Electronic Supplementary Information

Regulation of coordination and doping environment via target

molecular for boosting selective photocatalytic ability

1. Experimental section

1.1. Materials

Cadmium nitrate tetrahydrate (Cd(NO₃)₂·4H₂O), citric acid monohydrate (C₆H₁₀O₈), TC, ciprofloxacin (CIP), urea (CH₄N₂O), potassium ferricyanide (K₃Fe(CN)₆), potassium ferrocyanide (K₄Fe(CN)₆), potassium chloride (KCl), barium sulfate (BaSO₄), potassium bromide (KBr), anhydrous ethanol, triethanolamine (TEOA), tert-Butanol (t-BuOH). Unless otherwise specified, all are analytical pure.

1.2. Preparation

Firstly, 1.83 mmol Cd(NO₃)₂·4H₂O, 20 mmol C₆H₁₀O₈ and 25 mg TC were weighed into the beaker, and 7.5 mL deionized water was added to dissolve them through ultrasound. Then, the solution was left standing for 0.5 h and transferred into a threemouth flask for oil bath reaction at 80 °C to remove water and other volatile substances. Next, the dry mixture was transferred to a crucible and calcined at 600 °C for 3 h. After cooling to room temperature, brown-black powder solids were N-doped LC-CdO.

The materials synthesized by only changing the amount of $C_6H_{10}O_8$ to 0, 10 and 30 mmol were denoted as N-doped LC-CdO-CAM0, N-doped LC-CdO-CAM10 and N-doped LC-CdO-CAM30, respectively.

The materials synthesized by only changing the amount of TC to 0, 10 and 40 mg were denoted as CdO-OH, N-doped LC-CdO-TC10 and N-doped LC-CdO-TC40, respectively.

The materials synthesized by only using the same mass of urea or CIP instead of TC were denoted as N-doped CdO and CdO-CIP, respectively.

The material after CdO-OH was calcined by hydrogen argon mixture (10% H₂ / 90% Ar) at a low temperature of 150 °C for 24 h was denoted as CdO-H₂.

1.3. Photodegradation experiment

Simply, 0.1 g photocatalyst was weighed and put into the photoreaction bottle. After 100 ml of 20 mg/L TC solution was added, the photoreaction bottle was fixed in the photoreactor for experiment. Firstly, dark adsorption was carried out for 30 min to

achieve adsorption desorption equilibrium, and then light and aeration were turned on for the photocatalytic process. The light source used in the experiment was a 300 W xenon lamp, which used the visible light region greater than 420 nm, while the ultraviolet light region less than 420 nm was filtered out by a filter, so as to investigate the visible light catalytic performance of the material more intuitively. In the experiment, the stirring speed was 600 rmp and the air aeration rate was 1 mL/min. The photocatalytic degradation experiment was carried out for 1 h, during which the solution samples were taken every 4 min. After the experiment, the concentration of TC in the solution samples was detected by UV-visible spectrophotometer, and the total organic carbon content in the solution was measured by TOC analyzer.

1.4. Selective photodegradation experiment

The selective degradation process of antibiotic solution was the same as **Part 1.3**, and TC was replaced with CIP. The selectivity coefficient of photodegradation or mineralization (K'_{selectivity}):

$$K_{selectivity}(N - doped LC - CdO) = \frac{k (TC)}{k (CIP)}$$
(1)

$$K_{selectivity}(CdO - OH) = \frac{\kappa (IC)}{k (CIP)}$$

$$K_{selectivity}(N - doped LC - CdO)$$

$$(2)$$

$$K'_{selectivity} = \frac{K(CdO - OH)}{K(CdO - OH)}$$
(3)

Where k(TC) is the apparent rate constant of TC, k(TC) is the apparent rate constant of CIP, $K_{selectivity}$ (N-doped LC-CdO) is the coefficient of N-doped LC-CdO, $K_{selectivity}$ (CdO-OH) is the coefficient of CdO-OH, $K'_{selectivity}$ is the selectivity coefficient.

1.5. Cyclic experiment

In the cycling experiments, after the first photocatalytic reaction, the sample was centrifuged and sonicated for an hour. Subsequently, it was separated and then washed completely with DI water and ethanol. Afterwards, the collected sample was used for the next photocatalytic degradation experiment. This above process was repeated 3 times. The photocatalyst after 3 cycles was recovered and subjected to other tests.

