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**Title:** Surface Reconstruction Induced by Preconditioning in Different Electrolytes Impacts Electrooxidation of Solketal on Multi-Metal-Based Catalysts

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**Abstract:** The electrocatalytic oxidation of solketal (SOR) as the anodic reaction can be used to enhance hydrogen production efficiency due to its lower overpotential compared to the oxygen evolution reaction (OER). Additionally, solketal oxidation yields besides formate also high-value C3 products, providing an advantage over the typical alternative reaction for OER, glycerol oxidation. The multi-metal CoNiFeCu (1:1:1:0.5) catalyst is employed as electrocatalyst for SOR and different activation procedures are explored. The pre-conditioning of the catalyst in the presence of solketal supports a current density of 10 mA cm<sup>-2</sup> to be recorded at only 1.405 +/- 1 mV versus reversible hydrogen electrode (vs. RHE) during SOR, while promoting glyceric acid synthesis with 40% faradaic efficiency. Operando attenuated total reflection Fourier-transform infrared spectroscopy (ATR-FTIR) confirms that the activation of the catalyst contributes to an increased formation of products. However, most importantly, through utilizing the single-particle-on-the-nanoelectrode with identical-location transmission electron microscope (IL-TEM), it is unveiled that solketal suppresses Cu leaching and oxyhydroxide formation. Moreover, the structural transformations are correlated with the increased activity toward SOR.

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