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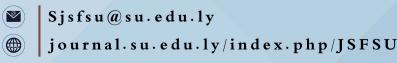
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Aqueous Extract of Winter Jasmine Leaves Mediated Biosynthesis of

Silver Nanoparticles

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ABSTRACT

This study reports a rapid and eco-friendly green method for the synthesis of silver nanoparticles from silver nitrate solution using winter jasmine leaves extract as a reducing agent of Ag⁺ to Ag⁰. A visible Absorption Spectrophotometer was used to monitor the formation of silver nanoparticles (AgNPs). The visible spectrum showed an absorption peak at 420 nm which reflects the surface plasmon resonance (SPR) of AgNPs. In addition to that, the synthesis of AgNPs was confirmed by the color change of the solution. By studying the most important factors affecting the formation of AgNPs, it was noted that the productivity of AgNPs in the solution increased by increasing the pH of the solution, and the basic medium was the appropriate medium for the synthesis process. Also, the amount of produced AgNPs increased with the increase in temperature, volume of extract, reaction time, the extract volume ratio of the winter jasmine leaves, and silver nitrate concentration. In conclusion, the aqueous extract of winter jasmine leaves represents a suitable material for the biosynthesis of AgNPs, and large amounts of AgNPs with appropriate sizes may be obtained by controlling the parameters that affect the synthesis process.

1 Introduction

Silver nanoparticles (AgNPs) have sparked the interest of researchers in recent years due to their wide range of applications, such as antibacterial agents [Le Ouay et al., 2015, Franci *et al*, 2015], antifungal agents [Elgorban et al, 2016, Medda *et al*, 2015], antioxidant agents [Keshari *et al*, 2020, Khorrami *et al*, 2018], antitumor agent [Hashemi et al., 2020, He *et al.*, 2016] and in dye degradation [Albeladi *et al.*, 2020, David *et al.*, 2020]. AgNPs have been synthesized using a number of physical and chemical methods. The physical methods include evaporation-condensation and laser ablation, whereas the

chemical methods include chemical reduction, and electrochemical techniques [Iravani *et al.*, 2014]. However, adopting such approaches to synthesize AgNPs has a number of disadvantages, including high cost, excessive energy consumption, and pollution of the environment [Borah *et al.*, 2020, Omar *et al.*, 2018]. Biosynthesis has lately been proposed as an alternative to those conventional methods since it is simple, costeffective, and an environmentally beneficial approach. Through this route, AgNPs could be produced by bacteria, fungi, and plants, which contain constituents that act as reducing and capping agents for synthesizing

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AgNPs [David et al., 2020, Omar et al., 2021]. These constituents include proteins, polysaccharides, polyphenols, and vitamins [Hashemi et al., 2020, David et al., 2020]. Many researchers have reported biosynthesis of AgNPs using the extract of many plants, such as Ocimum tenuiflorum, Solanum tricobatum, Syzygium cumini, Centella asiatica, and Citrus sinensis [Logeswari et al., 2015], Berberis vulgaris, Brassica nigra, Capsella bursa-pastoris, Lavandula angustifolia, and Origanum vulgare [Salayová et al., 2021], Salvia spinose [Pirtarighat et al., 2019], Moringa oleifera [Moodley et al., 2018], Cleome viscosa [Lakshmanan et al., 2018].

This study aimed to biosynthesize AgNPs using the aqueous extract of winter jasmine leaves, and silver nitrate as a precursor for silver. In addition to that, the effect of pH, reaction time, silver nitrate concentration, temperature, and the volume ratio of winter jasmine leaves was investigated.

2 Materials and Methods

2.1Chemicals

Silver nitrate solution with a concentration of 0.1 M (Winlab, UK), was used to prepare all diluted solutions, which were required to work as a source of silver ions. Sodium hydroxide and hydrochloric acid (BDH) were used to prepare solutions with 0.1 M concentration to adjust the pH of the reaction medium.

