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Facile and one-step liquid phase synthesis of uniform silver nanoparticles reduction by ethylene glycol

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ABSTRACT

A facile, simple to synthesis uniform and monodisperse, well size distribution of silver nanoparticle with 20–30 nm was reported. The silver nanoparticles were successfully synthesis by liquid phase method of EG (Ethylene glycol) in the presence of PVP (Polyvinyl pyrrolidone). The properties of uniform silver nanoparticles were investigated by HRTEM (High resolution transmission electron microscope), SEM (Scanning electron microscope), UV–vis (UV–visible spectroscopy), FT-IR (Fourier transform infrared spectra) and XRD (X-ray diffraction). The effects of different reaction conditions on the size distribution of Ag NPs were studied. The XRD peaks are consistent with the data on the standard card, corresponding to the face-centered cubic Ag plans of (111), (200), (220), (311) and (222). Besides, the d-spacing of silver nanoparticle is d = 0.223 nm by measuring the distance between the planes, which is in agreement with the value of the (111) lattice spacing of FCC (face centred cubic) silver. The research results would be useful to assess the facile synthesis of uniform and monodisperse silver nanoparticles.

1. Introduction

Silver nanoparticles have many excellent properties of low melting point, low cost, high efficiency, high stability, conductivity miniaturization and low pollution, which are widely used to flexible electrical, biomedical and optical applications [1–11]. In recent years, silver nanoparticles have attracted intensive research interest due to its enhancement of nanoscale morphology [12–17]. The fabrication of flexible circuits by silver nanoparticles is a potential area. Besides, the more flexible electrical applications were expanded such as micro-sensor, OLED, TFT, RFID [18–21]. The size distribution of silver nanoparticles is the key factor for the fabrication process.

To achieve stable and high-quality silver nanoparticles, many methods of synthetic have been explored, which mainly include three different ways based gas-condensation, mechanical alloying and solution-chemistry, respectively. The synthesis of silver nanoparticles by gas-condensation-based is characterized by high purity of the product, but the drawback is that the large equipment investment and the high energy consumption [22,23]. Due to the pollution of working atmosphere with ball mill and the size instability, mechanical attrition has greatly reduced the academic and industrial value of this method. As for the solution-chemistry synthesis method, it can be roughly divided into liquid phase synthesis method [24], Sol-gel [25], Hydrothermal [26], spray Pyrolysis [27] and others. Liquid phase synthesis method is one of the most effective methods for preparing nanoparticles, which is beneficial to provide molecular chemical reaction, composition and structure

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design [28]. To prepare different sizes and morphology of silver nanoparticles, liquid phase synthesis method control the nucleation diffusion growth by adjusting the reaction temperature, time, reactant concentration, the amount of dispersant and others [29,30]. Taking the reduction rate and production efficiency into account, polyol method of synthesis of uniform silver nanoparticles reduction is desired. Hence, liquid phase synthesis reaction is mild and easy to achieve uniform and stable nanoparticles.

Skrabalak S E et al. have facile synthesis of Ag nano-cubes with 45 nm in diameter by EG in the presence of PVP, which indicate that the polyol has a well behavior in the reaction of Ag nanoparticles and the PVP can serve as a good stabilizer [31,32]. Y Xia et al. [33,34] have synthesis uniform nano-cubic silver with 20 nm in diameter while EG restore the silver nitrate in the PVP protection. The ratio of reaction humidity, reactant concentration and stabilizer are the main factors which affect the size and morphology of silver nanoparticles in the reaction system. The research team has prepared different morphology of the silver nanoparticles. It is proved that the higher temperature (160 °C) is a key factor in the controllable synthesis. D Chen et al. [35] have synthesis 2–20 nm silver nanoparticles by EG with the PVP sever as the stabilizer. However, the studies only conduct the reaction at low temperatures in 0 °C, 30 °C, 60 °C. Besides, the yield is low and the purity is not enough. The reaction of ethylene glycol under high temperature reaction requires further investigation in order to explore the facile synthesis of uniform and monodisperse silver nanoparticles.

In this paper, the efficient and facile liquid phase synthesis is proposed to synthesis silver nanoparticles and the influence of three factors, silver nitrate concentration, silver nitrate and PVP mass ratio and reaction temperature on the synthesis experiment are investigated. We analysis the effects of different reaction conditions on the size distribution of Ag NPs by UV–vis spectroscopy, FT-IR, SEM and TEM studies. The synthesis achieves approach to uniform and well size distribution of Ag NPs within 20–30 nm.

2. Experiment methods

2.1. Materials

All chemical reagents are commercially available without further purification. EG (Ethylene glycol, 99.8%), silver nitrate ($AgNO_3$, 99.8%), PVP ($M_W \approx 30000$) were purchased from Sinopharm Chemical Reagent Co., Ltd.

2.2. Synthesis of Ag NPs

Ag NPs were produced by the reduction of silver nitrate by the polyol in the presence of PVP. In a typical synthesis procedure, 35 mL EG was heated at 160 °C in an oil bath with continuous mechanical string at 800 rpm for 1 h. 15 mL freshly EG solution (dissolved 0.1 M silver nitrate and 1 g PVP) were slowly dripping into the stirring solution at the rate of 1 mL min^{-1}. After the reduction reaction was complete, the mixture cooled down to room temperature. Finally, the Ag NPs was separated from the resulting solution by three centrifuging processes at 8000 rpm with anhydrous ethanol.

In order to study the silver nitrate concentration on synthesis of Ag NPs, silver nitrate samples of different concentration were prepared. The silver ion concentration was varied from 0.05 M, 0.1 M, 0.15 M, 0.20 M and 0.25 M with unchanged reaction parameters. To study the effect of silver nitrate and PVP mass ratio on synthesis of Ag NPs, samples with various silver nitrate and PVP mass ratio were prepared. The silver nitrate and PVP mass ratio was varied from 0.25:1, 0.5:1, 1:1, 2:1. To study the effect of the reaction temperature on synthesis of Ag NPs, five comparative trials at 100 °C, 120 °C, 140 °C, 160 °C and 180 °C under same reaction conditions were conducted.

2.3. Characterization

HRTEM observations are performed by Tecnai G2 F30 microscope at the acceleration voltage of 300 kV. The TEM samples were prepared by few drop of ethanol solution containing silver nanoparticles on the carbon coated copper grids and evaporating the solvent (ethanol) at room temperature. The SEM images are performed by scanning electron microscopy (SEM, Nova NanoSEM 450, FEI, Japan). UV–vis is to prove the presence of Ag NPs. The samples are placed in a 1 cm quartz cuvette over the wavelength of 200–800 at a resolution of 1 nm. The FT-IR is performed by VERTEX 70 (Bruker, Germany). In order to measure the crystallinity and phase composition of the Ag NPs, the X'Pert PRO X-ray diffractometer were used with CuKα radiation of wavelength of λ = 1.5406 Å in the 2θ range of 20°to 90° with a scanning rate of 10°/min.

3. Results and discussion

3.1. Preparation and characterization of Ag NPs

In this reaction system, PVP was used as the surfactant, EG (Ethylene glycol) as solvent and reducing agent, and silver nitrate was added to produce nano silver particles. The PVP can not only prevent the agglomeration between Ag NPs but also with silver ions to form a clathrate, reducing the chemical potential, making silver ions more easily reduced by EG [12,36,37]. When the system is heated, the reducing agent directly reduce silver ions to metallic silver nanoparticles with PVP absorbed onthe surface. The formation mechanism is displayed as Fig. 1.