Molekul, Vol. 18. No. 2, July 2023: 236 – 242

MOLEKUL
eISSN: 2503-0310

Articles
https://doi.org/10.20884/1.jm.2023.18.2.6972

Green Synthesis of Gold Nanoparticles using *Peronema canescens* Leaves Extract and Their Catalytic Performance for Dyes and Nitro Compounds

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Received November 03, 2022; **Accepted** May 05, 2023; **Available online** July 20, 2023

ABSTRACT. The most interesting and well-known research in the field of gold nanomaterials synthesis is the use of "green chemistry" to prepare gold nanoparticles (AuNPs). In this study, *Peronema canescens* leaf extract was used as the synthesis medium to successfully produce AuNPs in a way that was cheap, quick, and good for the environment. A UV-visible spectrophotometer, particle size analysis (PSA), and transmission electron microscopy (TEM) were used to find out more about the AuNPs that were prepared. The UV-visible spectrophotometer showed a surface plasmon resonance peak at 532 nm, which proves that AuNPs exist in the solution. TEM and PSA both showed that the AuNPs were mostly spherical and had an average diameter of 14.9 nm, respectively. In the presence of $NaBH_4$, the AuNPs were found to speed up the reduction of rhodamine B (RhB), metanil yellow (MY), and 4-nitrophenol (4-NP). The results show that the AuNPs that were prepared in a new way worked very well and could be used in catalysis.

Keywords: *Peronema canescens,* gold nanoparticles, green synthesis, catalytic activity, water pollutants

INTRODUCTION

Nanotechnology widely has many technologies in various fields of human life, including the synthesis of nanoscale materials. In addition, nanoscale materials offer shapes with various dimensions, one of which is the right solution to the issue of environmental pollution, such as water pollution. Efforts and innovations continue to be made to invent the right solution to reduce the impact of water pollutants, with the main condition that the solutions offered must consider the use of non-toxic, simple, time-saving, and eco-friendly materials.

The use of natural materials in the biosynthesis of gold nanoparticles (AuNPs), such as flower extract of *Ipomoea pescaprae* L. Sweet is proven to be able to produce spherical AuNPs with a size of 16.3 nm (Falahudin et al., 2020). Additionally, another study reported that fruit extracts from *Phyllanthus emblica* (Wang et al., 2021) and *Olea europaea* (Awad et al., 2019) were successfully used as a medium for the synthesis of AuNPs with triangular and irregular forms, respectively. Another potential use of bacteria types *Bacillus stearothermophilus* (Luo et al., 2014) and *Mariannaea* sp. (Pei et al., 2017) was successfully used as a bio-reduction agent in the production of AuNPs. Extract from *Egregia* sp. algae powder has good ability as a synthesis medium for AuNPs with high stability up to 4 weeks (Colin et al., 2018). The advantage of using the biosynthetic method in synthesizing AuNPs is that the formation process of AuNPs can be observed with the naked eye by changing the color of the solution to ruby red (Islam, 2019).

Technology for wastewater treatment in the degradation and removal of water pollutants continues to invent and design. In the few years, biosynthesis AuNPs have high potential and good catalytic performance in degrading water pollutants, such as methylene blue, congo red (López-Miranda et al., 2019), methyl orange, rhodamine B (Kim et al., 2021), and nitro compounds (Doan et al., 2020). This research shows that there is a negative impact caused by synthetic dye waste that is produced on the environmental ecosystem, especially the biota that lives around the water ecosystem. The inhibition of microbial water growth and plant fertility is an early marker because of bioaccumulation of synthetic dyes, then a serious threat lurks and affects human health. Early symptoms in humans are characterized by mild symptoms such as skin and eye irritation, then in the long-term cause chromosomal aberrations (Lellis et al., 2019). It seems like synthetic dyes, the massive use of nitro group compounds in the industrial world raises concerns about the impact of the waste generated. The results of testing for 2,4,6-trinitrotoluene group nitro compounds in test animals showed reproductive disorders which were characterized by reduced levels of the hormone testosterone and semen in the serum (Kovacic & Somanathan, 2014).

