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Nb Doping Strategy for Active Site Modification in Co_3O_4 to Enable Concurrent Hydrogen Production and Glycerol Valorization for Efficient Formate Production

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Electrocatalytic glycerol oxidation reaction (GOR) has emerged as a sustainable and energy-efficient alternative to the oxygen evolution reaction (OER), offering the dual benefit of hydrogen (H_2) generation and selective upgrading of biomass-derived glycerol into value-added chemicals like formate. However, the development of cost-effective, active, and stable electrocatalysts for GOR at low overpotentials remains a significant challenge. Herein, we report a niobium-doped cobalt oxide ($\text{Nb-Co}_3\text{O}_4$, designated as 3NCO) heterostructure directly grown on nickel foam (NF) via a hydrothermal strategy, serving as a high-performance, bifunctional electrocatalyst for HER, OER, and GOR. Nb incorporation introduces electronic modulation and lattice distortion within the Co_3O_4 framework, which enhances the density of active sites and optimizes charge transfer kinetics under alkaline conditions. As a result, the 3NCO/NF electrode exhibits excellent activity, requiring only 196 mV overpotential to achieve 10 mA cm^{-2} for HER, and 1.50 V vs. RHE for OER. Notably, in 0.1 M glycerol + 1 M KOH, GOR proceeds at just 1.19 V vs. RHE, achieving a remarkable potential drop of 330 mV compared to OER, and enabling overall electrolysis at 1.46 V in a two-electrode system. This work highlights the role of lattice Nb doping in boosting active site availability and electron transfer, positioning 3NCO/NF as a robust platform for energy-saving H_2 production and electrochemical glycerol valorization.

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