

Influence of irradiation time on the structural and optical characteristics of Ag nanostructure produced by atmospheric pressure plasma

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ABSTRACT

This article presents a method for producing silver nanoparticles through plasma at atmospheric pressure using electrochemical techniques. Initially, an electrochemical configuration is designed using atmospheric pressure plasma (a direct current (DC) source). Subsequently, silver nanoparticles were produced in an environment without a stabilizer and evaluated according to the treatment time. The results demonstrate that the plasma-based electrochemical method, in comparison to other methods, including traditional electrochemical, can increase the accuracy in the shape and structure of nanoparticles while simultaneously accelerating the synthesized processes of nanomaterials. Analysis revealed that the synthesized nanostructures range between 2 and 3 nanometers in size and exhibit rod-like shapes, among other morphologies.

1 Introduction

Today, nanotechnology materials play a crucial role in a world where science and technology are advancing at an unprecedented speed, making their use a high priority. Nanostructures with dimensions in the nanometer range exhibit physical and chemical properties that differ significantly from other materials, sparking increased interest from researchers and industry in this field. Among the various types of nanostructures, metal nanoparticles hold particular importance due to their exceptional electrical, optical, chemical, and magnetic properties. Colloidal nanoparticles of these metals are essential in technology, particularly in the production of glass and ceramics. Moreover, silver nanoparticles are known for their antimicrobial and antibacterial properties, making them ideal for specific applications in this field [1-14].

In recent years, numerous methods for synthesizing nanoparticles have been researched, each offering different advantages and limitations. The various techniques used to produce silver nanostructures include electrochemical methods, chemical deposition, hydrothermal processes, and plasma synthesis [15-19]. In particular, the electrochemical plasma method has proven to be a particularly innovative and effective approach. In this technique, electrochemical and plasma processes are synergistically combined, allowing precise control over the size, morphology, and physical and chemical properties of silver nanoparticles [20-22].

The electrochemical plasma method utilizes the unique interactions between electrochemical reactions and plasma environments to produce silver nanostructures with enhanced control over their properties. By varying the parameters of the electrochemical and plasma processes, researchers can customize the dimensions and surface properties of the nanoparticles to meet

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specific application requirements. This method not only offers improved reproducibility but also the potential for scalable production, making it a valuable tool in the field of nanomaterial synthesis [20-27].

In addition, the electrochemical plasma approach can facilitate the formation of nanoparticles with higher purity and uniformity compared to conventional methods. The synergy between electrochemical reactions and plasma generation enables more efficient nucleation and growth of silver nanoparticles, thereby minimizing the presence of unwanted by-products. This enhanced control and efficiency position the electrochemical plasma method as a promising candidate for both fundamental research and industrial applications in nanotechnology [20-27].

The plasma method provides precise control over nanoparticle properties, such as size, morphology, and structure, due to the unique interaction between electrochemical reactions and plasma environments. This level of control is difficult to achieve with other conventional methods like chemical deposition and hydrothermal synthesis [20-27].

Additionally, the plasma-based synthesis process enables the formation of nanoparticles with higher purity and uniformity. The combination of plasma and electrochemical reactions improves the nucleation and growth stages, reducing unwanted by-products and leading to more consistent nanoparticle structures. Furthermore, this method allows for quicker synthesis while maintaining particle quality, making it efficient and beneficial for scalable production. It enables faster adjustment of synthesis parameters to achieve the desired nanoparticle properties [20-27].

Another advantage of the plasma technique is its ability to synthesize nanoparticles without requiring high temperatures, making it suitable for temperature-sensitive or reactive materials. This characteristic expands its applicability, particularly in situations where high thermal energy could be detrimental to the material. Including these points in the introduction would underscore the superior control, purity, speed, and versatility of the plasma method, highlighting its relevance in advanced nanomaterial synthesis.

To emphasize the unique advantages of the plasma-based electrochemical method, we can mention its

environmental and energy advantages compared to other traditional nanoparticle synthesis methods. Unlike chemical reduction techniques, which often require toxic reagents and stabilizers, the plasma method can produce silver nanoparticles in a cleaner and more sustainable manner. The electrochemical plasma process operates without stabilizing agents, minimizing harmful chemical waste, which makes it particularly suitable for environmentally sensitive applications. Additionally, the plasma method operates at ambient conditions (room temperature and atmospheric pressure), significantly reducing energy consumption. This aspect not only enhances safety but also enables cost-effective scaling for industrial applications [20-27].

