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Research Article

Reduction of 4-nitrophenol Mediated by Silver Nanoparticles Synthesized using Aqueous Leaf Extract of *Peronema canescens*

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Abstract

In this study, we developed an alternative of 4-nitrophenol reduction mediated by silver nanoparticles (AgNPs) which was synthesized using aqueous extract of the *Peronema canescens* leaf through an eco-friendly approach. The reducing 4-nitrophenol to 4-aminophenol mediated by AgNPS in the presence of sodium borohydride as a hydrogen source proceeded rapidly at room temperature without any additional treatments. The AgNPS synthesis was simple and was carried out under mild conditions. Ultraviolet–visible spectroscopy was performed to examine the properties of the obtained AgNPs, which displayed an absorption peak at 431 nm. A transmission electron microscopy analysis revealed that the AgNPs were spherical in shape and had an average particle size of 19 nm as determined by particle size analysis.

Keywords: 4-nitrophenol; silver nanoparticles; *Peronema canescens*; nitro-aromatic compounds; AgNPs

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1. Introduction

Metal nanoparticles have demonstrated good catalytic activity in the UV and visible light regions, such as in the treatment of water pollutants [1]. 4-nitrophenol (4-NP) is an example of toxic and hazardous contaminant found in industrial and agricultural raw materials [2] and is a contributor to water pollutants. Previous studies have shown that the biosynthesized AgNPs has good activity against 4-NP compounds [3], and other result shows that the catalytic efficiency of 4-NP to 4-aminophenol reaches 100% [4]. In addition, the proposed method to biosynthesize AgNPs has good catalytic properties compared with methods that use Au [1], Pt [5], and Pd [6], which are much more expensive to produce.

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Silver metal has been used for thousands of years as raw material to manufacture various objects such as jewelry, tools, and coins. With the advance of technology, the particle size of silver has reduced to the nanoscale, which provides it with unique chemical and physical properties, and the applications of silver metal has broadened due to the size reduction of silver nanoparticles (AgNPs) [7]. AgNPs have good antiyeast, α-amylase [8], photo-catalytic [9], anticancer, antibacterial [10–12], and antiviral [13] activities. Moreover, they have applications in drug administration [14] and in sensing heavy metals, such as mercury [15], lead(II), and copper(II) ions [16].

Conventional methods for the production of silver nanoparticles use hazardous chemicals and materials in physical treatment and are considered to have a negative impact not only on the environment but also on all living organisms. Thus, a material synthesis process that has a simple methodology, low cost of production, and emphasis on eco-friendly principles can become an alternate method to synthesize silver nanoparticles. Previous studies have mentioned that the use of fruit [15], leaf, and stem extracts [13,17] of *Melastoma malabathricum*; root extracts of *Cassia toral* L [18]; microalgae [12]; *Cinamon bark* [11]; and biopolymer [19] have proven to be effective as a medium to produce silver nanoparticles. Other result shows that, extract of *Rhus chinensis* was useful in AgNPs synthesis, in the form of triangular, hexagonal, or oval. These results indicate that the bioactive compounds contained in extracts function as reducing and capping agents in the formation of AgNPs [20]. In addition, another advantage of using extracts is that during the synthesis process, the formation of AgNPs can be visually observed from the color change in the reaction solution [15].

On the other hand, Indonesia is rich in herbal plants that grow in the wild area, for example *P. canescens* which popular as *Pohon Sungkai*. The *P. canescens* extract has been used for a long time as an herbal medicine to treat toothache, cold, fever, and ringworm [21]. The methanol extract of *P. canescens* leaves is known to contain secondary metabolites such as alkaloids, flavonoids, terpenoids, steroids, and tannins [22]. In addition, studies have shown that the extract of young leaves of *P. canescens* has good activity as an herbal medicine and a dosage of 0.5625 mg kg w/w can decrease the mice body temperature by up to 29% [23].

According to a literature survey, the 4-nitrophenol (4-NP) reduction mediated by silver nanoparticles (AgNPs) synthesized using the aqueous extract of the *P. canescens* leaf has not been reported to date. The present study aims to synthesize AgNPs from the extract of the *P. canescens* leaf and investigate the performance of the as-prepared AgNPs in the reduction of 4-NP to 4-aminophenol (4-AP). To the best of our knowledge, this is also the first report on the synthesis of AgNPs using the aqueous leaf extracts of *P. canescens*.

