Supplementary Information (SI) for New Journal of Chemistry.

Supporting Information

Preparation of CoS_2/g-C_3N_4 composite catalyst and its application in photocatalytic reduction of CO_2

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2.3 Characterizations

2.3.1 The instrumental characterizations

Operating at a working voltage and current of 40 kV and 100 mA, the Kα ray wavelength of Cu was 1.5406 Å, and the powder X-ray diffraction (XRD) patterns of all samples were collected in the range of 2θ angles from 5° to 80°. The microscopic morphology of the sample was characterized by the scanning electron microscope (SEM, NOVA Nano SEM 450), transmission electron microscope (TEM, JEM-1400 EX) and high-resolution transmission electron microscope (HRTEM, JEM-2100 EX). The ultraviolet-visible (UV-vis) diffuse reflectance absorption spectrum was scanned in the range of 200-800 nm using a UV-vis spectrophotometer (Varian, Cary 500). The measurement of the specific surface area was carried out by using an ASAP 2020 instrument at 77 K using N_2 adsorption. The X-ray photoelectron spectroscopy (XPS) test was carried out using the PHI 5000C ESCA system. The excitation source was Al Kα rays with energy of 1486.6 eV, the pressure in the analyzer was less than 1×10^{-8} Torr, and the pass energy was 50 eV. And use X-rays with a power of 250 W and a target voltage of 14.0 kV. Cary Eclipse's fluorescence emission meter (FL) was used to test the fluorescence emission spectrum under excitation at a wavelength of 350 nm. The 133 eV/Falion 60S energy spectrometer (EDS) was used to detect the content of C, N, Co and S in the sample. The time-resolved fluorescence decay spectroscopy (TRFL) was measured using an FSL 980 instrument at room temperature. The electrochemical test was measured on an electrochemical workstation (Zahner, Zennium), using a three-electrode system (platinum counter electrode, saturated calomel electrode as reference electrode, working electrode). For the photocurrent and Mott-Schottky test, the electrolyte is 0.5 mol L^{-1} sodium sulfate, and the xenon lamp was used as the light source. The electrochemical impedance test (EIS) was carried out in a mixture of 0.1 mol L^{-1} KCl solution and 25 mmol L^{-1} $K_4Fe(CN)_3$ and $K_3Fe(CN)_6$ solutions.

2.3.2 CO_2 photocatalytic reduction (CO_2PR) evaluation

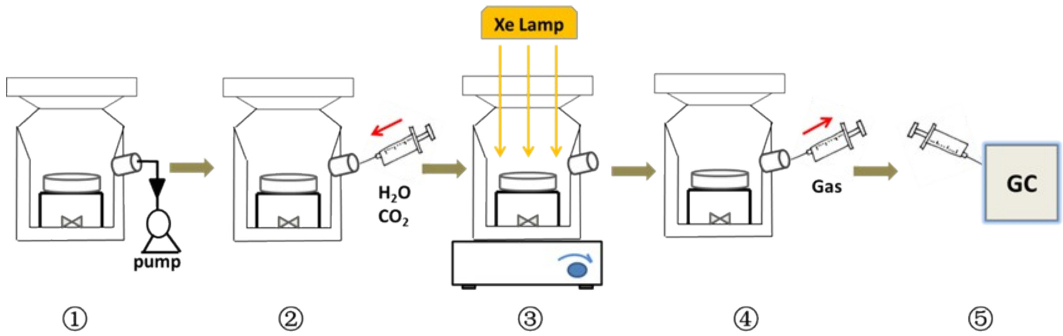

Fig. S1. Photocatalytic CO_2 reduction flowchart (①. Evacuate the reactor; ②. Inject 1 mL of ultrapure water and 200 mL of CO_2 into the reactor; ③. Irradiate the catalyst with Xe lamp for 4 h; ④. Extract 5 mL of reacted gas; ⑤. Inject gas into GC for analysis).

The gas-solid phase photocatalytic reduction experiments of CO_2 were carried out in a self-made reaction system (Fig. S1). 30 mg of sample was dispersed in 1 mL of water. The mixture was spread on a watch glass with a diameter of 6 cm, and dried to form a thin layer. The prepared sample was transferred to a reactor with a volume of 450 mL, and 1 mL of ultrapure water was injected into the reactor. Then, the reactor was evacuated and 400 mL of CO_2 (gas, 99.99%) was injected into the reactor. After 30 min of dark adsorption, a 300 W Xenon lamp was used to illuminate for 4 h. After the reaction, 5 mL of mixed gas was extracted and pumped into the gas chromatograph (GC) to analyze the gas composition. The external standard method was used to qualitatively and quantitatively test the gas, and the test was repeated three times for each sample. Three comparative tests were carried out under the conditions of using Ar instead of CO_2, no light and no catalyst.

