

Supporting Information

Highly Moisture-resistant Fe-doped Mesoporous Co₃O₄ Catalyst for Efficient Low-temperature CO Oxidation

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Characterization

Powder X-ray diffraction (XRD) patterns were recorded on a DX-2700 diffractometer (Dandong Haoyuan Instrument Co. Ltd.) using Cu K α radiation ($\lambda=0.15405$ nm) operated at 30 kV. Nitrogen adsorption and desorption isotherms were measured on a Micromeritics ASAP 2020 M analyzer at liquid nitrogen temperature (77 K). Prior to the measurements, the samples were degassed at 423K in vacuum for 6h. The specific surface area and pore size distribution were calculated using the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods, respectively. Field emission scanning electron microscopy (SEM) analysis was performed on a JEOL JSM4800 electron microscope. Transmission electron microscopy (TEM) observations were performed on a field emission JEM-3000F (JEOL) electron microscope operated at 300kV equipped with a Gatan-666 electron energy loss spectrometer and energy dispersive X-ray spectrometer. XPS (X-ray photoelectron spectroscopy) signals were collected on a VG Micro MK II instrument using monochromatic Al K α X-ray at 1486.6 eV operated at 200 W. All the elemental binding energies were referenced to the C (1s) line situated at 284.6 eV. Thermo-gravimetric analysis (TG-DSC) were carried out at a heating rate of 10 °C/min from ambient temperature to 600 °C with an air flow rate of 20 ml/min. H₂ temperature-programmed reduction (H₂-TPR) analysis was performed by using a Micromeritics Chemisorb 2750 apparatus. For each analysis, accurate amounts of calcined sample (50 mg) were purged in a flow of pure argon at 200 °C for 30 min to remove traces water (heating rate 10 °C/min). After cooling to room temperature, H₂-TPR experiments were performed using a 10 vol% H₂/Ar mixture at a flow rate of 25 ml/min. The sample was heated from ambient temperature to 800 °C at heating rate of 10 °C/min and H₂ consumption was detected by a thermal conductivity detector (TCD)

