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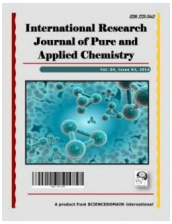

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Optimized Preparation of Silver Nanoparticles from Polyethylene Glycol and Formaldehyde

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Authors' contributions

This work was carried out in collaboration between all authors. The design of work was carried under direction of author DMK. Author NM involved in synthesis of the work and writing layout of the manuscript. All authors involved in writing, editing and proofing the manuscript and approved the final manuscript.

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ABSTRACT

The purpose of this work was to study the effect of various process and formulation parameters on size and shapes of silver nanoparticles (AgNPs) based on surface plasmon resonance (SPR). AgNPs were prepared by chemical reduction using formaldehyde (HCHO) as reducing agent and capped by polyethylene glycol (PEG). Effect of several processing variables including the concentration and volume of capping agent and reaction time is reported. The size of monodispersed nanoparticles was between 30-100 nm and was stable for three months at both room temperature and 4℃.

Keywords: Capping agent; full width at half-maximum; particle size; surface plasmon resonance.

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1. INTRODUCTION

The applications of nanotechnology in development is at its apace. Many metallic elements and their salts have various history of application in human history. Silver nanoparticles (AgNPs) have gained potential application in various fields including in biological sciences, food science, pharmaceuticals, packaging, electronic systems, mechanics, and information technology, among others. These is because, AgNPs tend to high surface-to-volume ratios which demonstrate unique properties with application values in electronic, water filtration, mechanical, photonic and optical, cell electrodes, biolabeling, integrated circuits sensors [1], deodorant fibers and antimicrobials [2-4].

The strategies to produce smaller size and expected shapes of to meet the required industrial applications are very complex. Different methods are already existing for preparation of AgNPs at small scales including chemical reduction [5,6], green method [7,8], sol-gel method [9], electrochemical reduction, heat evaporation, thermal decomposition in organic solvents [10], gas condensation [11,12], polyols process [13-15], Tollens process [16,17], pulsed laser ablation [18,19], chemical and photo-reduction in reverse micelles [20], and radiation in chemical reduction [2,21,22]. Chemical reduction method, one of the thermodynamically synthesis method [1] have been favored for preparation silver nanoparticles [4,23], however, the optimum production based on controlling some parameters is less reported. The advantage of chemical synthesis is its easiness in designing and process control, stabilization of nanoparticles, extraction of nanoparticles from solvent, surface modification and application, mass production and good chemical homogeneity.

In practice, successful preparation of metallic nanoparticles depends on both internal and external factors. The external factors depend on the processing conditions during processing. These include temperature and mixing shear rate applied [7]. The internal factors depend to formulation parameters including composition ratios, rheology, mixing sequence and the size and the shape and size of the mixing glassware. In AgNPs production, the reducing agent reduces silver ions (Ag+) to metallic silver (Ag^0), which is followed by agglomeration into oligomeric clusters if not controlled. It is common in chemical method that in the course of nanoparticles preparation, they are capped by capping materials protecting from sedimentation, agglomeration, or losing their surface properties [3]. Some common capping materials used are Some common capping materials used are sodium dodecylsulphate (SDS), citrate of sodium, polyvinyl alcohol (PVA), sodium polyacrylate, gelatin [5], polyethylenemine (PEI) [24], plyvinylypyrrolidone (PVP), polyethylene glycol (PEG) and cetyltrimethylammonium bromide (CTAB) [25].

In this research, we are used silver nitrate as metallic precursor, formaldehyde solution as reducing agent, polyethylene glycol as capping material and sodium hydroxide as the reaction promoter. The focus was to understand the influences of formaldehyde and concentration of capping materials on the shape and size distribution of silver nanoparticles. The processing was subjected in various formulations parameters to influence the reduction of nanoparticles from 100 nm to 30 nm.

2. MATERIALS AND METHODS

2.1 Materials and Reagents

All chemicals used in the experiment were analytic reagent (AR). Silver nitrate ($AgNO_3$ content 99.9%, Central Drug House (P) Ltd, New Delhi, India), polyethylene glycol (PEG) (Mw 6000-7500, Sisco Research Laboratories Pvt. Ltd, Mumbai, India), and formaldehyde (HCHO) solution (37-41% w/v) and sodium hydroxide (NaOH) pellets (98.0%) (SDFCL, Mumbai, India). All these reagents were used directly without further purification.

