Asian Journal of Chemistry; Vol. 25, Supplementary Issue (2013), S305-S307

ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in

Heat Mediated Synthesis of Silver Nanoparticles Using *Citrus limon* (Lemon) Extract†

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AJC-12860

In the present study, silver nanoparticles were rapidly synthesized by treating silver ions with *Citrus limon* (lemon) extract at higher temperature. The effect of process parameters like reductant concentration, mixing ratio of the reactants, concentration of silver nitrate and heating time period were studied. The formation of silver nanoparticles was confirmed by surface plasmon resonance as determined by UV-visible spectra in the range of 400-500 nm. X-ray diffraction analysis revealed the distinctive facets (111, 200, 220, 222 and 311 planes) of silver nanoparticles. Nanoparticles below 50 nm with spherical and spheroidal shape were observed from microscopic studies. The study offers a rapid method to synthesize silver nanoparticles within ten minutes of interaction with the bio-reductant.

Key Words: *Citrus limon*, Bio-reductant, Silver.

INTRODUCTION

Noble metal nanoparticles are widely used in the fields of medicine, biology, material science, physics and chemistry[1,2] in view of their application potential in areas such as photography, catalysis, biological labeling, photonics, optoelectronics and surface-enhanced Raman scattering[3,4]. Of the noble metal nanoparticles, silver is of particular interest because of its unique properties, such as good electrical conductivity, chemical stability, catalytic and antibacterial activity.

The quest for cleaner methods of synthesis has led to the development of bio-inspired approaches. Bio-inspired methods have been put forward to be advantageous over other methods of synthesis as they are cost effective and do not involve the use of toxic chemicals and extreme pressure conditions[5]. Silver nanoparticles have traditionally been synthesized using the salt of citrate[6] while gold nanoparticles have been synthesized using ascorbic acid as the reducing agent[7]. Since lemons are a rich source of citric acid and ascorbic acid[8,9], in the present study, fruits of *Citrus limon* (Lemon) were used as a bioreductant for the synthesis of silver nanoparticles.

EXPERIMENTAL

Lemons (*Citrus limon*) were squeezed to extract the juice which was later strained through a fine pore nylon mesh. The juice obtained was centrifuged at 10000 rpm for 10 min to remove any undesired impurities. This juice was used for the further experiments.

Synthesis of nanoparticles: In order to determine the optimum process parameters of the biological process, the factorial design of experiments, the one factor at a time method was employed in this study. This design of experiment implied that one experimental factor was varied at a time keeping the other factors constant.

Different concentrations of silver nitrate solution (10^{-2}, 10^{-3} and 10^{-4} M) were prepared and interacted with the juice of lemons in different mixing ratios (1:4, 2:3, 3:2, 1:1 and 4:1) at 80 ºC for different time periods.

Lesser concentrations of silver nitrate (10^{-3} and 10^{-4} M) did not yield significant results. Based on the results obtained, the standardized protocol was followed. 10^{-2} M silver nitrate was interacted with lemon juice (2 % citric acid concentration and 0.5 % ascorbic acid concentration) in the ratio of 1:4 and was heated to 80 ºC for 10 min.

Preliminary characterization of the silver nanoparticles was carried out using UV-visible spectroscopy. The purified powders obtained after 10 min of interaction at high temperature was subjected to X-ray diffraction analysis. The particle size range of the nanoparticles along with its polydispersity was determined using a particle size analyzer. Particle size was arrived based on measuring the time dependent fluctuation of scattering of laser light by the nanoparticles undergoing

†International Conference on Nanoscience & Nanotechnology, (ICONN 2013), 18-20 March 2013, SRM University, Kattankulathur, Chennai, India

Brownian motion. The morphology of the silver nanoparticles was determined using transmission electron microscopy (TEM).

RESULTS AND DISCUSSION

Commercially available sodium citrate has been used extensively for the synthesis of silver nanoparticles[6] while ascorbic acid though a weak reducing agent has been used for gold nanoparticles synthesis[7]. Lemon juice is a rich source of citric and ascorbic acid[8,9] and is also known for its water softening properties. Lemon juice was thus used in this study for the one point synthesis of silver nanoparticles.

The study was carried out for different time periods to determine the minimum time period of interaction. It was found that heating the reaction mixture to 80 ºC for 10 min was required for the formation of silver nanoparticles. Fig. 1 shows the UV-visible spectra of the silver nanoparticles obtained on varying the duration of heating. The absorption maximum of silver nanoparticles obtained on heating the reaction solution for 10 min was observed at 408 ± 0.033 nm. Further increase in the duration of heating led to a corresponding red shift in the absorption maxima.

