



Synthesis of Silver Nanoparticles in an Eco-friendly Way using *Lannea coromandelica* Aqueous Bark Extract

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Abstract

In this present study, silver nanoparticles (AgNPs) were synthesized through an easy, rapid, and eco-friendly pathway using *Lannea coromandelica* aqueous bark extract. The obtained AgNPs were characterized using Ultraviolet-Visible (UV-Vis) spectrophotometer, Fourier Transform Infrared (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscope (SEM). The results indicated that the pH of colloidal AgNPs played a vital role in forming AgNPs. The pH ranges used in this study were 6, 8, 10, and 12. The formation of AgNPs was confirmed by observing the surface plasmon resonance (SPR) band at each pH and obtaining a wavelength of 430.50, 419.50, 418.50, and 410.00 nm. A comparison of the FTIR spectra of *Lannea coromandelica* aqueous bark extract and AgNPs showed the contribution of the O-H group in reducing silver ions. XRD diffractogram showed that AgNPs formed at $2\theta = 37.8056^\circ (1\ 1\ 1)$, $44.0345^\circ (2\ 0\ 0)$, $64.3942^\circ (2\ 2\ 0)$, and $77.5003^\circ (3\ 1\ 1)$ with face-centered cubic (FCC) crystal structure, and the average particle size was 22.5047 nm. SEM results showed that the nanoparticles have a non-uniform and irregular shape.

1. Introduction

Silver nanoparticles (AgNPs) are one of the most studied metal nanoparticles and have experienced a very significant level of progress, not only because of the ease of the synthesis process but also their wide applications in various fields such as sensors, textile, food, electronics, medicine, agricultural, environmental safety and more [1, 2, 3]. In general, AgNPs are synthesized using two approaches, physical and chemical methods, which have several drawbacks, such as the use of large amounts of energy and the use of expensive and dangerous chemicals. This encourages efforts to develop eco-friendly nanoparticle synthesis methods [4, 5].

The approach of eco-friendly methods in the synthesis of nanoparticles is also known as biological methods or green synthesis. Generally, this method is conducted with the help of natural ingredients such as plant extracts, fungi, yeast, bacteria, viruses, and algae, which are known to contain secondary metabolites that reduce metal ions [6, 7]. Plant extract is a better synthesizer than other biological methods because of the

availability of abundant plant resources compared to other forms of natural resources. In addition, plant extracts are easy to obtain, safe to handle, and have wide secondary metabolite variability [8, 9]. Several studies have been performed on the synthesis of AgNPs using plant extracts such as *Melia azedarach* [10], *Eulophia herbacea* [11], *Astragalus tribuloides* [12], *Curcuma longa* [13], and *Cocos nucifera* [14].

Lannea coromandelica is a wild plant that is easily found in Indonesia. This plant comes from the Anacardiaceae family and is widely used by the community as a traditional medicinal plant such as wound medicine, cough medicine, an ulcer medicine, appetite enhancer, heals sprains, bruises, heart disease, dysentery, and thrush [15, 16]. Besides being potential as traditional medicine, it also has potential as a reducing agent because it has several active compounds. Britto and Durairaj [17] reported that the results of phytochemical tests on the *Lannea coromandelica* bark gave positive results for the content of flavonoids, alkaloids, saponins, terpenoids, phenols, and tannins. Then, Yun *et al.* [18]

also reported that the *Lannea coromandelica* bark contains a flavonoid compound in quercetin form, which can act as a reducing agent in the synthesis of AgNPs. Jain and Mehata [19] reported that quercetin contained in the *Ocimum sanctum* is mainly responsible for reducing Ag ions to AgNPs.

Based on the description above, AgNPs were synthesized eco-friendly using *Lannea coromandelica* aqueous bark extract with the pH parameter of colloidal AgNPs. Furthermore, the formed nanoparticles were confirmed using UV-Vis spectrophotometer, FTIR, XRD, and SEM instruments.

2. Methodology

2.1. Materials and Instruments

The materials used in this study were *Lannea coromandelica* bark, distilled water, bi-distilled water, silver nitrate (AgNO_3) crystals (Merck), sodium hydroxide (NaOH) crystals (Merck), and Whatman filter paper No. 1. The instruments used were UV-Vis spectrophotometer (Shimadzu UV-2600), centrifuge (TOMY MX-300), freeze dryer (MN10A), FTIR (Shimadzu IR Prestige21), XRD (Shimadzu XRD-7000), and SEM (JEOL JCM-6000plus).