Furthermore, the plasma method provides real-time control over particle synthesis through simple adjustments to parameters such as current and reaction time. This flexibility allows for rapid optimization of nanoparticle characteristics to meet specific application requirements, such as desired size, morphology, or crystallinity. Such dynamic control is harder to achieve with conventional methods, where synthesis conditions are often rigid and complex to modify mid-process.

Ongoing research is expected to further elucidate the mechanisms underlying this method and optimize the parameters for various applications. Further development of electrochemical plasma synthesis could pave the way for advances in the design and functionality of nanomaterials and expand their utility in various technological and scientific fields.

The effective application of nanostructures in various fields necessitates the production of nanoparticles with optimized properties, which, in turn, requires careful control of the synthesis process. Central to this optimization is the study of the factors that influence synthesis, particularly the role of time in shaping the properties of silver nanoparticles. Recent studies have shown that time is a crucial variable that influences the structural and functional properties of silver nanostructures produced by electrochemical plasma synthesis. Understanding and determining the optimal synthesis time is, therefore, crucial for enhancing the properties of these nanoparticles [27-30].

Research indicates that the duration of synthesis is a critical factor in determining the size, shape, and overall quality of silver nanoparticles. Extended or inadequate synthesis times can substantially alter the morphology and surface characteristics of the nanoparticles, leading to variations in their performance in real-world applications. Consequently, a thorough examination of the temporal evolution of the electrochemical plasma synthesis process is essential. This strategy not only aids in refining nanoparticle properties but also enhances the consistency and effectiveness of the synthesis procedure [31,32].

Given the influence of synthesis time on the rate, quality and properties of silver nanostructures, this study emphasizes the importance of a comprehensive investigation of time-dependent effects in electrochemical plasma synthesis. By methodically investigating how changes in synthesis time affect the properties of the produced nanoparticles, our research attempts to provide recommendations for achieving the desired nanoparticle properties and improving their potential applications.

In addition, this article deals with the particularities of the synthesis of silver nanostructures using electrochemical plasma techniques and their subsequent characterization. By analyzing the fabrication process in detail and evaluating the resulting nanostructures, this study aims to clarify the correlation between the synthesis time and the properties of the nanoparticles, thus improving the overall understanding and refinement of electrochemical plasma synthesis methods.

2 Experimental Section

2.1 Plasma reactor

A direct current source with a voltage of 2 kV was utilized to synthesize silver nanostructures through an electrochemical method combined with atmospheric pressure plasma. In the plasma structuring process, a hollow cylindrical stainless-steel tube with an outer diameter of 0.7 mm and a length of 9 cm serves as the cathode, positioned 5 mm above the liquid surface and 3 cm away from the anode. Argon gas at a flow rate of 200 sccm is employed for plasma generation (Figure 1). The typical current of the plasma used is 5 mA.

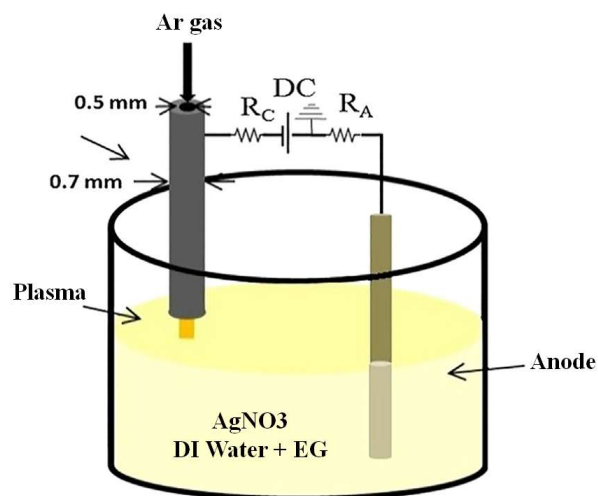


Figure 1. The apparatus diagram of Ag nanoparticles synthesis.

