Electronic Supplementary Material (ESI) for New Journal of Chemistry. This journal is © The Royal Society of Chemistry and the Centre National de la Recherche Scientifique 2023

Electronic Supplementary Information (ESI)

Photo-electro concerted catalysis of highly active Pt/CoP/C

Nanocomposite for Hydrogen Evolution Reaction

Yanzhu Ye^{a,b}, Yixiang Ye^b, Jiannan Cai^b, Zhongshui Li^{b,*}, Shen Lin^{b,*}

^a Fujian Institute of Education, Fuzhou 350001, China

^b College of Chemistry & Materials Science, Fujian Normal University, Fuzhou

350007, China

* Corresponding author at: College of Chemistry & Materials Science, Fujian Normal

University, Fuzhou 350007, China. E-mail address: zsli@fjnu.edu.cn (Z.S. Li);

shenlin@fjnu.edu.cn (S. Lin).

2. Experimental

2.1. Characterization

The composition of the composites was characterized by X-ray diffraction on a X'pert Pro diffractometer (PANaly, Holland) with filtered Cu-Kα radiation (40 kV, 40 mA). X-ray photoelectron spectra (XPS) were collected with monochromatic Al-Kα radiation source (1486.6 eV) on a VG ESCALAB250 spectrometer (Thermo Scientic, USA). The morphology and microstructure of the composites were recorded by scanning electron microscope (SEM, JSM-7500F, JEOL Ltd., Japan). TEM, HRTEM, HAAD-FSTEM and STEM-EDS mapping were recorded by transmission electron microscope (FEI Tecnai G2 F20). UV-Vis diffusive reflectance spectrum (UV-Vis DRS) is tested with a scanning wavelength range of 200-850 nm by the LAMBDA750S spectrophotometer produced by PerkinElmer, USA. The Pt content of the composites was determined by ICP-AES (CAP6300, Thermo Scientific, USA).

2.2. Electrochemical measurements

The electrochemical properties were tested on CHI750E electrochemical workstation at room temperature. A glassy carbon electrode (GCE) or carbon paper was used as the working electrode, and a saturated calomel electrode (SCE) as the reference electrode, and a carbon rod as the auxiliary electrode. The area of carbon paper was 1 cm², and that of GCE was 0.2475 cm². The potentials were converted according to the formula (RHE = E_{SCE} + 0.059 × pH + 0.241V). 2 mg as-synthesized catalyst was dispersed in the mixed solution (900 uL ethanol and 100 uL 5 wt% Nafion). After that, 20 uL dispersion solutions were taken out and coated in GCE or carbon paper, then naturally drying for testing.

Linear sweep voltammetry (LSV) was employed to evaluate the catalytic performance for HER. The electrolyte was 0.5 M H_2SO_4 with N_2 -saturation. The rotation speed and the scanning rate of LSV was 1600 rpm and 5 mV s⁻¹, respectively.

Chronoamperometry (20000 s) was tested at 10 mA cm⁻² current density to evaluate the durability of the catalyst with aerating N_2 . Hydrogen production was measured with an H-type electrolytic cell which was linked to gas chromatograph. It should be noted that the working electrode in H-type electrolytic cell was carbon paper coated with catalyst's ink, while the auxiliary and reference electrodes remained unchanged. The current densities of the electrochemical test curves were all normalized by geometric area.

2.3. Photo-electro catalysis tests

Xenon lamp light source (300W) provided simulated sunlight (200-1100 nm), and its wavelength could be adjusted by a filter (50 * 70 mm square sheet). In the photoelectro catalysis tests, the simulated sunlight (30 mm spot diameter, parallel light) was illuminated on the electrode surface through the bottom of the quartz electrolytic cell. The light source was 20 cm far from the electrode surface, and the constant operating voltage was 14 V.



Figure S1 (A) The full XPS spectrum of Pt/CoP/C and (B) the C1s spectrum of Pt/CoP/C.



Figure S2 UV-vis diffusive reflectance spectrum of Pt/CoP/C

Table S1. Comparison of increasing H_2 production quantities on the composites with that on different catalysts in the literatures

Composite	The increasing H ₂ production quantities under simulated sunlight irradiation (mmol h ⁻¹ g ⁻¹)	Data sources
Pt/CoP/C	877.83	This work
Pt/CoP	388.67	This work
$Co_{3}O_{4}/Cd_{0.9}Zn_{0.1}S$	139.78	Ref. ¹
Mn _{0.25} Cd _{0.75} S/Co ₃ O ₄	25.71	Ref. ²
NiCoP/Mn _{0.3} Cd _{0.7} S	118.5	Ref. ³
Pt-NLCDs@CdS	46.10	Ref. ⁴
C@CdS-HS/Pt	20.9	Ref. ⁵

References

- 1. Y. Han, Z. Liang, H. Dang and X. Dong, J. Taiwan Inst. Chem. Eng., 2018, 87, 196-203.
- 2. Q.-Z. Huang, J.-C. Wang, L.-Q. Ye, Q. Zhang, H.-C. Yao and Z.-J. Li, *J. Taiwan Inst. Chem. Eng.*, 2017, **80**, 570-577.
- 3. D. Kong, D. Yin, D. Zhang, F. Yuan, B. Song, S. Yao, J. Yin, Y. Geng and X. Pu, *Nanotechnology*, 2020, **31**, 305701.
- 4. J. Zhuang, S. Ren, B. Zhu, C. Han, Y. Li, X. Zhang, H. Gao, M. Fan and Q. Tian, *Chem. Eng. J.*, 2022, **446**, 136873.
- 5. S. Tang, Y. Xia, J. Fan, B. Cheng, J. Yu and W. Ho, *Chinese J. Catal.*, 2021, **42**, 743-752.