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Effect of pH in The Biosynthesis of Gold Nanoparticles: A Review

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Abstract:
This review aimed to investigate the effect of pH on morphology, and size of gold nanoparticles (AuNPs) biosynthesized using extracts of various plant parts and agricultural waste. By reviewing previous studies, it was found that the formation of AuNPs was more rapid in neutral and basic mediums than in acidic mediums. Furthermore, most of those studies indicated that the smallest sizes of biosynthesized AuNPs, produced by plant extracts, were in neutral and alkaline mediums. Regarding the morphology, it was found that multiple shapes of the AuNPs were obtained in an acidic medium of the reaction, while there was typically a dominant shape of the AuNPs in the basic medium. In conclusion, significant quantities of AuNPs with appropriate sizes and morphology could be obtained by controlling the medium of the reaction when AuNPs are biosynthesized using extracts of plant parts and agricultural waste.

Keywords: Biosynthesis, gold nanoparticles, pH, AuNPs size, AuNPs morphology.

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تأثير الأس الهيدروجيني في تخليق جسيمات الذهب النانوية: مراجعة

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الملخص
الهدف من هذه المراجعة كان للبحث في تأثير الأس الهيدروجيني على الشكل، والحجم لجسيمات الذهب النانوية التي تم تخليقها باستخدام مستخلصات من أجزاء نباتية مختلفة ومن النفايات الزراعية. ومن خلال استعراض الدراسات السابقة، تبين أن تكوين جسيمات الذهب النانوية كان أكثر سرعة في وسط متعادل وقاعدي منه في وسط حمضي. علاوة على ذلك، تشير معظم تلك الدراسات إلى أن أصغر أحجام جسيمات الذهب النانوية المخلقة بواسطة مستخلصات نباتية كانت في الوسط المتعادل والقاعدي. وبالنسبة للشكل، تبين أنه تم الحصول على أشكال متعددة لجسيمات الذهب النانوية في الوسط الحمضي للتفاعل، بينما كان هناك شكل سائد لجسيمات الذهب النانوية في الوسط القاعدي. ختاماً، يمكن الحصول على كميات كبيرة من جسيمات الذهب النانوية ذات الأحجام والأشكال المناسبة من خلال التحكم في وسط التفاعل، عندما يتم تخليق جسيمات الذهب النانوية باستخدام مستخلصات من أجزاء نباتية ونفايات زراعية.

الكلمات المفتاحية: تخليق حيوي، جسيمات الذهب النانوية، درجة الحموضة، حجم جسيمات الذهب النانوية، شكل جسيمات الذهب النانوية.

Introduction

Nanomaterials are those materials with at least one dimension of less than 100 nanometers [1]. This decrease in the dimensions, along with their unique shape and structure, causes a significant change in the chemical, physical, electrical, and mechanical properties of nanomaterials when compared to bulk materials, resulting in a wide range of applications [2,3].

Metal nanoparticles (MNPs) are a type of nanomaterial that includes gold, silver and iron nanoparticles. They have been manufactured in several ways, such as chemical, physical, and biological methods based on the used materials [4]. Researchers have recently become more interested in biological methods (biosynthesis or green synthesis) to synthesize MNPs, because they are simple, easy to implement, cheap, and environmentally benign compared to the other methods [2,3,5,6].

Many materials have been used in biological methods, including bacteria, fungi, algae, various plant parts [7,8], and agricultural residues [8,9]. Plant extracts contain biochemical substances that reduce metal ions, which aggregate with one another, and then the aforementioned biochemical substances surround these clusters of metal ions to prevent the dimensions of these clusters from exceeding the dimensions of the nanomaterials [10]. Among the bioactive chemicals that are thought to be responsible for green MNPs production are alkaloids, terpenoids, proteins, carbohydrates, and polyphenols [11].

MNPs, including gold nanoparticles (AuNPs), are biosynthesized without the use of any chemicals other than metal salts and those used to adjust the pH of the reaction solution, mostly hydrochloric acid and sodium hydroxide [12, 13].

As the sizes of gold nanoparticles produced via biosynthesis routes, as well as their shapes, are all affected by the pH of the reaction medium [14], this review aimed to investigate the role of pH on the size and the morphology of AuNPs biosynthesized using extracts of various plant parts and agricultural waste.

