

Supporting Information

Oxytetracycline-derived carbon dots for the fluorescence switch of trace ferric ion sensing

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Instrumentation methods

The size and morphology of the as-prepared OCDs were characterized using a transmission electron microscope (TEM, JEM-2100F microscope). The OCDs were ultrasonically dispersed in ethanol and TEM samples were prepared by adding two drops of OCDs ethanol solution on an ultrathin carbon film and then dried at room temperature overnight. Raman spectroscopy (LabRAM HR800) was conducted under a 532 nm excitation laser. The functional groups and chemical components were tested using a FT-IR Prestige-21 with a KBr disk in the range of 4000 to 500 cm^{-1} and an X-ray photoelectron spectrometer (XPS, ESCALAB 250Xi X-ray photoelectron spectrometer), respectively. The sample preparation for the FT-IR measurement was conducted by firstly mixing the as-prepared OCDs and KBr, then uniformly grinding and pressing them to make sample tablets prior to the FT-IR testing. The Fluorescence spectrum was performed on a Hitachi F-2500 typed and Perkin Elmer LS-55 typed fluorescence spectrophotometers using quartz cuvettes. The slit bands of both the excitation and emission were 5 nm. Fluorescence cell images were collected by the home-built multifunctional microscope equipped with bright-field and fluorescent modules.

Quantum yield measurement of OCDs

The quantum yield (QY) of the synthesized OCDs was calculated by using quinine sulfate in 0.1 M H_2SO_4 (QY₂ is 0.54) as a standard reference and was calculated using the following equation (Eq.S1):

$$\text{QY}_1 = \text{QY}_2 \frac{S_1 A_2 n_1^2}{S_2 A_1 n_2^2} \quad (\text{Eq.S1})$$

where, "S" is the integrated area of the fluorescence emission spectrum., "A" is the absorbance, and "n" is the refractive index of the solvent. The representation of subscript "1" is the parameter of OCDs, and the representation of subscript "2" is the parameter of quinine sulfate in reference solution.

Product yield measurement of OCDs

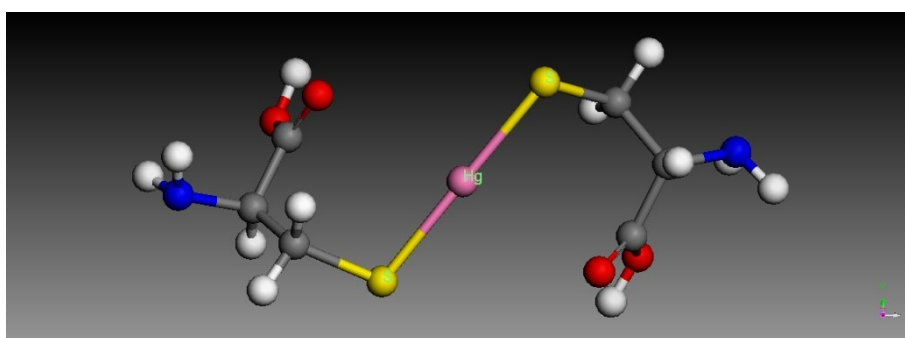
The product yield (PY) of the synthesized OCDs was determined according to Eq.S2:

$$\text{PY} = (m_{\text{OCDs}}/m_p) \times 100\% \quad (\text{Eq.S2})$$

where m_{OCDs} and m_p refer to the mass of as-prepared OCDs powder and precursor respectively.

Mechanism on masking effect of cysteine

The mercury is a thiophilic element and thus the mercaptide is formed after adding cysteine to the Hg(II)-containing solution, resulting in a dramatic drop of Hg(II) concentration. Moreover, not only the formation of Hg-mercaptide can avoid the interaction between Hg(II) with OCDs, but also it is non-fluorescent. Consequently, it displays the masking effect on Hg(II) ions.



Structure of Hg(II)-cysteine complex

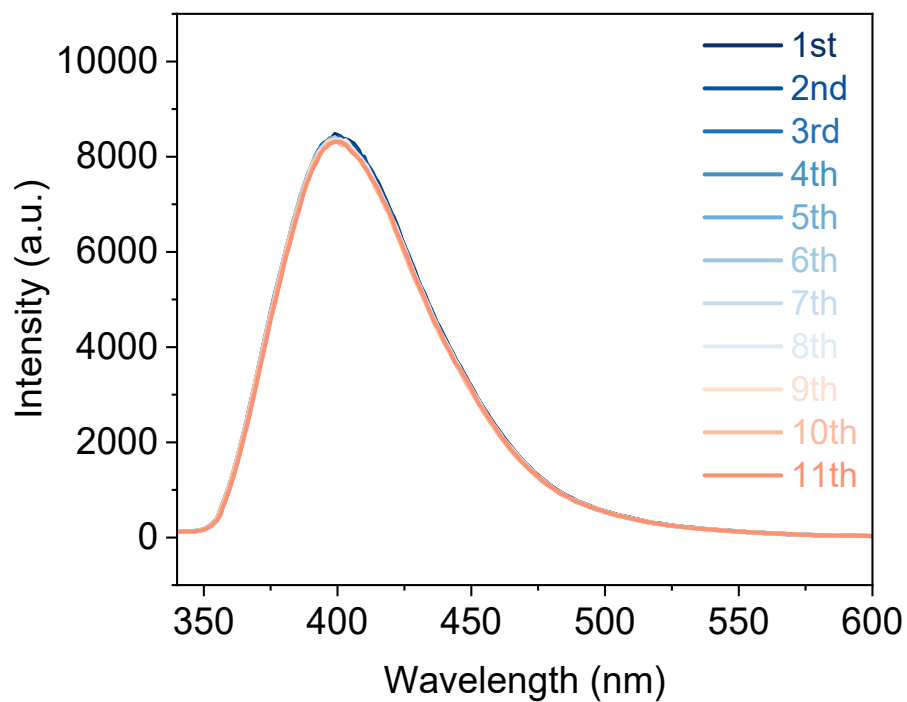


Fig. S1 Fluorescence emission spectra of OCDs (11 repetitions of the test).

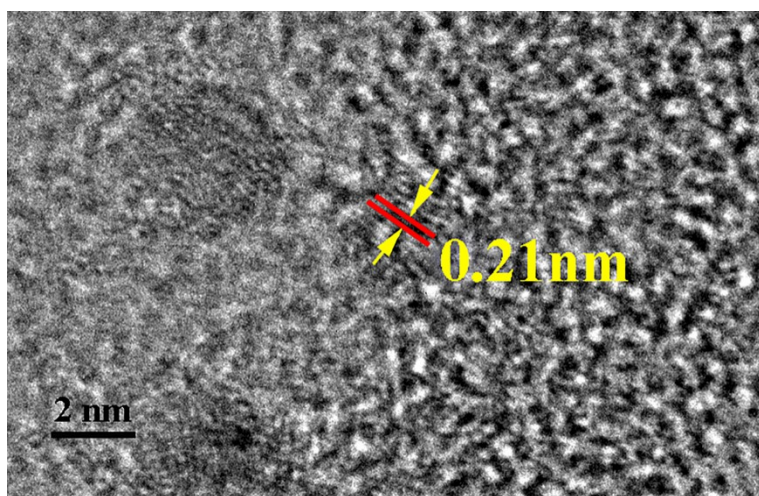


Fig. S2 HRTEM images of synthesized OCDs.

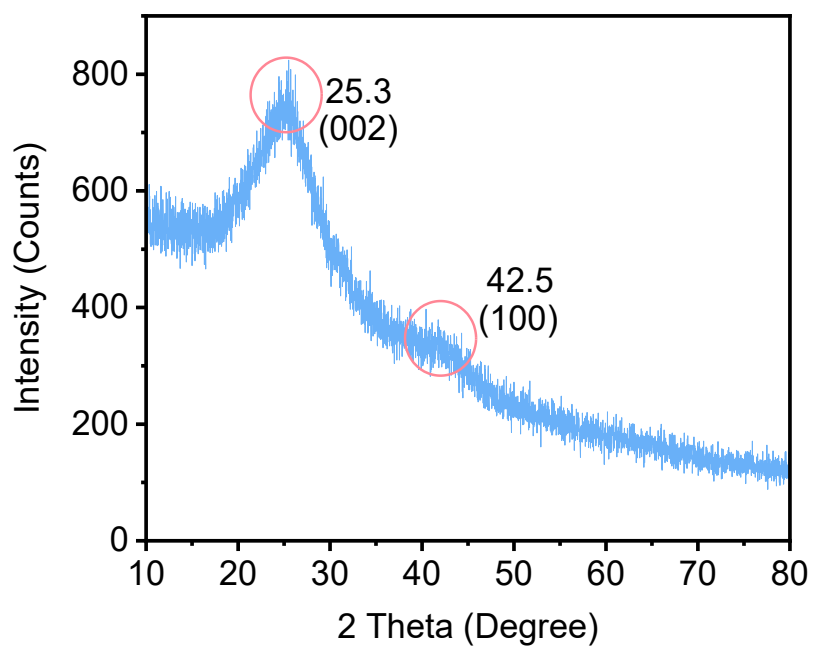


Fig. S3 XRD patterns of synthesized OCDs.