2. Materials and Methods

2.1 Materials

P. canescens leaves were collected from the garden of Universitas Bengkulu. Two main precursors were purchased from Merck; silver nitrate (Emsure), 4-nitrophenol. Sodium borohydride was purchased from Sigma-Aldrich (99.99%) and was used as received.

2.2 Instrumentations

A UV–Vis spectrophotometer (Agilent 60) was used to monitor the reaction of the AgNP synthesis at a scanning range of 380–800 nm and the reduction reaction of 4-NP mediated by the as-prepared AgNPs. The size distribution of the AgNPs was determined using a particle size analyzer (PSA) (Delsa™Nano – Beckman Coulter Inc.). A transmission electron microscope (TEM) (JEOL JEM 1400) was utilized to investigate the shape and morphology of the AgNPs in solution.

2.3 Experimental Procedures

2.3.1 Synthesis of silver nanoparticles using aqueous leaves extract of *P. canescens*

The fresh leaves of *P. canescens* were washed thrice with distilled water and dried for seven days at room temperature. The dried leaf (1 g) was mixed with 100 mL distilled water and heated in a hot plate at ±60 °C for 15 min under stirring. The leaf extract was filtered through filter paper (Whatman No. 1) and stored at ±5 °C.

An aqueous solution of silver nitrate (0.01 M) was prepared and used as starting material for the synthesis of AgNPs. In a bottle vial, about 5 mL of the aqueous extract of *P. canescens* was added drop wisely to 1 mL of the 0.01 M silver nitrate solution. The synthesis process was conducted at room temperature under gentle stirring [15]. The solution color, during the synthesis process, immediately changed from yellow to dark chocolate. The formation of AgNPs in the solution was monitored using an ultraviolet–visible (UV–Vis) spectrophotometer, and the shape and size of the AgNPs was confirmed by transmission electron microscopy (TEM) and particle size analysis (PSA).

2.3.2 Catalytic Activity Test

The catalytic activity of the as-prepared AgNPs was evaluated using 4-NP solution (2 mM) as reaction precursor and $NaBH_4$ solution (0.03 M) as hydrogen source. To investigate the intermediate and other possibilities, the following solutions were prepared: Sol-A, made by mixing 1 mL of the 4-NP solution and 10 mL aqua DM; Sol-B, consisting of 1 mL of the 4-NP solution, 10 mL aqua DM, and 5 mL of 0.03 M $NaBH_4$ solution; and Sol-C, prepared by mixing Sol-B with 150 μL of the as-prepared AgNPs. In general, all catalytic experiments were performed at room temperature under stirring [24]. The progress of the catalytic reaction was monitored at different time intervals using a UV–Vis spectrophotometer.

3. Results and Discussion

Figure 1(a) shows the *P. canescens* plant, and the air-dried leaves as the main source of the extract are shown in Figure 1(b). Visual assessment of the formation of silver nanoparticles; the color of the mixture solution during the reaction changed from transparent (Figure 1(c)) to light brown (Figure 1(d)); this is an initial indication of the formation of silver nanoparticles through the reduction of Ag^+ ions to Ag^0 [10]. The change in the color of the aqueous extract used to reduce silver ions has been previously proposed to stem from the oxidation reaction of the active compounds in the extract. This is based on the fact that the silver ion reduction process must be necessarily accompanied by the oxidation of the reducing compound or ions, constituting the so-called redox reaction [25]

The UV–Vis absorption spectrum of the leaf extract depicted in Figure 2(a) shows no peak. In contrast, the reaction mixture of the extract and silver nitrate solution showed a surface plasmon resonance band at 431 nm derived from the AgNPs after the reaction was kept for 48 h at room temperature (Figure 2(b)). This result is in line with a previous report on the use of *Vigna sp.* L seed extract as a reducing agent [26]. The UV–Vis spectroscopic results shown in Figure 2(b) and Figure 2(c) reveal the occurrence of surface plasmon resonance (SPR), a phenomenon specific of AgNPs [27].

