



Role of the anions in the hydrothermally formed silver nanowires and their antibacterial property



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ABSTRACT

Uniform silver nanowires were synthesized via a green and simple hydrothermal method through fine tailoring the anions in the precursors. The results indicate that the anions added to the reaction precursors played a key role in determining the morphology and composition of the final products. A new possible mechanism is proposed that in the hydrothermal process Ag_xA_y act as another oxidant except for Ag^+ ions. The obtained silver nanowires showed excellent and long-lasting antibacterial property.

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1. Introduction

The rapid development of nanotechnology enables the successful control of the dimensionality of silver to nano-scale level, which exhibits novel properties and superior performances that largely differ from that of bulk materials. For instance, two typical nanosilvers of silver nanoparticles (AgNPs) and silver nanowires (AgNWs) have attracted a great many of attentions in both academic and industrial fields, because of their unique electric, optical, thermal, chemical and physical [1–10] properties, as well as their excellent antibacterial property [11–13].

To solve the problem of oxidation and aggregation in practical use as antibacterial reagent, AgNPs have been incorporated or loaded to other support materials, such as mesoporous TiO_2 [14], carbon nanotubes [15,16], activated carbon fibers [17,18], glass fibers [19,20], ceramic [21], silicon [22,23] and papers [24,25]. Comparatively, AgNWs have advantages over AgNPs in practical applications by affording multiple binding points with supporting materials [13]. Therefore, much effort has been undertaken to the synthesis of AgNWs.

Hydrothermal method has proven to be effective and facile for the preparation of AgNWs, as it can not only avoid the post-treatment necessary for template assisted method [26–29], but also omit the use of harsh chemicals applied in other synthesis methods

[30,31]. As has been reported, experimental parameters like temperature, reaction time, pH and concentration of the reactant in hydrothermal process are important factors that affect the final products [32–34]. Tetsumoto et al. [33] found that in the AgNO_3 , NaCl and glucose system, the AgNWs could be easily generated at temperature of 150 °C. And a necessary reaction time of 24 h was required to guarantee the gradual transformation from Ag^+ to AgNWs. Yang et al. [34] reported the hydrothermal synthesis of AgNWs in the AgNO_3 and $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ system. They found that the higher the citrate concentration was, the faster the reaction rate would be. But once the citrate concentration exceeds certain limit, it would induce the formation of quasi-spherical particles rather than AgNWs. An increase in the pH of the reaction solution from 7.1 to 10.1 generated a higher percentage of spherical particles, because the nucleation and growth rates as well as the ratio of deprotonated citrate ions had changed. Jiu et al. [35] synthesized AgNWs using hydrothermal method by adding trace salts of NaCl, FeCl_3 and Na_2CO_3 . The shape and yield of AgNWs significantly depended on the nature and concentration of these additives. The temperature was responsible for the dissolution and the formation of Ag_xA_y (A is the anions in the added salts) to modify and improve the growth of AgNWs. The solubility of Ag_xA_y was important for the formation of AgNWs, which was sensitive to temperature. In another report [36], different exotic agents of NaOH, KBr and NaCl were added into the reaction systems. They found that different morphologies of the products were due to the different release rates of Ag^+ ions from these colloids to the solutions.

According to Jiu and Chen [35,36], the exotic agents of salts added to the reaction systems always playing pivotal role in the

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formation of the final products by forming Ag_xAg_y with different release rates of Ag⁺ ions to the solutions. To the best of our knowledge, there is still no report systemically investigates the effects of anions on the morphology and composition of the final products. In this paper, we present a simple and green hydrothermal route for the synthesis of AgNWs. The reaction time and anions in the precursors were found to act as the key factors in the hydrothermal process. A new growth path of the AgNWs was proposed after a systematic and careful study with a variety of characterizations. The as-prepared AgNWs were tested for their antibacterial property to explore their practical use potential in water treatment.

2. Experimental

2.1. Materials

All the chemical reagents of sodium hydroxide (NaOH), hydrogen chloride (HCl), sodium chloride (NaCl), sodium sulfate (Na₂SO₄), sodium acetate (Na₂C₂O₄), sodium carbonate (Na₂CO₃), sodium bromide (NaBr) and sulfide (Na₂S) were purchased from Shanghai Chemical Reagent Co. Ltd (Shanghai, China). Silver nitrate (AgNO₃) was primary reagent. Maltose was biochemical reagent purchased from Beijing AOBBOX biological technology Co. Ltd (Beijing, China). All the chemicals were used as received without further purifications. All the ultrapure water used in this study was produced by EPED system (EPED, China).

2.2. Preparation of AgNWs

In this particular designed system, silver nitrite was used as the silver source, and maltose was applied as the reducing agent. Sodium chloride was also introduced to the reaction system. 15 mL of AgNO₃ (0.02 M) and 15 mL of NaCl (0.02 M) were added to 30 mL of maltose aqueous solution (0.02 M) with magnetic stirring, and then the as-prepared mixture was transferred into a Teflon-lined stainless steel autoclave (100 ml volume) and maintained at 160 °C for 18 h in an electronic oven. After the autoclave cooled down naturally, the precipitate at the bottom of the reactor was sufficiently washed with water and then dried at 60 °C for further usage. In other anion systems, the NaCl was replaced by Na₂SO₄ (0.01 M), Na₂CO₃ (0.01 M), Na₂C₂O₄ (0.01 M), Na₂S (0.01 M), NaBr (0.02 M), and all the other procedures were the same.

