



# Facile Synthesis and Characterization of Monodisperse Silver Colloidal Nanoparticles Stabilized by Sodium Laurate

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## Abstract

A facile process was present in this paper for synthesizing nano silver particles colloidal in aqueous solutions using silver nitrate, sodium laurate, and hydrazine hydrate as silver precursor, stabilizer, and reducing agents, respectively. The generation of nano silver particles was confirmed by change of color from milk white to brown or dark. The prepared silver nanoparticles were analyzed by scanning electron microscope (SEM), transmission electron microscope (TEM), UV–Visible spectroscopy (UV–Vis), X-ray diffraction (XRD), and Fourier transform infrared spectroscopic (FT-IR). The SEM and TEM images indicated that the prepared silver nanoparticles have spherical shape, average size of 6 nm, and with a narrow distribution from 2 to 8 nm. The XRD information exhibited that the obtained metallic nanoparticles were highly crystalline in nature. FT-IR data demonstrated that the sodium laurate absorbed on the surface of silver nanoparticles. The effects of the sodium laurate and hydrazine hydrate on the silver nanoparticle formation were studied. The mechanism of the formation silver nanoparticles was discussed. The process raised in this study can be considered as an excellent candidate for the preparation of silver nanoparticles in a large-scale production. The simple method can be extended to synthesis of other noble metal nanoparticles.

**Keywords** Crystal morphology · Sodium laurate · Silver nanoparticles · X-ray techniques · Electron microscopy

## 1 Introduction

During the past two decades, inorganic nanoparticle study has been emerged as one of the most active areas of research all over the world (Abdelmoteleb et al. 2016;

Abkenar and Naderi 2016; Chauhan et al. 2012; Chowdhury et al. 2014; Dobrucka 2016; Dong et al. 2014b; Ghoreishi et al. 2011; Han et al. 2015; Li et al. 2015; Neelgund et al. 2015; Silva et al. 2014; Talib and Hui Fen 2016). This is due to their unique properties that are different from the bulk one. Noble metallic nanoparticles, because of their fascinating optical, chemical, magnetic, catalytic, and electronic properties, have gained considerable scientific and industrial attention (Dong et al. 2016a, b; Francis et al. 2010; Jayaprakash et al. 2014; Samari and Dorostkar 2015; Silva and Unali 2011). Among metal nanoparticles, silver nanoparticles have been the most popular theme of research owing to its novel properties and the possible application (Dong et al. 2014d; Nartop 2016; Nazdar et al. 2016). Most of these properties and the potential application are influenced by particle morphology and size, which are strongly dependent on the experimental conditions (Mochochoko et al. 2013; Mohammadi et al. 2016; Xiong et al. 2013; Zhao et al. 2013). In recent

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years, a wide range of techniques have been developed to prepare silver nanostructures with different size and shape. These include chemical reduction, electrochemical, sonochemical reduction, photochemical, polyol approaches, and biological methods (Augustine et al. 2014; Byeon and Kim 2012; Dong et al. 2014a, 2017; Francis et al. 2010; Mochochoko et al. 2013; Mohammadi et al. 2016; Patra et al. 2016; Rabinal et al. 2013; Xu et al. 2013; Zhao et al. 2013). Among these ways, the most commonly process for the fabrication of silver nanoparticles is chemical reduction in solutions, which usually involves the utilization of stabilizing agent to adjust the solubility, morphology, size, and stability of silver nanoparticles (Dong et al. 2014c; Jiang et al. 2011; Wang et al. 2005; Xiong et al. 2014; Zhao et al. 2010; Zhou et al. 2015). Various capping agents, such as organic thiol compounds, long-chain amines and fatty acids, polymers, and surfactant are generally employed to protect silver colloidal (Dong et al. 2016c; Le et al. 2010a, b; Nartop 2016; Silva et al. 2014; Tang et al. 2010). The protecting agent can be absorbed on the surface of metal nanoparticles, prohibiting them from agglomeration, and sedimentation (Lee et al. 2006; Rao and Trivedi 2005; Shim et al. 2008).

It is worth noting that majority of the techniques regarding the synthesis of silver nanoparticles utilize organic solvent as reaction medium or need some additional substance to let the organic capping agent dissolve in water. Lee et al. (2006) reported one-step reaction method for preparation of silver nanoparticles, using lauric acid as capping agent, silver nitrate as silver precursor,  $\text{NaBH}_4$  as reducing agent, and toluene as solvent. Liu et al. (2010) developed a method for fabrication of silver nanoparticles by adding ammonia to dissolve lauric acid in water.

