Supporting Information

A Serial of Ultrasensitive Electrocatalysts Fe-MOF/MWCNTs for

Fentanyl Determination

Zhidong Zhao ^{abc}, Yuan He ^{ad}, Xingrui Qi ^{ab}, Nian Li ^{ab}, Zijian He ^{ab},

Yufang Chen abdefg, Tao Jin *abdefg

^a Guangzhou Institute of Chemistry, Chinese Academy of Sciences, 510650, Guangzhou, P. R.

China

^b University of Chinese Academy of Sciences, 100000, Beijing, P. R. China ^c Guizhou Police College, 550005, Guiyang, P. R. China

^d CAS Testing Technical Services (Guangzhou) Co. Ltd., 510650, Guangzhou, P. R. China

^e CAS Engineering Laboratory for Special Fine Chemicals, Chinese Academy of Sciences, Guangzhou 510650, P. R. China

^f CASH GCC Shaoguan Research Institute of Advanced Materials, Nanxiong 512400, China ^g CASH GCC (Nanxiong) Research Institute of Advanced Materials Co., Ltd., Nanxiong 512400,

China

Corresponding Author Email: jintao@gic.ac.cn.

Synthesis of the five Fe-MOFs

Preparation of Fe-MOF-235 : H_2BDC (1.23 mmol) was added to 60 mL DMF and stirred for 10 min to form a clear solution. Then FeCl₃·6H₂O (0.738 mmol) was added to the solution and stirred for 5 min. The reactant mixture of 30 mL and 30 mL of ethanol were loaded into a Teflon-lined autoclave, heated at 80 °C for 24 h. After cooling to room temperature, the product was collected by centrifugation, washed several times each with ethanol and DMF, and dried at 60 °C for 12 h in a vacuum.^[1]

Preparation of Fe-MIL-88B : FeCl₃·6H₂O (1 mmol) and H₂BDC (1 mmol) were dissolved in a mixed solution of DMF (5 mL) and NaOH (2 M, 0.4 mL). After stirring for 30 minutes, the mixture was transferred to a Teflon-lined stainless steel autoclave, heated at 100 °C for 12 h. After cooling to room temperature, the product was collected by centrifugation, washed many times each with ethanol and DMF, and dried at 60 °C for 12 h in a vacuum.^[2]

Preparation of Fe-MIL-53 : FeCl₃·6H₂O (5 mmol) and H₂BDC (5 mmol) were dissolved in DMF (25 mL) and stirred for 30 min. The mixture was transferred to a Teflon-lined stainless steel autoclave, heated at 150 °C for 15 h. After cooling to room temperature, the product was collected by centrifugation, washed several times each with ethanol and DMF, and dried at 60 °C for 12 h in a vacuum.^[3]

Preparation of Fe-MIL-68 : FeCl₃·6H₂O (1 mmol) and H₂BDC (2 mmol) were dissolved into DMF (12 mL). Then HF (5 M, 120 μ L) and HCl (1 M, 120 μ L) were added to the above solution. After stirring for 30 minutes, the mixture was transferred to a Teflon-lined stainless steel autoclave, heated at 100 °C for 120 h. After cooling to room temperature, the product was

collected by centrifugation, washed many times each with deionized water and acetone, and dried at 60 $^{\circ}$ C for 12 h in a vacuum.^[4]

Preparation of Fe-MIL-101 : FeCl₃· $6H_2O$ (2.45 mmol) and H₂BDC (1.24 mmol) were added to DMF (15 mL) and stirred for 30 min. The mixture was transferred to a Teflon-lined stainless steel autoclave, heated at 110 °C for 24 h. After cooling to room temperature, the product was collected by centrifugation, washed several times each with ethanol and DMF, and dried at 60 °C for 12 h in a vacuum.^[5]



Fig.S1 TEM images and EDS spectrum of prepared Fe-MOF-235.



Fig.S2 TEM images and EDS spectrum of prepared Fe-MIL-88B.



Fig.S3 TEM images and EDS spectrum of prepared Fe-MIL-53.



Fig.S4 TEM images and EDS spectrum of prepared Fe-MIL-68.



