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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 Waals forces and hydrogen bonding. Cetyltrimethylammonium bromide (CTAB) acted as a stabilizer and/or capping agent.

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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

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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)	$10^4 k_{\text{obs}}(\text{s}^{-1})$
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

scaled up for large-scale synthesis (Esumi et al., 1990; Taleb et al., 1997; Zhu et al., 2000; Henglein, 2001; Sharma et al., 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 $(1-2) \times 10^{-6} \text{ } \Omega^{-1} \text{ cm}^{-1}$) 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 condenser 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.

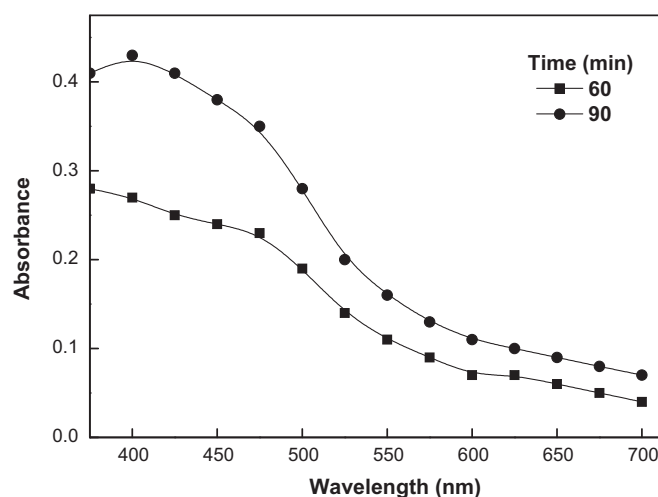


Figure 1 Spectra of AgNPs at different time intervals. Reaction conditions: [aniline] = $40.0 \times 10^{-4} \text{ mol dm}^{-3}$; [CTAB] = $10.0 \times 10^{-4} \text{ mol dm}^{-3}$; $[\text{Ag}^+] = 16.0 \times 10^{-4} \text{ mol dm}^{-3}$.

2.3. Preparation and characterization of AgNPs

In a typical experiment, AgNO_3 solution (0.01 mol dm^{-3}) and CTAB (0.01 mol dm^{-3}) were used, respectively, as a metal salt precursor and a stabilizing agent. Aniline solution (0.01 mol dm^{-3}) was also used as a reducing agent. The transparent colorless reaction mixture (Ag^+ ions + CTAB) was converted to the characteristic pale yellow color after the addition of aniline. Appearance of perfect transparent color was due to the formation of AgNPs (Huang et al., 1993). For TEM measurements, samples were prepared by placing a drop of working solution on a carbon-coated standard copper grid (300 mesh) operating at 80 kV. The Fourier transform infrared (FT-IR) spectra experiments were carried out on a Bruker Equinox 55 spectrophotometer. A few drops of the resulting sol were placed on KBr pellets and allowed to dry. IR spectra were recorded.

3. Results and discussion

3.1. General considerations

The pH of a reaction mixture plays an important role in the preparation and synthesis of differently shaped and sized nanoparticles and nanocomposites of Ag and Au ions in the presence of amino acids (Dey, 2005). It is well known that, the preparation of water soluble colloidal particles and silver sol strongly depends on the pH of the working solution and is unstable in aqueous solutions of $[\text{H}^+] > 1.0 \times 10^{-3} \text{ mol dm}^{-3}$ and its growth can be stopped by adding small amounts of mineral acids (Linnert et al., 1990). Therefore, the control of pH is a crucial problem that we address first. The control of pH is not as straightforward in micelles solutions as in ordinary solvents (Tondre and Hebrant, 1997). In