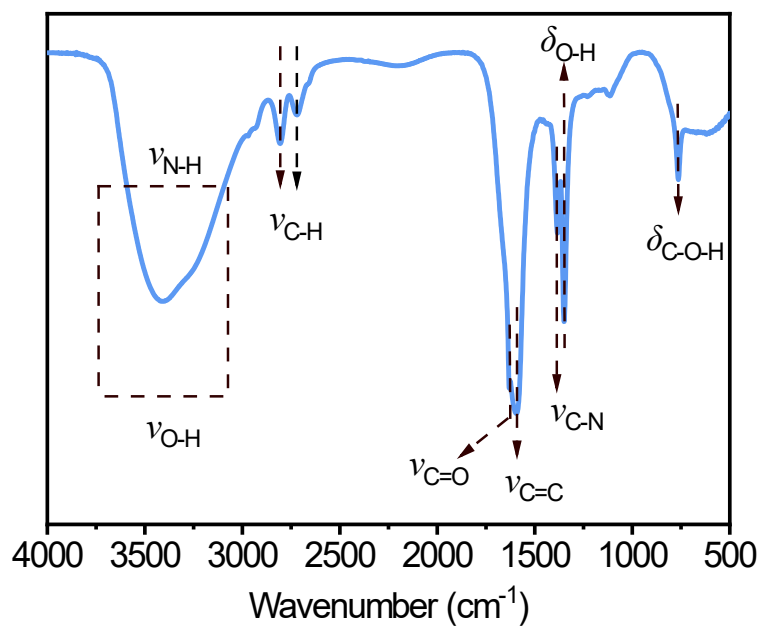


Fig. S4 FT-IR spectrum of synthesized OCDs.

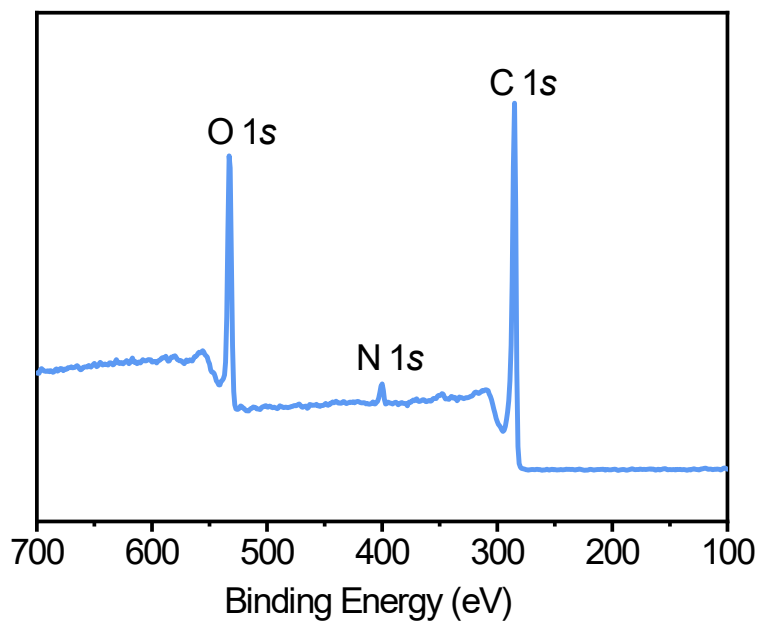


Fig. S5 Survey of XPS spectra of synthesized OCDs.

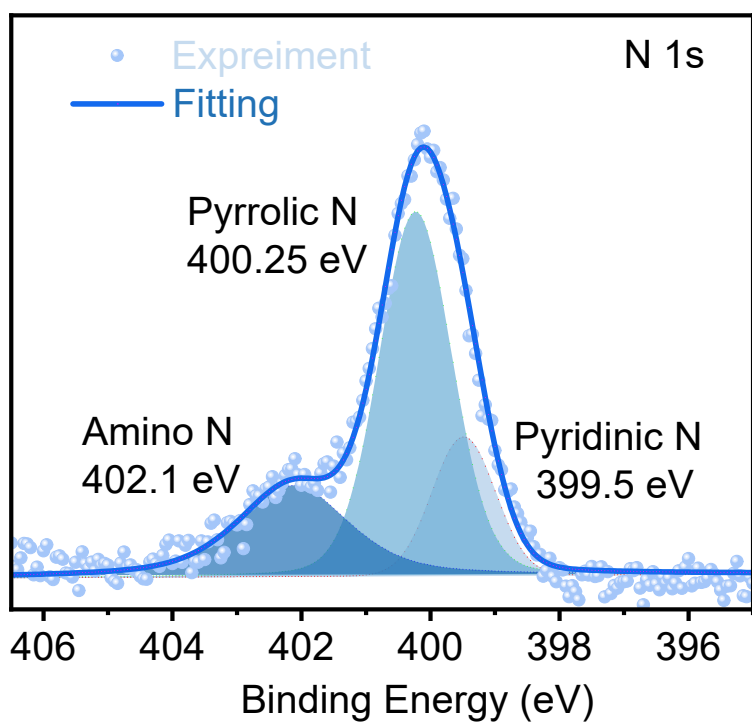


Fig. S6 High-resolution N1s profile of XPS spectra of synthesized OCDs.

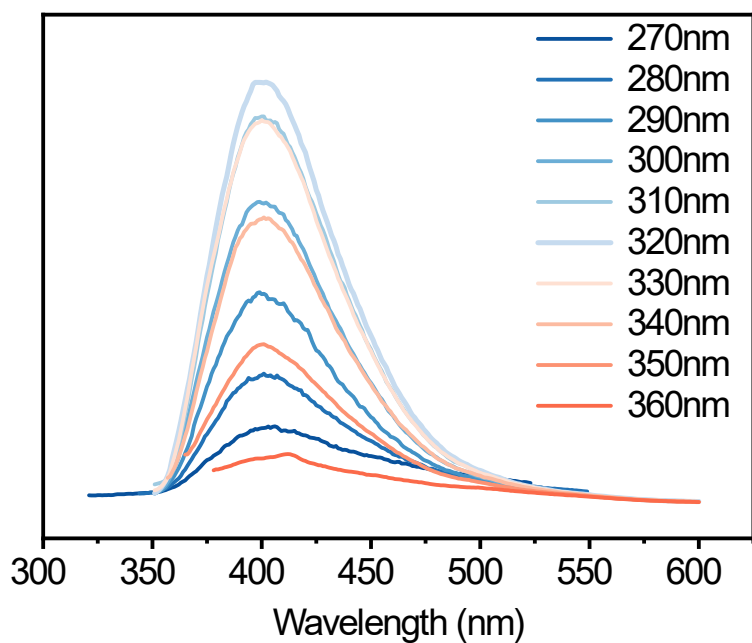


Fig. S7 Fluorescence emission spectra of OCDs at different excitation wavelengths.

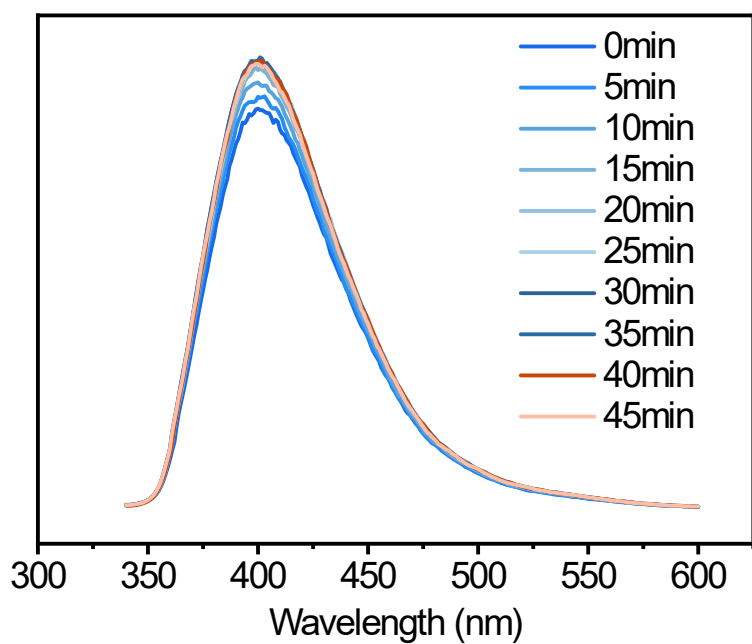


Fig. S8 Fluorescence emission spectra of OCDs at different time.

