

Oxalate Method for Assessing the Electrochemical Surface Area for Ni-Based Nanoelectrodes Used in Formaldehyde Sensing Applications

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Abstract : In this study, we used an accurate and precise method to measure the electrochemically active surface areas (Aecsa) of nickel electrodes. Calculated Aecsa is really important for the evaluation of an electro-catalyst's activity in electrochemical reaction of different organic compounds. The method involves the electrochemical formation of Ni(OH)₂ and NiOOH in the presence of adsorbed oxalate in alkaline media. The studies were carried out using cyclic voltammetry with polycrystalline nickel as a reference material and electrodeposited nickel nanowires, homogeneous and heterogeneous nickel films. From cyclic voltammograms, the charge (Q) values for the formation of Ni(OH)₂ and NiOOH surface oxides were calculated under various conditions. At sufficiently fast potential scan rates (200 mV s⁻¹), the adsorbed oxalate limits the growth of the surface hydroxides to a monolayer. Although the Ni(OH)₂/NiOOH oxidation peak overlaps with the oxygen evolution reaction, in the reverse scan, the NiOOH/ Ni(OH)₂ reduction peak is well-separated from other electrochemical processes and can be easily integrated. The values of these integrals were used to correlate experimentally measured charge density with an electrochemically active surface layer. The Aecsa of the nickel nanowires, homogeneous and heterogeneous nickel films were calculated to be Aecsa-NiNWs = 4.2066 ± 0.0472 cm², Aecsa-homNi = 1.7175 ± 0.0503 cm² and Aecsa-hetNi = 2.1862 ± 0.0154 cm². These valuable results were expanded and used in electrochemical studies of formaldehyde oxidation. As mentioned nickel nanowires, heterogeneous and homogeneous nickel films were used as simple and efficient sensor for formaldehyde detection. For this purpose, electrodeposited nickel electrodes were modified in 0.1 mol L⁻¹ solution of KOH in order to expect electrochemical activity towards formaldehyde. The investigation of the electrochemical behavior of formaldehyde oxidation in 0.1 mol L⁻¹ NaOH solution at the surface of modified nickel nanowires, homogeneous and heterogeneous nickel films were carried out by means of electrochemical techniques such as cyclic voltammetric and chronoamperometric methods. From investigations of effect of different formaldehyde concentrations (from 0.001 to 0.1 mol L⁻¹) on electrochemical signal - current we provided catalysis mechanism of formaldehyde oxidation, detection limit and sensitivity of nickel electrodes. The results indicated that nickel electrodes participate directly in the electrocatalytic oxidation of formaldehyde. In the overall reaction, formaldehyde in alkaline aqueous solution exists predominantly in form of CH₂(OH)O⁻, which is oxidized to CH₂(O)O⁻. Taking into account the determined (Aecsa) values we have been able to calculate the sensitivities: 7 mA mol L⁻¹ cm⁻² for nickel nanowires, 3.5 mA mol L⁻¹ cm⁻² for heterogeneous nickel film and 2 mA mol L⁻¹ cm⁻² for homogeneous nickel film. The detection limit was 0.2 mM for nickel nanowires, 0.5 mM for porous Ni film and 0.8 mM for homogeneous Ni film. All of these results make nickel electrodes capable for further applications.

Keywords : electrochemically active surface areas, nickel electrodes, formaldehyde, electrocatalytic oxidation

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