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Deciphering the Roles of Chemistry and Structure in Iridium Oxide Oxygen Evolution Nanocatalysts

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The development of proton exchange membrane (PEM) water electrolysis, which enables the production of de-carbonized hydrogen, is hindered by the low efficiency of the oxygen evolution reaction (OER). This is especially true in acidic environment where only noble metals (mainly Ru and Ir) can be used as electrocatalysts. Iridium oxide nanocatalysts (IrO_x) are promising due to their high OER activity and good resistance to acidic and oxidative conditions. However, finding a compromise between their electrocatalytic activity and stability remains challenging. Moreover, it is not yet clear which parameter (the material structure¹ or the initial iridium oxidation state²) controls these two properties.

To address these issues, we synthesized IrO_x nanoparticles (NPs) supported on carbon (IrO_x/C) and annealed them at temperatures ranging from 200°C to 800°C under air. To analyze the structure, morphology, and oxidation state of Ir in these catalysts, we used transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and both laboratory-based and synchrotron-based X-ray diffraction (XRD) techniques. We observed *in situ* the crystallization of IrO_x NPs, leading to crystallite size growth and change in Ir electronic state (see Figure 1a). We also observed the evolution of the IrO_x morphology with the annealing temperature (see Figure 1b-f). We assessed their electrocatalytic activity towards the OER and determined their stability number³ (*S*-number) using inductively coupled plasma mass spectrometry and electrochemistry. By correlating the changes in electronic structure, morphology and size to the evolution of the OER activity and stability of the IrO_x NPs, we identified some conditions where an optimal balance between activity and stability is achieved.



Figure 1 – **a.** XRD diffractograms obtained on IrO_x/C recorded *in situ* at 25, 100, 150, 200, 250, 300, 350, 400, 450, 500, 600, 700 and 800°C under air. Each measurement lasted 13 minutes. The hashes correspond to tetragonal IrO₂ (PDF card 00-043-1019) and the stars to Al_2O_3 (PDF card 04-025-2780), which is a measurement artefact related to the powder holder. **b-f.** TEM images of IrO_x/C NPs calcined at 200 °C, 300 °C, 350°C, 500°C and 800 °C.

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- 2. Claudel, F. et al. ACS Catal. 9, 4688–4698 (2019).
- 3. Geiger, S. et al. Nat. Catal. 1, 508-515 (2018).