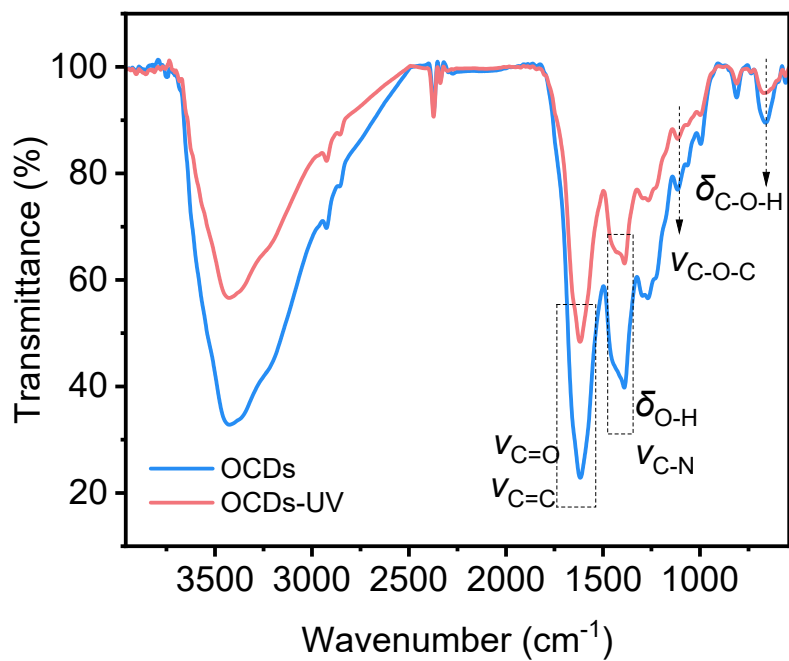


Fig. S9 FT-IR spectrum of OCDs powder before and after 1h ultraviolet irradiation.

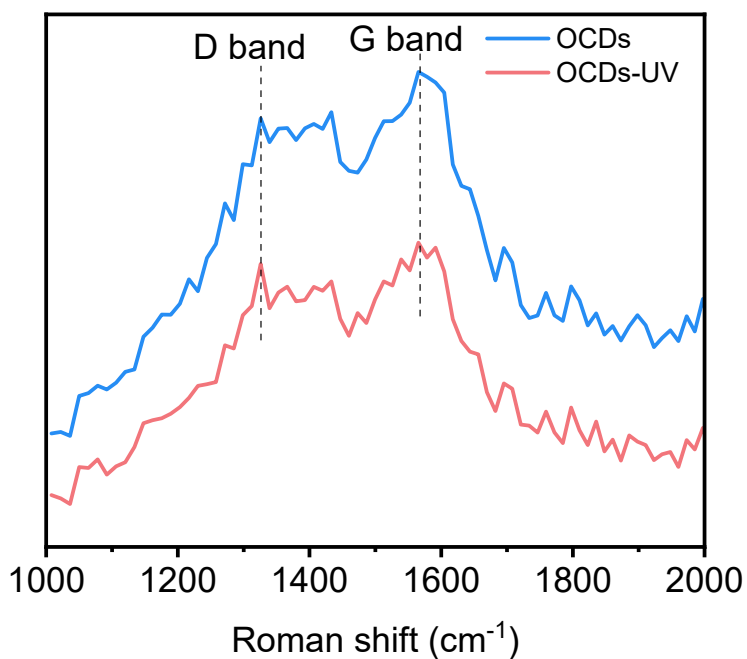


Fig. S10 Raman spectrum of OCDs powder before and after 1h ultraviolet irradiation.

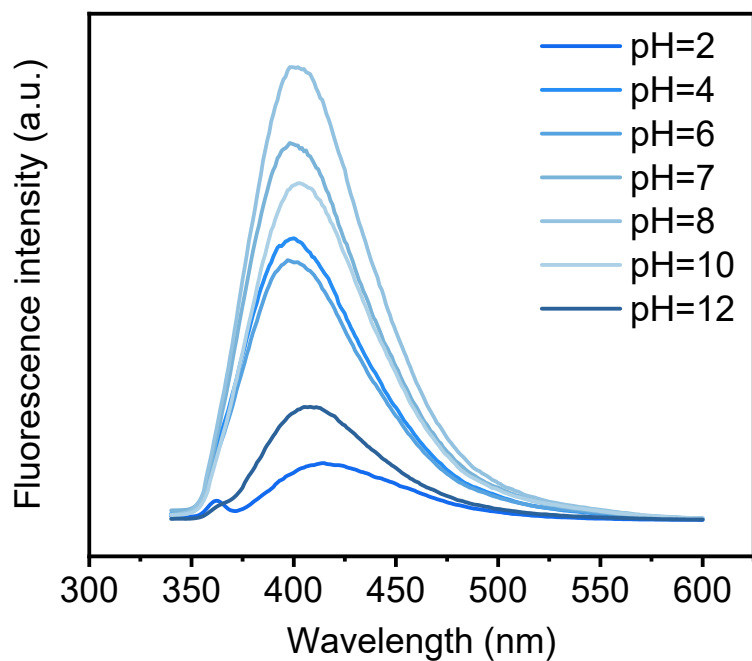


Fig. S11 Fluorescence emission spectra of OCDs at different pH conditions.

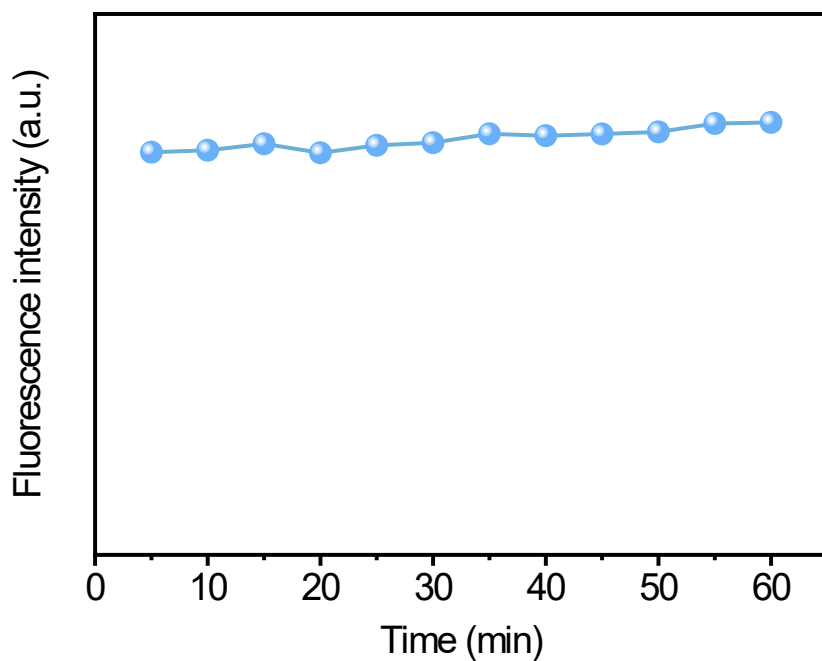


Fig. S12 Fluorescence emission intensities of OCDs-Fe(III) change upon the increase of time.

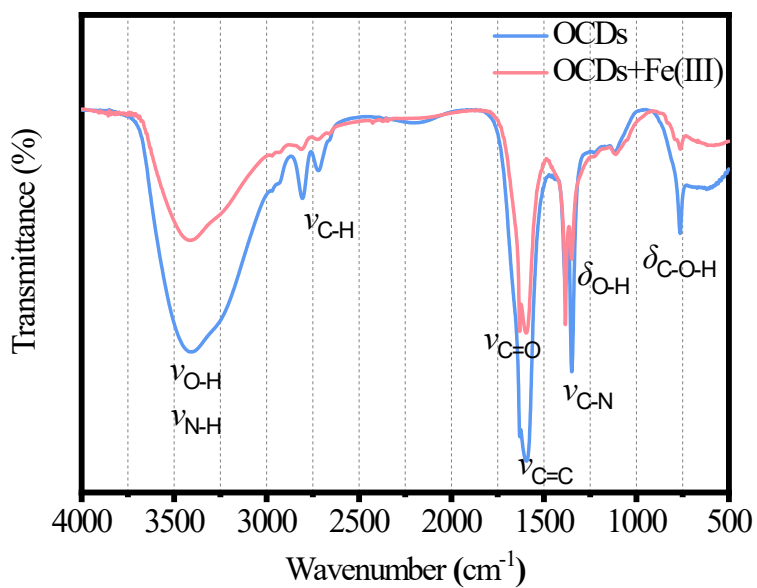


Fig. S13 FT-IR spectrum of OCDs and Fe(III)-OCDs.

