

## Synthesis and Characterization of Gold Nanoparticles (AuNPs) by Utilizing Bioactive Compound of *Imperata cylindrica* (L.) Raeusch

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### Abstract

Gold nanoparticles (AuNPs) have been successfully synthesized using bioactive compounds of *Imperata cylindrica* (L.) Raeusch leaf extract. In this study, precursors used  $\text{HAuCl}_4$   $7 \times 10^{-4}$  M,  $8 \times 10^{-4}$  M and  $9 \times 10^{-4}$  M. Concentration of *Imperata cylindrica* (L.) Raeusch used was 5%. The colloid of Gold nanoparticle (AuNPs) formed were characterized using Ultraviolet-visible Spectrophotometer (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR), Transmission Electron Microscopy (TEM), X-ray Diffraction (XRD). The formation of gold nanoparticles (AuNPs) at concentration  $9 \times 10^{-4}$  M showed the best results with a color change to red. UV-Vis showed an absorbance value of 1.4 and a wavelength of 530 nm. FTIR analysis showed the interaction of -OH functional group on phenolic compounds of *Imperata cylindrica* (L.) Raeusch leaf extract with  $\text{Au}^{3+}$  ions. There was a shift on the peak at wavenumber  $3414 \text{ cm}^{-1}$  to  $3404 \text{ cm}^{-1}$ , indicating the formation of Au nanoparticles. TEM analysis showed the morphology of AuNPs is sphere-shaped with a particle size of 20 nm. XRD analysis showed a crystal size average of gold nanoparticles is 12 nm.

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## 1. INTRODUCTION

Gold nanoparticles are colloids which have particle size variations with their unique on surface plasmon resonance (SPR). The ability of gold nanoparticles to absorb visible light can be used as a typical colorimetric indicator. The formation of gold nanoparticles was visually indicated by a color change during synthesis. The color change started from purple-blue, then turned to pink, and finally ruby red during the

formation of gold nanoparticles synthesis [1]. Meanwhile, it was reported that it started from yellow then turned to pink, blue and finally purple-gray [2]. On the other hand, the formation of gold nanoparticles was indicated by color change from light yellow to violet, and red-purple [3–4]. Synthesis of gold nanoparticles can be carried out using physical and chemical methods. Physical method was carried out when large-sized material (bulk) turned to be

nanometer-sized particles (top-down method). Meanwhile, the chemical method was carried out when the nucleus of particles combined to be nanometer-sized particles (bottom-up method).

The top-down method approach uses mechanical milling, chemical etching, sputtering, laser ablation, and electro-explosion techniques. Meanwhile, the bottom-up method approach uses spinning techniques, template support synthesis, plasma or flame spraying synthesis, laser pyrolysis, CVD, atomic molecular condensation, and biological synthesis via bacteria, yeast, fungi, algae, and plants [5]. Synthesis of gold nanoparticles using chemical methods is influenced by several factors including stirring speed, pH of the solution, temperature, and concentration. Besides, the reduction of  $\text{Au}^{3+}$  ions to  $\text{Au}^0$  ions involves the addition of chemicals as reducing agents and capping agents [6]. However, the stabilizing agents used in the synthesis of gold nanoparticles, such as,  $\text{NaBH}_4$  [7], citric acid and sodium dodecyl sulfate [8], polyvinyl pyrrolidone [9], and cetyltrimethylammonium bromide [10] are not environmentally friendly.

The latest research used environmentally friendly bio-reducing agents and stabilizing agents to synthesize the gold nanoparticle, such as, *Tinospora crispa* leaf extract [11], *Polyscias fruticosa* [12], *Citrus maxima* [13], *Plukenetia volubilis* [14], *Cornus mas* [15], *Nerium oleander* [16], and *Allium sativum* L [17]. The use of bio-reducing agents and natural stabilizing agents on gold nanoparticles synthesis is known as the green synthesis method. *Imperata cylindrica* (L.) Raeusch is a wild plant that can be used as herbal medicine. Positive (+) results on phytochemical tests of *Imperata cylindrica* (L.) Raeusch leaf extract contain bioactive compounds alkaloids, flavonoids, and steroids [18]. The water fraction of *Imperata cylindrica* (L.) Raeusch leaf extract was successfully used in the synthesis of gold nanoparticles with an extract concentration of 3.46% [4]. The uniqueness of the shape and size of gold nanoparticles distribution is influenced by the media used during synthesis. Gold nanoparticles can be spherical-shaped with range of diameter

from 40 -70 nm [19], spherical shape with particle size distribution between 2 -10 nm [16], nanofibers shape with particle size of 30 -180 nm [20], particle surface spherical shape with average size of diameter 35 nm [21], nanostar-shaped with particle size of 11 -68 nm [2], gold prism shaped nanoparticles using bromide media [22] and spherical micelles shape with particle size of 1 -2 nm [23].

