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EFFECT OF CONCENTRATION OF *IMPERATA CYLINDRICA L* LEAF EXTRACT ON SYNTHESIS PROCESS OF GOLD NANOPARTICLES

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ABSTRACT

EFFECT OF CONCENTRATION OF IMPERATA CYLINDRICA L LEAF EXTRACT ON SYNTHESIS PROCESS OF GOLD NANOPARTICLES. Gold Nanoparticles (GoldNPs) successful was performed using $HAuCl_4$ precursor as Au^{3+} ion source with $7x10^{-4}$ M concentration. The research aims to knows effect of concentration variation of Imperata cylindrica L leaf extract on synthesis process of gold nanoparticles. The research used of green synthesis method. Colloid of nanoparticles which is formed in analyzed using UV-Vis Spectrophotometer, FT-IR Spectroscopy, PSA, PZC, XRD and TEM. The results of synthesis showed the best concentration of *Imperata cilyndrica L* leaf extract at 3,46%, happen a shift of wavelength at UV-Vis from 216 nm to 530 nm with 1.779 absorbance value. The PSA analysis showed a particle size of 51.87 nm and a PZC value of -19.2 mV. The result of FT-IR indicated a shift of wavenumber in the hidroxyl group from 3354 cm^{-1} to 3390 cm^{-1} and showed a interaction of hydroxyl group at imperata cylindrica L leaf extract with Au^{3+} ion. TEM analysis shows the morphology of GoldNPs that spherical shape with a particle size of 20 nm. XRD calculation results show crystallite size of gold nanoparticles is 15.47 nm.

***Keywords**:* Imperata cylindrica L, Gold Nanoparticles (GoldNPs), Green Synthesis

ABSTRAK

PENGARUH KONSENTRASI EKSTRAK DAUN ILALANG *(IMPERATA CYLINDRICA L)* DALAM PROSES SINTESIS NANOPARTIKEL EMAS. Nanopartikel Emas (*GoldNPs*) berhasil dilakukan menggunakan prekursor $HAuCl_4$ sebagai sumber ion Au^{3+} dengan konsentrasi $7x10^{-4}$ M. Tujuan penelitian ini yaitu ingin mengetahui pengaruh variasi konsentrasi ekstrak daun ilalang dalam proses pembentukan nanopartikel emas. Metode yang digunakan pada penelitian ini yaitu metode *green synthesis.* Koloid nanopartikel yang terbentuk di karakterisasi menggunakan Spektrofotometer *UV-Vis*, Spektroskopi *FT-IR, PSA, PZC, XRD* dan *TEM*. Hasil penelitian menunjukkan konsentrasi ekstrak daun ilalang yang terbaik yaitu 3,46% terlihat adanya pergeseran panjang gelombang pada *UV-Vis* dari 216 nm menjadi 530 nm dengan nilai absorbansi tertinggi yaitu 1,779. Analisis *PSA* menunjukkan ukuran rata-rata nanopartikel emas sebesar 51,87 nm dan nilai *PZC* sebesar -19,2 mV. Hasil *FT-IR* menunjukkan adanya interaksi pada gugus hidroksil pada ekstrak daun ilalang dengan ion Au^{3+}, terlihat adanya pergeseran bilangan gelombang dari 3354 cm^{-1} menjadi 3390 cm^{-1}. Berdasarkan analisis *TEM* menunjukkan morfologi nanopartikel emas berbentuk *sphere* dengan ukuran partikel rata-rata 20 nm. Hasil perhitungan *XRD* menunjukkan ukuran kristalit nanopartikel emas sebesar 15,47 nm.

Kata Kunci: *Imperata cylindrica L*, Nanopartikel Emas (*GoldNPs*), *Green Synthesis*

INTRODUCTION

The recent of gold nanoparticles successful in the synthesis with variations particle size between 1-100 nm. Au nanoparticles have several advantages in such fields as catalysts [1], antibacterial [2], sensors [3] and drug delivery [4]. In addition to having superior properties, AuNPs also has a good particle size stability. Capping agent serves as a stabilizer particle size. Capping agent frequently used in the synthesis of AuNPs is polyvinyl pyrrolidone (PVP) [5], Cetyltrimethylammonium bromide/sodium dodecyl sulfate (CTAB / SDS) [6] and alginate [7].

In addition to capping agents, the formation of gold nanoparticles involves reducing compounds. Are usually, the reduction agents in the synthesis of AuNPs used sodium borohydrate ($NaBH_4$) [8], sodium citrate [9]. The use of chemical reducing is very harmful to the environment. To reduce the chemical reduction, the researchers successful to synthesized of gold nanoparticles using green chemistry method by utilizing natural bioreduktor that is environmentally friendly such as leaves extract of Bacopa monnieri extract [10], Rosa rugosa [11], and Euphorbia hirta L [12], Tinospora crispa leaf extract successfully used as reducing agent and capping agent in the synthesis of gold nanoparticles [13]. Secondary metabolites such as alkaloids, flavonoids, tannins, steroids, that contained at plant extracts very important role in the formation of gold nanoparticles. So far there has never been a synthesis of gold nanoparticles using leaf extracts of Imperata cylindrica L.

