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Quantitative measurement of gas products during electrochemical CO₂ reduction

The electrochemical CO₂ reduction reaction (CO₂RR) powered by renewable energy to produce valuable chemicals and fuels has become a promising strategy for using renewable energy to produce fuels and chemicals. The process can occur under ambient temperature and normal pressure conditions. This approach potentially aids in mitigating the climate change problem by initiating a carbon-neutral cycle. However, it is a significant challenge to obtain mechanistic information about the CO₂RR, as this is complicated by the multiple possible proton-electron transfer pathways and associated intermediates, and also accompanied by the competing hydrogen evolution reaction (HER). It is widely reported that the CO₂RR performance (such as activity and selectivity) is strongly dependent on the nature and structure of catalysts and electrolytes. High-resolution correlative characterization plays a central role in interrogating the complex electrochemical reactions that involve multiple pathways and various products.

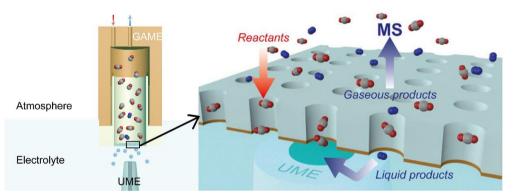
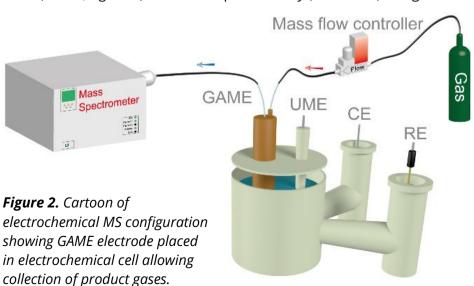


Figure 1. Game electrode. Lefthand side showing hollow structure of electrode leading to efficient collection of product species at the gas/liquid interface.

In relation to the CO₂RR, the mechanism and kinetics have not been fully understood, predominantly limited by the unclear electrochemistry-product distribution relationship. Recently, Dr. Guohui Zhang, Youxin Cui and Prof. Anthony Kucernak from Imperial College London have developed tools for real-time and in-situ measurements of the CO₂RR. Combining the recently developed "gas accessible membrane electrode" (GAME, figure 1) with mass spectrometry (GAME-MS) using a **Hiden**

Analytical QGA system, the evolution of gaseous products can be tracked synchronously during the electrochemical CO₂RR, figure 2. From the synchronized electrochemical-mass spectrometric results, the evolution of diverse CO₂RR products (H₂, C₂H₄, CH₄) is elucidated, providing insights about the onset potentials for individual





products and the Tafel slopes for the overall reaction, figure 3. It should also be highlighted that the estimated Tafel slopes for individual products can be obtained from the MSCVs enabled by the GAME-MS, which are critical to the interpretation of the electrokinetic behaviour. Such information will be useful in revealing the possible reaction pathways leading to these products. These approaches can be extended to study many other electrochemical reactions, providing insights for fundamental understanding and practical applications.

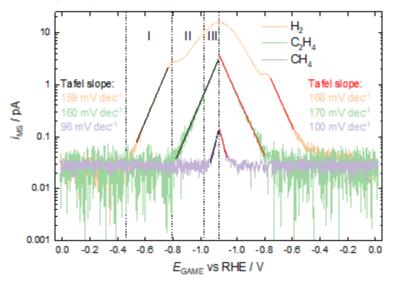


Figure 3. Mass spectrometric detection of product gases during cyclic voltammetry of a nanostructured copper electrode recorded in 0.5 M KHCO₃ at a scan rate of 1 mV s⁻¹ using a pure CO₂ atmosphere as a carrier gas.

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