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# Synthesis and Characterization of Silver Nanoparticles Using Bioreductant Andong Leaf Extract (*Cordyline fruticosa* (L) A. Chev.)

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Article Info	Abstract
Article Info Article history: Received: 26 <sup>th</sup> September 2023 Revised: 03 <sup>rd</sup> April 2024 Accepted: 26 <sup>th</sup> April 2024 Online: 31 <sup>st</sup> May 2024 Keywords: Silver Nanoparticles; Bioreductant; <i>Cordyline fruticosa</i> (L) A. Chev.	Silver nanoparticles are known to play a crucial role in various fields, prompting researchers to undertake synthesis and modification to obtain nanoscale particles. Silver nanoparticles were synthesized using a green synthesis-based chemical method with an extract from <i>Cordyline fruticosa</i> (L.) A. Chev. This study aims to determine the optimum conditions, including pH parameters, reaction time, and concentration, and assess the silver nanoparticles' stability and characteristics. The research findings revealed that the optimal conditions for synthesizing silver nanoparticles were an extract pH of 11, a reaction time of 60 minutes, a AgNO <sub>3</sub> concentration of 1.5 x 10 <sup>-4</sup> M, and an extract concentration of 0.008%. Characterization showed that silver nanoparticles were spherical, ranging from 5 to 20 nm and had an average hydrodynamic size distribution of 100.8 nm. In conclusion, leaf extract of <i>Cordyline fruticosa</i> (L.) A. Chev. can be utilized as a bioreductant and stabilizing agent, as indicated by stability test results, which showed considerable stability over a storage period of three
	months.

## 1. Introduction

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Research on nanoparticles is rapidly advancing due to their significant role in various fields such as industry [1], medicine [2], cosmetics [3], biotechnology [4], catalysis [5], and the environment [6, 7]. This has prompted many researchers to synthesize and modify nanoparticles to obtain nano-sized particles, particularly in synthesizing silver nanoparticles that are less than 100 nm. Silver nanoparticle synthesis can be performed using the chemical method (bottom-up) and the physical method (top-down). Using bioreductant in chemical synthesis often involves toxic and hazardous reducing agents like NaBH<sub>4</sub> [8], while physical synthesis requires consumption. With high energy technological advancements, research has been conducted using chemical methods with more environmentally friendly bioreductant, known as green synthesis, which involves various biological organisms such as bacteria, algae, fungi, and various plant extracts [9]. Compounds found in plant extracts, such as flavonoids, alkaloids, terpenoids, triterpenoids, phenols, tannins, saponins, steroids,

ascorbic acid, carbohydrates, proteins, and vitamins, can function as bioreductants and stabilizing agents [10].

The Cordyline fruticosa (L.) A. Chev. plant has been reported to contain various secondary metabolites in its leaves, such as saponins, polyphenols, steroids, triterpenoids, amino acids, and alkaloids, with tannins being the most dominant compounds [11]. Khandel et al. [12] reported that secondary metabolites like flavonoids and phenolics, which contain hydroxyl groups in their contribute antioxidant structures, to activity. Furthermore, phenolic compounds are considered effective hydrogen donors, making them potential reducing agents in the synthesis of silver nanoparticles. Rusnaenah et al. [13] successfully synthesized silver nanoparticles using extracts from Terminalia catappa leaves, which contain several secondary metabolites, including phenolics (tannins) and explained that the -OH functional groups are responsible for reducing Ag<sup>+</sup> to Ag<sup>0</sup>.

Previous studies have not reported on the synthesis of silver nanoparticles using *Cordyline fruticosa* (L.) A. Chev. leaves. Therefore, this research aims to synthesize



silver nanoparticles utilizing the bioreductant extract derived from *Cordyline fruticosa* (L.) A. Chev. leaves. The objectives include determining the optimal conditions for pH variations, reaction time, silver concentration, and the concentration of *Cordyline fruticosa* (L.) A. Chev. leaves extract, as well as assessing the stability and characteristics of the resulting silver nanoparticles.