Imperata cylindrica L is a plant in Indonesia has antioxidant properties and used as a herbal medicine. During this time, the Imperata cylindrica L plant are only used as animal feed and include disturbing weeds. The result of phytochemical test showed that secondary metabolites content at Imperata cylindrica L leaves is alkaloid, flavonoid, Tannins, steroids. Contained a flavonoid and the secondary metabolites the functioning act as reduction ion Au^{3+} into Au^0 [13]. From the background and literature, the research aims to knows effect of concentration variation of Imperata cylindrica L leaf extract on synthesis process of gold nanoparticles. Utilization of Imperata cylindrica L leaf extract is expected to be used as reductant on synthesis process of gold nanoparticles.

EXPERIMENTAL METHOD

Materials

Synthesis of gold nanoparticles using $HAuCl_4$ precursor, Imperata cylindrica L leaf extract, methanol, n-hexane, ethyl acetate pro analysis and aquadest. The sample using Imperata cylindrica L (ICL) were taken from Universitas Indonesia area.

Methods

As much 50 g ICL washed with water and dried for 7 days. The ICL were crushed using a blender to produce a powder. As much 25 g ICL powder macerated using 100 mL of methanol for 7 days. Filtrate the result of maceration in the extracted using n-hexane, ethyl acetate and water. The results of water fraction ICL leaf in the phytochemical test to determine of secondary metabolites such as alkaloids, flavonoids, steroids, tannins, saponins and polyphenols.

Taken as much 5 ml solution $HAuCl_4$ $7x10^{-4}$ M was added 1 mL of ICL extract solution water fraction (extract concentration variations code 1-5 is 0.692; 1.384; 2.076; 2.768; 3.46%). The solution was reacted at room light for 1 hour. The formation of gold nanoparticles be marked by color changes from yellow to red purple.

Characterization of gold nanoparticles using Shimadzu UV-Vis Spectrophotometer 2600 with wavelength from 200-800 nm set and equipped with wolfram lamp as a light source, functional group contained in extracts of Imperata cylindrica L leaf extract were characterized using Shimadzu Fourier Transform Infrared (FT-IR) Spectroscopy Prestige 21 with wavenumber from 400-4000 cm^{-1} set, and used a nernst lamp. The size distribution and particle charge GoldNPs were characterized using Particle Size Analyzer (PSA) and Zeta Potential (PZC) Malvern ZEN 1600 with dynamic light scattering system. The shape morphology and particle size of GoldNPs characterized by Transmission Electron Microscopy (TEM) JEM 1400 and using 350 keV of electron beam energy. Crystallite size and crystalline GoldNPs in characterization with Shimadzu X-Ray Diffraction (XRD) 610 with Cobalt us a electron source.

RESULTS AND DISCUSSION

The phytochemical test showed a positive flavonoids, polyphenols and tannins. The synthesis

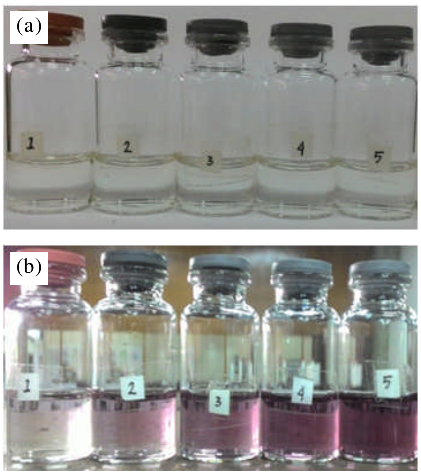

Figure 1. GoldNPs with a variety of extracts from left to right 1-5 (a) before and (b) after reaction.

of gold nanoparticles showed a color change from yellow to red purple it indicates the formation of gold nanoparticles. Figure 1 shows the conditions synthesis process of gold nanoparticles. GoldNPs formation process is a reduction and oxidation. The mechanism is Equalition (1).

$$Au^{3+} + 3e^{-} \longrightarrow Au^{0} \quad \text{.........................} \quad (1)$$

