



# Synthesis of silver nanoparticle at a gas/liquid interface in the presence of silver seeds and its application for electrochemical sensing



Ziying Yang, Chengcheng Qi, Xiaohui Zheng, Jianbin Zheng\*

Institute of Analytical Science, Shaanxi Provincial Key Laboratory of Electroanalytical Chemistry, Northwest University, Xi'an, Shaanxi 710069, China

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## ABSTRACT

Silver nanoparticles were synthesized by reducing  $[\text{Ag}(\text{NH}_3)_2]^+$  at a gas/liquid interface in the presence of silver seeds. Transmission electron microscopy (TEM) observations reveal that the size of these silver nanoparticles is around 35–40 nm with the average particle size of 37 nm. The silver nanoparticles were applied for the electrochemical sensor and electrochemical investigations indicate that the nanoparticles possess an excellent performance toward  $\text{H}_2\text{O}_2$ . The linear range is estimated to be from 5.0  $\mu\text{M}$  to 4.0 mM with a low detection limit of 1.7  $\mu\text{M}$ , a sensitivity of 166.7  $\mu\text{A mM}^{-1} \text{cm}^{-2}$  and a response time of 3 s. Additionally, the sensor exhibits good anti-interference.

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## 1. Introduction

The determination of  $\text{H}_2\text{O}_2$  is very important owing to its widespread applications in food, pharmaceutical, clinical and environmental analyses [1–3]. Nowadays, many techniques have been employed in the determination of  $\text{H}_2\text{O}_2$ , such as chromatography [4], chemiluminescence [5] and electrochemistry [6–8]. Among these methods, electrochemistry technique based on a simple and low cost electrode has been extensively applied for accurate determination of  $\text{H}_2\text{O}_2$  [9,10]. In comparison with enzyme-modified electrodes, non-enzymatic sensors have some advantages, including low cost, stability and easy to operate [11,12]. Therefore, enormous research effort has been paid on the non-enzymatic sensors. For example, Shang [13] reported that the non-enzymatic  $\text{H}_2\text{O}_2$  sensor based on nitrogen-doped graphene hollow AuPd nanoparticle hybrid films exhibited sound sensitivity and selectivity toward the detection of  $\text{H}_2\text{O}_2$ , which should be attributed to the high surface area and the synergistic effect between Au and Pd. Sun [14] fabricated a non-enzymatic  $\text{H}_2\text{O}_2$  sensor based on dumbbell-like PtPd– $\text{Fe}_3\text{O}_4$ NPs and the sensor exhibited excellent catalytic properties due to the interfacial interactions between two different nanostructures. In addition, many other metal nanoparticles, such as CuNPs [15], CoNPs [16] and  $\text{MnO}_2$  [17] were also employed to construct  $\text{H}_2\text{O}_2$  sensor.

As we know, the presence of nanoparticles (NPs) can improve the performance of sensors, due to their large specific surface areas and unusual structural features. Among all the nanomaterials, it is found that silver nanoparticles (AgNPs) not only exhibit the highest conductivity but also possess excellent catalytic properties. Compton and his coworkers have studied the electrochemical behavior of AgNPs towards  $\text{H}_2\text{O}_2$  reduction [18,19]. Bai fabricated a novel non-enzymatic sensor based on Ag/MnOOH nanocomposites for the detection of  $\text{H}_2\text{O}_2$  [20]. Therefore, synthetic techniques for the fabrication of AgNPs keep on being of enormous current research attention. Nowadays, physical, electrochemical and chemical methods have been employed to synthesize AgNPs [21]. For example, Lin synthesized AgNPs by the photoreduction of silver ions under UV irradiation [22]. Electrodeposition of AgNPs on DNA was also carried out in previous research [23]. Bai synthesized Ag–MnOOH–GO nanocomposites at a gas/liquid interface based on traditional silver mirror reaction [24]. Among these methods, the chemical reaction that happens at a gas/liquid interface is a special method different from others because the gas/liquid interface can control the progress of the reduction reaction in a mild way by adjusting the reaction temperature, the reaction time, the gas pressures, and so on. Unfortunately, it is difficult to prepare silver nanoparticles with a fairly narrow nanoparticle size distribution, due to the poor balance between the nucleation and growth processes, which greatly hinders them to be applied in real life. Therefore, it is necessary to carry out some studies to improve the balance between the nucleation and growth processes.

