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INFLOWMATION CHRONICLES
HIGHLIGHTS OF INTERESTING SCIENTIFIC APPLICATIONS

What is a Micro-Flow Reactor?

A micro system for chemical and biochemical reactions, including separation, fluid handling, and unit operations of chemical engineering, as well as analytical systems. Its small reaction volumes and high heat and mass transfer rates allow for precise adjustment of process conditions, short response times, and defined residence times, resulting in greater process control and higher yields and selectivity.

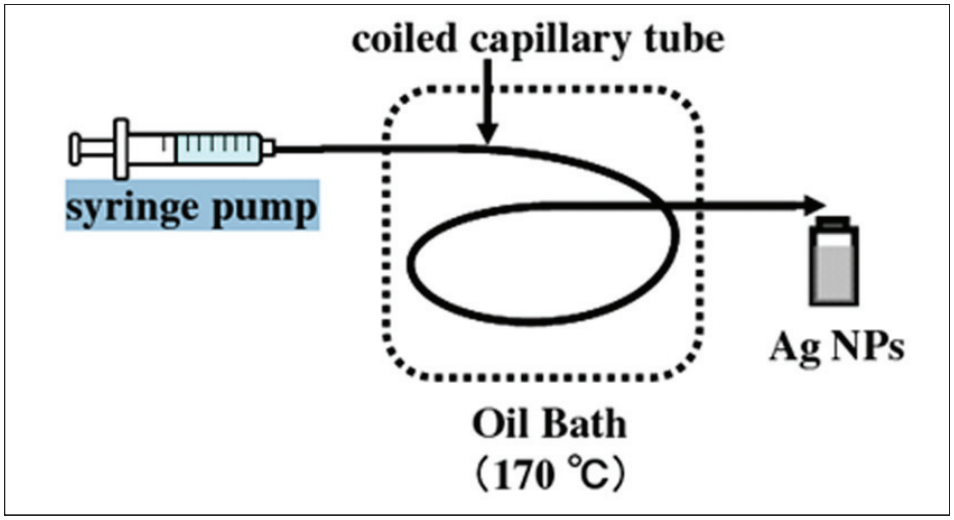

Figure 1: Schematic diagram of the experimental setup for the synthesis of silver nnoparticles in micro-capillary reactors.

Controlled synthesis of colloidal silver nanoparticles in capillary micro-flow reactor

In this study, using a polytetrafluoroethylene (PTFE) capillary tube as a micro-flow reactor, well-dispersed colloidal silver nanoparticles were controllably synthesized with different flow rates of precursory solution. Scanning transmission electron microscopy images and UV–visible absorbance spectra showed that silver nanoparticles with large size can be prepared with slow flow rate in the PTFE capillary reactor. The effects of tube diameters on the growth of colloidal silver nanoparticles were investigated. Experiment results demonstrated that using a tube with small diameter was more propitious for the controllable synthesis of silver nanoparticles with different sizes.

Introduction

In the recent decades, the study of nanoparticles (NPs) of noble metals has been an extremely active area because of their interesting properties which are different from those of bulk substances (Holm et al. 2006; Enders et al. 2007; Kumar et al. 2007; Xu et al. 2007; Shibata et al. 2002). Due to their ability to sustain resonant collective electron oscillations excited in the near-ultraviolet or visible range of the nelectromagnetic spectrum (Bohren and Huffman 1983), silver nanoparticles have provided a particularly rich and ongoing focus for intense research (Guerrini et al. 2006; Dawn and Nandi 2006). Many investigations have been focused on the control of the size of nanocrystals and their self-assembly into two dimensional (2D) or three dimensional (3D) superlattic structures (He et al. 2002; Wiley et al. 2007; Selvakannan et al. 2004). However, conventional synthesis methods are often impeded by their low production yields of nanoparticles, time-consuming processes, or higher costs (Brust et al. 1994; Plieni et al. 1998). Recently, micro-reactor technology has been developed to synthesize various organic (Kobayashi et al. 2004; Arana et al. 2003; Seong and Crools 2002), biological (Takayama et al.2001; Wang et al. 2003; Zanzotto et al. 2004), and inorganic materials (Chan et al. 2003; Nakamura et al. 2002; Yen et al. 2003; Shestopalov et al. 2004; Wang et al. 2002, 2004; Lin et al. 2004; He et al. 2005, 2007). In a micro-reactor, the reaction is confined to a very small area (down to micro-scale) and the heat transfer is much more efficient than that in the traditional batch reactor (Jahnisch et al. 2004). This advantage makes it possible to heat or cool the reaction system rapidly to get precisely defined reaction time. Furthermore, with a micro-reactor, the synthesis can be conducted continuously with a large-scale production.In this paper, using a polytetrafluoroethylene (PTFE) capillary tube as a micro-flow reactor, the synthesis of mono-disperse silver nanoparticles Is presented. With micro-capillary reactor, by changing the flow rate of precursory solution, the reaction time was defined preciously to obtain mono-dispersed silver nanoparticles with different sizes. Also, the effects of capillary tubes with different diameters onthe growth of colloidal silver nanoparticles were investigated. In capillary tube, due to the laminar flow of reaction solution, the reaction system is relatively static, to some degree; mass transfer is not so fast and efficient that particle can grow continuously as in the batch reaction system.