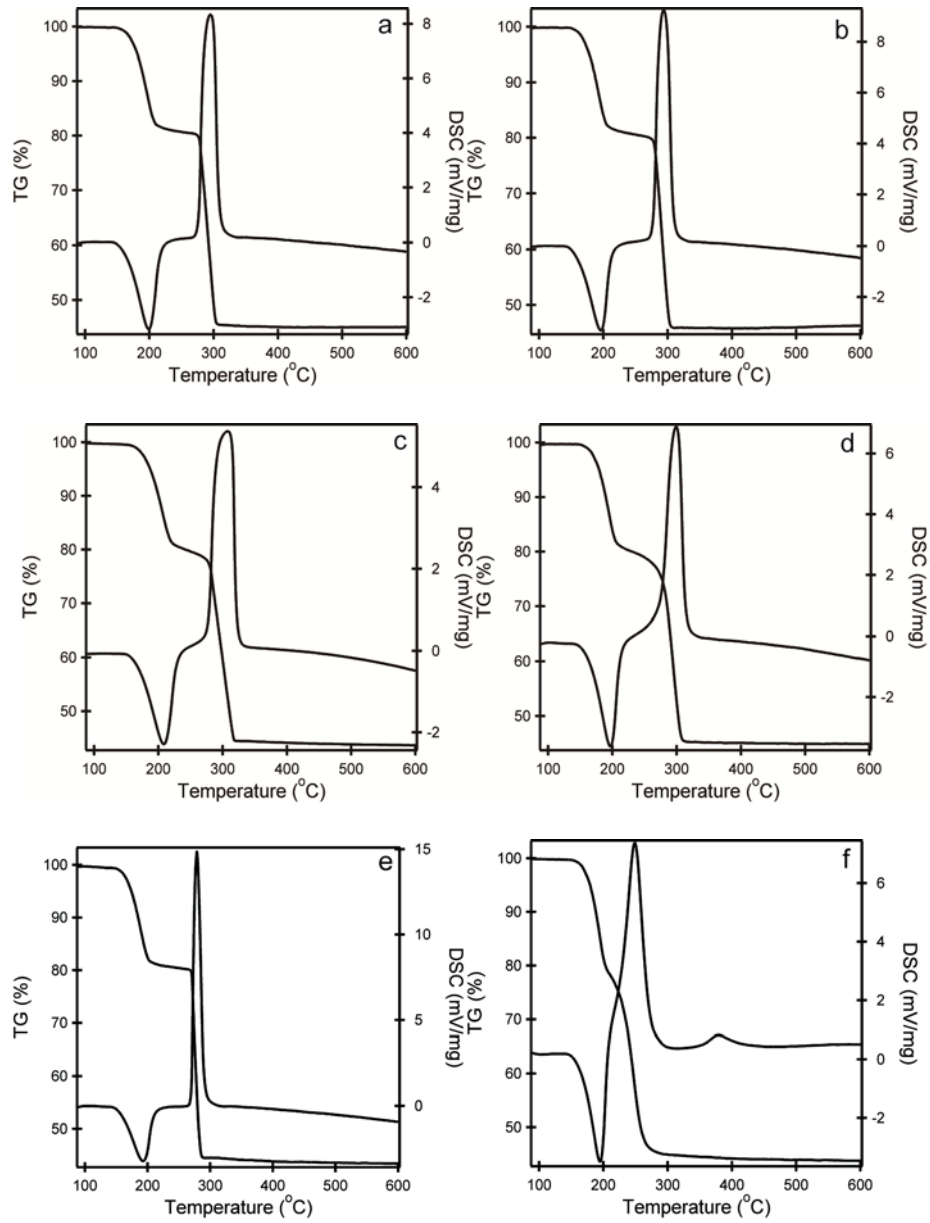


Fig. S1 TG-DSC curves of the oxalate precursors $\text{Fe}_{x/(x+y)}\text{Co}_{y/(x+y)}\text{C}_2\text{O}_4 \cdot 6\text{H}_2\text{O}$:
 $x/y=1/8$ (a) $x/y=1/4$ (b), $x/y=1/2$ (c), $x/y=1/1$ (d), $x/y=0/1$ (e), $x/y=1/0$ (f).

Table S1 The pore texture parameters of the catalysts

Sample	BET surface area (m^2/g)	Average pore diameter (nm)	Pore volume (cm^3/g)
CF8-1	139	3.0	0.28
CF4-1	156	3.0	0.29
CF2-1	185	3.3	0.27
CF1-1	205	3.3	0.26

TableS2 Surface composition of the sample CF2-1 before and after reaction

Element	Binding (eV)	Before reaction	After reaction under dry condition	After reaction under moisture condition (0.1 vol% H ₂ O)	After reaction under moisture condition (3.5 vol% H ₂ O)
		Percentage (%)	Percentage (%)	Percentage (%)	Percentage (%)
Co	780.0 795.2	19.3	21.7	19.5	14.3
Fe	Fe ²⁺	710.1	9.5	8.3	6.6
	Fe ³⁺	712.5	5.9	9.5	4.6
O	Lattice O	529.7	44.4	44.6	37.4
	Absorbed O	531.3 532.7	20.9	15.7	19.3

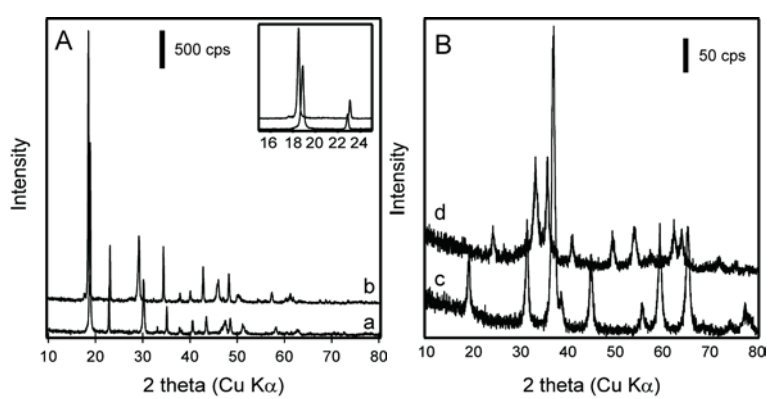


Fig. S2 The XRD patterns of the oxalate precursor (A) and after calcinated at 250 °C (B).
(CoC₂O₄·2H₂O (a), FeC₂O₄·2H₂O (b), Co₃O₄ (c), Fe₂O₃ (d))

