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Anode Catalysts for Electrocatalytic Alcohol Oxidation Coupled to CO₂ Reduction in Continuous-Flow Electrolyzers

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Abstract

Thanks to the intense scientific interest, the field of CO₂ electrolysis witnessed remarkable progress (industrially relevant partial current densities, selective electrocatalysts towards small molecule products, multi-cell stacks, etc.) in the past decade. However, despite all these efforts, the energy efficiency of CO₂ electrolyzers is still relatively low, which is rooted in the large cell voltages. A reason behind this is that the kinetically sluggish water oxidation (OER) is performed at the anode in parallel with CO₂ reduction (CO₂RR) at the cathode. This issue can be circumvented by coupling CO₂RR with alternative anode processes, resulting in smaller cell voltage (better energy efficiency) along with the possible generation of products at the anode with high market value. Thus, maximizing the potential of utilizing waste carbon sources to generate value-added products. Such alternative reactions include, for example, the electrocatalytic oxidation of chloride ions, aliphatic and aromatic alcohols, amines, urea, hydrazine, and numerous biomass-derived substances such as 5-(hydroxymethyl)furfural, sorbitol, etc.

In this study, CO₂RR to CO was driven at the cathode, and the electrocatalytic oxidation of glycerol (GOR) was performed at the anode of a small (1 cm² active surface area) microfluidic electrolyzer cell. Ir black was replaced by carbon-supported Pt nanoparticles, while Ag nanoparticles were used as the cathode catalyst. During our experiments, we identified four main factors influencing the GOR activity and selectivity: i) the quality and quantity of the used ionomer, ii) the electrolyte flow rate, iii) glycerol concentration, and iv) the applied anode potential. The latter was particularly important since Pt showed the highest activity toward GOR in a potential window where its surface starts to oxidize. The formation of a continuous oxide layer has to be avoided since it inhibits the alcohol oxidation reaction. By setting optimal conditions, a close to 100% Faradic efficiency (FE) toward CO formation on the cathode and between 70-80% FE toward GOR was achieved above 200 mA cm⁻² total current density. The amount of the formed products were tracked by mass spectrometry, gas-, and high-performance liquid chromatography, and nuclear magnetic resonance spectroscopy. Dihydroxyacetone, glycerate, glycolate, oxalate, tartronate, formate, and lactate were identified as GOR products. The missing charge was attributed to the formation and a small amount of CO₂. To improve the stability and selectivity of Pt in GOR, semipermeable metal oxide layers were introduced to the top of the catalyst layer. The thin films were synthesized by electrodeposition in different thicknesses. Based on our measurements, a set of criteria were defined regarding the composition of the anode catalyst layer (ionomer, co-catalyst

of criteria were defined regarding the composition of the anode catalyst layer (ionomer, co-catalyst overlayer, etc.), the composition of the electrolyte solution, and the applied electrochemical protocol to allow stable and selective GOR at high achievable current densities.

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