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Reducing Agent Concentration dependence on Size variation of Silver Nanoparticles growth

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Abstract: In the present work, AgNPs were prepared through chemical reduction method by varying the reducing agent concentration. Trisodium citrate ($Na_3C_6H_5O_7$) is used as reducing agent and PVA as capping agent. X-ray diffractometer (XRD) results showed that as reducing agent concentration increased, the average crystallite size decreased. FESEM results elicited decrement of particle size with increase of reducing agent concentration. EDS analysis confirmed the pure elemental silver formation. FTIR spectrum emphasized the intact of PVA with AgNPs. Photoluminescence studies revealed the enhancement in intensity with increase of reducing agent concentration due to decrease of particle size.
Keywords: nanoparticles; size control; concentration; photoluminescence.

Introduction:

The current trend in the synthesis of metal nanoparticles depends on the tenability of particle size. Nowadays silver nanoparticles (AgNPs) are of immense interest for scientists due to their potential usage in academic and industrial applications owing to their catalytic, optical and chemical stability properties [1-3]. Most common synthesis method is chemical reduction method [4]. As chemical reduction technique offers a systematic, efficient and simple procedure without decreasing the production rate. Temperature, pH, metal salts and reducing agent concentration and time of reaction effects the particle size critically. Tan et al. [5] reported AgNPs in the presence of aniline using sodium citrate. In this present work we have illustrated the dependence of particle size on trisodium citrate concentration.

Synthesis and characterization of silver nanoparticles:

In a typical synthesis process, 0.01 M of $AgNO_3$ solution is prepared with ethanol. Then, 0.05 M of trisodium citrate was added drop by drop followed by addition of 2ml PVA (1%). Then the solution was kept sealed under continuous stirring (i.e. 4 hrs) maintaining constant temperature of 75^{o}C. After the completion of the reaction, products were thoroughly washed for 3 times with ethanol and finally subjected to vacuum dry at 80^{o}C for 3 hrs. The above procedure is reiterated by varying the trisodium citrate concentration as 0.075, 0.10 and 0.125 M.

The XRD patterns of the samples were collected on a Rigaku D X-ray diffractometer. Elemental composition was examined through Oxford Inca Penta FET x3 EDS attached to Carl Zeiss EVO MA 15 scanning electron microscopy. The FESEM images were obtained using ZEISS, SUPRA55 field emission

scanning electron microscopy. FTIR spectra were recorded with ATR-FTIR, Bruker Vertex-80 spectrometer. Photoluminescence measurements were obtained using JobinYvon Fluorolog-3 spectrophotometer.

Results and Discussion:

Typical XRD profile of AgNPs at 0.10 M (Fig.1) shows the prominent peaks at $2\theta=38.15^{o}$, 44.24^{o}, 64.51^{o}, 77.42^{o} corresponding to (111), (200), (220) and (311) Bragg's reflections of the face-centered cubic structure of silver respectively (JCPDS No. 04-0783). The AgNPs size was determined from Debye-Scherrer's formula given as $D= K\lambda/\beta\cos\theta$, where D denotes the average crystallite size, λ is the X-ray wavelength, β is the angular line width at half maximum intensity and θ the half diffraction angle. As citrate concentration is increased from 0.05 M to 0.10 M, crystallite size is found to be decreased from 37 nm to 24 nm and again increased for 0.125 M. So, the optimum value for AgNPs synthesis is suggested to be 0.10 M.

FESEM images of the samples synthesized at 0.10 M and 0.125 M are displayed in Fig. 2, which confirms the existence of small and uniform spherical nanoparticles. Fig. 2(a) shows the AgNPs formation with average particle size around 25 nm (0.10 M) and for 0.125 M, it can be observed that larger particles of AgNPs are formed with 34 nm due to aggregation of nanoparticles. This could be due to decreased reduction rate. EDS spectrum reveals the strong signal in the silver region and confirmed the formation of pure AgNPs (Fig. 2(b)). Metallic nanocrystals of silver usually show distinctive optical absorption peak approximately at 3 keV due to surface plasmon resonance.

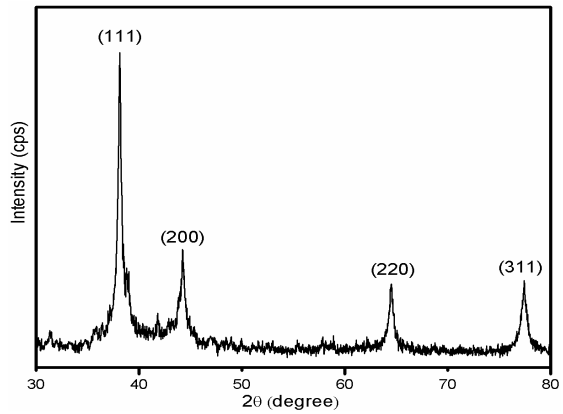

Fig. 1 Typical XRD profile of AgNPs prepared with 0.10 M trisodium citrate concentration.

