

Cite this: *Indo. Chim. Acta.*, 2020, 13, 2.

Received Date:
6th December 2020
Accepted Date:
30th December 2020

Keywords:

bay leaf extract;
gold nanoparticles;
sensors;
glucose;
biosynthesis.

DOI: <http://dx.doi.org/10.20956/ica.v13i2.11798>

Synthesis and Characterization of Gold Nanoparticles using the Bioreductor Bay Leaf (*Syzygium polyanthum*)

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Abstract. Bay leaf extract has been used successfully in the synthesis of gold nanoparticles. This is indicated by the color change from the gold solution which is yellow to burgundy after the addition of the bay leaf extract and the resulting wavelength is in the range of 500 - 550 nm which indicates that gold nanoparticles have been formed. The growth and stability of the resulting gold nanoparticles were observed using UV-Vis Spectroscopy. Particle Size Analyzer (PSA), Scanning Electron Microscope (SEM), X-Ray Spectrometer Diffractometer (XRD) and Fourir Transform InfraRed (FTIR) were used to characterize Nanoparticles are produced before they are made into blood sugar nanosensors. The maximum wavelength was obtained at a wave of 533 nm using a UV-Vis spectrophotometer. The particle size was determined using PSA with average particle a size distribution of 15.49 nm.

Introduction

Diabetes mellitus (DM) is defined as a chronic, multi-etiological metabolic disease characterized by increased blood glucose levels (fasting glucose ≥ 126 mg / dl or glucose ≥ 200 mg / dl) accompanied by disorders of carbohydrate, protein and lipid metabolism as a result of insufficiency. insulin function (Parisa, 2016). Diabetes mellitus is a chronic disease characterized by high blood sugar levels (Ensafi *et al*, 2017). The World Health Organization (WHO) predicts an increase in the number of people with diabetes mellitus in Indonesia from 8.4 million in 2000 to around 21.3 million in 2030. The International Diabetes Federation (IDF) predicts an increase in the number of people with diabetes mellitus (DM) in Indonesia from 9.1 million in 2014 to 14.1 million in 2035 (Decroli, 2019).

For people with diabetes mellitus, keeping blood glucose levels close to normal can reduce the risk of further complications. To maintain blood glucose levels in safe areas, tools to monitor blood glucose are needed. Currently, sensors for this purpose are so expensive that many people cannot afford them.

Therefore, intensive research is needed to develop a compliance sensor that is cheap, accurate and easy to use (Baghayeri *et al* 2016, Ensafi *et al*, 2017, Peng *et al*, 2016, Wu *et al*, 2016, Bijang *et al*, 2015, Misriyani, 2015).

In recent years, precious metal nanoparticles have attracted the attention of researchers because of their potential applications in the fields of optics, electronics, biological sensors and catalysts. Precious metal nanoparticles that are often used in various applications are gold nanoparticles because precious metal nanoparticles have a very high extinction coefficient and optical properties that depend on particle size and shape, medium dielectric constant, composition and distance between particles (Garcia *et al*, 2016, Murugavelu *et al.*, 2016, Tael *et al*, 2017). Broadly speaking, the synthesis of nanoparticles is carried out using the top-down method (physics) and the bottom-up method (chemistry). The top-down method reduces the size of metal solids to nano size mechanically, whereas the bottom-up method is done by dissolving it with a reducing and stabilizing agent to convert it into nano form (Iswarya *et al*, 2016, Zhou *et al*, 2016, Wan *et al*, 2017).

Seeing the risks and environmental impacts that can be caused as well as the high costs of the top-down and bottom-

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up method of nanoparticle synthesis, new innovations are needed to synthesize nanoparticles in an environmentally friendly and low cost manner. It has been found a way to synthesize nanoparticles by utilizing plants or microorganisms as reducing agents to produce nanoparticles which is known as nanoparticle biosynthesis.

