

Supporting Information

Investigating the Structural Evolution and Catalytic Activity of *c*-Co/Co₃Mo Electrocatalysts for Alkaline Hydrogen Evolution Reaction

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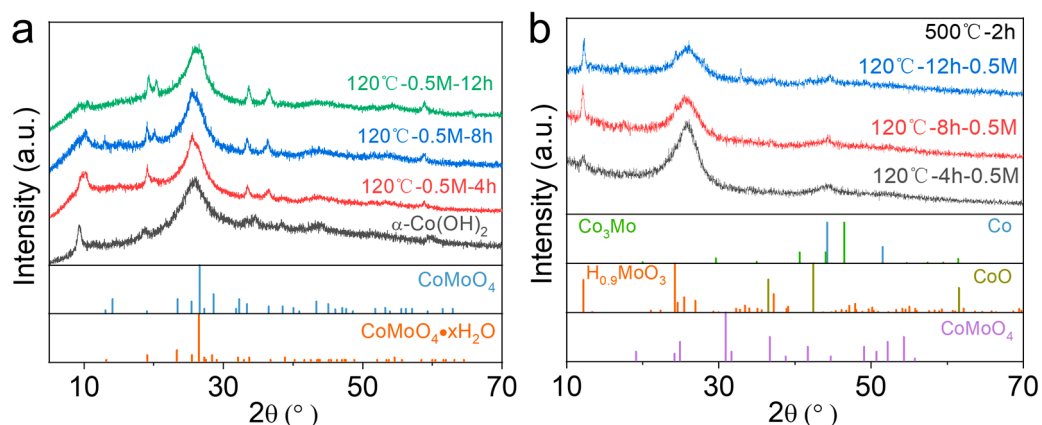


Figure S1. (a) The XRD patterns of sample-120°C-4h-0.5M, sample-120°C-8h-0.5M and sample-120°C-12h-0.5M. (b) The corresponding XRD patterns of catalysts annealed at 500 °C for 2 h in a mixed hydrogen-argon flow.

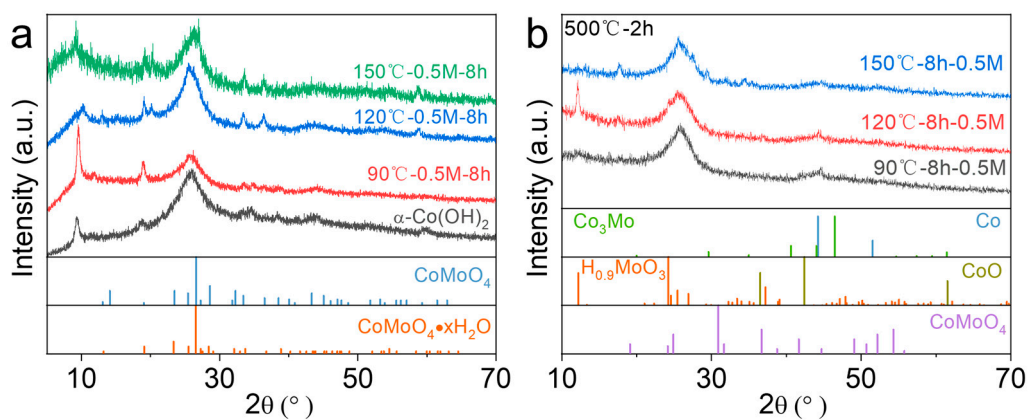


Figure S2. (a) The XRD patterns of sample-90°C-8h-0.5M, sample-120°C-8h-0.5M and sample-150°C-8h-0.5M. (b) The corresponding XRD patterns of catalysts annealed at 500 °C for 2 h in a mixed hydrogen-argon flow.

