Advancements in laboratory XAFS instrumentation for the investigation of electrocatalysts for water splitting

Carlos A. Triana

Department of Chemistry, University of Zurich, Winterthurerstrasse 190, CH-8057 Zurich, Switzerland
carlos.triana@chem.uzh.ch

Rational design of efficient electrocatalysts for industrial water splitting is essential to produce sustainable hydrogen fuel. However, understanding catalytic mechanisms under reaction conditions remains a major challenge. This is because the characterization of active species requires the use of front-line experimental techniques at high-energy synchrotron radiation sources to achieve the highest atomic-scale resolution. Fortunately, recent advancements in high-performance laboratory X-ray spectrometers offer synchrotron-quality resolution to access hard X-ray techniques at the lab without compromising on energy resolution. We present recent advances in laboratory X-ray absorption spectroscopy (XAS) instrumentation (Figure **1a**), and their applications to the investigation of electrocatalysts for water splitting. The tabletop XAS in-lab instrument permits the acquisition of X-ray absorption near edge spectra (XANES), giving insight on the valence state and electronic configurations of active catalytic centers, and the extended X-ray absorption fine structure (EXAFS), offering information of interatomic distances, coordination numbers, and disorder in the coordination shells (Figure **1b**).[1] The element-selectivity of the in-lab XAS instrument offers an exceptional technique to prove the atomic-level coordination environment, electronic and chemical states of active catalytic sites under reaction condition, no matter they are crystalline or amorphous catalysts.[2] The enhancement of the spectrometer capabilities to achieve adequate XAS signal-to-noise ratio will be discussed. The use of additives, attaining high performance at low/high energy ranges, sample preparation and design of electrochemical cells for *in situ*/*operando* experiments will be further examined (Figure **1c**).

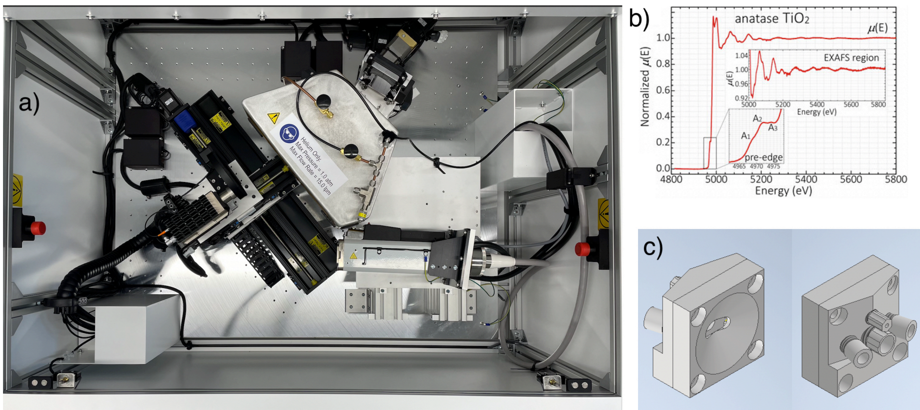

Figure 1. **a**) Laboratory X-ray absorption spectroscopy (XAS) instrument easyXAFS300. **b**) Representative XAS spectrum of anatase TiO_2. **c**) Home-designed electrochemical cells for *in situ*/*operando* XAS experiments.

[1] S.K. Beaumont, *Phys. Chem. Chem. Phys.* 2020, 22, 18747-56.

[2] C.A. Triana, C. Moyses Araujo, R. Ahuja, G.A. Niklasson, T. Edvinsson, *Sci. Rep.* **2017**, 7, 2044.