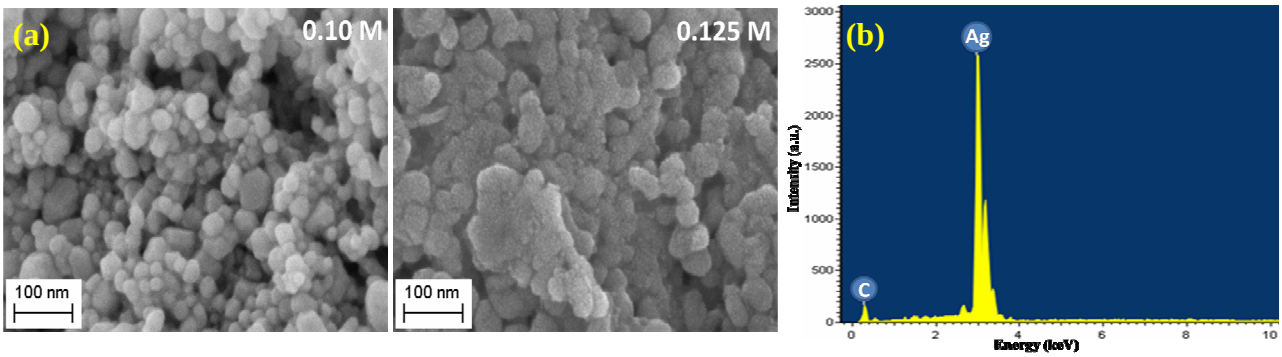

Fig. 2 FESEM images of AgNPs with (a) 0.10 M and 0.125 M of trisodium citrate concentration (b) Typical EDS spectrum of AgNPs prepared at 0.10 M citrate concentration.

The synthesized AgNPs were found to be photoluminescent at room temperature. PL spectrum of AgNPs when excited with 380 nm is shown in Fig. 3, which discloses the maximum intensity peak around 450 nm. It can be observed that as reducing agent concentration is increased from 0.05 M to 0.10 M, there is an enhancement in intensity whilst as reducing concentration is further increased to 0.125 M, the intensity of emission peak is decreased due to increase of particle size. Finally maximum intensity of emission peak is observed for 0.10 M reducing agent concentration with less particle size.

FTIR spectra of pure PVA and PVA capped silver nanoparticles (0.10 M) are shown in Fig. 4. The pure PVA sample shows strong absorption peaks at 3450, 3240, 1639, 1404, 1082 cm^{-1} corresponding to hydroxyl bands for free alcohol, hydrogen bond, the symmetric stretching of carboxylate anion (-COO-), O-H and C-H bending and C-O stretching respectively [6]. The spectrum of AgNPs had shown shifting in the bands due to the interaction of PVA with the surface of AgNPs by chemical adsorption. Hence, we can attest that prepared silver nanoparticles are capped by PVA.

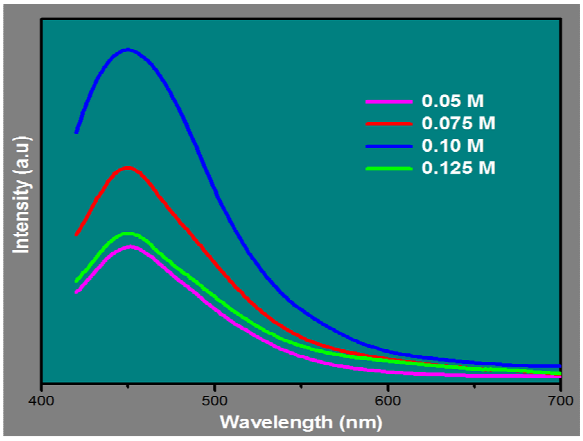

Fig. 3 PL spectra of synthesized silver nanoparticles at various reducing agent concentrations.

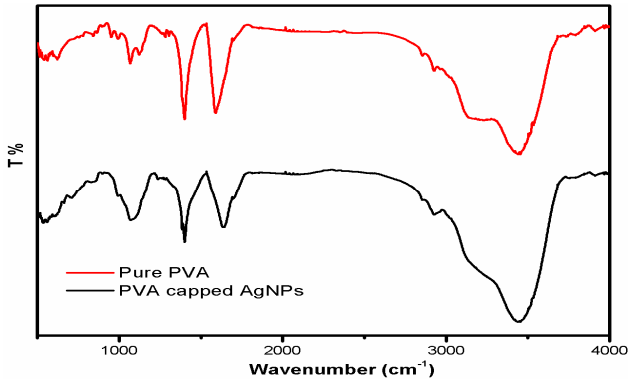

Fig. 4 FTIR spectra of pure PVA and PVA capped silver nanoparticles (0.10 M).

Conclusions:

Silver nanoparticles have successfully synthesized by chemical reduction method and demonstrated the effect of reducing agent ($Na_3C_6H_5O_7$) concentration on particle size. XRD pattern and SEM results confirmed the decrement of particle size with increase of reducing agent concentration. EDS profile revealed the formation of elemental silver. Photoluminescence studies elucidate the enhancement in intensity with increment in reducing agent concentration. Finally, FTIR spectra ascertain the encapsulation of silver nanoparticles by PVA.

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