2.2 Preparation of the aqueous extract of winter jasmine leaves

In September 2022, a sample of winter jasmine leaves was collected from the Faculty of Science - Gharyan and transferred to the lab in a nylon bag. These leaves were washed thoroughly with deionized water and dried in the open air, away from sunlight, then cut by hand into small pieces, in order to increase the surface area of these leaves. A quantity of winter jasmine leaves was added to a volume of deionized water in a ratio of 1g: 4 mL in a clean, dry beaker. After heating the mixture for 10 minutes, it was cooled by washing the outside of the beaker with tap water. Then the solution was filtered using Whatman No. 1 filter paper, and the filtrate was collected for later use in the biosynthesis of silver nanoparticles.

2.3 Biosynthesis of silver nanoparticles

The aqueous extract of winter jasmine leaves was added to a solution of silver nitrate with a concentration of 1 mM at a v/v ratio of 1:9, then the pH of the mixture was adjusted to 9 using sodium hydroxide solution before being kept at room temperature for 30 min. After that, the color change was observed, and the visible spectrum was recorded using a visible spectrophotometer (Jenway 6300 spectrophotometer, Staffordshire, UK).

2.4 Effect of pH

To 5 beakers, 1.5 mL of winter jasmine leaf extract and 8.5 mL of silver nitrate solution (1 mM) were added, in which the pH of the solution was initially 6. One of these solutions was left untreated and was among the readings that were monitored by the visible spectrophotometer. To the remaining four solutions, drops of sodium hydroxide solution (0.1 M) were added to obtain the following pH of 7, 8, 9, and 10. Hydrochloric acid (0.1 M) was used to reduce the pH when appropriate. All solutions were kept at room temperature for 30 min, then the color change of the previous solutions was observed, and the visible spectrum of each solution was recorded.

2.5 Effect of reaction time

Fifty mL of silver nitrate solution (1 mM) was transferred to a clean beaker, to which 5 mL of winter jasmine leaf extract was added. By adding drops of sodium hydroxide solution (0.1 M), the pH was adjusted to 9. After 5, 60, 120, and 180 min and 24 hours, 2 mL of the solution was withdrawn, and then the visible spectrum was recorded for each solution to monitor the effect of the time.

2.6 Effect of silver nitrate concentration

Two mL of winter jasmine leaf extract were added to 5 sample bottles, each of which contained 8 mL of silver nitrate solution with different concentrations: 0.5, 1, 2.5, 5, and 10 mM. The pH of each solution was adjusted to 9 by adding drops of sodium hydroxide solution, then all solutions were kept at room temperature for 30 min. After that, the visible spectrum for each solution was recorded.

2.7 Effect of extract volume ratio

To 6 sample bottles, different volumes of winter jasmine leaf extract were added to different volumes of silver nitrate solution (1 mM) provided that the total volume was 10 mL and the volume percentage of the extract in these solutions was 5, 10, 15, 20, 25 and 30%. The pH of each solution was adjusted to 9 using sodium hydroxide solution (0.1 M), and all solutions were kept at room

temperature for half an hour, then the visible spectrum of each solution was recorded.

2.8 Effect of temperature

One mL of the extract and 9 mL of silver nitrate solution (1 mM) were added to 5 sample bottles. Each solution's pH was adjusted to 9, and the solutions were heated for 20 min at different temperatures, namely 20, 30, 50, 60, and 70° C. Then the visible spectrum of each solution was recorded.

3 Results and Discussion

3.1 Confirmation of biosynthesized AgNPs

The color of the reaction medium changed rapidly from yellow to brown, as shown in Figure (1), when 1mL of aqueous extract of winter jasmine leaves was added to 9mL of 1mM AgNO₃ solution. The brown color was obtained by the excitation of surface plasmon vibrations, which are characteristic of AgNPs [Omar et al., 2018, Tripathy et al., 2010].



Figure. (1): Color change due to AgNPs formation

The visible spectrum of the reaction medium was used to monitor the bioreduction of pure Ag^+ ions to Ag^0 . The surface plasmon resonance (SPR) of AgNPs produced a peak with a wavelength of around 420 nm, which was not shown in the winter jasmine extract's visible spectrum as depicted in Figure (2). The appearance of this peak confirmed the formation of AgNPs.

