

Figure S1  $^1\text{H}$  NMR spectra of Tp (600 MHz,  $\text{CDCl}_3$ ): -OH (14.118 ppm, S, 3H), -CHO (10.154 ppm, S, 3H)

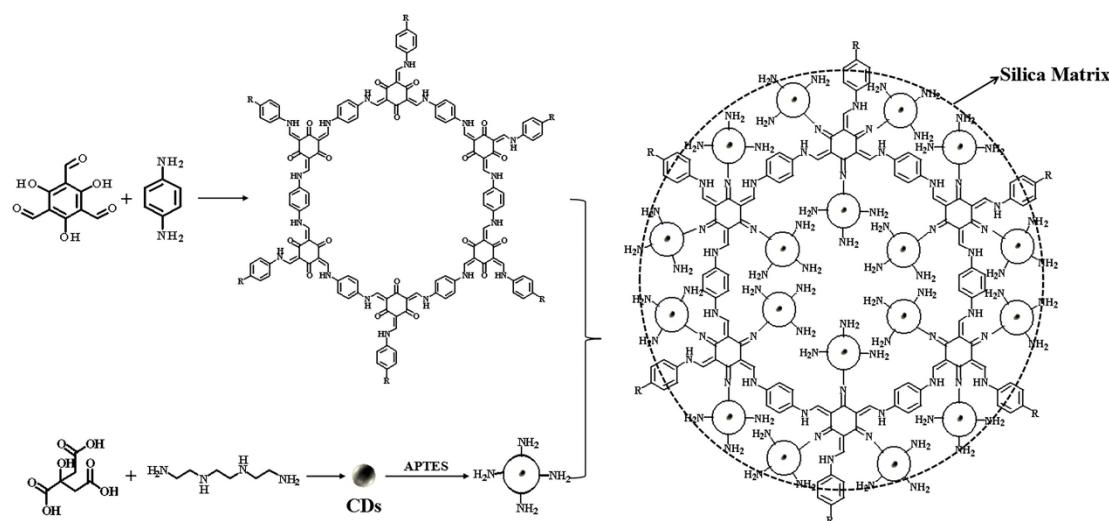
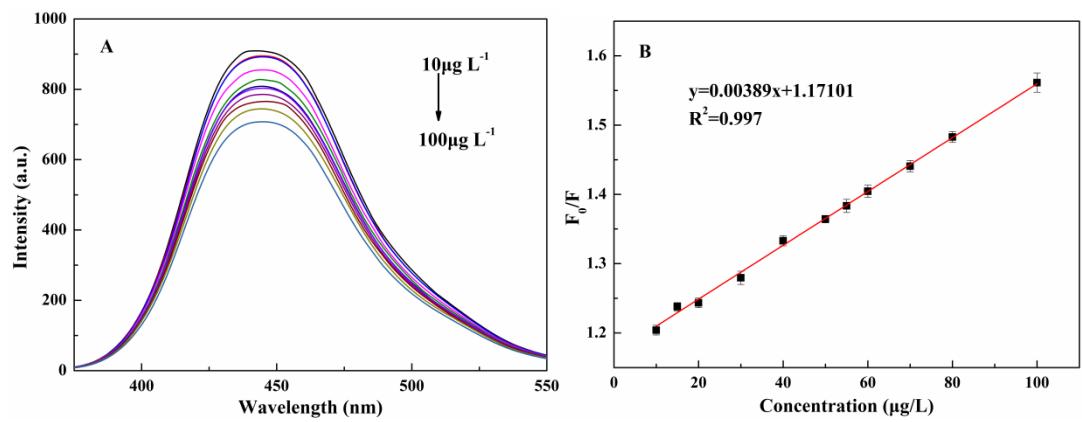


Figure S2 Preparation scheme of TpPa-1 COF@CDs



**Figure S3** Fluorescence spectrum of TpPa-1 COF@CDs quenched by series concentration Hg<sup>2+</sup> (A) and fluorescence titration curve for Hg<sup>2+</sup> (B) in pure water