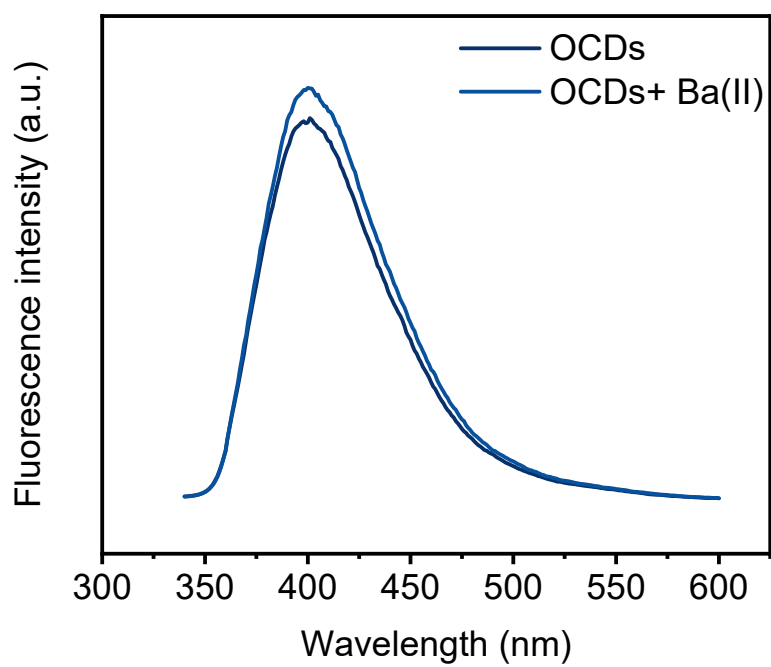


Fig. S14 Fluorescence spectra of OCDs with and without Ba(II).

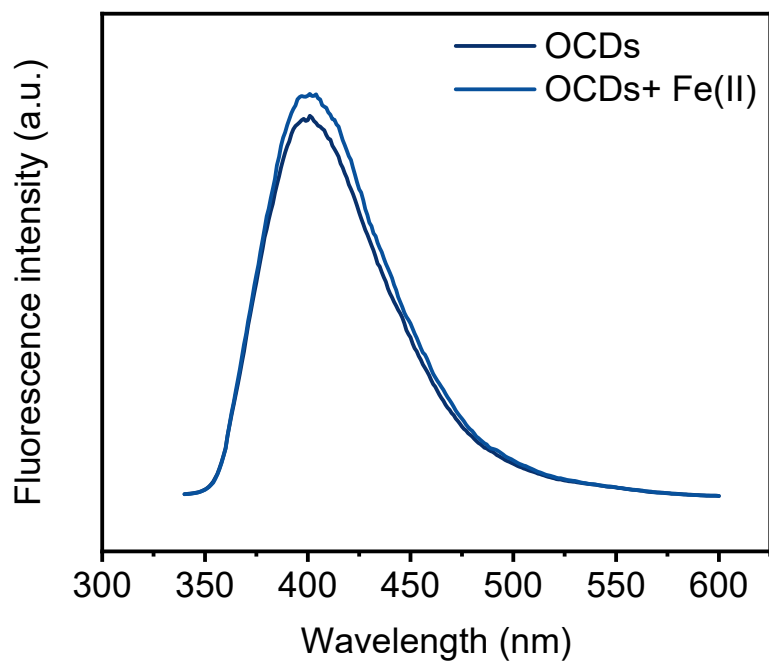


Fig. S15 Fluorescence spectra of OCDs with and without Fe(II).

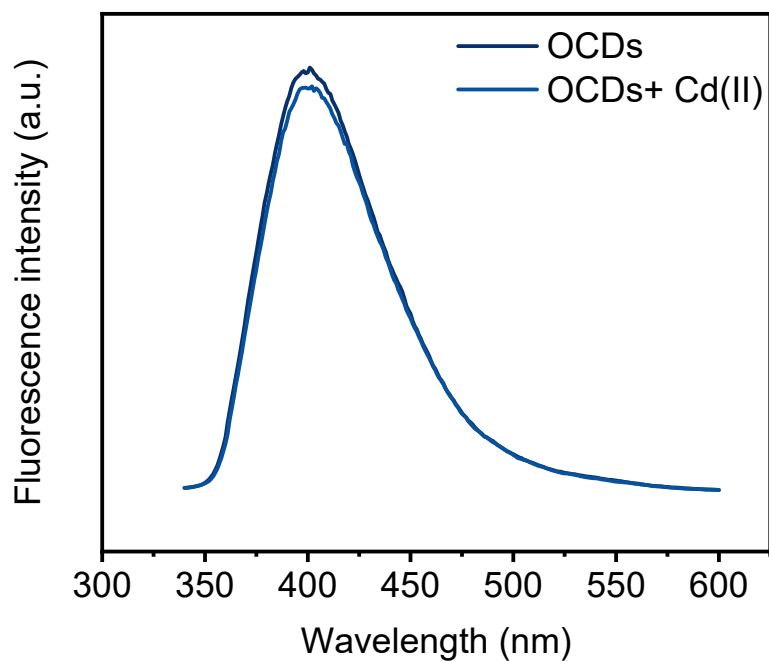


Fig. S16 Fluorescence spectra of OCDs with and without Cd(II).

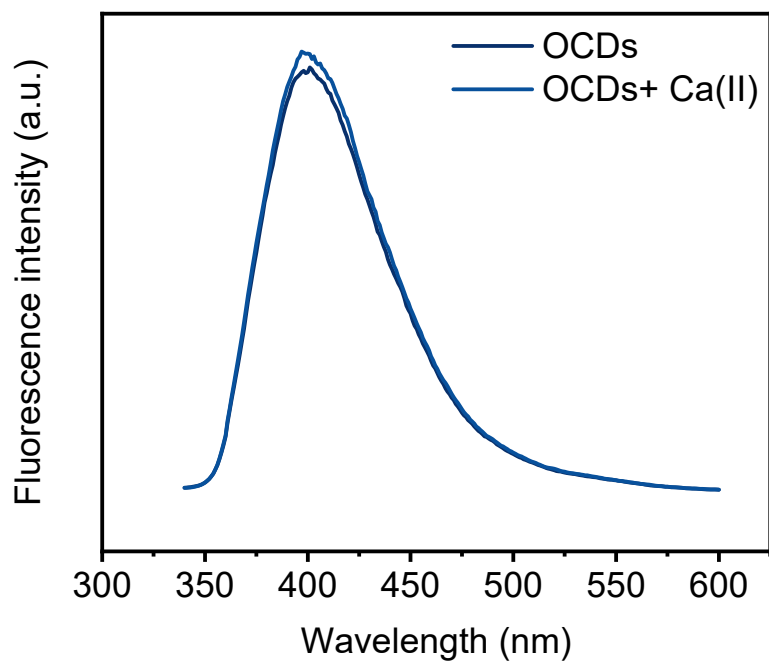


Fig. S17 Fluorescence spectra of OCDs with and without Ca(II).

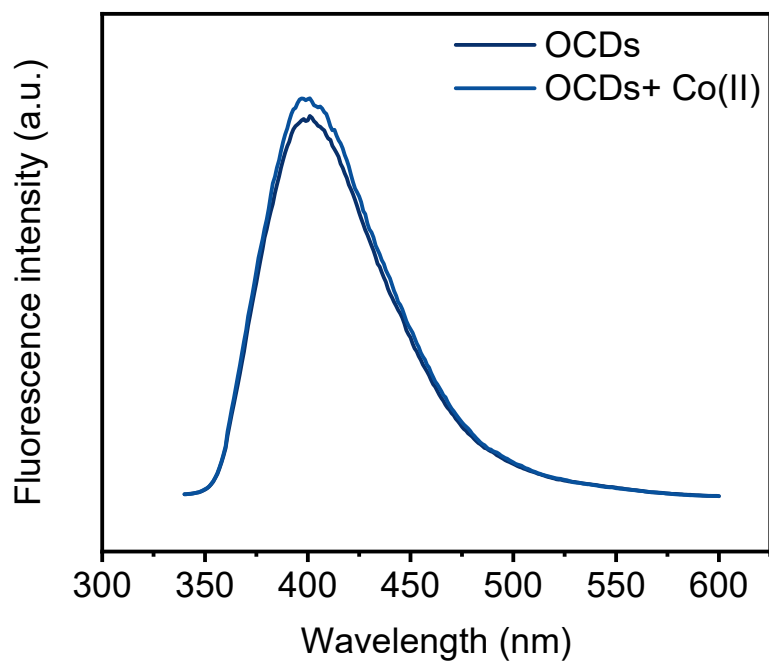


Fig. S18 Fluorescence spectra of OCDs with and without Co(II).

