

## Preface to Advanced Characterization Technique in Electrocatalysis and Photocatalysis

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The severe burning of fossil fuels brings about a worsening energy crisis and environmental issues which aroused global concern. Electrocatalysis and photocatalysis are promising approaches to store intermittent renewable solar and wind energy in fuels and chemicals, leading to reduced anthropogenic environmental damage. However, the sluggish kinetics of electrocatalysis and photocatalysis is a huge obstacle to the overall efficiency improvement of energy devices. There is still a knotty problem to rational designing high-performance catalysts, which largely depends on a deep understanding of the interface microenvironment and dynamic structure evolution of catalysts during the reaction.

The investigation of local environment of the active site in catalysts is a prerequisite for understanding their activity. Various techniques have been found valuable for the characterization of catalysts. Infrared (IR) and Raman spectroscopy provide important information about the chemical bonds and symmetry of the system by observing the vibrational modes, which are limited to weak detection signal and low sensitivity. Nuclear magnetic resonance (NMR) and electron paramagnetic resonance (EPR) are used to observe the chemical environment of certain species within the sample. The extended X-ray absorption fine structure (EXAFS) is an element selective technique and it can be used for determining the structural parameters of the selected element. However, the behavior of catalysts and reactants during the reaction remains a black box under these *ex-situ* characterizations. Therefore, *in situ* and *operando* techniques for these characterizations are indispensable to comprehend the structural transformation of catalysts and the behavior of the intermediate at interface. By analyzing the atomic structure and electronic state of the active site, the relationship between structure and properties is established, which provides a new perspective for catalytic reaction and promotes *in situ* spectroscopy in various systems. Due to the urgency of the development of advanced characterization techniques, as supported by *Chinese Journal of Structural Chemistry*, a special issue for the **Advanced Characterization Technique in Electrocatalysis and Photocatalysis** was organized by the Guest Editors.

This special issue contains 7 papers and 1 News, which are

related to advanced characterization techniques in electrocatalysis and photocatalysis reactions. We would like to express our faithful appreciation to all the authors for their contribution to this special issue which covers *in-situ* SR-FTIR, soft XAS, *in-situ* HP-STM, *operando* EC-STM, *in-situ* MAS NMR, *in-situ* TEM, *in-situ* SERS and *operando* HERFD-XANES. In the news by Prof. Huang, the lattice strain observed not only affects the electrochemical performance of LMR, but also has implication in other layered cathode materials for Li-ion batteries. The recent progress in the research of dynamic mechanisms for valuable electrocatalytic reactions based on *in-situ* SR-FTIR methodology is summarized methodically as reported by Prof. Liu. The recent developments of soft XAS techniques applied in 2D photo/electrocatalysts mainly focusing on surface active sites have been reviewed by Prof. Zhong. The high pressure (HP) STM and electrochemical (EC) STM for the *in-situ/operando* characterization at solid-gas and solid-liquid interfaces with atomic resolution were introduced by Prof. Song. The development of *in-situ* MAS NMR techniques was outlined and detailed applications in material synthesis and heterogeneous catalysis were discussed by Prof. Hou. The recent advances in *in-situ* TEM together with 3D ET for catalyst studies were reviewed by Prof. Han. The role of temperature change in hotspots for the conversion of p-aminothiophenol on Au by employing variable-temperature SERS measurements was studied by Prof. Huang. The structural evolution of NiPc with uniform Ni-N<sub>4</sub>-C<sub>8</sub> moiety during the reaction tracked by *operando* HERFD-XANES was studied by Prof. Jiang.

We hope this special issue could provide the readers with an innovative recognition of relationship between structure and performance during the reaction. We have learned a lot about interesting *in-situ* and *operando* characterization techniques in this special issue. Undoubtedly, the content in this issue is just a start, but we will do better in future issues, and look forward to our new contributions as this journal grows. We do hope that readers will enjoy the range of topics presented here and perhaps initiate ideas to push their research to the next stage.



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