Plant extracts and agricultural waste extracts for nanoparticle synthesis:

Recently, the design of environmentally acceptable experimental techniques for the fabrication of NPs has been considered an essential field of nanotechnology [15]. Further, a safe technique for reducing and stabilizing AuNPs is provided by producing them using plant extracts and agricultural waste extracts. Therefore, with the aid of various bio components found in many plant extracts and agricultural waste, it is possible to reduce Au salts and stabilize AuNPs [8-10]. Convenient and rapid routes to reductively synthesize AuNPs employing different plant extracts have been comprehensively studied [16].

For the fabrication of plant-mediated AuNPs, plant extracts can act as effective reducing agents by their different functional groups (OH, NH2…etc.) presented in compounds such as polyphenols, flavonoids, alkaloids, and tannins. Also, the production of AuNPs has been achieved through a controlled reduction of gold ions to atoms [17]. In most cases, it is not necessary to add stabilizing and capping agents, because the combination of bio molecules present in the plant extracts can do their roles [18].

To generate NPs by plant Extracts from different parts of the plants including stems, leaves, latexes, fruit pericarps, fruit juice, seeds, and roots, the extracts are simply added to the metal salt solution at different temperatures. Then the reaction takes place in a few minutes as shown in figure (1). Nanoparticles of gold and other metals have been synthesized using this method [19].

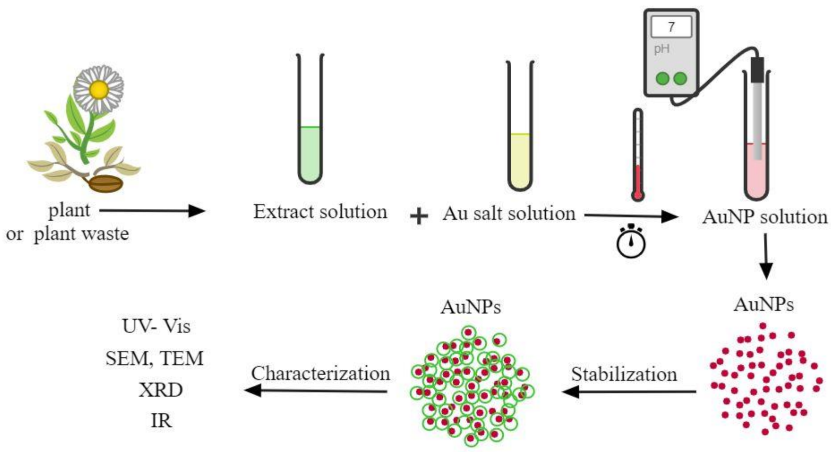

Figure 1: Schematic diagram illustrating the plant-mediated synthesis of gold.

The production of these NPs is influenced by several factors, including the type and concentration of plant extract and salt used the pH of the medium, the temperature, and the reaction time. These parameters play a crucial role in determining the quantity, the shape, and the size of the resulting NPs [20].

Besides that, the successful synthesis of AuNPs is verified through two key observations. Firstly, a noticeable alteration in the color of the extract solution. This color can be changed by adjusting the concentration ratio between the Au salt and the extract. Moreover, the variations of the color are resulting from the changes in the size, shape, and composition of particles [21]. Additionally, the Surface plasmon resonance (SPR) can be used to confirm AuNPs synthesis, with the aid of UV-Vis spectrophotometer (~580 nm). SPR is defined as an optical phenomenon occurring at metal surfaces, when the ray beams strike the surfaces at a specific angle. It is a feature that can be linked to the collective oscillation of conduction electrons as produced by an electromagnetic field, causes these nanoparticles to typically display very intense colors when dispersed in liquid mediums [22]. Further, it has been found that each of the size, shape, aggregation, and composition of nanosized metal particles, as well as the proximity of other metal nanoparticles, significantly affect SPR frequency [23]. These findings provide strong evidence for the successful synthesis of AuNPs [24]. Figure 2 represents typical SPR spectra of the synthesized AuNPs.

