



Synthesis of water soluble glycine capped silver nanoparticles and their surface selective interaction



Nityananda Agasti^{a,*}, Vinay K. Singh^b, N.K. Kaushik^a

^a Department of Chemistry, University of Delhi, Delhi 110007, India

^b Department of Chemistry, Sri Aurobindo College, University of Delhi, Delhi 110017, India

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ABSTRACT

Synthesis of biocompatible metal nanoparticles has been an area of significant interest because of their wide range of applications. In the present study, we have successfully synthesized water soluble silver nanoparticles assisted by small amino acid glycine. The method is primarily based on reduction of AgNO₃ with NaBH₄ in aqueous solution under atmospheric air in the presence of glycine. UV–vis spectroscopy, transmission electron microscopy (TEM), X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, thermogravimetry (TG) and differential thermal analysis (DTA) techniques used for characterization of resulting silver nanoparticles demonstrated that, glycine is an effective capping agent to stabilize silver nanoparticles. Surface selective interaction of glycine on (111) face of silver nanoparticles has been investigated. The optical property and crystalline behavior of silver nanoparticles were found to be sensitive to concentration of glycine. X-ray diffraction studies ascertained the phase specific interaction of glycine on silver nanoparticles. Silver nanoparticles synthesized were of diameter 60 nm. We thus demonstrated an efficient synthetic method for synthesis of water soluble silver nanoparticles capped by amino acid under mild reaction conditions with excellent reproducibility.

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1. Introduction

Biomedical applications of nanomaterials have been one of the significant and challenging focuses. Surface functionality and biocompatibility decides the utility of metal nanoparticles for biomedical applications [1,2] especially in cancer diagnosis and therapy [3,4]. Biocompatibility of metal nanoparticles is highly dependent on their size, shape and composition [5,6]. The challenge of synthetically controlling the size and crystal morphology of metal nanoparticles has been met with biomolecule directed nanoparticle synthesis. Synthesis of nanoparticles using biological capping agents such as microorganisms [7], plant extract [8], bone extract [9] has been an attractive alternative over other chemical reagents since they are renewable, cost effective, eco-friendly and biocompatible. Efforts have been made to use biomolecules like peptides and amino acids to synthesize nanomaterials. For instance, Brown et al. [10] investigated the polypeptide- directed synthesis of gold nanocrystals and effect of polypeptides on the morphology and orientation of the resulting

gold nanocrystal. Xu and Polavarapu [11] synthesized gold nanoparticles by using amino acid as capping agent. Yang et al. [12] synthesized silver nanoplates by using glycyl–glycine as stabilizing agent. Tiopronin (N-(2-mecaptopropionyl)-glycine) is used as a protecting ligand for synthesis of Ag [13] and Au [14] nanoparticles. Glutathione has been used for preparation of Au NP [15], Ag NP [16] and Au_{core}–Ag_{shell} nanoparticles [17]. Zakaria et al. [18] demonstrated the interaction of amino acids alanine, glutamate, cysteine, lysine, arginine and histidine with citrate capped Au nanoparticles. Lysine and aspartic acids have been used as capping agent for synthesis of Au nanoparticles [19]. Efforts have also been made to synthesize silver nanoparticles with cysteine [20] and glutamic acid [21] as capping agents. However, no attempt was made to synthesize silver nanoparticle using amino acid glycine. Glycine being an amino acid having a –COOH and –NH₂ group and its solubility in water can play an important role as a capping agent for synthesis of biocompatible metal nanoparticles.

Recognizing the wide utility of metal nanoparticles synthesized in water and subsequently linked to biomolecules, we report the synthesis of water soluble silver nanoparticles using glycine as capping agent. The glycine capped silver nanoparticles were obtained in the form of a dry powder after evaporation of the aqueous component, this powder being extremely stable in air and readily redispersible in water. AgNO₃ was reduced to silver nanoparticles in presence of glycine. Varied concentration of

* Corresponding author at. Present address: Department of Chemistry, Ramjas College, University of Delhi, Delhi 110007, India. Tel.: +91 9999691964; fax: +91 11 27667447.

E-mail address: nnagasti@gmail.com (N. Agasti).

glycine affecting the crystallinity of silver nanoparticles has been investigated through X-ray diffraction studies. Furthermore, based on XRD studies, surface selective interaction of Ag (111) with glycine has been observed. To ascertain the surface coordination of silver particles with glycine FTIR, TGA and DTA techniques were employed.

2. Experiments

2.1. Material and characterization

AgNO_3 was purchased from Merck. Glycine was from SRL and NaBH_4 from spectrochem and was used as received. Millipore water ($>18.0\text{M}\Omega$), purified using the Millipore Milli-Q gradient system, was used in whole experiment. Prior to experiments, all glassware were washed thoroughly with aqua regia and rinsed with Millipore water.

Absorption spectra of the nanoparticles were recorded on a Varian Cary 100 dual beam spectrophotometer at room temperature. Baseline correction was performed prior to all measurements. The particle size was characterized by using a TECNAI G2HRTEM 200 Kv transmission electron microscope. TEM samples were prepared by spreading a drop of colloid into a carbon coated grid and drying at room temperature. FTIR spectra of samples were recorded on a PerkinElmer FTIR Spectrometer Spectrum 2000 at room temperature. Specimen for analysis was prepared as a KBr pellet of dry samples. FTIR spectra of pure glycine and glycine capped silver nanoparticles at varying concentrations of glycine were recorded (Supporting information). The X-ray diffraction pattern was recorded by using Rigaku RU 200B X-ray diffractometer with Ni filtered $\text{Cu K}\alpha$ radiation. The thermogravimetric analyzer Shimadzu-TA-60-WS was used to evaluate thermal properties of samples at a heating rate of 30°Cmin^{-1} under N_2 atmosphere.

