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Title: In-situ electrochemical reconstruction and modulation of adsorbed hydrogen coverage in cobalt/ruthenium-based catalyst boost electroreduction of nitrate to ammonia

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Abstract: The electroreduction of nitrate offers a promising, sustainable, and decentralized route to generate valuable ammonia. However, a key challenge in the nitrate reduction reaction is the energy efficiency of the reaction, which requires both a high ammonia yield rate and a high Faradaic efficiency of ammonia at a low working potential (≥ -0.2 V versus reversible hydrogen electrode). We propose a bimetallic Co-B/Ru₁₂ electrocatalyst which utilizes complementary effects of Co-B and Ru to modulate the quantity of adsorbed hydrogen and to favor the specific hydrogenation for initiating nitrate reduction reaction at a low overpotential. This effect enables the catalyst to achieve a Faradaic efficiency for ammonia of 90.4 \pm 9.2% and a remarkable half-cell energy efficiency of 40.9 \pm 4% at 0 V versus reversible hydrogen electrode. The in-situ electrochemical reconstruction of the catalyst contributes to boosting the ammonia yield rate to a high level of 15.0 \pm 0.7 mg h⁻¹ cm⁻² at -0.2 V versus reversible hydrogen electrode. More importantly, by employing single-entity electrochemistry coupled with identical location transmission electron microscopy, we gain systematic insights into the correlation between the increase in the catalyst's active sites and its structural transformations during the nitrate reduction reaction.

Nitrate electroreduction is a promising method for sustainable NH₃ production, but the challenge lies in achieving high yield and efficiency at low potentials. Here the authors report a bimetallic Co-B/Ru₁₂ catalyst for improved activity and unveil the dynamic change of the active sites using operando techniques and single entity experiments.

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