
Titik Taufikurohmah1*, Djodjok Soepadjo2, Rusmini3, and Hari Armadiano4
1, 3 Chemistry Department, Universitas Negeri Surabaya, INDONESIA.
(E-mail: titiktaufikurohmah@unesa.ac.id, rusmini@unesa.ac.id)
2 Japanese language Department, Universitas Negeri Surabaya, INDONESIA.
(E-mail: dsoepardjo@unesa.ac.id)
4 R&D Department PT. Gizi Indonesia

Abstract—Cluster diameter analysis of the results of nanomaterial synthesis can be done using UV-Visible instruments with complex mathematical calculations. Another way is to use the Transform Electron Microscope (TEM) instrument directly. Both have advantages and disadvantages of each. This research compares the results of the two instruments on the results of nanogold and nanosilver synthesis. The results obtained diameter through calculations from UV-Visible nanogold spectra has a diameter of 20.62-20.68 nm, 20 ppm nanosilver has a diameter of 17.32 nm, while the combined nanogold-nanosilver has a diameter of 17.22-17.33 nm. The results of measuring the diameter of the cluster using the TEM instrument give more varied results. It was concluded that measurement of cluster diameter using UV-Visible gives a picture of the dominant cluster diameter that exists in colloidal nanomaterials both nanogold and nanosilver. The results of the measurement of the diameter of the clusters with the TEM instrument give a real picture of the variation of the diameter of the clusters produced by synthesis. Thus this study has the impact of making it easier for researchers to determine instrument choices as needed.

Keywords— instrument, cluster diameter, synthesis, nanogold, nanosilver.

I. INTRODUCTION

The development of nanomaterial technology has covered various fields of application, mainly in the medical field, namely drug and cosmetic formulation technology [1]. The use of nanogold as an antiaging in cosmetics has been applied in many countries in the world. As antiaging in cosmetics, nanogold have antioxidant activity [2]. Nanogold has been tested to recovery skin tissue damage caused dangerous material phenol, study case [3]. Tissue labeling method has been used to determine the part of tissue that recovery by nanogold [4]. The application of nanogold in pharmaceutical formulas has been used in drug and modern cosmetics [5]. In vitro and in vivo test of toxicity of nanogold giving explanation that nanogold safe to body [7]. The size of nanogold cluster in the colloidal system make it possible to cellular interaction [8]. This interaction analysis with Atmospheric scanning electron microscopy [9]. Similarly, silver nanoparticles has analysis in vivo assessment of some adverse bioeffect including bio-protectors activity [10]. Nanosilver as a reliable antimicrobial with a very broad spectrum [11]. Nanogold has high antioxidant activity [2], Nanogold and nanosilver not easily damaged and are easily synthesized in the laboratory [12]. Nanogold has the ability to reduce free radicals such as antioxidants from natural ingredients even better [2]. Nanogold heals tissue damage because it has the ability to increase cell proliferation and collagen biosynthesis [1].

Based on these advantages, nanogold and nanosilver are selected as important materials in medicine and cosmetic formulas [13]. Synthesis of nanogold and nanosilver is done with a variety of conditions to get the diameter of the cluster of atoms (cluster) as desired [14]. Cluster size is related to the ease of absorption of material. The easier the material is absorbed the greater the usefulness in medicine and cosmetics. Cluster diameter measurement and analysis are important in every nanomaterial synthesis [15]. The use of the right instruments will produce accurate cluster diameter measurements. Need a study that analyzes and compares the results of accurate nanomaterial cluster diameter measurement results.

This research compares the results of cluster diameter analysis to the results of nanogold and nanosilver synthesis [16]. The results of nanogold and nanosilver synthesis were characterized and analyzed using two instruments, UV-Visible instruments and TEM instruments. To determine the diameter of the nanogold and nanosilver clusters using complex mathematical calculations on the UV-Visible instrument. Whereas with the TEM instrument the cluster diameter analysis is directly from the scale listed on the resulting image.

Both the results of the measurement of the diameter of the cluster discussed the advantages and disadvantages to meet the demands of researchers. Selection based on the needs of researchers will be answered in a detailed discussion of each instrumentation result. This research began with nanogold and nanosilver synthesis and a combination of both. The basic ingredients of nanogold are HAuCl4 and nanosilver in the form of AgNO3 by reducing sodium citrate in aquo media [17]. Nanogold various concentrations, nanosilver 20 ppm and a combination of both. The research data are absorbance and maximum wavelength data from UV-Visible spectra. Data from TEM instruments are in the form of images and sizes of nanogold and nanosilver clusters [18].