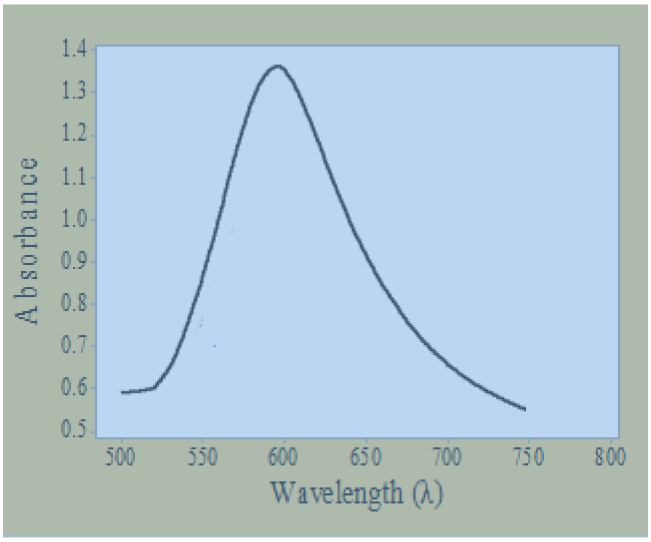

Figure 2: SPR spectra of the synthesized AuNPs.

However, further research is needed to optimize the synthesis conditions and understand the mechanisms behind the formation of these NPs using plant extracts.

The optimal pH for the synthesis of AuNPs using different aqueous extracts of plants:

In addition to precursors and reducing agents, pH has a considerable impact on the synthesis of nanoparticles. It was found that pH 7 and 9 gave good results for the synthesis of AuNPs resulting from the reaction between Commiphora wightii leaves extract and tetrachloroauric acid (HAuCl4). This indicates that neutral and basic mediums facilitated the biosynthesis of AuNPs. While in the acidic medium, the anionic electrostatic repulsion of the bioactive molecular groups in the solution led to the agglomeration of AuNPs and a lowering in the reduction process of Au ions [25]. The same results were obtained in the study related to the synthesis of AuNPs using acer pentapomicum leaves extract; it was proved that the neutral and basic pH (7-8) were required to complete the bio-reduction process for the biosynthesis of AuNPs [26].

In the study related to the synthesis of AuNPs using Sambucus wightiana whole plant extract as a reducing and stabilizing agent, the pH was adjusted by using phosphate buffer, and it was concluded that the AuNPs exhibited maximum absorption at neutral and acidic medium, whereas the peak height of the SPR peaks decreased at basic medium [27].

The synthesis of AuNPs utilizing licorice root extract and HAuCl4.3H2O solution required a pH of 5, which generated a narrow band at 540 nm. This means that the highest reduction of gold ions occurred in acidic medium. In comparison, the pH 4 solution presented a weak SPR intensity due to the insufficient production of reducing species, which resulted in a low yield of AuNPs [28]. According to the study by Chun-Gang et al., basic medium worked optimally for the biosynthesis of AuNPs using citrus maxima peel extract, which probably quickened the reaction rate and enhanced homogenous nucleation [29]. The role of the red cabbage extract's pH on the synthesis of AuNPs was evaluated by Lekeufack and Brioude, who found that at higher pH (11-12), the AuNPs were well dispersed. On the contrary, under acidic conditions, the reaction rate was slow, and small AuNPs seeds underwent secondary nucleation and heterogeneous nucleation as a result [30].

The pH effect has been studied over the range of (2-14) in the green synthesis of AuNPs by C. buchanani extract, and it was observed that the preferred pH was (7) for AuNPs synthesis. This may be due to the availability of sufficient biomolecules in the extract that are responsible for reducing Au ions in the solution and converting them to elemental Au^0, to form AuNPs according to a certain mechanism. Whereas a strong alkaline medium with a pH of (14) caused the solution to darken black, which is the consequence of the precipitation of gold nanoparticles [31]. In contrast, in a one-pot green synthesis of AuNPs capped and stabilized with gall extract of Pistacia integerrima, AuNPs were optimally synthesized at pH (4-5) and (10-11). However, enhanced AuNP synthesis were observed at alkaline pH [32].

The optimum pH obtained from the experiment of AuNPs green synthesis by aqueous garlic (Allium sativum L.) extract was (4) [33]. High values of pH (basic medium) led to rapid reduction of Au ions to AuNPs in the synthesis of AuNPs by Cacumen Platycladi leaf extract. This medium may promote homogeneous nucleation and decrease anisotropy growth, whereas the opposite occurs in an acidic medium; slow reduction rate resulting in the heterogeneous nucleation and secondary nucleation of small Au seeds [34].

Another study revealed that the alkaline pH (~8) was preferred for the biosynthesis of AuNPs utilizing Fagonia indic aqueous extract, which may indicate that a higher pH induced more nucleation and growth of metal nanoparticles in general [35].

