Evaluation of Ethylene Glycol Oxidation on Cobalt Oxide films by using Scanning Electrochemical Cell Microscopy

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Scanning electrochemical cell microscopy (SECCM) has been widely used to investigate structure-activity correlations in the nanoscale^[1]. The using of the droplet hanging at the tip of a nanopipette as a probe allows for the measurement of the local activity of a catalyst, with the probed area being dependent on the size of the used nanopipette (from 50 nm to 10 µm). However, achieving high-resolution activity maps of metal oxides for oxidation reactions in alkaline media is limited by the excessive wetting and the instability of the droplet during the measurements, which limit the resolution of the SECCM in alkaline media^[2]. Despite some studies and strategies proposed to overcome this problem, the phenomena is not totally understood. In this work, we study how the wetting and the recorded currents for ethylene glycol oxidation reaction (EGOR) and oxygen evolution reaction (OER) are affected by the atmosphere surrounding the droplet during the experiment and the size of the pipette. For that, smooth, thin films of single-faceted Co₃O₄ (111) deposited on gold were used as a model system. The understanding of the phenomena occurring during the SECCM measurement helped to adopt strategies to get higher resolution in the SECCM measurements and capture any heterogeneity in the EGOR activity on single and clusters of Co₃O₄ nanoparticles found within the films fabricated by spray coating of cobalt hydroxide and its subsequent calcination.

[1] D. Martin-Yerga, Curr. Opin. Electrochem., 2023, 42, 10.1016/j.coelec. 2023.101405

[2] S. Varhade et al., Electrochim. Acta, 2023, 460, 10.1016/j.electacta.2023.142548.