#### 2. Experimental

#### 2.1. Tools and Materials

The materials used in this research were *Cordyline fruticosa* (L) A. Chev. leaves, methanol (CH<sub>3</sub>OH), sodium hydroxide (NaOH), and silver nitrate (AgNO<sub>3</sub>). The tools used were pH meter (Hanna microprocessor-211), rotary evaporator (RE100-PRO), UV-Vis spectrophotometer (UV-1280 Shimadzu), FTIR (IR Prestige-21 Shimadzu), PSA (Microtrac Nanotrac Wave II), TEM (1400-Jeol Jem), and centrifuge (TOMY MX307).

#### 2.2. Cordyline fruticosa (L) A. Chev. Leaf Extraction

Fresh leaves were initially cleaned and subsequently dried by exposure to air without direct sunlight. Once dried, the leaves were finely cut into small pieces and then ground using a blender before being sieved through a 40-mesh sieve. The resulting leaf powder was carefully stored in a clean container, shielded from light exposure. Subsequently, 350 grams of the powdered material underwent maceration in CH<sub>3</sub>OH solvent for 3×24 hours. Following maceration, the resultant extract was concentrated using a rotary evaporator until a thick consistency was achieved.

#### 2.3. Synthesis of Silver Nanoparticles

The synthesis of silver nanoparticles was conducted by varying several parameters, including pH, reaction time, silver concentration, and extract concentration.

### 2.3.1. Determination of Optimum pH of Cordyline fruticosa (L) A. Chev. Leaf Extract

In each test tube, 5 mL of leaf extract solution with a concentration of 0.01% was combined with NaOH solution until the pH reached variations of 10, 11, and 12, respectively. Subsequently, 5 mL of AgNO3 solution with a concentration of 1×10<sup>-4</sup> M was added to each test tube. The mixture was then heated at boiling water temperature for 60 minutes. After heating, the solution was cooled using and measured cold water using UV-Vis spectrophotometer at a wavelength ranging from 300 to 700 nm. The optimal pH obtained from these measurements was utilized for the subsequent parameters.

#### 2.3.2. Determination of Optimum Reaction Time

In each test tube, 5 mL of leaf extract solution with a concentration of 0.01% was combined with NaOH solution until reaching a pH of 11. Subsequently, 5 mL of AgNO<sub>3</sub> solution with a concentration of  $1 \times 10^{-4}$  M was added to each test tube. The mixtures were then heated to the boiling water temperature for 15, 30, 45, 60, 90, and 120 minutes. After that, the solutions were cooled using cold water and measured using UV-Vis spectrophotometer at a wavelength ranging from 300 to

700 nm. The optimal reaction time, as determined from these measurements, was utilized for the subsequent parameters.

# 2.3.3. Determination of Optimum Concentration of AgNO<sub>3</sub>

A leaf extract solution with a concentration of 0.01% was added to 5 mL of NaOH solution in each test tube until the pH reached 11. Then, 5 mL of AgNO<sub>3</sub> solution was added at varying concentrations of 0.5×10<sup>-4</sup> M, 1×10<sup>-4</sup> M, 1.5×10<sup>-4</sup> M, 2×10<sup>-4</sup> M, and 3×10<sup>-4</sup> M. The mixtures were then heated to the boiling water temperature for 60 minutes. After heating, the solutions were cooled using and measured UV-Vis cold water using spectrophotometer at wavelengths ranging from 300 to 700 nm. The optimal concentration of AgNO3 determined from these measurements was used for the subsequent parameters.

