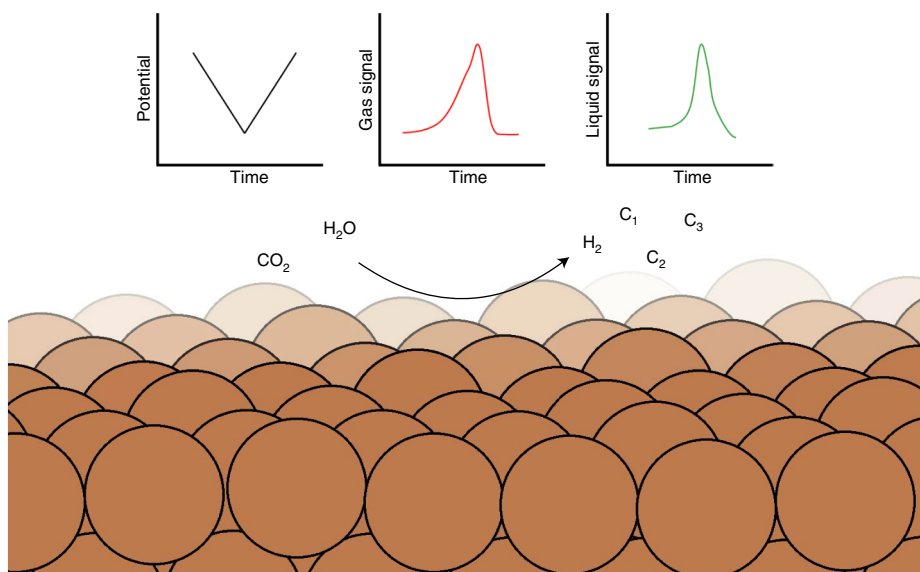


## CARBON DIOXIDE ELECTROREDUCTION

### Products in real time

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Electrocatalytic processes often involve transformations and restructuring that can only be captured by measurements under operando conditions. These dynamic processes can induce the formation of certain products under very specific transient conditions. Furthermore, complex reaction mechanisms encompass the formation of intermediate species that often cannot be detected, since current analytical techniques do not offer the required temporal resolution. Now, Ioannis Katsounaros, Payman Khanipour and co-workers at the Helmholtz Institute Erlangen-Nürnberg for Renewable Energy present a technique to determine the formation of gaseous and liquid products of electrochemical reactions in real time.

The electrochemical real-time mass spectrometry (EC-RTMS) approach possesses high potential and temporal resolution — one to a few seconds — and sensitivity. The setup consists of a scanning flow cell where the gaseous products are first extracted from the electrolyte outlet stream using a hydrophobic membrane. The degassed electrolyte is subsequently nebulized and the mist containing the liquid products is extracted using a spray chamber. The gaseous and liquid products are

analysed by mass spectrometry in parallel to the electrochemical measurement.

The power of EC-RTMS is showcased by studying the electrochemical reduction of CO<sub>2</sub> on a mechanically polished Cu electrode in 0.1M KHCO<sub>3</sub> electrolyte. The researchers alternate potential pulses at  $-0.2$  (before the reaction onset) and  $-1.1$  V (relevant for C<sub>2+</sub> formation) versus the reversible hydrogen electrode of 120 and 60 s, respectively, and monitor the formation of C<sub>1</sub>, C<sub>2</sub> and C<sub>3</sub> gaseous and liquid products. The results are consistently reproduced after each pulse cycle. After anodizing the surface at 1.2 V during 60 s and repeating the alternate pulses, the formation of C<sub>2+</sub> products is found to increase, whereas that of C<sub>1</sub> products is suppressed. These results are further confirmed by cyclic voltammetry complemented with EC-RTMS on the pristine and anodized Cu electrode, and are ascribed to the formation of oxidized copper species after anodization, as demonstrated by X-ray photoelectron spectroscopy measurements.

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