Supplementary Information

Carboxyl ligand-regulated Cd metal complexes as efficient catalyst for electrocatalytic reduction of CO_2 to CO

Jia Li[1], Tianxia Liu[1,2,3*], Boming Lu[1]

1. School of Chemistry and Chemical Engineering, North Minzu University, Yinchuan 750021, P.R.China

2. Ningxia Key Laboratory of Solar Chemical Conversion Technology, North Minzu University, Yinchuan 750021, P.R.China

3. Key Laboratory for Chemical Engineering and Technology, State Ethnic Affairs Commission, North Minzu University, Yinchuan 750021, P.R.China

*Corresponding author.

E-mail address: 2005053@nmu.edu.cn (T. X. Liu)

CONTENT

1 Preparation of electrode

Firstly, a homogeneous dispersion was prepared by mixing anhydrous ethanol and Nafion solution in a 100:1 volume ratio. Subsequently, 5 mg of catalyst powder was combined with 2 mg of carbon black in a sample vial. Thirdly, 0.5 mL of the pre-formulated Nafion/ethanol mixture solution was introduced. The mixture was then subjected to 30 minutes of ultrasonication to achieve a uniformly dispersed catalyst ink. The evenly dispersed catalyst suspension was coated on one end of 1.5×1 cm rectangular carbon paper with a 10 μL pipette, and the rectangular area of 0.5 cm^2 was evenly coated on both sides to ensure that the total coating area reached 1 cm^2. The carbon paper electrode coated with catalyst was subjected to thermal treatment in an oven at 60℃ for 30 min before collection, and used after being cooled gradually to ambient temperature.

2 Material characterization method

Field emission scanning electron microscope (FE-SEM, carezeiss SIGMA 500, Germany) and transmission electron microscope (TEM, JEM-2100, Japan) were employed to characterize the morphology features of the obtained materials and the information of material components. In the amplification range of 25 μm ~ 200 nm, the energy dispersive X-ray energy spectrum (EDS) and morphology of metal complex were analyzed, and the different morphology and the overall distribution of elements of the catalyst were observed. The diffraction data of Cd-BA, Cd-BDC and Cd-BTC were obtained on the Cu-Kα target, and the powder X-ray diffraction (XRD, Smartlab-SE type, λ = 1.5406 Å) was carried out at 40 kV, 30 mA, with a step size of 0.01. The infrared absorption spectrum was obtained by FT-IR 4800 in the measuring spectral range from 4000 to 400 cm^{-1}, enabling the identification of surface functional groups were systematically characterized. X-ray photoelectron spectroscopy (XPS) was used 200 W Al-Kα X-ray source for excitation, and recorded it on ThermoScientific ESCALab 250Xi acquisition spectrometer, and obtained the XPS full spectrum and the high-resolution spectra of all constituent elements, so as to analyze the surface elemental composition and chemical bonding state of the sample. All XPS spectra were corrected based on the C 1s peak of 284.8 eV. The specific surface area and pore size distribution characteristics of the samples were analyzed and determined by Micromeritics ASAP2460 specific surface area analyzer based on the nitrogen adsorption-desorption isotherm technique.

Table S1 metal complexes as catalysts for electrochemical CO_2 reduction

Compound	FECO (%)	Ref
$[Ni(DMCy^{2tBu})](PF_6)_2$	14±2	1
CCC-NHC	34	2
Re(I)NHC	60	3
$Re(bpy)(CO)_3Cl$	90	4
$[Fe1](BF_4)_2$	37	5
$[Co(tBu3tpy)_2]_2+$	37	6
CuPc-CNT	44	7
Ag(I) bis-BIAN	51	8

