

## Intensified Electrochemical H<sub>2</sub>O<sub>2</sub> Synthesis and Highly Efficient Pollutant Removal Enabled by Nickel Oxides with Surface Engineered Facets and Vacancies

**Authors :** Wenjun Zhang, Thao Thi Le, Dongyup Shin, Jong Min Kim

**Abstract :** Electrochemical hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) synthesis holds significant promise for decentralized environmental remediation through the electro-Fenton process. However, challenges persist, such as the absence of robust electrocatalysts for the selective two-electron oxygen reduction reaction (2e<sup>-</sup> ORR) and the high cost and sluggish kinetics of conventional electro-Fenton systems in treating highly concentrated wastewater. This study introduces an efficient water treatment system for removing substantial quantities of organic pollutants using an advanced electro-Fenton system coupled with a high-valent NiO catalyst. By employing a precipitation method involving crystal facet and cation vacancy engineering, a trivalent Ni (Ni<sup>3+</sup>)-rich NiO catalyst with a (111)-domain-exposed crystal facet, named {111}-NivO, was synthesized. This catalyst exhibited a remarkable 96% selectivity and a high mass activity of 59 A g<sup>-1</sup> for H<sub>2</sub>O<sub>2</sub> production, outperforming all previously reported Ni-based catalysts. Furthermore, an advanced electro-Fenton system, integrated with a flow cell for electrochemical H<sub>2</sub>O<sub>2</sub> production, was utilized to achieve 100% removal of 50 ppm bisphenol A (BPA) in 200 mL of wastewater under heavy-duty conditions, reaching a superior rapid degradation rate (4 min, k = 1.125 min<sup>-1</sup>), approximately 102 times faster than the conventional electro-Fenton system. The hyper-efficiency is attributed to the continuous and appropriate supply of H<sub>2</sub>O<sub>2</sub>, the provision of O<sub>2</sub>, and the timely recycling of the electrolyte under high current density operation. This catalyst also demonstrated a 93% removal of total organic carbon after 2 hours of operation and can be applied for efficient removal of highly concentrated phenol pollutants from aqueous systems, which opens new avenues for wastewater treatment.

**Keywords :** hydrogen peroxide production, nickel oxides, crystal facet and cation vacancy engineering, wastewater treatment, flow cell, electro-Fenton

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