S.A Aromal and D. Philip performed a study on the biosynthesis of AuNPs using the aqueous extract of fenugreek seeds (Trigonella foenum-graecum), and they found that at lower pH, the SPR band was broad, indicating poly-dispersed AuNPs. The SPR band sharpened at a higher pH (6-7), which was a result of mono-dispersed AuNPs [36].

Finally, we have concluded that the formation of AuNPs was more rapid in neutral and basic mediums than in acidic mediums; this may be due to the ionization of the bioactive groups present in several parts of plants. It should also be mentioned that the Au ions cannot hydrolyze at an acidic pH, whereas at a higher pH, they partially hydrolyze to form bio-organic Au^{+3} complexes on the surface of the particles. As a result, a large number of functional groups were available for gold binding at higher pH to produce a lot of AuNPs. Table 1 shows the optimal pH for the synthesis of AuNPs using different plant extracts that were used as reducing and capping agents, as indicated below:

Table 1: The optimal pH for the synthesis of AuNPs using different plant extracts.

The reducing agent	**The optimal pH**			**References**
	Acidic	**Neutral**	**Basic**	
Commiphora wightii leaves extract	-	7	9	[25]
Acer pentapomicum leaves extract	-	7	8	[26]
Sambucus wightiana leaves extract	3, 5	7	-	[27]
licorice root extract	5	-	-	[28]
Citrus maxima peel extract	-	-	9,11	[29]
red cabbage extract	-	-	11-12	[30]
C. buchanani extract	-	7	-	[31]
gall extract of Pistacia integerrima	4-5	-	10-11	[32]
Garlic (Allium sativum L.) extract	4	-	-	[33]
Cacumen Platycladi leaf extract			10-12	[34]
Fagonia indica aqueous extract	-	-	8	[35]
fenugreek (Trigonella foenum-graecum) seeds extract	6	7	-	[36]
Zingiber officinale root extract	-	-	9	[37]

The effect of pH on the size of AuNPs:

The pH has played a very important role in controlling the size of green-synthesized AuNPs. In the investigation of Bogireddy et al., they synthesized AuNPs by using Coffea arabica seed extract (CAS) as a reducing and capping agent. The size of AuNPs was significantly influenced by the pH of this extract when combined with Au ion solution. The results showed that the smaller-sized AuNPs (~28 nm) were generated when the pH of the reaction mixture was higher (pH >10), while the bigger-sized AuNPs (~69 nm) were synthesized when the pH was lower (pH >3), probably as a result of the limitation of the capping agent availability [38]. In contrast, the average size of biosynthesized and stabilized AuNPs using the antidiabetic potent plant Cassia auriculata was (15–25 nm) at a wide range of pH (3–10), which indicates that the pH has no effect on the size and stability of AuNPs [39].

In another investigation, the size of AuNPs decreased from 90 to 40 nm when the pH ranged from (3) to (7), according to Transmission Electron Microscopy (TEM) experiments in the article discussing the biosynthesis of AuNPs using black cardamom [19].

Mostafa Khalil et al. found that the strong anti-oxidant capabilities of the high phenol content of the aqueous extract of olive leaves assisted in the reduction of gold cations to AuNPs, and the reduction process was accelerated in the basic medium. This result was supported by TEM studies at pH (3) and (10), which revealed that in an acidic medium, the particle size was larger than in a basic medium. This is because a higher pH will facilitate the synthesis of smaller nanoparticles and improve the efficiency of surface capping on AuNPs [15].

The obtained optimized particle size of AuNPs synthesized from aqueous extract of garlic (Allium sativum L.) as a reducing and stabilizing agent was also characterized using TEM, and it was 15±3 nm at the low pH (~4) [40].

According to the investigation of the biosynthesized AuNPs produced by Garcinia kola pulp extract, the particle size ranged from 18 to 38 nm, with an average size of 28 nm when the optimum pH was 7 [41].

The optimal size of stable AuNPs synthesized from Acer Pentapomicum leaves extract was determined by Scanning Electron Microscopy (SEM), and they ranged from 18 to 25 nm when the mixture reaction was neutral to basic (7-8) [42].

Finally, we found through this study that the smallest sizes of biosynthetic gold nanoparticles produced by plant extracts from different parts of the plant was in neutral and alkaline mediums, which is due to the high availability of bioactive groups in these two mediums.

The effect of pH on the morphology of the AuNPs:

Many studies have reported that the morphologies of the AuNPs depend strongly on the pH variation of the reaction medium [38, 43- 47]. Hence, the shape of the nanoparticles differs depending on the concentration of hydrogen ions [43].