Gold nanoparticles can be applied as phenols detector from industrial waste [24], drug release [25], mediation of phototherapy in cancer [26], anti-microbial [27], potential in equine regenerative medicine [28], bio-sensors for the phenol detector from industrial effluents [29], in vitro studies for cancer [30], release of doxorubicin and verapamil [31], potential treatment of cancer and bacterial diseases [32], adaptive response in human astrocytes [33], and biomarkers detector [34].

In this research, gold nanoparticles was prepared through green synthesis method using the reaction of  $\text{HAuCl}_4$  solutions with *Imperata cylindrica* (L.) Raeusch leaf extract. The bio-active compounds contained in the *Imperata cylindrica* (L.) Raeusch leaf extract function as a medium, capping agent, as well a natural reductor agent for  $\text{Au}^{3+}$  to  $\text{Au}^0$  ions. Natural reduction agents of *Imperata cylindrica* (L.) Raeusch leaf extract can be used as a substitute for materials that are not environmentally friendly. Herein, we investigate a simple method and environmentally friendly in the gold nanoparticles synthesis. The produces in the gold nanoparticles synthesis using *Imperata cylindrica* (L.) Raeusch leaf extract are expected in small size of nanoparticles, morphological shape, and good of crystallinity.

## 2. EXPERIMENTAL SECTION

### 2.1. Materials and Instruments

Sample of *Imperata cylindrica* (L.) Raeusch leaf were taken from Depok Area (West Java, Indonesia). Gold metal 99.9% was PT Antam (Jakarta, Indonesia), while  $\text{HNO}_3$  and  $\text{HCl}$  were purchased from Sigma-Aldrich (Missouri, United States).

Characterization of gold nanoparticles (AuNPs) using UV-Vis Spectrophotometer Shimadzu 2600, with wavelength range 200 -800 nm from tungsten lamps. Meanwhile, the interaction of functional groups in *Imperata cylindrica* (L.) Raeusch leaf extract with gold colloids was characterized using the Fourier Transform Infra-red Spectroscopy (FTIR) Shimadzu Prestige 2, the source of the Nernst lamp at wavenumber 4000 -400  $\text{cm}^{-1}$ . Furthermore, the morphology of gold nanoparticle was characterized using Transmission Electron Microscopy (TEM) JEM 1400 with a source of energy electrons of 350 KeV. Minewhile, the crystallinity of gold nanoparticles was characterized using X-ray diffraction (XRD) Shimadzu 610 with Cu electron source.

## 2.2. Methods

### 2.2.1. Preparation of *Imperata cylindrica* (L.) Raeusch Leaf Extract

About 10 g of *Imperata cylindrica* (L.) Raeusch leaf was boiled and stirred with 100 mL of distilled water for 1 hour at 60<sup>0</sup> C until the color of the liquid solution changes from watery to light yellow. After that, the yellow colored extract was filtered through Whatman No. 1 filter paper.

### 2.2.2. Synthesis of Gold Nanoparticles

The precursor used was  $\text{HAuCl}_4$  solution with various concentrations of (7, 8, 9  $\times 10^{-4}$  M) respectively and *Imperata cylindrica* (L.) Raeusch leaf extract used stock solution by 5%. About 5 mL  $\text{HAuCl}_4$  solutions was added with 1 mL *Imperata cylindrica* (L.) Raeusch leaf extract (v/v). The reaction was carried out for 24 hours at room temperature until the color changes from yellow to red.

## 3. RESULT AND DISCUSSION

### 3.1. UV-Vis Spectrophotometer

The colloid of gold nanoparticles has a surface plasmon resonance phenomenon which are shown from the absorbance and absorption

values at the wavelength measured by the UV-Vis spectrophotometer. Figure 1 shows the UV-Vis spectra of *Imperata cylindrica* (L.) Raeusch leaf extract and gold nanoparticles that were formed. The absorption peak of 320 nm with an absorbance value of 0.6 in *Imperata cylindrica* (L.) Raeusch leaf extract indicates that there is an active functional group in the secondary metabolites of *Imperata cylindrica* (L.) Raeusch leaf extract which is conjugated.