KDS Model 100 syringe pump

Key features for this application:

- **High precision and accuracy**
- **Smooth flow**
- **User selectable flow rates**

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Experiment…Synthesis and chemicals

Figure 1 depicts a schematic diagram of the experimental setup. As a typical procedure (Hiramatsu and Osterloh 2004), about 0.011 M precursor reaction solution was prepared by dissolving 50 mg silver acetate and 2.5 ml oleylamine in 25 ml 1,2-dichlorobenzene. The precursory solution was transferred into a syringe and injected into the PTFE microcapillary tube with different flow rates using the KDS 100 syringe pump by KD Scientific (Holliston, MA). The capillary tube was coiled and immersed into the 170 C oil bath. The residence time of the reaction solution inthe heating zone was controlled by changing flow rates of reaction solution. All reagents were purchased from Aldrich Chemical Co., and used without further purification. The sample was washed with methanol for several times, and the final sample was dispersed in chloroform.

Results and Discussion

With micro-reactor technology, the reaction time can be controlled preciously by pumping the precursory solution with different flow rates. For example, in this study, using a PTFE capillary tube with the diameter of 300 cm, if the tube length immersed in oil bath is 200 cm, when the flow rates of the precursory reaction solution are 70.7, 28.2, and 14.2 mL/min; the reaction time can be defined as 2, 5, and 10 min, respectively. Figure 2 presents the UV–visible absorbance spectra of silver nanoparticles synthesized with different flow rates in the PTFE capillary tube with the structures mentioned above. In Fig. 2, the flow rates were 70.7, 28.2, and 14.2 mL/min, and the maximum absorption peaks of as-synthesized silver nanoparticles were at 410, 414, and 422 nm, respectively, indicating that the growth of silver nanoparticles can be controlled by different flow rates. With slow flow rate, large particles were produced. Furthermore, as shown in Fig. 2, the full width at half-maximum (FWHM) values of absorbance peaks of silver nanoparticles synthesized at different flow rates were 67, 50, and 89 nm, respectively. These small FWHM values of absorbance spectra showed that silver nanoparticles synthesized in the PTFE micro-reactor were well dispersed and particle size distributions were narrow. These results can be further demonstrated by STEM observation.

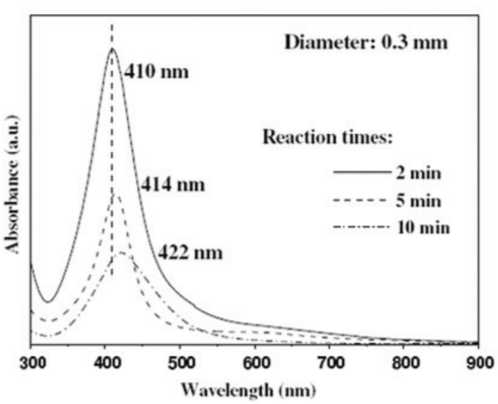

Figure 2: UV—visible absorption spectra of silver colloidal nanoparticles synthesized with different flow rates in capillary tubes with a diameter od 300 μm.

Conclusion

In this paper, using PTFE capillary tube as micro-reactor, well-dispersed silver nanoparticles with different sizes were synthesized by changing the flow rates of precursory solution. STEM images and UV–visible absorbance spectra demonstrated that big particles can be synthesized with slow flow rate of precursory solution. The effects of capillary tube on the growth of colloidal silver nanoparticles were investigated. In micro-flow reactor, reaction solution flows in laminar mode and the reaction system is in a relatively static state; mass transfer is confined in a relatively small area. Thus, using a capillary tube with the appropriate structure, such as diameter of the tube, is very important to controllably synthesize well-dispersed silver nanoparticles. The experiment results demonstrated that using a tube with small diameter was more propitious to realize the controllable synthesis of silver nanoparticles with different sizes.

Reference

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Sheng Tai He, Yu Lan Liu, Hideaki Maeda

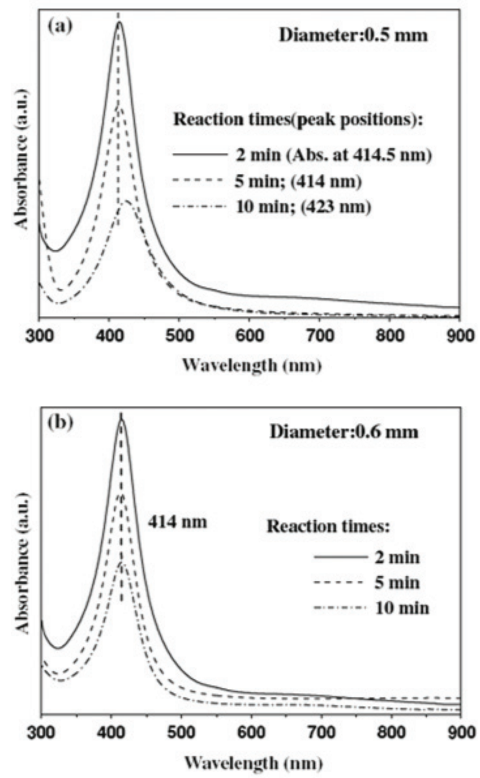

Figure 3: UV—absorption spectra of silver nanoparticles synthesized in PTFE capillary micro-flow reactor with different diameters: (a) μm and (b) 600 μm.