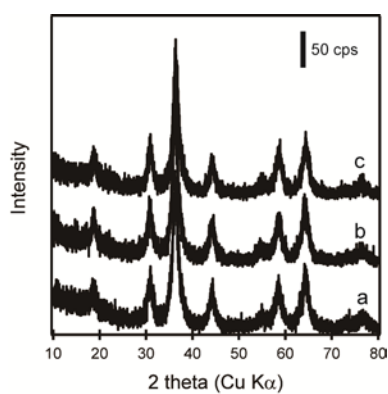


Fig. S3 The XRD patterns of CF2-1 after reaction under dry (a) and moisture conditions: 0.1 vol% H₂O (b) and 3.5 vol% H₂O.

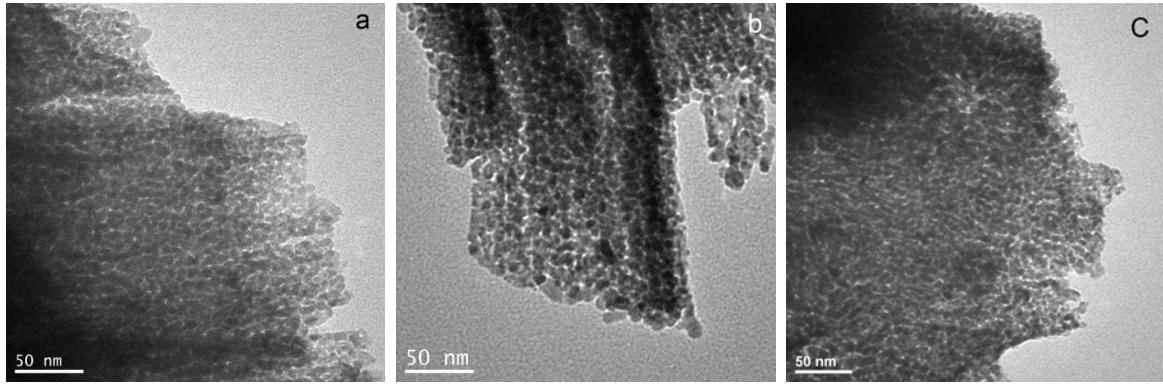


Fig. S4 TEM images of CF2-1 after reaction under dry (a) and moisture conditions: 0.1 vol% H₂O (b) and 3.5 vol% H₂O.

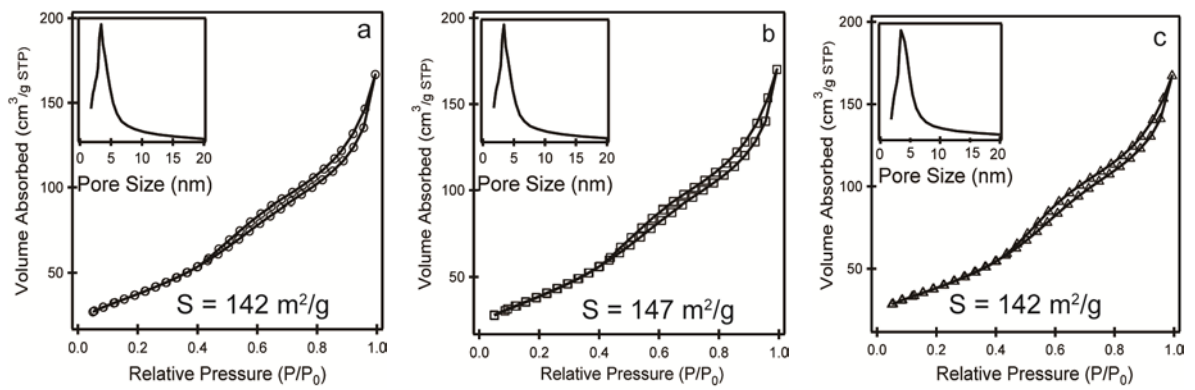


Fig. S5 The N₂ adsorption-desorption isotherms of CF2-1 after reaction under dry (a) and moisture conditions: 0.1 vol% H₂O (b) and 3.5 vol% H₂O.