$HAuCl_4$ solution used as the Au^{3+} ion source and has a 216 nm absorbance value [14]. The reaction of gold nanoparticles formed for 1 hour at room temperature. The best concentration of Imperata cylindrica L leaf extract is 3,46%. The color is more purple seen of Figure 1b code 5. The absorbance measurements using UV-Vis spectrophotometer was performed in the wavelength range 200-800 nm. Figure 2 shows the UV-Vis spectrum of GoldNPs colloid synthesized with various concentrations of ICL extracts. The qualitative secondary metabolites contained the extracts of ICL have UV-Vis spectrum at wavelength of about 250-350 nm. Flavonoid compounds containing aromatic conjugated system and can show strong absorption band in the UV region. From UV-Vis spectra (Figure 2(a)) shows the concentration of ICL extract are best a value of 3.46% because has absorbance value of 1,779 and a wavelength of 530 nm. Synthesis process of gold nanoparticles influenced by

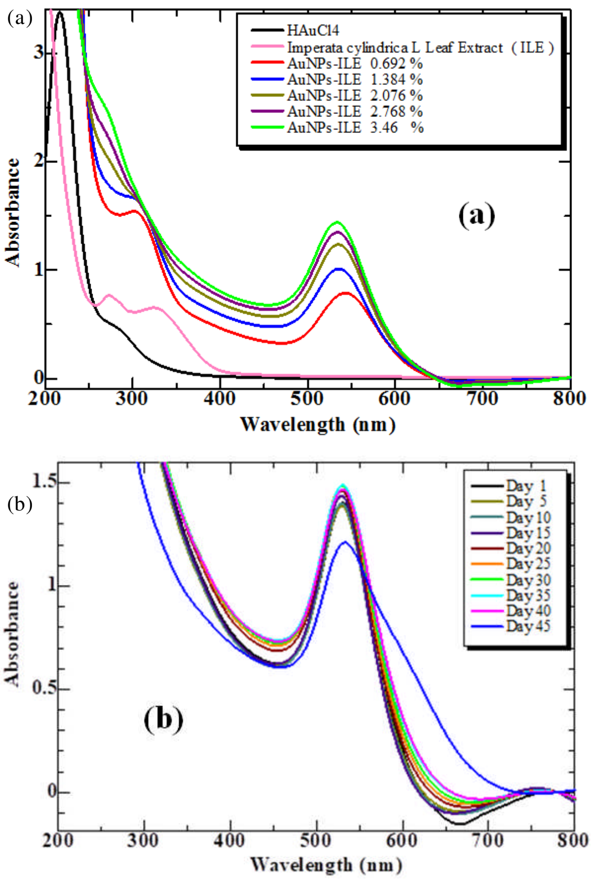

Figure 2. UV-Vis spectra GoldNPs (a) various concentrations of the extract and (b) the stability of GoldNPs.

reaction time. The longer of reaction time at synthesis process of gold nanoparticles, increasingly much of nanoparticles are formed and the higher at absorbance value [15]. The best results are further characterized.

Gold nanoparticles have Surface Plasmone Resonance value when interaction with visible light [16]. Figure 2(b) shows the gold nanoparticles formed as stable for 40 days. Day 45 absorbance significant decline, indicated agglomeration happen at gold nanoparticles.

FT-IR shows the presence of -OH groups at ICL extracts can ion reduction Au^{3+} into Au^0. In addition, the hydroxyl groups may act as a capping agent in the formation of gold nanoparticles. Indicated by the shift of the hidroxyl group at wavenumber 3354 cm^{-1} to 3390 cm^{-1} shown in Figure 3(a). The presence of more electronegative oxygen at carbonyl group that interaction with Au^0 induce a shift wavenumber of 1720 cm^{-1} to 1751 cm^{-1} so that it takes more energy to perform vibration in group C=O [17].

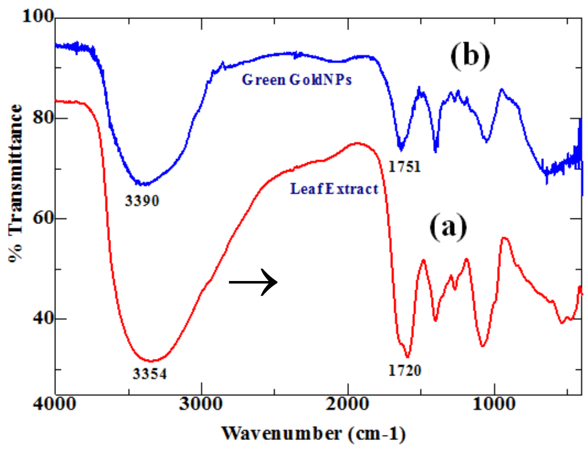

Figure 3. FT-IR spectra of (a) Imperata cylindrica L leaf extract and (b) GoldNPs.

TEM characterization was performed to determine the morphological form, density difference and GoldNPs particle size.