The addition of *Imperata cylindrica* (L.) Raeusch leaf extract as a natural bio-reducing agent is aimed to reduce  $\text{Au}^{3+}$  to  $\text{Au}^0$  ion. Besides, the functional groups contained the secondary metabolites of *Imperata cylindrica* (L.) Raeusch leaf extract acting as a capping agent and a stabilizer of gold nanoparticles [35].

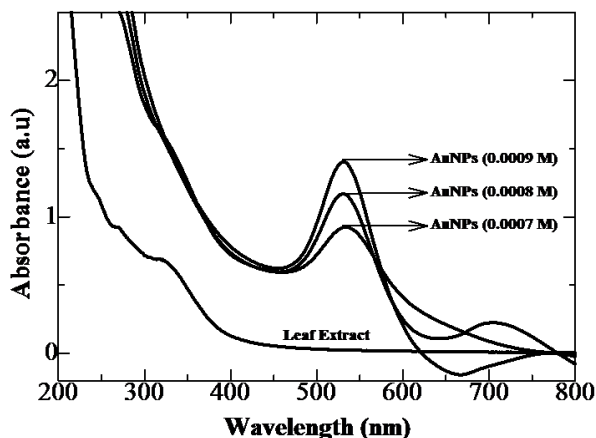


Fig. 1. UV-Vis spectrum of *Imperata cylindrica* (L.) Raeusch and AuNPs

Surface plasmon resonance phenomena of gold nanoparticles at the concentration of  $\text{HAuCl}_4$  solution (7 $\times 10^{-4}$  M; 8 $\times 10^{-4}$  M and 9 $\times 10^{-4}$  M) shows the absorbance value of 0.9; 1.1; and 1.4 with the wavelength value of 532 nm; 530 nm; and 530 nm, respectively. The absorbance value is increased along with the increasing of  $\text{HAuCl}_4$  solution concentration. This indicates the number of gold nanoparticles formed. The wavelength value of gold nanoparticles is related to the size of the particles produced. The lower the result of wavelength value (shift to ultra violet), the smaller size of the nanoparticles. The colloidal spectrum of gold nanoparticles can be seen at 500 -600 nm [36].

### 3.2. FTIR Spectroscopy

HAuCl<sub>4</sub> solutions ( $9 \times 10^{-4}$  M) was used in FTIR analysis. Interaction of active groups with colloid of gold nanoparticles can be seen in the shift of absorption peaks at wavenumbers 3414 cm<sup>-1</sup> to 3404 cm<sup>-1</sup>, 1612 cm<sup>-1</sup> to 1634 cm<sup>-1</sup>, 1382 cm<sup>-1</sup> to 1403 cm<sup>-1</sup>, and 1063 cm<sup>-1</sup> to 1049 cm<sup>-1</sup>. FTIR spectrum of *Imperata cylindrica* (L.) Raeusch leaf extract at wavenumber 3414 cm<sup>-1</sup> indicates the existence of vibration stretching functional groups (–OH), 1612 cm<sup>-1</sup> stretching of carbonyl or carboxylic (C=O), 1382 cm<sup>-1</sup> of bending carboxylic (–O–H), and 1063 cm<sup>-1</sup> in the presence of stretching vibrations (C–N).

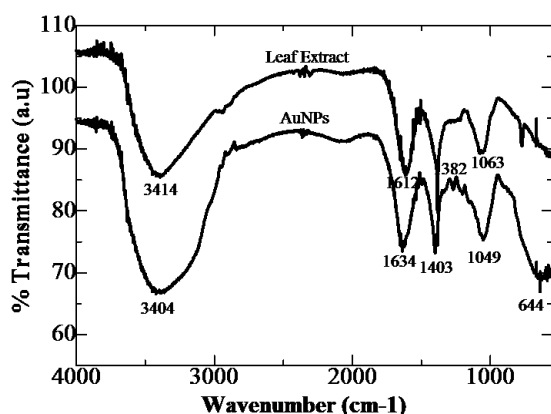


Fig. 2. FTIR spectrum of *Imperata cylindrica* (L.) Raeusch and AuNPs

The results of the FTIR spectrum are similar to previous studies on functional groups in leaf extracts [6]. The existence of a new absorption peak at wave-number 644 cm<sup>-1</sup> predicts the presence of gold metal formed. Based on the FTIR spectrum obtained, the active functional group that acts as a reduction agent for Au<sup>3+</sup> to Au<sup>0</sup> ions and stabilizer is sourced from phenolic compounds (flavonoids), amino acids, proteins and polypeptides [13].

