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Nature factory of silver nanowires: Plant-mediated synthesis using broth of *Cassia fistula* leaf

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ABSTRACT

In this paper, we demonstrated a green protocol in which silver nanowires with diameters in the range of 50–60 nm and lengths up to tens of micrometers were synthesized with extract of the *Cassia fistula* leaf as reductant and capping agent at room temperature. Different techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), selected area electron diffraction (SAED), high-resolution transmission electron microscopy (HRTEM), X-ray diffraction (XRD) and UV–vis spectroscopy were employed to characterize the nanowires. Particularly, TEM images recorded at intervals revealed that the resulting silver nanowires evolved from spherical nanoparticles via one-dimensional aggregation. The results also showed that reaction temperature played a crucial role in the formation of silver nanowires.

1. Introduction

Morphology-controlled fabrication of nanostructures has become one of the most important and challenging aspects of modern nanotechnology due to their potential applications in optical, electronic and mechanical nanodevices. For instance, considerable effort has been made to develop new synthetic routes to one-dimensional metal nanostructures such as nanorods, nanowires and nanotubes in the last decade [1–4]. Silver nanowires (AgNWs) have attracted much attention owing to the unique electrical and thermal conductivities of bulk silver. Various methods have been successfully developed for the synthesis of AgNWs. Until now, the most widely used methods are solution-phase chemical approaches, which can be divided into templated and non-templated processes. Templated process involves hard and soft templates. Highly ordered materials including microchannels in alumina or polymer membranes [5,6], mesoporous materials [7–9], carbon nanotubes [10] and DNA [11,12] can be used as hard templates to direct the growth of nanowires. However, removal of the rigid templates from the nanowires generally requires rigorous condition or complicated process, which consequently will limit the scale of production. Therefore, it is rational to develop the soft-template or non-template methods. Murphy and co-workers have synthesized AgNWs with uniform diameters in the presence of silver seeds with cetyltrimethyl ammonium bromide (CTAB) served as soft template [13]. Xia's group has synthesized AgNWs by reducing silver nitrate with ethylene glycol (EG), and polyvinylpyrrolidone (PVP) was introduced as structure-directing reagent rather than a soft template [14,15].

Although AgNWs can be well controlled through different methods mentioned above, they were synthesized either at high temperature or via synergetic action of several reagents. There is a growing need to develop environmentally benign alternatives to these existing methods. Biosynthesis of nanoparticles as an emerging highlight of nanobiotechnology has received increasing attention. To date, a variety of bacteria [16–23], fungi [24–26], and plants [27–36] have been demonstrated to produce metal nanoparticles with different morphologies. For example, single crystalline triangular gold nanoparticles could be modulated by plant extracts of *Tamarind* leaf, *Aloe vera* and lemongrass [32–34]. Our group showed that sundried *Cinnamomum camphora* leaf could be used to synthesize silver nanoparticles and gold triangular nanoparticles in aqueous solutions at ambient conditions [36]. And recently, He et al. reported the biosynthesis of gold nanowires using the cell free extract of *Rhodopseudomonas capsulate* [23]. However,

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shape control of the biogenic metal nanoparticles has met very limited success. In this article, we describe a simple green synthesis of well-defined AgNWs by reducing silver nitrate with the broth of sundried *Cassia fistula* leaf at room temperature, no additives were added to direct the anisotropic growth. Different techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), selected area electron diffraction (SAED), high-resolution transmission electron microscopy (HRTEM), X-ray diffraction (XRD) and UV–vis spectroscopy were employed to characterize the AgNWs. A possible mechanism underlying the fabrication process of the nanowires was proposed based on TEM images recorded at intervals. To the best of our knowledge, this is the first report on plant-mediated biosynthesis of AgNWs.

2. Experimental details

2.1. Materials

C. fistula trees were cultivated by Department of Virescence, Xiamen University, China. The freshly harvested *C. fistula* leaves were washed and exposed to the sun until they were completely dried. Then they were crushed into powder and screened by a 20-mesh sieve. Silver nitrate ($AgNO_3$) was purchased from Sinopharm Chemical Reagent Co. Ltd, China and was used as received.