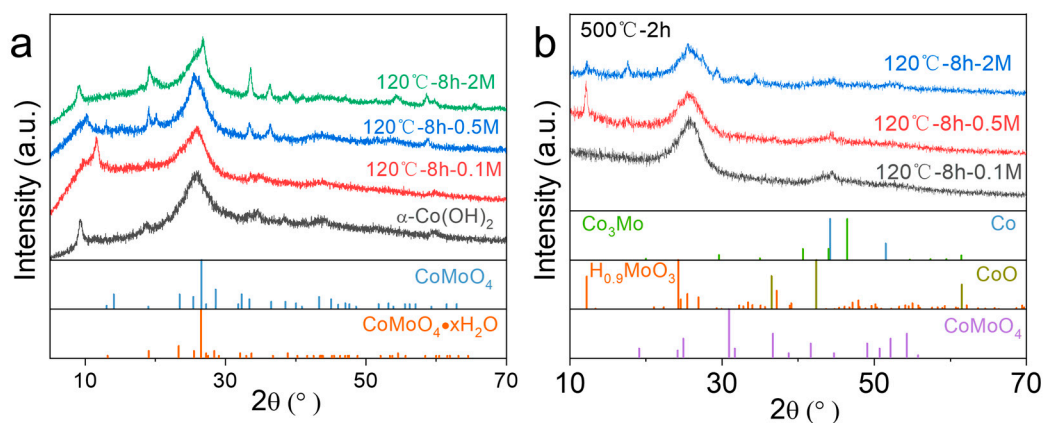


Figure S3. (a) The XRD patterns of sample-120°C-8h-0.1M, sample-120°C-8h-0.5M and sample-120°C-8h-2M. (b) The corresponding XRD patterns of catalysts annealed at 500 °C for 2 h in a mixed hydrogen-argon flow.

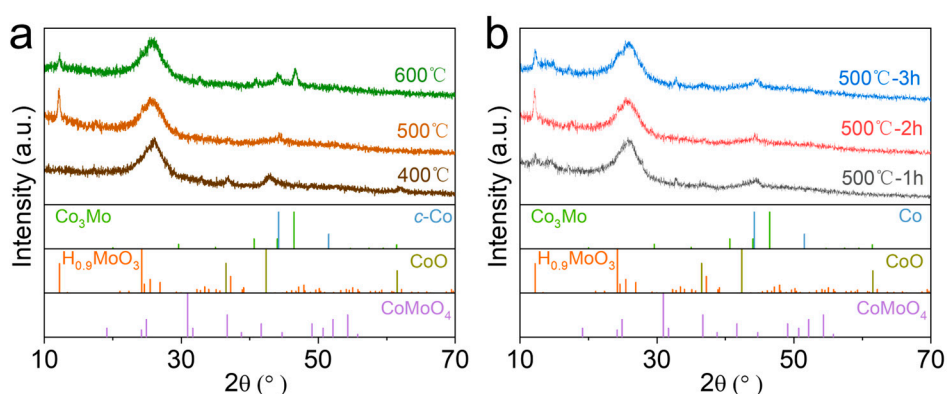


Figure S4. The XRD patterns of $c\text{-Co/Co}_3\text{Mo}$ catalysts obtained by annealing sample-120°C-8h-0.5M at (a) 400°C, 500°C, and 600 °C for 2 h in a mixed hydrogen-argon flow, respectively; (b) 500 °C for 1, 2, and 3h in a mixed hydrogen-argon flow, respectively.

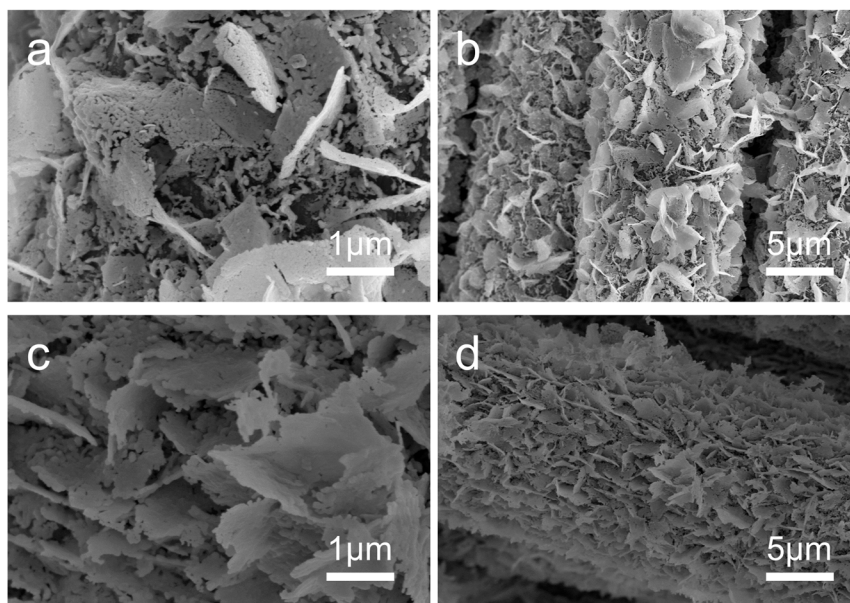


Figure S5. SEM images of catalysts obtained by annealing (a, b) sample-90°C-8h-0.5M and (c, d) sample-150°C-8h-0.5M at 500 °C for 2 h in a mixed hydrogen-argon flow, respectively.