**Table S1 Relevant parameters of Nenjiang River sample**

Test items	Temperature (°C)	pH	Electric conductivity (ms m <sup>-1</sup> )	Dissolved oxygen (mg L <sup>-1</sup> )	Permanganate (mg L <sup>-1</sup> )	Five-day biochemical oxygen demand (mg L <sup>-1</sup> )				Oil (mg L <sup>-1</sup> )	Volatile phenol (mg L <sup>-1</sup> )	COD (mg L <sup>-1</sup> )	Total nitrogen (mg L <sup>-1</sup> )	Total phosphorus (mg L <sup>-1</sup> )	Mercury (mg L <sup>-1</sup> )
						Ammonia nitrogen (mg L <sup>-1</sup> )									
Test value	12.0	8.0	14.8	8.38	3.8	1.3	0.126	—	—	17	0.42	0.02	—	—	—
Test items	Selenium (mg L <sup>-1</sup> )	Arsenic (mg L <sup>-1</sup> )	Hexavalent chrome (mg L <sup>-1</sup> )	Cyanide (mg L <sup>-1</sup> )	Anionic surfactant (mg L <sup>-1</sup> )	Sulfide (mg L <sup>-1</sup> )	Boron (mg L <sup>-1</sup> )	Stibium (mg L <sup>-1</sup> )	Formaldehyde (mg L <sup>-1</sup> )	Hydrazine hydrate (mg L <sup>-1</sup> )	Butylxanthic acid (mg L <sup>-1</sup> )	Active chlorine (mg L <sup>-1</sup> )	—	Titanate (mg L <sup>-1</sup> )	—
Test value	—	0.0007	—	—	—	—	—	0.0004	—	—	—	—	—	—	—

“—” represented that the test items was not detected in Nenjiang River sample.

**Table S2 The fluorescence lifetime of TpPa-1 COF@CDs solution before and after Hg<sup>2+</sup> addition**

System components	$\tau_1$ (ns)	$\tau_2$ (ns)	$T_{av}$ (ns)	$R^2$
TpPa-1 COF@CDs	4.0366	13.5251	11.99	0.8169
TpPa-1 COF@CDs+Hg <sup>2+</sup>	2.4534	9.5247	7.08	0.9867

**Table S3 Results of actual water sample recoveries (n=5)**

Analytical samples	Detected value ( $\mu\text{g L}^{-1}$ )	Added ( $\mu\text{g L}^{-1}$ )	Found ( $\mu\text{g L}^{-1}$ )	Recovery (%)	RSD (%)
Nenjiang River	— <sup>a</sup>	0	— <sup>b</sup>	—	—
	— <sup>a</sup>	20.00	19.15	95.76	5.66
	— <sup>a</sup>	60.00	59.04	98.40	4.38
		90.00	94.21	104.68	7.62

—<sup>a</sup> and —<sup>b</sup> represented that the concentration of Hg<sup>2+</sup> in blank water sample was below detection limit using GB 3838-2002 method and the fluorescence method of this work, respectively.

**Table S4 Comparison of TpPa-1 COF@CDs with other fluorescent COFs**

Names of Fluorescent COFs	Reaction functional monomer	Reaction time (h)	Adsorption capacity (mg g <sup>-1</sup> )	Fluorescence (μg L <sup>-1</sup> )	LOD	Recyclable times	References
AH-COF	2,5-dihydroxy-3,6-diallyltetraphthalohydrazide (synthesized through three organic reaction procedures); 1,3,5-trisformylbenzene	72 h	—	20	4	10	
TNPP	4-(bis(4-bromophenyl) amino) benzaldehyde, tris (4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2yl) phenyl) amine, thiosemicarbazone	84 h	47.8	22.8	3	49	
COF based amine and sulfonyl groups	2,4,6-trihydroxybenzene-1,3,5-tricarbaldehyde, 2,5-diaminobenzene sulfonic acid	72 h	98.42	640	5	19	
TFPPy-CHYD	1,3,6,8-tetrabromopyrene, 4-formylphenylboronic acid, triphenylphosphine	72 h	758	3.41	6	52	
NOP-28	cyanuric chloride, anhydrous AlCl <sub>3</sub> , diphenylsulfane	24 h	658	12	—	54	
TPE-S-COF	4,4'-dithiodianiline, 4',4'',4''',4''''-(ethene-1,1,2,2-tetrayl) tetrakis(([1,1'-biphenyl]-4-carbaldehyde))	120 h	42.4	38.7	3	16	
TpPa-1 COF@CDs	p-phenylenediamine, 1,3,5-triformylphloroglucinol, tartaric acid, ethylenediamine	20 h	235	0.75	5	This work	

“—” represented that it is not mentioned in the previous report.

**Table S5** The previous methods of detection of Hg<sup>2+</sup> using carbon dots as fluorescent probe

Carbon source	Detection limit (μmol/L)	Concentration range (μmol/L)	References
catechol	0.015	0.015-10	29
folic acid	0.1	0-16	30
ampicillin sodium	0.033	0-50	32
ammonium citrate	0.41	0-20	33
2,4,6-triaminopyrimidine	0.011	0-10	34