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The Role of pH in Green Synthesis of Silver Nanoparticles Using Various Plant Extracts Parts: A brief review

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A B S T R A C T

Synthesizing silver nanoparticles (AgNPs) from extracts of various plant parts and of agricultural waste is strongly affected by pH. These green synthesis methods cconsidered simple, cost effective, ecofriendly, and biologically effective. This brief review aimed to survey the role of pH in the green synthesis of AgNPs using those media, with a focus on its effect on AgNPs morphology and size. This review article revealed that the alkaline pH (9 - 10) is the optimum condition for synthesizing more stable and tiny uniform AgNPs. In addition, according to the reviewed articles, the spherical shape is the most dominant shape of these articles in the basic medium and the size of these particles decreases at elevated pH values and ranged from 5-40 nm.

1. Introduction

Silver nanoparticles, also known as AgNPs, are a class of nanomaterials that are gaining popularity because of their many uses, which include water purification, anticancer, antioxidant, and antibacterial/antifungal agents. [Omar et al, 2021, Donga & Chanda, 2021, Hembram et al, 2018]. Many physical and chemical techniques are used for synthesizing AgNPs, but there are many disadvantages of these techniques, including high costs, excessive energy use, usage of dangerous chemicals, and the release of toxic contaminants into the atmosphere [Omar et al, 2018]. Therefore, biological techniques for overcoming these drawbacks have emerged in recent years [Mostafavi et al, 2022]. These approaches, known also as biosynthesis or green synthesis, are based on the application of diverse media including bacteria, fungus, extracts of various plant parts [Saravanan et al, 2021] and extracts of agricultural waste [Omar et al, 2021, Omar et al, 2018].

These extracts contain biochemical materials that reduce silver ions to form silver atoms, which then aggregate with one another leading to AgNPs formation. The previously mentioned biochemical materials then surround these aggregations, preventing their dimensions from exceeding the dimensions of the nanomaterials and thus stabilizing them [Hembram et al, 2018, Shah et al, 2015]. Among these bioactive chemicals are phenolic alkaloids, terpenoids, proteins, carbohydrates, and polyphenols, which are assumed to be responsible for green production of metallic nanoparticles including AgNPs [Marslin et al, 2018].
Synthesizing AgNPs from botanical extracts and agricultural residues is carried out without the use of any chemicals other than silver salts and those chemicals used to modify pH of the reaction medium, mainly hydrochloric acid to make it acidic and sodium hydroxide to make it basic [Kredy, 2018, Christopher et al, 2015, Abdelmonem & Amin, 2014, Castro et al, 2013]. pH has a significant role in the synthesis of

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AgNPs from plant parts and agricultural waste. It has an impact on the quantity of the product. Also, its particle size and form [Shah, 2015]. Therefore, the purpose of this brief review was to examine the role of pH in the green synthesis of AgNPs using plant extracts and agricultural waste extracts, with a focus on pH effect on AgNPs morphology and size.

1 Plant extracts and agricultural waste extracts for nanoparticle synthesis:

More focus has recently been placed on the production of AgNPs from botanical extracts and agricultural waste. These techniques' biological applicability, cost, ease of use, and lack of toxicity make them suitable substitutes for other techniques [Shaik et al, 2018].

AgNPs can be easily generated from plant extracts and agricultural waste. The reducing componds utilized in the AgNPs production are either obtained through a standard extraction process or are utilized as soluble powder that has been previously extracted [Rajeshkumar & Bharath, 2017]. The extract solution is simply combined with the silver salt in aqueous environment and kept at an appropriate temperature for synthesis. The fact that AgNPs can be successfully produced by these reactions at room temperature is a major advantage. [Dhand et al, 2016, Alsammarraie et al, 2018]. Nonetheless, the AgNPs' shape and size are extremely dependent on the reaction coditions including pH, temperature, Ag^{+} concentration, reaction time, and extract composition and concentration…etc [Rajeshkumar & Bharath, 2017].

The phytochemicals found naturally in plants are used for producing AgNPs, these compounds include flavonoids, terpenoids, catechins, and polyphenols as well as other functional groups from aldehydes, ketones, and carboxylic acids [Sivakumar et al, 2021]. Alkaloids and flavonoids are examples of water-soluble phenolic compounds found in the botanical extracts, which serve as reducing and capping agents during the AgNPs production [Komes et al, 2011]. Silver nuclei are formed when the Ag+ ions are reduced by the functional groups present in the botanical extract. AgNPs are formed when these nuclei continue to expand as a result of the ions' accumulation at the nucleus due to subsequent reduction. [Li et al, 2007]. As the reducing agent typically acts as a covering and stabilizing agent, external chemicals are not needed for capping or stabilizing. [Sathishkumar et al, 2009]. Figure (1) shows the synthesis process of the AgNPs from plant extracts.