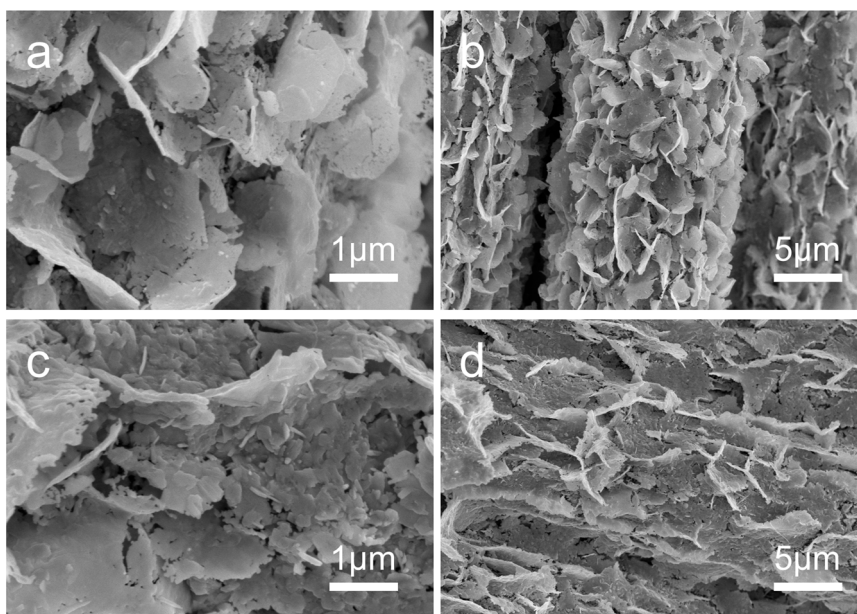


Figure S6. SEM images of catalysts obtained by annealing (a, b) sample-120°C-4h-0.5M and (c, d) sample-120°C-12h-0.5M at 500 °C for 2 h in a mixed hydrogen-argon flow, respectively.

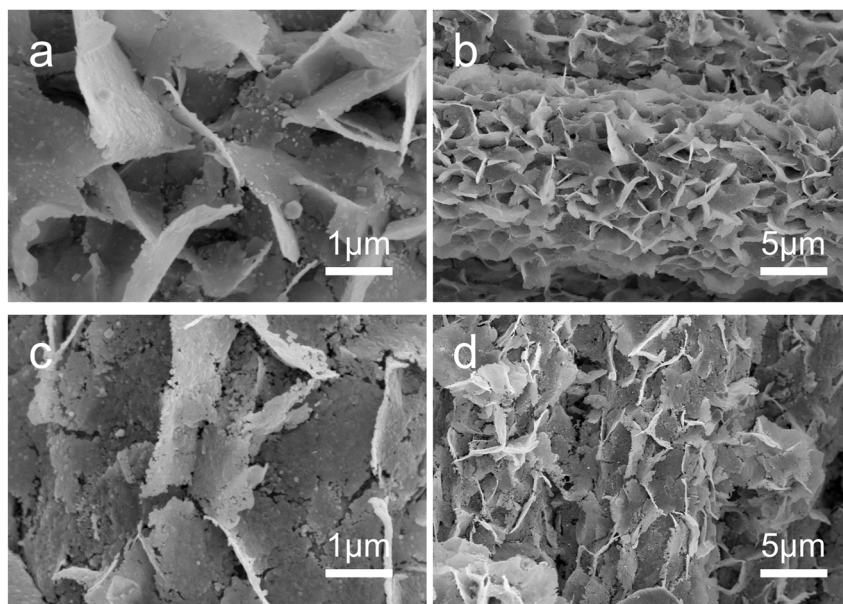


Figure S7. SEM images of catalysts obtained by annealing sample-120°C-8h-0.5M at (a, b) 400 °C and (c, d) 600 °C for 2 h in a mixed hydrogen-argon flow, respectively.

