Microplasma assisted synthesis of silver nanoparticles capped with PVA, PVP and Sucrose

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Abstract
Silver nanoparticles (Ag NPs) have been synthesized by exposing Ar gas microplasma to liquid solution containing Ag⁺ ions. Sucrose, Polyvinyl Alcohol (PVA) and Polyvinylpyrrolidone (PVP) were added separately as capping agents in aqueous solution to avoid agglomeration of Ag NPs. There are two main mechanism involved in this experiment (1). interaction of electrons from microplasma with Ag⁺ ions present in aqueous solution and (2). Control of Ag NPs size by capping agents by surrounding Ag NPs. We have performed a comparison of capping performance of Sucrose and PVA in terms of stability of Ag NPs with the help of Ultraviolet-Visible spectroscopy (UV-vis). X-ray diffraction (XRD) analysis of Ag NPs capped with PVA and Sucrose shows average crystal size of 25 nm and 60 nm respectively. Dynamic Light Scattering (DLS) analysis was employed to measure the size distribution of Ag NPs capped with Sucrose, PVA and PVP capping agents. DLS showed that PVA-capped Ag NPs formed are more uniform in size as compared to Sucrose-capped. Average size of Ag NPs measured from DLS is 65 nm when capped with PVA and 120 nm when capped with Sucrose. Fourier Transform Infrared Spectroscopy (FTIR) spectrum obtained have bands of wavenumbers corresponding to functional groups of Sucrose and PVA which are present around Ag NPs.

1. Introduction

Nanotechnology has become an highly emerging research field in 21st century among all the disciplines. Development of nanotechnology is being witnessed in all fields like materials, medicines and electronics [1, 2]. It has therefore, covered many topics including microelectronics, polymer-based biomaterials, nanoparticle drug delivery, miniemulsion particles, fuel cell electrode polymer bound catalysts, layer-by-layer self-assembled polymer films, electrospon nanofibers, imprint lithography, polymer blends and nanocomposites [3].

Designing of multifunctional NPs have made them potentially capable of detecting and targeting cancer cells, delivering and releasing drugs in a control manner with great sensitivity and precision[4]. Bulk silver in itself do exhibit many applications in daily life but silver nanoparticles are unique in its own nature as compared to bulky silver due to change in optical, chemical and physical properties. A dynamical enhancement in these properties is due to an increase in surface-to-volume atomic ratio of Ag NPs [5]. Ag NPs have been used in numerous fields including medical purposes (antibacterial activity, sterilizing medical tools, diagnosis of various diseases) [6]. However, Ag NPs reveals good electrical conductivity, catalytic activity, chemical stability and antimicrobial activity due to quantum size effect and surface effect as compared to bulk form of silver [7].

There are number of synthesis methods for the preparation of Ag NPs including chemical reduction method [8–10], laser ablation [11], thermal decomposition [12], ultrasound assisted synthesis [13] and green synthesis method [14, 15] etc. In chemical reduction method there are chances of some toxic chemical is being absorbed on surfaces of NPs which can have severe effect in medical applications. Above mentioned synthesis techniques also take time in hours to produce Ag NPs as described by Guzman [16] in chemical reducing method and by Jain [14] in green synthesis. These techniques also have some drawbacks e.g., by-products which are formed along with Ag NPs e.g. as mentioned by Raveendran et al [17] that when β-D-glucose is added in pure water as
reducing agent to form Ag NPs and starch tagged Ag NPs are formed. Microplasma assisted synthesis technique was chosen because it is an eco-friendly technique as it doesn’t contain any toxic chemical reducing agent [18].

Microplasma has been newly reported for the synthesis of Ag NPs [19]. Precursor can be in all three forms i.e., solid, liquid and gas. If precursor is used in solid form usually solid rod then plasma will produce NPs by sputtering of that precursor rod [20–22]. If precursor is in form of liquid, then there will be an interaction between electrons of plasma and Ag \( +\) ions present in aqueous solution [23–25]. Ordinary plasmas have equilibrium in terms of temperature and number density of their species but microplasma exhibits high number density and temperature of electrons as compared to ions and other species of plasma [26, 27] which makes them suitable for reduction of Ag \( +\) ions. Microplasma has been used in place of chemical reducing agent and hence changed the synthesis method type from chemical to physical reduction method.