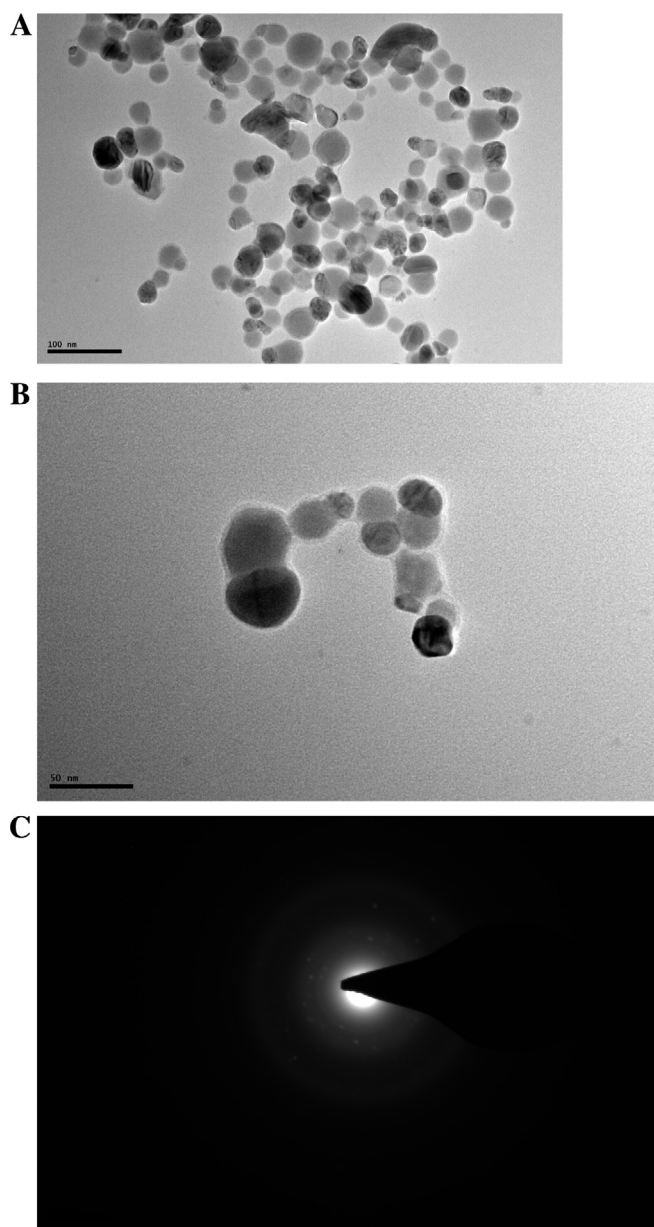


Figure 2 TEM images of Ag-nanocrystals. Reaction conditions: $[\text{CTAB}] = 10.0 \times 10^{-4} \text{ mol dm}^{-3}$; $[\text{Ag}^+]/[\text{aniline}] = 0.7$ (A), and 0.35 (B). Selected area diffraction images of randomly selected Ag-nanocrystals (C).

order to see any change in the macroscopic pH during the nucleation and growth processes, pH was recorded as a function of [CTAB] and aniline. The pH values were found to be nearly constant with increasing [CTAB] and [aniline] (pH was found to be constant) (Table 1). It is not surprising because ionic micelles show a marked difference in the effective local pH to exist at their micellar surface over those in bulk aqueous solvent. The different pH in the micellar pseudo phase of ionic micelles is understandable because most of the ionic (acidic and basic) species should be in the micellar phase and in addition pKs in micellar systems are almost of different times from the pKs measured in water.