2.2. Green synthesis of silver nanowires

In a typical synthesis of AgNWs, the carefully weighed *C. fistula* biomass, 1.0 g, was added to 25 ml deionized water in a 100 ml beaker, and then the mixtures were boiled for 5 min, leading to rupture of the walls of leaf cell and release of intracellular materials into solution. After boiling, the solution was filtered and proper amount of deionized water was added to keep the volume of the filtrate 25 ml. The pH of the broth is about 5.4. Later on, the filtrate was mixed with 25 ml aqueous $AgNO_3$ solution (2 mM) and the mixtures were thereafter shaken at a rotation rate of 150 rpm in the dark at 30 °C for 48 h.

2.3. Characterization

UNICAM UV-300 spectrophotometers (Thermo Spectronic) were used to measure the UV–vis spectrum of AgNWs. High-resolution TEM images were obtained and Energy dispersive X-ray (EDX) analysis was performed on Tecnai F30 Microscope. SEM observations were carried out on LEO-1530 Electron Microscope (LEO, Germany). The crystal structures of the AgNWs were investigated using a X'Pert Pro X-ray Diffractometer (PANalytical BV, The Netherlands) operated at a voltage of 40 kV and current of 30 mA with Cu Kα radiation. The resulting AgNWs were obtained by centrifuging the resulting solution at 12,000 rpm for 10 min. The precipitates were completely dried at 60 °C and then analyzed by FTIR Nicolet Avatar 660 (Nicolet, USA).

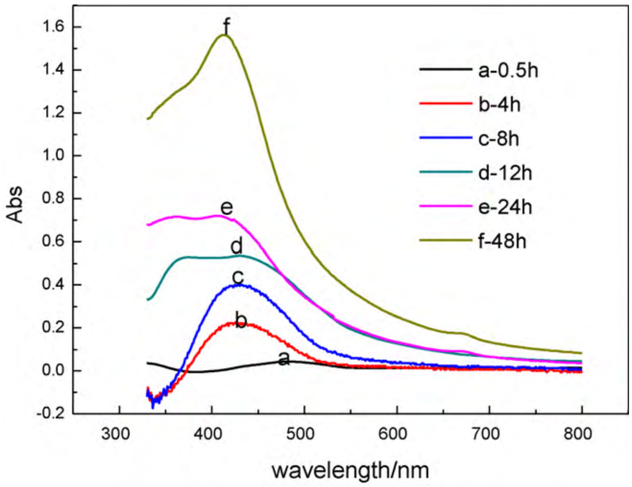

Fig. 1. UV–vis absorption spectra of silver colloids taken at various times: (a) 0.5 h, (b) 4.0 h, (c) 8.0 h, (d) 12 h, (e) 24 h, and (f) 48 h.

3. Results and discussion

3.1. The characterization of silver nanowires

UV–vis absorption spectrum, which strongly depends on the shape and size of nanoparticles, the surrounding dielectric medium, the coupling of colloids and absorbed solutes [37,38], is usually used to characterize the optical properties of metal nanoparticles. Fig. 1 shows the UV–vis absorption spectra of the sample taken at different stages. The appearance of a weak plasmon peak (curve a) indicated that there were a few nanoparticles formed in the solution. At $t = 4$ h, a plasmon peak centered at 430 nm was observed. The intensity of this plasmon peak increased as the reaction time was prolonged (curves b and c). While the reaction time was up to 12 h, a new peak appeared at 360 nm, which can be attributed to the transverse plasmon response of AgNWs, indicating the formation of short nanowires [39,40]. As the length of nanowires grew with time, the intensity of the transverse plasmon peak increased [39]. Nevertheless, the presence of a plasmon peak centered at 415 nm