1.6. Electrochemical experiment

EIS, Mott-Schottky experiment and photocurrent responses were performed on the electrochemical workstation. It included a standard three-electrode, which contained a Hg/Hg_2Cl_2 electrode and a Pt wire as the reference electrode and the counter electrode, respectively. EIS Nyquist analysis was performed in the electrolyte of 0.5 M Na₂SO₄ solution. Its working electrode was obtained through depositing a sample film. Mott-Schottky experiment was determined in dark over the frequency of 1000 Hz.

1.7. Radical capture experiment

The radical capture experiments were carried out by adding TEOA (1 mmol, a quencher of photo-generated holes (h⁺)), t-BuOH (1 mmol, a quencher of hydroxyl radicals (\cdot OH)) and aerating with nitrogen (inhibit the production of superoxide free radicals (\cdot O₂⁻)), respectively. The remaining experimental procedures were the same as the photocatalytic experiment.

1.8. Characterizations

The morphology of the synthesized samples was investigated by SEM with a field emission JSM6700F microscope and a tungsten filament 3400N microscope, and by TEM with a Hitachi H-7650. The XRD analyses were performed with a D8 ADVANCE diffractometer, operating with a parallel Cu-K α radiation at 40 kV and 40 mA. Fourier transform infrared spectra (FT-IR) were recorded on a Nicolet Nexus 470 FT-IR (America thermo-electricity Company). UV–Vis DRS spectra were recorded in the 200–800 nm wavelength range, using a UV2600 UV–Vis spectrophotometer with BaSO₄ as a reflectance sample. XPS (PHI5300) with monochromatic Al K α excitation source was conducted to study surface electronic structures of electrocatalysts. Electron paramagnetic resonance (EPR, Bruker-A300) was performed to evaluate oxygen vacancies concentration and free radical signal. The electrochemical performance was assessed in a standard three-electrode system by an electrochemical workstation (CHI 660E). PL spectra obtained on a Cary Eclipse fluorescence spectrophotometer with an excitation wavelength of 390 nm. *In-situ* ATR-FTIR were recorded on a Thermo Scientific Nicolet iS50 FTIR, and the light source was a low-power 420 nm wavelength LED lamp. The total organic carbon analyzer (multi N/C 2100, Germany) was used to measure the total organic carbon content (TOC).

1.9. Computational details

DFT calculation was performed with the CP2K package, using the hybrid Gaussian and plane-wave approach. The generalized gradient approximation (GGA) of Perdew-Burke-Ernzerhof (PBE) exchange-correlation functional was chosen for all calculations. The DZVP-MOLOPT-SR-GTH basis set in combination with Goedecker-Teter-Hutter (GTH) pseudopotentials were employed. D3 correction was used to describe the system. The input files were created using Multiwfn program. For the auxiliary basis set of plane waves, a 400 Ry cut-off was used. The convergence of energy was converged to 1×10^{-6} Hartree. The materials were calculated in a periodic cubic simulation box of $19.13 \times 19.13 \times 25$ Å³. The CdO model consisted of 120 Cd atoms and 120 O atoms, and the bottom two layers were fixed in the process of geometric optimization. The adsorption energies (E_{ads}) of the antibiotic molecule adsorbed on the photocatalysts (N-doped LC-CdO, LC-CdO or CdO-OH) were calculated from the equations:

$$E_{abs} = E_{slab + antibiotic} - E_{slab} - E_{antibiotic}$$
(1)

Where $E_{slab + antibiotic}$, E_{slab} and $E_{antibiotic}$ are the energies of photocatalyst with antibiotic, the relaxed bare photocatalyst, and the free antibiotic molecule, respectively.

The Mulliken population was calculated using the free academic version of CASTEP 19.1, and the GGA of PBE exchange-correlation functional was chosen for all calculations.

The density of states was used with the Plane Wave Self-Consistent Field (PWSCF) code in the open source software Quantum ESPRESSO (QE) package. Generalized gradient approximation (GGA) with Perdew-Burke-Ernzerhof (PBE) exchange-correlation function and norm-conserving pseudopotential was used for all calculations. When $2 \times 2 \times 2$ supercell was used as optimization unit. In the structural optimization

process, the ultrasoft pseudopotential was used to effectively calculate the atomic force, where the plane wave cut-off energy and density of the electron wave function were 50 Ry and 500 Ry, respectively. The convergence tolerance for the energy and force was set at 10^{-4} Ry and 10^{-5} Ry/bohr, respectively. A $3 \times 3 \times 3$ Monkhorst-Pack k-points grid was used for geometric optimization.

The mutagenicity of degradation intermediates was assessed using T.E.S.T. software based on quantitative structure-activity relationship (QSAR) prediction.