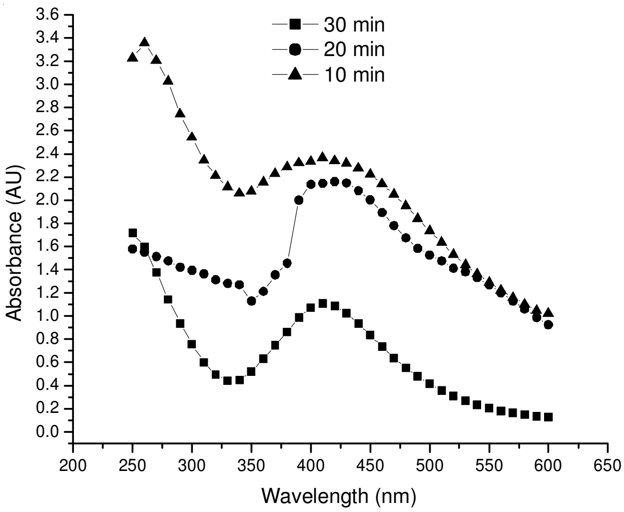

Fig. 1. UV Visible spectra of silver nanoparticles obtained on varying the duration of heating

Fig. 2 shows the XRD pattern of the synthesized silver nanoparticles. The peaks could be assigned to the five different facets of silver namely, (111), (200), (220), (311) and (222) planes[10]. A few unassigned peaks were also noticed in vicinity of the characteristic peaks. Intense Bragg reflections suggest that strong X-ray scattering centres in the crystalline phase and could be due to capping agents. Independent crystallization of capping agents were ruled out due to the process of centrifugation and are dispersion of pellet in millipore water after nanoparticle formation as part of purification process. The average particle size distribution of the nanoparticles in solution as determined by particle size analyzer was found to be 148 nm with a polydispersity of 0.309.

Fig. 3 represents the TEM micrograph of the synthesized silver nanoparticles. TEM images were taken randomly and spherical nanoparticles with a size ranging from 25-50 nm were observed.

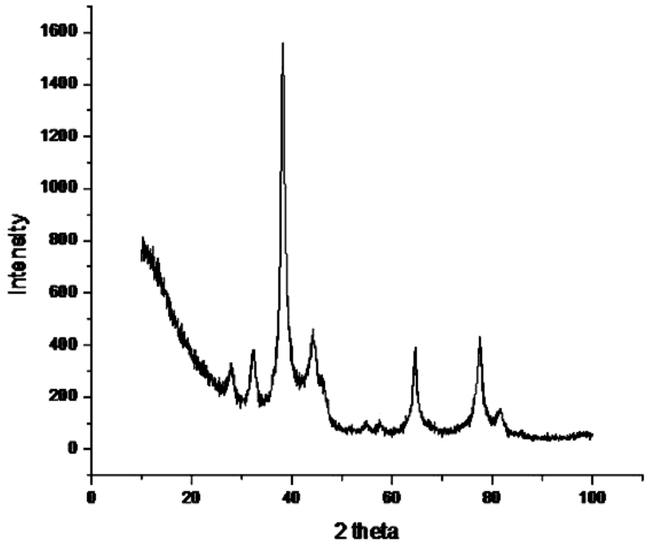

Fig. 2. XRD pattern of the biosynthesized silver nanoparticles

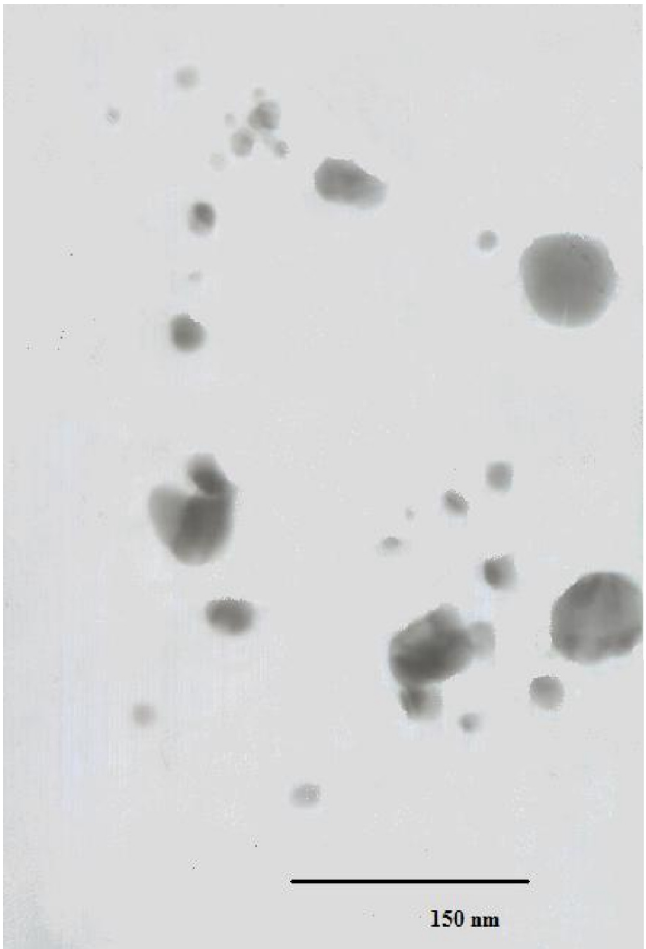

Fig. 3. TEM image of synthesized silver nanoparticles

Conclusion

A rapid synthesis protocol for silver nanoparticles was developed in the present study. This preparatory method is a novel and cost effective method that excludes the use of external stabilizing/capping agents. With an interaction time of just ten minutes, nanoparticles below 50 nm size with nearly spherical shape were produced.

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