#### 2.3.4. Determination of Optimum Concentration of Leaf Extract

Leaf extract solutions with varying concentrations of 0.002, 0.004, 0.006, 0.008, and 0.01% were prepared, with 5 mL of each solution added to separate test tubes. NaOH solution was then added to each test tube until the pH reached 11. Subsequently, 5 mL of AgNO<sub>3</sub> solution with a concentration of  $1.5 \times 10^{-4}$  M was added to each test tube. The mixtures were then heated to boiling water temperature for 60 minutes. After heating, the solutions were cooled using cold water and measured using UV-Vis spectrophotometer at wavelengths ranging from 300 to 700 nm. The optimal leaf extract concentration determined from these measurements was used for further research.

# 2.4. Stability Test

Silver nanoparticles were re-synthesized using the optimal conditions determined previously: a 0.008% leaf extract solution was adjusted to pH 11 with NaOH, followed by the addition of a  $1.5 \times 10^{-4}$  M AgNO<sub>3</sub> solution. The mixture was then heated to boiling water temperature for 60 minutes. After heating, the solution was cooled and stored in a closed bottle. Absorptions were measured using a UV-Vis spectrophotometer at wavelengths ranging from 300 to 700 nm at various intervals: every 1 day, 2 days, 3 days, 1 week, 2 weeks, 3 weeks, 1 month, 2 months, and 3 months of storage.

#### 2.5. Characterization of Silver Nanoparticles

The solution for characterization was synthesized using the optimal conditions determined in the previous tests, specifically: an extract pH of 11, a reaction time of 60 minutes, an AgNO<sub>3</sub> solution concentration of  $1.5 \times 10^{-4}$  M, and an extract solution concentration of 0.008%, with a 1:1 ratio of extract solution to AgNO<sub>3</sub> solution. The resulting colloidal silver nanoparticle solution was then characterized using FTIR, PSA, and TEM.



Figure 1. Ag<sup>+</sup> reduction reaction

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Storage time	$\lambda_{max}$ (nm)	Peak intensity (Absorbance)	FWHM (nm)
After synthesis	414	0.971	98
1 day	414	0.966	98
2 days	414	0.953	99
3 days	414	0.992	105
1 week	414	0.905	97
2 weeks	415	0.876	98
3 weeks	413	0.816	98
1 month	414	0.907	100
2 months	414	0.828	102
3 months	414	0.827	104

Table 1. Profile of surface plasmon resonance of silver nanoparticles synthesis during storage



Figure 2. UV-Vis spectra of (a) extract, (b) AgNO<sub>3</sub>, and (c) colloidal silver nanoparticle

FTIR was performed to analyze the functional groups in synthesized silver nanoparticles. Solid samples for FTIR analysis were prepared by centrifuging the colloidal silver nanoparticles at 15,000 rpm for 20 minutes and then drying the resulting solid at 40°C in an oven.

PSA was used to determine the size distribution of the silver nanoparticle colloids. Samples for PSA analysis were prepared in 10 mL volumes and sent to the characterization instrument operator. The results included particle size distribution data and polydispersity index values.

TEM was employed to determine the morphology and size of the silver nanoparticles. Samples for TEM analysis were also prepared in 10 mL and sent to the characterization instrument operator. The results were provided as TEM images, which were further analyzed using ImageJ software to measure the nanoparticle sizes.

# 3. Results and Discussion

# 3.1. Synthesis of Silver Nanoparticles

Silver nanoparticles are synthesized by reducing  $Ag^+$ from  $AgNO_3$  to  $Ag^0$  using leaf extract as a bioreductant. The secondary metabolites contained in the leaves have antioxidant properties due to the presence of -OH functional groups, which are easily oxidized. Typically, - OH are acidic; when NaOH is added as a base, proton release occurs due to deprotonation, forming phenoxide anions that have three pairs of free electrons. These anions are used to reduce metal ions, thus forming  $Ag^0$ [14].