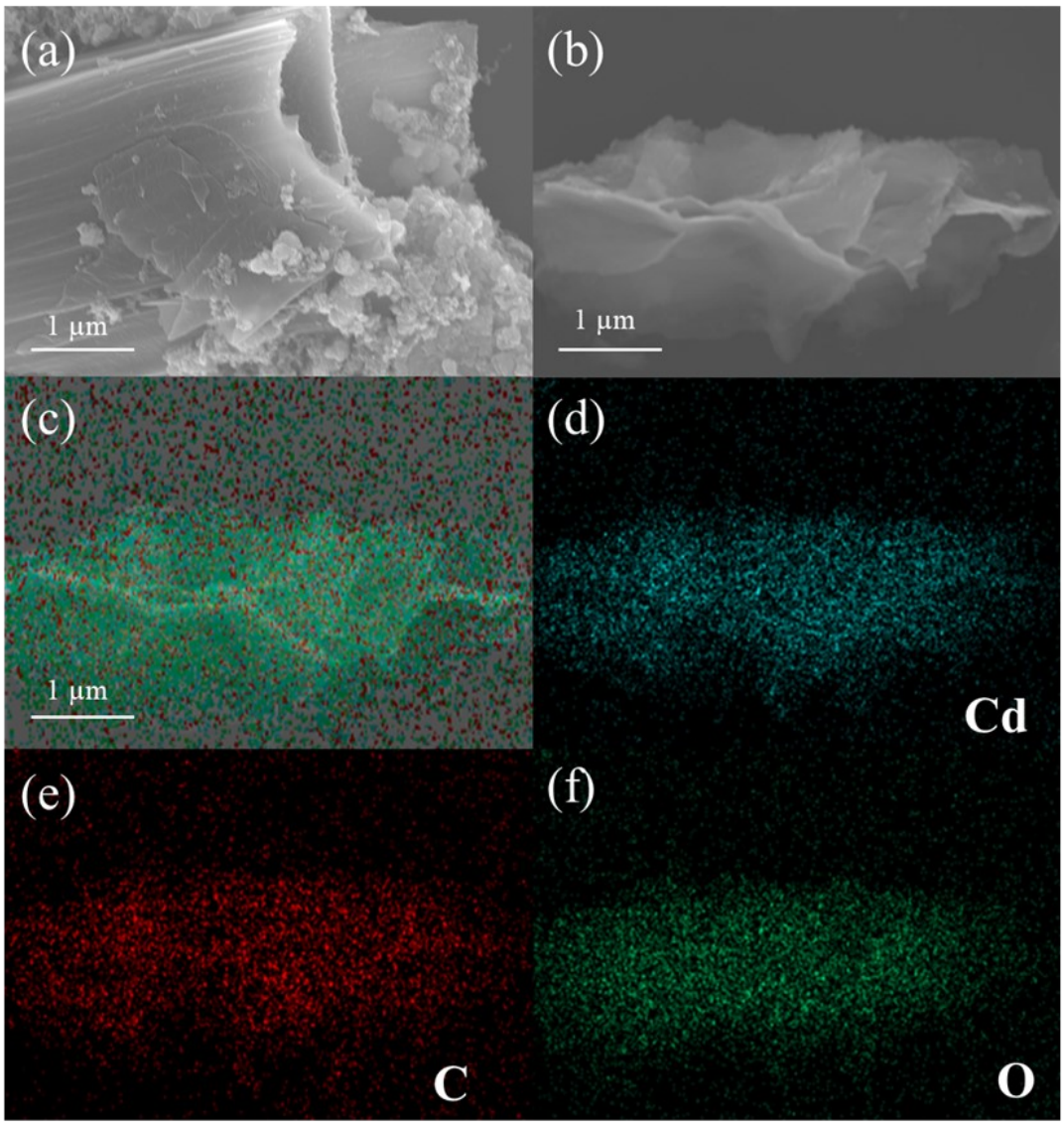

Fig. S1 After electrolysis SEM and EDS of Cd-BDC

Reference

1 T. Qiu, G. P. A. Yap and J. Rosenthal, ACS Appl. Energy Mater., 2019, 2, 8560-8569.

2 J. D. Cope, N. P. Liyanage, P. J. Kelley, J. A. Denny, E. J. Valente, C. E. Webster, J. H. Delcamp and T. K. Hollis, Chem. Commun., 2017, 53, 9442-9445.

3 C. J. Stanton, C. W. Machan, J. E. Vandezande, T. Jin, G. F. Majetich, H. F. Schaefer, C. P. Kubiak, G. Li and J. Agarwal, Inorg. Chem., 2016, 55, 3136-3144.

4 S. Sato, B. J. McNicholas and R. H. Grubbs, Chem. Commun., 2020, 56, 4440-4443.

5 S. Suhr, N. Schröter, M. Kleoff, N. Neuman, D. Hunger, R. Walter, C. Lücke, F. Stein, S. Demeshko, H. Liu, H.U. Reissig, J. van Slageren and B. Sarkar, Inorg. Chem., 2023, 62, 6375-6386.

6 N. Elgrishi, M. B. Chambers and M. Fontecave, Chem. Sci., 2015, 6, 2522–2531.

7 N. M. Latiff, X. Fu, D. K. Mohamed, A. Veksha, M. Handayani and G. Lisak, Carbon, 2020, 168, 245-253.

8 D. Krisch, H. Sun, K. Pellumbi, K. Faust, U.P. Apfel and W. Schöfberger, Catalysts, 2022, 12, 545.