The large number of surface atoms of nanomaterials due to large surface to volume atomic ratio have large surface area along with large surface energy. Nanostructures and Nanomaterials have large number of atoms on surface as compared to respective bulk materials because surface to volume ratio of atoms got increases as size of material decreases. The nanomaterials not only have large surface area but also have large surface energy. Due to this high surface energy, atoms present on surface of nanomaterial have dangling bonds which are highly unstable and can get bonded to same/other kind of atoms present nearby. If these atoms formed bonds with same kind of atoms then nanomaterials get agglomerated [28]. To avoid this agglomeration, capping agents are added in solution of nanoparticles which lowers the surface energy of dangling atoms and make them stable.

In this article we have investigated the capping action of different kind of capping agents with Ar gas microplasma assisted synthesized Ag NPs. To study the effect of capping agents from different classes on the Ag NPs size, Sucrose, PVA and PVP have been chosen. We have chosen Sucrose, PVA and PVP as capping agents because they belong to three different class and we wanted to study their effects on size of Ag NPs. We have tried to compare capping capability of Sucrose and Polyvinyl Alcohol (PVA) in terms of Ag NPs size with the help of UV-vis spectroscopy which is up to best knowledge has never been done before especially with the physical technique.

2. Experimental methods

2.1. Experiment arrangement

The experimental setup of experiment is schematically described in figure 1(a). Ar gas cylinder is fed to the stainless capillary tube via flow-meter. The gas electric discharge is produced with the help of DC power supply (ORTEC Model 459 USA) to provide 3–5 kV voltage. The counter electrode is dipped in beaker containing \( \text{AgNO}_3 \) aqueous solution. The inner diameter of stainless capillary tube is of the size of \( \leq 1 \) mm. The distance between tip of capillary tube and surface of aqueous solution is maintained between 3–5 cm. The flow-rate of Ar gas is fixed at 600 sccm.

3. Description

3.1. Materials

Silver Nitrate (.AgNO\(_3\); Duxan 169.87 gmol\(^{-1}\)) was used as a precursor for producing nanoparticles. Sucrose (\(\text{C}_{12}\text{H}_{22}\text{O}_{11}; 342.30\) gmol\(^{-1}\) Duxan), Polyvinyl alcohol (PVA, Duxan) and Polyvinylpyrrolidone (PVP, Duxan) were used as stabilizing agents. All reagents used were of analytical grade. Deionized water was used throughout the experimentation.

3.2. Preparation of Ag NPs

A number of 10 ml aqueous samples of 3 mM AgNO\(_3\) were prepared in de-ionized water. These aqueous solutions were then grouped into three on the basis of type of capping agent added. Sucrose (2.7 mM), Polyvinyl alcohol (2% W/V) and Polyvinylpyrrolidone (2% W/V) were added as capping agent. Then the precursor containing Ag \( +\) ions in aqueous form along with capping agent was placed under the microplasma (figure 1(b)) for the reduction of Ag \( +\) ions to neutral Ag atoms for the optimum exposure time of 10 min. In order to control agglomeration of these neutral Ag atoms capping agent has been added. Color of the sample changed upon the formation of Ag NPs figure 2. Color of the solution depends on many factors including concentration of ingredients, plasma exposure time etc.

3.3. Characterizations

X-ray Diffraction Analysis is used to find detail crystalline structures of particles. Ultraviolet-Visible spectroscopy (UV–1201 SHIMADZU, Japan) is used to confirm the presence of Ag NPs in colloidal form. UV–vis light is passed through sample and the corresponding absorbance spectrum is obtained. For Ag NPs maximum
absorbance peak occurs at 400–430 nm [29]. Transmission Electron Microscopy (TEM) was not available unfortunately so Mie theory was employed as an alternative approach to measure the Ag NPs size and its assumptions were validated with the help of Scanning Electron Microscopy (SEM) and Energy Dispersive x-ray (EDX) analysis. In order to measure Ag NPs size indirectly Mie theory was used and in order to validate results from Mie theory, Scanning Electron Microscopy (SEM) and Energy Dispersive x-ray (EDX) analysis are presented. Dynamic Light Scattering is used to find the particle size distribution dispersed in the solvent. It involves the measurement of scattered light from the Ag NPs, and mostly relies on Rayleigh scattering from the suspended nanoparticles. FTIR spectrum is used to identify the functional groups used in capping of Ag NPs [30]. FTIR analysis is done in the range of 500–4500 cm$^{-1}$ [31].

Figure 1. (a) Schematic diagram of microplasma (b) photograph of atmospheric pressure DC microplasma.