Despite that a lot of procedures have been exploited to production of silver nanoparticles, the development of a facile synthesis of uniform silver nanoparticles with desired sizes and shapes, by using simple and easy synthetic routes, remains a great challenge (Seoudi et al. 2011; Wang et al. 2014). Sodium laurate is an anionic surfactant, which can be easily dissolved in water. In the present research, we reported an equally facile method for the synthesis of silver nanoparticles using sodium laurate as capping agent and  $\text{N}_2\text{H}_4$  as reducing agent. In this method, sodium laurate can be easily dissolved in water by stirring, preventing silver nanoparticles from agglomeration and oxidation. Furthermore, the reductant  $\text{N}_2\text{H}_4$  is more economical and less toxic than  $\text{NaBH}_4$ . The obtained silver nanoparticles were stable with a narrowly sized distribution.

## 2 Experimental Methods

### 2.1 Materials

Sodium laurate, silver nitrate, and hydrazine hydrate solution (80%) were used in the experiment. All the above chemicals brought from Sinopharm Chemical Reagent Co. Ltd., were of analytical grade and used as received without further purification. The water used in the experiment was doubly distilled water. All glassware used in the laboratory experiments was cleaned with a fresh solution of aqua regia (the 3:1 of  $\text{HCl}/\text{HNO}_3$  solution), washed thoroughly with doubly distilled water, and dried before use.

### 2.2 Synthesis of Silver Nanoparticles

The synthesis of silver nanoparticles was simply obtained by the reduction of silver nitrate with hydrazine in sodium laurate aqueous solution. In a normal synthesis, 0.5 g sodium laurate was added to 20 mL distilled water in a flask. The solution was stirred by a magnetic stirrer at room temperature for about 10 min to obtain a clear solution. At this time, 15 mL silver nitrate solution (containing silver nitrate 0.1 g) was added in. The mixture was stirred for another 10 min. Then 0.5 mL hydrazine hydrate solution was slowly dropped into the above mixture with constant stirring. At the initial stage of the reaction, the color of the mixture was promptly changed into yellow by the addition of hydrazine hydrate solution, indicating the formation of silver nanoparticles. After the hydrazine hydrate solution was added, the reaction was kept for another 1 h to produce a clear colloidal solution containing nano-scale silver particles. Finally, the silver nanoparticles were collected and then separated from the surfactant by centrifugation at 12,000 rpm for 20 min, washed with distilled water and ethanol in sequence, and further dried in vacuum at 40 °C for 12 h.

To check the effects of different parameters, several experiments were carried out by changing the amount of sodium laurate and hydrazine hydrate solution, while other reaction parameters were kept constant.

### 2.3 Characterization

Transmission electron microscope (TEM) performed Tecnai G2 F20 microscope at an acceleration voltage of 200 kV to analyze the size, size distribution, and morphology of prepared silver nanoparticles. SEM analyses were observed on a JSM-6700F scanning electron microscope at an acceleration voltage of 200 kV. The TEM samples were obtained by dropping small amount of nano silver particle solution onto carbon-coated copper grids and

evaporating the solvent (ethanol) at room temperature. The silver particle ethanol solution was prepared by mixing the silver particles and ethanol by the help of ultrasonic agitation. The particle sizes of silver nanoparticles were determined statistically by image analysis.

Synthesis of silver nanoparticles by reducing silver ion in sodium laurate solution can be clearly discovered by UV–Vis spectroscopy. The absorption spectra of silver nanoparticle colloids obtained at different reaction conditions were operated at wavelengths ranging from 200 to 750 nm at a resolution of 0.5 nm by using a Specord S 600 UV–Vis spectrophotometer. Doubly distilled water was used as a blank solution.

Crystalline metallic pattern of silver nanoparticles was characterized on an X'Pert PPO X-ray diffractometer with  $\text{CuK}\alpha$  radiation of wavelength of  $\lambda = 1.5406 \text{ \AA}$  in the  $2\theta$  range from  $20^\circ$  to  $90^\circ$  with a scanning rate of  $0.05^\circ/\text{s}$  at room temperature. The sample for XRD analysis was gathered by centrifuging reaction medium at 12,000 rpm for 20 min and dried in vacuum at  $40^\circ\text{C}$  for 12 h.

The Fourier-transform infrared (FT-IR) analysis was operated on a Tensor 27 spectrophotometer to characterize the surface structure of silver nanoparticles in the wavelength range of  $4000\text{--}400 \text{ cm}^{-1}$ . The synthesized silver nanoparticles were blended with KBr powder and pressed into a pellet for measurement. Background correction was made on the basis of the spectrum from the reference pure KBr powder which was pressed into a pellet.

