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Fabrication and characterisation of electrodeposited reduced graphene oxide modified molybdenum microdisk electrode for the reduction of dissolved carbon dioxide in aqueous solutions

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[Shafiee, Saiful Arifin](#)^a; [Adlee, Siti Aisyah Saiful](#)^a; [Rashidi, Daniel Mohamad](#)^a; [Mahat, Mohd Muzamir](#)^{b, c}; [Ismail, Mohamad Wafiuddin](#)^a; [+7 authors](#)

^a Department of Chemistry, Kulliyah of Science, International Islamic University Malaysia, Jalan Sultan Ahmad Shah, Bandar Indera Mahkota, Pahang, Kuantan, 25200, Malaysia

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Abstract

The rising atmospheric carbon dioxide (CO₂) concentration and its dissolution in aquatic systems have intensified interest in electrochemical sensing technologies capable of sensitive detection of dissolved CO₂. To address this, molybdenum (Mo) microdisk electrodes were surface modified with reduced graphene oxide (rGO) via a one-step electrochemical deposition method, following

graphene oxide synthesis using a modified Hummers' approach. The morphology, elemental composition, and crystalline phases were characterised using scanning electron microscopy, energy dispersive X-ray, X-ray diffraction, and X-ray photoelectron spectroscopy confirming a successful rGO deposition alongside minor molybdenum oxide (MoO₂) phases on the electrode surface. The scalable electrodeposition process employed herein eliminates the need for chemical reductants, offering an environmentally benign route for the development of graphene-based electrocatalysts. The cyclic voltammetry evaluation, conducted at a scan rate of 0.1 V s⁻¹ within a potential window of -1.25 to 0.25 V (vs Ag/AgCl), demonstrates an enhanced CO₂ reduction activity in 0.5 M sodium bicarbonate (NaHCO₃) at -1.0 V vs Ag/AgCl compared with bare Mo, evidenced by higher cathodic currents, a linear response to CO₂ bubbling time ($R^2 > 0.9981$) from 2 to 20 min, and quasi-reversible electron transfer behaviour. The electrodes displayed high reproducibility with relative standard deviation, RSD = 1.57%. This research work aims at proposing a low cost, facile, sensitive and selective assembly of three electrode electrochemical reduction of dissolved CO₂ which operates at room temperature in ambient conditions and highlights the potential for future studies exploring rGO integration on other metal microelectrodes. © 2026 Elsevier B.V.

Author keywords

Carbon dioxide reduction; Dissolved carbon dioxide gas; Electrochemical sensor; Electrodeposition; Graphene oxide; Reduced graphene oxide

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Corresponding authors

Corresponding author

W.H. Danial