1 The ideal pH for synthesizing AgNPs using different aqueous plant extracts:

Reducing silver ions mainly affected by the pH. The results obtained from ultraviolet spectra when preparing AgNPs using the extract of Aldermana leaves, concluded that the optimal medium for preparing these particles was at pH = 10. It was observed that an absorption peak did not appear within the scope of 400-600 nm in the acidic media, and the absorption band began to appear with increase in the pH value. The ionization of the phenolic chemicals found in the Aldermana plant may be the cause of that. [Rousta & Ghasemi, 2019]. Similar findings were obtained in the study of the preparation of AgNPs using Pistiastratiotes L. extract, where it was affirmed that, basic pH 10 medium is required to complete the bio-reduction process for synthesizing the AgNPs [Traiwatcharanon et al., 2016].

In the study on the production of AgNPs using an aqueous extract of Agrimonia eupatoria L., the aqueous extract of the plant is an effective reducing agent and stabilizer for Agrimonia nanoparticles. Based on research on the ideal pH values for this biosynthesis, it can be concluded that an acidic environment was more suitable, and the most ideal pH value was 6 for this plant [Marković et al., 2024]. The same results were obtained in the study of the synthesis of AgNPs using Tilia cordata flower extract, and it was shown that a pH of 6 (which is for the plant extract) is required to complete the bioreduction process for synthesizing AgNPs [Corciova et al., 2018].

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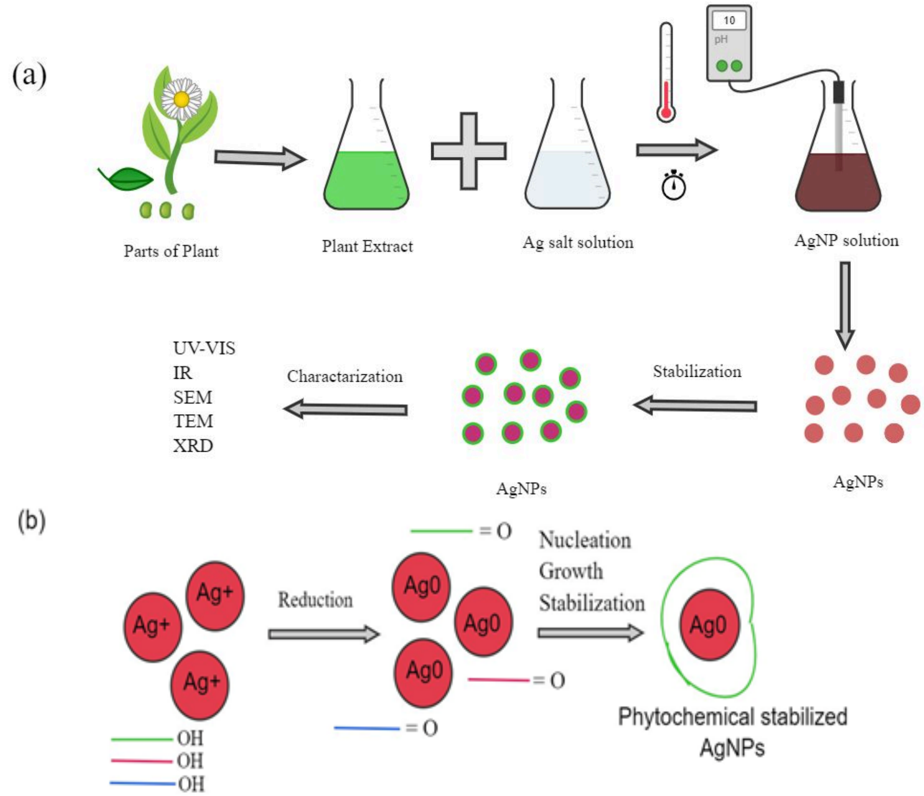

Figure 1: (a) Schematic diagram represents a synthesis of AgNPs by plants. **(b)** Mechanism of formation of AgNPs using plants.