### 3 Results and Discussion

#### 3.1 Preparation and Characterization of Silver Nanoparticles

Fatty acid-protected nano silver colloids can be obtained expediently by chemical reduction method in aqueous solution. In this study, nano silver particles were prepared by reduction of silver ion with hydrazine using long-chain sodium laurate as stabilizing agent. It is usually acknowledged that silver nanoparticles exhibit yellow, red or brown color in water. The characteristic color changes are attributed to the excitation of the surface plasmon resonance in the metal nano colloids. After the addition of hydrazine solution, the color of reaction mixture varied from milky white to yellow, pale red then wine red, and finally brown or black, demonstrating the formation of silver nanoparticles. The photos of the silver nanoparticles before and after synthesis are shown in Fig. 1. It is believed that the color variations are due to the formation of nano silver particles in the solution: silver ions are first coordinated with sodium laurate, which are then reduced to zero-valent silver.

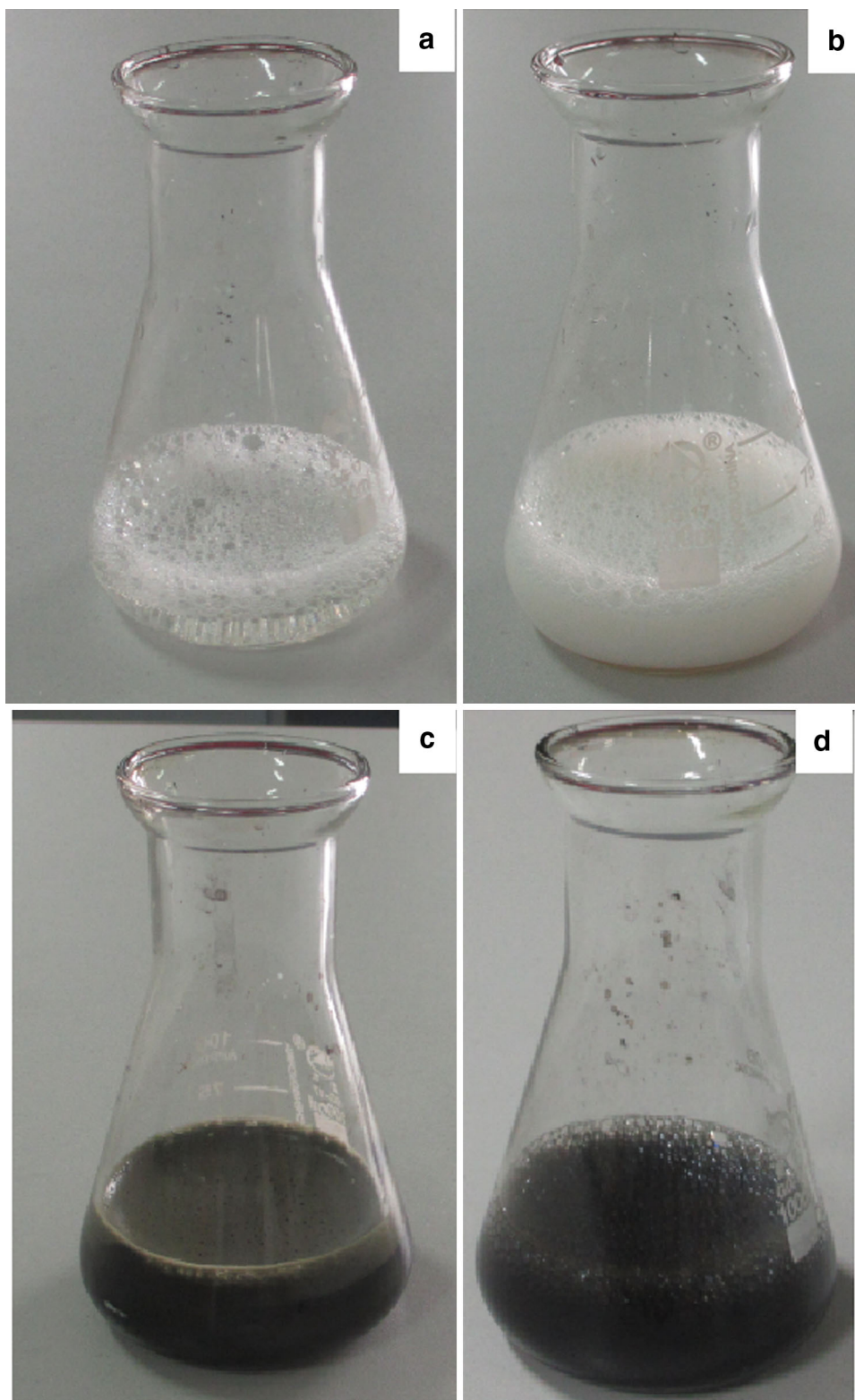
The formation of silver nanoparticles was also confirmed by UV–Vis absorption spectra. The typical UV–Vis spectral analysis of the prepared silver nanoparticles is shown in Fig. 2. It is generally recognized that UV–Vis absorbance spectroscopy could be used to study the size and morphology of metal nanoparticles in aqueous suspensions, owing to that the peak positions and shapes are sensitive to particle size and shape. As shown in Fig. 2, an optical absorption band with a maximum at 403 nm was observed, illustrating the presence of spherical or roughly spherical silver nanoparticles in the obtained colloid solution. The frequency and width of the plasmon absorption lie on the particle size and shape, as well as the dielectric constant of the surrounding medium. According to the Mie theory, if the absorption peak is symmetric, the size distribution of nano particles in the colloid solution is narrow. Contrarily, if the peak is asymmetric, it demonstrated that colloid is polydispersed. It also can be seen in Fig. 2 that the peak is very symmetric in shape, showing the obtained silver nanoparticles are monodispersed.

