

Supporting Information

In Situ Preparation of MOF-derived Fe₂O₃ Nanorods for Visible-Light-Driven Oxygen Evolution

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1 Experimental

1.1 Materials

Hexahydrate ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and terephthalic acid (H_2BDC) were purchased from Sigma-Aldrich. All reagents utilized in this study were of analytical grade and employed without the need for additional purification. Distilled water used in the experiments was prepared using a distilled water purification system.

1.2 Characterization

The phase structures of prepared samples were studied with an X-ray diffractometer with Cu $K\alpha$ radiation of $\lambda = 1.5406 \text{ \AA}$. Field-emission scanning electron microscopy (FESEM) were tested by S-4800 (Japan) instrument. The TEM data was measured using JEOL JEM-2100 F (USA). UV visible light adsorption spectra using a Shimadzu UV-3600 with 0.1 nm data interval in the range of 200-800 nm. FT-IR spectra were recorded using a Bruker EQUINOX-55 FTIR instrument in the range of 400-4000 cm^{-1} . Thermogravimetric analysis (TGA) was conducted in the 30-800 $^\circ\text{C}$ range at a 10 $^\circ\text{C}/\text{min}$ heating rate under air flow using a PerkinElmer TGA 4000 thermal analyzer.

1.3 Electrochemical measurements

The electrochemical measurements were recorded at room temperature (25 $^\circ\text{C}$) in cell quartz with three-electrode system configurations (thin-film FTO as a working electrode, Ag/AgCl as the reference electrode, and Pt mesh as the counter electrode). 0.1 M Na_2SO_4 solution was used as the supporting electrolyte. The light source was a 300 W Xenon lamp (PLS-SXE300). The Nyquist plots were measured in the frequency range between 0.01 and 10^5 Hz at 1.23 V vs. RHE under dark conditions with an AC voltage perturbation of 10 mV. The applied potential was converted to the RHE values using the following Nernst equations:

$$E_{(RHE)} = E_{(Ag/AgCl)} + E_{(Ag/AgCl \text{ vs RHE})} + 0.059 \times pH$$

$$E_{(Ag/AgCl \text{ vs NHE})} = 0.196V$$

1.4 Photocatalytic water oxidation

The photocatalytic oxygen evolution tests of the samples were evaluated. 20 mg

photocatalyst, 0.13 g La_2O_3 and 0.1 g AgNO_3 were dispersed in 100 mL water. La_2O_3 was used to maintain the pH value of the solution and AgNO_3 was used as the electron sacrificial reagent for the photocatalytic oxygen evolution reaction. Before the visible-light irradiation, the suspension was fully deaerated to ensure the air was completely removed. A 300 W Xenon lamp was used as the visible-light source. O_2 evolved was measured by the gas chromatography (GC, D7900).

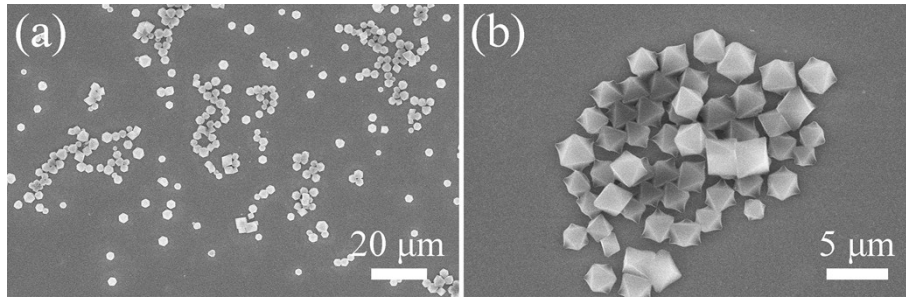


Fig. S1. (a) and (b) SEM images of MIL-101(Fe) with different magnifications.