One of the herbal plants considered to have potential as an antihyperglycemia is bay leaf (*Syzygium polyanthum*). Bay leaves are common plants and are easily found in Indonesia. Several studies have shown that the main content of flavonoid compounds in bay leaf extract can significantly reduce blood glucose levels. The flavonoid glycoside compounds found in bay leaves function as a hydroxyl radical scavenger so that they can prevent diabetogenic action (Parisa, 2016)

Seeing the potential and prospects for the production of gold nanoparticles using bay leaves (*Syzygium polyanthum*) and diabetes mellitus, this research will develop the synthesis and characterization of gold nanoparticles from bay leaves (*Syzygium polyanthum*) as a blood sugar level sensor. The gold nanoparticles were then applied as a sensor for blood sugar levels. Furthermore, it is continued to carry out selectivity tests, detection limits, work ranges, response speed and economic calculations as a baseline for feasibility of production on an industrial scale.

Experimental

Material and Methods

This Bay leaves, glucose anhydrid, aquabides, distilled water, Whatmann paper No.42, pure gold metal, aquaregia, tetrachloroauric acid (HAuCl_4), NaOH, and polyacrylic acid.

Tools used in research this is a commonly used glassware in the laboratory, UV-Vis spectrophotometer (Shimadzu UV-2600), Fourier Transform InfraRed (FTIR) Shimadzu IR Prestige21, X-ray Diffraction (XRD) Shimadzu 7000, Particle Size Analysis (PSA) Vasco, Scanning electron microscope (SEM) JEOL-JSM-6510LV, hotplate, multistireer, centrifugation, freeze dryer and analytical balance.

Experimental Procedures

Bay Leaf Preparation and Extract

The leaves are taken and then washed with distilled water and dried until the washing water flows. After that, the leaves are evenly cut 2 cm x 2 cm and weighed 10 grams, then boiled with 50 mL of distilled water in 500 mL of Erlenmeyer. Then the stew is allowed to boil for 5 minutes. After that it was cooled to room temperature, the boiled water was poured and filtered using Whatman No 42 filter paper. The extract was stored in the refrigerator when not

in use. Furthermore, bay leaf extract can be used for the synthesis of gold nanoparticles.

Manufacture of 1000 ppm HAuCl_4 solution

1000 ppm HAuCl_4 stock solution was prepared by weighing 1 gram of gold metal then dissolving it in 8 mL of aquaregia while heating. Furthermore, the solution is added with aquabides into a 1000 mL volumetric flask until the miniscus mark, then the solution is homogenized

Synthesis of Gold Nanoparticles

The synthesis of gold nanoparticles was carried out by mixing 40 mL of 50 ppm HAuCl_4 solution with 2 mL of bay leaf boiled water extract. Furthermore, 10 mL of 1% PAA was added to the solution then the mixture was stirred with a magnetic stirrer for 2 hours. The formation of gold nanoparticles is characterized by changing the solution from yellow to burgundy color. Furthermore, the gold nanoparticle solution formed is catalyzed by UV-Vis spectroscopy after 1 hour (Yasser, 2017).

Characterization with a UV-Vis Spectrophotometer and PSA

Nanoparticle solutions were analyzed using a UV-Vis spectrophotometer at intervals of 1, 2, 3, and 4 days. The colloid absorbance of the gold nanoparticles was analyzed with a maximum wavelength in the 200-700 nm region. The gold nanoparticle solution was then analyzed for particle size using PSA. PSA was used to determine the average size of gold nanoparticles.

Characterization by X-Ray Diffraction (XRD), Fourier Transform Infrared (FTIR) and Scanning Electron Microscopy (SEM)

Gold nanoparticle colloids have been prepared beforehand, centrifuged at a speed of 10,000 rpm for 30 minutes at 27 °C for 10-20 times. Then the colloid was dried with a freeze dryer for 24 hours. Furthermore, gold nanoparticles were taken to be characterized by X-Ray Diffraction (XRD), Fourier Transform Infrared (FTIR) and Scanning Electron Microscopy (SEM).