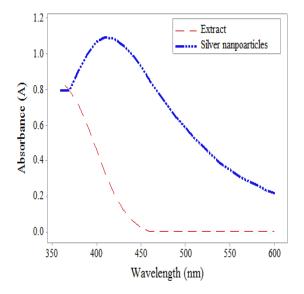


Figure. (2): Visible spectrum of winter jasmine extract and AgNPs solution

Several studies have found that polyphenols and flavonoids (One of the active biochemical components of the winter jasmine plant under investigation) appear to be responsible for the bioreduction of silver ions and the formation of AgNPs. [Iravani et al., 2014, Omar et al., 2018, Awwad et al., 2012, Shahmoradi et al., 2018].

3.2 Effect of pH

The absorbance of (SPR) peak was found to gradually increase with increasing pH as shown in Figure (3), and the color of solutions became darker (Figure (4)), implying that the rate of formation of AgNPs is more rapid in the basic pH than in acidic pH. Several previous studies have reported that the formation of AgNPs occurs rapidly in neutral and basic pH [Omar et al., 2018, Vivek et al., 2012], and this may be due to the ionization of the phenolic group present in the extract of winter jasmine leaves. The slow rate of formation and aggregation of AgNPs at acidic pH could be related to the electrostatic repulsion of anions present in the reaction solution [Omar et al., 2018, Vivek et al., 2012]. It should also be mentioned that Ag⁺ ions cannot hydrolyze in an acidic medium, whereas at pH values above 8, Ag⁺ ions partially hydrolyze to form bio-organic Ag complexes $[Ag(OH)_x]$ on the surface of the particles and AgOH and Ag₂O colloid in the medium, hence the degree of hydrolysis and colloid formation increases with increasing pH of the solution. As a result of this impact, no significant changes in the values of the wavelength at the highest absorbance (λ max) were detected in the extremely alkaline region, and a large number of functional groups were available for silver binding at higher pH. On the other hand, they facilitated the binding of a larger number of silver nanoparticles, resulting in a large number of nanoparticles with smaller diameters [Tripathy et al., 2010, Vivek et al., 2012, Veerasamy et

al., 2011]. 3.0 pН 5 2.5 9 Absorbance (A) 2.0 10 1.5 1.0 0.5 0.0 400 450 500 550 600 Wavelength (nm)

Figure. (3): Absorbance of (SPR) peak increased with increasing pH values



Figure. (4): The color of solutions became darker with increasing pH values

3.3 Effect of reaction time

Figure (5) shows the visible spectra of AgNPs obtained during varying periods (5 minutes to 24 hours), the absorbance of the SPR peak increased with increasing interaction time from 5 min to 24 h and the value of λ_{max} shifted from 420 to 450 nm, indicating a redshift. The red shift was most likely produced by the SPR being

suppressed by the combined effects of increased silver nanoparticle particle size in colloidal solution and anisotropy in their morphologies [Tripathy et al., 2010]. After 24 hours, there was no significant change, and this reflects the stability of the yield. The active organic compounds in the extract of the winter jasmine leaves prevented the nucleation and growth of silver nanoparticles when they exceeded the required size, which is consistent with the results of a previous study [Tripathy et al., 2010].

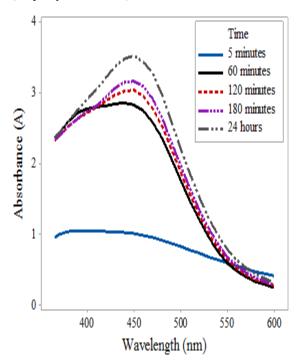


Figure. (5): The absorbance of (SPR) peak increased with increasing interaction time.

3.4 Effect of silver nitrate concentration

The results revealed that the higher the silver nitrate concentration, the higher the (SPR) peak values, as represented in Figure (6) when varied concentrations of silver nitrate solution (0.5, 1, 2.5, 5, 10 mM) were utilized. With increasing concentrations of silver nitrate, the (SPR) peak of AgNPs became increasingly evident. Due to the obvious abundance of silver ions in the reaction media, more silver particles are synthesized. All five (SPR) peaks produced a narrow and sharp peak with good symmetry, indicating a homogeneous and approximate size distribution of spherical AgNPs, and these results are consistent with previous research [Rao et al., 2017]. Additionally, figure (7) shows that when the concentration of silver nitrate increased, the color of the silver nanoparticle solution darkened, which was another proof of forming more of AgNPs.