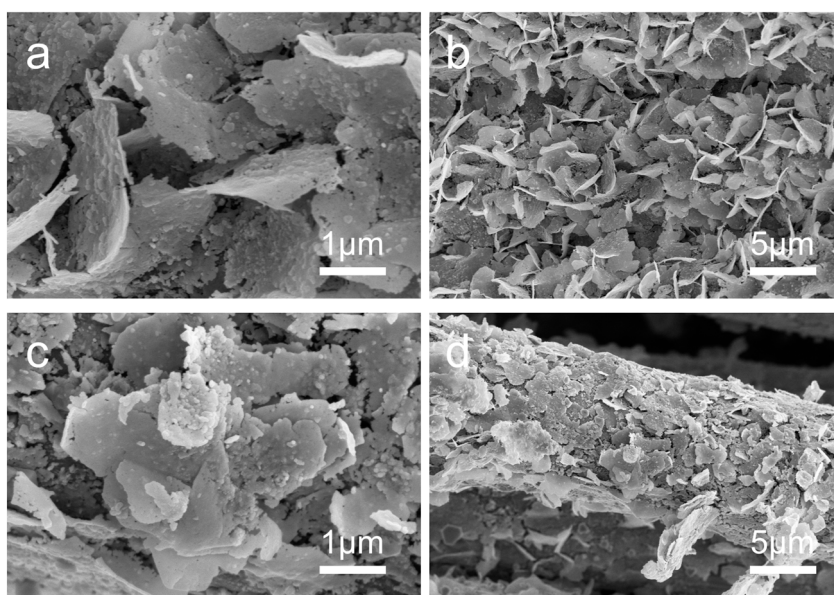


Figure S8. SEM images of catalysts obtained by annealing sample-120°C-8h-0.5M at 500 °C for (a, b) 1 h and (c, d) 3 h in a mixed hydrogen-argon flow, respectively.

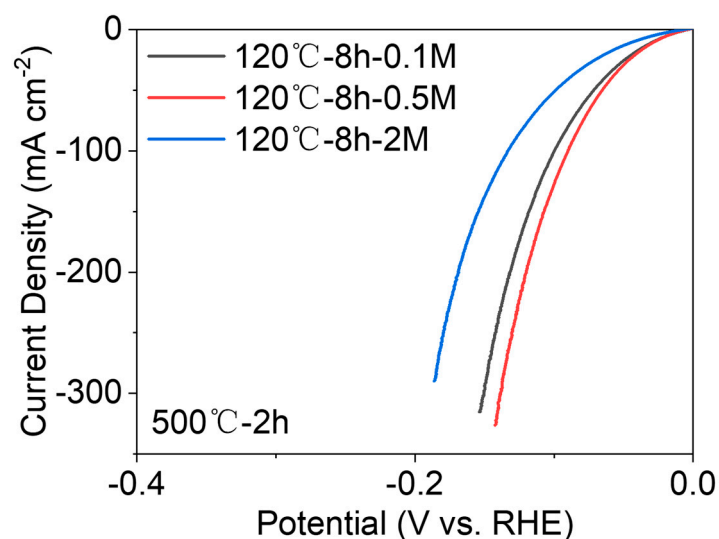


Figure S9. LSV curves of catalysts obtained by annealing sample-120°C-8h-0.1M, sample-120°C-8h-0.5M, and sample-120°C-8h-2M at 500 °C for 2 h in a mixed hydrogen-argon flow, respectively.

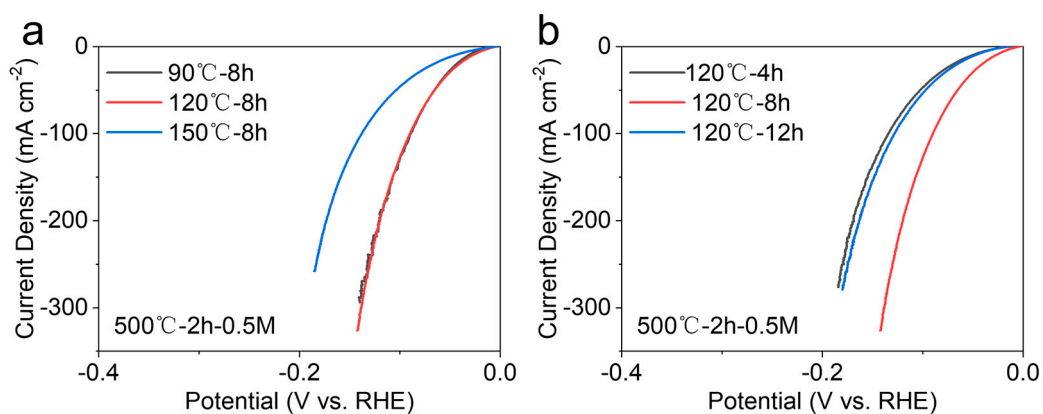


Figure S10. (a) LSV curves of catalysts obtained by annealing sample-90°C-8h-0.5M, sample-120°C-8h-0.5M, and sample-150°C-8h-0.5M at 500 °C for 2 h in a mixed hydrogen-argon flow, respectively. (b) LSV curves of catalysts obtained by annealing sample-120°C-4h-0.5M, sample-120°C-8h-0.5M, and sample-120°C-12h-0.5M at 500 °C for 2 h in a mixed hydrogen-argon flow, respectively.