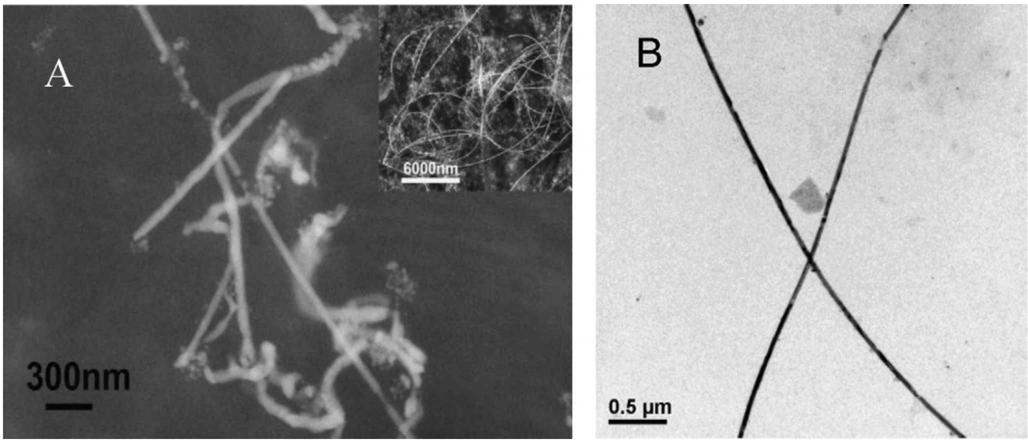

Fig. 2. Typical SEM (A) and TEM image (B) of as-prepared silver nanowires. The inset of (A) is the low magnification of silver nanowires.

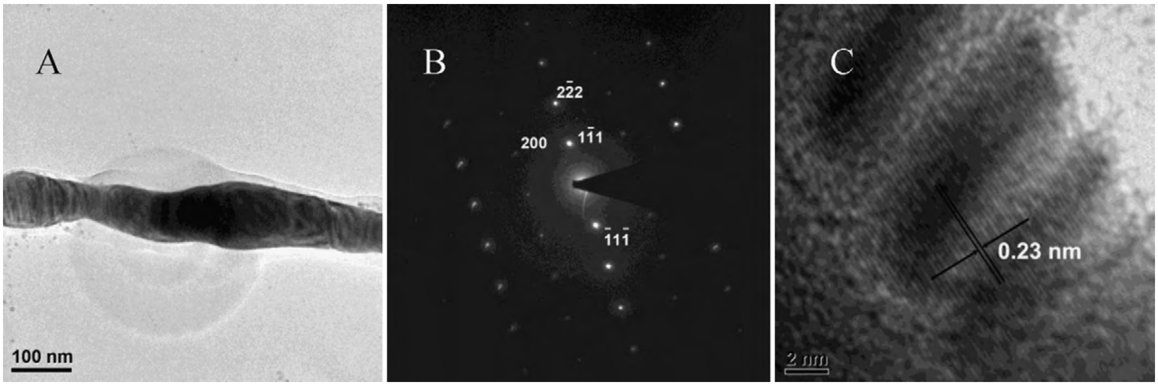

Fig. 3. TEM image (A), SAED pattern (B) and HRTEM (C) of the individual silver nanowires.

indicated the coexistence of a few other silver nanoparticles in the final products.

