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Synthesis, characterization and electrocatalytic activity of silver nanorods towards the reduction of benzyl chloride

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Abstract

This paper describes a simple method of synthesizing silver nanorods using the polyol process, where propylene glycol serves both as a reducing agent and as a solvent in the presence of a capping reagent such as polyvinylpyrrolidone (PVP). The diameter and length of silver nanorods could be controlled by changing the $AgNO_3$/PVP ratio. The end-to-end assembly of the silver nanorods was found. The silver nanorods were characterized by using scanning electron microscopy, transmission electron microscopy, X-ray diffraction and absorption spectroscopy. The catalytic activity of a glassy carbon electrode with Ag nanorods exhibits extraordinary electrocatalytic activities towards the electro-reduction of benzyl chloride.

Keywords: Silver nanorods; Polyol process; Reducing agent; Capping reagent; Electro-reduction

1. Introduction

Nanomaterials of noble metals have received much attention due to their potential application in microelectronics, optical and catalytic properties [1–5]. The synthesis of metal nanoparticles of desired size and shape has enormous importance in nanotechnology because of their characteristic shape and size dependent optical, electronic and chemical properties. The novel properties of these materials have found extensive uses in microelectronics, electronics, magnetic devices, non-linear optics, chemical and biochemical sensors and in catalysis [5–7]. Among the known nanoparticles, Ag is widely studied because of its characteristic optical, spectroscopic and catalytic properties. They have been widely exploited for use in photography, catalysis, biological labeling, photonics and surface enhanced Raman scattering, among others. A rich variety of recipes are now available in the literature for the synthesis of Ag nanoparticles. However, it has been observed in most cases, that due to the existence of a highly exposed surface, the nanoparticles tend to aggregate and form large clusters even during the preparative process. Therefore, a need to control the particle–particle interactions so as to obtain a stable dispersion has been acutely felt by researchers in this community. It is generally accepted that the preparation of discrete silver nanoparticles with well-defined shape and well-controlled dimensions is more difficult than preparation of gold ones due to the higher reactivity of silver compounds. Here we report the formation of silver nanorods, through direct reduction of $AgNO_3$ by the solvent (propylene glycol) at 170 °C in the presence of a stabilizer polymer PVP. By varying the concentration of silver salt and PVP as well as the temperature, one can conveniently control the shape of the resulting nanoparticles. These results are in agreement with those reported by Xia and co-workers in a number of recent papers using the polyol process [8–12]. It was shown that silver nanorods formed in a modified polyol process as a result of the aggregation of previously formed nanoparticles.

An exciting and hitherto unexploited silver nanorod has been needed for electrode applications. Electrocatalytic reduction of organic halides (RX) has been a central topic in organic electrochemistry for the last few decades [13–17]. Among various examined electrode materials, silver has been found to possess extraordinary electrocatalytic properties [18–32].

In the present paper, we report a simple chemical synthesis of silver nanorods. The as-prepared silver nanorods were characterized by transmission electron microscopy (TEM), scanning electron microscopy (SEM), powder X-ray diffraction (XRD) and UV absorption spectrum measurements. The

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electrochemical reduction of benzyl chloride is evaluated by cyclic voltammetry and is interpreted in comparison with that of conventional silver electrodes. Here we report the results of an investigation on the potentialities of silver nanorods as an electrocatalyst in the electro-reduction of benzyl chloride.

2. Experimental

2.1. Materials

Poly (*N*-vinyl-2-pyrrolidone) (PVP, MW ~ 40 000, supplied by Fluka), silver nitrate ($AgNO_3$) and the solvents used in the study were analytical grade reagents and were used as received.

2.2. Synthesis of silver nanorods

The synthesis was performed within a three-neck round beaker, which was heated in an oil bath up to 170 °C and then 10 ml of $AgNO_3$ and 20 ml of PVP solution in glycol were added in turn. The refluxing was maintained for 90 min; small aliquots were then withdrawn during the reaction for TEM. The mixture was diluted with water and nanorods were collected by centrifugation.