2. Figures and Tables



Fig. S1 SEM images of (a) CdO-OH and (b) N-doped LC-CdO.

In **Fig. S2a-2b** and **2f**, different amounts of TC had no effect on the morphology and crystal structure of CdO. The materials prepared with different amounts of citric acid monohydrate were shown in **Fig. S2c-2e**. The CdO synthesized without CAM was more likely to produce regular octahedron; The CdO synthesized by adding a small amount of CAM (10 mmol) had both rod-like and irregular lumps, and a small amount of regular octahedron; The CdO synthesized by adding excessive CAM (30 mmol) was composed of a large number of small balls, which was similar to the results reported in the literature¹⁻³. Although the morphology was not the same, from the XRD pattern (**Fig. S2g**), it can be seen that their crystal structure was consistent. Thus, the change of morphology was only caused by the addition of $C_6H_{10}O_8$, and the crystal structure would not change with the addition of $C_6H_{10}O_8$ and TC.



Fig. S2 SEM images of (a) N-doped LC-CdO-TC10, (b) N-doped LC-CdO-TC40, (c) Ndoped LC-CdO-CAM0, (d) N-doped LC-CdO-CAM10 and (e) N-doped LC-CdO-CAM30; XRD patterns of samples with (f) different TC addition and (g) different citric acid monohydrate addition.

The HRTEM images of CdO-OH and N-doped LC-CdO and shown them in Fig. S3. CdO-OH exposed complete (111) crystal faces, while the crystal faces of N-doped LC-CdO surface were (111) crystal faces and (200) crystal faces interlaced, indicating that the addition of TC would reconstruct the microstructure of CdO surface⁴. Notably, (200) planes exposed to a higher plane index could potentially expose more lowcoordination atoms⁵. The addition of TC had no effect on the morphology and crystal structure of CdO, but only changed the surface microstructure.



Fig. S3 HRTEM images of (a) CdO-OH and (b) N-doped LC-CdO.



Fig. S4 XPS high-resolution spectra of O 1s. (OL: lattice oxygen; OS: surface hydroxyl

group; O_A : adsorbed oxygen)

According to the XPS high-resolution spectra (**Fig. S5**), with the increase of TC content, the low coordination content also increased gradually, but the increase was not obvious after 25 mg. Therefore, the optimal dose of TC was 25 mg.



Fig. S5 XPS high-resolution spectra of samples with different TC addition. (O_L : lattice oxygen; O_S : surface hydroxyl group; O_A : adsorbed oxygen)

As demonstrated in XPS spectra (**Fig. S6**), CdO-UR contained lattice N, because under high temperature conditions, urea would decompose into a large amount of ammonia to form N doping. However, there was no lattice N in CdO-CIP, indicating that the N element in CIP was easy to escape during the calcination process. In addition, the content of low coordination Cd of these two materials did not increase significantly compared with CdO-OH, indicating that CIP and urea as nitrogen sources could not lead to low coordination effect. In **Fig. S6g-6i**, treatment of CdO-OH with hydrogen at low temperature significantly reduced the hydroxyl group on the surface of CdO-H₂, while increasing the content of low coordination Cd.



Fig. S6 XPS high-resolution spectra of N-doped CdO, CdO-CIP and CdO-H₂. (O_L: lattice oxygen; O_S: surface hydroxyl group; O_A: adsorbed oxygen)

In this experiment, the calcination process was carried out in the air, because a large amount of oxygen in the air would fill the oxygen defect position, which was not conducive to the formation of oxygen vacancy⁵⁻⁷. In **Fig. S7**, no oxygen vacancies were generated in all the samples, indicating that the formation of low coordination was not caused by vacancy engineering.



Fig. S7 EPR signals of different samples.



Fig. S8 (a) EDS spectra of CdO-OH and N-doped LC-CdO; (b) The mapping images of N-doped LC-CdO. (scale: 100 nm)

The results of refinement showed that the cell size of N-doped LC-CdO did not change significantly compared with CdO-OH, and no shift of peak position was observed. This indicated that N doping had little effect on the lattice of the CdO, which might be due to the low doping content (**Table S1** and **S2**). Meanwhile, the structure of the two materials was optimized by theoretical calculation (**Fig. S9d**), and the results also showed that doping did not significantly change the crystal structure. However, some deviation between the optimization results and the actual refinement results was due to the selection of element pseudopotential and the correlation between optimization parameters and optimization accuracy. Therefore, due to the low doping amount of N in this work, N doping did not affect the lattice expansion or contraction of CdO.