Fig.S5 TEM images and EDS spectrum of prepared Fe-MIL-101.



Fig.S6 CV curves of different scan rate values in the range of 10–600 mV $\cdot s^{-1}(a)$ and the Linear relationship of Ipa and $\sqrt{\nu}$ of bare CGE.



Fig.S7 CV curves of different scan rate values in the range of 10-600 mV·s⁻¹(a) and the Linear

relationship of Ipa and \sqrt{v} of MWCNTs/CGE.



Fig.S8 CV curves of different scan rate values in the range of 10–600 mV·s⁻¹(a) and the Linear relationship of Ipa and $\sqrt{\nu}$ of Fe-MOF235/MWCNTs/CGE.



Fig.S9 CV curves of different scan rate values in the range of 10–600 mV·s⁻¹(a) and the Linear relationship of Ipa and $\sqrt{\nu}$ of Fe-MIL-88B/MWCNTs/CGE.



Fig.S10 CV curves of different scan rate values in the range of 10–600 mV·s⁻¹(a) and the Linear relationship of Ipa and $\sqrt{\nu}$ of Fe-MIL-53/MWCNTs/CGE.



Fig.S11 CV curves of different scan rate values in the range of 10–600 mV \cdot s⁻¹(a) and the Linear relationship of Ipa and $\sqrt{\nu}$ of Fe-MIL-68/MWCNTs/CGE.



Fig.S12 CV curves of different scan rate values in the range of 10–600 mV·s⁻¹(a) and the Linear relationship of Ipa and $\sqrt{\nu}$ of Fe-MIL-101/MWCNTs/CGE.



Fig.S13 The Linear relationship of Log Ipa and Log v of, Fe-MOF-235/MWCNTs(a) ,Fe-MIL-88B/MWCNTs(b) ,Fe-MIL-53/MWCNTs(c) ,Fe-MIL-68/MWCNTs(d) ,Fe-MIL-101/MWCNTs(e).



Fig.S14 CV curves of fentanyl recorded by Fe-MOF-235/MWCNTs(a) ,Fe-MIL-88B/MWCNTs(b) ,Fe-MIL-53/MWCNTs(c) ,Fe-MIL-68/MWCNTs(d) ,Fe-MIL-101/MWCNTs(e) repeated for 10 times using a single modified GCE .

Reference

[1] Wang, Q.; Astruc, D., State of the Art and Prospects in Metal-Organic Framework (MOF)-Based and MOF-Derived Nanocatalysis. Chemical Reviews 2020, 120 (2), 1438-1511.

[2] Yu, X. Y.; Feng, Y.; Guan, B. Y.; Lou, X. W.; Paik, U., Carbon coated porous nickel phosphides nanoplates for highly efficient oxygen evolution reaction. Energy & Environmental Science 2016, 9 (4), 1246-1250.

[3] Yan, L. T.; Cao, L.; Dai, P. C.; Gu, X.; Liu, D. D.; Li, L. J.; Wang, Y.; Zhao, X. B., Metal-Organic Frameworks Derived Nanotube of Nickel-Cobalt Bimetal Phosphides as Highly Efficient Electrocatalysts for Overall Water Splitting. Advanced Functional Materials 2017, 27 (40).

[4] Zhao, S. L.; Wang, Y.; Dong, J. C.; He, C. T.; Yin, H. J.; An, P. F.; Zhao, K.;
Zhang, X. F.; Gao, C.; Zhang, L. J.; Lv, J. W.; Wang, J. X.; Zhang, J. Q.; Khattak, A.
M.; Khan, N. A.; Wei, Z. X.; Zhang, J.; Liu, S. Q.; Zhao, H. J.; Tang, Z. Y., Ultrathin metal-organic framework nanosheets for electrocatalytic oxygen evolution. Nature Energy 2016, 1, 1-10.

[5] Jahan, M.; Liu, Z. L.; Loh, K. P., A Graphene Oxide and Copper-Centered Metal Organic Framework Composite as a Tri-Functional Catalyst for HER, OER, and ORR. Advanced Functional Materials 2013, 23 (43), 5363-5372.