2.2. Preparation of glycine capped silver nanoparticles

The synthesis followed a one pot procedure as shown in part (i) of Fig. 1. Solutions of glycine were prepared with different concentrations of glycine (50 mM, 100 mM, 150 mM and 200 mM) dissolved in of millipore water. 20 ml solution of each concentration was taken in separate round bottom flasks and was set on stirring. 10 ml of freshly prepared aqueous solution of 4 mM ice-cold NaBH_4 was added drop wise into the glycine solution. Then 10 ml of aqueous solution of 1 mM AgNO_3 was added into the above mixture drop wise. Four sets of reactions with silver to glycine ratio

1:50 mM, 1:100 mM, 1:150 mM, 1:200 mM were investigated. The AgNO_3 was immediately reduced to silver nanoparticles upon addition of NaBH_4 , manifested by an immediate color change of solution from colorless to dark yellow. The color of the solution varies with respect to concentration of glycine as shown in part (ii) of Fig. 1. All the solutions were homogenous and clear. The mixture was allowed to stir for 5–6 h. Colloidal solutions of silver nanoparticles were subjected to centrifugation and washed with deionized water to remove any uncoordinated glycine. The silver nanoparticles were then dried and characterized.

3. Results and discussion

Fig. 2 depicts the absorption spectra recorded for glycine capped silver nanoparticles at varying concentration of glycine with respect to metal ($\text{Ag}^+:\text{Glycine}$; 1 mM:50 mM, 1 mM:100 mM, 1 mM:150 mM, 1 mM:200 mM). The absorption spectra show intense absorption in the range of 399–406 nm, which signifies the surface plasmon resonance (SPR) band of silver nanoparticles [22] and thus confirm the formation of silver nanoparticles. Effect of concentration of glycine on formation of silver nanoparticles was also investigated in our study. The absorption spectra for the glycine (50 mM) capped silver nanoparticles and glycine (100 mM) capped silver nanoparticles showed SPR bands at 406 nm and 399 nm, respectively. As shown in Fig. 2B, the blue shift of SPR indicates a change in particle size to smaller dimensions and is consistent with HRTEM images in Fig. 5A and B. It is well evident from the literature [23] that a polaritonic blue shift occurs with decreasing cluster size and a red shift occurs with increasing cluster size, due to electromagnetic retardation. This trend of blue shift of SPR band did not continue with further increase in glycine concentration, which can be attributed to increase in dielectric constant of the medium on increasing concentration of glycine. This nonuniform optical property of Ag NPs with respect to concentration of capping ligand glycine can be supported by a previous report [21]. Fig. 2B shows, with increase in glycine concentration beyond 100 mM, the SPR band becomes broader, with a decrease in intensity and also suffers a red shift at 402 nm and 404 nm for glycine (150 mM) and (200 mM) capped silver nanoparticles respectively. Red shift of SPR band indicating increase in particle size is in well agreement with the HRTEM images and X-ray diffraction (XRD) data. Both HRTEM images and XRD data reveals increase in particle size on increasing concentration of glycine from 100 mM to 200 mM.

Effect of concentration of glycine on particle size and surface selective interaction with silver was corroborated by X-ray

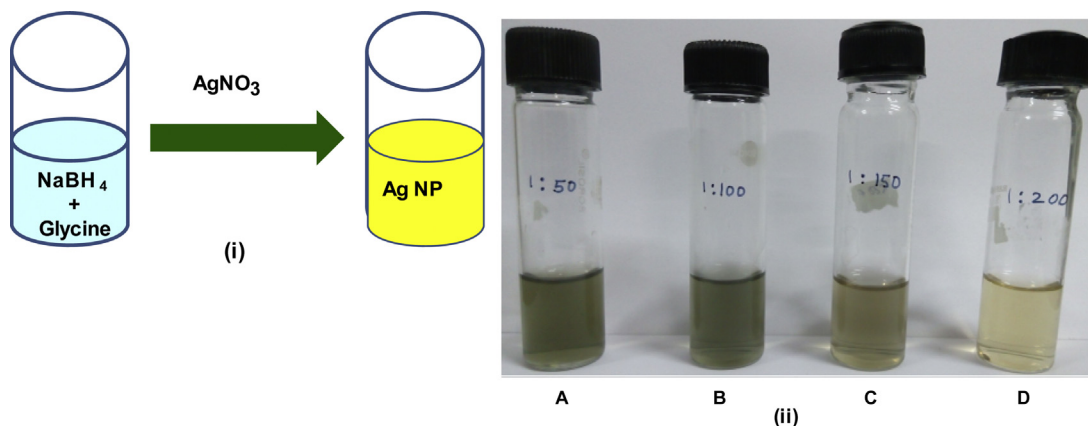


Fig. 1. (i) Schematic illustration of synthesis of glycine capped silver nanoparticles. (ii) Photographs of glycine capped Ag nanoparticles with varying silver to glycine ratios [A]: 1:50 mM, [B]: 1:100 mM, [C]: 1:150 mM and [D]: 1:200 mM.

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