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Supporting Information

A hydroxy-containing three dimensional covalent organic framework bearing silver nanoparticles for reduction 4-nitrophenol and degradation organic dyes

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S1 Characterized instruments

The crystallinity of prepared samples of 3D-COF and AgNPs-3D-COF were characterized by powder X-ray diffraction (PXRD) and recorded using a Bruker D8 Advance X-ray diffractometer at 40 kV and 40 mA over 2° to $80^{\circ} 2\theta$ range with a Cu Ka radiation. The microstructures of the prepared materials 3D-COF and AgNPs-3D-COF were studied by scanning electron microscope (SEM) (AMRAY 1000B) and transmission electron micrographs images (TEM, JEM-2010). The porosity of 3D-COF and AgNPs-3D-COF were investigated by nitrogen adsorption-desorption isotherms measured at 77 K on an Autosorb IQ2 absorptiometer (Quantachrome Instruments). The thermostability of 3D-COF and AgNPs-3D-COF were measured by thermogravimetric analysis (TGA) carried out on a Netzch STA449F3 analyzer under N₂ atmosphere at a heating rate of 10 °C/min from room temperature to 800 °C. X-ray photoelectron spectroscopy (XPS) test was conducted to demonstrate the chemical component in an Escalab 250Xi instrument from Thermo Scientific equipped with an Al Ka microfocused X-ray source, and the C1s peak at 284.8 eV was used as internal standard. Solid-state nuclear magnetic resonance (NMR) experiments were conducted using an Agilent VNMRS 600 MHz NMR spectrometer at room temperature.

S2 Synthesis of AgNPs-3D-COF

AgNO₃ aqueous solution (0.5 mL 5.0 mM) was added into the dispersion of 3D-COF (20.0 mL, 2.5 mg mL⁻¹) in deionized water (DW). The above mixture was continuously stirred for 15 min at room temperature. Subsequently, NaBH₄ aqueous solution (1.0 mL, 50 mM) was poured into the aqueous dispersion of 3D-COF and AgNO₃, and the mixture was stirred for 10 min at room temperature. Finally, the reaction mixture was centrifuged (16 000 rpm, 30 min) two times and the generated solid was collected and washed with DW and ethanol two times. The treated solid was dried via lyophilization under vacuum to obtain pure AgNPs-3D-COF nanocomposite.



Figure S1. FTIR spectrum of 3D-COF and starting materials TFPM and DHBD.



Figure S2. TGA curve of 3D-COF.



Figure S3. N₂ adsorption-desorption isotherms for 3D-COF.



Figure S4. Pore-size distribution of 3D-COF.

Figure S5. High-resolution TEM image of AgNPs-3D-COF.



Figure S6. High-resolution C 1s XPS of 3D-COF.



Figure S7. High-resolution N 1s XPS of 3D-COF.



Figure S8. The UV-vis absorption spectra for catalytic reduction of 4-NP in the presence of NaBH₄, AgNPs-3D-COF and NaBH₄ + AgNPs-3D-COF, respectively.



Figure S9. Time-dependent UV-vis absorption spectra for catalytic reduction of 4-NP in the presence of Pd/C catalyst.



Figure S10. Time-dependent UV-vis absorption spectra for catalytic reduction of MB in the presence of Pd/C catalyst.



Figure S11. Time-dependent UV-vis absorption spectra for catalytic reduction of CR in the presence of Pd/C catalyst.



Figure S12. PXRD pattern of AgNPs-3D-COF after tests.

Table S1. Elements content of C, N, O and Ag

Elements	С	N	0	Ag
wt%	78.74	5.25	7.48	8.53

 Table S2. Comparison of the ability of various catalysts for catalyzing reduction 4

 NP.

Entry	Catalyst	$k (\min^{-1})$	References
1	Ag@Pd-Ag nanocubes	1.69	[1]
2	Ag@Ag-Pd NFs	1.28	[2]
3	Ag@PEDOT	0.66	[3]
4	Au@Ag@PDA	0.5	[4]
5	Ag-Au-rGO	2.08	[5]
6	Au@Ag/MOF	2.98	[6]
7	Ag ⁰ @CMP	0.08	[7]
8	Fe ₃ O ₄ @PPy-MAA/Ag	0.17	[8]
9	AgNPs-3D-COF	2.99	This work

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