2.3. Structural characterization

XRD patterns were obtained by a powder diffractometer (XRD-SHIMADZU XD-D1) using a Ni-filtered Cu Kα X-ray radiation source. The morphology of the silver nanorods was investigated by SEM (FEI, Model: Quanta 200). TEM, electron diffraction and energy dispersive X-ray analysis (EDAX) were performed on a Philips CM12/STEM instrument. For UV–vis absorption investigation, a certain amount of the obtained Ag nanorod powders was dispersed in ethanol and determined with a TU-1201 model spectrophotometer.

2.4. Electrochemical characterization

A single glass compartment cell with three electrodes was employed for the cyclic voltammetry studies. Pt wire and

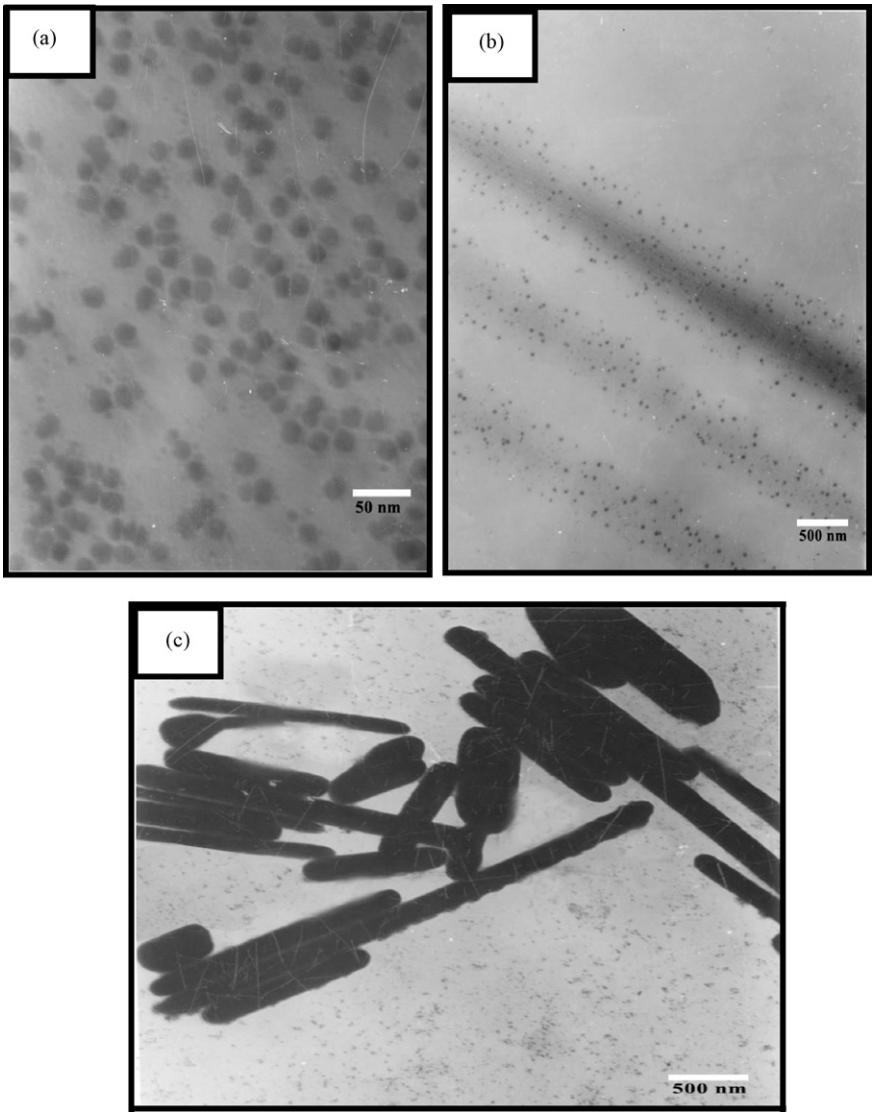

Fig. 1. (a–c) TEM images of silver nanorods formation during the course of reaction.