Fig. S9 Rietveld refinement results of the XRD patterns of (a) CdO-OH and (b) Ndoped LC-CdO; (c) XRD patterns and (d) Optimized 2×2×2 supercell.



Fig. S10 UV-Vis DRS results of CdO-OH and N-doped LC-CdO.



Fig. S11 Photodegradation and pseudo-first-order kinetic curves of different samples.



Fig. S12 (a) Selectivity of photodegradation and (b) pseudo-first-order kinetic curves for TC and CIP with different samples.



Fig. S13 (a) Mineralization and (b) pseudo-first-order kinetic curves of different samples.



Fig. S14 Dark adsorption results of different samples.

The possible degradation path of TC was analyzed by density functional theory (DFT) calculation and mass spectrometry (MS). The Mulliken Population between different atoms (**Fig. S15a**) was calculated, and the smaller the value, the more likely the bond was to break. The easily broken bonds were indicated with a green background in **Fig. S15b**. In order to better understand the products of TC decomposition, the MS data (**Fig. S16**) at 0, 20 and 40 min of degradation were analyzed. At 0 min, m/z=445 corresponded to the molecular weight of TC. At 20 min, TC gradually decomposed into smaller molecules, and at 40 min, the intensity of m/z=445 decreased to a very low level, which mean that TC molecules almost did not exist. With the progress of reaction time, the peak intensity of MS decreased sharply, which also indicated that the total amount of organic pollutants decreased. This further corroborated the TOC data results.

Accordingly, a more in-depth analysis of Mulliken Population and mass spectrometry data inferred possible intermediate path presented in Fig. S17. The C4-O1, C2-C3, C14-N1, C21-N1, C22-N1 and C20-N2 bonds in TC were prone to fracture, resulting in hydroxyl detachment, amino detachment, N-methyl detachment and ring opening, forming P1 (m/z=419), P2 (m/z=375) and P3 (m/z=375). Due to the continuous attack of free radicals, these three intermediates were also continuously decomposed by oxidation. P1 (m/z=419) shed hydroxyl and -CO(NH₃) into P4 (m/z=297), and further denitrification to form P7 (m/z=268). Simultaneously, P2 (m/z=375) shed hydroxyl and aldehyde groups into P5 (m/z=297), and then deaminated to form P7 (m/z=268). P3 (m/z=375) was also stripped of amino and hydroxyl groups, then ring-opened to P6 (m/z=318), and then further ring-opened and dehydrogenated to P8 (m/z=274) and P9 (m/z=274). Subsequently, the methyl group, hydroxyl group, etc. were separated, and P7 (m/z=268) as well as P8 (m/z=274) became P10 (m/z=230). Gradually, they became smaller molecules P11 (m/z=140), P12 (m/z=112), P13 (m/z=127) and P14 (m/z=112). With the progress of reaction time, these small molecules would eventually become carbon dioxide and water.

To understand the toxicity changes of TC products in the degradation process,

T.E.S.T. software was used to predict and evaluate these intermediate products. In **Fig. S18**, mutagenicity of intermediates decreased to mutagenic negative through toxicity prediction.



Fig. S15 (a) Mulliken Population analysis and (b) chemical structure of TC.



Fig. S16 The m/z of degraded TC with N-doped LC-CdO at (a) 0 min, (b) 20 min and (c) 40 min.



Fig. S17 The proposed degradation pathway of TC.



Fig. S18 The mutagenicity of TC as well as its possible degradation intermediate.



Fig. S19 (a) Free radical capture experiment of N-doped LC-CdO; (b) DMPO-'OH and

(b) DMPO- $\cdot O_2^-$ ESR signals.



Fig. S20 Schematic diagram of photocatalytic degradation mechanism of N-doped

LC-CdO.



Fig. S21 Partial density of states of N-doped CdO.



Fig. S22 (a) Cycling experiment; (b) SEM image and (c) XRD pattern of N-doped LC-CdO after three cycles.



Fig. S23 The bader charge of all Cd atoms in the first layer.



Fig. S24 (a)The model and adsorption energy of TC by LC-CdO; The model and adsorption energy of CIP by (b) CdO-OH and (c) N-doped LC-CdO.