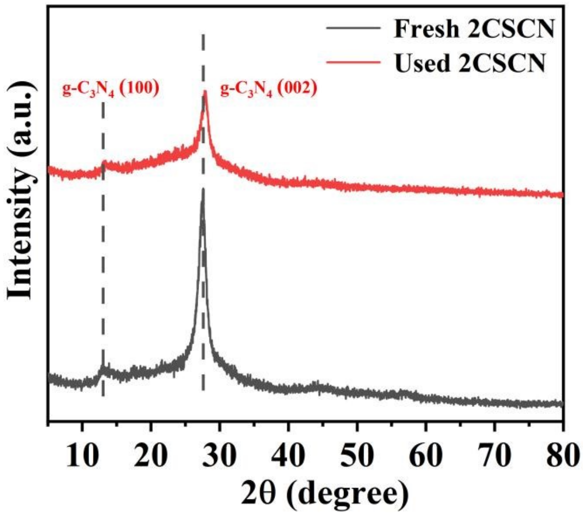

Fig. S2. XRD patterns of 2CSCN before the reaction and after 5 cycles of testing.

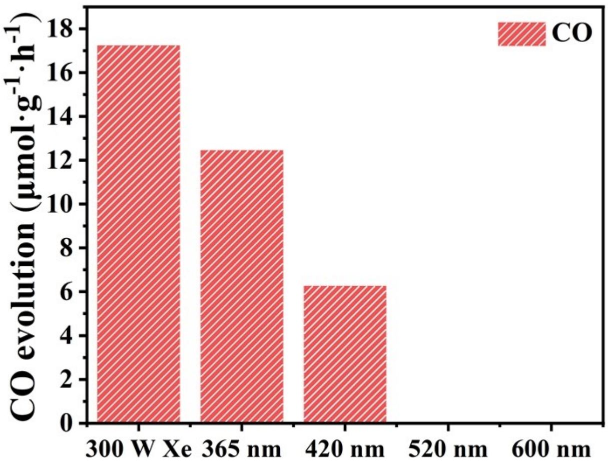

Fig. S3. CO production rate of 2CSCN under different light irradiation.

Table S1. Comparison of photocatalytic performance of our catalyst with other reported similar materials.

Sample	Light source	Reaction condition	Performance ($\mu mol \cdot g^{-1} \cdot h^{-1}$)	Reference
CoS_2/g-C_3N_4	300 W Xe lamp	H_2O	CO (17.24)	1
Pt/g-C_3N_4	LED-397/427/452	H_2O	CO (13.20)	
g-C_3N_4/Ag-TiO_2	300 W Xe lamp	H_2O	CH_4 (9.33) CO (6.33)	2
NiO/g-C_3N_4	300 W Xe lamp	H_2O	CO (4.17)	3
$SnFe_2O_4$/g-C_3N_4	300 W iodine tungsten lamp	H_2O	CO (7.56)	4
AgX/pCN	low-power 15 W energy-saving daylight lamp	H_2O	CH_4 (10.92)	5
MoS_2/g-C_3N_4	300 W Xe lamp	H_2O	CO (8.37)	6
$CuInS_2$/Au/g-C_3N_4	300 W Xe lamp with 400 nm cut- off filter	H_2O	CH_4 (0.15) CO (2.43)	7
Bi_2S_3 QDs/g-C_3N_4	300 W Xe lamp	H_2O	CH_4 (12.52) CO (54.74)	8
Cu_2O QDs/g-C_3N_4	Photocatalytic activity evaluation system (CEL-SPH2N-D9, Beijing)	H_2O	CH_4 (0.08) CO (8.12)	9
NiS_2 QDs/g-C_3N_4	300 W Xe lamp	H_2O	CO (10.68)	10
CoZnAl-LDH/RGO/g-C_3N_4	300 W Xe lamp	H_2O	CO (10.11)	11
g-C_3N_4/CoCo-LDH	300 W Xe lamp	H_2O	CO (71.39)	12
Co-$CuInS_2$	300 W Xe lamp	H_2O	CO (15.24)	13
Co_9S_8@$ZnIn_2S_4$/CdS	300 W Xe lamp	H_2O, MeCN, Na_2SO_3	CO (82.10)	14

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