



Short communication

Electrochemical synthesis of silver nanoparticles in solution



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ABSTRACT

Silver nanoparticles in the bulk solution were obtained by the potentiostatic electrolysis in undivided cell using silver anode at the potential of the mediator reduction (tetraviologen calix[4]resorcine) at room temperature in DMF/0.1 M Bu₄NPF₆ media. The metal nanoparticles aggregate to form larger particles eventually.

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1. Introduction

In recent years, the metal nanoparticles are of great scientific interest due to their unique physical and chemical properties and a wide variety of potential applications in the field of catalysis, biomedicine, optics, electronics and others [1–6]. Methods of preparation of metal nanoparticles are quite diverse. They are conventionally divided into physical and chemical. The electrochemical methods of obtaining of metal nanoparticles in solution have found only limited application [7–13], although the electrochemical reduction of the metal ions and complexes is the classical method of producing of metal, metal plating, metal black on the electrode surface on an industrial scale [14].

Based on the earlier expressed idea [15] of the electrochemical production of the metal particles in the bulk solution through the mediator reduction of their ions or complexes, we recently [16] have carried out the electrosynthesis of the palladium nanoparticles by reduction of [PdCl₄]²⁻ using viologen-cavitand MVCA-C₅⁸⁺·8Cl⁻ (Scheme 1) as a mediator and stabilizer of Pd nanoparticles. In this article, we report the electrochemical synthesis of silver nanoparticles in the bulk solution using also MVCA-C₅⁸⁺·8PF₆⁻ as a mediator. Silver nanoparticles (typically 5–50 nm) spectral selectively (~400 nm) absorb solar energy; exhibit catalytic and antibiotic properties; and are the most common and widely used nanoparticles [17]. Known methods of their preparation and properties sufficiently completely are summarized in the review [17]. Currently, the most widely used method of their synthesis is the reduction of silver salts by sodium borane, first proposed by Creighton [18], Lee and Meisel [19].

2. Experimental section

The investigation was carried out using the methods of CV, electrolysis, dynamic light scattering (DLS) and Atomic force microscopy (AFM).

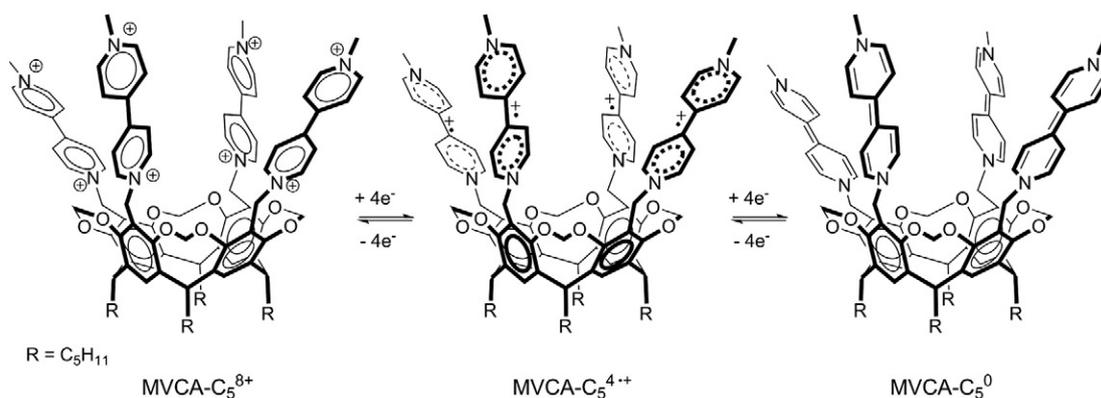
CV recordings were done on a P-30S potentiostat (without IR-compensation) in argon atmosphere. The working electrode was a glassy carbon (GC) disk electrode (d = 3.4 mm) pressed into Teflon. The electrode was cleaned by mechanical polishing with diamond paste before each measurement. Platinum/silver wire was a counter electrode. The potentials were measured relative to the saturated calomel electrode (SCE), E₀' (Fc/Fc⁺) = +0.41 V. Aqueous SCE was connected by a bridge filled with background solution. The temperature was 295 K. The diffusion nature of the peak currents *i*_p was proven using the theoretical shape of the voltammogram and the linear dependence *i*_p – *v*^{1/2} by varying the potential scan rate *v* from 10 to 200 mV/s and the adsorption nature was substantiated by the presence of an adsorption maximum.

The electrolysis was carried out in a three electrode undivided glass cell in a potentiostatic mode in argon atmosphere at room temperature (T = 295 K) using a P-30S potentiostat. During the electrolysis, the solution (DMF, 0.1 M Bu₄NPF₆, 0.5 mM MVCA-C₅⁸⁺·8PF₆⁻, V = 10 ml) was stirred with a magnetic stirrer. We used platinum plate (S = 3.12 cm²) as cathode, silver wire (S = 0.3 cm²) as anode and SCE as reference electrode. The solution after the electrolysis were controlled by CV on the indicator GC disk electrode (d = 3.4 mm) directly into the electrolyzer.

DLS measurements were performed by Malvern Instrument Zetasizer Nano. The measured autocorrelation functions were analyzed by Malvern DTS software.

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Scheme 1. Reversible electrochemical reduction of MVCA-C₅⁸⁺.