Data from the two instruments will be analyzed by different methods. This is to answer the research problem, namely how the results of nanogold and nanosilver cluster diameter measurements using UV-Visible and TEM instruments. What are the advantages and disadvantages of
both in preparation for their use in various cosmetic and drug formulas.

II.  RESEARCH METHODS

A. Tool
Some of the tools used in this research, namely a beaker glass 500mL, 10mL measuring cup, 100mL measuring flask, watch glass, analytic balance, hot plate, magnetic stirrer UV-Visible Spectrophotometer Shimadzu 1800, and TEM (Transmission Electron Microscopy) JEOL JEM 1400.

B. Materials
Some of the materials used in this research that 1000 ppm HAuCl₄ solution, 1000 ppm AgNO₃ solution, sodium citrate p.a (≥ 99% Merck), and distilled water (PT Bratachem).

III. EXPERIMENTAL SETUP

A. Synthesis and Analysis of Nanogold
Nanogold is synthesized from 1000 ppm HAuCl₄ base material which is yellow. Heat water (1000-x) ml until boiling. 2 g of sodium citrate is put into boiling water. X ml HAuCl₄ is added, then wait 5 minutes until the solution changes from yellow to burgundy. The heating is stopped and the resulting colloidal red color is nanogold. Nanogold is ready to be analyzed with UV-Visible instruments and TEM instruments. If x is 20 ml, the resulting nanogold concentration is 20 ppm.

B. Synthesis and Analysis of Nanosilver
Nanosilver was synthesized from a colorless 1000 ppm AgNO₃ base material. Heat water (1000-x) ml until boiling. 2 g of sodium citrate is put into boiling water. X ml of HNO₃ is added, then wait 5 minutes until the solution changes from colorless to yellow-gray. The heating is stopped and the resulting yellow-gray colloid is nanosilver. Nanosilver is ready to be analyzed with UV-Visible instruments and TEM instruments. If x is 20 ml, the concentration of nanosilver produced is 20 ppm.

IV. RESULTS AND DISCUSSION

Figure 1. UV-Visible Spectra of Nanogold at Variant Concentrate
Nanogold absorb visible light, and maximum absorption occur 517-520 nm. The absorption value related with concentration. The greater concentrate following the greater absorbance of colloidal gold in the spectrogram at Figure 1. Nanogold with variant concentration 5, 10, 15, 20, and 25 ppm give variant absorbance. Nanogold 20 ppm and nanogold 25 ppm have absorbance value almost overlapping.

Figure 2. UV-Visible Spectra of Nanosilver and Nanogold at Variant Cocentrate
Based on Figure 1 and Figure 2, Nanogold absorption is seen at various concentrations at a wavelength of 517-520 nm. While the combined nanosilver and nanogold various concentration absorption wavelengths 429.50-433.00. This shows the uptake shifts near the 433.50 nm nanosilver uptake. Thus the nanosilver cluster covers the
nanogold cluster so that what is read by the instrument is the nanosilver cluster.

Table 1. Analyze result of UV Visible Nanogold and Nanosilver

<table>
<thead>
<tr>
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<th>(\lambda) maximum (nm)</th>
<th>Absorbance</th>
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<td>0,464</td>
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<table>
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<th>No</th>
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<th>Absorbance</th>
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<td>0,521</td>
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<td></td>
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<td>0,544</td>
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</table>

Mathematical Calculations

Complex mathematical calculations for calculating nanogold and nanosilver cluster diameters include the following stages:

\[ E_g = E_g(\infty) + \frac{14.84}{8R^2} \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{2.6}{kR} \]

\[ E_g(\infty) \text{ from reference } = 1.3 \text{ eV} \]

\[ m_e \text{ and } m_h = 0.25 \]

\[ k = 6.5 \]

so the value of \( R \) (cluster diameter) can be calculated as

\[ \frac{hc}{\lambda} = E_g(\text{bulk}) + \frac{14.84}{8R^2} \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.8e^2}{4\pi e \varepsilon_0} \]

1. **NS 20 ppm (433,50 nm)**

\[ \frac{hc}{\lambda} = E_g(\text{bulk}) + \frac{14.84}{8R^2} \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.8e^2}{4\pi e \varepsilon_0} \]

\[ \frac{1240.6}{433.50} = 1.3 + \frac{14.84}{1} \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R} \]

\[ 2.862 - 1.3 = \frac{14.84}{R^2} \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R} \]

\[ 1.562 = \frac{29.68}{0.0625R^2} - \frac{2.6}{6.5R} \]

\[ 1.562 \times 0.0625R^2 = 29.68 - 0.025R \]

\[ 0.0976R^2 + 0.025R - 29.68 = 0 \]

can be searched by the abc formula

\[ x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a} \]