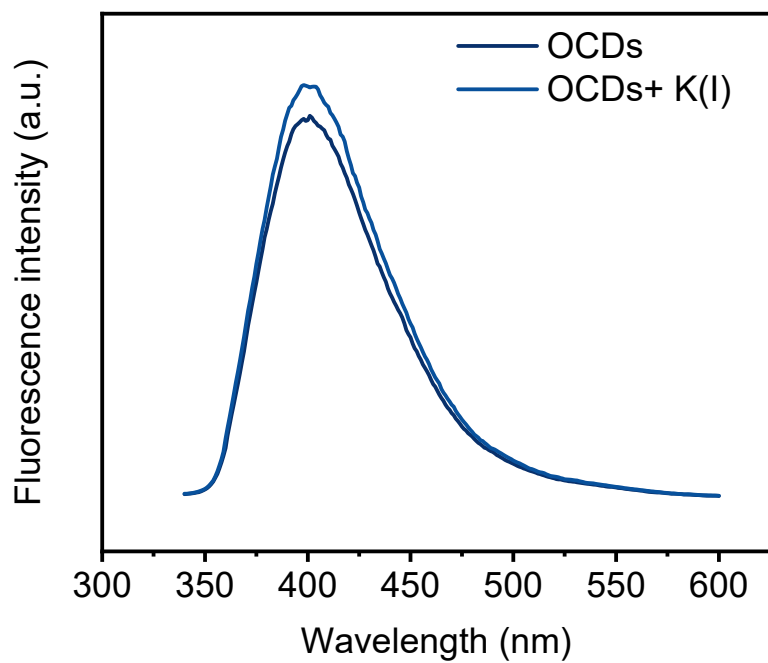


Fig. S19 Fluorescence spectra of OCDs with and without K(I).

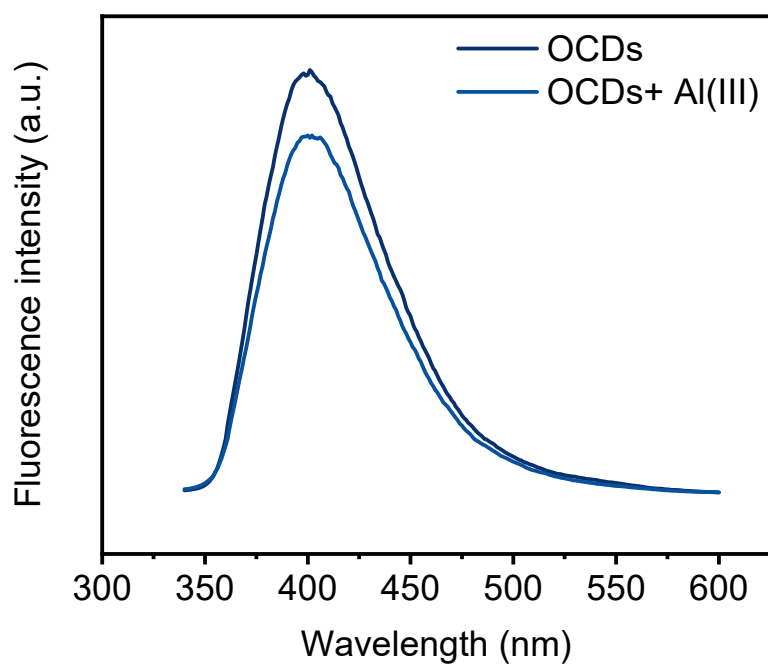


Fig. S20 Fluorescence spectra of OCDs with and without Al(III).

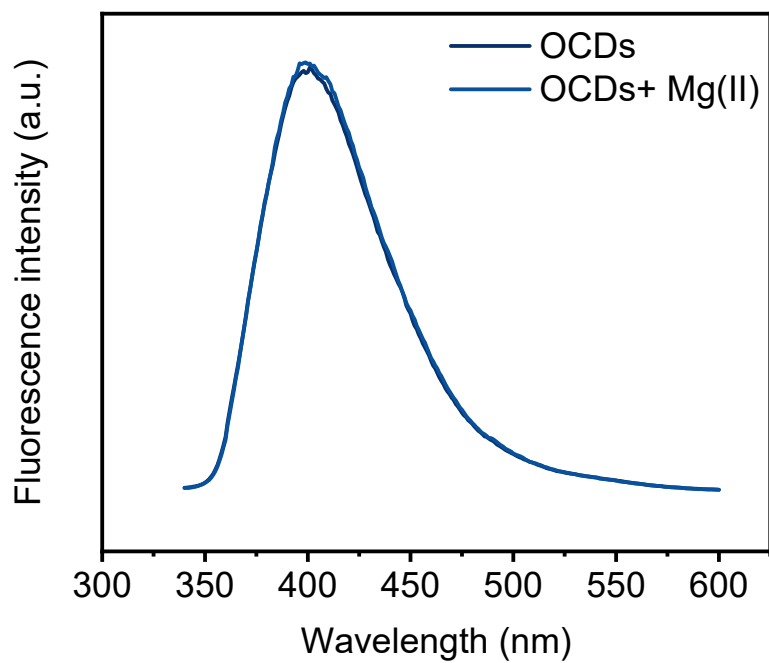


Fig. S21 Fluorescence spectra of OCDs with and without Mg(II).

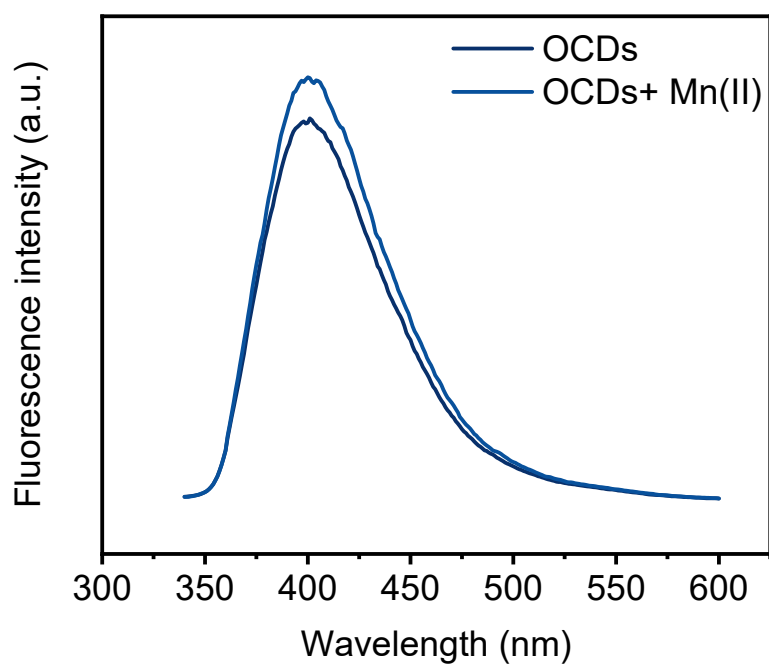


Fig. S22 Fluorescence spectra of OCDs with and without Mn(II).

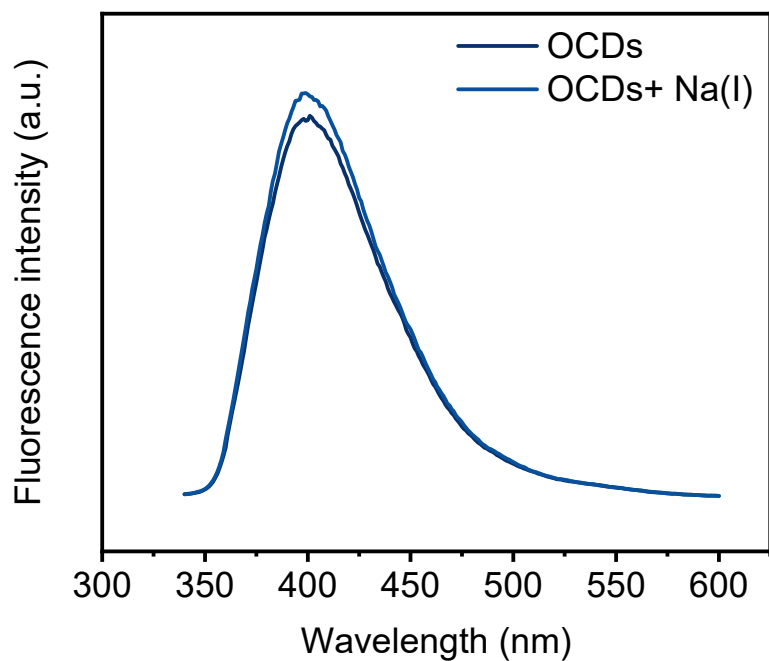


Fig. S23 Fluorescence spectra of OCDs with and without Na(I).