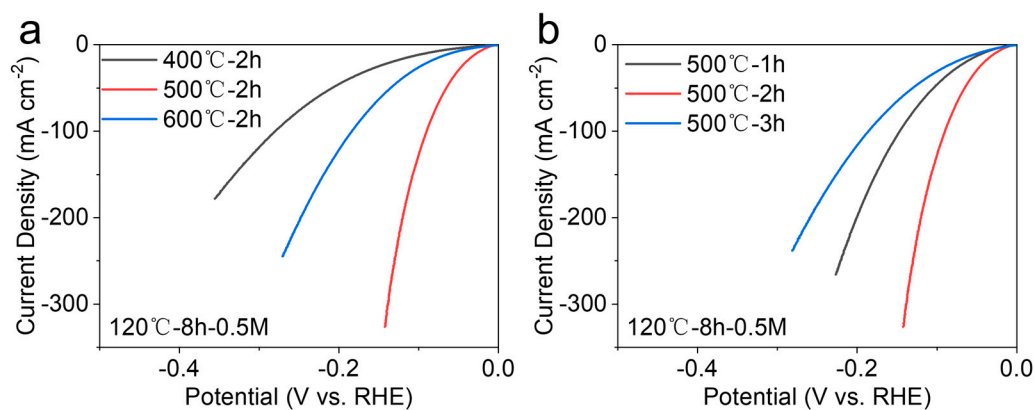


Figure S11. (a) LSV curves of catalysts obtained by annealing sample-120°C-8h-0.5M at 400, 500, and 600 °C for 2 h in a mixed hydrogen-argon flow, respectively. (b) LSV curves of catalysts obtained by annealing sample-120°C-8h-0.5M at 500 °C for 1, 2, and 3 h in a mixed hydrogen-argon flow, respectively.

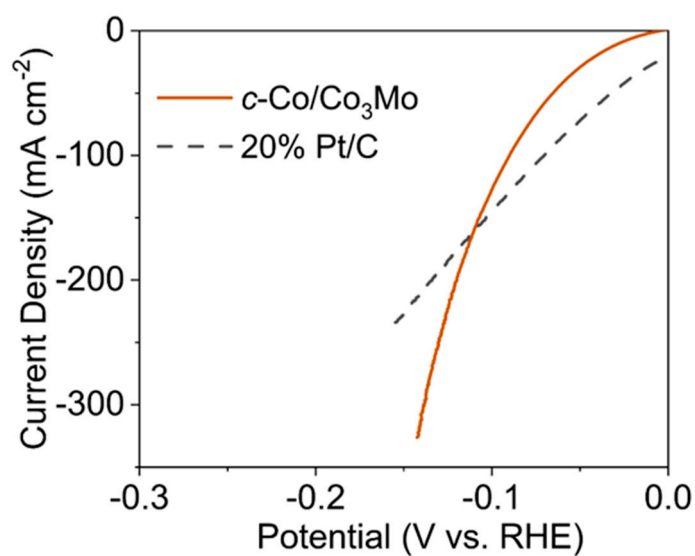


Figure S12. LSV curve of 20% Pt/C and *c*-Co/Co₃Mo electrodes.

