



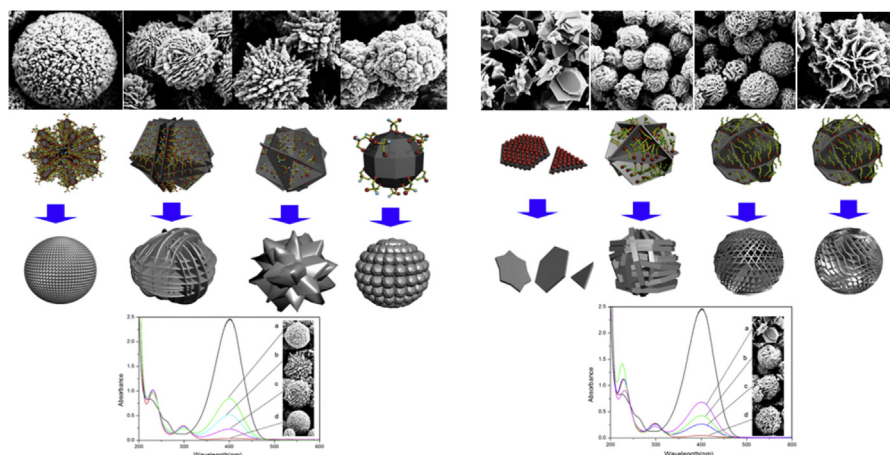
Facile controlled synthesis of silver particles with high catalytic activity



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GRAPHICAL ABSTRACT



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ABSTRACT

Properties of metal particles (MPs) are largely dependent on preparation method and morphology. Herein, we report a new facile synthesis route of silver particles (AgPs) in which growth pattern of AgPs can be easily controlled and some novel morphologies are achieved. In this strategy, cheap organic acids are selected as growth regulators for the morphology control of AgPs, and the concentration of organic acid plays a key role in the formation of AgPs. Furthermore, the effects of number of carbon atom in organic acids on the morphology of AgPs were investigated. With increase of number of carbon atom in organic acids, the morphology of AgPs will change from plate to sphere. These results presented here are vital to both exploration of growth theory and constructing novel morphologies of AgPs not only in this paper but also possibly other system. Moreover, catalytic activity of these prepared AgPs was studied toward reduction of *p*-nitrophenol by NaBH₄.

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1. Introduction

In the past few years, nano/micro metal particles (MPs) have possessed very broad applications in many fields such as surface-enhanced Raman scattering, solar cell, electronic paste display, catalysis etc. owing to their unique physical and chemical proper-

ties [1–6]. In some cases, whether commercial value or application characteristics of metal particles are largely dependent on their size and morphology. So controlled synthesis of metal particles is highly desirable and many efforts have been devoted to this aspect [7–17]. Thus far, several technologies including seeded growth and template methods have been developed to controllably synthesize metal particles with special morphology [18–20]. Among them, seeded growth involving preferential growth on pre-formed seed surface is probably the most widely used and effective protocol because of its advantages in structural control and variety [21,22]. However, despite its importance, the development of seeded growth for metal particle has remained a considerable challenge to achieve micron metal particles with desired structure. As for template methods, surfactants are often used as template and the final morphology of the metal particles rely on the templates formation in the reaction solution. The multi-step process is necessary for preparing big size AgPs and surfactants may become an obstruction for rapid large-scale preparation [23,24].

Usually, synthesis of noble metal particles by chemical reduction is a good model system, which generally includes three starting materials, namely, metal-containing salts or acids, reducing agent, and water. This synthetic scheme has been widely studied and a substantial amount of important information regarding this system has been deposited in literatures [25,26]. Recently, several groups achieved homogeneous or heterogeneous growth of noble metal particles and obtained interesting morphologies using different technologies [27,28]. However, these methods are invalid for preparation of AgPs with large size. Herein, we reported a new facile method to synthesize large size AgPs in which homogeneous and heterogeneous growth pattern could be precisely controlled and some novel morphologies of AgPs were achieved. On this base, catalytic activity of these AgPs were also examined through reduction of *p*-nitrophenol by NaBH_4 .

2. Results and discussion

2.1. Citric acid system

In this route, citric acid acted as stabilizers and growth regulators, and quadruple ascorbic acid was added to AgNO_3 solution for ensuring that AgNO_3 will be completely reduced. Experimental results indicated that the concentration of citric acid played a key role in the formation of AgPs. Fig. 1 shows SEM images of AgPs obtained under different citric acid concentration with diameter between 1 μm and 4 μm .

Without citric acid, irregular spherical AgPs with rough surface were obtained as shown in Fig. 1(a). Interestingly, with the increase of citric acid concentration, the rough part of Ag surface start to grow and form needle protrusion like anemones (0.0001 M) as shown in Fig. 1(c). With the further increase of citric acid concentration, these protuberant crystals on Ag surface gradually transferred from large and random to small but consistent, and resulted in AgPs like a plush ball as shown in Fig. 1(e and f).