2.2. Preparation of *Lannea coromandelica* Aqueous Bark Extract

Lannea coromandelica bark was collected from *Lannea coromandelica* trees at the campus of Universitas Hasanuddin, Makassar, Indonesia. The bark was washed several times with distilled water to remove the dust particles, then room-dried to remove the residual moisture, and the dried *Lannea coromandelica* bark was cut into small pieces. One gram of *Lannea coromandelica* bark was added to 100 mL of bi-distilled water in an Erlenmeyer's flask of 250 mL capacity. The mixture was heated carefully for about 15–20 minutes and was filtered through a Whatman filter paper No. 1. The aqueous extract of *Lannea coromandelica* can be used directly for the synthesis of AgNPs.

2.3. Synthesis and Characterization of AgNPs with *Lannea coromandelica* Aqueous Bark Extract

The synthesis of AgNPs on pH variations was performed by reacting 10 mL of AgNO_3 0.5 mM and 1 mL of *Lannea coromandelica* aqueous bark extract 1%. Then, NaOH 0.1 mM solution was added with a predetermined pH variation of pH 6, 8, 10, and 12. The reaction was observed from the color change and measured using UV-Vis spectrophotometer at 1, 3, 6, and 24 hours. Colloidal AgNPs that have been formed was centrifuged at 10,000 rpm for 30 minutes. The precipitate formed was dried using a freeze dryer for \pm 6 hours to obtain the sample in powder form. Furthermore, AgNPs powders were characterized using FTIR, XRD, and SEM instruments.

The characterization of the UV-Vis spectrophotometer was carried out on a solution containing colloidal AgNPs, which was inserted into a

cuvette and then measured at a wavelength of 200–700 nm. FTIR characterization was carried out on two mg of AgNPs powder, mixed with 100 mg of KBr, and made into pellets, and then the FTIR spectrum was analyzed in the wavenumber range of $4000\text{--}400\text{ cm}^{-1}$. XRD characterization was carried out on two mg of AgNPs powder, made into pellets, and XRD was analyzed at 2θ 20–80°. SEM characterization was carried out on two mg of AgNPs powder, made into pellets, and analyzed by SEM at 15 kV reduction voltage acceleration with 5000 \times and 8000 \times magnification.

3. Results and Discussion

One of the parameters that significantly affect the formation of AgNPs is pH. Several studies reported that the formation of AgNPs depends on the pH of the reaction; the absorbance value increases gradually with increasing pH, indicating the level of formation of AgNPs is higher in basic conditions than in acidic conditions. This suggests that the basic condition is more favorable for producing AgNPs [20, 21, 22, 23, 24]. In this study, the effect of pH on the formation of AgNPs was observed at pH 6, 8, 10, and 12. The color changes observed visually at 1, 3, 6, and 24 hours indicated the formation of AgNPs marked by a brownish color change. Increasing pH causes the color of the solution to become more concentrated. According to Kocazorbaz *et al.* [25], the darker the resulting color, the more Ag^+ ions are reduced to Ag^0 . Therefore, the difference in the intensity of the resulting color can indicate the ratio of the number of nanoparticles formed. Furthermore, the nanoparticles formed were confirmed using UV-Vis spectrophotometer, FTIR, XRD, and SEM instruments.

Table 1. Comparison of pH variations used in the synthesis of AgNPs in an eco-friendly way using plant extract

Plant extract	Variation of pH parameter	UV-Vis absorption (nm)	Ref.
<i>Averrhoa carambola</i>	4, 7, 10	430, 415, 410	[20]
<i>Cuminum cyminum</i>	4, 7, 10	430, 426, 422	[21]
<i>Nepeta leucophylla</i>	5–9	415–440	[22]
<i>Kalanchoe pinnata</i>	5, 7–12	420–440	[23]
<i>Scrophularia striata</i>	5, 5, 8, 10, 11, 12	around 400–430	[24]
<i>Azadirachta indica</i>	9–13	383–415	[26]
<i>Ferula persica</i>	6, 8, 10, 12	around 400–440	[27]
<i>Jujube core</i>	10, 12	around 420	[28]
<i>Convolvulus fruticosus</i>	6, 8, 10, 12	around 427–450	[29]
<i>Lannea coromandelica</i>	6, 8, 10, 12	410–430.50	Present study

3.1. UV-Vis Spectrophotometer Analysis

Instrumentation characterization using UV-Vis spectrophotometer aims to confirm the formation and growth of nanoparticles. The formation of AgNPs was characterized by the appearance of SPR bands in the wavelength range of 400–450 nm [30, 31]. The UV-Vis spectra of colloidal AgNPs with pH variations can be seen in Figure 1.