Due to the technological boom, chemical, physical, and biological approaches are widely used in synthesizing and producing AuNPs. The novelty of this study is in the utilization of green chemistry approaches by using bioactive compounds in *Peronema canescens* leaves extract as a medium to produce eco-friendly AuNPs, low toxicity, economical, efficient, and simple when compared to prior methods. Furthermore, the bioactive compounds contained in *P. cenescens* extract have a double function as bioreduction and stabilizing agents. The reduction of synthetic dyes and 4-NP by sodium borohydride was evaluated to determine the performance of AuNPs' catalytic activity.

EXPERIMENTAL SECTION

Chemical and Instrument

Tetrachloroauric acid (Sigma Aldrich) was used as a precursor in the synthesis of AuNPs. *P. canescens* leaves extract was used as a bioreduction and capping agent, which was obtained at the University of Bengkulu, Indonesia. Metanil yellow (MY), rhodamine B (RhB), and 4-nitrophenol (4-NP) as models of water pollutants were purchased from Sigma Aldrich and demineralized water.

Agilent carry 60 UV-visible spectrophotometer was used to detect surface plasmon resonance (SPR) peaks and catalytic activity of AuNPs. Particle size analyzer (PSA) (DelsaTMNano – Beckman Coulter Inc.) and transmission electron microscope (JEOL JEM 1400) were used to determine the mean diameter and morphology of AuNPs synthesized by *P.canescens* leaves extract. Transmission electron microscopy (TEM) was used to investigate the shape and size of the nanoparticles that were synthesized.

Preparation of *P. canescens* Leaves Extract

The washed *P. canescens* leaves were dried at room temperature for 1 week. Furthermore, the dried *P. canescens* leaves, cut into small pieces (1 gram) were put into an Erlenmeyer filled with demineralized water (100 ml). The extraction process for *P. canescens* leaves is conducted for 15 minutes at 60 °C while stirring using a magnetic stirrer (Magnetic stirrer SH-3) at around 300 rpm, then the extract is filtered and stored in the refrigerator for further use (Yudha et al., 2021).

Synthesis of Gold Nanoparticles (AuPNs)

The synthesis of AuNPs on the medium of *P. canescens* leaves extract was conducted at room temperature, 10 ml extract of *P. canescens* leaves into the sample bottle, then slowly inserting 0.5 ml of 0.01 M $HAuCl_4$ into the sample bottle containing the extract. The color change that occurs in the synthesis solution from yellow (extract) to violet (AuNPs), is an early indication of the formation of AuNPs (Falahudin et al., 2020).

Activity Catalytic of Gold Nanoparticles (AuPNs)

The catalytic performance of AuNPs in degrading water pollution was tested using a solution of dyes (RhB, MY) and 4-NP. Dyes solution (RhB and MY) was used as the first test solution as water pollutants, 10 mg/L dyes solution was added to the sample bottle, then 500 μL 0.06 M $NaBH_4$ was slowly added to the dye's solution. Furthermore, 100 μL of AuNPs catalyst was added to the solution while stirring at room temperature, and the catalytic activity of AuNPs was monitored using a UV-Visible spectrophotometer, for 15 minutes reaction with a measurement time interval of every 2 minutes.

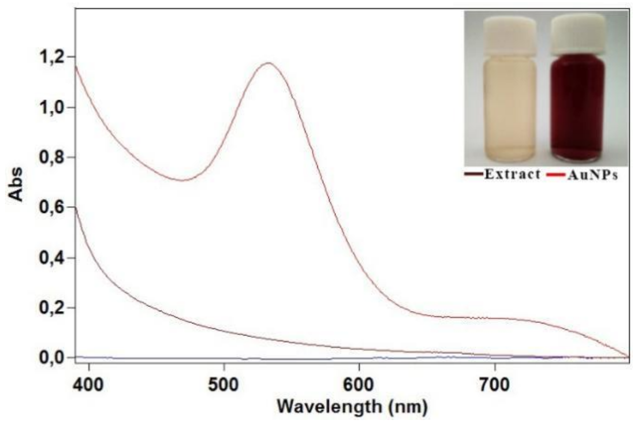
4-nitrophenol solution 2 mM represents a class of nitro compounds as a second model of water pollutants. Solution A was prepared by 10 ml of Aqua DM into a sample bottle, slowly adding 1 ml of 4-NP solution and stirring at room temperature. Then, 5 mL of 0.03 M $NaBH_4$ was added to the 4-NP solution, and solution B was prepared with the combination of solution A, followed by the addition of 150 μL AuNPs while stirring at room temperature. The catalytic activity was measured every 2 minutes using a UV-visible spectrophotometer for 15 minutes of reaction.