Figure 3. Surface plasmon resonance spectra of silver nanoparticles with pH variations

Figure 2 displays the spectra results, indicating that the AgNO<sub>3</sub> solution shows an absorption peak at a wavelength of 305 nm. In contrast, the synthesized silver nanoparticles exhibit a peak at a wavelength of 414 nm. The surface plasmon resonance spectrum within the wavelength range of 400–450 nm confirms that all Ag<sup>+</sup> ions have been reduced to Ag<sup>0</sup>. Similar research by Bagur *et al.* [15] demonstrated that the AgNO<sub>3</sub> solution produces a peak spectrum with a  $\lambda_{max}$  below 350 nm, whereas the synthesized silver nanoparticles show a peak at a wavelength of 425 nm. This surface plasmon resonance spectrum in the 400–450 nm range corroborates the complete reduction of Ag<sup>+</sup> to Ag<sup>0</sup>.

#### 3.1.1. pH Optimization

This study compared the utilization of pH without adding a base (pH 5) to basic pH conditions (pH 10, 11, and 12). Analysis revealed that the solution at pH 5 exhibited a wavelength of 385 nm, suggesting the absence of silver nanoparticle formation (Figure 3). Conversely, solutions at pH 10, 11, and 12 displayed a wavelength of 410 nm, indicating the presence of silver nanoparticles. Notably, silver nanoparticle formation occurred exclusively under basic pH conditions, as the protons from hydroxyl groups underwent deprotonation, forming a negative charge that facilitated the binding of these groups to metal cations [16].

These findings align with prior research conducted by Moldovan *et al.* [17], which concluded that basic pH supports silver nanoparticle formation, while acidic conditions lead to aggregation and generating larger particles. The absorbance of the three pH variations was examined to ascertain the optimum pH. Results demonstrated that at pH 11, the absorbance was notably higher, indicating a greater production of nanoparticles compared to pH 10 and 12. Consequently, subsequent synthesis endeavors were conducted at pH 11, which was identified as the optimal pH condition.

#### 3.1.2. Reaction Time Optimization

The experiment aimed to ascertain the ideal duration necessary for producing silver nanoparticles in optimal quantities. Various heating durations, ranging from 15 to 120 minutes, were tested. Analysis depicted in Figure 4 revealed a direct relationship between the heating duration and the absorbance intensity, signifying the progressive reduction of silver ions leading to the formation of silver nanoparticles. The synthesis of silver nanoparticles exhibited an ascending trend, with absorbance values increasing from 0.481 at 15 minutes to 0.659 at 60 minutes. Subsequently, absorbance declined to 0.636 at 90 minutes and further decreased to 0.606 at 120 minutes.

Notably, heating for 60 minutes resulted in the highest absorbance intensity, indicating the optimal formation of silver nanoparticles. This observation aligns with prior research conducted by Dada *et al.* [18], which demonstrated that all Ag<sup>+</sup> ions had been successfully reduced to Ag<sup>0</sup>, completing the silver colloid nucleation process and yielding small, stable particle sizes under optimal conditions. Hence, the optimal duration for synthesizing silver nanoparticles is determined to be 60 minutes.

#### 3.1.3. Silver Concentration Optimization

Variations in silver concentration were conducted to confirm the ability of the extract used to reduce silver ions in line with increasing silver concentration. As the AgNO<sub>3</sub> concentration increased, the yellow color in the silver nanoparticle colloid intensified, indicating a proportional increase in nanoparticle formation. This validates that the extract can effectively reduce silver ions in tandem with increasing silver concentration up to a concentration of  $3 \times 10^{-4}$  M.



Figure 4. Surface plasmon resonance spectra of silver nanoparticles with varying reaction times

Figure 5 shows the spectrum of silver nanoparticles with wavelengths ranging from 405 to 415 nm. The concentration of  $1.5 \times 10^{-4}$  M is considered the optimal silver concentration. At a concentration of  $0.5 \times 10^{-4}$  M, the measured wavelength is 403 nm with low absorbance intensity, attributable to the insufficient presence of Ag<sup>+</sup> ions and, thus, a limited number of nanoparticles formed. At concentrations of  $1 \times 10^{-4}$  M and  $1.5 \times 10^{-4}$  M, the measured wavelength is 410 nm, with a higher absorbance intensity observed at  $1.5 \times 10^{-4}$  M due to the increased nanoparticle formation compared to the  $1 \times 10^{-4}$  M concentration.