\* Corresponding author. Tel.: +86 29 88302077 (L); fax: +86 29 88303448 (O).  
E-mail address: [zhengjb@nwu.edu.cn](mailto:zhengjb@nwu.edu.cn) (J. Zheng).

In this paper, we present a facile preparation route to synthesize AgNPs by reducing  $[\text{Ag}(\text{NH}_3)_2]^+$  at a gas/liquid interface in the presence of silver seeds. Then we fabricate a non-enzymatic  $\text{H}_2\text{O}_2$  sensor based on AgNPs and investigate the electrochemical performance of the sensor toward  $\text{H}_2\text{O}_2$ .

## 2. Experimental

### 2.1. Reagents and materials

Silver nitrate ( $\text{AgNO}_3$ ) and sodium citrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ ) were purchased from Xi'an Chemical Reagent (Xi'an, China). Ammonia solution ( $\text{NH}_3$ , 25%), sodium hydroxide ( $\text{NaOH}$ ) and hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 30%, v/v aqueous solution) were received from Tianjin Tianli Chemistry Reagent Co., Ltd (Tianjin, China). Acetaldehyde ( $\text{C}_2\text{H}_4\text{O}$ , 40%) and sodium borohydride ( $\text{NaBH}_4$ , 98%) were obtained from Shanghai Yuanju Biotechnology Co., Ltd (Shanghai, China). 0.1 M phosphate buffered saline (PBS, pH=7.2) was used as the supporting electrolyte. Reagents and chemicals were of analytical reagent grade.

### 2.2. Apparatus

Transmission electron microscopic (TEM) images were carried out by Tecnai  $\text{G}^2$  F20 S-TWIN (FEI, USA). UV–visible spectra were scanned by Cary 50 probe UV–visible spectrophotometer (Varian, Australia). Electrochemical measurements were carried out in a conventional three-electrode electroanalysis system controlled by EC 550 electrochemical workstation (Gaoss Union Technology Co., Ltd., Wuhan, China) and CHI 660 electrochemical workstation (Shanghai CH Instrument Co. Ltd., China). A conventional three-electrode cell was used, including a glassy carbon electrode (GCE, geometric area=0.07  $\text{cm}^2$ ) as the working electrode, an Ag/AgCl (3 M KCl) electrode as the reference electrode and platinum foil as the counter electrode. All potentials given in this work were referred to the Ag/AgCl electrode.

### 2.3. Synthesis of AgNPs

5 mL of 10 mM freshly prepared  $[\text{Ag}(\text{NH}_3)_2]^+$  solution was diluted to 46 mL by doubly distilled water. Then, 4 mL of 38.8 mM sodium acetate was added to this aqueous solution and this aqueous solution will become light yellow when 10  $\mu\text{L}$  of 5 mM  $\text{NaBH}_4$  aqueous solution was added under vigorous stirring. At last, the mixture and another beaker that had 20 mL of  $\text{CH}_3\text{CHO}$  solution were put in a closed container. The volatilized  $\text{CH}_3\text{CHO}$  gas can be used as reductant and the reaction was performed for 3 h under room temperature with continuous magnetic stirring. After which, AgNPs were separated from the solution by centrifugation at 8000 rpm for 5 min. The collected products of solid state were washed for three times by doubly distilled water and ethanol. Then, the obtained powder was dried in a vacuum oven at 40°C for 12 h.