The shape and morphology of metal nanoparticles were also observed by SEM and TEM. Figure 3 shows the SEM image of the synthesized silver nanoparticles. It is clear that the silver nanoparticles are homogeneous and spherical in shape. The typical TEM image and corresponding size distribution of the as-prepared silver nanoparticles are illustrated in Fig. 4. In Fig. 4a, the TEM image reveals that the nano particles are small and monodispersed. The prepared silver nanoparticles are spherical in shape, with a narrow distribution from 2 to 8 nm, as shown in Fig. 4b, which may attributed to the anionic surfactant sodium laurate adsorbed on the surface of the silver nanoparticles.

The XRD analysis was used to examine the crystalline nature of silver nanoparticles. The typical XRD pattern of sodium laurate protected nano silver particles, as illustrated in Fig. 5, demonstrates five characteristic diffraction peaks  $2\theta$  at  $38.2^\circ$ ,  $44.5^\circ$ ,  $64.7^\circ$ ,  $77.5^\circ$ , and  $81.8^\circ$  for the marked indices of (111), (200), (220), (311), and (222), respectively. These data indicate a face-centered cubic (FCC) crystal structure of metallic silver nanoparticles. The intense reflection at (111) is relatively higher than other peaks, showing the growth direction of nanocrystals. These information manifests that the synthesized silver nanoparticles may be likely be enriched in (111) crystal facet. No diffraction peaks pertaining to  $\text{Ag}_2\text{O}$  or other impurities are observed in Fig. 5, suggesting that the sodium laurate adsorbs on surface of silver nanoparticles and prevents them from oxidizing. In addition, some low-intensity peaks were also detected. These may be due to some sodium laurate involved in the process as stabilizing agents and adsorbed on the surface of silver nanoparticles.

FT-IR measurements were conducted to confirm the existence of sodium laurate molecules on the surfaces of

**Fig. 1** The photos of **a** sodium laurate solution, **b** silver nitrate and sodium laurate mixture, **c** silver nanoparticles synthesized at 10 min, **d** synthesized at 1 h



silver nanoparticles. The FT-IR spectra of pure sodium laurate is illustrated in Fig. 6a. The band centered between 3450 and 3500  $\text{cm}^{-1}$  corresponds to stretching vibrations of hydroxyl group ( $-\text{OH}$ ). Two absorption peaks between

2750 and 3000  $\text{cm}^{-1}$  are attributed the asymmetric and symmetric  $\text{CH}_2$  stretching. The peaks around 1500  $\text{cm}^{-1}$  are due to the propyl stretching vibrations and C–H bending vibration. In comparison with Fig. 6a, the peaks in