The ability of the pH reaction to change the biomolecules' elecrtical charges, which may affect their ability to coat and stabilize, as well as the subsequent growth of the nanoparticles, is an important effect when studying the synthesis of AgNPs using *E. camaldulensis* leaves. Change in color was immediately observed in basic medium (pH 9 and 11), and nanoparticle agglomeration was observed at pH 11. pH 7 showed maximum absorption at characteristic surface plasmon resonance (SPR) peaks; (SPR) is a phenomenon of free electrons oscillations on the surface of the Nano metallic particles due to their interaction with visible light [Ider et al, 2017]; Therefore, neutral pH (7) is preferred as the optimal pH [Liaqat et al., 2022].

Pometia pinnata (Matoa) leaf extract was used as a bioreducing agent to convert silver precursors into nanoparticles. This study showed that the biosynthesis process was affected by multiple values of pH. Therefore, the synthesis process of AgNPs was conducted to research how pH affects the synthesis of AgNPs in acidic and alkaline conditions (4, 5 and 11). The characterization of the AgNPs were described by color change and the use of UV-Vis spectrophotometer device. After 24 hours of synthesis, when pH was 4 the color shifted to yellow, while brown at pH 5, and dark brown when the pH became 11. The data of the UV-Vis spectrophotometer differed depending on the pH value, as one peak appeared at 350nm at pH 4, two peaks at pH 5 at 360 and 447 nm, respectively, and two peaks at pH 11 at 422 and 673 nm, respectively [Handayani et al., 2020].

The effect of pH over the range (3-11) was studied in the green synthesis of AgNPs by Rudanti fruit, it was observed that in the acidic conditions, the peak becomes broader and the particle size increases. At pH 9, a narrow peak with a uniform size distribution is reached by the absorbance, which increases with pH [Anigol et al., 2017].

According to research on the biosynthesis of AgNPs, the ideal pH for neem leaf extract-based AgNPs synthesis was 13. Additionally, it was discovered that the pH accelerates the reduction reaction, and the color change was observed very quickly when mixing $AgNO_3$ with aqueous neem leaf extract, that is, in a few minutes the sample color became dark brown. Furthermore, it was found that the creation of the nanoparticles is inhibited at acidic pH, or pH less than 7. Whereas, the production of the nanoparticles was enhanced at higher pH by the bioavailable functional groups in neem leaf extract, this may be due to the lack of ionization of the biological groups responsible for reducing Ag^+ to Ag^0 in the acidic medium [Verma & Mehata, 2016].

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The pH effect has been studied over the range of (5.7-9) in the green synthesis of AgNPs using Aloe fleurentinorum extract. The appropriate pH value for this preparation was at pH = 8 [Jamil et al., 2024].

Synthesizing AgNPs by aqueous extract of Azadirachta indica leaves required pH 13, where sharp peak appeared. The color of the solution did not change at pH 5 and 7, which confirms that the acidic and neutral mediums are not suitable for the formation of AgNPs from this plant extract [Ansari et al., 2023].

In a study conducted by Kahraman about the synthesis of silver nanoparticles using Alchemilla vulgaris and Helichrysum arenariumthe, it was found that the pH affected the absorption curve of the solution of the plant extract and silver salt. Changing the pH affected the rate of reduction of the Ag^+ ion to Ag^0, by observing a change in the color to dark brown. It is also worth noting that the process of synthesis of AgNPs depended on the role of the active substances of the extract in the reaction medium, which was affected by pH. When the pH dropped to 5, the absorption peak at 412 nm increased. [Kahraman, 2024]. In other study, gallic acid was used for synthesizing AgNPs and the optimal pH was 9 for the synthesis of more AgNPs with a smaller average size of 5.77nm [Ahani & Khatibzadeh, 2022].

According to the investigation conducted by Nahar et al. the form and size of AgNPs were affected by change in the pH, by changing the charge of the biomolecules of the plant extract, which may affect their capping ability and the stability of the resulting particles. This research indicated that pH = 9 was the optimal pH for the synthesis and stability of AgNPs by Cinnamomum tamala leaf extract [Nahar et al., 2020].

Husam M. kredy conducted a research on synthesizing AgNPs using Lawsonia inermis extract, the color intensity of the reaction medium was observed at pH 9. There was no reaction at pH 3, but monodispersed silver nanoparticles were obtained at pH 9 [Kredy, 2022].