### 2.4. Electrode modification

The glassy carbon electrode (GCE) was prepared by a simple casting method. Prior to use, the GCE was polished with 1.0 and 0.3  $\mu\text{m}$  alumina powder to obtain mirror like surface, respectively, and rinsed with doubly distilled water, followed by sonication in ethanol solution and doubly distilled water successively. Then, the GCE was allowed to dry in a stream of nitrogen. 1.0 mg of AgNPs was dispersed into chitosan (1 mL, 0.5%) and sonicated for 5 min. The suspension (6  $\mu\text{L}$ ) was dropped onto the pre-polished mirror like surface of GCE and then dried in air at room temperature.

## 3. Results and discussion

### 3.1. Characterizations of AgNPs

Here, we present a facile preparation route to synthesize AgNPs by reducing  $[\text{Ag}(\text{NH}_3)_2]^+$  at a gas/liquid interface in the presence of silver seeds as illustrated in Fig. 1. The seed-mediated growth method can improve the balance between the nucleation and growth, which is beneficial to prepare AgNPs with a fairly narrow nanoparticle size distribution [25,26]. Citrate-stabilized Ag seeds were first prepared from the  $\text{NaBH}_4$  reduction of  $[\text{Ag}(\text{NH}_3)_2]^+$  [27]. Then, the volatilized  $\text{CH}_3\text{CHO}$  gas can be used as reductant, resulting in the growth only occurring on the Ag seeds in a mild way. AgNPs with a fairly narrow nanoparticle size distribution were obtained.

The formation of AgNPs was identified by UV–vis absorption spectroscopy. As shown in Fig. 2A, the spectrum of AgNPs dispersed in water has an obvious absorption peak at about 420 nm, which may be ascribed to the excitation of surface plasmon of AgNPs [28].

Fig. 2B–D shows the typical TEM images of AgNPs under different magnifications. As shown in Fig. 2D, the average particle size of the prepared nanostructures is about 37 nm. From Fig. 2B and C, it can be seen clearly that the size of these silver nanoparticles is around 35–40 nm and the shape control over the spherical AgNPs is improved. These can be contributed to the presence of silver seeds which improves balance between nucleation and growth stages [26]. In addition, the gas/liquid interface and the use of the precursor  $[\text{Ag}(\text{NH}_3)_2]^+$ , other than  $\text{Ag}^+$ , are also essential to homogeneous particle size. Because the gas/liquid interface and the use of the precursor  $[\text{Ag}(\text{NH}_3)_2]^+$  can control the progress of the reduction reaction in a mild way, resulting in the growth only occurring on the seeds and avoiding the self-nucleation. With this understanding, the balance between the nucleation and growth can be improved by the stepwise reduction method, in which the fast nucleation stage is carried out and the following growth stage is slowed down. Although AgNPs we synthesized have almost homogeneous particle size, it is seen clearly by the TEM patterns that AgNPs aggregate in some extent, which can be contributed to the high surface energy and large number of dangling bonds [29,30]. That, of course, the detail influences of other factors on the aggregation will be studied carefully in our subsequent work.

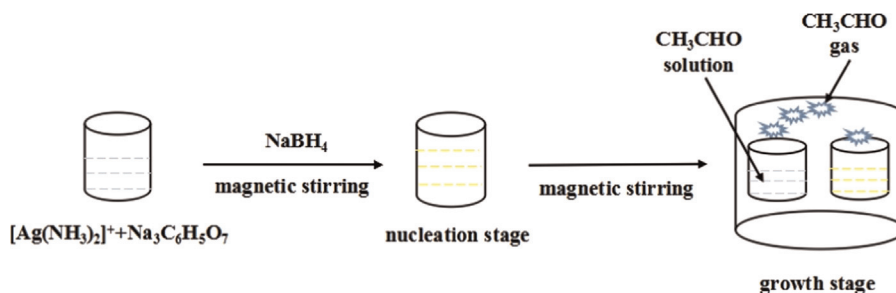


Fig. 1. Experimental procedure of synthesis of silver nanoparticles.

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