The SEM and TEM characterizations of the as-synthesized AgNWs are shown in Fig. 2. As presented in Fig. 2A, the obtained product was composed of nanowires and a few silver nanoparticles, which was in accordance with the above UV–vis spectrum (curve f of Fig. 1). The inset of Fig. 2A suggested that the length of AgNWs were up to more than 10 μm. The TEM image in Fig. 2B shows AgNWs ranging in diameter from 40 to 60 nm. Fig. 3B is the SAED pattern of the corresponding AgNWs. The electron diffraction spots could be attributed to the diffraction of ⟨1 1 0⟩ zone, indicating that the as-synthesized nanowires were single crystalline [41]. HRTEM was employed to observe the structure of the resulting nanowires. Fig. 3C shows the fine nanostructure of one section of the AgNWs presented in Fig. 3A. The spacing of the lattice plane is 0.23 nm, which is consistent with the *d* value of (1 1 1) planes of the silver crystal, confirming the crystalline nature of nanowires. The EDX profiles of the sample show the presence of Ag, C and Cu (Fig. 4). Herein, the signal of Cu and C may be due to the background from the supporting copper grid. To further confirm the structure of the AgNWs, a typical XRD pattern was taken from the sample treated for 48 h. In Fig. 5, four diffraction peaks can be assigned to the (1 1 1), (2 0 0), (2 2 0) and (3 1 1) of face-centered cubic (fcc) silver crystal, respectively. The calculated lattice constant based on the (1 1 1) plane is 4.084 Å, which is in accordance to literature value of 4.086 Å (JCPDS file No. 04-0783 from ASTM).

FTIR analysis was used to identify the possible biomolecules responsible for the reduction of the Ag^+ ions and capping of the AgNWs. A number of vibration bands can be seen in the region 1000–4000 cm^{-1}(Fig. 6). The absorption peaks located at around 1075 cm^{-1} can be assigned as the absorption peaks of –C–O–C– or –C–O–. The weak band centered at 1250 and 1461 cm^{-1} is due to C–H deformation vibrations. The absorption spectra at about 1538 and 1628 cm^{-1} can be assigned to the stretching vibration of –C=C–. Moreover, we also observed two bands in the region 2800–3000 cm^{-1}, which can be attributed to the stretching vibration of C–H. In addition, there is a broad peak located at around 3363 cm^{-1}, which can be assigned to the O–H stretching

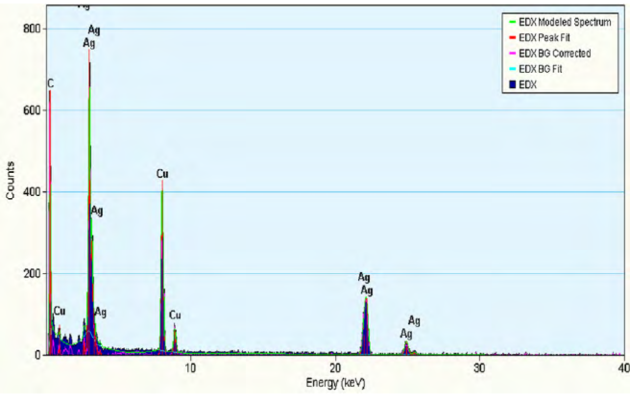

Fig. 4. EDX spectroscopy of the same sample as in Fig. 2A.

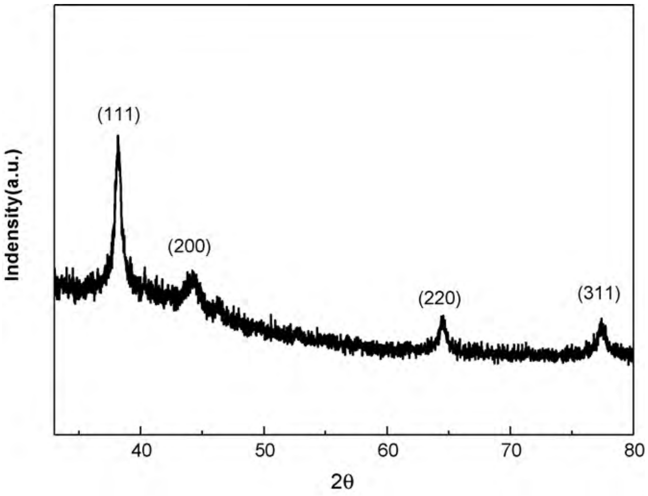

Fig. 5. XRD pattern of the as-prepared nanowires. Labeled peaks correspond to the characteristic diffraction peaks of elemental Ag(0).