Result and Discussion

Characterization with a UV-Vis Spectrophotometer

The nanoparticles that had been synthesized were then analyzed using a UV-Vis spectrophotometer at intervals of 1, 2, 3, and 4 days. The absorbance of gold nanoparticle solutions was analyzed with a maximum wavelength in the visible area, namely 400-700 nm. The table of the results of the UV-Vis spectrophotometer analysis is as follows:

UV-Vis absorption analysis began by measuring the maximum wavelength of HAuCl_4 50 ppm, PAA 1%, bay leaf



extract (*Syzygium polyanthum*), and AuNPs after 5 minutes of synthesis time. The results of UV-Vis spectrum measurements shown in Table 1 indicate that gold nanoparticles were successfully synthesized due to a bathochromic change, namely a shift in the maximum wavelength of H₂AuCl₄ from 308 nm to 533 nm. In general, the absorption that appears at a wavelength of 500–550 nm is a typical peak of gold nanoparticles (Rohiman *et al.*, 2014).

Table 1. Absorption UV-Vis

Sample	Wavelength (nm)	Absorbance
PAA 1%	215	0.539
Extract	313.50	10.000
H ₂ AuCl ₄ 50 ppm	308.00	0.358
AuNPs	533.80	0.611

The analysis using a UV-Vis spectrophotometer was continued to see the growth pattern and stability of the synthesized gold nanoparticles with day variations and the absorption was obtained at a wavelength of 533.80–534.0 nm shown in **Figure 1**. Based on the research of Rohiman *et al.* (2014), it was concluded that Typical peaks of gold.

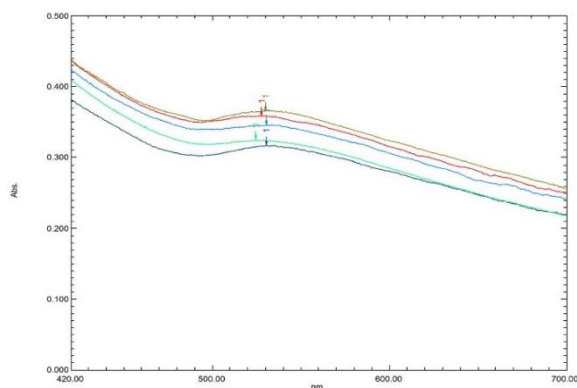


Figure 1. UV-Vis spectrum of synthesized gold nanoparticles based on day variations.

The analysis using a UV-Vis spectrophotometer was continued to see the growth pattern and stability of the synthesized gold nanoparticles with day variations and the absorption was obtained at a wavelength of 533.80–534.0 nm shown in **Figure 1**. Based on the research of Rohiman *et al.* (2014), it was concluded that Typical peaks of gold.

Nanoparticles occur at a wavelength of 500–550 nm, this proves that gold nanoparticle formation and absorbance increases with increasing interaction time which is the gold nanoparticle absorption region.

Characterization of Gold Nanoparticles by FTIR

FTIR characterization aims to identify possible functional groups in the water extract of bay leaves that have a role in reducing Au³⁺ ions to Au⁰. IR absorption measurements were carried out on bay leaf sample extracts before and after the reaction took place to form nanoparticles. The IR spectrum of binahong leaf extract and gold nanoparticles shows the typical absorption for several functional groups shown in **Figure 2**.

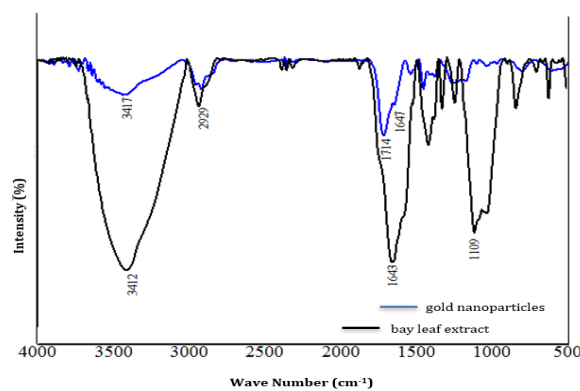


Figure 2. FTIR Spectrum

The FTIR spectrum of the bay leaf extract shown in **Figure 2**. shows a broad and strong band at 3412 cm⁻¹ absorption is a characterization of the O-H group. Another IR band appeared at 1643 cm⁻¹ indicating the presence of an amide group which was strengthened by the appearance of 1109 cm⁻¹ absorption which is the C-N group contained in the bay leaf extract.