Prediction of reduction mechanism of Au<sup>3+</sup> to Au<sup>0</sup> ions first step from the electrons source derived from the three functional groups hydroxyl (–OH) in phenol on the flavonoid compounds in the *Imperata cylindrica* (L.) Raeusch leaf extract. The functional group of hydroxyl (–O–H), atom of hydrogen undergoes a termination bond and resulting of conjugation

bond in the phenol structure. The three electrons on the three of hydrogen atom are used as a source of electrons in the reduction of Au<sup>3+</sup> to Au<sup>0</sup> ions to colloid shape of gold nanoparticles.

### 3.3. Transmission Electron Microscopy

TEM is used to analyze the shape and size of gold nanoparticles. Gold nanoparticles with a concentration of HAuCl<sub>4</sub>  $9 \times 10^{-4}$  M have a spherical shape and average particle size of 20 nm. There is a density difference between the gold nanoparticles and the capping agent of *Imperata cylindrica* (L.) Raeusch leaf extract. Gold's density is darker than that in capping agents (Figure 3).

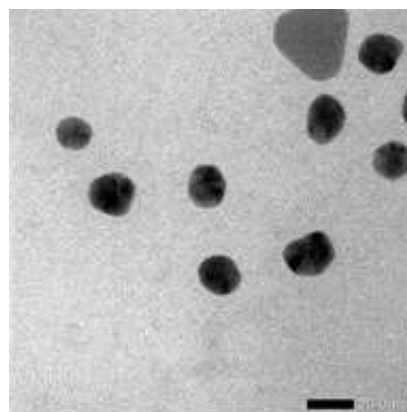


Fig.3. TEM of AuNPs

TEM shows the gold nanoparticles that are synthesized using *Imperata cylindrica* (L.) Raeusch leaf extract. The shape of gold nanoparticles is different at TEM analysis, indicating the functional groups presence of C=C, –OH, and C–N in the secondary metabolites contained in the *Imperata cylindrica* (L.) Raeusch leaf extract which functions as a capping agent.

### 3.4. X-Ray Diffraction

The crystallinity of gold nanoparticles with a concentration of HAuCl<sub>4</sub>  $9 \times 10^{-4}$  M is shown in Figure 4. The XRD results show the peak crystallinity of gold nanoparticles which is typical at 2 theta between 20°–80°.

Diffraction angle of 38.11°; 44.45°; 64.78°; and 77.55° is a form of face-centered cubic (FCC) crystal which has a value of hkl (111),

(200), (220) and (311). This data corresponds to JCPDS No. 01-089-3697.

The higher of  $\text{HAuCl}_4$  solutions concentration used, it is predicted the more of gold nanoparticles will be formed, as well as the crystallinity and the small crystal size of gold nanoparticles. Gold nanoparticles have an average crystal size of 16 nm which is calculated using the Scherrer equation.

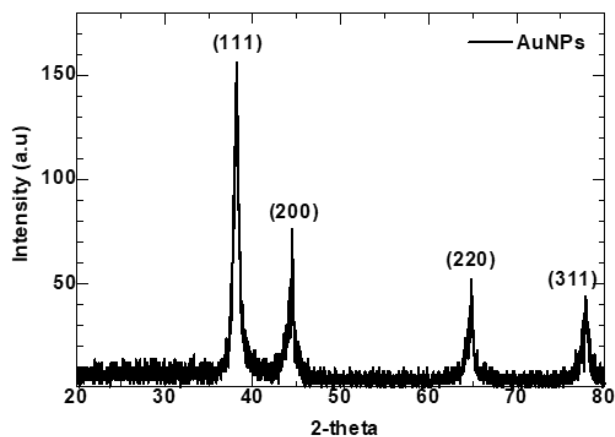


Fig.4. Pattern XRD of AuNPs

#### 4. CONCLUSION

Secondary metabolites contained in the leaf extract of *Imperata cylindrica* (L.) Raeusch can be used as a medium on the synthesis of gold nanoparticles. *Imperata cylindrica* (L.) Raeusch leaf extract has a 5% stock solution concentration. Synthesis of gold nanoparticles was carried out for 24 hours at room temperature. The concentration of  $\text{HAuCl}_4$  solution  $9 \times 10^{-4}$  M has the best results with absorbance value of 1.4 and a wavelength of 530 nm, respectively. The specific peak of gold nanoparticles can be seen at wave-number of  $644 \text{ cm}^{-1}$ . Gold nanoparticles have a crystal size of 16 nm in the form of spherical particles.

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