Mechanistic Investigation of Catalytic Materials via Electrochemistry and In-Operando Synchrotron Techniques

In the context of the growing demand for increasingly efficient, selective, and sustainable catalytic systems, understanding the fundamental mechanisms governing material activity represents a crucial challenge for advanced research. In this framework, the integration of electrochemical techniques with operando spectroscopic methodologies represents a strategic approach to investigate in detail the structural and electronic transformations occurring during catalytic processes. This study proposes a multidisciplinary approach, combining electrochemical measurements with soft X-ray absorption spectroscopy (XAS), which will be performed at the BACH beamline (CNR) at Elettra Synchrotron radiation facility, in order to obtain a dynamic, real-time view of the phenomena underlying catalytic activity. To this end, specifically designed electrochemical cells will be employed, capable of ensuring controlled experimental conditions compatible with synchrotron radiation. This approach will enable real-time correlation between the electronic properties of materials and their catalytic activity, providing a detailed understanding of reaction mechanisms. Samples will be appropriately synthesized, deposited onto conductive substrates, and characterized to ensure purity, stability, and reproducibility. Preliminary electrochemical characterization will be carried out using cyclic voltammetry (CV), linear sweep voltammetry (LSV), and chronoamperometry (CA) techniques, including the use of a rotating disk electrode (RDE) to separate kinetic and diffusion contributions. These investigations will be complemented by additional spectroscopic and surface analysis methods such as Raman spectroscopy and X-ray Photoelectron Spectroscopy (XPS).

The PhD project is structured in different phases:

Phase 1 – Setup, training, and preliminary characterization

The candidate will be involved in the planning of research activities, alongside training on the main experimental techniques, including electrochemistry and advanced synchrotron-based spectroscopies. In parallel, will work on the synthesis and optimization of catalytic materials, followed by comprehensive physico-chemical characterization (morphology, structure, and composition) and electrochemical measurements aimed at evaluating sample activity and stability. Preliminary results will be critically analyzed and presented in internal contexts.

Phase 2 – Advanced experimental activity

The activity will focus on the optimization of electrochemical cells for operando measurements and on the acquisition of experimental data at the synchrotron (BACH beamline – Elettra). In this phase, XAS spectroscopy data will be collected and integrated with complementary techniques such as Raman, XPS, and other surface analyses. The collected data will be analyzed to identify correlations between electronic properties and catalytic activity, enabling iterative optimization of the studied materials. Results will be presented at national and international conferences or workshops.

Phase 3 – Modeling, consolidation, and thesis writing

The activity will be devoted to the in-depth and systematic analysis of the collected data, including comparison among the different systems studied, leading to the writing of the PhD thesis. The results will be critically integrated into a coherent scientific framework, alongside preparation for the final defense and dissemination of outcomes through seminars and conference.

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