At silver concentrations of  $2 \times 10^{-4}$  M and  $3 \times 10^{-4}$  M, two measured wavelengths, namely 410 and 415 nm, were observed, accompanied by a broadening of the peak towards the redshift, indicative of larger nanoparticle sizes. This finding aligns with previous research by Gusrizal *et al.* [19], which demonstrated an increase in absorbance intensity with rising silver concentration and a consequent increase in silver nanoparticle size. Therefore, a silver concentration of  $1.5 \times 10^{-4}$  M was chosen for further synthesis.

### 3.1.4. Optimization of Extract Concentration

From the spectrum analysis, it is evident that there is a correlation between absorbance and extract concentration, reaching a peak at 0.008%. This signifies an optimal reduction of available  $Ag^+$  ions and the subsequent formation of numerous silver nanoparticles. However, there is a decline in absorbance beyond this concentration, likely due to the depletion of silver ions available for reduction.

Octavianus *et al.* [20] conducted similar research, exploring variations in wild almond leaf extract concentrations, with the optimal concentration determined to be 0.008%. Consistent with these findings, the surface plasmon resonance spectrum data in Figure 6 reveal the highest absorbance at an extract concentration of 0.008%, indicating optimal nanoparticle formation compared to other concentrations. Therefore, it is decided to employ an extract concentration of 0.008% for further synthesis.



Figure 5. Surface plasmon resonance spectra of silver nanoparticles with varying concentrations of AgNO<sub>3</sub>



**Figure 6**. Surface plasmon resonance spectra of silver nanoparticles with varying concentrations of leaf extract

#### 3.2. Stability Test

The stability test data depict the ability of silver nanoparticles to maintain their size against aggregation. The surface plasmon resonance spectrum generated is observed to determine changes in the spectrum's wavelength, absorbance intensity, and peak width [21].

Table 1 presents the surface plasmon resonance spectra over four months. After three months of storage, the silver nanoparticle colloids exhibited absorption peaks within the range of 413-415 nm, accompanied by a slight wavelength shift of approximately 1-2 nm towards longer wavelengths, commonly referred to as a bathochromic or red shift [22]. This shift remains within the typical range for nanoparticles, typically between 400-450 nm. Additionally, there was a 15% decrease in absorption intensity, suggesting a reduction in the number of silver nanoparticles present in the colloid due to nanoparticle precipitation. Furthermore, the FWHM value experienced a slight increase from 98 nm to 104 nm, indicating the stability of the silver nanoparticles.

Figure 7 demonstrates the stability of the synthesized silver nanoparticles, indicating their suitability for storage over three months. This suggests that the *Cordyline fruticosa* (L) A. Chev. leaf extract, employed as both a bioreductant and stabilizing agent, obviates the need for additional stabilizing agents [23]. This finding aligns with research by Rahayu *et al.* [24], who found that silver nanoparticles synthesized using Dayak onion bulb extract remained stable throughout 4 months of storage.



Figure 7. Surface plasmon resonance spectra of silver nanoparticles after three months of storage



Figure 8. FTIR spectra of leaf extract and silver nanoparticles

#### 3.3. Characterization of Silver Nanoparticles

FTIR characterization is employed to identify the functional groups present in leaf extracts and silver nanoparticles by analyzing the intensity of infrared light absorbed by the material. The FTIR spectra results of *Cordyline fruticosa* (L) A. Chev. leaf extract reveal several key absorption peaks. At the wavenumber 3388.93 cm<sup>-1</sup>, there is a broad absorption peak indicative of -OH functional groups from alcohols and phenols. The absorption at 1727.86 cm<sup>-1</sup> indicates the presence of the C=O carboxylate functional group.