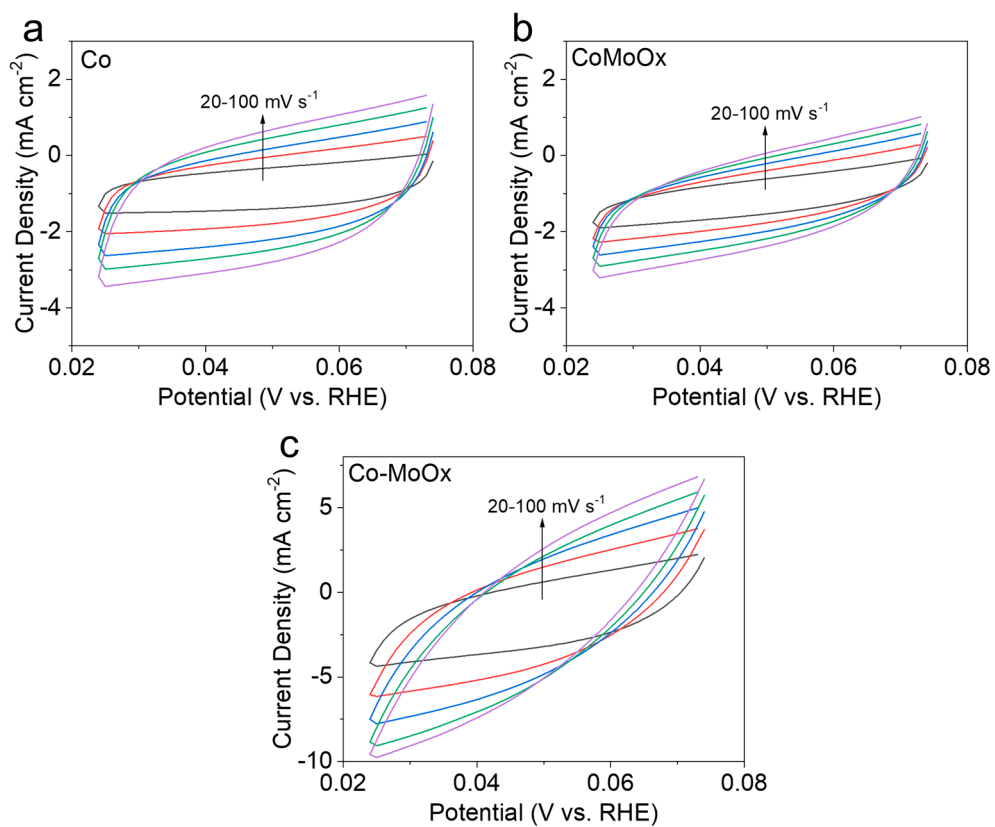


Figure S13. CV curves of (a) *c*-Co, (b) *c*-Co/Co₃Mo, and (c) Co₃O₄/CoMoO₄ at varied scan rates.

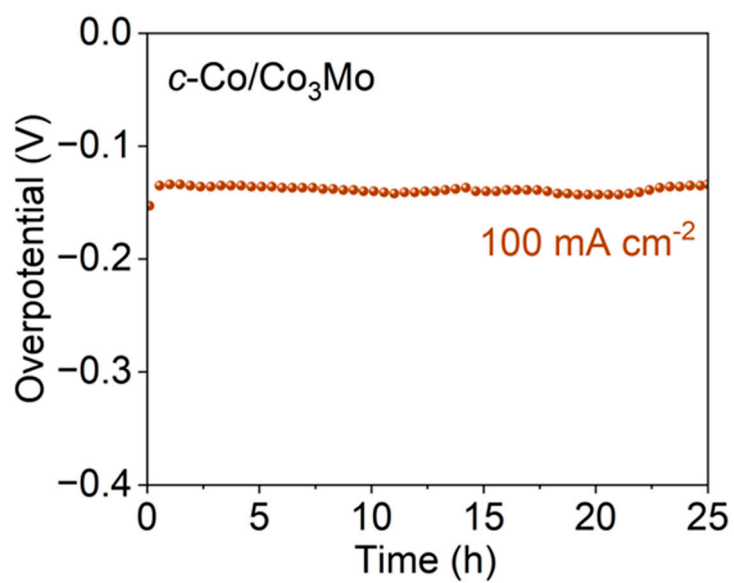


Figure S14. Chronopotentiometry curve of *c*-Co/Co₃Mo at a constant current density of 100 mA cm^{-2} .

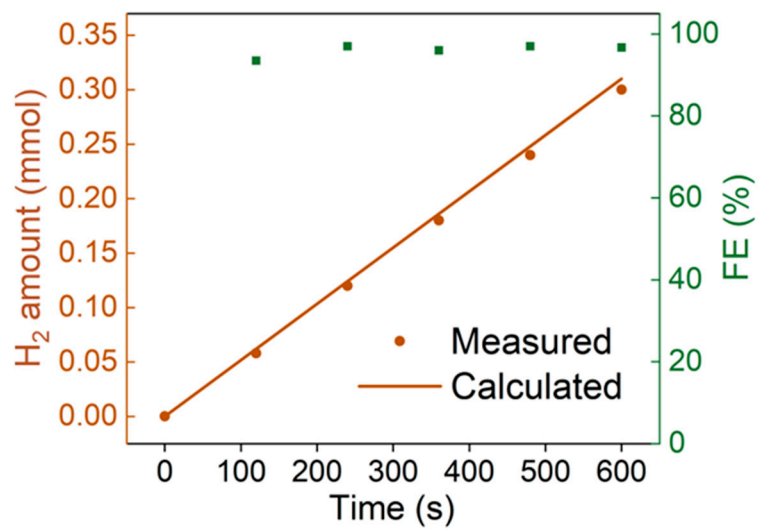


Figure S15. Faraday efficiency of *c*-Co/Co₃Mo at 1 M KOH.

