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**Title:** Enhancing Cascade Reaction Efficiency by Local pH Regulation for Integrated Anodic H<sub>2</sub>O<sub>2</sub> Generation and Ammoximation

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**Abstract:** Cascade reaction strategies integrating electrochemistry with chemical transformations offer routes for the synthesis of value-added chemicals. However, the efficiencies of such integrated processes get compromised due to competitive electrochemical reactions and incompatibility between electrochemical and chemical transformations. We report an integrated electrochemical-chemical coupling of anodic H<sub>2</sub>O<sub>2</sub> generation with ammoximation for oxime synthesis. An FTO/Sb<sub>2</sub>WO<sub>6</sub> anode was designed and optimized to anodically produce H<sub>2</sub>O<sub>2</sub> with a maximum Faradaic efficiency (FE) of 87%. H<sub>2</sub>O<sub>2</sub> from the anode oxidizes NH<sub>3</sub> to NH<sub>2</sub>OH, which subsequently reacts with cyclohexanone to yield cyclohexanone oxime with 99% selectivity and a maximum electron efficiency (EE) of 81%. Continuous and adjustable H<sub>2</sub>O<sub>2</sub> input ensures synchronization with the ammonia oxidation reaction while minimizing over-oxidation of the reaction intermediates. Operando scanning electrochemical microscopy (SECM) revealed local pH shifts caused by the proton-coupled electron transfer and its effect on the FE of H<sub>2</sub>O<sub>2</sub> synthesis and competing NH<sub>3</sub> oxidation, providing mechanistic insights for optimizing the reaction microenvironment. By regulating the electrolyte composition to modulate the interfacial pH, side reactions were suppressed and H<sub>2</sub>O<sub>2</sub> generation was promoted, thereby enhancing cascade selectivity. This work highlights local pH regulation as a tool to improve reaction compatibility and efficiency in cascade electrosynthesis.

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