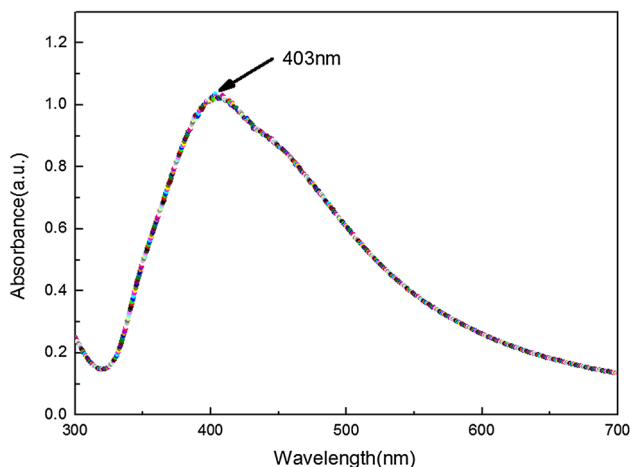


Fig. 2 The typical UV-Vis spectra of silver nanoparticles

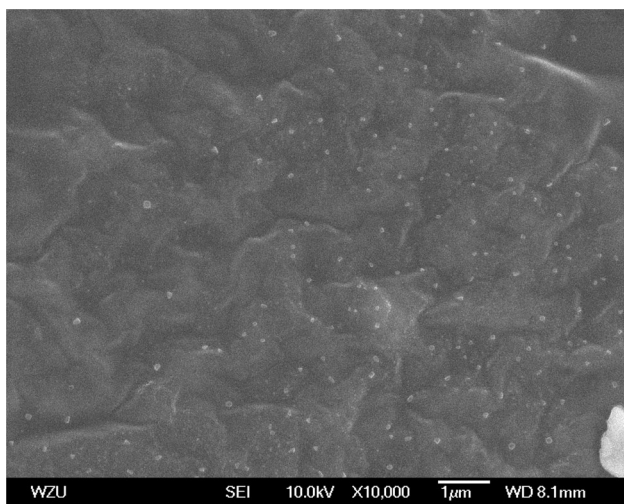


Fig. 3 The typical SEM of the silver nanoparticles

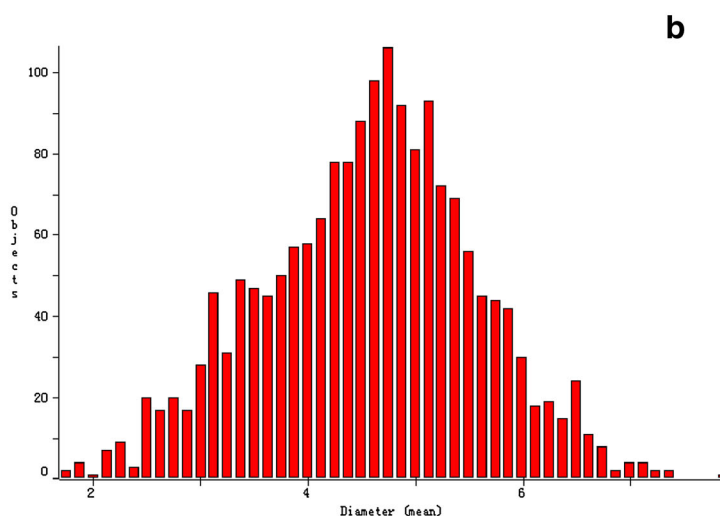
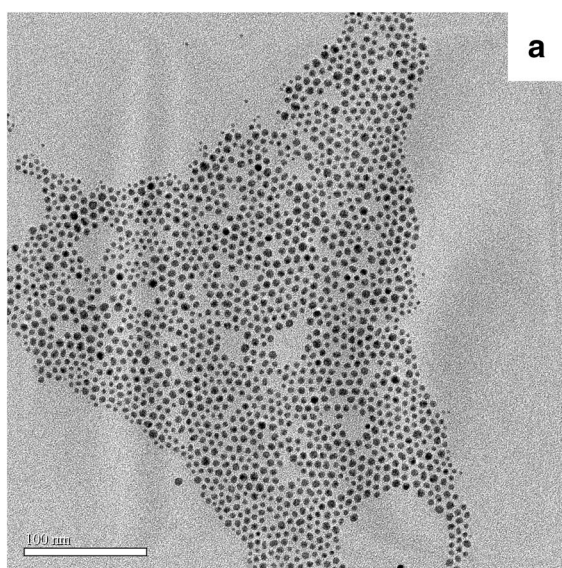
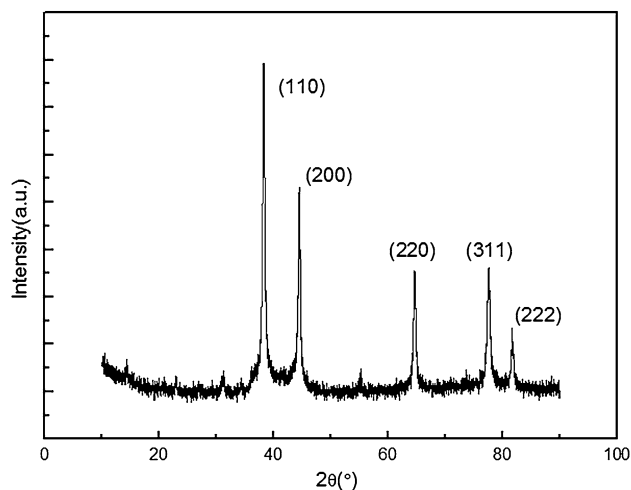