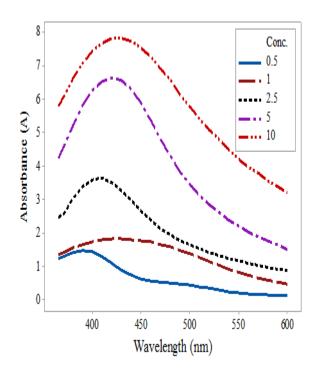


Figure. (6): Effect of silver nitrate concentration

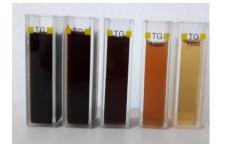


Figure. (7): How the color of the reaction mixture darkened as the concentration of silver nitrate increased

3.5 Effect of extract volume ratio

While fixing the rest of the parameters (time, temperature, AgNO3 concentration, and pH), the different volume percentages of winter jasmine extract were utilized, and when the visible spectrum of these several solutions was scanned it was observed that as the volume percentage of the extract increased, the absorbance values increased as well, as shown in figure (8), this was due to an increase in the amount of bioactive compounds in the extract that is responsible for reducing silver ions and turning them into AgNPs as it has been evidenced by comparable research [Kiruba et al., 2013]. When using a small volume percentage of the extract to react with silver nitrate, the process of forming silver nanoparticles was very slow, but when the volume

percentage of the extract was increased, the reaction speed increased, which was a sign of the formation of larger amounts of silver nanoparticles in the solution, as evidenced by the increase in the (SPR) absorption peak (figure (8)) and the darkening of the solution's color as shown in figure (9).

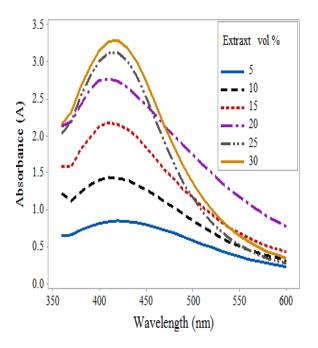


Figure. (8): Effect of extract volume percentage (%) on the synthesis of AgNPs.

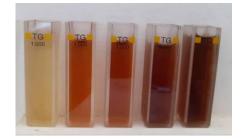


Figure. (9): The darkening of the color of the solution increases as the volume of the extract increases

3.6 Effect of temperature

When different temperatures were utilized in the synthesis process, it was revealed that the greater the temperature of the AgNPs solution, the higher the absorbance value of the (SPR) peak, specifically at 60 and 70 °C as shown in Figure (10). Furthermore, the (SPR) peak became sharper as the temperature increases, suggesting the synthesis of a significant number of similar-sized AgNPs. A previous study has noted the same observations [Singh et al., 2021].

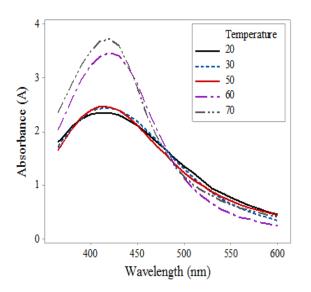


Figure. (10): effect of temperature on the synthesis of AgNPs.

4 Conclusions

Through its interaction with silver nitrate solution, the aqueous extract of winter jasmine leaves represents a suitable material for the biosynthesis of AgNPs, and large amounts of AgNPs with appropriate sizes may be obtained by controlling the parameters that affect the synthesis process (pH, reaction time, extract volume ratio, temperature, and silver nitrate concentration). It was determined that increasing the pH of the solution enhanced the productivity of AgNPs in the solution and that the basic medium was the best medium for the synthesis process. The amount of AgNPs produced increased as the temperature, volume of extract, reaction time, the extract volume ratio of the winter jasmine leaves, and silver nitrate solution concentration increased.

Characterization of AgNPs synthesized by the aqueous extract of winter jasmine leaves would require additional research, as well as using winter jasmine plant extract to synthesize gold, copper, and zinc nanoparticles.

5 Limitations of the Study

The shape and size of the silver nanoparticles that formed have not been characterized, which would have offered useful information.

Conflict of Interest: The authors declare that there are no conflicts of interest.

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