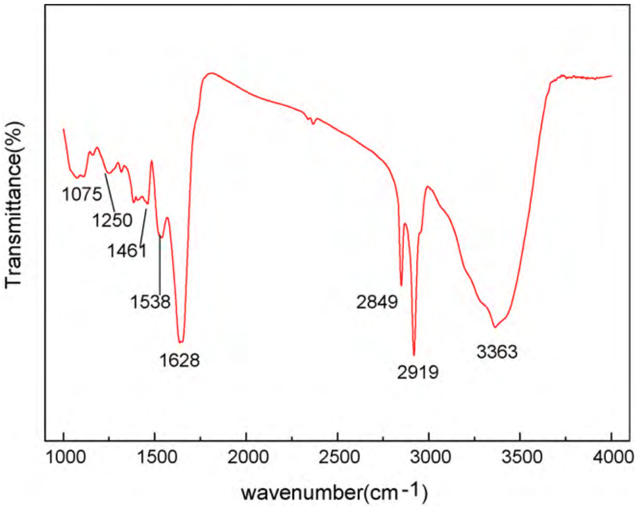

Fig. 6. Typical FTIR absorption spectrum of silver nanowires.

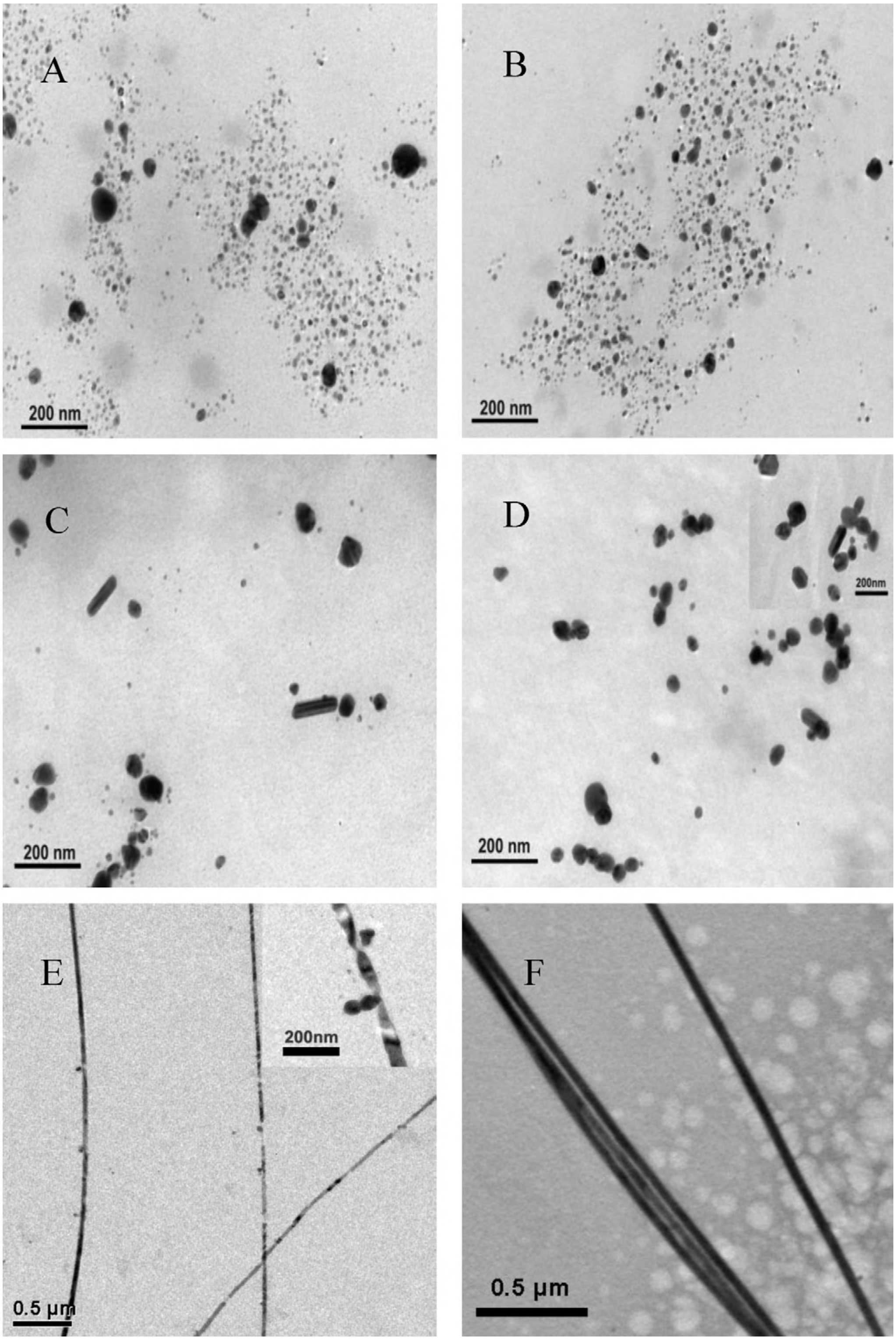

Fig. 7. TEM images of the sample taken at different stages: (A) t = 0.5 h; (B) t = 4 h; (C) t = 8 h; (D) t = 12 h; (E) t = 24 h; and (F) t = 48 h. The scale bar of (A–D) is 200 nm; the scale bar of (E–F) is 500 nm. The inset of (D) and (E) is the one part of the as-synthesized product.

vibration, indicating the presence of hydroxyl groups [42]. Therefore, we conclude that some water-soluble polyhydroxy components, e.g. alkaloids, flavonoids and polysaccharose, are the capping ligands of the AgNWs.

For the fcc metals, surface energies associated with different crystallographic planes are usually different. The surface free energies of the crystallographic planes decreased in the order $\gamma_{\{111\}} < \gamma_{\{100\}} < \gamma_{\{110\}}$ [43]. In the polyol synthesis of AgNWs, the advocated mechanism is that the AgNWs are evolved from multiply twinned nanoparticles [40,44,45]. In these cases, surfactants or capping agents are usually used to induce the formation of 1-D structures as they can adsorb selectively on the surfaces of a metal and control the growth rates of various faces of the metal. Gao et al. reported that AgNWs capped by PVP are bounded by {1 0 0} planes with {1 0 0} growth direction [44]. In another report, they employed X-ray photoelectron spectroscopy (XPS) to investigate the surface chemical state of AgNWs and the results showed that a strong interaction between the carboxyl oxygen atom (C=O) of PVP and {1 0 0} facets of the Ag core, which might lead to anisotropic growth of silver nanoparticles, resulting the formation of AgNWs [45]. Sun et al. also demonstrated that oxygen atoms of the pyrrolidone unit may facilitate the adsorption of PVP onto {1 0 0} facets of silver nanoparticles and induce them to grow at {1 1 1} planes [40]. Likewise, the oxygen atoms herein may facilitate the adsorption of the polyhydroxy components onto specific crystallographic planes like {1 0 0} planes of the nanoparticles to stabilize the nanoparticles and then induce the growth of AgNWs.