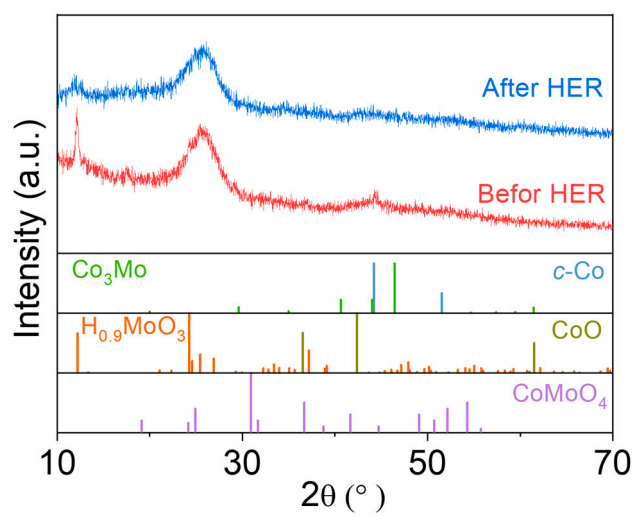


Figure S16. XRD patterns of *c*-Co/Co₃Mo before and after HER test.

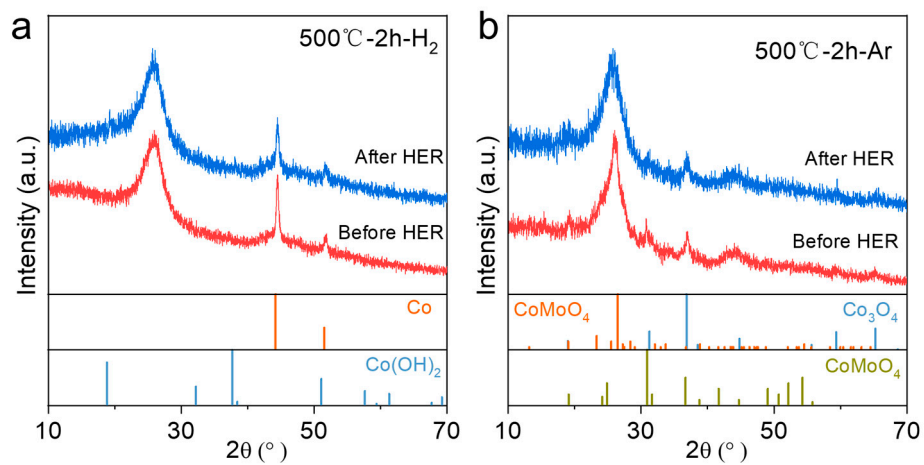


Figure S17. XRD patterns of (a) *c*-Co and (b) $\text{Co}_3\text{O}_4/\text{CoMoO}_4$ before and after HER test.

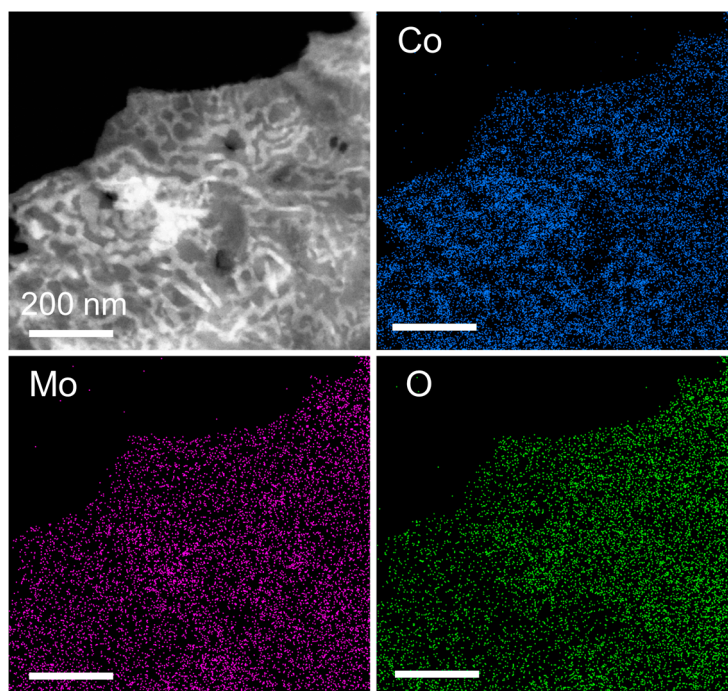


Figure S18. EDX elemental mapping images of *c*-Co/ Co_3Mo after HER test.

