The Effect Of Microwave Irradiation on Synthesis of Gold Nanoparticles Using Ethanol Extract of White Bol Guava Leaves

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Abstract: The aim of this research was to determine the effect of microwave radiation on the synthesis of gold nanoparticles using ethanol extract of white bol guava leaves. 50 mL of 25 ppm HAuCl₄ solution was synthesized using ethanol extract of white bol guava leaves to convert Au³⁺ to Au⁰ with 0.75 mL and synthesized using microwave and without microwave. Characterization of gold nanoparticles (Au-NPs) using Scanning Electron Microscope (SEM) and X-ray Diffraction (XRD) instruments. Based on the results, the microwave irradiation has a significant effect on the formation of Au-NPs using ethanol extracts of white bol guava leaves, based on the analysis using XRD, the size of Au-NPs was 17.13 nm with microwave irradiation and 17.57 nm without without microwave irradiation. Based on the results of SEM-EDS obtained Au-NPs by the microwave irradiation of 54.92%, while without a microwave irradiation of 42.17%.

Keywords: white bol guava leaves, ethanol extract, microwave, gold nanoparticle

1. Introduction

The research of nanotechnology has shown the creation of a new product with better performance. The directs research in chemistry to synthesize nano-sized particles (nanoparticles). The metals that are widely developed into nanoparticles are Au (gold), Pt (platinum), Ag (silver), and Pd (palladium). The most studied is Au because Au ions are known have free radical scavenging activity that can cause damage in various parts of cells that cause aging [1].

The properties of Au-NPs different with Au. The Au has a solid yellow and inert shape while gold nanoparticles are burgundy. The interaction of particles and the formation of Au-NPs networks is key in determining the properties of nanoparticles. Au-NPs also have different sizes and have different shapes such as spheres, octahedral, decahedral, tetrahedral, nanotriangles, nanoprisms, hexagonal platelets and nanorods [2].

Synthesis of nanoparticles can be synthesized using microwave irradiation. The biggest advantage of microwave irradiation was the produced of heat evenly. Microwave irradiation, the formation of nanoparticle nuclei is also more uniform and the time required is also faster, compared to conventional heating for nanoparticle synthesis [3].
The natural materials as bioreductors contribute to the reduction of inorganic reducing agents. These bioreductors have advantages such as not producing by-products or only producing environmentally friendly by-products that are easily separated.

White bol guava leaves contain secondary metabolites, namely flavonoids, terpenoids, steroids and phenolics. The characteristic of flavanoid compounds which are easily oxidized is the release or donor of electrons to inhibit antioxidant molecules to become free radicals. Based on its antioxidant properties and the ease of oxidation it can facilitate the process of forming Au-NPs which a reduction process.

2. Experimental Details

2.1. Sample preparation
White bol guava leaves are washed thoroughly with distilled water. The leaves are dried in the air, then cut into small pieces., Weighed as much as 1000 g then macerated with 10 L of ethanol 96% for 3 x 24 hours. Then filtered using a vacuum pump then ethanol is evaporated so that a thick extract of white guava leaves is ready to be used as bioreductor.

2.2. Synthesis of Au-NPs without Microwave Irradiation
A total of 50 mL of HAuCl$_4$ 25 ppm solution was added as much as 0.75 mL of ethanol extract of white bol guava leaves. Then the mixture is stirred for 120 seconds and allowed to stand for 60 minutes, then the mixture was centrifuged.

2.3. Synthesis of Au-NPs with Microwave Irradiation
A total of 50 mL of HAuCl$_4$ 25 ppm solution was added as much as 0.75 mL of ethanol extract of white bol guava leaves, irradiation microwave at 80 oC during 120 seconds. Then the mixture is stirred for 120 seconds and allowed to stand for 60 minutes, then the mixture was centrifuged.

2.4. X-Ray Diffraction Analysis
X-Ray Diffraction (XRD) analysis of Au-NPs was carried out using powder X-Ray diffractometer instrument (Shimadzu 7000) operated at a voltage of 40.0 kV and a current of 30.0 mA with CuKα radiation in 0-2θ scanning range of 20-80 configuration. The crystallite domain size was calculated using Debye- Scherrer formula.

2.5. Scanning Electron Microscopy (SEM) Analysis
The sample was prepared by placing Au-NPs powder on sample holder and subsequently drying in air, before transferring it into the microscope operated at an accelerated voltage of 130Kv (Hitachi SU 3500).

3. Results and Discussion
In general, the formation of Au-NPs can be seen of the change in the color of solution, where the solution becomes blackish purple. Based on this study, Au-NPs solution changed from brownish yellow to blackish purple in the minute 60. As time increases, the color of the solution becomes increasingly concentrated due to the reduction reaction between biorector and the HAuCl$_4$ precursor, shown in Fig.1.