The stability of AgNPs is essential for various purposes. Thus, a low stability of AgNPs results in aggregation and agglomeration, which has an impact on their characteristics, particularly their size, affording nano-sized,

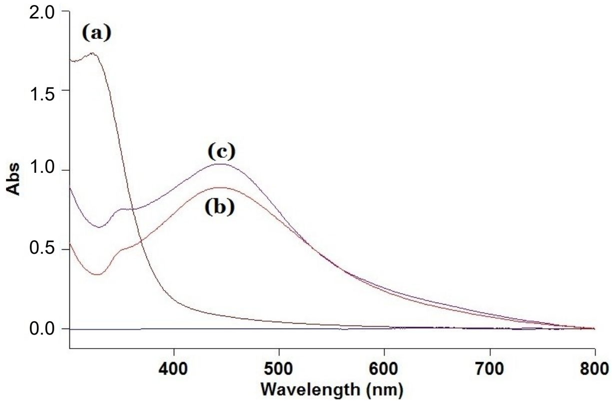

Figure 2. UV-Vis spectra of (a) aqueous extract of *Peronema canescens* (b) reaction mixture after 48 h, and (c) reaction mixture after 22 d.

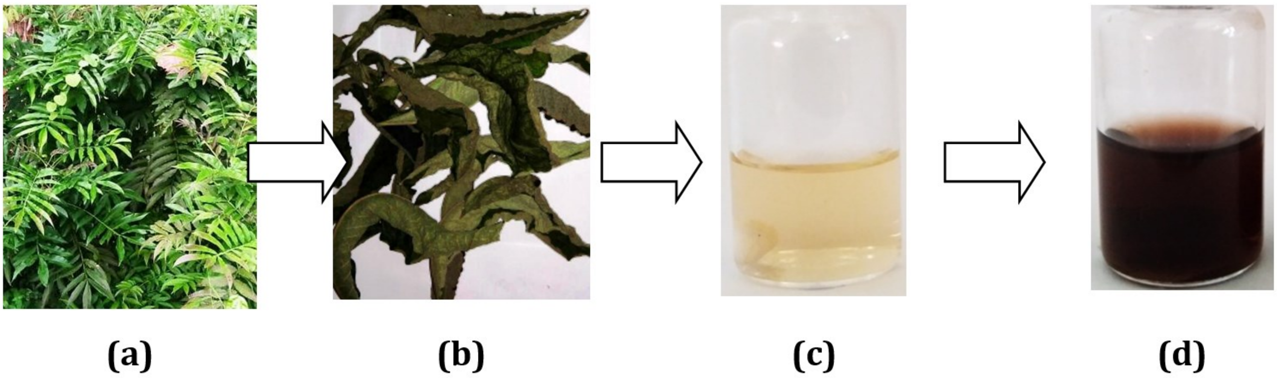

Figure 1. *P. canescens* plants: (a), *P. canescens* air-dried leaves, (b) aqueous extract of *P. canescens* air-dried leaves, and (d) reaction mixture of the extract and $AgNO_3$.

micro-sized, or bulk particles with different properties. Therefore, the investigation of the stability of AgNPs in the reaction medium is very important to ensure the practical application and cost-effectiveness of the resulting material. Interestingly, the as-prepared AgNPs solution was stable even after standing at room temperature for 22 days, which was supported by the stable UV–Vis spectrum at 431 nm (Figure 2c) and lack of aggregation in the bottle's bottom. The bioactive components of the *P. canescens* leaf extract are alkaloids, flavonoids, terpenoids, steroids, and tannin, which can be predicted to play important roles in the formation and stabilization of the AgNPs [14]. In addition, the stability may stem from some organic compounds present in *P. canescens* leaves, such as peronemins [28].

TEM analysis confirmed that the AgNPs mainly consisted of spheres of various sizes (Figure 3). The particle size of the as-prepared Ag nanoparticles was analyzed by PSA and the results are presented in Figure 4. The Figure 4 shows a particle size distribution graph from which it can be inferred that the average size of the AgNPs was 19 nm, which is similar to that obtained by TEM. Taken together, these results demonstrate the potential of *P. canescens* leaf extract as a medium for the synthesis of AgNPs and also act as a stabilizer of the silver nanoparticles.