Fig. 4 The typical a TEM image and b size-distribution histogram of the silver nanoparticles

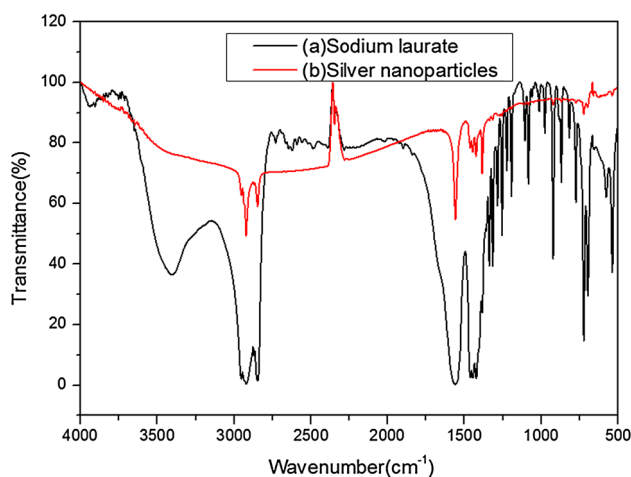
Fig. 6b are weak and slight shift, indicating a chemical bond between the silver atom and sodium laurate molecules is formed. By this way, the sodium laurate molecules are absorbed on the surface of silver nanoparticles, preventing the nano silver particles from oxidation and agglomeration.

### 3.2 Effect of Amounts of Sodium Laurate on Silver Nanoparticles

In this reaction, sodium laurate was used as stabilizer due to its nice solubility. Silver nanoparticles were induced by reduction of silver ions using hydrazine in sodium laurate solution. It is apparently that the size, size distribution, and shape of the silver nanoparticles are affected by the amounts and nature of the capping agent. In order to investigate the effect of stabilizer on silver nanoparticles, various quantities of sodium laurate (0.1, 0.5, and 1.0 g) were used as capping agent to synthesize silver colloids. The UV-Vis absorbance spectra of silver nanoparticles synthesized at different amounts of sodium laurate when the other parameters were kept constant are shown in Fig. 7. Silver colloid synthesized at lowest amount of sodium laurate shows a SPR peak at 406.5 nm with a long trail, indicating a broad size distribution. As the amount increased to 0.5 g, the SPR peak was blue shift from 406.5 to 403 nm, suggesting the decrease the size of silver nanoparticles. However, when the amount of sodium laurate was further increased to 1.0 g, there is a slight red shift from 403 to 404 nm, which may be due to the increase the size of silver particles. This phenomenon can be explained that when too much sodium laurate was employed, the concentration of silver ions was decreased, which may lead