So the value of \( R \) is:

\[ R = -0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0976 \times (-29.68)} \]

\[ R = \frac{-0.025 \pm 3.403}{0.195} \]

\[ R = 17.32 \text{ nm} \]

2. **NG 5 ppm (517,00 nm)**

\[ \frac{hc}{\lambda} = E_g(\text{bulk}) + \frac{14.84}{8R^2} \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.8e^2}{4\pi e \varepsilon_0} \]

\[ \frac{1240.6}{517.00} = 1.3 + \frac{14.84}{1} \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R} \]

\[ 2.400 - 1.3 = \frac{14.84}{R^2} \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R} \]

\[ 0.100 = \frac{29.68}{0.0625R^2} - \frac{2.6}{6.5R} \]

\[ 0.100 \times 0.0625R^2 = 29.68 - 0.025R \]

\[ 0.0687R^2 + 0.025R - 29.68 = 0 \]

R can be searched by the abc formula (x)

\[ x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a} \]

\[ R = -0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0687 \times (-29.68)} \]

\[ R = \frac{-0.025 \pm 2.856}{0.137} \]

\[ R = 20.66 \text{ nm} \]

3. **NS 10 ppm (517,50 nm)**

\[ \frac{hc}{\lambda} = E_g(\text{bulk}) + \frac{14.84}{8R^2} \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.8e^2}{4\pi e \varepsilon_0} \]

\[ \frac{1240.6}{517.50} = 1.3 + \frac{14.84}{1} \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R} \]

\[ 2.397 - 1.3 = \frac{14.84}{R^2} \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R} \]

\[ 0.097 = \frac{29.68}{0.0625R^2} - \frac{2.6}{6.5R} \]

\[ 0.097 \times 0.0625R^2 = 29.68 - 0.025R \]

\[ 0.0686R^2 + 0.025R - 29.68 = 0 \]

R can be searched by the abc formula (x)

\[ x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a} \]

\[ R = -0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0686 \times (-29.68)} \]

\[ R = \frac{-0.025 \pm 2.854}{0.137} \]

\[ R = 20.65 \text{ nm} \]

4. **NS 15 ppm (518.00 nm)**

\[ \frac{hc}{\lambda} = E_g(\text{bulk}) + \frac{14.84}{8R^2} \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{1.8e^2}{4\pi e \varepsilon_0} \]

\[ \frac{1240.6}{518.00} = 1.3 + \frac{14.84}{1} \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R} \]

\[ 2.395 - 1.3 = \frac{14.84}{R^2} \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R} \]
1.095 = \frac{29.68}{0.0625 R^2} - 2.6 \cdot \frac{1}{6.5R}
1.095 \times 0.0625 R^2 = 29.68 - 0.025R
0.0684 R^2 + 0.025R - 29.68 = 0
x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}
R = \frac{-0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0684 \times (-29.68)}}{2 \times 0.0684}
R = \frac{-0.025 \pm 2.851}{0.137}
R = 20.62 \text{ nm}

5. NS 20 ppm (520.00 nm)
\frac{hc}{\lambda} = E_g (bulk) + \left( \frac{h^2}{8R^2} \right) \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{18e^2}{4\pi \varepsilon_0 R}
1240.6 \cdot 520.00 = 1.3 + \left( \frac{14.84}{R^2} \right) \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R}
2.386 - 1.3 = \left( \frac{14.84}{R^2} \right) \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R}
1.086 = \frac{29.68}{6.5} - \frac{2.6}{6.5R}
1.086 \times 0.0625 R^2 = 29.68 - 0.025R
0.0679 R^2 + 0.025R - 29.68 = 0
x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}
R = \frac{-0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0679 \times (-29.68)}}{2 \times 0.0679}
R = \frac{-0.025 \pm 2.838}{0.136}
R = 20.68 \text{ nm}

6. NS 25 ppm (518.00 nm)
\frac{hc}{\lambda} = E_g (bulk) + \left( \frac{h^2}{8R^2} \right) \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{18e^2}{4\pi \varepsilon_0 R}
1240.6 \cdot 518.00 = 1.3 + \left( \frac{14.84}{R^2} \right) \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R}
2.395 - 1.3 = \left( \frac{14.84}{R^2} \right) \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R}
1.095 = \frac{29.68}{6.5} - \frac{2.6}{6.5R}
1.095 \times 0.0625 R^2 = 29.68 - 0.025R
0.0684 R^2 + 0.025R - 29.68 = 0
x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}
R = \frac{-0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0684 \times (-29.68)}}{2 \times 0.0684}
R = \frac{-0.025 \pm 2.851}{0.137}
R = 20.62 \text{ nm}