2.2 Preparation of Silver Nanoparticles

Colloidal silver particles were synthesized by the reduction of $AgNO_3$ with formaldehyde as reducing agent, PEG as surfactant agent and NaOH as catalyst. The initial concentration of AgNO3 was 1 mM, which mixed at constant molar ratio of either HCHO or NaOH solution to $AgNO_3$ at 10 factors. The volume of PEG used in the recipe are 10, 15, 20, 25 and 30 mL and NaOH solution was added to the reaction system to initiate the reduction, as well as to achieve a reaction time of several minutes. All measurements were performed at room temperature (25℃). Magnetic stirring was applied throughout the entire synthesis process. The reaction time for other control experiments is 30 min if not mentioned otherwise. The prepared

aqueous dispersion of the silver NPs was used for subsequent experiments without any additional modifications.

2.3 Characterizations

2.3.1 Dynamic light scattering analysis (DLS)

The particle size distribution (PSD) and zeta potential (ZP) of the silver nanoparticles was measured by dynamic light scattering (DLS) using a Brookhaven (Holtsville, NY) Zeta Plus instrument [26]. DLS data were analyzed at 25℃ and with a fixed light incidence angle of detection of 173º in optically homogeneous square polystyrene cells, using the backscattering technique. The mean hydrodynamic diameter (PS, Z-average) and PDI of the analyzed samples were obtained by calculating the average of 14 runs. All of the presented results of the particle sizes are listed as average values from three independent measurements.

2.3.2 UV-VIS spectrophotometer analysis

The ultraviolet (UV)-visible (VIS) spectra of the silver dispersion as the factor of reduction of metallic silver nitrate to nanoparticles was analyzed by Perkin Elmer (Boyton Beach, BL) Lambda 35 UV-VIS spectrometer according to [26].

2.3.3 Transmission electron microscopy (TEM)

To observe the structures of AgNPs, HR-transmission electron microscopy (HRTEM) operating at 120 KV were conducted for dried samples [24]. A small amount of the dried sample was dispersed in ethanol and the suspension was treated sonication for 10 min and then 10 µL of solution was dropped onto a Formvar-covered copper grid placed on filter paper. After evaporation of ethanol, conventional TEM micrographs were recorded.

3. RESULTS AND DISCUSSION

The mechanism of nanoparticle formation have been reported to in main two phases, particle nucleation and growth by coagulation and coalescence. These phases can be controlled to produce specific tailored particles characteristic by controlling process and formulation parameters such as temperature, reaction time and concentration. These results are based on several optimizing experiments, which lead to decide on few factors for presentation of our laboratory protocol. The results presented are based on dropwise addition (injection) of silver nitrate solution in the mixture of PEG and HCHO and subsequently addition of NaOH in the dispersion.

3.1 Theoretical Reduction of $AgNO_3$ by Formaldehyde

There are two theories that can be used in preparation of silver nanoparticles using formaldehyde as reducing agent. The first is that the presence of NaOH in the reaction leads to the Canizarro's reaction, which is essentially the auto-oxidation-reduction of formaldehyde. Consequently, formic acid (HCOOH) and methanol (CH_3OH) are formed, where by methanol is easily converted to formic acid. The HCOOH is produced as the sodium salt, sodium formate (HCOONa). HCHO undergoes Cannizarro's reaction as follows:

$$2HCHO + OH^- \rightarrow HCOO^- + CH_3OH \quad (1)$$

$$Ag^+ + HCOO^- \rightarrow Ag + CO_2 + \tfrac{1}{2}H_2 \quad (2)$$

In addition, stoichiometric reaction between formaldehyde and silver ion in an alkaline solution by injection can be written as:

$$2Ag^+ + HCHO + 3OH^- \rightarrow 2Ag + HCOO^- + 2H_2O \quad (3)$$

$$2Ag^+ + HCHO + OH^- \rightarrow 2Ag + HCOO^- + \tfrac{1}{2}H_2 \quad (4)$$

Theoretically, all equations hold the facts based on the formulation procedures. The equations (1) and (2) normally occur when the mixing process is based on injection of silver nitrate in the reaction mixture. The primary solution is formed between HCHO and NaOH in presence or absence of stabilizing agent. The Canizarro's reaction occurs at this point with formation of sodium formate and methanol. Further injection of the metallic silver nitrate to the mixture allows for the reduction of silver ion to nanoparticles. Equations (3) and (4) are based on the addition of HCHO and then stabilizer in the silver nitrate in the mixing vessel. Generally, formaldehyde reacts with the hydroxyl ion by nucleophilic addition reaction to produce hydride and formate ions. It is the hydride ions which reduce silver ion to silver atom and may become hydrogen itself as a by-product.

