Int. J. Electrochem. Sci., 16 **(2021)** Article ID: 210240

International Journal of
ELECTROCHEMICAL
SCIENCE
www.electrochemsci.org

In-Situ and Real-Time Monitoring of Oxygen Evolution during Kolbe Reaction by Scanning Electrochemical Microscopy

Zemin Yu[1], *Yuanbo Wang*[1,2], *Xingyan Cao*[2], *Yihan Li*[2], *Tianen Ma*[2], *Liqiu Zhang*[2,*], *Lichun Liu*[2], *Hongyan Yue*[1,*]

[1] School of Materials Science and Engineering, Harbin University of Science and Technology, Harbin 150040, China.
[2] Nanotechnology Research Institute & College of Biological, Chemical Science and Engineering, Jiaxing University, Jiaxing, 314000, China
[*]E-mail: liqiu0524@zjxu.edu.cn, hyyue@hrbust.edu.cn

doi: 10.20964/2021.02.31

Received: 9 November 2020 / *Accepted:* 12 December 2020 / *Published:* 31 December 2020

In this study, the oxygen evolution during the Kobe reaction was *in-situ* and real-time monitored by scanning electrochemical microscopy (SECM) in tip-generation substrate-collection (TG/SC) mode. A typical Kolbe reaction involving the acetate electro-oxidation was used as a study model. To this end, the TG/SC method of acetate electro-oxidation was performed at a 10 μm-diameter tip Pt disk microelectrode positioned ~1.2 μm away from a 50 μm-diameter substrate Pt disk microelectrode. The substrate Pt was employed as an efficient catalyst to monitor the byproduct O_2 produced at the tip by the catalytic reduction at an appropriate electrode potential. The linear scanning voltammetry (LSV) results indicated that O_2 evolution during acetate oxidation depended on both the electrode potential and acetate concentration. At low potentials (<2.4V vs. Ag/AgCl), the current of O_2 evolution dominated the acetate oxidation reaction in 0.1 M $HClO_4$ electrolyte, while O_2 evolution was inhibited at the joint condition of higher potentials (>2.4V) and elevated concentrations of acetate, resulting in better current efficiencies of acetate oxidation. The inhibition effect of O_2 evolution was also confirmed in alkaline electrolytes, consistent with the reported literature dealing with other traditional detection techniques. In sum, the proposed detection technique based on *in-situ* and real-time dynamic monitoring of oxygen evolution was accurate and sensitive, thereby promising for the study of broad range of reactions involving the generation of oxygen species.

Keyword: Microelectrodes, Kolbe reaction, oxygen evolution, SECM, TG/SC

FULL TEXT