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“Localized generation of the catalytic metallic nanostructures and pH mapping with scanning electrochemical microscopy”

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Abstract

In the first part of the thesis, the development of a method of preparation of ligand-free copper (CuNS), gold (AuNS), and bimetallic nanostructures (NS) was described. NS were obtained by localized electrorefining of polycrystalline metal wires from the microelectrodes on indium tin oxide (ITO) and glassy carbon (GC) supports, using scanning electrochemical microscopy (SECM). The morphology of the obtained NS and thus their catalytic properties were adjusted by the electrorefining parameters, *e.g.* the potential of electrodeposition, translation rate of the microelectrode and composition of the electrolyte. A number of various NS prepared on a single support were studied for their catalytic activity toward oxygen reduction reaction (ORR) in alkaline media and carbon dioxide reduction reaction (CO_2RR) by SECM feedback mode. Their electrocatalytic activity toward the afore-mentioned reactions depends on electrorefining conditions.

In the second part preparation of syringaldazine (Syr) modified carbon nanoelectrode for local pH imaging was described. pH was determined from the mid-peak potential of a cyclic voltammogram of an adsorbed Syr. Voltammetric pH nanosensor exhibited a stable quasi-reversible voltammetric response within the pH range of 2 – 12 with fast response and high spatial resolution. It was successfully applied for mapping of local alkalization of buffered electrolyte in the vicinity of a Pt microelectrode during ORR.