3.2 Qualitative Analysis of Silver Nanoparticles Preparation

In the course of preparation of AgNPs, we took care of several factors to observe their effects in the intensity of colour change as the indicator of nanoparticle formation. The volume of reducing and capping agents, temperature of reaction and holding time of the reaction were observed in Fig. 1. Several mixing procedures were carried out to optimize the reaction conditions at 80℃ for the nanoparticles. Simultaneously additional, sequentially addition and injection of silver nitrate was carried out to observe the colour changes. In simultaneous addition of all materials and then holding the experiment at 80℃, no clear colour was observe even at extended time of reaction. The glassy translucent colour was observed, which indicates presence of PEG in the reaction. Holding the mixture of silver nitrate and PEG at 80℃ and further addition of HCHO in the mixture, no colour was observed until some drops of NaOH added. NaOH triggered colour changes from shiny glassy into slight yellow and then yellow.

Addition of more than 3 mL of NaOH into the solution led to pale yellow colour and then blackish color. The colour change indicated the formation of silver nanoparticles in the reaction; however, when large amount of NaOH used the nanoparticles were aggregated into blackish colour, which indicated large particle size formation. This is very interesting observation as higher amount of NaOH in reacting species, increased the reduction mechanism and chemical kinetic of silver nitrate. Similarly, the colour change to blackish was apparently when more amount of HCHO into the solution of $AgNO_3$, PEG and 2 mL NaOH. The injection of $AgNO_3$ into hot solution of PEG, HCHO and NaOH the colour changes were immediately observed from glassy to yellow indicating the formation of silver nanoparticles. Holding the reaction for extended time at 80℃ there was slight changes of colour from pale yellow to wool-like-blackish on the top or opaque with phase-separation. As the reaction time was extended to 1 h, the color of the solution did not change significantly but more precipitate was formed. Additionally, rise in reaction temperature from 80 to 90℃ after colour change accelerated the colour change in the solution and segregation. Consequently, qualitative studies led to design new experiments based on the volume of and concentration of PEG and reaction time.

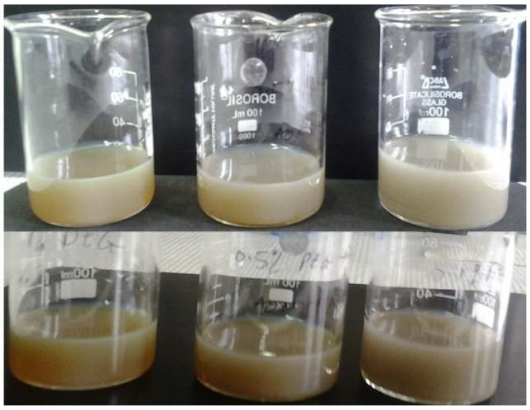

Fig. 1. Some optimization processes for AgNPs preparation

Literature shows that weak reducing agents have slow reactions with metallic precursors which need the activation by basic solution of NaOH or sodium bicarbonate to form a basic or neutral reacting species [1,3]. Addition of NaOH in reacting species increases the pH of solution and favors for more reduction and formation of AgNPs. However, increase in pH of solution results onto precipitation of the products which settle at the bottom of solution due to diffusion of hydroxyl ion (OH^-) into the stable electric double layer which attracts the collisions of particles. In addition, at higher pH PEG undergoes phase separation processes leading to the precipitation of large particles due to hydrophobicity system, with identical compositions of the equilibrium phase. The dynamic phase-separation of PEG may lead to metal complexation and partition due to chemical participation of solvated anion from the polyethylene oxide chains. Further analysis shows that, at the temperature between 80-100℃ sodium hydroxide reacts with PEG and precipitation beside PEG acting as reducing, solvent and chelating agent in the reaction [27,28].

