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# Synthesis and Characterization of Colloidal Silver Nanoparticles of about 16nm Diameter by a Chemical Reduction Method

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## ABSTRACT

The vast use of silver nanoparticles (AgNPs) in rapidly growing number of biomedical applications and sensing has given the importance of different synthetic methods for the preparation of AgNPs. Threre are lots of effective synthetic procedure that are lacking to synthesize nanoparticles with maintaining proper size and shape in colloidal medium. Here in this article synthesis of colloidal AgNPs and its characterization is described. The sizes of the synthesized AgNPs were of about 16±2nm. Some important modern techniques like UV-Vis spectroscopy, powdered X-ray diffraction (XRD), Scanning electron microscopy (SEM), High resolution transmission electron microscopy (HRTEM) were used for proper characterization synthesized colloidal AgNPs. Silver nitrate ($AgNO_3$) and sodium borohydride ($NaBH_4$) were used to synthesize colloidal AgNPs where the later one plays vital dual role by reducing $AgNO_3$ and giving stabilization of synthesized nanoparticles.

**Keywords:** Colloidal, AgNPs, XRD, SEM, HRTEM.

## INTRODUCTION

Metal nanoparticles are the good number studied nanopartcles among which AgNPs paid an excellent interest due its vast applications. Last 30 years has noticed the tremendous use of AgNPs in different fields like electronics, medicines, healthcare, cosmetics, food packaging, agriculture, environmental cleaningup programme, energy production and tank-storages.[1–8] AgNPs have potential antimicrobialactivity and for this reason this nanoparticles are able to prevent infections.[9] Water filter coated with AgNPs have been marketed by different manufacturers.[10] AgNPs can be synthesized by using variety of methods in solution such as electrochemical, photochemical, chemical reduction, microwave processing, ultrasound processing.[11–15] Several strategies are derived to produce metal nanoparticles like AgNPs of meticulous geometry depending on precise necessities for variety of utilization are based on its geometry.[16,17] In the past nearly 22 years, a fresh route emerged allied with the research of non-spherical nanoobjects both planar like circular plates, triangles, penta- and hexagons, etc. and 3D like cubes, prisms, tetrahedra, rods, wires, etc.

Now scientists paying good attention on making nanoparticles via colloidal system as in solution phase synthesis methods are very much adaptable and suitable method. Here the prime aim to synthesize colloidal silver nanoparticles which are spherical in nature because spherical particles that have the least amount surface for a given volume are thermodynamically more stable.[18] The chemical reduction technique is preferable for this particular nanoparticles synthesis because it uses ordinary and very economical reducing agents like $NaBH_4$, salt of ascorbic acid, salt of citric acid',' and elemental hydrogen.[19–21]

## EXPERIMENTAL

### Chemicals and reagents

$AgNO_3$ was purchased from Indian Drug and Pharmaceuticals LTD., Hyderabad, India. Sodium borohydride ($NaBH_4$) and Milli-Q water were purchased from Merk, Mumbai, India. All the chemicals used for the synthesis of colloidal nano particles are of highest purity available. All the reactions were performed by using Milli-Q water.

### Synthesis of Silver nanoparticles

Yellow coloured colloidal AgNPs were synthesized by reducing $AgNO_3$ with a mild reducing agent sodium borohydride. $Ag^+$ ions were reduced to $Ag^\circ$ by $BH_4^-$ ions which is used as a reducer as well as a stabilizer.[22,23] One millimolar of aqueous silver nitrate solution (1 mM) of about 1 mL was taken in a 2 mL micro-centrifuge tube and cooled it by keeping it in ice for 10 minutes. In a small beaker, aqueous $NaBH_4$ solution (2 mM) of about 3 mL was also cooled in ice. After 10 min, drop wise $AgNO_3$ solution was added to the $NaBH_4$ solution with constant stirring. The ratio of the reactants $AgNO_3$ and $NaBH_4$ in the recation was (1:2).[24,25]

$$AgNO_3 + \text{milli Q-water} \longrightarrow AgNO_3\ (aq)$$

$$NaBH_4 + \text{milli Q-water} \longrightarrow 2NaBH_4\ (aq)$$

$$2AgNO_3(aq) + 2NaBH_4(aq) \longrightarrow 2Ag^0\ (aq) + B2H_6 + H_2 \uparrow + 2NaNO_3\ (aq)$$

### UV-Vis Spectroscopy

The formation AgNPs were performed by utilizing UV-Visible spectroscopy. This study was performed by SHIMADZU Spectrophotometer model no. TCC-240A at 25°C. A rectangular cell of path lenghth 1 cm was used for this purpose. AgNPs were prepared by reduction of $AgNO_3$ with sodium borohydride.

### X-ray diffraction study

Colloidal AgNPs were lyophilized to get solid AgNPs for the purpose of powdered XRD study. The X- ray diffraction pattern of AgNPs was recorded with the help of the intrument Brucker AXS (Model D8, WI, USA) by using CuK$\alpha$ supply ($\lambda$ =1.5409 Å) at the time of diffractogram recording. The $2\theta$ range and the scan rate were set up 0° to 80° and 5°/min respectively. This experiment was done by keeping the current 40 mA at 25°C.

