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Edge sites of MoS₂ catalysts are active for both HDS and HER reactions: the proof of concept

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Introduction. Since the pioneer work of Jaramillo et al. in 2007¹, MoS₂ has been considered promising to replace platinum (Pt) for the Hydrogen Evolution Reaction (HER) electrocatalysis. If the activity of MoS₂ has been linked with the total edge site concentration for both hydrotreatment reactions and HER reaction, no evidence has been given yet that well-performing MoS₂ catalysts for hydrotreatment reactions could lead to improved HER catalysts as well. In this study, we compare the thiophene hydrodesulfurization (HDS) and HER activities of a series of MoS₂ catalysts prepared with or without citric acid (CA) and with or without cobalt (Co) or Pt single atoms as promoter.

Experimental/methodology. Three series of mono- bi- and trimetallic (MoS₂/C, Co-MoS₂/C and Pt1%-CoMoS₂/C) catalysts with different amounts of CA were prepared by an incipient wetness impregnation method². The thiophene HDS tests were carried out within a differential glass reactor at 623 K. Electrochemical measurements were carried out using a bi-potentiostat (Autolab PGSTAT302N) with a catalyst loading of 300 μg_{powder} cm⁻².

Results and discussion. The comparison between the activity in thiophene HDS and HER on MoS₂ catalysts (Figure 1) reveals that for both reactions the addition of CA is beneficial on the three composition series up to an atomic ratio of CA/metal of 2. This positive effect could be linked to a decrease in slab length as revealed by High Angle Annular Dark Field Scanning Transmission Electron Microscopy. The introduction of Co or (Co+Pt) further increases the activity towards both reactions, the best catalyst being Pt1%-CoMoS₂/C prepared with the atomic ratio of CA/metal of 2. This parallel in activity ranking is in agreement with active sites being the same for both reactions, providing rational guidelines on how to improve the HER activity.

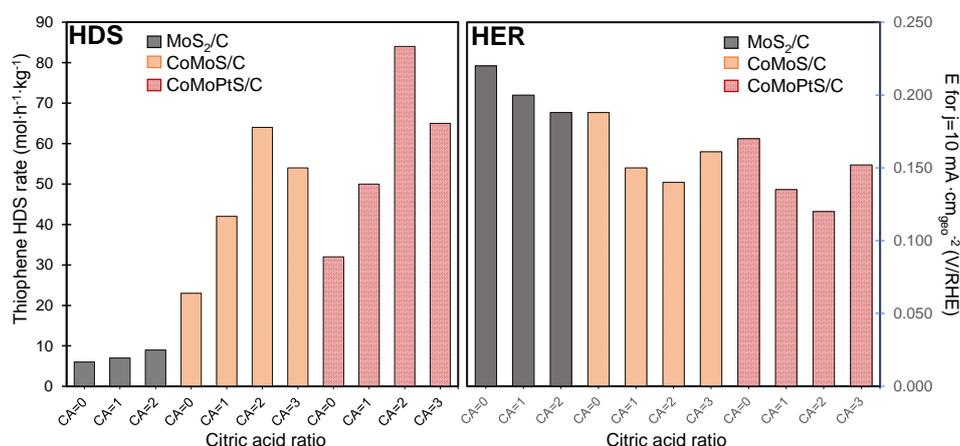


Figure 1. Effect of promotion and citric acid addition in MoS₂/C activity for HDS and HER reactions.

Finally, the dissolution of Mo, Co, and Pt elements during HER was monitored *in situ* using an electrochemical flow cell connected to an inductively coupled mass spectrometer for the best-tested catalyst. Mo dissolves, particularly in the first cycles in agreement with the leaching of partially sulfided Mo entities². Thus, improving the Mo sulfidation degree should lead to more stable catalysts.

References

[1] Jaramillo, T.; Jørgensen, K. P.; Bonde, J.; Nielsen, J.H.; Horch, S.; Chorkendorff, I. *Science* **2007**, 317, 100-102.

[2] Zavala, L. A.; Kumar K.; Martin V.; Maillard F.; Maugé F.; Portier X.; Oliviero L. * and Dubau L. * *ACS Catal.* **2023**, 13, 1221–1229.