3.2. The formation mechanism of silver nanowires

In order to have a good insight into the growth mechanism of AgNWs, we investigated their shape evolution by sampling from the solution at different stages. Fig. 7A shows the initial product taken at 0.5 h: a mixture of spherical silver nanoparticles with an average diameter of ~12 nm and a few other larger ones displaying a diameter of ~50 nm. It was speculated that the smaller nanoparticles were slowly consumed by the larger ones over time through an Ostwald ripening process [46,47]. TEM image of the sample reacted for 4 h shows that the size of silver nanoparticles increased (Fig. 7B). When the reaction time is up to 8 h, large nanoparticles with an average diameter of 60 nm dominated. Furthermore, a few nanorods and the aggregation of some nanoparticles is also observed in Fig. 7C. When the reaction time was prolonged to 12 h (Fig. 7D), it was observed that the amount of aggregation increased and part of them evolved into prolate nanorods structure. After the reaction was continued for another 12 h, the aggregation of silver nanoparticles disappeared and shaped nanowires with diameter ranging from 50 to 60 nm were observed (Fig. 7E). However, it is worth mentioning that a proportion of the nanowires were not continuous. From the TEM image, it seems that those discontinuous nanowires were formed by several long nanorods arrayed in a line. Furthermore, it was observed that the nanoparticles surrounding the broken-nanowires tended to integrate themselves into the breaks between the nanorods, as shown in the inset of Fig. 7E, which further confirms our expectation that with extended reaction times, those adjacent nanorods would connect to each other to form the desired nanowires. At $t = 48$ h, as shown in Fig. 7F, long nanowires with average diameter of 60 nm and well-defined shape were yielded, which was coherent with our former expectation.