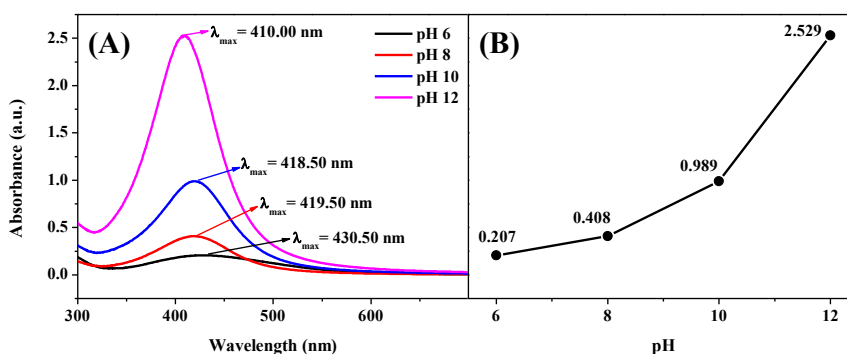


Figure 1. UV-Vis spectra of colloidal AgNPs (A) at pH 6, 9, 10, and 12, (B) absorbance vs. pH

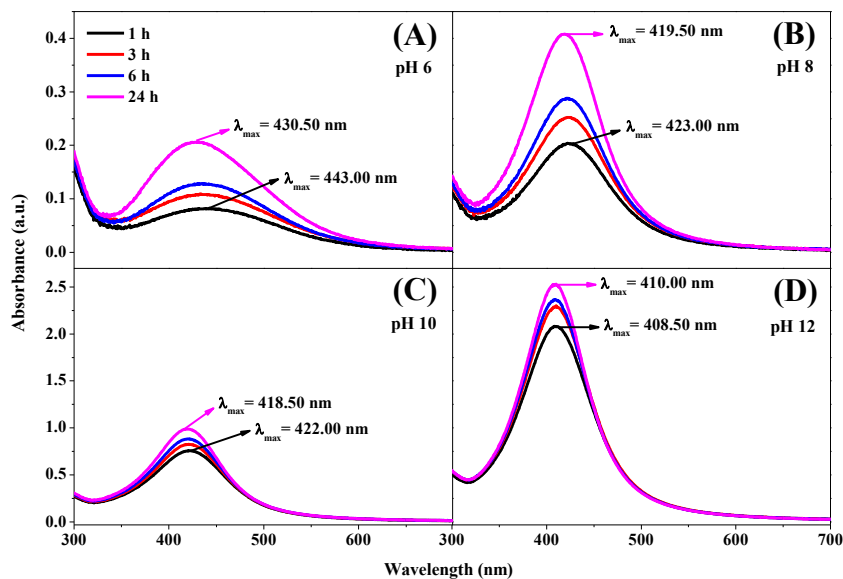


Figure 2. UV-Vis spectra of colloidal AgNPs at pH (A) 6 (B) 8 (C) 10 and (D) 12 at 1, 3, 6, and 24 hours

Figure 1 shows that the absorbance peaks appear in the wavelength range of 410.00–4350.50 nm, indicating that AgNPs have been formed. The lowest absorbance value was indicated by colloidal AgNPs synthesized at pH 6 and continued to increase with the increasing pH of the solution. Hence, it can summarize that the increase in pH of the solution is directly proportional to the absorbance of the resulting AgNPs. According to Marciniak *et al.* [32], this is due to the presence of hydroxide ions which can enhance the reduction capacity of biomolecules acting as reducing agents to carry out metal ion reduction reactions. Therefore, the higher the pH of the solution, the more colloidal AgNPs are formed. Furthermore, the maximum wavelength shift at 1, 3, 6, and 24 hours were observed to estimate the stability of colloidal AgNPs. The maximum wavelength shift of colloidal AgNPs with a function of time can be seen in Figure 2.

Based on Figure 2, colloidal AgNPs at pH 12 shows a maximum wavelength shift that is not excessively large at 1 to 24 hours of observation (408.50 to 410.00 nm). According to Apriandanu *et al.* [33], a slight maximum wavelength shift can be used as a parameter of nanoparticle stability. On this basis, this condition was considered the optimum pH for forming AgNPs with the reducing agent of *Lannea coromandelica* aqueous bark extract. Hashemi *et al.* [27], Naghizadeh *et al.* [28], and Shirzadi-Ahodashti *et al.* [29] also reported that the optimum pH for the formation of AgNPs was 12.

3.2. FTIR Analysis

The FTIR characterization aimed to identify the possible functional groups in the *Lannea coromandelica* aqueous bark extract that have a role in reducing Ag^+ to Ag^0 . A comparison of the FTIR spectra of the *Lannea coromandelica* aqueous bark extract before and after the formation of AgNPs can be seen in Figure 3, and information on the functional groups of the absorption can be seen in Table 2.