Figure 2. Ag NPs capped with (a) 2.7 mM Sucrose (b) 2% W/V PVA (c) 2% W/V PVP.
4. Results and discussion

4.1. X-ray diffraction analysis

Two samples each of 10 ml aqueous solution of AgNO₃ were prepared capped with PVA and Sucrose, both added separately. After exposure under Ar gas microplasma for 10 min, the samples were centrifuged for 3 times, the centrifuged powder were placed in oven at 80 °C for 24 h. The dried powder of Ag NPs was characterized using XRD analysis as shown in Figure 3.

In Ag NPs, four crystal faces have been observed i.e. (111), (200), (220) and (311) at 38.2°, 44.4°, 64° and 77° respectively, as reported by [7, 32, 33]. From each spectrum, average Ag Np size has been calculated with the help of Scherrer’s formula, by calculating first FWHM $\beta$ in radians and then placing in equation (1),

$$D = \frac{K\lambda}{\beta \cos \theta_B},$$

where $\beta$ is the FWHM (radians) of a diffraction peak, $\lambda$ is the x-ray wavelength, $\theta_B$ is the diffraction angle, and $K$ is the Scherrer’s constant ($K = 1$ for usual crystal). The average size of Ag NPs obtained were 25 nm (capped with PVA) and 60 nm (capped with Sucrose). In case of Sucrose capped Ag NPs, noise in XRD spectrum has been observed due to many reasons, few of them may be that sample of Sucrose capped Ag NP required more high speed centrifuging or due to $\text{NO}_3^-$ ions, ionic Ag, OH groups present in that sample [32, 33].

4.2. Ultraviolet-visible spectroscopy

In UV-vis spectrometer, glass cuvette is used to hold reference solvent and sample. The freshly prepared samples were transported to the spectrometer. The UV-vis absorption spectrum was obtained by plotting absorbing

![Figure 3. Ag NPs XRD spectrum capped with (a) 2% W/V PVA and (b) 2.7 mM Sucrose.](image)
intensity against the wavelength. UV-vis absorbance spectrum of Ag NPs with different capping agents is shown in figure 4.

Ag NPs capped with PVA shows maximum absorbance of 1.7 a.u at 412 nm and average particle size calculated theoretically with the help of Mie theory is 15 nm while in case of PVP capping agent Ag NPs showing maximum absorbance of 1.44 a.u at 416 nm and NP size calculated from Mie theory is 17 nm. Absorbance intensity reflects the concentration of particles [34] so number of Ag NPs are more capped and hence more uniform when they are capped with PVA.

In case of Sucrose capped Ag NPs, mole optimized Sucrose selected are 2.7 mM after taking following moles ratio of AgNO₃:Sucrose = 1.1, 1, 0.9, 0.7 as compared to those selected by YL Thong [19]. UV-vis spectrum of Ag NPs capped with Sucrose at different mole ratio with AgNO₃ is shown in figure 5.

UV-vis spectrum of Ag NPs capped with Sucrose have large broadness as compared to other spectra. This broadness shows size distribution of Ag NPs indirectly. It has been observed from Sucrose capped spectrum that Ag NPs are not of uniform size yet maximum absorbance occurs at 417 nm. FWHM lies at 538 nm and the particle size calculated from Mie theory is 93 nm [35]. It may also be possible that in physical reduction method Sucrose didn’t work as efficient as it does in chemical reducing method.

PVP and PVA were added separately in aqueous solution of AgNO₃ and tested with the quantities weighing 1, 2 and 3 g W/V [36]. Experimental results depicted in figures 6–7 demonstrate that 2 grams of each would give optimize results.

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**Figure 4.** UV-vis spectrum of Ag NPs capped with (a) 2% W/V PVP (b) 2% W/V PVA (c) 2.7 mM Sucrose.

**Figure 5.** UV-vis spectrum of Ag NPs capped with AgNO₃:Sucrose ratio of (a) 7:10 (b) 9:10 (c) 10:10 (d) 11:10.
4.2.1. Ag NPs size measurement using Mie Theory

As the main objective of this study is the comparison of the capping capability of capping agents with the help of UV-vis spectroscopy. As the main objective of this study describes that we are comparing the capping capability of capping agents by doing UV-vis analysis. Mie theory has been used to evaluate the Ag NP size, highlighting that the validity of the approach has been already proved in previous work [35] through a comparison with TEM measurements. For that purpose, measurement method of Ag NPs size was chosen to be Mie theory because Transmission Electron Microscopy (TEM) was not available unfortunately but indirect measurement through Mie theory has been done before and compared with TEM results in literature as done by [35]. In Mie Theory, from UV-vis spectrum particle size can be estimated. Mie theory applies electrodynamics classically to cluster of simple spherical shapes which based on the solid state. It have assumed that resultant Ag NPs are spherical and widely spatially separated. The surrounding medium of Ag NPs assumed to be distilled water. The detail derivation of equation (2) can be found in [35]. The dielectric parameter of surrounding medium is equivalent to square of refractive index [37]. The refractive indexes of distilled water, Sucrose solution in water, PVA solution in water and PVP solution in water at wavelength range of 400–500 nm are 1.33, 1.5, 1.68 and 1.47 respectively [38, 39]. The values of refractive indexes of capping agents and distilled water are close to each other so calculations of Ag NPs size are done only with value of dielectric parameter of distilled water.
\[ \gamma(R) = \gamma_0 + \frac{Av_f}{R} \]  

