The supplementary files for Synergistic promotion of selective oxidation of glycerol to C_3 products by Mo-doped $BiVO_4$-coupled FeOOH co-catalysts through photoelectrocatalysis process

1. Characterization

The morphological characterization of the samples was carried out using field emission scanning electron microscopy (FE-SEM, TESCAN MIRA3 LMH) at an accelerating voltage of 10 kV. High-resolution transmission electron microscopy (HRTEM) and selected area electron diffraction (SAED) analyses were conducted with a field emission electron microscope (TEM JEOL 2100F) at 200 kV, complemented by energy dispersive X-ray spectroscopy (EDS) for detailed sample characterization. Additionally, X-ray diffraction (XRD) analysis was performed using a Rigaku SmartLab diffractometer with CuKα radiation, covering a 2θ range of 20° to 80°. The light absorption properties of the samples were examined using an FLS 980 UV-Vis spectrophotometer (Edinburgh Instruments), measuring wavelengths from 200 to 800 nm. The valence states and elemental compositions of the samples were analyzed using X-ray photoelectron spectroscopy (XPS, ESCALAB250Xi) with Al-Kα radiation, alongside UV-visible diffuse reflectance spectroscopy. A PerkinElmer Lambda 750 UV-visible spectrometer, with $BaSO_4$ as the reference material, was employed for the measurements. Photoluminescence (PL) spectra were recorded using an FLS980 fluorometer from Edinburgh Instruments Ltd.

2. Supplementary Figures

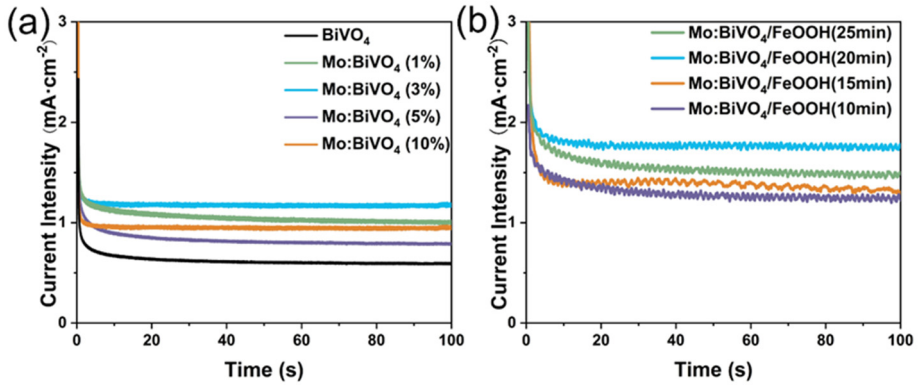


Figure S1 (a) i-t curves of the photocurrent test in glycerol solution for Mo:$BiVO_4$ photoelectrodes with different Mo doping concentrations (x% Mo:$BiVO_4$), (b) i-t curves of the photocurrent test in glycerol solution for Mo:$BiVO_4$/FeOOH photoelectrodes with different FeOOH loading times (Mo:$BiVO_4$/FeOOH (x min))