### Scanning electron microscopy

Surface morphological study and size of the synthesized colloidal AgNPs were done by SEM (model Zeiss EVO 18 special edition, Japan) with operating voltage 15 Kv. Dilution of the colloidal AgNPs was done by ethanol and then a droplet was casted on glass slit. Then it was reserved in vacuum desiccators for drying. Finally it was gold coated for SEM.

### HRTEM for morphological study

HRTEM is a dominant instrument to study their interface structures, morphology, particle size and bare surfaces of the synthesized metal nanoparticles like AgNPs. The geometric shape and size of the sample was measured with the help of JEOL-HRTEM-2011-Tokyo(Japan) by keeping the accelerating voltage range 80-85Kv. A drop of 50 times diluted AgNPs solution was casted on copper grid (carbon coated) having mesh size 300C (PRO Sci Tech). AgNPs containing Cu grids were incubated for about 6 h before taking the final images.

## RESULT AND DISCUSSION

### UV-Visible Spectroscopy

The synthesized AgNPs were primarily characterized by UV-Vis spectroscopic study

(Fig. 1). The generation of UV-Visible yellow colour obviously indicates the creation of AgNPs and it confirmed by the surface Plasmon resonance. The UV-Visible spectroscopy clearly shows the Plasmon absorption band of AgNPs with peak maxima at ~404nm. This value indicates that the size of the synthesized AgNPs is of about 16±2nm which is already mentioned in various literature.[26]

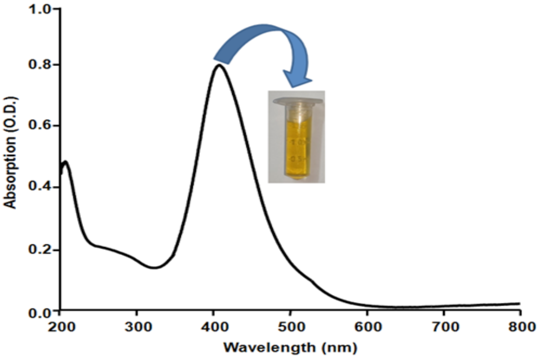

Fig. 1. UV-Vis absorption spectra of AgNPs

### X-ray diffraction study

The pattern of XRD recorded for the sample AgNPs (shown in Fig. 2) is displayed that the four peaks were stand for the (111), (200), (220) and (311) planes of fcc silver respectively. The Bragg's peaks are broad in nature which indicates the development of nanoparticles. The four distinct diffraction peaks were represented in Table 1. This value in accord with the reported literature (JCPDS file no. 84-0713 and 04-0783).[27,28]

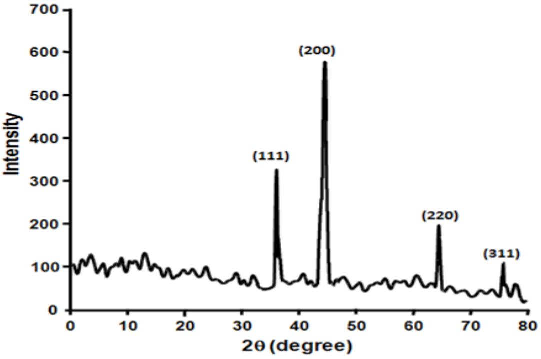

Fig. 2. XRD pattern of AgNPs

**Table 1: The different values of 2θ vs planes hkl**

| 2θ (degree) | 38.15 | 46.10 | 67.35 | 77.70 |
|---|---|---|---|---|
| hkl | 111 | 200 | 220 | 311 |

### Scanning electron microscopy

Figure 3(a) represents SEM image of the AgNPs which reveals the surface morphology of the synthesized nanoparticles. Most of the AgNPs are spherical in nature represented by Fig. 3(b-c). which are the zoomed in picture of Figure 3(a).