Briefly, pH is considered an important factor affecting the synthesizing process of the AgNPs, and this process increases in basic media, while it decreases or does not occur in acidic and neutral media. This can be attributed to the fact that some functional groups in the plant extract, such as OH, which are present in polyhydroxy phenolic molecules, do not ionize in acidic and neutral environments and therefore do not play their role as reducing agents for the silver ions and converting them into nanoparticles. Table 1 shows the ideal pH for the synthesis of AgNPs from different botanical extracts used as reducing and coating agents.

Table 1: The ideal pH for synthesizing AgNPs using different botanical extracts.

Reducing Agent	Optimal pH			References
	Acidic	Neutral	Basic	
Dermaneh leaf extract	-	-	10	Mohammad et al,2019
Pistiastratiotes L. extract			10	Pranlekha et al,2015
aqueous extract of Agrimonia eupatoria L.	6	-	-	Ana et al,2023
Tilia cordata Flowers extract	6	-	-	Andriea et al,2018
E. camaldulensis leave extract	-	7	-	Nida et al,2022
Pometia pinnata (Matoa) Leaves Extract	5	-	11	Handayani, 2020
Rudanti fruit	-	-	9	Anigol, 2017
Neem leaves extract	-	-	13	Aparajita, 2016
Extract of Aloe fleurentinorum	-	-	8	Yasmin, 2024
Azadirachta indica leaves extract	-	-	13	Madeeha et al, 2023
Alchemilla vulgaris	5	-	-	Havva, 2024
gallic acid	-	-	9	Mina et al ,2022
Cinnamomum tamala leaf extract	-	-	9	Shahin et al,2020
Lawsonia inermis extract	-	-	9	Husam, 2018

1 pH effect on the morphology of the AgNPs:

Literature review on the relationship between pH and morphology of AgNPs has shown that the shape of these particles relies on the pH level of the reaction [Restrepo & Villa, 2021, Sohrabnezhad et al., 2016, Nasir et al., 2023]. Several researches revealed that changing pH could lead to different shapes of AgNPs [Villanueva-Ibáñez et al, 2015, Mukherji et al, 2019]. According to many reports, various morphologies of AgNPs were created by adjusting the pH of the medium [Fernando & Zhou, 2019, Devi et al, 2019]. Additionally, the

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phytochemicals (mostly polyphenols) of the botanical extract played role in the reduction and stabilization of the formed AgNPs, which became smaller and uniform in basic conditions [Devi et al, 2019].

The Surface Plasmon Resonance (SPR), depending on the pH, can describe the shape and size of the AgNPs [Riaz et al, 2021]. Velgosová et al conducted a reseach work on green synthesis of AgNPs, it was found that the formed particles have limited size range and spherical shape at higher pH, which appeared by the symmetrical and narrow SPR band at pH values around (8-10) and remain stable by time. Whereas, the AgNPs SPR band generated (or produced) in the pH 4 and pH 6 solutions began to broaden after period of time [Velgosová et al 2016].

The morphology of AgNPs tend to become more spherical at higher pH values. For example, Sathishkumar et al. found that the shape of AgNPs, that were created using bark extract from Cinnamon zeylanicum, tended to become spherical instead of elliptical at higher pH values (pH ≥ 5) [Sathishkumar et al, 2009]. This finding demonstrated the critical impact that pH plays in regulating the size and form of the production of AgNPs. Andreescu et al also noted a comparable pH effect, along with the quick and total reduction of the silver species at higher pH values [Andreescu et al, 2007].

In research by Seifipour et al [Seifipour et al, 2020], AgNPs were synthesized using tragopogon collinus extract under different conditions and their results revealed that the TEM images depicted crystals of AgNPs at pH10.

According to the investigation conducted by Anigol et al about the pH role on the form and size of AgNPs prepared using capparis moonii fruit extract, the size increased in acidic conditions and decreased in basic conditions, tiny spherical nanoparticles formed at elevated pH, and their amount increased. One explanation for this outcome could be that the rate of the reaction was higher at the elevated pH, and smaller particles eventually crystallized as a result of that. This process included the nucleation and development of tinier particles from the nuclei of Ag [Anigol et al, 2017].

