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ORIGINAL ARTICLE

Preparation and characterization of silver nanoparticles using aniline

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Abstract UV–Vis spectroscopy, transmission electron microscopy (TEM) and selected area electron diffraction (SAED) have been employed to characterize silver nanoparticles (AgNPs) using aniline and silver nitrate as reductant and oxidant, respectively. A broad surface resonance plasmon (SRP) band appears at 400 nm, indicating that the AgNPs are spherical. The TEM images show that AgNPs aggregated in an unsymmetrical manner, leading to the formation of beautiful silver nanocrystals. Aniline adsorbed onto the surface of Ag-nanocrystals through electrostatic, van der Walls forces and hydrogen bonding. Cetyltrimethylammonium bromide (CTAB) acted as a stabilizer and/or capping agent.

1. Introduction

Synthesis of different morphologies of advanced silver nanomaterials (nanotubes, nanowires, nano cubes, nanorods, and nanosheets (Mulvaney et al., 2002; Yu and Yam, 2005; Xie et al., 2007; Kim et al. 2009)) has been the subject of a large number of investigations due to their potential applications in the various fields such as biomolecular detection (Duran et al., 2005), as biosensors (Xiong et al., 2008), in catalysis (Sun and Seff, 1994), photography (Albrecht et al., 2006), and as antimicrobials (Sondi and Salopek-Sondi, 2004; Brigger et al., 2004; Guzman et al., 2008). The literature is replete regarding the use of different reducing agents (ascorbic acid, hydrazine, ammonium formate, dimethylformamide and sodium borohydride (Sondi and Salopek-Sondi, 2004)). The morphology strongly depends on the experimental conditions such as, method of preparation, nature of reducing agents and stabilizers. The simplest and the most commonly used bulk- solution synthetic method for metal nanoparticles is the chemical reduction (Solomon et al., 2007; Harada et al., 2009; Won et al., 2010; Khan et al., 2011; Khan et al., 2012) of metal salts. Using chemical reduction methods for the synthesis of nanoparticles with different morphologies can be advantageous over other biosynthetic processes because it involves the reduction of an ionic salt in the presence of surfactant using a reducing agent as well as it is cost effective, easily scaled up for large-scale synthesis (Esumi et al., 1990; Taleb et al., 1997; Zhu et al., 2000; Henglein, 2001; Sharma et al.,

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Table 1 Values of k_{obs} as a function of [aniline] and [CTAB] for the formation of AgNPs.

10^4 [Aniline] (mol dm^{-3})	10^4 [CTAB] (mol dm^{-3})	10^4 k_{obs}(s^{-1})
0.0	10.0	0.0
5.0		10.2
10.0		10.1
15.0		10.2
20 .0		10.0
30.0		10.4
40.0		10.3
50.0		10.2
40.0	2.0	Yellowish turbid
	6.0	4.0
	10.0	10.1
	15.0	14.0
	20.0	Yellowish turbid

2009) and further there is no need to use high pressure, energy and temperature. Biological microorganisms bacteria (Saifuddin et al., 2009), fungi (Duran et al., 2007), yeast (Kowshik et al., 2003) or plant extracts (Haverkamp and Marshall, 2009) have emerged as an alternative to chemical synthetic procedures. The presence of various natural products (carbohydrates, alkaloids, steroids, proteins and/or peptides) in plant extracts, and the tendency of Ag ions to form a variety of complexes with carbohydrates and proteins, all combine to give systems of considerable complexity.

Henglein (1993) and his coworkers reported the formation of long-lived clusters of silver by a chemical method (Ag^+ ions – sodium borohydride reaction in the presence of a polyanion) for the first time (Linnert et al., 1990). Reduction of silver(I) by chemical methods proceeds through a one-step process to produce a colored silver sol because the surface of a metal has free electrons in the conduction band and positively charged nuclei.

Aniline (aromatic amine) is a water soluble weak reducing agent. In this paper, we report a simple one-pot chemical reduction method for the preparation of AgNPs using aniline and shape-directing CTAB as reducing- and stabilizing agents, respectively. Studies revealed that the reaction conditions ([CTAB] and [aniline]) content have great influence on the morphology of silver nanoparticles. The conditions for synthesis were optimized by changing the concentrations of aniline, CTAB and Ag^+ ions.

2. Experimental

2.1. Materials and instruments

Doubly distilled (first time from alkaline $KMnO_4$) and deionized water (specific conductance (1–2) $\times 10^{-6}$ ω^{-1} cm^{-1}) was used as solvent for preparing the stock solutions of all reagents. Aniline (Merck India, 99%), $AgNO_3$ (Merck India, 99%), and cetyltrimethylammonium bromide (Merck India, 99%), were used as received. Aniline solutions were prepared daily and stored in amber colored glass bottles. UV–visible Recording Spectrophotometer, UV-260 Shimadzu, with 1 cm quartz cuvettes, Transmission electron microscope (JEOL, JEM-1011; Japan and Bruker Equinox 55 spectrophotometer were used to record the spectra, size and size distribution and ir spectra, respectively.

2.2. Rate determination

Required amounts of $AgNO_3$, CTAB and water, were taken in a three necked reaction vessel fitted with a double-walled contender to arrest evaporation. The reaction was started by adding a thermally equilibrated solution of aniline. The growth of AgNPs formation (formation of yellowish-brown colored silver sol) was followed spectrophotometrically by pipetting out aliquots at definite time intervals and measuring the absorbance at 400 nm (λ_{max} of pale-yellow color). Duplicate runs gave results that were reproducible to within $\pm 5\%$. The apparent rate constants (k_{obs}, s^{-1}) were calculated from the initial part of the slopes of the plots of $\ln(a/(1-a))$ *versus* time with a fixed time method (Khan and Talib, 2010; Huang et al., 1993). The pH of the reaction mixture was also measured at the beginning and end of each kinetic experiment.

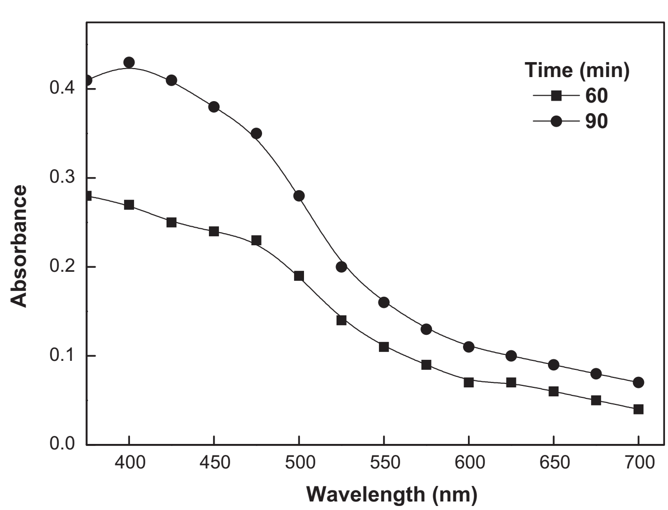

Figure 1 Spectra of AgNPs at different time intervals. *Reaction conditions*: [aniline] = 40.0×10^{-4} mol dm^{-3}; [CTAB] = 10.0×10^{-4} mol dm^{-3}; [Ag^+] = 16.0×10^{-4} mol dm^{-3}.

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