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Title of the doctoral dissertation: **Potential-driven generation of Ni(OH)₂-type nanoparticles embedded in non-conductive poly(Salen) matrices for electrocatalytic oxidation of ethanol and urea**

Abstract

This PhD dissertation presents the fabrication, characterization, and application of Ni(OH)₂-type catalysts embedded in poly(Salen) matrices, named poly[NPs-Ni(OH)₂Salen], for the electrooxidation of small organic molecules such as ethanol and urea. This electrochemical oxidation is of significant importance for energy conversion and wastewater treatment. However, developing efficient catalysts with properly tuned structures that ensure high catalytic activity remains a major challenge. In particular, the fast generation and stability of Ni³⁺OOH-type active sites are crucial for achieving high electrocatalytic performance.

To address this challenge, NiSalen complexes with distinct chemical structures were electrodeposited under potentiostatic and potentiodynamic conditions to prepare poly(NiSalen) precursors with distinct morphologies. Subsequently, poly[NPs-Ni(OH)₂Salen] catalysts were generated *via* a potential-driven transformation of the poly(NiSalen) precursors in alkaline media. The mechanism of this transformation, which had not been previously elucidated, is presented in detail.

Deposition of poly(NiSalen) precursors with various morphologies influenced the arrangement of Ni(OH)₂ NPs embedded in the poly(Salen) matrices. The morphologies of both precursors and the Ni(OH)₂-type catalysts derived from them were imaged using SEM and TEM. These results highlighted interdependencies among the structures of the precursors, the morphologies of the resulting catalysts, and their electrocatalytic performance. Moreover, modifications to the structure of poly(NiSalen) precursors, based on different configurations of methyl substituents at the imine bridge, and the addition of methoxy groups to the aromatic rings, were separately studied to assess their influence on the electrocatalytic activity of the derived catalysts.

However, when urea was electrocatalytically oxidized, additional optimization of the catalyst operating conditions was performed. Taking into account the strong adsorption of the electrooxidation products. The influence of alkaline media concentration on the potential-driven transformation process was investigated by monitoring the *in situ* generation ability of Ni³⁺OOH-type active centers. The optimal operating conditions of poly[NPs-Ni(OH)₂Salen] catalysts for urea electrooxidation were determined based on comprehensive electrochemical analysis, including multiple scan-rate dependent studies, Tafel slope evaluation, and activation energy determination. At the end, long-term stability tests were conducted in a real system mimicking conditions to confirm the applicability of poly[NPs-Ni(OH)₂Salen] catalysts for the remediation of urea-rich wastewater.