Preparation and electrocatalytic activity of bimetallic

Ni-Cu micro- and nanoparticles

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**Electronic Supplementary Information**

**Description of the materials used and procedures for the synthesis of nanoparticles by the method of successive chemical reduction (to Experimental Section in the article)**

*Materials*

Nickel nitrate (Ni(NO3)2·6H2O), copper nitrate (Cu(NO3)2·3H2O), polyvinyl alcohol (Mw = 9,000-10,000 g mol‒1), hydrazine hydrate (N2H4·H2O, 64%) and sodium hydroxide (NaOH) were purchased from "Ridder" LLP (Karaganda, Kazakhstan) and used without further purification. *p*-Nitrophenol was purchased from Sigma-Aldrich. Distilled water and medical ethyl alcohol (96%) were used to prepare aqueous-ethanol solutions.

*Synthesis of monometallic Ni and Cu, and bimetallic Ni-Cu particles*

The Ni/Cu and Cu/Ni particles were prepared by chemical reduction using two synthetic procedures. For the experiments, weighed portions of metal salts were taken which contain 2.00 g of each metal, i.e. the Ni:Cu ratio was 1:1 by weight, or 0.034 mol of Ni(NO3)2·6H2O and 0.032 mol of Cu(NO3)2·3H2O.

According to *the first procedure*, the Ni (or Cu) nitrate was dissolved in 200 ml of aqueous-ethanol solution (volume ratio was 1:1) under stirring at 80°C (or 60°C) for 30 min. To the solution, a mixture consisting of hydrazine hydrate (1.023 mol or 0.788 mol, respectively, for Ni and Cu salts reduction) and sodium hydroxide (0.273 mol or 0.063 mol) dissolved in 30 ml of distilled water was slowly added. The stirring of the reaction mixture was continued for 1 h. Next, the mixture was cooled to 5°C in an ice bath and the metal particles were separated by centrifugation at 2500 rpm. To the obtained metal particles, 150 ml of fresh water-ethanol solution were added and the suspension was sonicated for 30 min. Separately, a solution of the second metal nitrate in 100 ml of aqueous-ethanol mixture was prepared. This solution was then poured into the first metal particle suspension and stirred at room temperature for 30 min. After that, the temperature of the reaction mixture was raised to that required for the second metal reduction. Then, the alkaline solution of the reducing agent prepared by mixing above amounts of hydrazine hydrate and sodium hydroxide was added to the resulting suspension under stirring for 1 h. The reaction mixture was cooled to 5°C, centrifuged, washed with distilled water and ethyl alcohol and dried at 80°C and a pressure of 0.06 MPa. Monometallic Ni and Cu particles were prepared by the first part of this synthesis procedure.

*The second procedure* for Ni-Cu particles synthesis differs from the first one only in that the particles of the first metal reduced from its salt were not separated from the reaction mixture. After the reduction of the first metal cations is completed, its particles were sonicated in the same reaction mixture, and then all components for the second metal cations reduction were introduced in the suspension.

By the procedures 1 and 2, bimetallic Ni/Cu and Cu/Ni particles were also synthesized with the addition of a polymeric stabilizer, polyvinyl alcohol (PVA). In this case, a weighed portion of the salt of the first metal was dissolved in 150 ml of the 3% PVA aqueous-ethanol solution. When reducing the second metal, the PVA stabilizer was not used due to a possible obstacle to the interaction with the first metal.