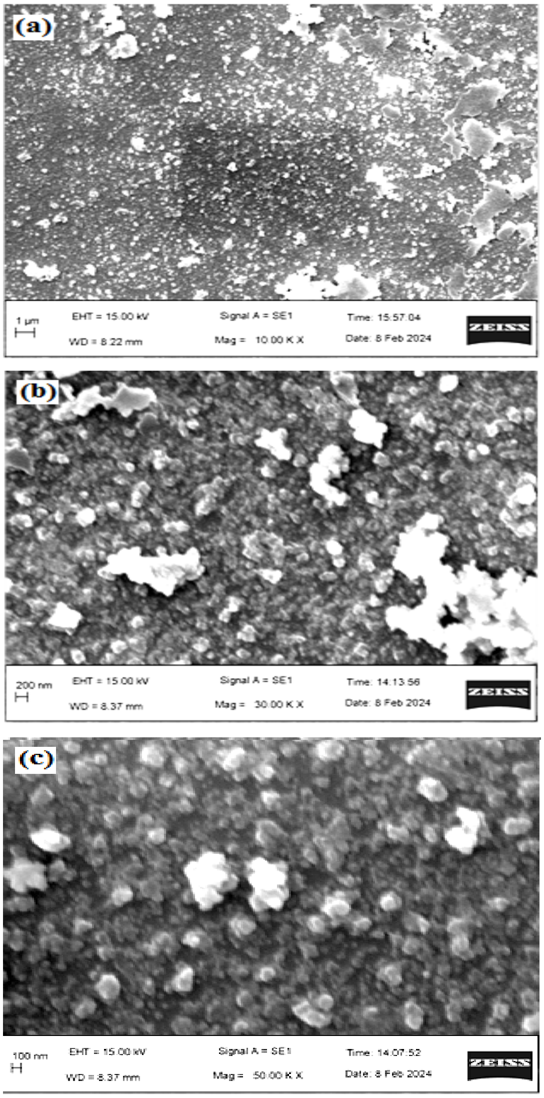

Fig. 3. SEM images of AgNPs nanoparticles with different magnification (a-c)

### HRTEM for morphological study

HRTEM study proofs Fig. 4(d-f) that the morphological structure of synthesized AgNPs is spherical in nature and average diameter is of about 16±2nm. The SAED picture with many bright points is shown by Fig. g which is the evidence of the formation of crystalline AgNPs.

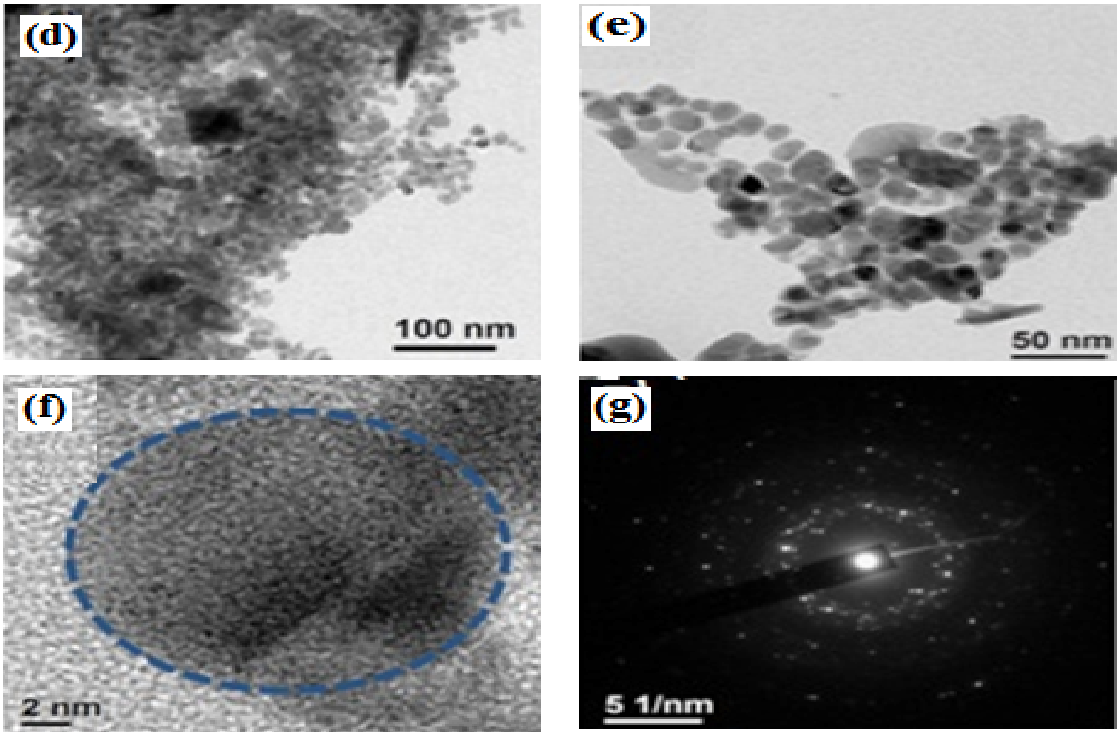

**Fig. 4. TEM image of synthesized colloidal AgNPs (d). Zoomed in images are (e and f) of the same. Bright SAED pattern is represented by Figure 4(g).**

## CONCLUSION

This article represents successful synthesis of colloidal AgNPs by chemical reduction method where aqueous silver nitrate was reduced by aqueous sodium borohydride. The UV-Vis study shows absorption maxima of synthesized nanoparticles is ~404nm which is a preliminary indication of generation of AgNPs having average diameter of about 16 ±2nm. Further SEM and TEM images confirm this diameter. The nature of the metal nanoparticles fcc and crystalline was confimed by the XRD study and SAED pattern respectively. This article is significant in the fileld of synthesis of AgNPs because the unique properties of colloidal AgNPs makes them precious building blocks for developing sophisticated materials and technologies with a broad range of applications.

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### Conflict of Interest

The author declares no conflict of interest.

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