During production, a platinum electrode is used in the reaction cell. The electrolyte used in this setup consists of a silver nitrate solution with concentrations ranging from 1 millimolar to deionized water. When the silver nitrate solution is dissolved in water, silver and nitrate ions are formed. When using the plasma, the discharge current is maintained in a stable state and regulated by the direct current source.

This experimental setup enables precise control over the synthesis of silver nanostructures by integrating electrochemical processes with plasma technology. Utilizing a cylindrical stainless-steel cathode alongside a controlled argon flow and regulated current allows for the production of silver nanoparticles with the desired properties. The alteration of reaction time yields insights into how the synthesis duration impacts the properties of the produced nanostructures.

2.2 Material

The reaction medium (inner diameter: 8 cm; length: 3 cm) contains a solution of deionized (DI) water and ethylene glycol (75:25), along with silver nitrate (AgNO_3) at room temperature. The synthesis of silver nanoparticles (Ag NPs) utilizes AgNO_3 sourced from Merck.

2.3 Material characteristic

The morphology of the Ag NPs was analyzed by TEM (Zeiss-EM10C-80 KV). The UV-Vis absorption was measured using a UV-Vis–NIR spectrophotometer (PG Instruments model T80+). Additionally, the XRD pattern was recorded using an X'Pert MPD (Philips) X-ray diffraction meter with Co-K α radiation (1.79 Å).

3 Result and discussion

3.1 Characterizations of Ag nanostructures

The results shown in Fig. 2 illustrate the progression of the reduction of silver nanoparticles in a solution over time. In particular, the transmission electron microscopy (TEM) image taken after 5 minutes shows that only spherical silver nanoparticles of different sizes are present, with an average diameter of about 2-3 nanometers. This observation indicates that the nanoparticles at this early stage are predominantly small and spherical.

However, after 10 minutes of processing, the TEM image reveals a more intricate scenario. It depicts the coexistence of smaller, isolated particles alongside larger silver nanoparticles, suggesting a shift toward particle aggregation. This increase in particle size signifies a propensity for particle amalgamation. The observed aggregation likely signifies a directional coalescence of nanoparticles, possibly attributed to the ongoing reduction process and subsequent nanoparticle interactions.

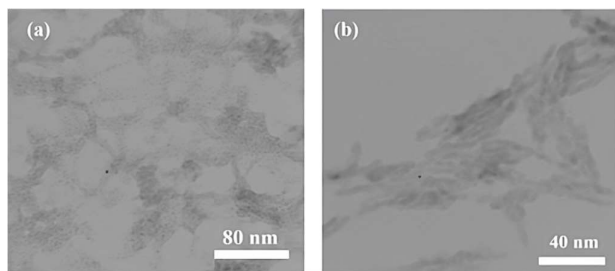


Figure 2. Transmission electron microscope (TEM) images of Ag nanoparticles synthesized by using plasma in deionized water and ethylene glycol (75:25) along with containing silver nitrate at different times (a) 5 min, (b) 10 min.

The transition from predominantly small, spherical nanoparticles to a mixture of small and larger particles illustrates the dynamic nature of the nanoparticle

synthesis process. The growth and aggregation behavior of the nanoparticles over time provides insights into the kinetics of the reduction process and the factors affecting the particle size distribution. Understanding these changes is crucial for optimizing the synthesis conditions to achieve nanoparticles with the desired size and uniformity, which is critical for their effective application in diverse technological and scientific domains.

Furthermore, plasma can impact the formation of oriented nanostructures via electrostatic forces, which are pivotal in governing the growth and orientation of nanostructures in specific directions. The electric fields generated by plasma can align nanostructures as they develop, exerting forces on charged particles like ions or nanoparticles. Through the manipulation of the electric field's intensity and direction, the growth of aligned nanostructures can be precisely controlled.

To delve deeper into the material type and ascertain the structural and chemical composition of the nanoparticles generated, an X-ray diffraction (XRD) analysis was conducted. For instance, the XRD pattern of the sample is depicted in Fig. 3. As illustrated in the figure, notable peaks are evident at diffraction angles of 38.17°, 44.31°, 64.5°, and 77.05°. These distinct peaks are indicative of particular crystal planes present within the nanoparticle samples.

