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Methylviologen mediated electrochemical synthesis of catalytically active ultrasmall bimetallic PdAg nanoparticles stabilized by CTAC

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Abstract

Efficient electrosynthesis of the monodisperse ultrasmall spherical mono- and bimetallic nanoparticles (NP) of Ag and Pd stabilized in the CTAC shell in solution bulk was carried out by methylviologen mediated reduction of equimolar amounts of Pd(II) and Ag(I) (1.5 mM) in the presence of CTAC (75 mM) at controlled potentials of the $MV^{2+}/MV^{\cdot+}$ redox couple in water/ 0.1 M NaCl medium at room temperature. Metal ions are quantitatively converted to NP upon consumption of the theoretical amount of electricity. Sizes of isolated PdNP and AgNP are 5 ± 1 nm and 18 ± 5 nm, respectively. Bimetallic PdAgNP were obtained in three ways: (i) by preliminary synthesis of PdNP and subsequent reduction of AgCl (Pd/AgNP); (ii) by preliminary synthesis of AgNP and subsequent reduction of $PdCl_2$ (Ag/PdNP); (iii) by joint reduction of $PdCl_2$ and AgCl (Ag-PdNP). In all cases, a solid-solution alloy PdAgNP is obtained. The driving force for the alloy formation is the energy gain of ~ 33 kJ/mol of an alloy. In the first way, the NP size (8 ± 2 nm) and the Pd content in the alloy (76%) are slightly higher than in the other two methods (6 ± 1 nm, 50 and 68%), but the alloy crystallite size (2-5 nm) in NP is the same. PdNP and alloys NP exhibit a high catalytic activity toward the *p*-nitrophenol reduction and the

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