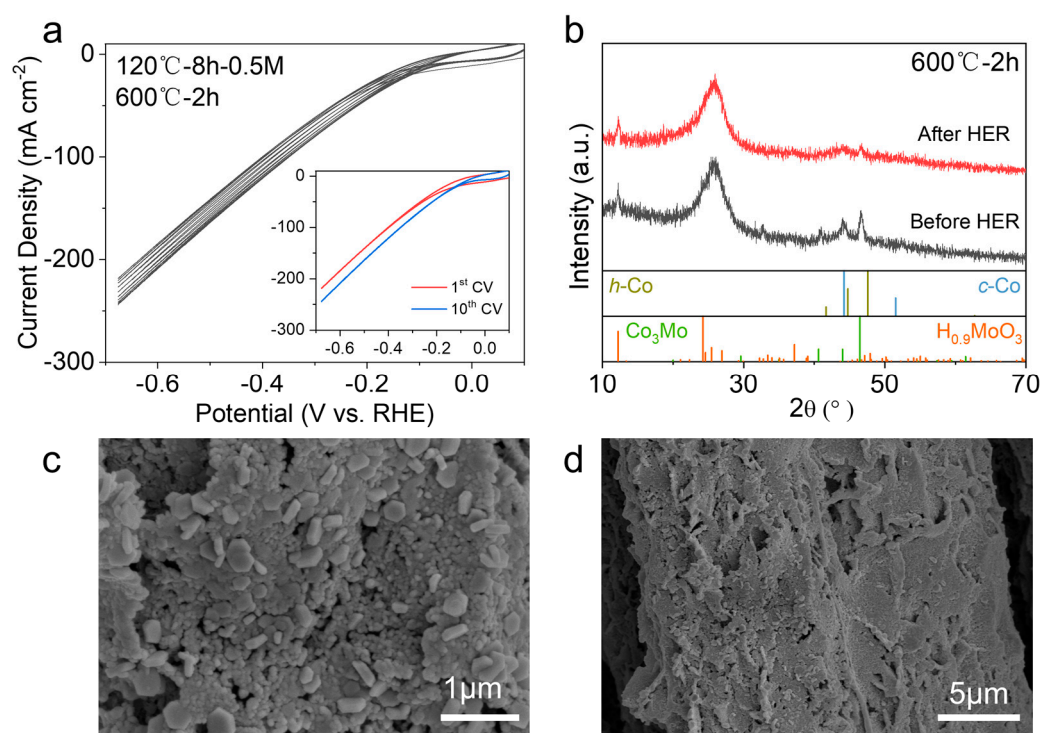


Figure S19. (a) Evolutive CV curves from the 1st to the 10th CV in 1 M KOH at 50 mV s⁻¹ between 0.124 and -0.676 V (vs RHE), (b) XRD patterns, (c, d) The SEM image after HER test was obtained when the sample was annealed at 120°C-8h-0.5M, 600°C, in a hydrogen-argon mixed flow for 2 h.

Table S1. The results of EDX for the c-Co/Co₃Mo catalyst.

Element	Wt %	Atomic %
O	24.30	56.01
Co	61.76	38.64
Mo	13.94	5.36

Table S2. The comparison of HER performance for c-Co/Co₃Mo with some related reports.

Catalyst	Overpotential at 10 mA cm ⁻² (mV)	References
c-Co/Co ₃ Mo	28	This work
RCMO@NF	68	<i>ACS Appl. Mater. Interfaces</i> , 2019 , <i>11</i> , 9002– 9010
Co ₃ Mo/CoMoO ₃ NPS	34	<i>J. Mater. Sci. Technol.</i> , 2023 , <i>137</i> , 184-192
NF/CoNiMo-500	34	<i>Chem. Eng. J.</i> , 2022 , <i>450</i> , 138206
PVP- MoS ₂ -RGO	66	<i>Small</i> , 2018 , <i>14</i> , e1803361
CoP@3DOM-FeP	67.2	<i>J. Mater. Chem. A</i> , 2021 , <i>9</i> , 23574-23581
Mn ₂ P-MnP/PNC	63	<i>Chem. Eng. J.</i> , 2023 , <i>469</i> , 143879
PdSe ₂	138	<i>Adv. Funct. Mater.</i> , 2021 , <i>31</i> , 2102321

Table S3. TOF for c-Co/Co₃Mo at overpotentials of 100, 150, and 200 mV for HER.

Overpotential	100 mV	150 mV	200 mV
TOF s ⁻¹	0.052	0.14	0.24