In the first set of experiments, a solution of aniline (10 cm^3 , 0.01 mol dm^{-3}) was added to a series of solutions of silver nitrate (8 cm^3 , 0.01 mol dm^{-3}) and CTAB ($=2\text{--}20\text{ cm}^3$, 0.01 mol dm^{-3} , total vol. 50 cm^3). The most interesting features of the present observations are the fast change in the color of the AgNO_3 -CTAB in the presence of aniline. At lower [CTAB] ($<4.0 \times 10^{-4}\text{ mol dm}^{-3}$), the reaction mixture becomes turbid and a yellowish-white precipitate appears whereas, the formation of perfect transparent yellow color is observed in [CTAB] ($>4.0 \times 10^{-4}\text{ mol dm}^{-3}$). The appearance of color is caused by the surface plasmon resonance of Ag-NPs in the visible region (Burda et al., 2005). Formation of transparent silver sol was observed between $4.0 \times 10^{-4} \leq [\text{CTAB}] \leq 14.0 \times 10^{-4}\text{ mol dm}^{-3}$ with $[\text{Ag}^+] = 16.0 \times 10^{-4}\text{ mol dm}^{-3}$ and $[\text{aniline}] = 20.0 \times 10^{-4}\text{ mol dm}^{-3}$. At higher [CTAB] ($\geq 16.0 \times 10^{-4}\text{ mol dm}^{-3}$), the reaction mixture becomes turbid and a white yellowish gray precipitate appears. In the second set of experiments, different volumes of Ag^+ ion solutions in the range of $1\text{--}10\text{ cm}^3$, 0.01 mol dm^{-3} were added to a reaction mixture containing aniline (10 cm^3 , 0.01 mol dm^{-3}) and CTAB (4 cm^3 , 0.01 mol dm^{-3}). Yellowish-black precipitate is formed at $[\text{AgNO}_3]$ ($>5.0 \times 10^{-3}\text{ mol dm}^{-3}$).

3.2. Spectra and TEM images of AgNPs

It is well known that AgNPs exhibit yellowish brown color in aqueous solution due to the excitation of a surface plasmon resonance band in the UV-visible region (Liz-Marzan, 2006).

As the aniline solution was mixed with an aqueous solution of the silver nitrate and CTAB, it started to show the appearance of pale-yellow color due to the reduction of the silver ion; which indicated the formation of silver nanoparticles. It is generally accepted that UV-Vis spectroscopy could be used to examine size and shape-controlled nanoparticles in aqueous solution. UV-Vis spectra recorded from the reaction medium at different time intervals are depicted in Fig. 1 which consisted of a single sharp surface plasmon resonance band at 400 nm . The most characteristic part of the silver sol is a narrow plasmon absorption band observable in the $350\text{--}600\text{ nm}$ regions. A broad surface plasmon resonance band is due to the aggregation and/or adsorption of aniline onto the surface of Ag-nanocrystals.

TEM images of the silver sol are shown in Fig. 2. These results indicate the adsorption and/or deposition of silver nanoparticles onto the surface of roughly sphere-shaped polydispersed particles for $[\text{Ag}^+]/[\text{aniline}]$ ratios of 0.8, 0.4 and 0.3, respectively. The Ag-nanocrystals that emerged in the images have a variety of shapes: spherical, triangle and irregular. Selected area electron diffraction reveals the presence of ring patterns in the single face-centered cubic (fcc) crystalline nature of the spherical nanoparticles with a preferential growth direction along the Ag (110), (200), (220), (311) and (331) planes. In agreement with the UV-vis spectrophotometric observations, the TEM images reveal that Ag-nanocrystals are polydisperse, irregular in deposition and roughly spherical with a rather similar diameter of 30 nm . Our results are quite consistent with those of various investigators (Sondi and Salopek-Sondi, 2004) for the synthesis of silver nanoparticles by the chemical reduction method. Bakshi et al. Bakshi (Bakshi, 2010) and El-Sayed et al. Petroski et al. Petroski et al. (1998) suggested that faceted nano-fibers or rodlike nanoparticles are formed during the reduction of metal salts by weak reducing agents where growth occurs over a longer period.

3.3. Effect of [reactant], pH and mechanism of AgNPs

The effect of [aniline] on the growth of AgNPs was studied between $2.0 \times 10^{-4} \leq [\text{aniline}] \leq 30.0 \times 10^{-4}\text{ mol dm}^{-3}$ at fixed

