Synthesis of silver nanoparticles used chemical reduction method by glucose as reducing agent

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Abstract. Silver nanoparticles have been successfully synthesized using a chemical reduction method at room temperature using glucose as a reducing agent, silver nitrate as a source of nitrate, polyvinyl alcohol (PVA) as a stabilizing agent. The purpose of this research was to determine the effect of the mole ratio of reducing agent Glu / Ag and percentage (%) PVA (b/v) in the silver nanoparticle synthesis of the resulted product. Product characterization was analyzed by UV-Vis Spectrophotometers, XRD, and TEM. The Analysis of the UV-Vis spectra showed that the most stable particles were silver nanoparticles used PVA 3% and Glu / Ag 1: 8 on λ\text{max} 415 to 417 nm. TEM's characterization showed that silver nanoparticles were spherical with a dominant size range of 12.28 nm – 38.45 nm. The newly synthesized silver nanoparticles may have a potential for antibacterial applications.

1. Introduction
Silver nanoparticles were one of the most widely studied materials in the field of nanoscale technology and were used for a variety of applications that lead to the formation of silver nanoparticles and silver ions. It was also had the ability as an antimicrobial agent and was more environmentally friendly than other metal nano. It was a nanotechnology-based product. It was antibacterial and anti-virus will be very helpful in dealing with various problems that can be caused by bacteria and viruses [1]. Colloidal silver nanoparticles can be used in special applications with textiles, one of that antimicrobial properties, to produce antimicrobial products which can be carried out through engineering particles such as silver with sizes between 1 to 100 nanometers. Silver was a commonly used metal, because of its low toxic properties to the human skin. The purpose of silver ions is made nano because viruses, bacteria, and pathogens are the smallest particles that live in biological organisms. In order for silver to work effectively, the size of silver must be smaller than viruses, bacteria, and other pathogens [2].

In principle, the degradation of bacteria and viruses by silver ions was that silver particles will damage and penetrate the bacterial cell wall, then enter the bacterial thiol group and bind to sulfhydryl groups in bacteria so as to prevent the production of enzymes in bacteria. Furthermore, silver particles will inhibit DNA growth and eventually bacteria die [3]. Silver ions are neutral in water, acid resistant, salt and weak. Good stability against heat and light. Has long-lasting antibacterial power. The smaller the particle size, the more negligible the influence of gravity. While increasing the surface charge and chemistry allows the repulsive force between particles so that the particles can be dispersed in water. There are many methods used for silver nanoparticles syntheses such as chemical reduction methods [4], electrochemistry [5], \gamma -radiation [6], photochemistry [7] and laser ablation. However,
the most popular method in the formation of silver nanoparticles is the chemical reduction. In this method, a reduction reaction process was used on silver salts such as silver nitrate, silver sulfate, silver perchlorate, and other silver-containing salts. This method was easy and relatively low cost [4,8]. Other substances used for making silver nanoparticles were stabilizers, binding agents, reducing agents, distilled water, and catalysts to accelerate reactions. Stabilizers of surface-active substances include anionic zap, cationic zap, amphoteric, and nonionic zap. The stabilizer functions to prevent the aggregation of nanoparticles by creating a protective layer between particles to complicate the movement of atoms. Research that had been carried out substances that are often used as stabilizers in these nanoparticles were sodium dodecyl sulfate (SDS), polyphenyl pyrrolidone (PVP), polyvinyl alcohol (PVA), but the substance that was often used in PVP [9]. Reducing agents were used to reduce what was present in metal salts to atoms, reducing agents commonly used were sodium borohydride (NaBH₄), hydrazine (N₂H₄), formaldehyde (HCHO), glucose (C₆H₁₂O₆.H₂O), trisodium citrate (2Na. C₆H₅O₇.H₂O), etc [10]. The most commonly used substance was glucose because glucose was a weak reducing agent[11] . The catalyst was used to speed up the reaction. Reducing agents will work in an alkaline atmosphere. in other words, the addition of alkali can complete the reaction in the short term. The compounds used generally contain hydroxide ions (OH), such as NaOH. Subsequently, the substance used as a binding agent. The function of this substance is to regulate the concentration of free metal ions in solution. Just as the stabilization functions to prevent the formation of aggregation, particle size can be regulated. Substances commonly used are urea (CO (NH₂)₂) for reasons of convenience and the cost was relatively cheap [12].