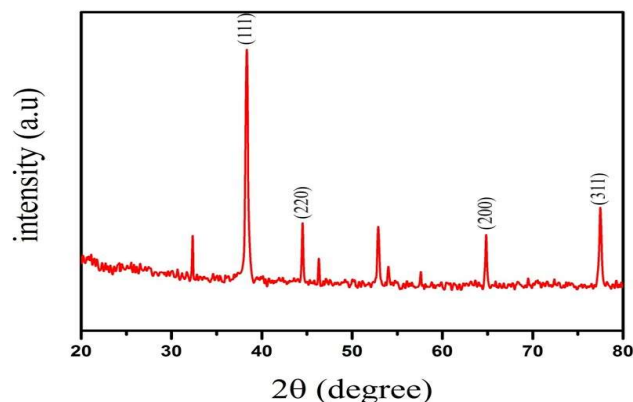


Figure 3. X-ray diffraction pattern of Ag nanoparticles synthesized by using plasma in deionized water and ethylene glycol (75:25) along with containing silver nitrate.

Utilizing Bragg's law and the angles obtained from the diffraction pattern, we can calculate the d-distance spacing between the crystal planes. This calculation

offers insights into the crystallographic structure of the nanoparticles, facilitating a comprehensive understanding of their phase composition and structural characteristics. Through the analysis of these XRD patterns, they can evaluate the crystallinity quality, phase purity, and potential lattice deformations in the synthesized nanoparticles, which is essential for refining their properties and potential applications.

Based on equations 1 and 2 and taking into account the atomic radius of silver (1.44 \AA), we determined the indices and lattice parameters for the face-centered cubic (fcc) structure. Specifically, in the fcc crystal structure, the main diffraction planes are characterized by the Miller indices, where all three indices hhh, kkk, and lll are either all even or all odd. This characteristic aligns with the diffraction planes observed in the X-ray diffraction pattern

$$a_{fcc} = 2r\sqrt{2}, \quad (1)$$

$$d_{(hkl)} = \frac{a}{\sqrt{h^2+k^2+l^2}}. \quad (2)$$

As a result, the observed peaks correspond to the crystal planes (111), (220), (200), and (311), in alignment with the characteristic planes of the fcc crystal structure of silver. The Miller indices for these planes reflect the arrangement and spacing of the atomic planes within the fcc lattice, confirming the presence of the fcc crystal structure in the synthesized silver nanoparticles.

The analysis of these diffraction peaks provides crucial information about the crystallographic orientation and phase purity of the nanoparticles. By comparing the observed diffraction peaks with the expected positions for an fcc lattice, one can validate the crystallinity and structural integrity of the nanoparticles. This detailed structural characterization is essential for understanding the material properties and optimizing the synthesis conditions for desired applications.

Figure 4 indicates the absorbance spectrum of silver colloids prepared from a solution containing 0.2 mM silver nitrate in deionized water at time intervals of 10, 20 and 30 minutes with a discharge current of 5 mA, showing microplasma at room temperature (25°C). With time, the spectra show higher absorbance (larger

peak area), indicating higher production efficiency. The positions of the peaks for all three modes are close to each other and at a certain distance (390 nm to 410 nm), indicating the average size of the nanoparticles. However, the FWHM with time in the 10-min time interval is wider compared to the FWHM in the 30-min time interval.

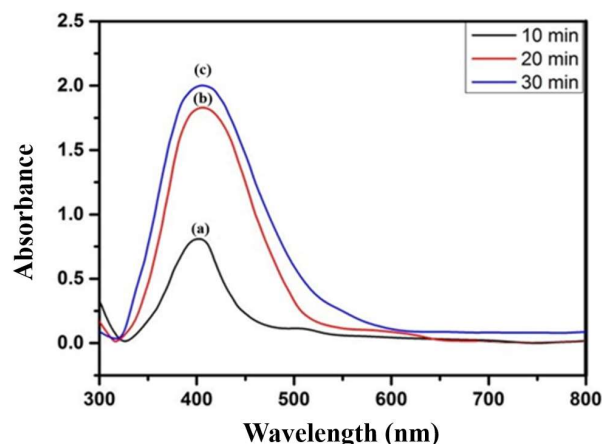


Figure 4. The absorption spectrum of silver colloids produced from a solution containing 0.2 mM, silver nitrate, in deionized water and ethylene glycol (75:25) in time intervals (a) 10, (b) 20 and (c) 30 minutes with microplasma discharge current 5 mA.