In a different work, AgNPs were prepared using Camellia Sinensis extract which contains polyphenols as the active groups that are responsible for the reduction and stabilization of the AgNPs. The morphology of these particles were strongly affected by fluctuations in pH and temperature. It was discovered that AgNPs with various morphologies (spherical, polygonal, and capsule) formed at pH 5 and 25 °C. However, when the temperature was raised from 25 °C to 65 °C, the reduction rate increased causing these particles to start attaining the spherical shape with varying diameters at the same pH. Moreover, an increase in pH from 5 to 11 at 65 °C led to increasing in the monodispersity of AgNPs with spherical shape, which was explained by the facilitation of the reduction by hydroxide ions [Riaz et al, 2021].

To sum up this section, the morphology of the green synthesized AgNPs depends strongly on pH and various morphologies can be formed in acidic conditions while the spherical shape is mostly prevailing in basic conditions.

2. pH effect on the AgNPs size:

AgNPs produced in the Nurfadhilah et al. study from an aqueous extract of D discolor leaves. Size, density, dispersion, and stability variations were observed by varying the pH of the solution. At pH 7, 9, and 11, the produced AgNPs had sizes in the range of 80 nm, <50 nm, and <35 nm, in that order. The generated nanoparticles tend to be smaller and can grow up to 5 nanometers in size when the pH rises [Nurfadhilah et al, 2018].

The size and stability of the AgNPs may be impacted by the pH of the solution, according to a different study by Velgosová et al. On the other hand, AgNPs produced in basic solutions with pH values of 8 and 10 are small, polyhedral, stable, and have a narrower size interval. Their average size is 15 nm [Velgosová et al 2016].

In their research, Kaur et al. found that the production of AgNPs from Litchi Chinensis leaf extract produced particles that were highly stable at physiological pH, ranging in size from 10 to 50 nm, with an average size of 43 nm at the ideal pH of 7 [Kaur et al, 2021].

A study was designed to use oak fruit peel extract (Jaft) to manufacture AgNPs. Dynamic light scattering (DLS) and transmission electron microscopy (TEM) analyses revealed that the produced AgNPs were spherical, with an average size of 40 nm at pH 9 [Heydari & Rashidipour, 2015].

The sea buckthorn berry extract was used to study the green synthesis of AgNPs. Dynamic light scattering (DLS) analysis and UV-visible spectroscopy revealed that the pH had an impact on the manufactured particle size. The produced particles were found to be spherical in shape, with an average size of 27.3 ± 0.2 nm at pH 10 and to exist in a monodisperse form, according to transmission electron microscopy (TEM) data [Wei et al, 2020].

Chowdhury et al. carried out an investigation to create AgNPs at 25°C using carambola fruit extract at various pH levels. The produced particles' size decreased as the pH of the solution increased, as demonstrated by the ultraviolet and visible absorption, which showed that the peaks' intensities grew narrower and higher. AgNPs' average particle size was revealed by transmission electron microscopy (TEM) to be 16, 13, and 12 nm for pH values of 4, 7, and 10, respectively [Chowdhury et al, 2019].

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An extract from the bulb of Crocus Haussknechtii Bois was used to produce small AgNPs. Optimizing the experimental conditions such as temperature and pH could produce spherical nanoparticles with average size 10–25 nm at pH 7 [Mosaviniya et al, 2019].

In a study conducted by Nadzirah and his group, AgNPs were prepared using Polygonum minus extract as bioreducing agent. In this context, the results found that the temperature rising led to accelerate the AgNPs production, and the monotonically increasing absorbance indicated that the basic pH was more suitable for synthesizing AgNPs at pH 8. TEM inspection revealed spherically shaped AgNPs with good diffusion. The size of their particles varied from 6 to 21 nm [Nadzirah et al, 2023].

Finally, this review concluded that the basic medium is where the synthesis of tiny and stable AgNPs occurs when plant extracts are used as a bioreducing agent.

1 Conclusions

Research studies on the green production of silver nanoparticles using plant extracts and agricultural waste have become increasingly popular. Optimizing the size and shape of the generated AgNPs is largely dependent on the synthesizing process, which is influenced by numerous factors. One of these characteristics is the pH, and variations in the medium's pH can have an impact on the size and form of these particles. This review revealed that the synthesis process increases in basic media, while it decreases or does not occur in acidic and neutral media. In addition, smaller and highly organized spherical AgNPs can be synthesized under alkaline conditions. Whereas, various morphologies and larger AgNPs can be formed in acidic conditions. However, the shape and size of the green produced AgNPs are also influenced by other variables, such as temperature and plant extract content; additional research in this area is needed.

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

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