In this study, the source of silver used is AgNO₃ (silver nitrate), the reducing agent used was glucose, the stabilizing agent used is polyvinyl alcohol (PVA), the binding agent was used ammonium hydroxide. The mole ratio between the reducing agent / Ag varied 1: 4, 1: 8 and 1: 12 and% of PVA varied 1%, 3% and 5%. This research will prove the effect of mole ratio and% stabilizers on the carving of the formed Ag nanoparticles.

2. Material and Method

2.1. Material
Glucosa, polyvinyl alcohol, acetic acid (CH₃COOH), ammonium hydroxide (NH₄OH), Silver Nitrate (AgNO₃) were purchased from Merck.

2.2. Procedure

2.2.1. Synthesis of Silver Nanoparticles
0.017 grams of silver nitrate were dissolved in 100 ml of water while being distilled until silver nitrate dissolved and ammonium hydroxide was added. The silver ion solution formed is put into glucose solution in the ratio of the mole ratio of glucose to Ag + (1: 4, 1: 8 and 1: 12). In this solution PVA was added with a concentration of 1, 3 and 5%, then heated at 60°C in an erlemeyer flask. During the heating process, the mixture is stirred using a magnetic stirrer until it is brown. Then the heating is stopped but the impregnation is continued until the mixture temperature becomes room temperature. The formed colloidal silver nanoparticles were observed and measured until the day 14 after synthesis by UV-Vis spectrophotometer. Furthermore, colloidal silver nanoparticle samples were characterized by TEM and XRD.

2.3. Characterization
The powder X-ray diffractogram of synthesized and calcined samples was recorded on a Rigaku Miniflex diffrajectometer with CuKa radiation between 1.5 and 10° (2θ) with a scanning rate of 1°/min. TEM micrographs of the samples were obtained with a JEOL 100CX microscope with 100 kV of acceleration voltage. SEM merk JEOL-JSM 6360 LA.
3. Results and Discussion

3.1. Effect Molar Ratio of Glucose/Ag and % PVA

Molar ratio effect of Glucose / Ag (1: 4, 1: 8 and 1: 12) and PVA addition% (1%, 3% and 5%) shown in Figure 1. It explains the effect of adding the Glucose / Ag molar ratio and The PVA% measured by UV-Vis Spectrophotometer obtained wavelength results from colloidal silver nanoparticles measured on days 1,4,7,10 and 14. Figure 1. A was a colloidal silver nanoparticle image with a ratio of molar ratio Glucose / Ag 1: 4. Fig 1.B was a colloidal silver nanoparticle image with molar ratio Glucose / Ag 1: 8 and 1.C was a colloidal image of silver nanoparticles with a ratio of molar ratio Glucose / Ag 1: 12. From the data, it shown that the addition of PVA affects the stability of silver nanoparticles. The maximum wavelength had increased every day until measurements on day 14.

![Figure 1](image)

**Figure.1** The Curve of the maximum wavelength of silver nanoparticles (A) Glu/Ag 1 : 4 (B) Glu/Ag 1 : 8 and (C) Glu/Ag 1 : 12

From the graphs of A, B and C silver nanoparticles with the addition of PVA 1% the maximum wavelength shifted from (412 nm, 420 nm, and 418 nm) on day 14. At the addition of 3%, PVA gives a measurement of the maximum wavelength shifting from (415 nm, 416 nm, and 417 nm) on day 14. However, the addition of 5% PVA gives the maximum wavelength measurement results which were unstable (406 nm, 445 nm, and 439 nm). The addition of 1% PVA produces silver nanoparticles that quite stable due to a small shift in maximum wavelength. The addition of 3% PVA produces relatively stable silver nanoparticles because of the smallest maximum wavelength shift. Different when compared with the addition of PVA 5% to produce unstable silver nanoparticles, it shown from the shift in the maximum wavelength that is 406 nm, 445 nm, and 439 nm and there are black deposits in the sample. Black sediment indicates that nanoparticles colloids undergo agglomeration [13].
Stabilizers play an important role in controlling the size of silver nanoparticles. If the stabilizer added above the optimum conditions can prevent the stability of the silver nanoparticles, causing agglomeration [14]. Therefore in this study, the addition of 3% PVA is the optimum concentration condition in synthesis because it produces silver nanoparticles with a stable maximum wavelength spectrum.

