S-containing Molybdenum Oxide Films as pH Neutral Hydrogen Evolution Electrocatalyst prepared by Electrodeposition

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Molybdenum sulfides, molybdenum oxides, and S-containing molybdenum oxides have been shown to be promising electrocatalysts for hydrogen evolution. In this context, amorphous Mo–O–S films on a fluorine-doped tin oxide (FTO) substrate were prepared by cyclic voltammetry using two different electrodeposition baths containing different Mo and S precursors. Although ammonium tetrathiomolybdate (ATTM) was successfully used as a source for Mo and S, this precursor was identified as having some limitations. Therefore, an alternative electrodeposition bath containing thiourea and ammonium molybdate was presented in this work. Amorphous thiourea-molybdate-based films were annealed under an inert atmosphere, and the thermal effect was subsequently evaluated by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), UV-Vis, and Raman analysis. Finally, harsh galvanostatic electrolysis conditions were applied to perform a preliminary evaluation of the catalytic behaviour of the Mo–O–S films in a phosphate buffer solution (pH 7). The Mo–O–S films prepared from the thiourea-molybdate bath showed promising stability and activity results, with a Tafel slope of 85 mV·dec⁻¹ in the range of overpotentials lower than 100 mV even after prolonged HER operation. These findings open an alternative approach to the possible mechanism of Mo–O–S formation.

Keywords: molybdenum oxide; molybdenum sulfide; thiourea; electrodeposition; hydrogen evolution reaction

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