**Fig. 5** XRD pattern of silver nanoparticles

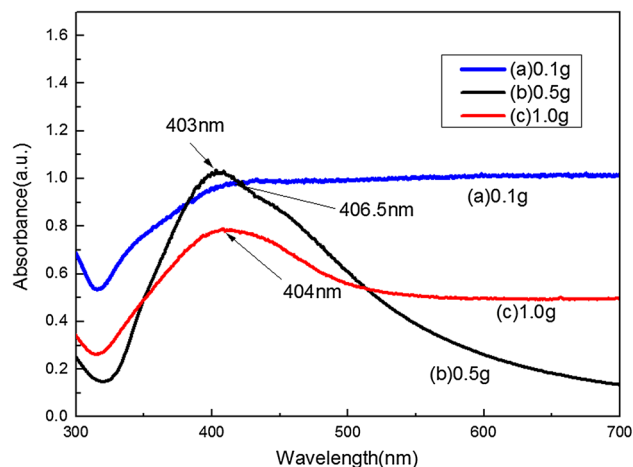


**Fig. 6** FT-IR spectra of **a** sodium laurate and **b** silver nanoparticles

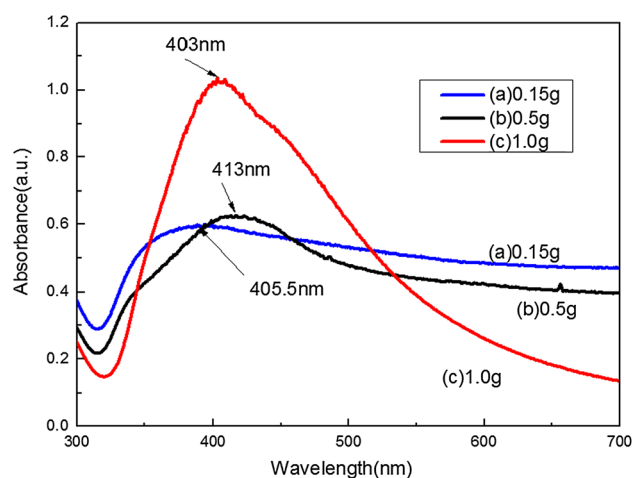
to a slow reaction rate (slow nucleation and slow growth of nuclei). Usually, fast nucleation and slow growth of nuclei are attributed to small and uniform particles. However, slow nucleation and slow growth of nuclei may lead to relative big particles. From the above, it can be concluded that the amounts of sodium laurate play an important role in the formation of silver nanoparticles. The appropriate amount for the synthesis of silver nanoparticles is 0.5 g.

### 3.3 Effect of Amounts of Hydrazine on Silver Nanoparticles

Effect of reducing agent on the metallic nanoparticles has been reported widely. In the present research, hydrazine was used as reducing agents to prepare silver nanoparticles. Hydrazine is less toxic and much cheaper than sodium borohydride. Hydrazine can not only reduce the silver ion, but also fabricate nitrogen during the synthesis process,



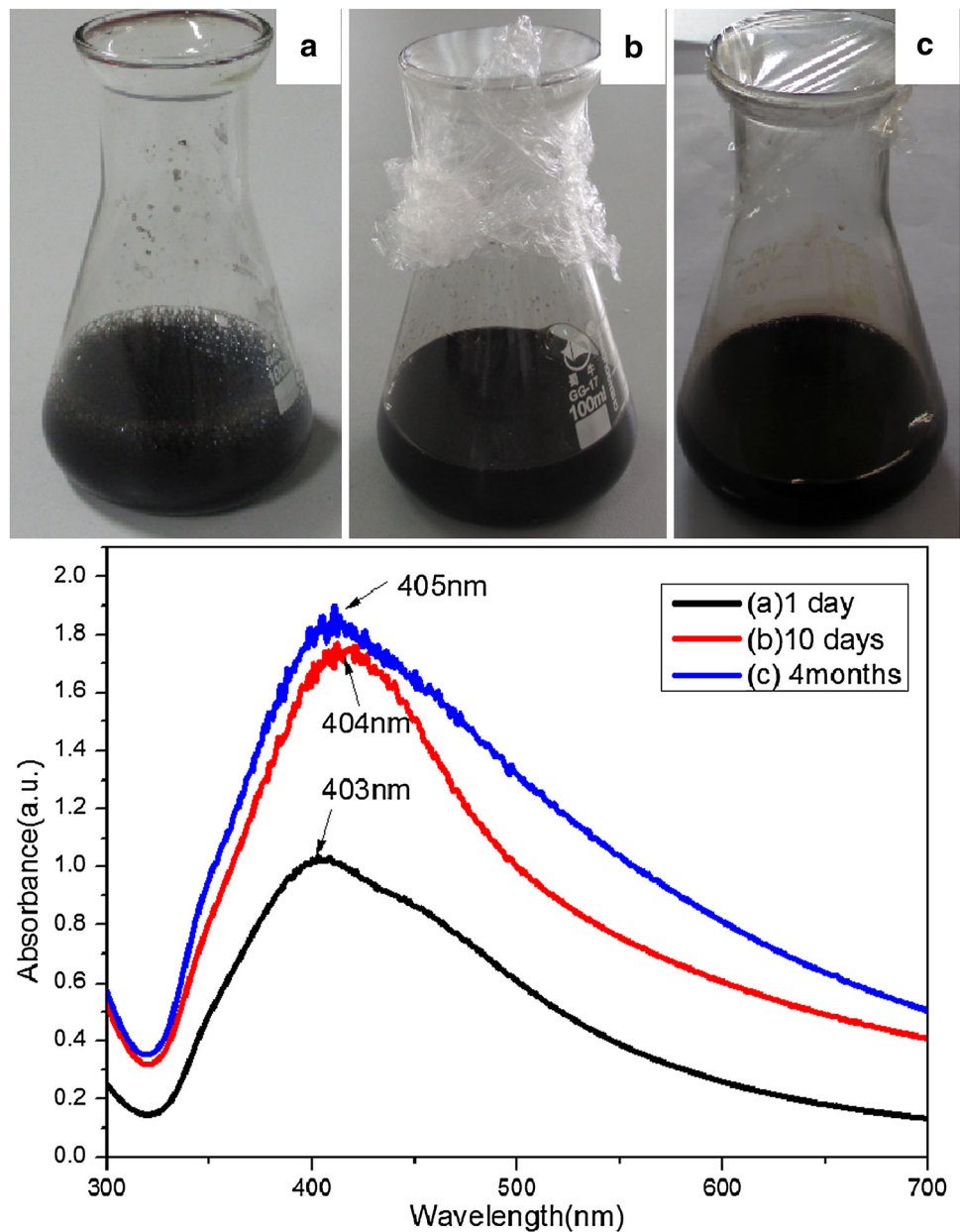
**Fig. 7** Effects of sodium laurate amount on UV-Vis spectra of as-synthesized nano-silver colloids