Based on the traditional nucleation-growth mechanism for metal particles, the formation of AgPs involves two steps. Firstly, silver nitrate was reduced by ascorbic acid to silver atom. Then some silver atom quickly aggregated and formed numerous primary silver crystal seeds. Secondly, Ag atoms were continuously adsorbed by the active sites of the silver seeds at the solid–liquid interface, which lead to the growth of AgPs. As documented in many literatures, the (111) plane of silver crystal may possess the lowest surface energy, and the preferential adsorption of suitable additives, such as sodium citrate may further lower the energy and stabilize the (111) plane, that often results in the formation of nano-plates structure [29]. But in our work, when ionic sodium

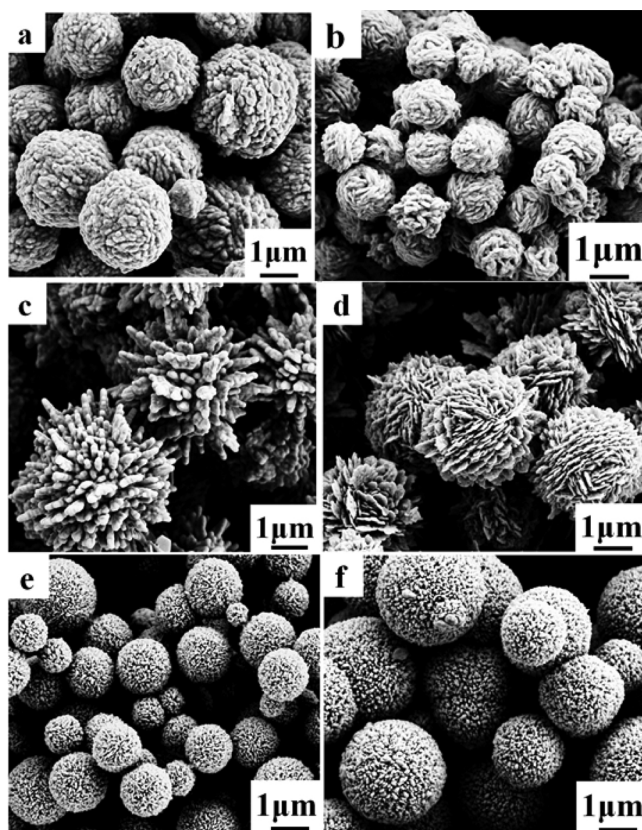


Fig. 1. SEM images of AgPs obtained under different concentrations of citric acid (a) 0 M, (b) 0.00001 M, (c) 0.0001 M, (d) 0.001 M, (e) 0.01 M, and (f) 0.1 M.

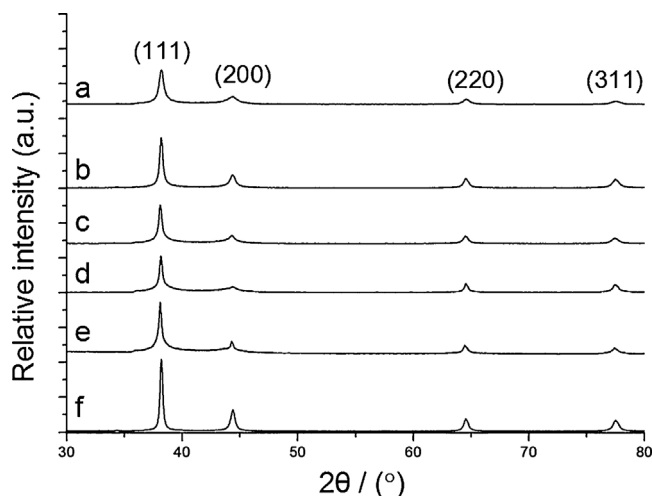


Fig. 2. XRD patterns of AgPs formed in the presence of citric acid with different concentrations (a) 0 M, (b) 0.00001 M, (c) 0.0001 M, (d) 0.001 M, (e) 0.01 M, and (f) 0.1 M.

citrate was replaced by nonionic citric acid, an excited opposite phenomenon and novel morphologies occurred. Fig. 2 shows the XRD patterns of corresponding AgPs. The five diffraction peaks are indexed to the corresponding crystal surface of (111), (200), (220) and (311). These data reveal that all samples of AgPs possess the same face-centered cubic crystal structure. But it is obvious that with the increase of the concentration of citric acid, the relative peak strength of (220) and (311) was improved. Compared with sodium citrate as additive, the phenomenon indicates that reducing polarity of additives would decrease the tendency of preferential

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