3.3 Dynamic Light Scattering Analysis (DLS)

With DLS results, three factors were taken in consideration; holding time of the reacting species, volume and concentration of the capping agent. Holding the nanoparticles at the same temperature for extended time has been noted to change the particle size. Reaction time in nanoparticles preparation for more than 10 min observed to accelerate particle aggregation as in Fig. 2. Within 5 minutes of reaction, nanoparticles were found to be monodisperse with low dispersity index, indicating their stability

and shelf life. The particle distribution was increasing as the holding time increase from 10 to 30 min. This showed that, the polymeric layer at the interface hinder the growth surface of monosized metallic nanoparticles, remaining monodisperse. In this case, the diffusion process is likely to be the rate-limiting step of subsequent growth of initial nuclei, which can favor the formation of uniformly sized nanoparticles once the reaction time is controlled. As the reaction time increased, the dispersed silver nanoparticles was prone to agglomeration as the reaction extends and could lead to decreased yield of product [14].

The effects of concentration of PEG demonstrated that low concentration leads to large nanoparticles, especially in injection of silver nitrate in solution containing other recipes. Increased concentration leads to decreased particle size until reach the optimal concentration. At high concentration of PEG, nanoparticles shown large particle size in Fig. 2. The nanoparticles observed to be widely dispersed and the distribution was much skewed. In this regard, at lower concentration of PEG, the aggregation of particles was favored due to lower adsorption of PEG around the surface of nanoparticles. When the optimum concentration of PEG met, the lowest particle size was observed due to steric hindering effects. The stability of nanoparticles from this dispersion was very stable at room temperature and at 4℃ for more than 3 months. Higher PEG concentrations decrease the interfacial tension because of localization of the PEG molecules and strong adsorption at the interface, resulting into stable particle size and an increased surface area in the dispersed hydrophilic phase.

Additionally, more concentration of PEG incorporated with NaOH and HCHO was not able to reduce the added silver nitrate due to restricted interactions. Consequently, the reaction deficit of reacting recipes may lead to poor yield of products because some of silver nanoparticles tend to retain in aqueous phase. This is in agreement by Agnihotri and others [6], that the higher concentration of reactants was required for synthesizing larger silver nanoparticles (≥ 50 nm) based on full width at half maximum (FWHM), the phenomenon which is going to discussed in next section [27,28].

The concept of effect of volume of PEG in the solution is similar to that of concentration. The volume implication is based on the contents of PEG in the reaction process and access of reacting species to initiate the reaction [27-29]. It can be easily understood by considering the fact that increased amount of polymer produces steric resistance for the diffusion and consequently results in a diffusion controlled growth, which favors the formation of spherical particles. However, based on the results in Fig. 2, as the contents of silver nitrate exceed a certain limit, the diffusion free interaction of species is controlled which could limit the yield of nanoparticles. From classic chemistry, dilute NaOH can easily precipitate aqueous silver nitrate into brown solution. In presence of PEG molecules, the addition of NaOH in the reacting solution formed brown solution, which gradually change into yellowish green color as reaction was completed. Changes in colour suggested that silver ions were reduced into extremely fine silver particles [6]. This have been confirmed in our work that, the nanoparticles produces by using only NaOH was in the range of atomic size, 250-500 nm. There, using high volume of PEG more distributed nanoparticles were observed. This phenomena have been observed on the effects of increasing the volume of HCHO [24].

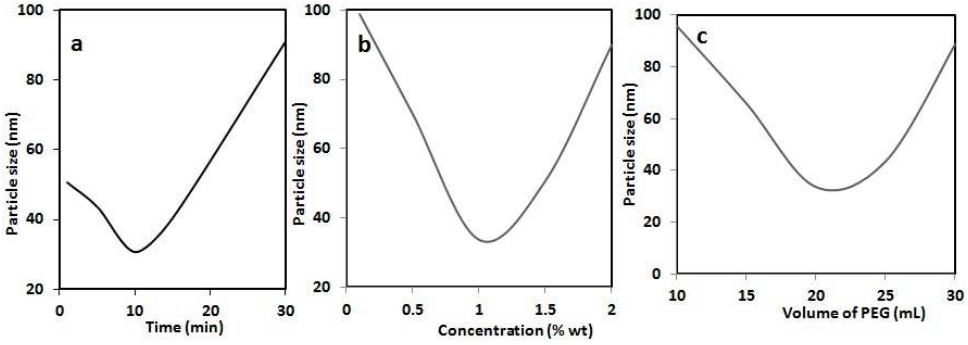

Fig. 2. Effects of reaction time (a), concentration (b) and volume of capping agent (c) on silver particle size