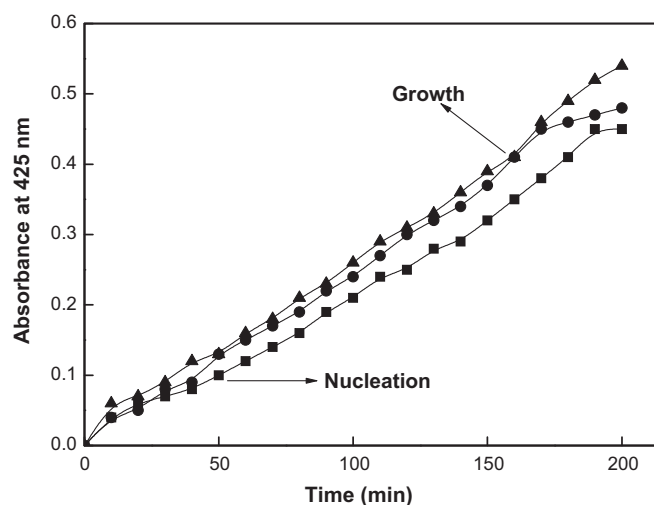
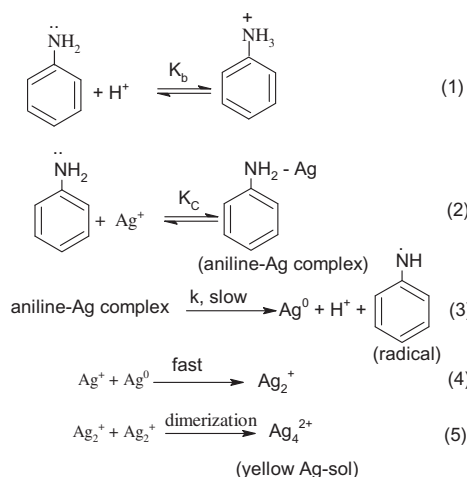


Figure 3 Plots showing the effects of [aniline] on the absorbance of AgNPs. Reaction conditions: $[\text{Ag}^+] = 40.0 \times 10^{-4}\text{ mol dm}^{-3}$; $[\text{CTAB}] = 10.0 \times 10^{-4}\text{ mol dm}^{-3}$; [aniline] = 20.0 (■), 30.0 (●) and $40.0 \times 10^{-4}\text{ mol dm}^{-3}$ (▲).

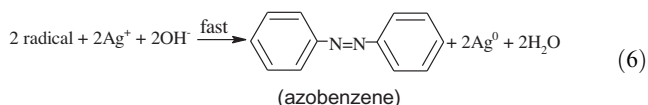


Scheme 1 Mechanism to the reduction of Ag^+ by aniline.

$[\text{Ag}^+] = 16.0 \times 10^{-4} \text{ mol dm}^{-3}$, $[\text{CTAB}] = 10.0 \times 10^{-4} \text{ mol dm}^{-3}$ (Table 1). Interestingly, we did not observe the formation of perfect transparent silver sol at lower $[\text{Ag}^+]$ ($\leq 2.0 \times 10^{-4} \text{ mol dm}^{-3}$). Measurements of reaction rates were also hampered due to the formation of a gray precipitate at higher $[\text{Ag}^+]$ ($\geq 35.0 \times 10^{-4} \text{ mol dm}^{-3}$). The reaction-time profile (absorbance *versus* time plots) suggests that autocatalysis is involved in the silver sol formation (Fig. 3). It was noted that the extent of the induction period (nucleation) depends on [aniline]. Thus, we concluded that the aniline- Ag^+ reaction has nucleation, growth and autocatalytic reaction paths (autocatalysis is due to the formation of a metal nucleation center which acts as a catalyst for the reduction of other Ag^+ present in solution).

Henglein and his coworkers monitored the stepwise growth of silver clusters by spectroscopic methods in the presence of polymers. These investigators used the radiation method in the reduction of Ag^+ into Ag^0 (hydrated electrons in pulse radiolysis) and also reported the mechanism for the production of metallic colloidal Ag particles (Linnert et al., 1990; Ershov and Henglein, 1998). On the basis of above observed results and previous observations, the following mechanism for the formation of AgNPs has been proposed (Scheme 1).