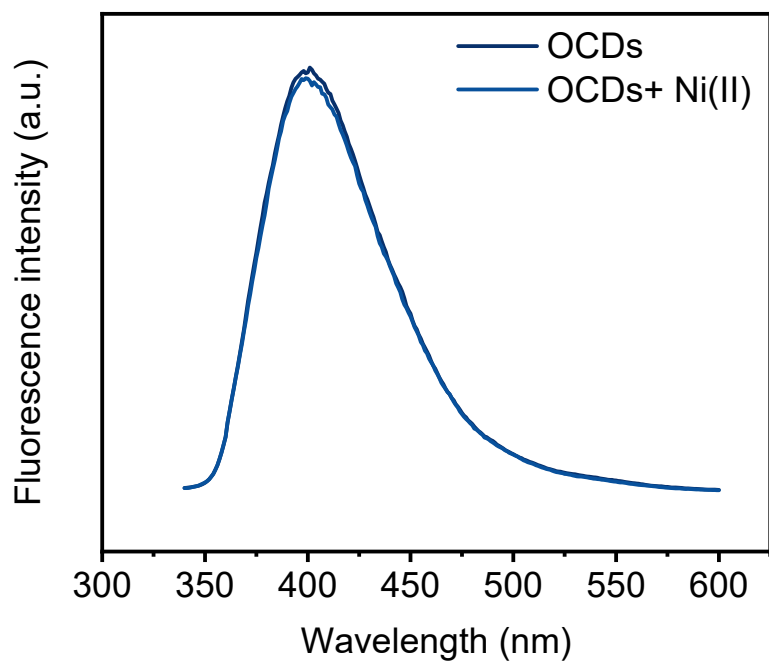


Fig. S24 Fluorescence spectra of OCDs with and without Ni(II).

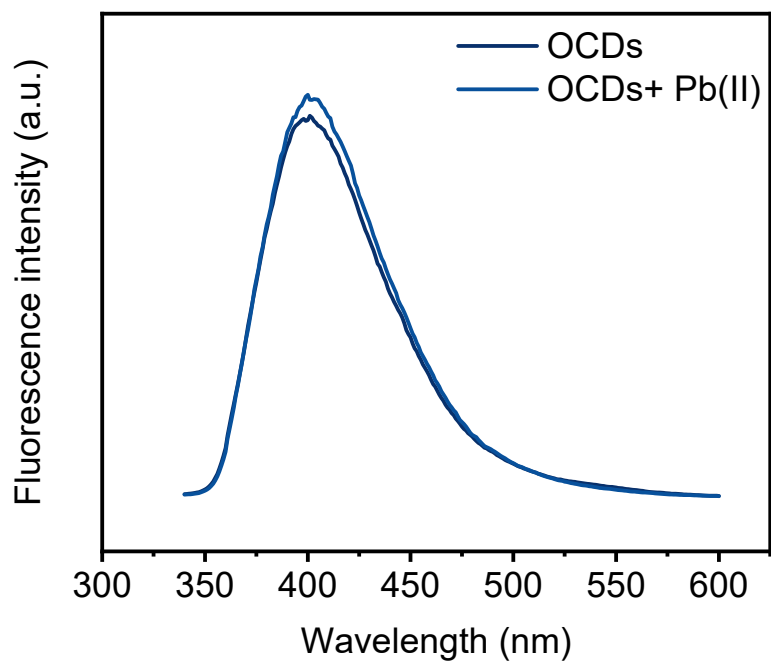


Fig. S25 Fluorescence spectra of OCDs with and without Pb(II).

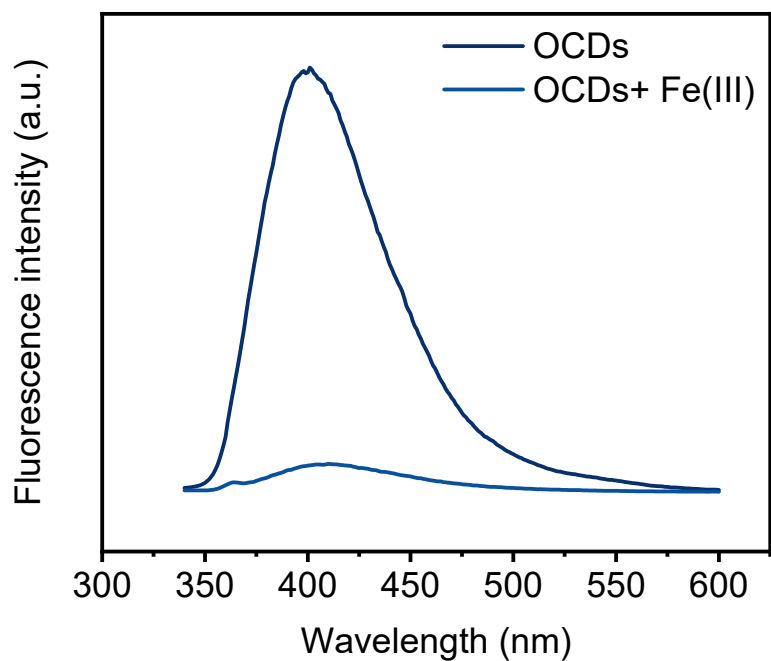


Fig. S26 Fluorescence spectra of OCDs with and without Fe(III).

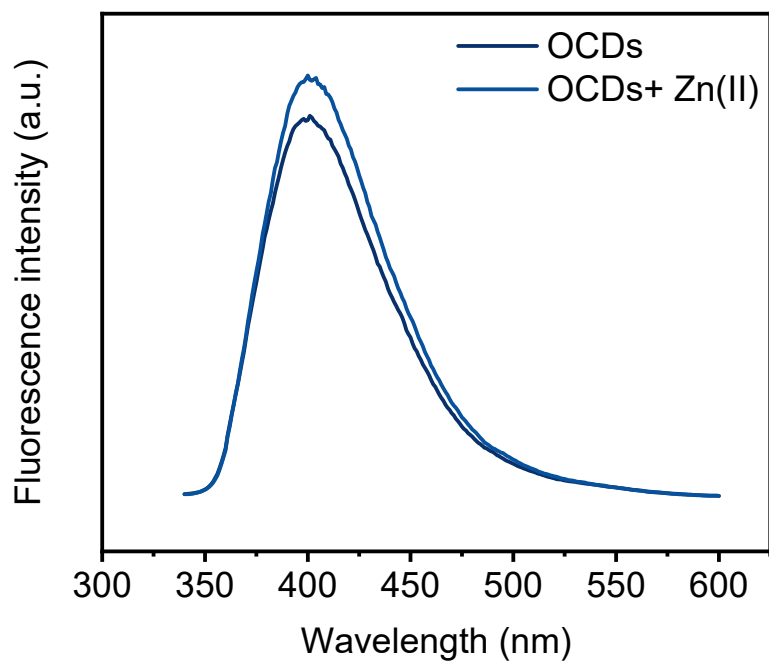


Fig. S27 Fluorescence spectra of OCDs with and without Zn(II).

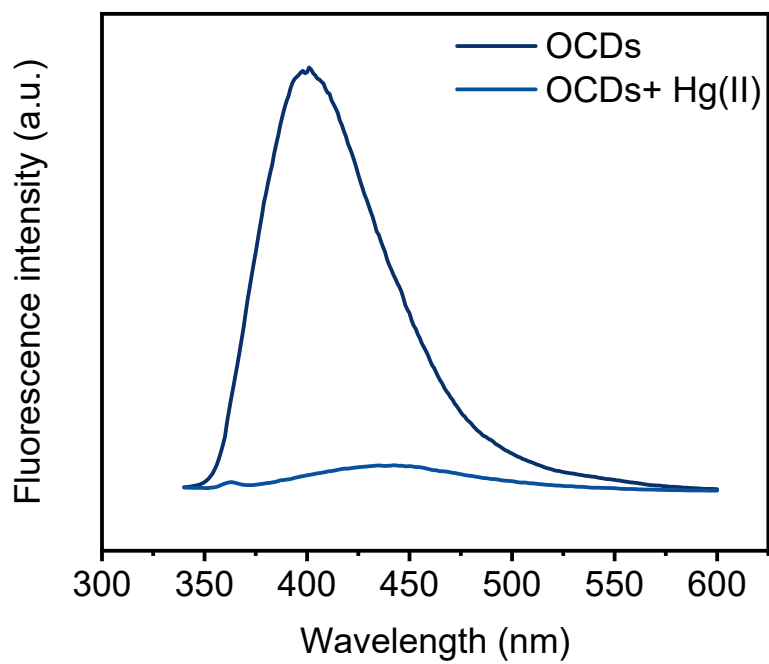


Fig. S28 Fluorescence spectra of OCDs with and without Hg(II).

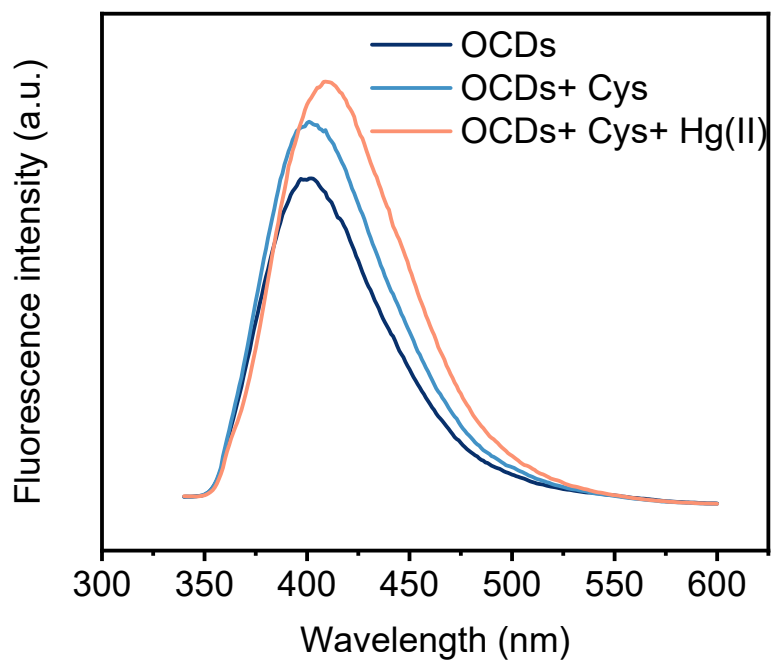


Fig. S29 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Hg(II).