Figure 2 explains that the absorption increases with increasing of time for colloidal silver nanoparticles until day 14. Figure 2 A, B and C show that the Glucose / Ag 1: 4-mole ratio of silver colloid nanoparticles on day 1 had an absorption of 0.179, 0.514 and 0.213 then on day 14 the peak absorption increased to 0.346, 0.718 and 0.413. At Glucose / Ag 1: 8 mole ratio, nanoparticle colloid on day 1 had a peak absorbance of 0.215, 0.954 and 0.514 then on day 14 the peak absorption increased to 0.918, 1.798 and 0.936. However, it was different when compared to the Glucose / Ag 1: 12-mole ratio of silver colloid nanoparticles on day 1 had an absorption of 0.246, 0.741 and 0.241 then on the 14th day the peak of absorption increased to 0.435, 1.568 and 0.546. Glucose / Ag 1: 8-mole ratio shows that the absorption is relatively stable and significant compared to Glucose / Ag 1: 4-mole ratio and Glucose / Ag 1: 12. Comparison of the number of reducing agents Glucose affects the stability of the formed nanoparticles [18]. Based on the above conditions it can be concluded that the mol ratio of Glucose / Ag 1: 8 and the addition of 3% PVA is the optimum condition to produce silver nanoparticles. Therefore this sample will be characterized by TEM, SEM and XRD analysis.

3.2. XRD analysis
The peak of Ag nanoparticle diffraction shwan in Figure 3. Based on Figure 3, the diffraction peaks appear at 2θ with high intensity at 38.0849, 44.2563, 64.3639, 77.5192, 81.3964 with Miller index
{111}, {200}, {220}, {311}, and {222}. The Miller index is the Crystal lattice \{hkl\} field which states the Crystal system was a material. Crystal System of silver nanoparticles, namely Faces Centered Cubic (FCC) according to JCPDS Ag data No. 01-071-4613. Based on the results of XRD obtained the size of silver nanoparticles was 18.61 – 30.01 nm.

![XRD Pattern of Silver Nanoparticles](image1.png)

**Figure 3** XRD Pattern of Silver Nanoparticles

3.3. **SEM-EDX analysis**

Silver nanoparticles with a glucose / Ag 1: 8 ratio reducing agent and 3% PVA are the optimum conditions for the mole ratio of reducing agents and stabilizers agent with silver moles used. From the results of SEM images in fig.4, the surface morphology of the resulting material is spherical in uniform size.

![SEM images of Silver Nanoparticles](image2.png)

**Figure 4.** SEM images of Silver Nanoparticles
EDX analysis analyzes the composition of elements produced from product materials. Figure 5 shows 76.75% silver elements produced from silver nanoparticles material produced. From the results of EDX analysis, more than half of the composition components of the material produced were silver, the rest in the form of oxygen elements derived from oxides used in the synthesis of silver nanoparticles.

![Figure 5. EDX - Silver Nanoparticles.](image)

3.4. TEM analysis

Based on the TEM results of silver nanoparticles with a comparison of Glucose / Ag 1: 8 reducing agent and 3% PVA can produce the smallest silver particle size of 12.28 nm – 38.45 nm. According to Sosilawati et al, 2015 [15] reducing agents can affect the nanoparticles formed. The addition of 3% PVA is the optimum condition of the addition of agent stabilizers so that it can inhibit the agglomeration process so that the resulting composition is not getting bigger. Silver nanoparticle synthesis conducted by Zhang, et al 2016 states that by varying the ratio of mole of reducing agent to precursor Ag, silver nanoparticles of various sizes [16]. This shown that the synthesized silver nanoparticles have proven distribution.
4. Conclusion
Comparison of Glucose/Ag 1: 8 mole reducing agent and 3% PVA% produce silver nanoparticles whose absorption is the most significant and relatively stable at wavelength measurements of 415-417 nm. Therefore, the addition of PVA 3% and the ratio of mole ratio of Glucose / Ag 1: 8 was the optimum condition in the synthesis of silver nanoparticles. The results of TEM analysis showed that the morphology of silver nanoparticles was spherical with a particle size of 12.28 nm – 38.45 nm. The results of the XRD analysis showed that the silver nanoparticles produced had a Face Centered Cubic (FCC) crystal structure.

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