7. NS 20 ppm + NG 5 ppm (433.00 nm)
\frac{hc}{\lambda} = E_g (bulk) + \left( \frac{h^2}{8R^2} \right) \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{18e^2}{4\pi \varepsilon_0 R}
1240.6 \cdot 433.00 = 1.3 + \left( \frac{14.84}{R^2} \right) \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R}
2.865 - 1.3 = \left( \frac{14.84}{R^2} \right) \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R}
1.565 = \frac{29.68}{6.5} - \frac{2.6}{6.5R}
1.565 \times 0.0625 R^2 = 29.68 - 0.025R
0.0978 R^2 + 0.025R - 29.68 = 0
x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}
R = \frac{-0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0978 \times (-29.68)}}{2 \times 0.0978}
R = \frac{-0.025 \pm 3.411}{0.1956}
R = 17.31 \text{ nm}

8. NS 20 ppm + NG 10 ppm (431.00 nm)
\frac{hc}{\lambda} = E_g (bulk) + \left( \frac{h^2}{8R^2} \right) \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{18e^2}{4\pi \varepsilon_0 R}
1240.6 \cdot 431.00 = 1.3 + \left( \frac{14.84}{R^2} \right) \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R}
2.878 - 1.3 = \left( \frac{14.84}{R^2} \right) \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R}
1.578 = \frac{29.68}{6.5} - \frac{2.6}{6.5R}
1.578 \times 0.0625 R^2 = 29.68 - 0.025R
0.0987 R^2 + 0.025R - 29.68 = 0
x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}
R = \frac{-0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0987 \times (-29.68)}}{2 \times 0.0987}
R = \frac{-0.025 \pm 3.422}{0.1973}
R = 17.22 \text{ nm}

9. NS 20 ppm + NG 15 ppm (434.00 nm)
\frac{hc}{\lambda} = E_g (bulk) + \left( \frac{h^2}{8R^2} \right) \left( \frac{1}{m_e} + \frac{1}{m_h} \right) - \frac{18e^2}{4\pi \varepsilon_0 R}
1240.6 \cdot 434.00 = 1.3 + \left( \frac{14.84}{R^2} \right) \left( \frac{1}{0.25} + \frac{1}{0.25} \right) - \frac{2.6}{6.5R}
2.859 - 1.3 = \left( \frac{14.84}{R^2} \right) \left( \frac{2}{(0.25)^2} \right) - \frac{2.6}{6.5R}
1.559 = \frac{29.68}{6.5} - \frac{2.6}{6.5R}
1.559 \times 0.0625 R^2 = 29.68 - 0.025R
0.0974 R^2 + 0.025R - 29.68 = 0
x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}
R = \frac{-0.025 \pm \sqrt{(0.025)^2 - 4 \times 0.0974 \times (-29.68)}}{2 \times 0.0974}
The size cluster diametre of nanogold and nanosilver that calculate with kompleks mathematical calculation give following data, Table 2. Nanosilver 20 ppm have cluster diametre 17.32 nm. Nanogold 20 ppm have cluster diametre 20.68, only one value. This value appear and discribe of the size of cluster dominant in the colloidal system.

Table 2. Cluster Diametre (CD) of Nanogold and Nanosilver by Mathematic Calculation

<table>
<thead>
<tr>
<th>No</th>
<th>Sample</th>
<th>Concentration (ppm)</th>
<th>E maximum (nm)</th>
<th>Absrbanse</th>
<th>CD (nm)</th>
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<tbody>
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<td>0,544</td>
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</table>

Figure 3. TEM of Nanogold 20 ppm (Left) and Nanosilver 20 ppm(Right)

Based on Figure 3, calculated using the scale shown in the TEM image it can be determined that the diameter of the 20 ppm Nanogold cluster has varying sizes. In Figure 2, there are 9 nanogold clusters with a size of 20.67; 20.62; 20.73; 19.10; 19.21; 18.31; 14.41; 12.35; and 8.21 nm. Figure 3 show that these sizes of the clusters not only one size as was UV-visible analysing with mathematical calculation. Thus the dominant cluster at size 20.xx nm from TEM images while from UV-Visible the diameter of the nanogold cluster is 20.68.
V. CONCLUSION

It was concluded that diameter cluster was read in the UV-Visible instrument was a cluster with dominant size that was results of synthesis. The TEM instrument provides the real of cluster diameter sizes that variant size corresponding with actual conditions of the synthesis results. The combination of nanogold and nanosilver shows that the nanosilver cluster covers the nanogold cluster so that what is observed by the instrument is the nanosilver cluster.

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