Based on the above TEM results, we conclude that AgNWs formed not through point-initiated vectorial growth but rather by the recrystallization of adjacent nanoparticles in a linear aggregation. And a formation mechanism of the AgNWs was elucidated in light of Fig. 8.

At the very beginning of the biosynthesis, silver ions were reduced to metallic silver by certain components existing in the broth of *C. fistula* leaf. And silver nuclei were formed at the initial reaction stage. By gathering the surrounding silver atoms, the nuclei gradually grew into relatively small nanoparticles which may act as the seeds for the growth of larger nanoparticles. Then, as it was stated in the very beginning of this section, large nanoparticles with main two distinctive sizes were generated after 0.5 h of reaction. It is well known that the surface energy of larger particles is lower than that of smaller ones, therefore these small nanoparticles were apt to dissolve into the solution and grow onto larger ones via an Ostwald ripening process [47]. Afterwards, the adjacent large nanoparticles moved, met and then adhered to form a short chain-shape structure. Zhao et al. observed similar phenomenon by employing N, N-dimethylformamide to prepare AgNWs [48]. They proposed that *Van der Waals* forces induce the aggregation of nanoparticles through the so-called depletion flocculation. By prolonging the reaction time, the newly formed silver atoms deposit onto the concave regions of the connected nanoparticles through capillary phenomenon, leading to the formation of long nanorods.

Sun et al. reported that PVP may passivate the side surface of the nanorods, leaving the ends of nanorods uncovered, which remain to be attractive or reactive towards new form atoms [14]. In our experiment, some biomolecules in the broth of *C. fistula* leaf may play the same role of PVP and induce the growth of nanowires. Herein, the ends of these nanorods may be bounded by {1 1 1} facets

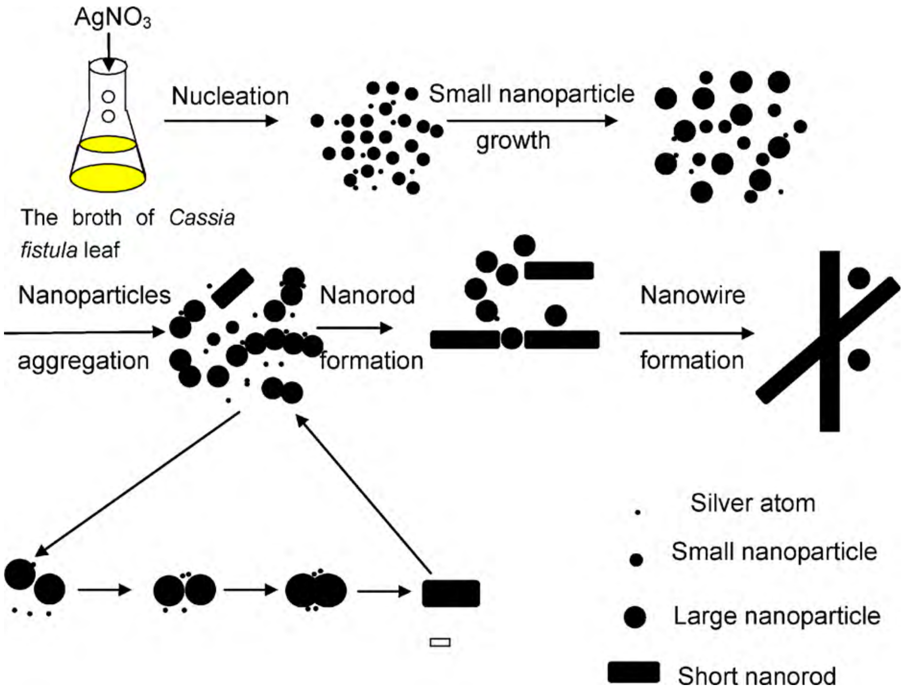

Fig. 8. Schematic illustration of the growth mechanism of silver nanowires.

