Quasi simultaneous operando XRD/XAS studies in heterogeneous catalysis

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Understanding the structural evolution of heterogeneous catalysts under reaction conditions is essential for establishing reliable structure-activity relationships. Operando X-ray diffraction (XRD) and X-ray absorption spectroscopy (XAS) provide complementary insights into the crystallographic and electronic changes occurring during catalytic processes. However, obtaining a comprehensive picture of catalyst working mechanisms is often hindered by the difficulty of integrating data collected from separate experiments, typically optimized for individual techniques.

To address this challenge, we employed *quasi simultaneous* operando XRD/XAS to investigate the evolution of nanostructured catalysts during the conversion of biomass-derived molecules in flow conditions, enabling the simultaneous acquisition of structural and electronic information within a single experiment. Two case studies will be presented: i) In-promoted Ni catalysts for the steam reforming of acetic acid [1] and ii) bimetallic Ni-Cu catalysis for the production of propanediols from glycerol, using in-situ generated hydrogen via aqueous phase reforming of ethanol. The catalytic processes involve an initial reduction step in hydrogen at high temperature, followed by the introduction of liquid reactants under high-temperature and/or high-pressure conditions.

We will first describe the technical solutions developed to perform the catalytic reactions while simultaneously acquiring XRD/XAS data. We will then present a comprehensive view of the structural and electronic evolution of the catalysts under working conditions, combining *quasi-simultaneous* XRD/XAS measurements with ex situ characterization and ab initio calculations.

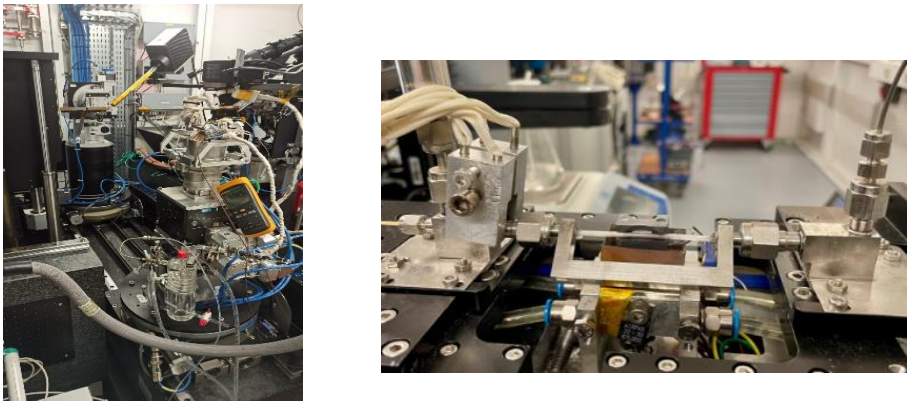


Figure 1. *Parts of the experimental setup used for steam reforming of acetic acid*

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