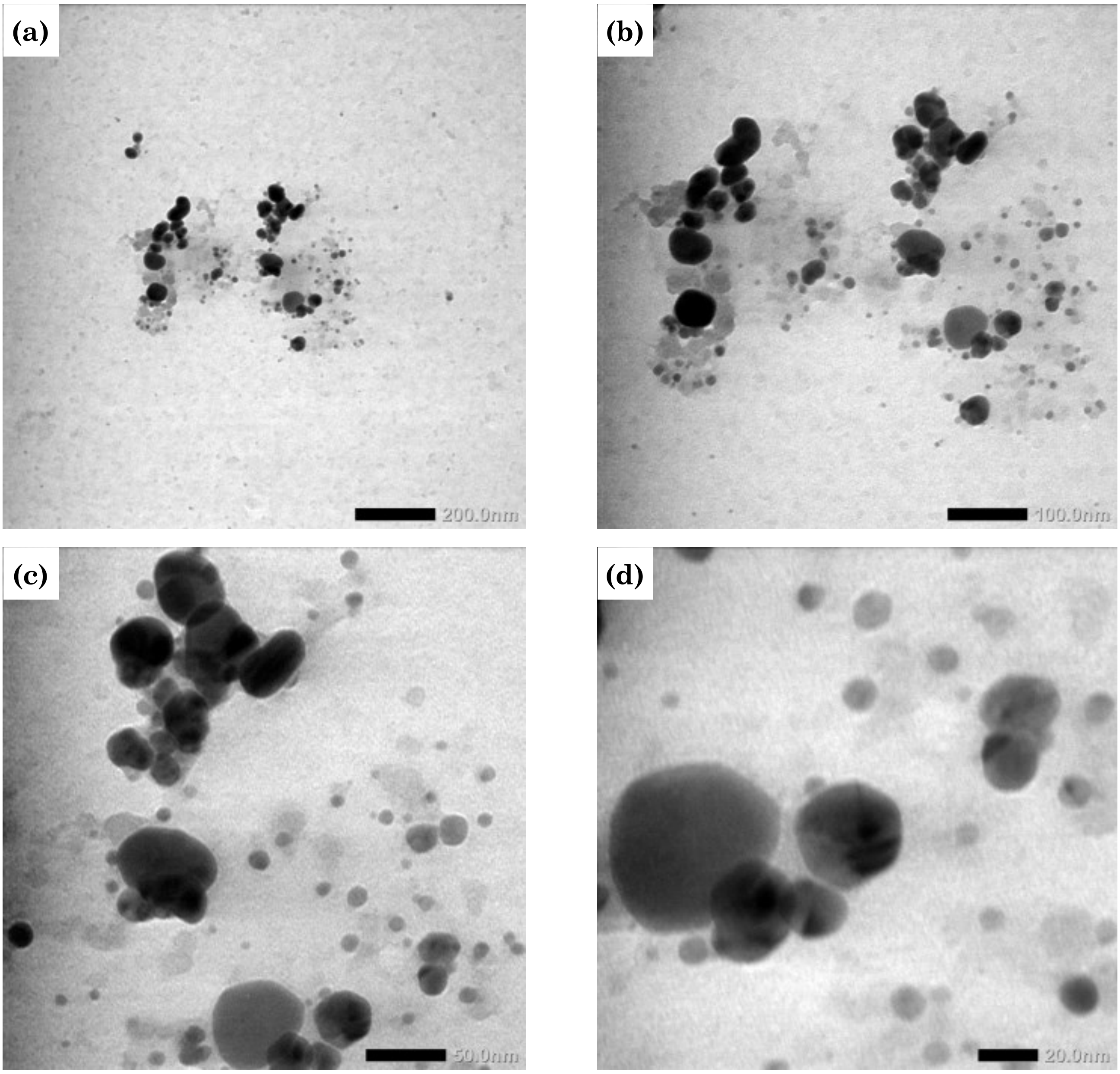

Figure 3. TEM image of as-prepared silver nanoparticles: (a) scale-bar 500 nm, (b) scale-bar 200 nm, (c) scale-bar 50 nm, (d) scale-bar 20 nm.