In the spectra (**Fig. S25a-b**) of CdO-OH adsorption of TC under dark conditions, 1550-1000 cm⁻¹ belong to the absorption peak range of TC, which included C=C in benzene ring (1492 cm⁻¹), C-C stretching (1453 and 1438 cm⁻¹), C-N and N-H (1360-1190 cm⁻¹), C-C breathing ring band (1175 cm⁻¹), carbonyl group (1132 cm⁻¹), C-H in-plane vibrations (1120-1000 cm⁻¹) and associated hydroxyl group (about 3300 cm⁻¹).⁶⁻¹⁰ In the initial adsorption process, hydroxyl rapidly adsorbed on the surface of CdO-OH within 4min, resulting in a large number of hydroxyl coordination, while the relative N-doped LC-CdO slowed down this phenomenon. Under the same conditions, the spectrum of N-doped LC-CdO adsorption TC (**Fig. S25c-d**) contained all the peaks mentioned above, and generated new absorption peaks at 2936,2849,1400,1375 and 1295 cm⁻¹. Among them, 1400 and 1375 cm⁻¹ were attributed to CH₃ and C-H asymmetric bending, respectively.^{8,11,12} Meanwhile, the peaks at 2936 and 2849 cm⁻¹ were correlated to stretched vibration peak of -CH₂-, which might be caused by the combination of doped N with methyl carbon in TC to form a new C-N bond (1295 cm⁻¹).¹³



Fig. S25 In-situ ATR-FTIR spectrum at (a) 1550-1000 cm⁻¹ and (b) 4000-2200 cm⁻¹ of TC adsorption on the surface of CdO-OH and In-situ ATR-FTIR spectrum at (c) 1550-1000 cm⁻¹ and (d) 4000-2200 cm⁻¹ of TC adsorption on the surface of N-doped LC-CdO.

Sample	Cd /Atomic %	O /Atomic %	N /Atomic %
CdO-OH	21.95	78.02	0.03
N-doped LC-CdO	22.14	77.12	0.74

 Table S1 XPS content of different samples.

 Table S2 EDS content of different samples.

Sample	Cd /Atomic %	O /Atomic %	N /Atomic %	
CdO-OH	50.33	49.65	0.01	
N-doped LC-CdO	54.82	45.06	0.12	

Table S3 Selectivity coefficient of photodegradation for TC and CIP with different

samples.					
Sample	Contaminant	k (min ⁻¹) K _{Selectivity}		K` _{Selectivity}	
CdO-OH	ТС	0.0317	1 90		
	CIP	0.0176	1.80	4.8	
N-doped LC-CdO	ТС	0.1370	0.67		
	CIP	0.0158	8.67		
Sample CdO-OH N-doped LC-CdO	Contaminant TC CIP TC CIP	k (min ⁻¹) 0.0317 0.0176 0.1370 0.0158	K _{Selectivity} 1.80 8.67	K` _{Sele}	

Table S4 Selectivity coefficient of mineralization for TC and CIP with different

samples.						
Sample	Contaminant	k (min⁻¹)	K _{Selectivity}	K` _{Selectivity}		
CdO-OH	тс	0.0100	2 1 2			
	CIP	0.0047	2.13	2.38		
N-doped LC-CdO	тс	0.0253	5.00			
	CIP	0.0050	5.06			

		•	-	-			
		Light Time	Contaminan	К	K' _{Selectivity}		
Sample		(min)	t	(min ⁻		Ref.	
		source (min	(1111)		¹)		
1	CdO _{0.5} S _{0.5}	visible	60	TC	0.022	2.08	14
2	TiO ₂	UV	180	2,4-D	0.020	1.58	15
3	C/TiO ₂	UV	90	CIP	0.028	3.00	16
4 TiO ₂ (001)	T:0 (004)	UV-	262		0.009	1.37	17
	$HO_2(001)$	visible	360 2,4	2,4-D			17
5 ZnO/CuFe ₂ O ₄	UV-		MB	0.038	2.56	10	
	ZnO/CuFe ₂ O ₄	120 visible				18	
6	Bi ₂ O ₃ -doped TiO ₂	visible	80	MG	0.054	1.76	19
7	Ag/Ag ₃ VO ₄ /g-C ₃ N ₄	UV	120	тс	0.018	3.2	20
8	TiO ₂	UV	240	2,4-D	0.006	2.32	21
9	Ag/Zn-TiO ₂	UV	120	E-pHB	0.043	4.8	22
10	Ag ₂ S-TiO ₂	UV	120	E-pHB	0.052	3.57	23
11 n		UV-	600	Atrazine	0.005	2.04	24
	meso-TiO ₂	visible					24
	N-doped LC-CdO	visible		тс	0.137	4.8	This
12			20				work

Table S5 Photocatalytic selectivity coefficient and apparent rate constant over

different reported photocatalytic materials.

Notes: 2,4-D (2,4-Dichlorophenoxyacetic acid), MG (Malachite green) and E-pHB (Ethyl p-hydroxybenzoate).

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