Absorption peaks at 2926.01 cm<sup>-1</sup> and 2854.65 cm<sup>-1</sup> correspond to the -CH (aliphatic) functional groups. Aliphatic compounds are further supported by the presence of  $CH_2$  and  $CH_3$  groups at wavenumbers 1454.33 cm<sup>-1</sup> and 1377.17 cm<sup>-1</sup>, respectively, which show sharp absorption and weak intensity characteristic of aliphatic CH bending vibrations. Additionally, absorptions at 1649.14 cm<sup>-1</sup> and 1512.19 cm<sup>-1</sup> indicate stretching vibrations from the aromatic C=C functional group. A peak with moderate intensity at 1047.35 cm<sup>-1</sup> suggests stretching vibrations from the CO functional group [25].



Figure 9. (a) particle size distribution of silver nanoparticles and (b) silver nanoparticles shape

The FTIR spectrum of silver nanoparticles, shown in Figure 8, reveals a decrease in the absorption intensity of several functional groups. These groups include the –OH group, with an absorption band at 3442.94 cm<sup>-1</sup>, aliphatic CH groups at 2920.23 and 2852.72 cm<sup>-1</sup>, the C=O group at 1741.42 cm<sup>-1</sup>, aromatic C=C groups at 1645.28 and 1541.12 cm<sup>-1</sup>, and the CO group at 1053.13 cm<sup>-1</sup>. This decrease in absorption intensity suggests that these functional groups are involved in the reduction of Ag<sup>+</sup> ions to Ag<sup>0</sup>. FTIR spectra analysis of *Cordyline fruticosa* (L) A. Chev. leaf extract indicates the presence of flavonoid and phenolic compounds, which function as bioreductants. This finding implies forming a layer of organic compounds on the surface of the silver nanoparticles, acting as stabilizing agents to prevent aggregation [26].

Figure 9a shows the average size distribution of silver nanoparticles at 100.8 nm. The size distribution data indicate that the synthesized silver nanoparticles produce particles within the 1-100 nm nano-size range, with some particles exceeding 100 nm. Similar results were obtained in studies by Daniel *et al.* [27], using Epomea carnea, which produced silver nanoparticles ranging from 30-130 nm, and by Dua *et al.* [28], using Eupatorium adenophorum leaf extract, which produced nanoparticles with an average size of 117.75 nm, distributed from 30-400 nm. The presence of silver nanoparticles above 100 nm can be attributed to the amount of extract functioning as a capping agent on their surfaces [29].

Figure 9b illustrates several dispersed nanoparticles, forming small aggregates coated with a thin organic layer derived from plant extracts, which acts as a capping agent [30]. TEM characterization reveals silver nanoparticles varying in size from 5 nm to 20 nm with a spherical morphology. This finding is consistent with research by Raj *et al.* [31], which produced spherical silver nanoparticles with an average size of 18.12 nm. Therefore, it can be concluded that *Cordyline fruticosa* (L) A. Chev. leaf extract effectively functions as a stabilizing agent in synthesizing silver nanoparticles.

# 4. Conclusion

This research successfully synthesized silver nanoparticles using Cordyline fruticosa (L) A. Chev. leaf extract as a bioreductant. The synthesis was conducted under optimum conditions: a AgNO3 concentration of 1.5×10<sup>-4</sup> M, a leaf extract concentration of 0.008% at pH 11, and a heating time of 60 minutes. FTIR analysis confirmed that flavonoid and phenolic compounds in the leaf extract played a crucial role in the synthesis process. The resulting silver nanoparticles were spherical, ranging from 5 to 20 nm and an average hydrodynamic size of 100.8 nm. The nanoparticles demonstrated good stability, as the colloidal solution remained stable for three months. These findings indicate that Cordyline fruticosa (L) A. Chev. leaf extract is effective both as a reducing agent and a stabilizing agent in synthesizing silver nanoparticles.

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