

Applications of X-ray Spectroscopy for in situ Study of CO₂ Conversion Electrocatalysts

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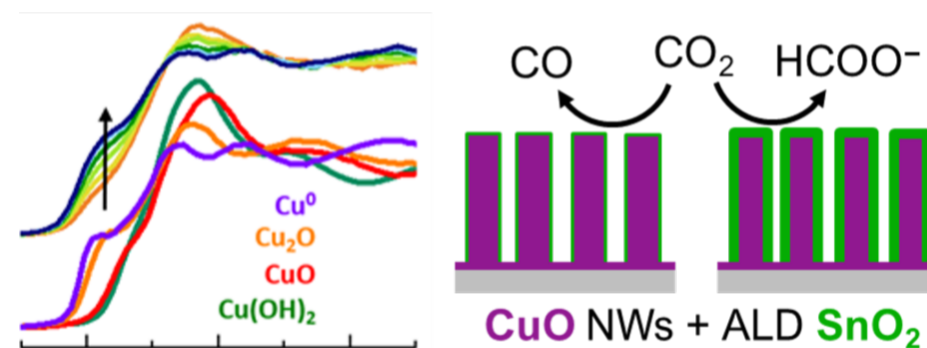
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Recent developments in electrocatalyst design for carbon dioxide conversion are revealing that various design principles -- such as catalysts based on metal oxides, doped metals or metal alloys, and metal atoms in molecular coordination environments -- demonstrate behaviors which differ from their simple metal counterparts, revealing strategies toward enhancing selectivity toward high-value products while suppressing undesired ones. Continued rational development of catalysts demands that we have a detailed understanding of the structure-function relationships which dictate selectivity. However, under the harsh reaction conditions of CO₂ reduction (e.g. highly negative potential, local pH extremes) many of these catalysts are prone to significant structure changes, making it difficult to understand the true catalytically active form of the electrode materials. "Post mortem" analyses often fail to accurately represent the active form of catalysts, so methods are demanded which are capable of examining the electrode during operation, e.g. in situ or operando.

X-ray absorption spectroscopy (XAS) techniques can be uniquely powerful in investigating electrochemical systems under operating conditions. The high energies of X-ray photons can enable them to be used under ambient conditions and to pass through liquid electrolyte. With a tunable energy source (e.g. synchrotron), different elements can be selectively probed due to their distinct absorption edges. A wide range of information can be revealed using X-ray spectroscopy methods, including composition, oxidation states, and local coordination environment. But in situ XAS is usually bulk sensitive, whereas catalysis occurs at surfaces, so complimentary surface-sensitive methods such as X-ray photoelectron spectroscopy (XPS) are valuable. When conducted using "quasi in situ" methods, XPS can provide a good compromise between surface sensitivity and in situ conditions. Performing both XAS and XPS allows one to gain a detailed understanding of dynamic electrocatalysts. In this talk I will explain the approaches we use for both, including their pros and cons, in the framework of our study on Cu-Sn catalysts[1] with compositions tunable to achieve selective CO₂ conversion to either carbon monoxide or formate.

References:

[1] Schreier, M. et al. Solar conversion of CO₂ to CO using Earth-abundant electrocatalysts prepared by atomic layer modification of CuO. *Nature Energy* 2, 17087 (2017).

Acknowledgements:

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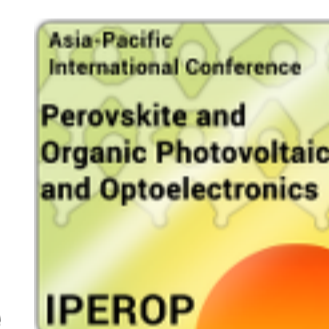
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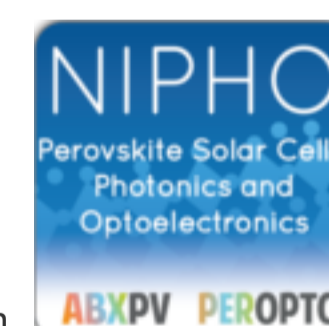
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