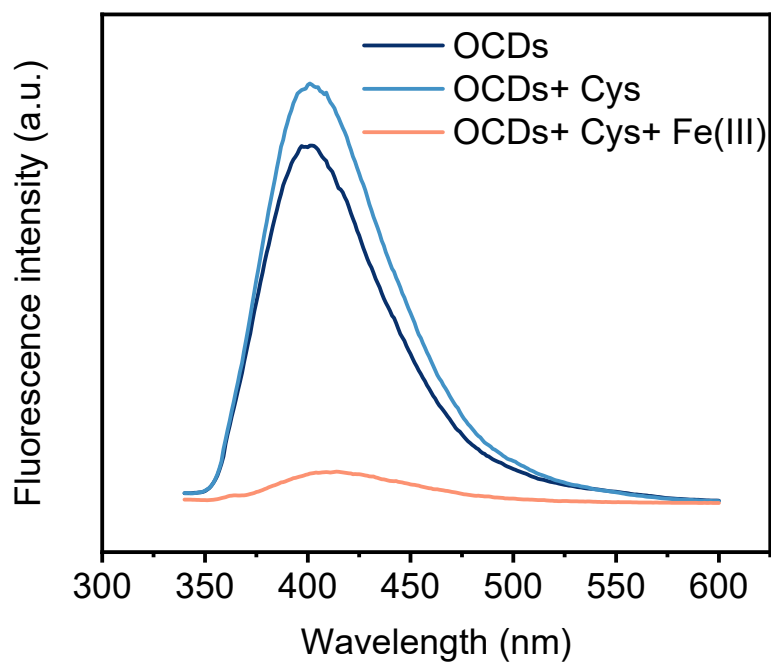


Fig. S30 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Fe(III).

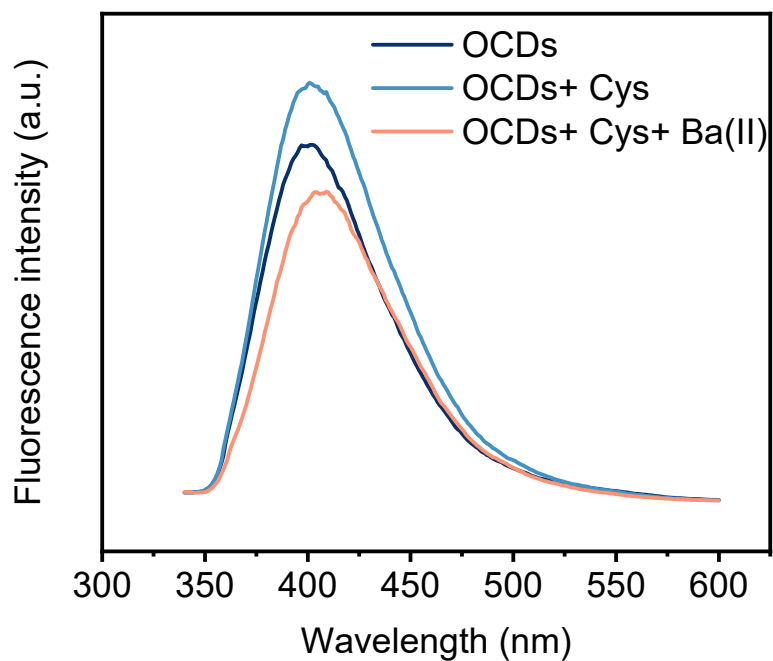


Fig. S31 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Ba(II).

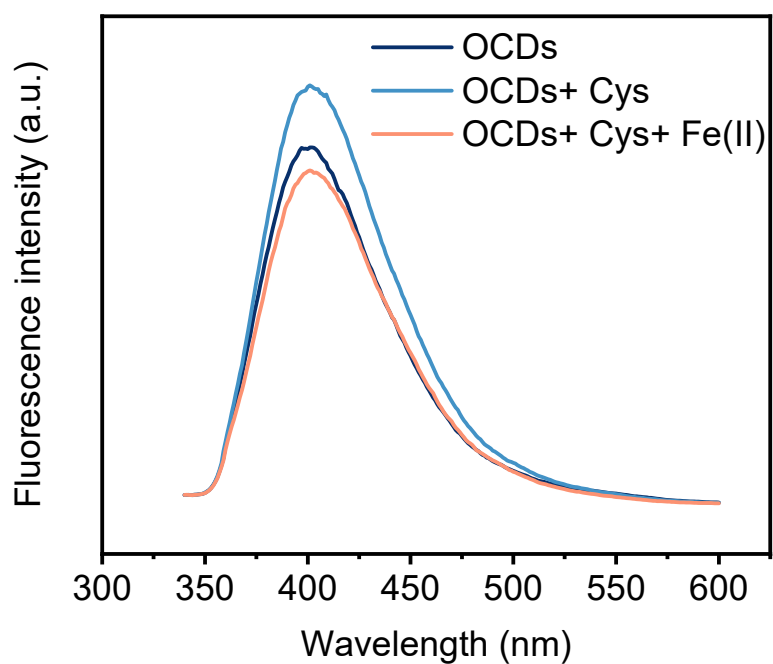


Fig. S32 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Fe(II).

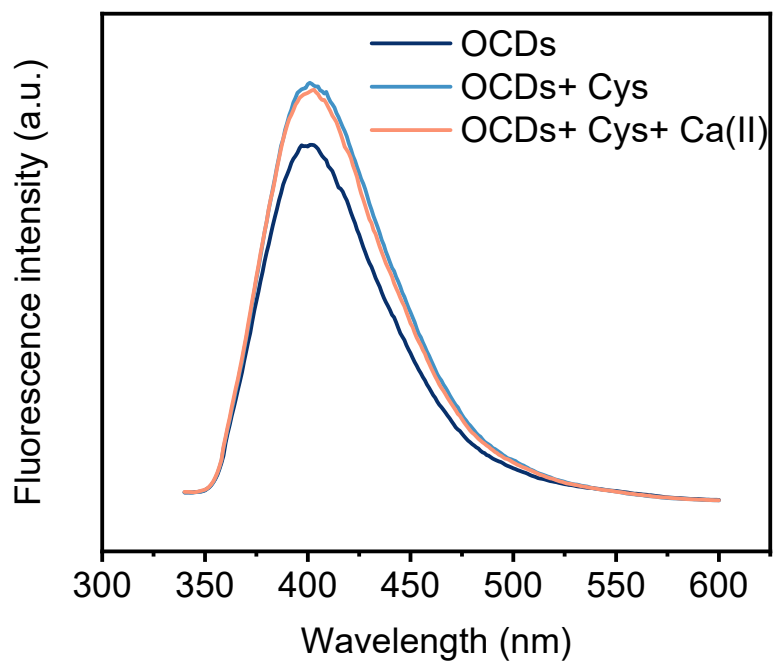


Fig. S33 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Ca(II).

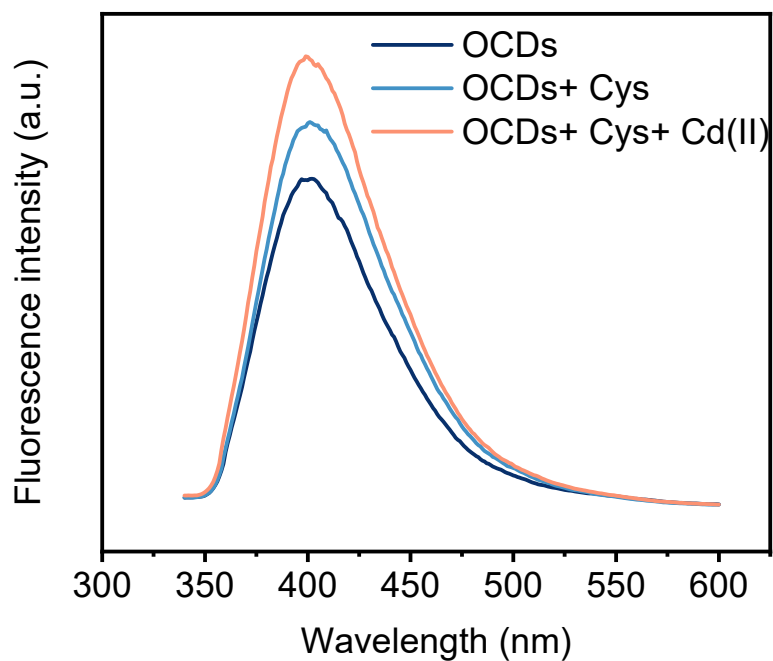


Fig. S34 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Cd(II).