The purple color formed indicates a reduction from Au$^{3+}$ to Au$^{0}$. The discoloration that occurs during synthesis indicates that the growth of the resulting clusters is increasingly greater, where when Au atoms have not interacted with each other (colorless solution).
In a certain amount, the gold cluster change to be brownish purple color which is followed to become dark purple, when the cluster gets bigger and when entering nano size, gold becomes purple. Au atoms will interact with fellow metal bonds and produce clusters in very large numbers.

There was a significant color change between the microwave and non-microwave methods, namely in the microwave method a rapid color change occurred from yellow to blackish purple in the next 5 minutes and minutes, whereas in the method without microwave, the color change from yellow to blackish purple occurred when 60 minutes and then the next day.

![Figure 1](image1.png)

**Figure 1.** The change of HAuCl4 and Biorector Solution color (a) using microwave irradiation (b) without microwave irradiation

With microwave irradiation, the formation of nanoparticle nuclei is also more uniform and the time required is also faster when compared to conventional heating for nanoparticle synthesis [4]. It can be concluded that, the microwave method can cause color changes that are relatively fast with the Au-NPs produced.

The results of the synthesis of Au-NPs obtained blackish brown powder as much as 0.0430 g (microwave) and 0.0400 g (without microwave).

![Figure 2](image2.png)

**Figure 2.** A. Au-NPs powder using *microwave* B. Au-NPs powder without *microwave*

The formation of Au-NPs was synthesized using ethanol extract of white bol guava leaves bioreductor supported by XRD analysis. The peaks of Au-NPs diffraction pattern with microwave irradiation are shown at 2θ values, namely 37.9120, 34.0776 and 44.0894 with the FWHM values of 0.388, 0.367 and 0.517, and the peaks of the diffraction pattern of Au-NPs without microwave irradiation are indicated at the values of 2θ namely 37.8604, 34.0310, and 44.0535 with FWHM values of 0.414, 0.389 and 0.545, respectively.

In addition, Miller's indexes of each peak are (111), (200), (202) and (311). The orientation of (202) and (311) the peaks are weak, and much stronger in orientation (111) and (200). It shows that Au-NPs are dominant in (111) and (200). The results of the XRD analysis are similar to the results of previous studies with the Miller’s index of Au-NPs (111), (200), (220), and (311) [5]. The highest intensity was (111) which describes the cubic lattice parameters.

At Angle 2θ produces several peaks of gold nanoparticles. However, those who have high intensity with the microwave method are at an angle of 2 37, 9120 with a FWHM value of 0.388 and without
using a microwave, which has a high intensity at an angle of 2.44, 0535 with an FWHM value of 0.548 so that it can be used as a reference for calculating the size of Au-NPs. The size of Au-NPs was calculated using the Debye Scherer equation obtained the size of Au-NPs using a microwave that is 17.13 nm and without a microwave that is 17.57 nm.

Figure 3. Diffractogram of Au-NPs (a) with microwave irradiation (b) without microwave irradiation

Analysis of Au-NPs with SEM instruments was carried out with the aim to determine the morphology and tendency of nanoparticles to aggregate.

Figure 4. Morphology of AuNP (a) using microwave (b) without microwave

The SEM result by using microwave and without microwave method show the morphology of the images reflected from the BSE (Backscattered Electron). This reflection gives the difference in molecular weight of the atoms that make up the surface, where atoms with high molecular weight will be lighter colors than atoms with low molecular weight.

Au-NPs are depicted in a brighter image, this is reinforced by the results of EDS which provides information on the composition of elements or elements, which are contained in the results of deposition of gold nanoparticles.

Based on Figure 5, the composition of elemental analysis results of the microwave method with EDS consisting of Gold (Au) of 54.92%, Carbon (C) of 27.4% and oxygen (O) of 17.68%. And the results of the analysis method without microwave with EDS consisted of Gold (Au) of 42.17%, Carbon (C) of 34.52%, and oxygen (O) of 23.31%.
Figure 5. (a) EDS result of Au-NPs (a) using microwave irradiation (b) without microwave irradiation

4. Conclusion
The microwave irradiation has a significant effect on the formation of Au-NPs using ethanol extracts of white bol guava leaves, based on the analysis using XRD, the size of Au-NPs was 17.13 nm with microwave irradiation and 17.57 nm without without microwave irradiation. Based on the results of SEM-EDS obtained Au-NPs by the microwave irradiation of 54.92%, while without a microwave irradiation of 42.17%.

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