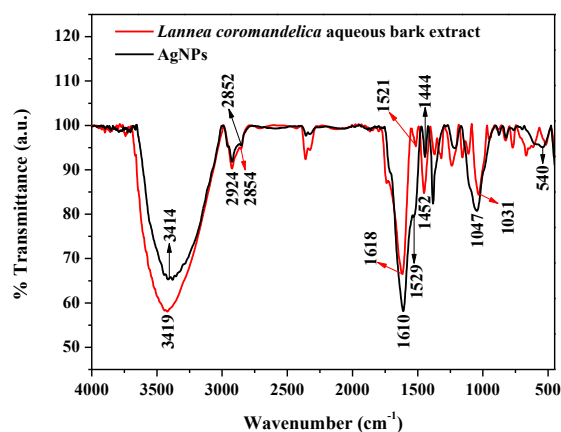


Figure 3. FTIR spectra of *Lannea coromandelica* aqueous bark extract and AgNPs

Table 2. FTIR absorption data of *Lannea coromandelica* aqueous bark extract and AgNPs

Wavenumber (cm ⁻¹)		Functional groups
<i>Lannea coromandelica</i> aqueous bark extract	AgNPs	
3419	3414	O-H stretching
2924 and 2854	2924 and 2852	-Csp ³ -H
1618	1610	C=O
1521	1529	C=C aromatic
1452	1444	C-H bending
1031	1047	C-O phenol
-	540	Ag-O [34, 35]

The FTIR spectra profile of *Lannea coromandelica* aqueous bark extract showed absorption with a strong intensity at 3419 cm⁻¹, which indicated the presence of O-H groups originating from alcohol or phenol compounds. This was reinforced by absorption with strong intensity at 1031 cm⁻¹, which was the presence of vibrations of the C-O bond of phenol. Absorption at 1618 cm⁻¹ and 1521 cm⁻¹ indicated the presence of vibrations of the C=O and C=C aromatics. The bending vibration of the C-H bond of an alkane at 1452 cm⁻¹.

The FTIR spectra profile of AgNPs is not significantly different from the spectra profile of *Lannea coromandelica* aqueous bark extract. The difference can be seen from the decrease in the absorption intensity in the area of the hydroxyl functional group. Changes in the absorption intensity can provide clues to the functional groups that play a role in forming AgNPs. The appearance of absorption at 540 cm⁻¹ in the FTIR spectra of AgNPs is the vibration of the Ag-O bond, strengthening the notion that hydroxyl groups are involved in the synthesis of AgNPs. The same thing was also reported by Wan Mat Khalir *et al.* [34] and Chetia *et al.* [35] that the hydroxyl functional group was responsible for reducing silver ions.

3.3. XRD Analysis

XRD characterization aimed to study the crystalline properties of the synthesized AgNPs. The characteristic peaks observed in the XRD pattern confirmed the presence of the AgNPs shown in Figure 4.

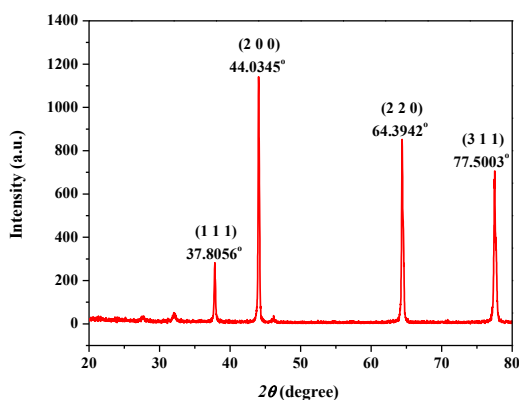


Figure 4. XRD diffractogram of AgNPs

Figure 4 shows that the diffraction peaks at 2θ are 37.8056°, 44.0345°, 64.3942°, and 77.5003°. The four peaks were correlated on the Bragg planes (1 1 1), (2 0 0), (2 2 0), and (3 1 1) according to published data in ICCD No. 04-0783. Table 3 confirmed that the results from

XRD (2θ) and the standard diffraction angle (2θ) of AgNPs are in agreement [36].