The analysis using TEM on GoldNPs sample which was successfully synthesized using ICL leaf extract with concentration of 3.46% and $HAuCl_4$ $7x10^{-4}$ M is shown in Figure 4. The TEM results show that the

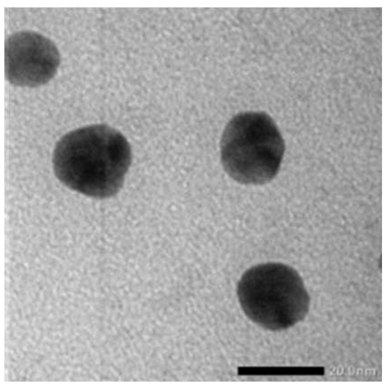

Figure 4. TEM of GoldNPs

resulting GoldNPs particles are sphere and have good homogeneity. The morphology of GoldNPs is strongly influenced by the diversity of secondary metabolites in the extract. The GoldNPs core has an evenly distributed active side that causes the GoldNPs to be spherical shape [18]. The existence of the intermediate particles force (shown on the PSA results that have a negative potential zeta value) will produce a good distribution and will reduce the occurrence of agglomeration [19]. GoldNPs have higher density than capping agent. Seen GoldNPs have darker images because they have a greater density than a capping agent and have a particle size of ± 20 nm at 150000x times magnifications.

Measurements using PSA and Zeta Potential function to know the average distribution of particle size and charge on a particle system. PSA showed the one peaks which states the intensity of distribution particle size (Figure 5(a)). At the peak has a particle size of 51.87 nm with 70.3% intensity. The capping agent from Imperata cylindrica L can be used as an stabilizing of GoldNPs [20].

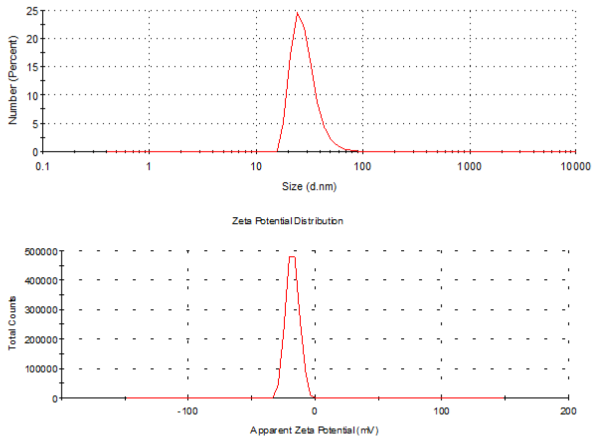

Figure 5. Spectrum image of GoldNPs (a) PSA, (b) PZC.

Zeta potential is measured using a zetasizer and zeta potential value associated with the stability of a dispersed particle. Result of PZC characterization showed the potential value of -19.2 mV for gold nanoparticles (Figure 5(b)). This indicated the repulsion style force between of particles of -19.2 mV. The more negative results of potential zeta value the stronger particles force, the less agglomeration occurs [21].

The XRD showed a peak appearing at 2θ : 38.12; 44.44; 64.79 and 77.79 respectively (Figure 6). The value of corresponding from the JCPDS No. 65-2870. Gold which has a miller index (111), (200), (220) and (311) and 2θ is 38.18; 44.39; 64,65; 77.54 respectively [22]. Based on calculations using Scherrer equation obtained an average crystallite size of gold nanoparticles is ± 15.47 nm. Effect of Imperata cylindrica L proving the structure of GoldNPs to be Face Center Cubic (FCC) [23].

Further purification of the flavonoid fraction to strengthen the stability of gold nanoparticles.

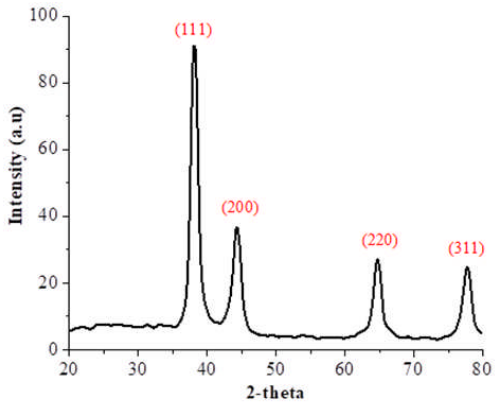

Figure 6. Pattern XRD of GoldNPs

CONCLUSION

Variations of Imperata cylindrica L leaf extracts are very influential on the process of synthesis of gold nanoparticles. The higher the ICL concentration given, the more Au^{3+} ions are reduced to Au^0 (GoldNPs). The color change turns purple after the addition of ICL leaf extract for 1 hour reacts. Seen from result UV-Vis highest absorbance value that is on giving of extract 3,46%. Leaf extract also serves as a capping agent for gold nanoparticles no agglomeration occurs. The value of -19.2 mV from PZC indicates a repulsive force between particles causing nanoparticles to not occur agglomeration and of the TEM image of the particle is well distributed with an average particle size of 20 nm. The gold nanoparticles have an FCC crystal structure. From the existing research data can be concluded that gold nanoparticles successfully synthesized using Imperata cilyndrica L leaf extracts.

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