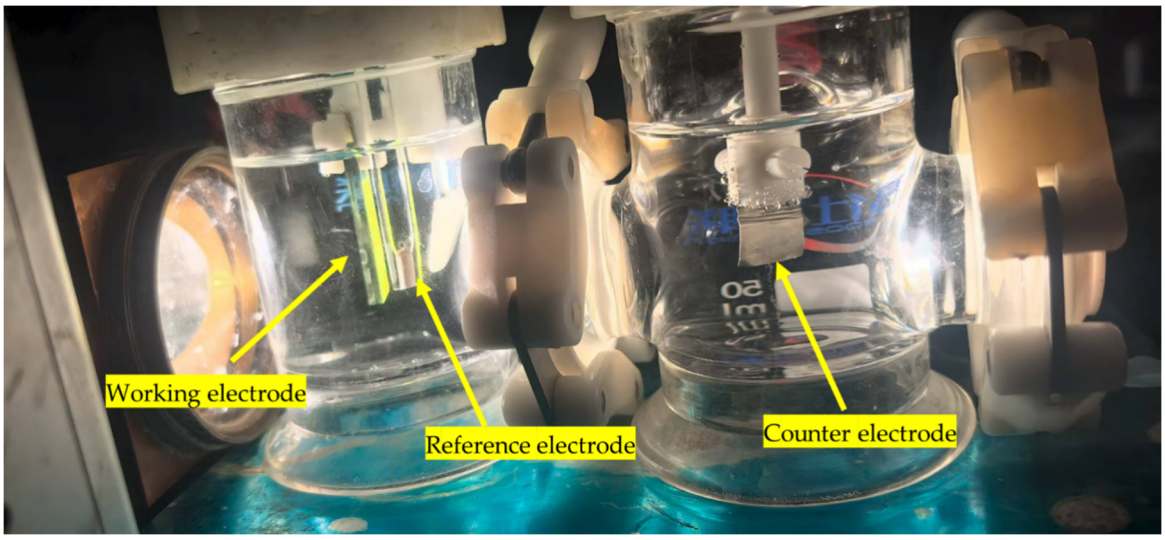


Figure S2 PEC reaction apparatus: H-type electrolytic cell

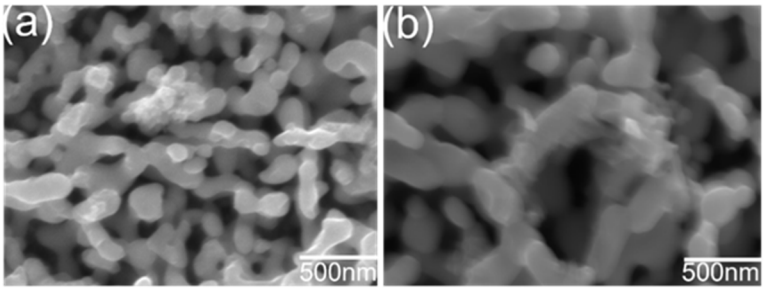


Figure S3 SEM image of Mo:$BiVO_4$/FeOOH photoelectrode before 10 hours glycerol conversion reaction, (b) SEM image of Mo:$BiVO_4$/FeOOH photoelectrode after 10 hours glycerol conversion reaction

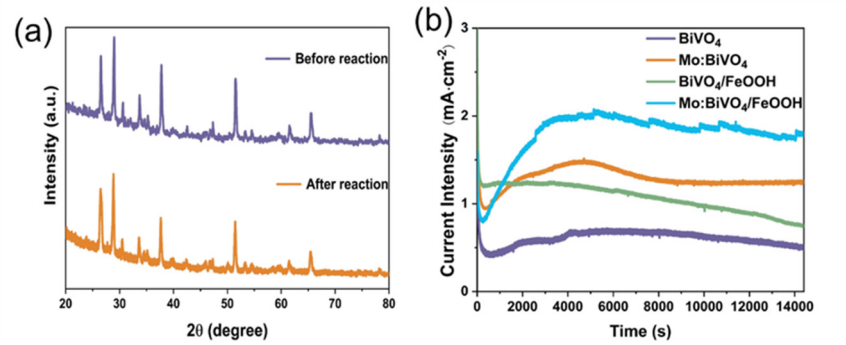


Figure S4(a) XRD plots of Mo:$BiVO_4$/FeOOH photoelectrode before and after 10 cycles of reaction, (b) Photocurrent diagram of photovoltaic electrodes converted by four hours of glycerol

Table S1 Summary of PEC glycerol selective oxidation over different photoanodes

Catalyst	Glycerol conversion rate ($\mu mol \cdot cm^{-2} \cdot h^{-1}$)	Target Product and Selectivity	References
Ta:$BiVO_4$	72	DHA, 97%	[15]
$BiVO_4$	48	GLAD, 37%	[1]
Mo:$BiVO_4$	56	GLAD, -	[9]
NOOH/$BiVO_4$	76	GLAD, 25%	[18]
FeOOH/$BiVO_4$	49	GLAD, 58%	[19]
Mo:$BiVO_4$/FeOOH	79	GLAD, 49%	This work