The IR band of the gold nanoparticle reaction results in **Figure 9** shows that there is a shift in the wave number from 3412 cm⁻¹ to 3417 cm⁻¹, which indicates that there is an interaction between O-H groups and Au. Another IR band undergoes a change from 1643 cm⁻¹ to 1647 cm⁻¹ originating from the C = O amide group, and there is a C = O ketone group on the gold nanoparticles absorbed 1714 cm⁻¹. This indicates that the compounds containing these groups in the bay leaf extract are responsible for reducing gold to gold nanoparticles.

Characterization of Gold Nanoparticles using XRD and PSA

The synthesized powder of gold nanoparticles was analyzed using XRD. Characterization using XRD was carried out to identify the crystalline phase in the material by determining the lattice structure parameters and to obtain the particle size (Marlinda, 2016). The characteristic peaks observed in the XRD AuNPs pattern confirm the presence of the gold nanoparticles shown in **Figure 3**.

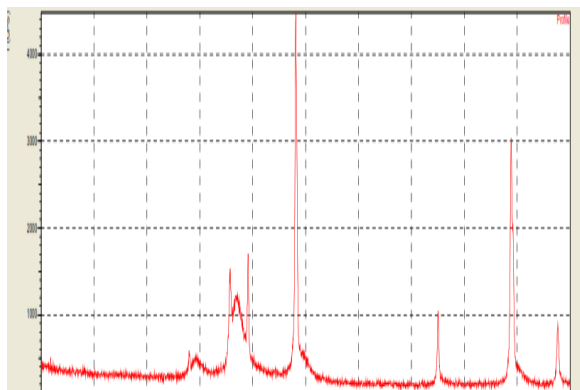


Figure 3. XRD Diffraction Patterns of gold nanoparticles

Based on **Figure 3.**, it can be seen that the diffraction peaks are at angles of 2θ 44.06° , 64.42° , and 39.53° . The respective peaks obtained correspond to the fields (1 1 1), (2 0 0), and (3 1 1) reported in the Joint Committee on Powder Diffraction Standards (JCPDS) for gold metal (Au) standards. This XRD pattern shows that the gold nanoparticles produced are essentially crystalline. The approximate size of the gold particles can be calculated using the Debye-Scherrer equation. Based on the calculation using this equation, the average size of the particles is 13.35 nm, while the measurement using the Particle Size Analyzer (PSA) is not far, which is 15.49, which means that gold nanoparticles have been successfully synthesized.