In UV-Visible spectroscopy, metal nanoparticles such as silver show a phenomenon called surface plasmon resonance (SPR). SPR occurs when free electrons in the conduction band of the nanoparticle collectively oscillate in resonance with the electromagnetic field of the incident light. This resonance results in an absorption peak in the visible or near-visible region. For Ag NPs, smaller nanoparticles typically produce an SPR absorption peak at shorter wavelengths, while larger nanoparticles shift this peak to longer wavelengths due to a change in the collective oscillation behavior of the electrons. In this study, peak positions between 390 nm and 410 nm are observed. This narrow range indicates relatively uniform nanoparticle size, as large variations in size result in broader or multiple peaks. The average peak position provides insight into the particle size: the closer the peak positions are, the lower the variation in nanoparticle size.

The absorption peaks in the UV range observed in silver nanoparticles are due to surface plasmon resonance (SPR). As a noble metal, silver has a high density of free electrons in its conduction band. When

light of a certain wavelength interacts with the nanoparticles, it can excite these electrons and create a resonance that leads to strong absorption at certain wavelengths. For this reason, silver nanoparticles often have a characteristic peak in the visible range so that they can be detected with UV-Vis spectroscopy. For silver nanoparticles, this SPR peak can be influenced by several factors, including the size of the nanoparticles, with larger particles shifting the SPR peak towards longer wavelengths; the shape, as spherical nanoparticles generally have a single SPR peak, whereas anisotropic shapes (e.g. rods) can produce multiple peaks; and the surrounding medium, with changes in the refractive index of the medium around the nanoparticles influencing the position of the SPR peak. This SPR-based peak is a diagnostic feature used to characterize the size and distribution of nanoparticles in a sample. It is a well-known optical property of silver nanoparticles that is often used for their detection and analysis in various applications.

3.2 Mechanism

In this method, chemical reactions and fragmentation processes within a solution are triggered by electron irradiation. Hydrated electrons, which are highly effective reducing agents, facilitate the reduction of silver ions (Ag^+) to metallic silver atoms (Ag). These silver atoms collide with other silver ions under the influence of attractive forces and van der Waals interactions, forming silver dimers (Ag_2^+). Subsequently, these dimers gradually evolve into silver nanoparticles in the solution.

The reduction of silver ions (Ag^+) can occur either through direct interaction with the highly reducing hydrated electrons or via reactive radicals in the environment, highlighting the complexity of the reduction process. Particularly under electron irradiation, silver particles have the potential to increase in size and aggregate, ultimately resulting in the creation of silver nanoparticles.

The process is characterized by the formation of silver nanoparticles through the combined effects of electron-induced reduction and nanoparticle growth dynamics. The electron irradiation method provides a controlled environment for the synthesis of silver nanoparticles and allows precise control of particle size and

distribution. This technique utilizes the strong reducing power of hydrated electrons to initiate and drive the process of nanoparticle formation, providing a versatile approach to producing nanomaterials with specific properties and applications.

4 Conclusions

The aim of this article is to present a novel and advanced method for the rapid and efficient synthesis of silver nanostructures with different dimensions. In this study, an electrochemical-plasmonic approach is used to synthesize silver nanostructures with various sizes and shapes. Laboratory analysis revealed that the synthesized nanostructures range between 2 and 3 nanometers in size and exhibit rod-like shapes, among other morphologies.

The utilization of atmospheric pressure plasma in synthesizing nanostructures highlights its superior capabilities compared to conventional techniques, which typically necessitate high thermal energy. Atmospheric pressure plasma provides a notable advantage by facilitating the fabrication of nanostructures in reactive or temperature-sensitive materials. This characteristic empowers researchers to create nanostructures with precision under gentler conditions, thereby expanding the array of materials and applications.

In addition, the electrochemical plasma method offers unique possibilities, such as a high degree of flexibility in the fabrication of nanostructures and the ability to tailor specific features, including morphology and crystalline properties. This flexibility is particularly advantageous for applications that demand precise control over the physical and chemical properties of the nanostructures.

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