In order to explore the further application of the as-prepared AgNPs, the catalytic activity of the AgNPs in the reduction of 4-NP to 4-AP was investigated in the presence of $NaBH_4$ as hydrogen source. Figure 5 shows a shift in the absorption peak of the solution from 317 nm (Sol-A) (Figure 5(a)) to 400 nm (Sol-B), which is indicative of the formation of the 4-nitrophenolate intermediate [24] (Figure 5(b)) after the addition of $NaBH_4$ into the 4-NP solution [29]. The addition of AgNPs to Sol-B causes a gradual decrease in the absorption intensity at a wavelength of 400 nm and an increase in the absorption intensity at 298 nm, which is consistent with the formation of 4-AP in the catalytic process (Figure 5(c)) [30]. These results demonstrate the role of the AgNPs in the catalytic reduction of 4-NP to 4-AP in which the silver nanoparticles surface acts as mediator for the electron transfer from BH_4^- donors to 4-NP acceptors in aqueous solution [31]. The 4-NP reduction process is still an active area of research. For instance, recent research results claim that the hydrogen in the metal-catalyzed 4-NP reduction comes from water rather than from $NaBH_4$. However, $NaBH_4$ is required as a support for water ionization to produce H^+ ions [32].

According to the spectrum depicted in Figure 5, the use of 150 μL of an AgNPs solution (equivalent to 12.5 mol% Ag) as a catalyst is sufficient to reduce 0.002 mmol of 4-NP to 4-AP. This process is characterized by a decrease in the absorbance of the nitrophenolate ions until its complete disappearance after 30 minutes. Although the concentration of the product (4-AP) was not calculated quantitatively, the disappearance of the absorbance peak of the 4-nitrophenolate ions and the appearance of a new peak attributable to 4-AP directly indicates that a high conversion is achieved with the amount of catalyst investigated.

4. Conclusions

In conclusion, the reduction of 4-nitrophenol to 4-aminophenol was successfully carried out in the presence of AgNPs which was synthesized using extract of *P. canescens* leaf. The reduction reaction of 4-nitrophenol was performed without any other treatment such as heating or additional pressure. This result also show that the used silver nanoparticles was synthesized using the extract of *P. canescens* leaf for the first time in a simple and eco-friendly manner.

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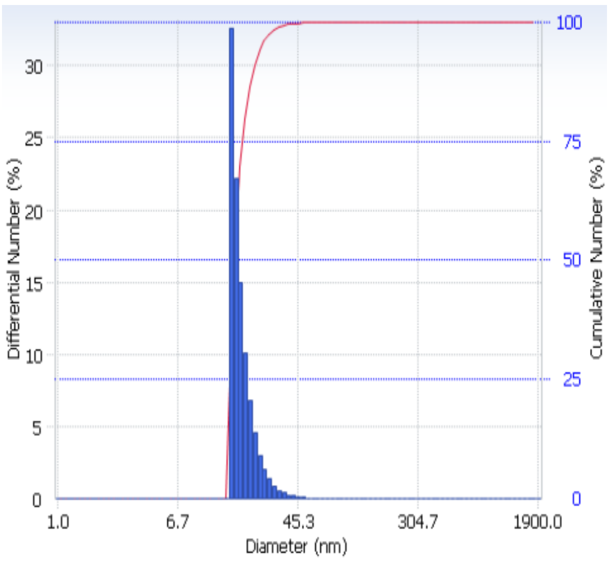

Figure 4. PSA pattern of reaction mixture between silver nitrate and aqueous extract of *P. canescens* leaf.

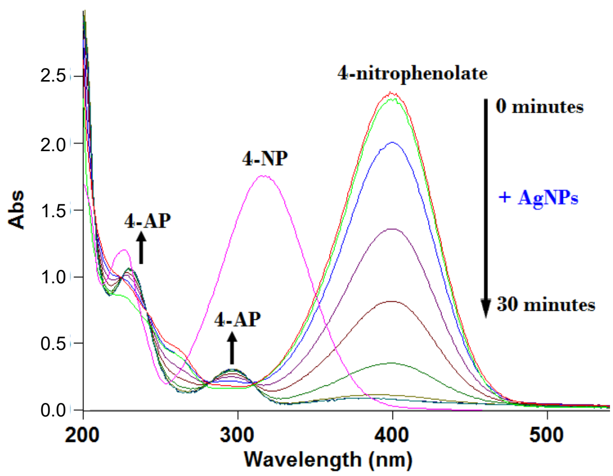

Figure 5. UV–visible spectrum of 4-nitrophenol, 4-nitrophenolate ions (4-nitrophenol in the presence of sodium borohydride), and after addition of AgNPs (0-30 min) catalyst in the reaction mixture (overlay of UV–visible spectra showing the progress of the reduction reaction at 5, 10, 15, 20, 25, 30 min.

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