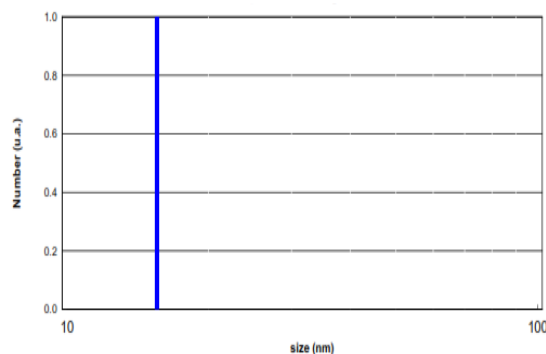


Figure 4. PSA analysis results for gold nanoparticles

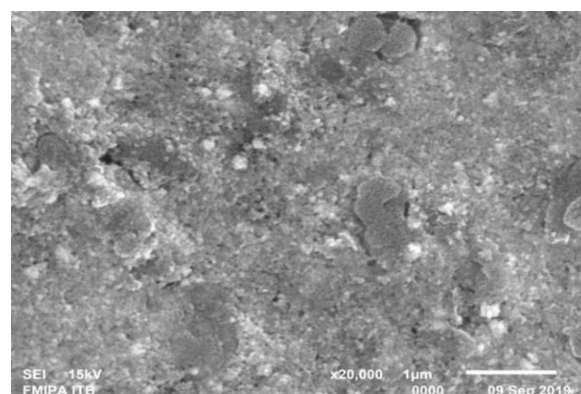
Characterization using PSA aims to determine the diameter size and distribution of gold nanoparticles in the sample. In measuring the PSA, three data were obtained, namely the intensity, number and volume of sample distribution so that it could describe the overall condition of the sample (Nikmatin *et al*, 2011).

The results of measuring gold nanoparticles using PSA in the sample are shown in **Figure 4.** The results obtained

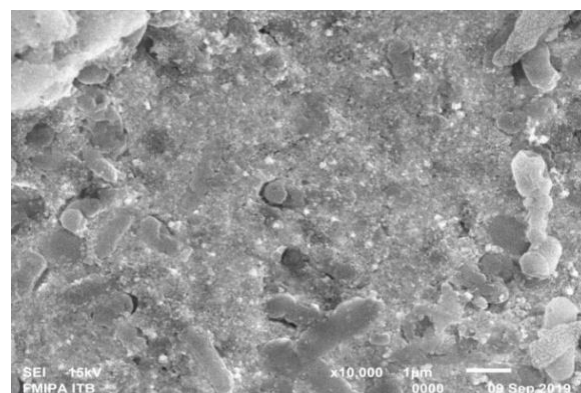
indicate that the diameter size of gold nanoparticles is 15.49 nm. This size indicates that the synthesized particles have included the proper size of the nanoparticles. According to Nagarajan (2018) the size of the nanoparticles is on a scale of 0-100 nm.

Characterization of Gold Nanoparticles by SEM

SEM characterization aims to determine the surface morphology of gold nanoparticles. The enlargement of the bio-reduced gold nanoparticle sample image was carried out on a scale of $10\ \mu\text{m}$ and $20\ \mu\text{m}$ with an HV of 15 kV as shown in Figure 5. Figures 5 a and b show that the resulting nanoparticles have a round and non-uniform shape. This is in accordance with the research of Ahmad and Ikram (2015) that gold nanoparticles have varying structures. In Figure 5, it can be seen that the clumps began to form, indicating that agglomeration has occurred in the sample, this may be due to the long storage of the sample, then measurements are taken.



(a)



(b)

Figure 5. Results of analysis of gold nanoparticle samples by SEM at (a) a scale of $20\ \mu\text{m}$ and (b) $10\ \mu\text{m}$ scale

Conclusion

Based on the research that has been done, it can be ignored that the bay leaf extract is a gold nanoparticle

biosynthetic agent. The results of the research on the characterization of gold nanoparticles using UV-Vis spectroscopy after 5 minutes were at a wavelength of 533 nm. XRD characterization showed that the synthesized product was a crystal of gold nanoparticles with a particle size distribution of 13.35 nm. SEM results showed evidence of gold nanoparticles having a non-uniform surface structure and a spherical shape. Characterization by FTIR showed the synthesis of gold nanoparticles of tannin compounds contained in the extract of bay leaf (Synthesis and Characterization of Gold Nanoparticles using the Bioreductor Bay Leaf (*Syzygium polyanthum*) The average size of the results of characterization of gold nanoparticles with PSA was 15.49 nm.

Conflict of Interest

No potential conflict of interest was reported by the authors

Acknowledgements

Thank you to the Hasanuddin University Research and Community Service Institute (LP2M-UH) for research grants under the Academic Advisory Lecturer Research (PDPA) scheme for 2020. The same is conveyed to UNHAS Undergraduate Chemistry Students and Analysts in the field of Analytical Chemistry, Chemical Laboratory Integrated, Analytical Chemistry Laboratory, UNHAS Chemistry Department, Bogor Agricultural University Chemistry Department and Sepuluh Nopember Institute of Technology Department of Mechanical Engineering for all their assistance.

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