3.4 UV-VIS Spectrophotometer Analysis

UV–Vis spectroscopy is used to confirm sample formation by showing the surface plasmon resonance (SPR) [4]. As shown in Fig. 3, silver nanoparticles exhibit a sharp extinction peak at wavelength around 450 to 480 nm for silver nanoparticles with the average sizes between 30-40 nm. The absorption maximum for the silver nanoparticles increased with increasing their particle size. Similarly, the full width at half maximum (FWHM) of the nanoparticles have increased with increase in particle size from 30 to 98 nm. Reaction time has the lowest FWHM due to small particle formation, followed by volume of PEG used and finally the concentration of PEG. The full width at half maximum (FWHM) of the corresponding peaks determines dispersity of the nanoparticles in particular formulation, where a large FWHM is attributed to peak broadening and hence, polydispersity. Increase in particle size decreases in the intensity is due to change in the free electron density. Particle aggregation trends occur with extraction yields a variation of the width and the red-shift of the maximum in the absorption spectrum. From this scenario, the concentration of the capping agent in the reacting species is the truest determinant of reaction processes. That is working on optimization of concentration of species can reflect the final products than relying of the volume used or reacting time. However, other factors can affect adversely the processes when are not considered during the process.

Reference [26] described the UV spectra band and the position of band occurs after 304 4 reactions minutes, however some intensity observed before that time is due to nucleation process which can occur. The increase in reaction time increases the spectra intensity which shifts the position of spectral peak to higher wavelengths. At optimal reaction conditions the smallest size of particles is formed and then further peaks are formed towards higher wavelengths which indicate the increase in diffusional growth and formation of large particles due to aggregations [7]. Similarly, increase in concentration of capping agent based on molar ratios between metallic precursors and the capping tends to shift the plasmon band/peaks to higher wavelengths due to increase in particle reduction and provides physical barrier which prevents the agglomeration progression and then enhance the coating and stabilization of the formed nanoparticles.

The SPR band depends much on the size, shape, morphology, surface-adsorbed species, composition and dielectric environment of the prepared nanoparticles [4,7]. The SPR of silver nanoparticles tends to shift to longer wavelengths with increasing particle size, the spherical nanoparticles show single SPR band and anisotropic particles show as a minimum two SPR bands [21].

3.5 Transmission Electron Microscopy (TEM)

The as highlighted above, concentration of capping materials have the highest effect to both stability and formation of metallic nanoparticles [16]. It dictates the particle size, shapes and morphology of nanoparticles. The TEM micrographs in Fig. 4 show that increase in concentration in PEG increased the dispersion of AgNPs in the emulsion. The whole effects may be attributed with reduction in production of new AgNPs at higher concentration of PEG due to occupation of free space with PEG matrix. This prevents creation of active nucleic sites and reduces the agglomerations of particles, hence increase in particle stability [14,30]. The PEG attaches to the surface of AgNPs and prevents the [29] diffusion of AgNPs from the surrounding solution causing diffusion processes as the rate limiting step. In this case, more monodispersed and stable nanoparticles are obtained.

The effect of capping agent on metallic nanoparticle formation has been reported equally [27,29]. Its increase has been found to form more small, stable and spherical nanoparticles. The effects are due to steric resistance for diffusion process which favors the spherical particle formation with spatial phase separations of reactants. The PEG matrix in the reacting species mediates the interface immiscible phases and the intensity of inter-phase transports and affects the rate of interactions between the phases. This dissolution-growth process is also known as Ostwald ripening, in which large particles grow at the expense of small particle by diffusing through the interface. As a result, nuclei with small size may become unstable and dissolve back into the solution; dissolved species will then deposit onto the surface of large particles. However, in this case, a higher reagent through such nucleation prevents further nucleation and largely hinders the subsequent growth of existing nuclei.