RESULTS AND DISCUSSION

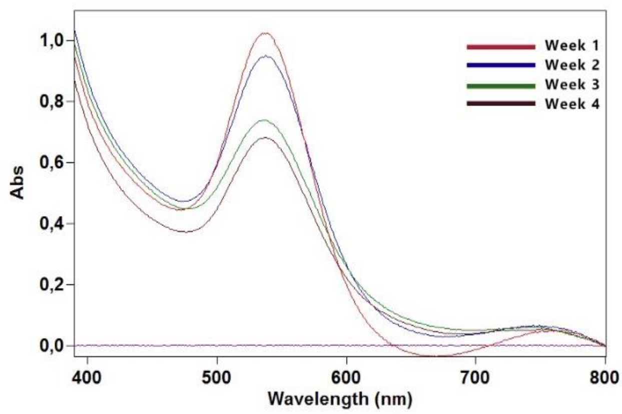
AuNPs have been successfully synthesized via a biosynthesis approach using *P. canescens* leaves extract as a medium synthesis. The process of forming AuNPs can be observed visually by the color change in the synthesis solution. **Figure 1a**, It appears that there is a color transition from yellow (extract) to purple (AuNPs), the phenomenon of changing the color of the solution is an early indication of the formation of AuNPs (Singh et al., 2018) followed by UV-visible spectrophotometer analysis. The UV-visible spectrophotometer analysis is proven and useful for studying metal nanoparticles, including AuNPs. Analysis using a UV-visible spectrophotometer showed the formation of specific peaks in the visible region at a wavelength at 523 nm (González-Ballesteros et al., 2017). This event is known as the surface plasmon resonance phenomenon and is a special phenomenon of noble metal nanoparticles that generates strong electromagnetic fields at the particle surface, increasing all radiation properties such as scattering and absorption (Huang & El-Sayed, 2010). The presence of a specific absorption peak indicates that *P. canescens* leaves extract has the ability as a bioreduction by reducing Au^{+3} to Au^0 , As evidenced by the peak shift from 310 nm ($HAuCl_4.3H_2O$) (Chan et al., 2020) to 523 nm (AuNPs). These results conform to previous research showing that the content of *P. canescens* leaves extract such as alkaloids, terpenoids-steroids, flavonoids, and tannins (Ibrahim & Had, 2012), plays a key role as a bioreduction and capping agent in the formation of AuNPs (Ghazal et al., 2018).

The ability of *P. canescens* leaves extract as a medium for the synthesis of AuNPs plays an important role in preventing particle aggregation, this phenomenon can be observed by the formation of granules or precipitates in the solution which can be observed visually (Kim et al., 2016). This function certainly has implications for the stability of the AuNPs produced and impacts on the performance of AuNPs as a catalyst later. **Figure 1b** shows the UV-visible spectra of AuNPs after 4 weeks of synthesis at room temperature, in the first week of synthesis, there was a slight decrease in absorbance at 532 nm, and after one month of synthesis, there was a significant decrease in absorbance. This phenomenon indicates a reduction in the ability of *P. canescens* leaves extract as a capping agent in maintaining the stability of AuNPs (Krishnaswamy et al., 2014), which slowly cause agglomeration in AuNPs solution (Abbasi et al., 2015).