(2)

where \( \gamma_0 \) is the velocity of bulk scattering (for Ag, \( \gamma_0 = 5 \times 10^5 \text{ s}^{-1} \)), \( A \) represents scattering process (\( \frac{3}{4} \) for Ag) and \( v_f \) is the fermi velocity (for Ag, \( v_f = 5 \times 10^{12} \text{ ms}^{-1} \)). From UV-vis spectrum, FWHM is calculated in terms of \( \Delta \lambda \) in units of nm then it is converted into \( \text{s}^{-1} \) units using relation,

\[ \gamma(R) = \frac{2\pi v_f}{\Delta \lambda} \]  

(3)

then putting value of \( \gamma \) from equations (3) into (2), we get the value of \( R \) which is the size of Ag NPs.

In order to validate Mie theory it was necessary that Ag NPs formed are ideally be spherical and no impurity is present around Ag NPs. For this purpose, Scanning Electron Microscopy (SEM) and Energy Dispersive x-ray (EDX) analysis was done. Ag NPs capped with PVA formed are seemed spherical by SEM result shown in figure 8 (left) and those Ag NPs capped with Sucrose are somewhat pyramid shape as shown by SEM results figure 8 (right). Elemental Composition is determined by EDX analysis. In figure 9 (top), elemental composition of Ag NPs capped with PVA sample is shown. It has been found that concentration of carbon and oxygen are more than Ag NPs. Presence of carbon is due to PVA capping agent and oxygen is due to oxidation of Ag NPs sample. There is also possibility that sample of Ag NPs capped with PVA is not well centrifuged so that it shows large concentration of carbon. Under the light of above discussion i.e. unavailability of TEM facility. Mie theory has been applied to calculate Ag NPs size from UV-vis spectra which are supported by SEM and EDX results to some extent.

### 4.3. Stability analysis of Ag NPs

The stability of Ag NPs was assessed for the period of about 15 days with different capping agents. Three samples were prepared such that each sample contained 3 mM AgNO\textsubscript{3} concentrated aqueous solution. These three samples were grouped on the basis of capping agent type as well as the amount of capping agent.

Ag NPs stability analysis was done with the help of UV-vis spectroscopy. The UV-v is spectra of Ag NPs are sketched in figures 10–12. From figure 10 it can be noted that when freshly prepared Ag NPs capped with Sucrose were subjected to UV-vis spectroscopy, the maximum absorbance on 1st day was 0.5 a.u at \( \lambda = 407 \text{ nm} \) and later it reduced to 0.15 a.u at \( \lambda = 428 \text{ nm} \) after 15 days. Broadness of peak also increases with each passing day. The capping effect is basically the result of Brownian and electrostatic forces with which the capping agent is capped around the NPs. It might be possible that on first day of formation of Ag NPs, the electrostatic forces will be in equilibrium with forces due to Brownian motion so a relatively uniform peak was formed. However, with passage of time, this equilibrium breaks gradually and capping of Ag NPs therefore loses and hence particles starts agglomerating. In case of Sucrose, the capping agent might not be as strongly bonded in capping Ag NPs as that of PVA.

Now if we study the effect of different concentration of same capping agent as shown in figures 11, 12, it can be observed that when PVA 1 g/L is added, concentration of Ag NPs decreases gradually with time but yet in contrast with PVA 2 g/L. Figure 12 remains almost the same for the period of 15 days. It therefore proves that Ag NPs size remain almost constant for 15 days. It might be possible due to the fact that when 2g PVA is added, the...
number of organic PVA is becoming sufficient enough to cap all Ag NPs from all around so that there will be no way left for Ag NPs to agglomerate further.

Sizes of Ag NPs capped with (a) Sucrose (b) PVA 1 g/L c) PVA 2 g/L against time is shown in figure 13. The plot itself describes clearly that when Ag NPs are capped with 2 g/L PVA, their size will remain constant or changes minutely as compared to Sucrose and PVA 1 g/L. Ag NPs size increases from 17 nm to 50 nm greatly
Figure 11. UV-vis spectrum of Ag NPs capped with PVA 1 g/L recorded on day (a) 1, (b) 2, (c) 3, (d) 4, (e) 5, (f) 7, (g) 15.