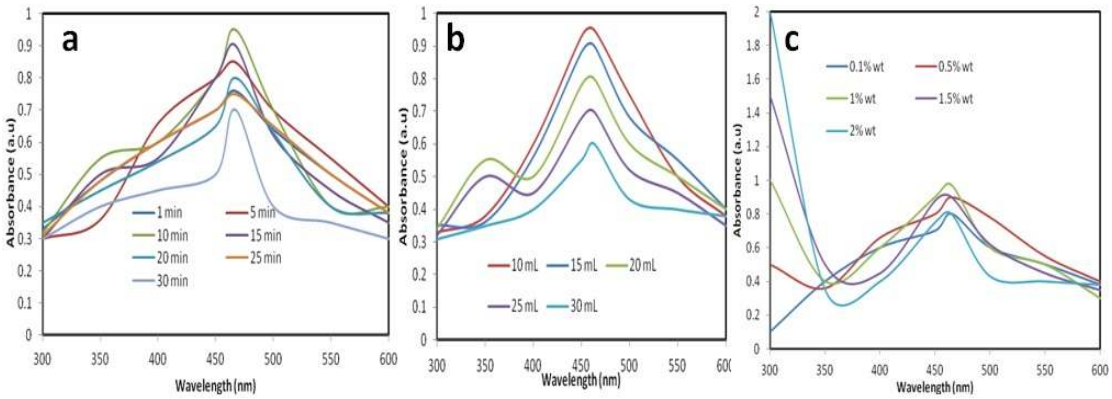

Fig. 3. UV spectra due to effects on (a) reaction holding time, (b) volume of PEG and (c) concentration of PEG in preparation of AgNPs

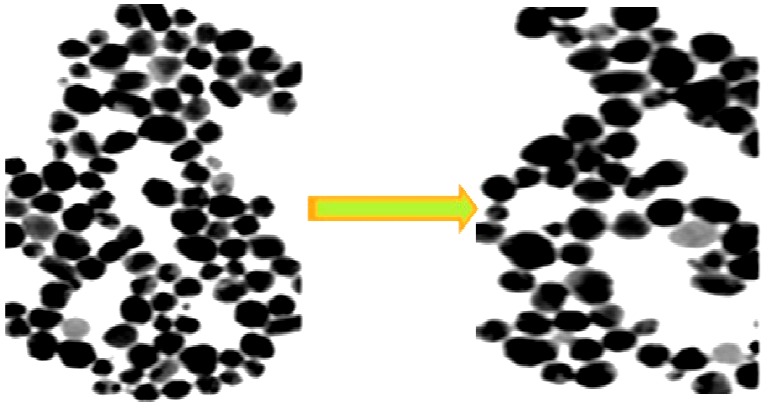

Fig. 4. Micrograph of TEM showing dispersion of AgNPs in the highest and the lowest concentrations of PEG in reacting species

4. CONCLUSION

The synthesis of AgNPs and other metallic nanoparticles have both process and formulation dependences. The particle size, shape, structural crystals, stability, morphology and orientation of can undergo dynamic modifications on changing one factor during processing. In many processes tried to produce the AgNPs using HCHO as reducing agent and PEG as capping agent the injection of reagents into the hot reacting vessel resulted into better yield at shot time. A change in color intensity was the definite qualitative way to decide the formation of AgNPs. At higher color intensity indicated the depletion of one of the reacting reagent which can prevent further reaction and subsequently the agglomerations of particles could start. Increase in volume and concentration of capping agent as well as the holding time of reacting species at higher temperature were noticed to affect the particle size of AgNPs. An increased temperature results in an increased solubility, and thus a reduced supersaturation of growth species in the solution. As a result, nuclei with small sizes may become unstable and dissolve back into the solution; dissolved species will then deposit onto the surfaces of large particles as Ostwald ripening phenomena described.

However, concentrations of changing reacting species have more noticeable effects on particle formation. The capping agent observed to dictate the diffusional processes in the reacting species by occupying the free volume at the interface between metallic precursor and reducing agent. This has showed that, increase of capping agent at the interface lead to monodispersed particles and stable yields for at least three months. This agent tends to cap the metallic clusters due to coating in the non-polar aqueous medium.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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