In a study by Bogireddy and his group, AuNPs were synthesized using Coffea arabica seed as reducing and capping agents [38]. In this context, their results revealed that along with some spherical particles, the biosynthesis process generated large quantities of anisotropic AuNPs with a variety of shapes. While bigger-sized AuNPs are fabricated when the pH of the reaction mixture is low (pH < 3) probably as a result of the limitation of the capping agent availability (OH– functional groups) which provided by the constituents of plant, smaller-sized quasi-spherical nanoparticles are fabricated when the pH of the reaction mixture is higher (pH > 10).

In a similar investigation, AuNPs were synthesized by Singh and Srivastava using black cardamom extract as a reducing agent. They have reported that AuNPs at pH (3, 7) have a spherical flower-shaped morphology whereas two types of particles are produced at pH (11), one of which is triangular and the other almost spherical [16].

In research by Khalil and Ismail [15], AuNPs were synthesized using olive leaf extract as bioagent. Their results revealed that the TEM images depicted a mixture of shapes such as triangular, hexagonal and spherical shapes obtained at pH (10), while smaller spherical nanoparticles were formed at pH (3).

Ghodake et al. [44] also reported a single-step biological route for fabricating AuNPs using pear fruit. In that study, they observed significant changes in the nanostructures by modulating the pH of the reaction mixture where Au ions bind with pear biomass in a pH-dependent manner. Under acidic conditions (pH 4), the obtained mixture consisted of triangular, partially hexagonal and spherical nanoparticles, whereas the gold particles induced under neutral conditions showed plate-like structures. Moreover, the gold nanostructures produced under alkaline conditions pH (9) consisted of different shapes, including triangular nanoparticles, hexagonal nanoparticles and others.

Wacławek et al., studied the impact of Tarragon extract on the synthesis of AuNPs [45]. According to this study, the initial pH value influenced the shape of the formed AuNPs.Triangle and spherical shaped nanoparticles were mainly generated when the initial pH was acidic (2.8- 4.0) during the synthesis. Further, it was found that no triangular nanoparticles formed when the medium was further increased to pH (5).

Castroet utilized the sugar beet pulp for the fabrication of AuNPs. It was reported that the shapes of the fabricated nanoparticles were controlled by changing the pH values of the reaction medium: at low pH (2-4), triangular and hexagonal nanoparticles were obtained, whereas gold nanowires were produced at higher initial pH (10). Furthermore, AuNPs at pH (7) have rods, spherical and triangular shaped morphology [46].

Another study [47] demonstrated a biosynthesis of gold nanoparticles using an extract of grape leaves and seeds. The results showed that the shape of the produced nanoparticles was controlled by varying the initial pH value of the reaction medium: hexagonal structures particles at pH (4.3) and spherically in shape at pH (8.6).

Briefly, many scientists were able to produce AuNPs in anisotropic shapes, using plant extracts when the reaction conditions were adjusted concerning pH. However, it is important to note that the correlation between pH and AuNPs shape is complex and it can be influenced by many variables such as the concentration of the plant extract, the reaction temperature, and the presence of any additional chemicals in the reaction mixture [48].

Finally, it could be say that, even when utilizing the same plant extract, there are multiple shapes of the AuNPs in an acidic medium of the reaction, while there is typically a dominant shape of the AuNPs in a basic medium.

Conclusion:

The field of nanotechnology recently recognized the importance of developing environmentally friendly methods for synthesizing nanoparticles (NPs). One such method involves, using plant extracts and agricultural waste extracts to reduce Au ions, and then produce and stabilize gold nanoparticles (AuNPs). Extensive research has been conducted to explore convenient and efficient ways to synthesize AuNPs using different plant extracts. The production of AuNPs is affected by various factors including the pH of the medium parameter, which is important in determining the amount, shape, and size of the obtained AuNPs. This review revealed that various plant extracts are effective in producing AuNPs, which is an acceptable green chemistry procedure. The review also revealed that different values of pH can influence the size and the shape of AuNPs, The majority of research articles (reviewed in the current study) indicate that the basic medium is the optimum condition. Controlling the pH and other factors that affect the biosynthesis process could result in significant amounts of AuNPs with appropriate sizes and morphology. More research is needed to review the impact of other factors, such as temperature and reaction time, on the size and morphology of AuNPs produced via the biosynthesis route.

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