Particle size analysis was used to determine the particle size and size distribution in colloidal AuNPs. Based on a histogram of particle size analysis clearly showed the average diameter of AuNPs synthesized by *P. canescens* leaves extract was 14.9 nm (**Figure 2a**), with a polydispersity index (PDI) is 0.570, this value indicates that the colloidal AuNPs formed to have an even distribution in the sample (Bailly et al., 2019). Transmission electron microscopy analysis was used to see the morphology of the formed AuNPs. **Figure 2b** shows the results of TEM analysis of AuNPs with various magnifications from 200 to 20 nm, AuNPs have a spherical shape without the formation of agglomeration between particles. The absence of aggregation formed indicates that the presence of secondary metabolites in *P. canescens* leaves extract other than as a reducing agent, also plays a role as a capping agent to prevent aggregation between AuNPs (Xing, 2021). In line with PSA results, the size of the AuNPs seen in the TEM analysis shows a value that is

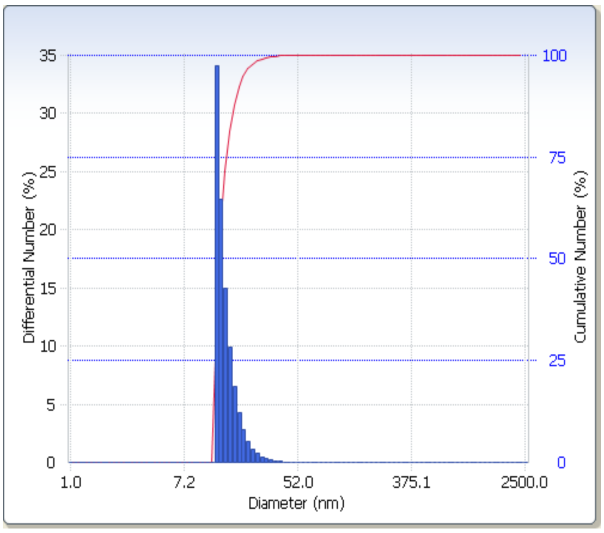


(a)

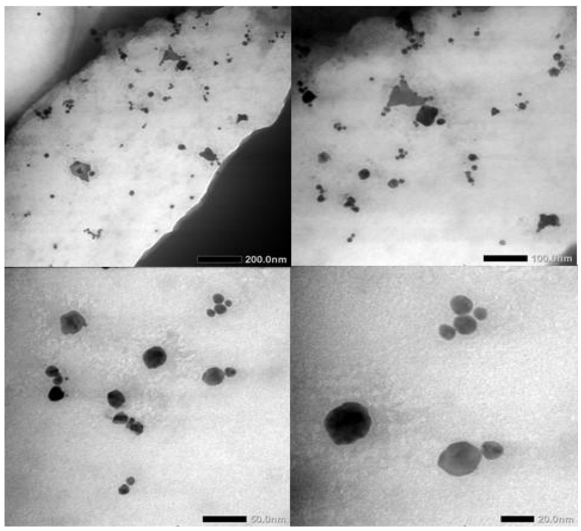


(b)

Figure 1. UV-visible spectroscopy AuNPs stability after (**a**) 1 hour and (**b**) 4 weeks synthesis



(a)



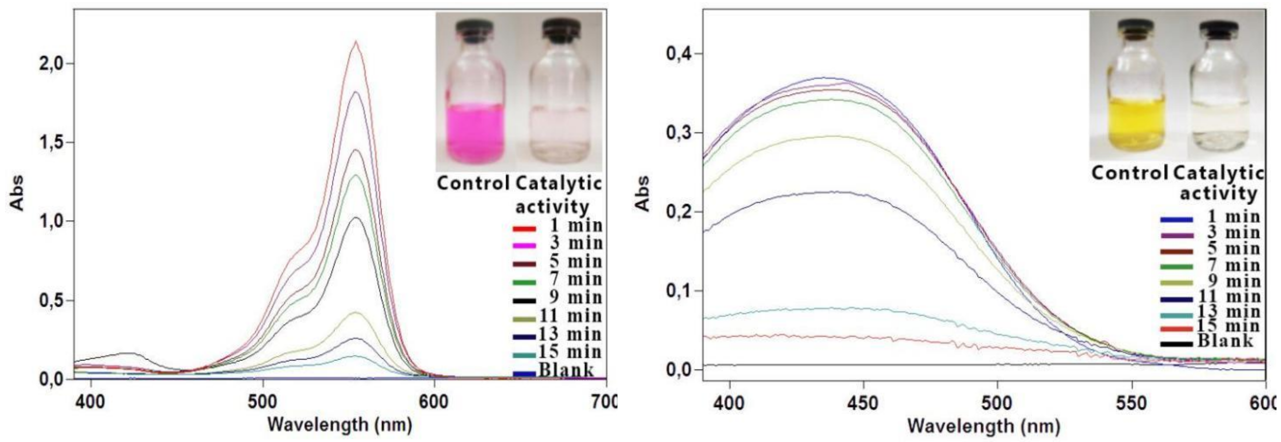
(b)