Figure 12. UV-vis spectrum of Ag NPs capped with PVA 2 g/L recorded on day (a) 1, (b) 2, (c) 3, (d) 4, (e) 5, (f) 7, (g) 15.

Figure 13. Ag NPs size versus time.
with time when capped with Sucrose as demonstrated in figure 13. When Ag NPs are capped with PVA 2 g, NPs size remains almost constant as depicted from figure 13.

4.4. Dynamic Light Scattering

Three samples of Ag NPs, capped separately with Sucrose, PVA, PVP were also characterized using dynamic light scattering analysis. The results are shown in figures 14–16.

In figure 14, for Ag NPs capped with Sucrose, average size obtained is 120 nm which is larger than the sizes gauged through XRD (60 nm) and UV-vis (93 nm) spectrum. The main reason behind the increase in size of Ag NPs is that Dynamic Light Scattering (DLS) technique measures hydrodynamic size rather than the physical size [40, 41]. As mentioned by Domingos [42], DLS is more sensitive for comparatively larger sized particles. Hence it might be possible that size of Ag NPs get agglomerated at the time of DLS sampling. So this is evident reason why Ag NPs size measured by DLS are larger as calculated from XRD or UV-vis spectrum. As samples have not been centrifuged so they contained impurities along with Ag NPs whose sizes may or may not be larger than Ag NPs size.

DLS spectra of Ag NPs capped with PVA are shown in figure 15. The average Ag NPs sizes obtained are 65 nm, respectively. Ag NPs size capped with PVA have small counts. Reason behind this small comparative
counts might be due to lack of sonication. These results demands separate and detailed study which will be done in future. Our results for DLS of Ag NPs capped with PVA are in agreement with Zielińska [7] results in terms of counts/intensity. But there is difference in Ag NPs size due to difference of synthesis method. Figure 16 exhibits DLS results of Ag NPs capped with PVP. Average Ag NPs size obtained is 76 nm. This DLS result for Ag NPs capped with PVP is quite different when compared with UV-vis result. It is highly probable that some impurities may got added in sample before characterizing through DLS.

4.5. FTIR Analysis

FTIR spectroscopy was carried out for the assessment and identification of functional groups involved in capping of Ag NPs. For this aim, colloidal solutions of Ag NPs in deionized water with capping agents (Sucrose and PVA) dissolve in them separately were analyzed.

Three prominent and one minute peak was observed in both types of IR spectra figure 17. 1st peak observed around 607 cm$^{-1}$ with 45% transmittance, 2nd peak observed around 1640 cm$^{-1}$ with 70% transmittance, 3rd peak which is due to very little absorbance of IR photons is observed around 2122 cm$^{-1}$ in case of Sucrose capped and around 2210 cm$^{-1}$ in case of PVA capped Ag NPs at absorbance of only 4% and last peak observed around 3300–3320 cm$^{-1}$ with 50% transmittance.

The absorbance observed at region 607 cm$^{-1}$ may be due to stretching of Ag-Ag bonds as mentioned by Iqbal et al [23]. The peaks at 1640 and 3320 cm$^{-1}$ are due to $-\text{OH}$ functional group present in the sample either from PVA (C2H4O)n or moisture from atmosphere [43, 44]. The peaks at 2110 and 2122 fall in the region 2100–2260 cm$^{-1}$ which belongs to the stretching vibrations either due to $-\text{C=N}$ or $-\text{C=C}$ bond as given in IR spectra reference table [45, 46].

5. Conclusion

In this paper we have investigated the microplasma assisted synthesis of Ag NPs. In particular, we showed that by changing concentration/type of capping agent with fixed microplasma parameters (gas flow rate, input voltage, gas type etc), one can tune Ag NPs size and hence affects contributing to the stability of Ag NPs. The average size of Ag NPs synthesized at mole ratio of 0.9 with Sucrose and at 2 g W/V PVA and PVP each were 120, 65 and 76 nm, respectively as measured by Dynamic Light Scattering technique. The results shows that PVA capped Ag NPs were most stable for the period of 15 days as compared to Sucrose and PVP capping agent, as with PVA capping agent, the Ag NPs size only changes within the limit of <5 nm. These stability results are of practical importance for cancer therapy specifically [47].
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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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Figure 17. FTIR spectrum of Ag NPs colloidal solution.
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