The investigation of the surface of samples was carried out by intermittent contact mode AFM using Scanning Probe Microscope Multi-Mode V (Veeco).

MVCA-C₅⁸⁺·8PF₆⁻ were synthesized by the literature procedure [20]. The salt Bu₄NPF₆ (“Aldrich”) and DMF (“Ekos-1”) were used as purchased without additional purification.

3. Results and discussions

The cyclic voltammogram (CV) of MVCA-C₅⁸⁺ exhibits the two diffusion reversible reduction peaks in DMF/0.1 M Bu₄NPF₆ media (Figure 1).

The first reduction peak ($E_{p1}^{\text{red}} = -0.39$ V) corresponds to the formation of tetra(cation-radical) MVCA-C₅⁴⁺, and the second peak ($E_{p2}^{\text{red}} = -0.82$ V) represents the formation of the neutral compound MVCA-C₅⁰ (Scheme 1) [20–25].

The silver ions are reduced more easily ($E_p^{\text{red}} = 0.20$ V) with the formation of metallic silver deposited on the electrode surface and oxidized at $E_p^{\text{ox}} = 0.60$ V (Fig. 1). For the bulk metallic silver, a continuous growth of the current of the anodic dissolution of silver is observed in the near field of potentials to form Ag⁺ ions (Fig. 1). These voltammetric data show the principal possibility of obtaining the silver particles in the bulk solution by the electrolysis in undivided cell in

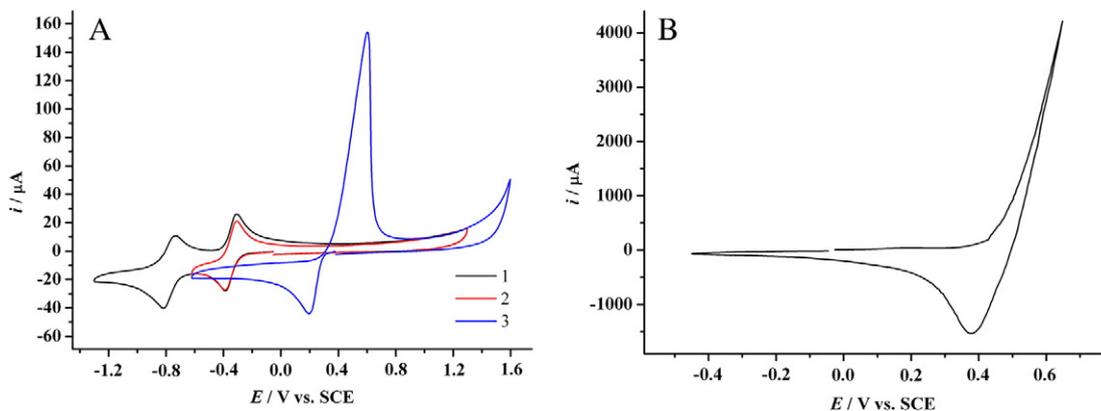


Fig. 1. CV in DMF/0.1 M Bu₄NPF₆ media of (A) MVCA-C₅⁸⁺ (0.5 mM) (1, 2) and Ag⁺ (1.5 mM) (3) using the glassy carbon electrode, (B) metallic Ag (S = 0.3 cm²). $\nu = 100$ mV/s.

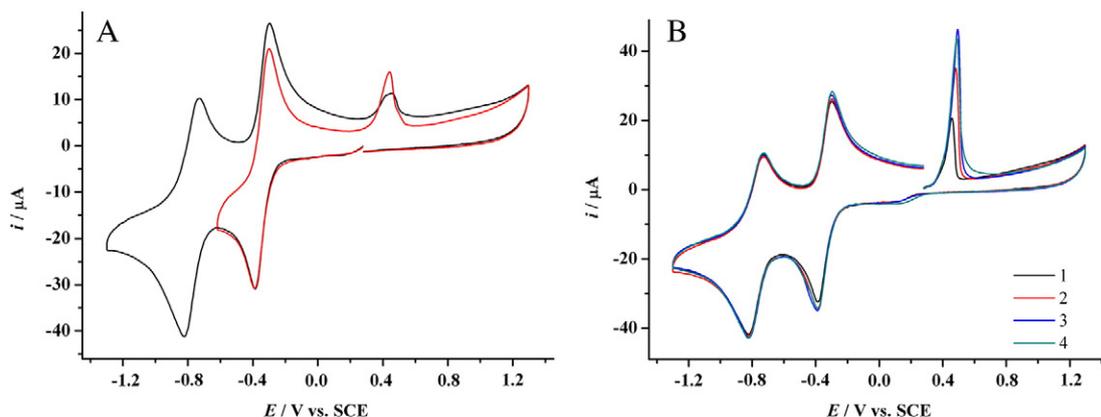


Fig. 2. CV for the system MVCA-C₅⁸⁺ (0.5 mM)-Ag⁰ recorded in the electrolysis cell after electrolysis at $E = -1.1$ V ($Q = 3$ F/mol MVCA-C₅⁸⁺) in DMF/0.1 M Bu₄NPF₆ media using the glassy carbon electrode with the potential scan in (A) negative and (B) positive directions depending on incubation time the indicator electrode in the solution: 0.5 min (1), 1 min (2), 2 min (3), 3 min (4). $\nu = 100$ mV/s.

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