Figure 2. Particles size analysis and transmission electron microscopy of AuNPs synthesized by *P. canescens* leaves extract

little bit different. Differences in measurement methods on both instruments may be the cause of the size difference in AuNPs measured between PSA and TEM. Analysis using PSA gave the average particle size, while the TEM technique gives the size of each Au nanoparticle (Husen & Iqbal, 2019). In addition, the influence of differences in sample preparation (particle number, mass, optical properties, and volume) and the polydispersity of the solution used also give different nanoparticle measurement results, depending on the instrument response (Sujitha & Kannan, 2013).

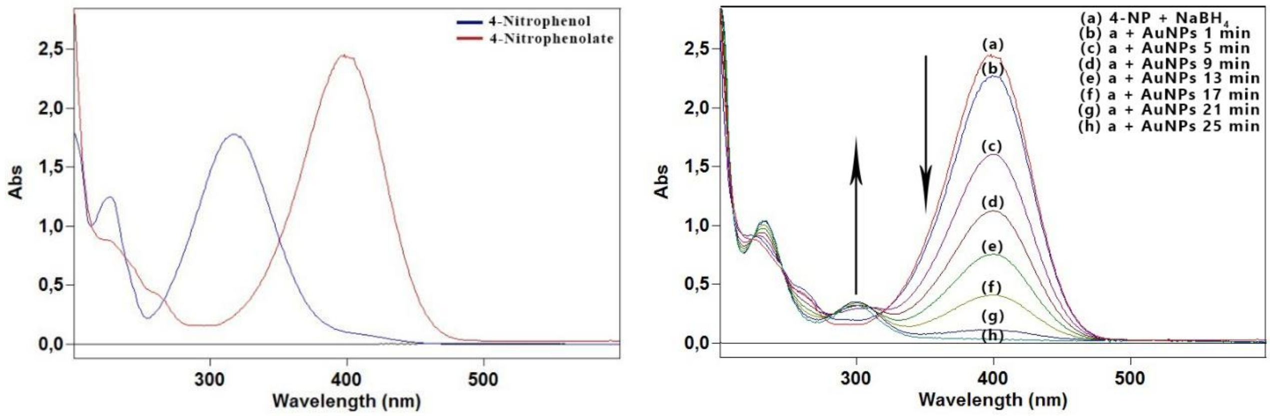
Catalytic activities of AuNPs synthesized by *P. canescens* leaves extract was studied in degrading water pollutants. RhB and MY are used as models of water pollutants for dye degradation, it is due to their massive use in the industrial world. Reduction with strong reagents like $NaBH_4$ in the presence of nano-catalysts is an alternative way to reduce organic dyes that works very well. Catalytic progress of AuNPs in degradation of RhB was monitored by UV-visible spectrophotometer. The presence of AuNPs as a catalyst in the reduction of RhB using $NaBH_4$ (sodium borohydride) solution serves to increase the efficiency of the catalysis reaction. In the absence of catalyst, the reduction process of RhB takes a long time. The redox potential values of RhB (-0.48) and sodium borohydride (-1.33 V) show a significant difference, which causes the reduction process to occur slowly, so that the presence of AuNPs in the reaction functions to facilitate electron relay between $NaBH_4$ (donor) and RhB (acceptor) (Alle et al., 2020). In **Figure 3a**, the analysis of the UV-visible spectrophotometer shows the specific peak of RhB with a maximum wavelength at 554 nm (Vijayan et al., 2019). The RhB degradation process begins with the addition of 150 μL AuNPs solution into a solution containing RhB and $NaBH_4$. The phenomenon of decreasing the absorbance of RhB solution was monitored by spectrophotometer UV-visible, decolorization of RhB process could be observed visually after 15 minutes of reaction time, which was indicated by a change in the color of RhB solution from pink-red to colorless (Nguyen et al., 2018). Additionally, the decrease in absorbance in the RhB solution is an indication of the degradation process going well with leuco-RhB as a reaction product (Paul et al., 2016). Furthermore, MY is used as the second dye as a water pollutant model, because of its carcinogenic properties to living things and the environment. MY degradation process using a reducing agent ($NaBH_4$) at room temperature. **Figure 3b**. showed that the catalytic performance of AuNPs in degrading MY occurred rapidly after 15 minutes of reaction time. This phenomenon was observed with a decrease in absorbance using a UV-visible spectrophotometer at a wavelength at 434 nm and a change in the color of the solution from yellow to colorless.