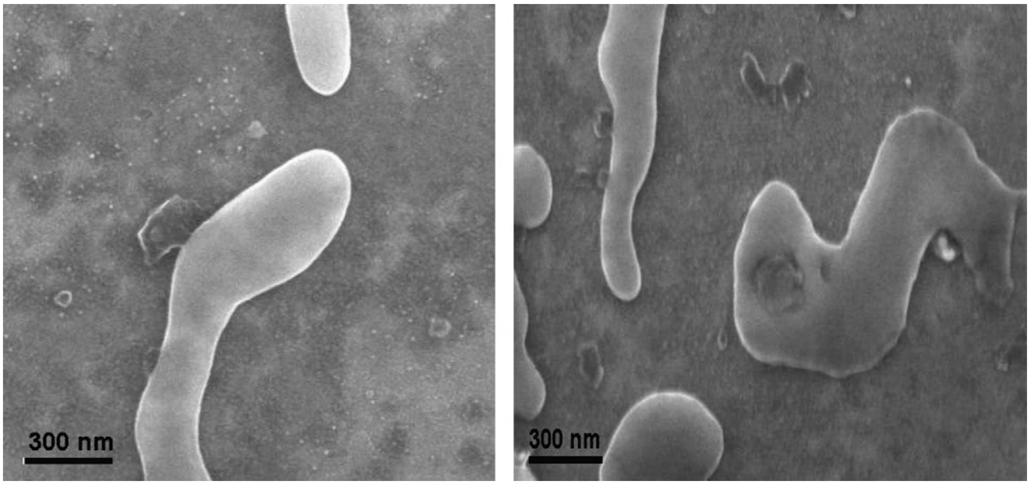

Fig. 9. SEM images of nanowire after it had been further calcined in air at 400 °C for 2 h.

whose interaction with biomolecules is very weak and thus reactive to the new form atoms. Once the reaction takes place, the silver atoms are transported to the nanorods ends through diffusion and the adjacent nanorods are linked to each other, resulting in the formation of AgNWs.

It is worth mentioning that reaction temperature is crucial for the formation of AgNWs. When an experiment was carried out at 60 °C with other conditions remaining the same, only spherical nanoparticles and short nanorods were obtained, suggesting that elevated temperatures might change the interaction between biomolecules and the faces of silver and hinder the coalescence of nanoparticles in solution, which was not favorable in the formation of AgNWs. In addition, we also studied the effect of calcination on these AgNWs. The morphology of AgNWs has altered after being heated in air at 400 °C for 2 h. Only a few irregular rods and spherical nanoparticles were observed, which were shown in Fig. 9, and the diameters of these rods were wider than the as-prepared nanowires.

4. Conclusions

In summary, we have developed a simple and green method to synthesize AgNWs with diameters in the range of 40–60 nm and lengths up to tens of micrometers using the broth of *C. fistula* leaf as reductant and capping agent. No chemical reagent or surfactant template is required in this protocol, which consequently endows the bioprocess with the advantage of being environmental-friendly. The results in this study showed that, rather than formed through a point-initiated vectorial growth, the AgNWs formed by the recrystallization of adjacent nanoparticles in a linear aggregation. In addition, reaction temperature was found to play an important role in this protocol and a low temperature was favorable in the formation of AgNWs. The shape and size of AgNWs could not be maintained after being calcined at 400 °C for 2 h.

Acknowledgments

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