2.3. Characterization

The morphologies of the products were observed by a JSM-6390A scanning electron microscope (SEM, JEOL, Japan) and a JEM-2100 transmission electron microscope (TEM, JEOL, Japan). The UV–vis spectra were obtained from Agilent 8453 spectrophotometer system (UV–vis, Agilent, USA). The crystalline structures of all the samples were examined by X-ray diffraction meter (XRD, X'pert PRO with CuK α radiation). The chemical states of the products were studied using the X-ray photoelectron spectroscopy (XPS) measurement performed on Axis Ultra, Kratos (UK) at monochromatic Al K α radiation (150 W, 15 kV and 1486.6 eV). The Ag⁺ ion concentration released in the disinfection process was checked by the ICP measurement (ICPE-9000, Shimadzu, Japan).

2.4. Antibacterial property test

The as-prepared AgNWs were loaded on the activated carbon fibers (ACFs) surface to prepare the antibacterial material of AgNWs-loaded ACFs. Antibacterial property of the material was tested using the disk-diffusion method [37]. The water disinfection

experiment was carried out in a circulated setup using the AgNWs-loaded material as the filter. Typical gram-positive and gram-negative bacteria of *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*) were selected as the model bacteria with initial concentration of 7×10^5 cfu mL⁻¹. The antibacterial efficiency was determined by plate counting method. Specifically, an aliquot of the reaction solution was diluted with saline, and an appropriate dilution of the sample was incubated at 37 °C for 24 h on a culture media of nutrient agar. The number of viable cells was then counted, and the antibacterial efficiency was calculated. All the glassware and materials were sterilized in autoclave at 120 °C for 15 min before experiments.

3. Results and discussion

Fig. 1a presents the SEM image of the AgNWs synthesized at 160 °C for 18 h in the NaCl system. It can be seen clearly that AgNWs with a diameter of 60–140 nm were successfully synthesized by the facile hydrothermal method. The UV–vis absorption spectra in Fig. 1b show absorption peak at ~ 380 nm and ~ 410 nm. The appearance of plasmon peak at ~ 410 nm indicates the formation of silver nanowires, while the weak absorption peak at ~ 380 nm was attributed to the resonance of the transverse plasmon mode [38]. The XRD pattern shown in Fig. 1c also demonstrates that metallic Ag with face-centered-cubic (*fcc*) structure and high crystallinity has been successfully synthesized. The significant diffraction peaks at 2θ of 38.3°, 44.32°, 65.54° and 77.40° can be assigned to the (1 1 1), (2 0 0), (2 2 0), and (3 1 1) planes of *fcc* silver, respectively (JCPDS file No. 04-0783). Fig. 1d shows a typical TEM image of a single AgNW. The inset HRTEM image of the selected area reveals clear lattice fringe with *d* spacing of 0.235 nm, corresponding to (1 1 1) plane. SAED pattern in the bottom inset of Fig. 1d can be indexed to *fcc* structure silver with a lattice constant of 4.08 Å, which is in consistency with the analysis of XRD result.

Fig. 2 shows the SEM images of the products obtained at various reaction times of the synthesis process in the NaCl system. It illustrates that the formation of AgNWs underwent a transformation process from particles to wires. Specifically, the AgCl colloidal solution aggregated with heating and grew from about 1 μ m to 2–3 μ m in the first 4 h (Fig. 2a and b). At the reaction time of 8 h, apparent wires appeared. From then on, the product showed a transformation from the mixture of particles and wires to pure wires. The corresponding XRD pattern in Fig. 2f demonstrates that pure AgCl was obtained before the reaction. The relative content of Ag crystal/AgCl increased with the reaction time, with pure Ag crystals obtained eventually. Both of the SEM and XRD measurements revealed that AgNWs were gradually grown from AgCl during the synthetic process. Besides, the XRD (Fig. 2f) and SAED (Fig. 1d) results show that the AgNWs grow along the (1 1 1) direction. The use of coordination reagent such as polymer and coordination compound is necessary to control the morphology of AgNWs by selective adsorption and desorption on different surfaces [39]. As no polymer or coordination compound was introduced, maltose, reducing agent applied in this study, is concluded to simultaneously act as the coordination reagent to kinetically control the growth rate of the AgNWs. Maltose can be oxidized into carbon-based compounds under hydrothermal condition [33]. These compounds are adsorbed on the silver seeds and govern the subsequent growth. The surface energies associated with different crystallographic planes of cubic Ag hold as follows: $\gamma(111) < \gamma(100) < \gamma(110)$ [40]. In terms of surface-energy minimization, facets with higher surface-energy tend to absorb the carbon-based polymers. The (1 1 1) plane has the lowest surface-energy, which may result in no adsorption of carbon-based polymer. The addition of silver atoms to the (111) plane yielded the anisotropic growth of the material to wire.

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