The reduced 4-nitrophenol (4-NP) compound was evaluated for the catalytic performance of AuNPs in the presence of $NaBH_4$ at room temperature. **Figure 4a** shows the UV-visible spectra of 4-NP with a specific peak of 317 nm, in the presence of $NaBH_4$ in the solution the color of the solution became yellow and the peak of the solution shifted to 400 nm to form 4-nitrophenolate ions which would later form 4-aminophenol (4-AP) (Chairam et al., 2017). The solution's absorption peak moving from 317 nm (4-NP peak) to 400 nm (4-nitrophenolate ions) followed by the increase in absorbance value after the formation of 4-nitrophenolate ions is caused by the use of a relatively high $NaBH_4$ solution, which causes a lot of hydrogen bubbles (H_2) to form during the reaction (**Figure 4b**) (Varshney et al., 2020). Also, the process of reducing 4-NP with $NaBH_4$ solution to form 4-AP is very slow; that it takes more than a day, and the properties of 4-nitrophenolate, which is very stable in solution without a catalyst (Otari et al., 2016). The catalytic progress of AuNPs observed by UV-visible spectrophotometry, the presence of AuNPs synthesized by *P. canescens* leaves extract to the mixture solution, causes a gradual decrease in the



(a) (b)

Figure 3. UV-visible spectra catalytic activity of AuNPs in the presence of $NaBH_4$ (**a**) RhB solution and (**b**) MY solution



(a) (b)

Figure 4. UV-visible spectra (**a**) reaction between 4-NP and $NaBH_4$ to form 4-nitrophenolate ions (**b**) conversion of 4-nitrophenolate ion in the presence AuNPs.

absorbance of 4-nitrophenolate ions at 400 nm in line with the length of time the AuNPs contact in solution for 25 minutes and occurs the color change of the reaction mixture solution from yellow to colorless (Yudha et al., 2021) (**Figure 4b**). The decrease in absorbance that occurred at a wavelength of 400 nm, followed by the formation of a new peak at 300 nm as an indication of the formation of a reaction product 4-AP (Seo et al., 2017). In the early stages of the reaction, the presence of NaBH4 solution in 4-NP forms a stable intermediate compound namely, 4-hydroxylaminophenol (4-Hx). Then, the addition of colloid catalyst AuNPs into the solution causes a reduction process from 4-hydroxylaminophenol to 4-AP, which occurs on the surface of AuNPs (Zhang et al., 2020). In a solution, the change from 4-NP to 4-AP happens quickly because of the 4-nitrophenolate ions. No experiment has tried to figure out how much 4-AP was obtained, but the fact that the absorption peak of 4-nitrophenolate ions decreased suggests that almost all of the 4-NP was converted into 4-AP. In the 4-NP to a 4-AP reduction reaction, AuNPs catalyst contributes to accelerating the hydrogenation process by acting as an electron relay from a donor (BH_4^-) to an acceptor (4-NP) (Khoshnamvand et al., 2020).

CONCLUSIONS

The high stability of AuNPs has been successfully synthesized via a green synthesis approach using *P. canescens* leaves extract as a medium synthesis with a specific peak at 523 nm based on spectrophotometer UV-visible analysis. Analysis of AuNPs showed that the particles have a spherical shape with an average diameter of 14.9 nm based on TEM and PSA analysis, respectively. The performance of AuNPs as a catalyst was tested by degrading compounds rhodamine B (RhB), metanil yellow (MY), and 4-nitrophenol (4-NP) as model pollutants. The results clearly show that the AuNPs synthesized via green synthesis, shows good ability and promise as an alternative solution to replace chemical methods that are not environmentally friendly.

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