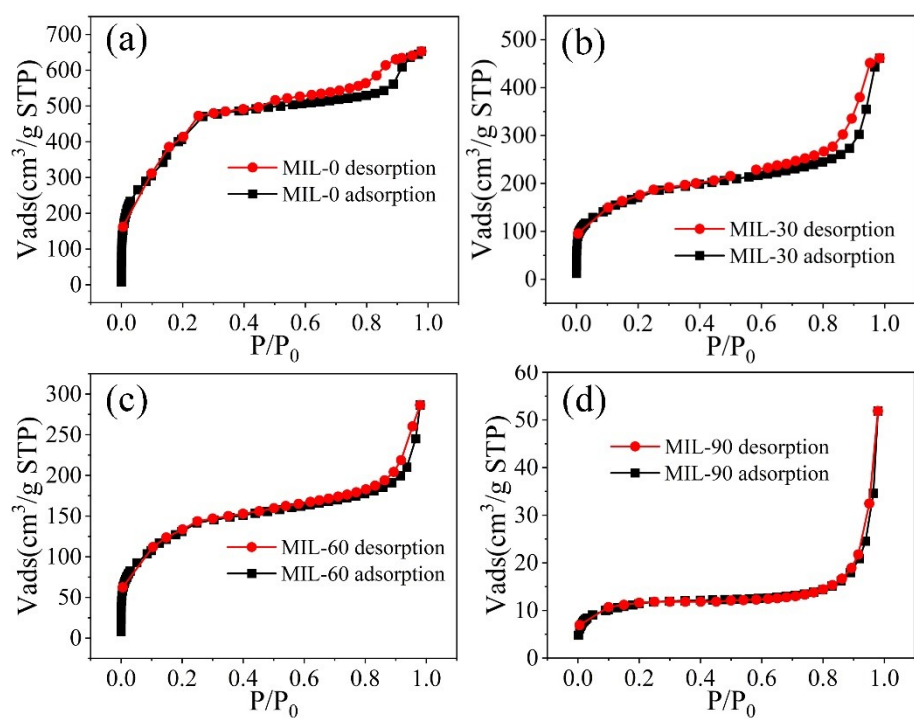


Fig. S2. N_2 adsorption-desorption isotherms of (a) MIL-0, (b) MIL-30, (c) MIL-60 and (d) MIL-90.

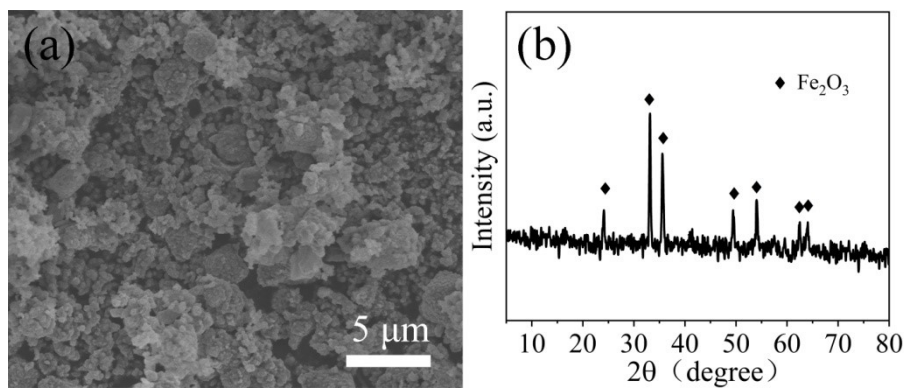


Fig. S3. (a) SEM image and (b) XRD pattern of Fe₂O₃ obtained by calcining the MIL-101(Fe).

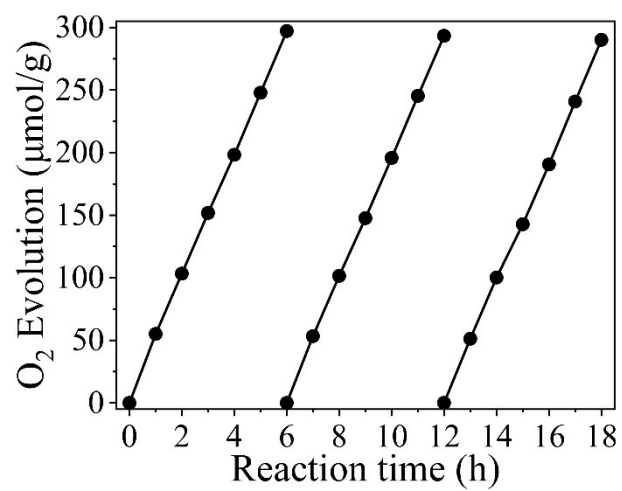


Fig. S4. Cyclic O₂ production over MIL-90 photocatalyst.

Table S1 Catalytic performance of iron oxide-based electrocatalyst

Catalyst	Electrolyte	Current density (mA/cm ²)	Reference
Ti:Fe ₂ O ₃	1M NaOH	0.09	[1]
Fe ₂ O ₃ nanorod arrays	1M KOH	0.12	[2]
Fe ₂ O ₃	0.1M Na ₂ SO ₄	0.25	[3]
Fe ₂ O ₃	1M NaOH	0.10	[4]
Fe ₂ O ₃	0.1M NaOH	0.08	[5]
Fe ₂ O ₃ nanorods	0.1M Na ₂ SO ₄	0.11	Present work

Reference

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