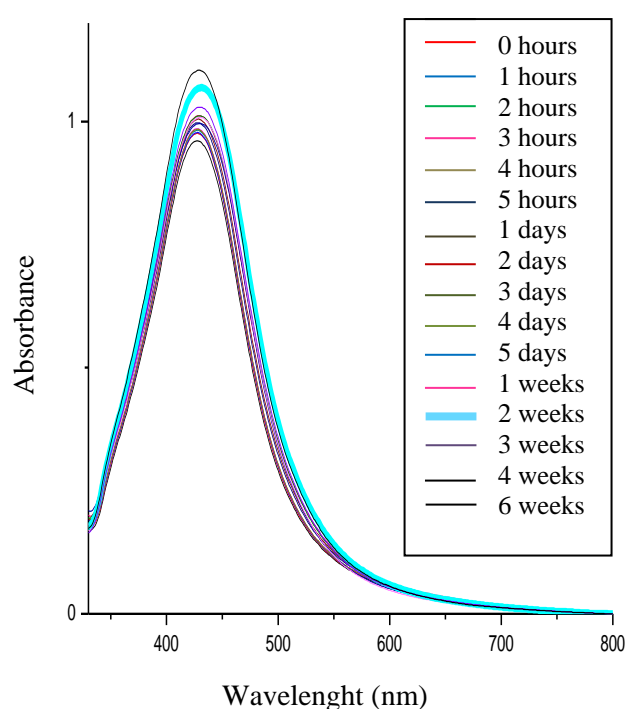


Figure 6. The spectrum of silver nanoparticles stability.

5. Conclusions

Reduction of Ag (I) ions with para-hydroxy benzoic acid which also as a stabilizer can produce silver nanoparticles that formed at alkaline pH. The silver nanoparticles produced proved to be stable for 6 weeks storage, which was characterized by a small wavelength shift as 4 nm. The morphological form of the silver nanoparticles produced has a spherical morphology with a uniform size as 34 ± 1.78 nm.

6. References

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