In Scheme 1, Eqs. (1) and (2) represent the protonation of aniline and the formation of aniline-Ag complex, respectively. Eq. (3) (rate-determining step) is a one step one-electron oxidation reduction mechanism leading to the formation of Ag^0 and radical. In the next steps, Ag^0 atoms formed a complex with Ag^+ ions yielding Ag_2^+ ions (Eq. (4)), which are ultimately converted into the stable species of silver after dimerization to yield Ag_4^{2+} (Eq. (5)). The proposed mechanism is further supported by the detection of the radical with acrylonitrile (Singnorella et al., 1996). Radical was converted into the stable oxidation products of aniline, i.e., azobenzene after the dimerization and oxidation by available Ag^+ ions (Eq. (6)).



The nature of azobenzene as the oxidation product of aniline was confirmed by comparison of its FT-IR spectra against an authentic sample of azobenzene.

On the basis of the Scheme 1 mechanism, the following rate law consistent with the experimental results has also been derived.

$$\frac{d[\text{Silver} - \text{sol}]}{dt} = K K_b K_c [\text{Ag}^+][\text{aniline}] \quad (7)$$

3.4. Probable reaction site to the growth of AgNPs

It is well established that micelles are dynamic aggregates of amphiphilic molecules, surfactant, that create highly anisotropic interfacial regions lining the boundary formed by the highly polar aqueous and nonpolar hydrocarbon regions, imparting new chemical and physical properties to the system. Micelles, as well as other association colloids, can alter nanoparticle's shape, size and other surface properties to different extents depending up on their molecular structure i.e. nature of head group, length of hydrophobic tail and type of counterions (Tascioglu, 1996). As a result, various properties such as oxidation-reduction potentials, dissociation-ionization constants, quantum efficiencies, reaction mechanisms, size, and shape of inorganic nanocrystals are changed. In the present study, role of CTAB can be explained by incorporation, and/or solubilization of aniline, aniline- Ag^+ complex and/or silver nanoparticles onto the surface of CTAB micelles. Aniline may be assumed to be totally present in the Stern layer of cationic CTAB micelles due to the ion-pair formation between the positive head group ($-\text{N}^+(\text{CH}_3)_3$) and lone-pairs of $-\text{NH}_2$ group of aniline. On the other hand, the presence of the non-polar phenyl group is responsible for the solubilization of aniline into the micellar palisade layer (a few carbon atoms deep toward the core) hydrophobically. Amino group has a strong tendency to coordinate with Ag^+ . In micelle-mediated reactions, it is not possible to precisely locate the exact site of the reaction but, at least, localization of the reactants can be considered (micellar surfaces are water-rich and do not provide a uniform reaction medium because a micelle is a porous cluster with a rough surface and deep-water-filled cavities).

It is well known that the nature of reducing agents (reduction potential, E_0) is an important factor, also the control of the size, shape and the size distribution of the Ag-nanoparticles. Interestingly, the reactivity of paracetamol toward Ag^+ ions is much higher than the corresponding oxidation of many other organic substrates (ascorbic acid, cysteine, aspartic acid, hydrazine, citric acid and its sodium salt), although it is a weaker reducing agent (Al-Thabaiti et al., 2008; Khan et al., 2009; Ahmad et al., 2010; Rafey et al., 2011). This is not very surprising judging from the fact that the presence of OH^- ions in the form of NaOH is responsible for the higher pH of the reaction mixture. As a result, paracetamol easily transfers the proton to Ag^+ ions leading to the formation of Ag^0 . It can now be stated confidently that the pH of the working media is particularly important along with the E_0 .

4. Conclusions

In this paper, we demonstrated a one-pot, simple, easy chemical reduction method in the synthesis of AgNPs in the presence of cationic micelles of CTAB. Mechanism, rate-law, probable reaction site and selected area electron diffraction were discussed for the first time in the preparation of

monodisperse and aggregated roughly spherical AgNPs by a simple chemical reduction method using aniline. The nanoparticles formation (pale yellow color) was monitored by recording the temporal course of their absorption spectra. The single-crystallinity of AgNPs was also confirmed by the selected electron diffraction patterns. It was found that the shape and size distribution of the AgNPs strongly depend on the experimental conditions, [aniline], [CTAB] and pH. Further progress in this area will provide green paths in the synthesis of controlled shape and size of AgNPs as well as speed up the removal of contaminants from drinking water.

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