Table 3. Experimental and standard diffraction angle of AgNPs

Experimental diffraction angle (2θ in degrees)	Standard diffraction angle (2θ in degrees) ICCD No. 04-0783
37.8056	38.116
44.0345	44.277
64.3942	64.426
77.5003	77.472

The average particle size of AgNPs can be determined from the 2θ angle that is characteristic of AgNPs using the Debye-Scherrer equation as follows:

$$D = \frac{k \lambda}{\beta \cos \theta}$$

where *D* is the average particle size of AgNPs (nm), *k* is the Debye-Scherrer constant (0.9), *λ* is the x-ray wavelength (0.15406 nm), *β* is the FWHM (Full Width at Half Maximum) (rad) of each peak, and *θ* is the angle diffraction. The data from the analysis of the average crystal size of AgNPs can be seen in Table 4.

Table 4. Average particle size and lattice parameter of AgNPs

2θ (deg)	FWHM (deg)	<i>D</i> (nm)	<i>d</i> -spacing (Å)	<i>h</i> ² + <i>k</i> ² + <i>l</i> ² [37]	<i>hkl</i>	<i>a</i> (Å)
37.8056	0.1949	32.2049	2.3777	3	1 1 1	4.1183
44.0345	0.1761	32.4323	2.0548	4	2 0 0	4.1096
64.3942	0.1938	17.7159	1.4457	8	2 2 0	4.0891
77.5003	0.2243	7.6657	1.2307	11	3 1 1	4.0818
Average particle size		22.5047	Average lattice parameter		4.0997	

Based on Table 4, the particle size calculated using the Debye-Scherrer equation ranged from 7.6657 to 32.4323 nm, with an average particle size of 22.5047 nm. These results indicated that the reduction process could produce nano-sized particles (<100 nm) [23, 24]. The size of the AgNPs was smaller than the reducing agent used for the plant extract *Convolvulus fruticosus* [29]. Based on Table 3, the peak at the 2θ angle obtained has a face-centered cubic (FCC) crystal orientation. The FCC structure means that silver atoms will be located at each corner, and atoms will also occupy positions on all cube surfaces. The lattice parameter '*a*' for the FCC structure can be calculated by the following equation:

$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2}$$

where *a* is the lattice parameter, *d_{hkl}* is *d*-spacing (nm), and *h*, *k*, and *l* are the Miller indices of the Bragg plane. The average lattice parameter is 4.0997 Å (Table 4), which was in agreement with the standard lattice parameter value (*a* = 4.086 Å) from standard ICCD No. 04-0783 for AgNPs [22].

3.4. SEM Analysis

SEM characterization aimed to show the morphology of the formed AgNPs. The SEM test results were tested using SEM 5000× and 8000× magnification and reduced voltage acceleration of 15 kV, the AgNPs produced in this study are shown in Figure 5.

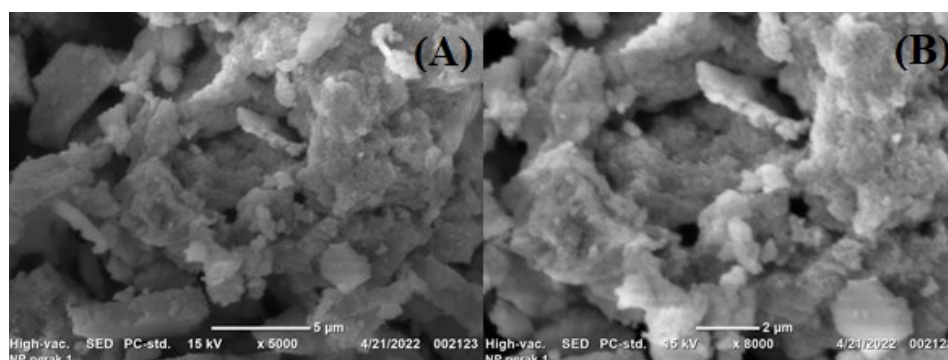


Figure 5. Image of SEM AgNPs with the magnification of (A) 5000× (B) 8000×

Figure 5 shows that the morphology of AgNPs has a surface structure with non-uniform and irregular particle shapes. Observations also showed that the resulting nanoparticles tend to agglomerate. The same thing was also reported by Masakke *et al.* [38] that the AgNPs that had been synthesized were random in shape, with sizes that tended to vary due to the aggregation of nanoparticles.

4. Conclusion

In summary, AgNPs have been synthesized without toxic materials in a simple eco-friendly way. *Lannea coromandelica* aqueous bark extract was determined to act as a reducing agent in this investigation. The formation rate of AgNPs depends on the pH, with pH 12 as the optimum condition. The FTIR spectra revealed that the O-H groups of the extract interacted with the nanoparticles and formed Ag-O vibrational bonds at 540 cm^{-1} . The AgNPs produced have an FCC crystal structure, and the average particle size was 22.5047 nm. The SEM showed that the resulting nanoparticles were of non-uniform and irregular shape.

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