**Fig. 8** Effects of hydrazine amount on UV-Vis spectra of as-synthesized nano-silver colloids

which may prevent the silver nanoparticles from oxidation (Dong et al. 2014a). To study the effect of reducing agent on silver nanoparticles, various amounts of hydrazine were utilized as reducing agent to prepare silver nanoparticles, when other parameters were kept constant, as demonstrated in Fig. 8. Silver nanoparticles obtained at lowest amount of hydrazine exhibits a SPR peak at 405.5 nm with a long trail, suggesting a broad size distribution. As the amount increased to 0.5 g, the SPR peak was small red shift from 405.5 to 413 nm, revealing the increase the size of silver nanoparticles. However, the shape of the spectra is more symmetric, suggesting that the obtained silver nanoparticles were ranged in a relative small size distribution. When the amount of hydrazine was further increased to 1.0 g, the absorbance spectra were blue shift from 413 to 403 nm, with a symmetric shape, indicating smaller size with

**Fig. 9** The photos and UV–Vis spectra of silver nanoparticles **a** before and **b** after 10 days, **c** 4 months of storage



narrowly distribution silver nanoparticles are obtained. When the weight of hydrazine was increased, the reduction rate of silver ion was improved, and more nuclei were formed during the nucleation step, which lead to generation of smaller particles with narrowly distribution. Hence, the hydrazine also play virtue role in the synthesis of silver nanoparticles.

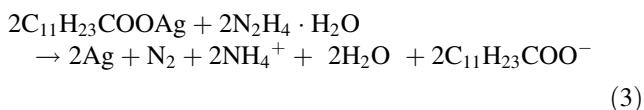
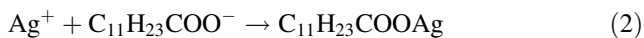
### 3.4 The Stability of Silver Nanoparticles

Obviously, the application depends on the stability of silver nanoparticles. Their application will be limited if the silver nanoparticles aggregate and precipitate in a short time. The

photos and UV–Vis absorbance spectra of silver colloids before and after 10 days, 4 months' storage are shown in Fig. 9. It is can be seen that no sign of sedimentation was found in the sodium laurate-protected nano silver particles colloid after 10 days, or 4 months storage. Furthermore, from the UV–Vis absorbance spectra of silver nanoparticles before and after 10 days, 4 months storage, as can be seen, a slight red shift from 403 to 404 and 405 nm was observed. These phenomena exhibit that the gained nano silver particle colloid is highly stable due to the excellent stabilizing effect of sodium laurate.

### 3.5 Possible Mechanism

The above studies illustrate that small, monodispersed and stable nano silver particle colloid can be prepared using hydrazine hydrate as the reducing agent and sodium laurate as the stabilizing agent in aqueous solution. The reactions are as follows:



### 4 Conclusions

In summary, stable and small sized sodium laurate stabilized silver nanoparticles were prepared by reducing silver nitrate using hydrazine as the reducing agent and sodium laurate as the capping agent. The average size of prepared silver nanoparticles was about 6 nm with a narrow distribution from 2 to 8 nm. The synthesis process present in this study was facile without adding any other chemicals to dissolve capping agent. Furthermore, the reaction can be operated at room temperature without heating. Sodium laurate not only prevents the agglomeration but also protects the silver nanoparticles from the oxidation. The sodium laurate and hydrazine play important roles in the formation of small size particles. The prepared nano silver particle colloids were highly stable and did not show any sign of sedimentation even after 4 months storage. The method can be extended to other noble metals, such as Au, Pt, and Pd.

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