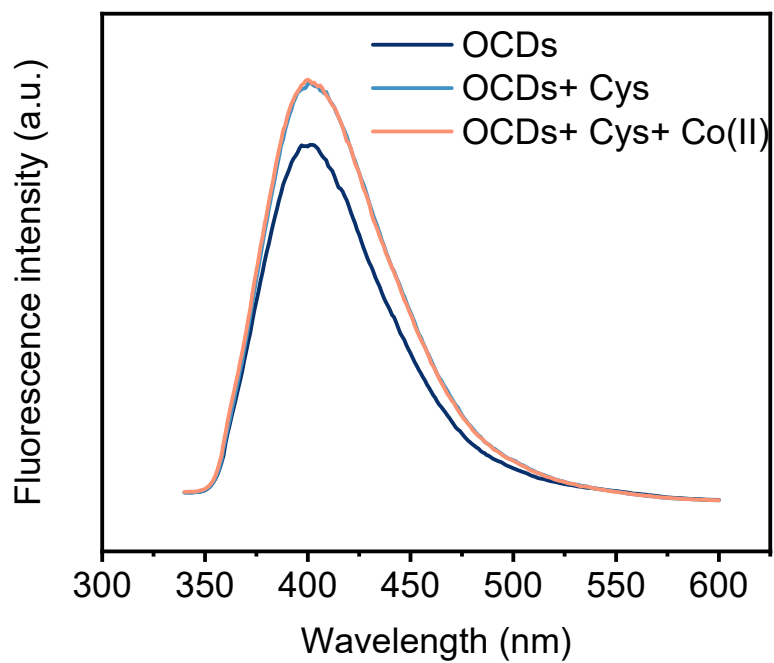


Fig. S35 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Co(II).

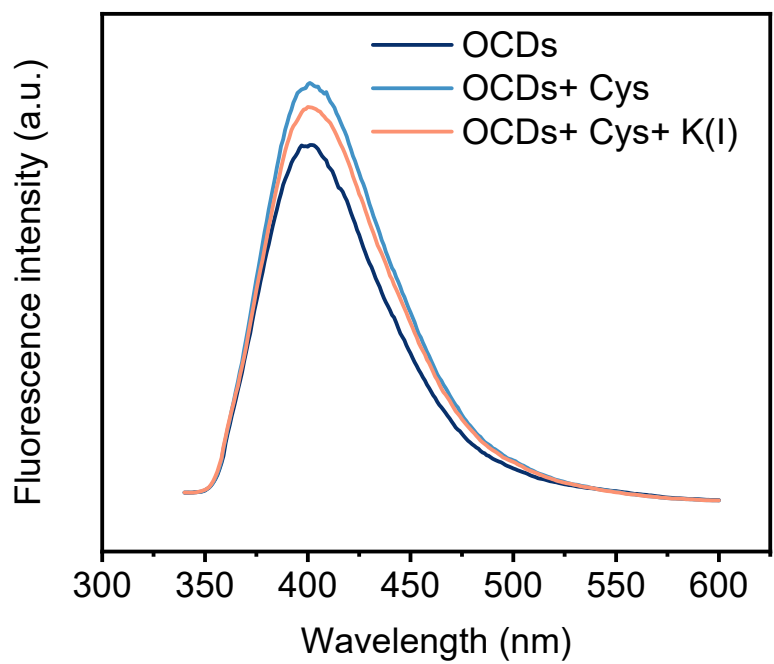


Fig. S36 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + K(I).

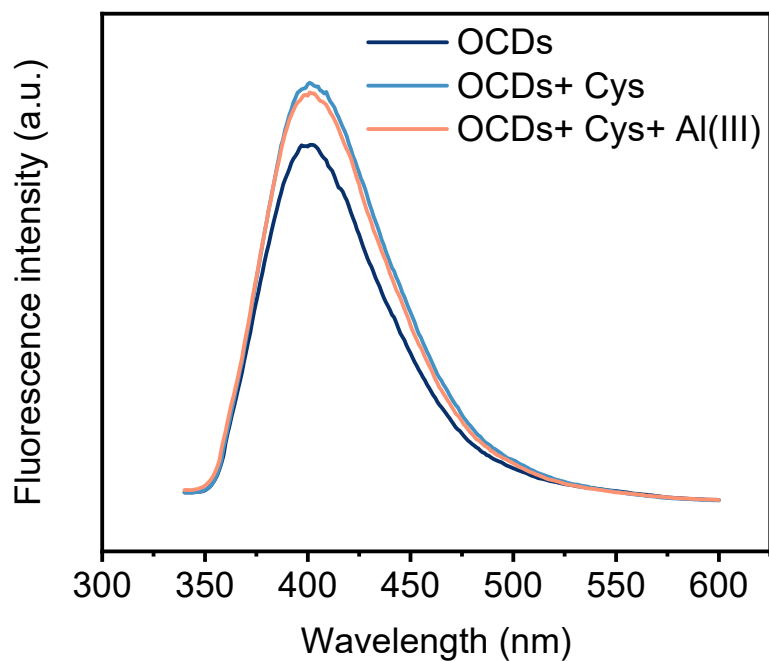


Fig. S37 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Al(III).

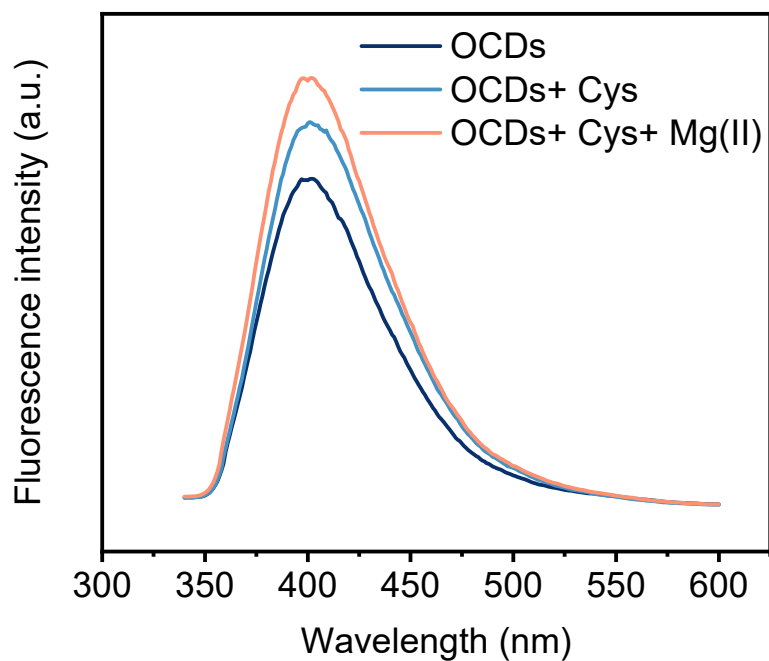


Fig. S38 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Mg(II).

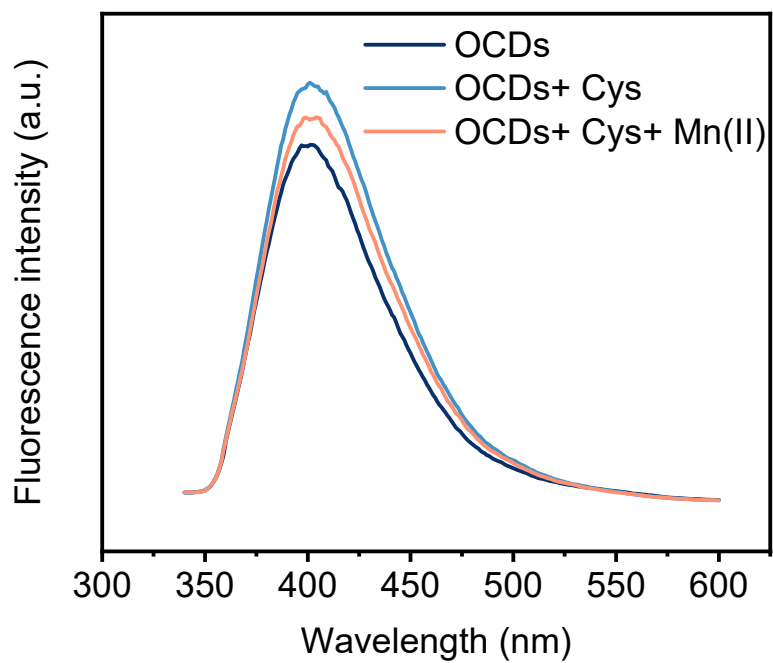


Fig. S39 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Mn(II).

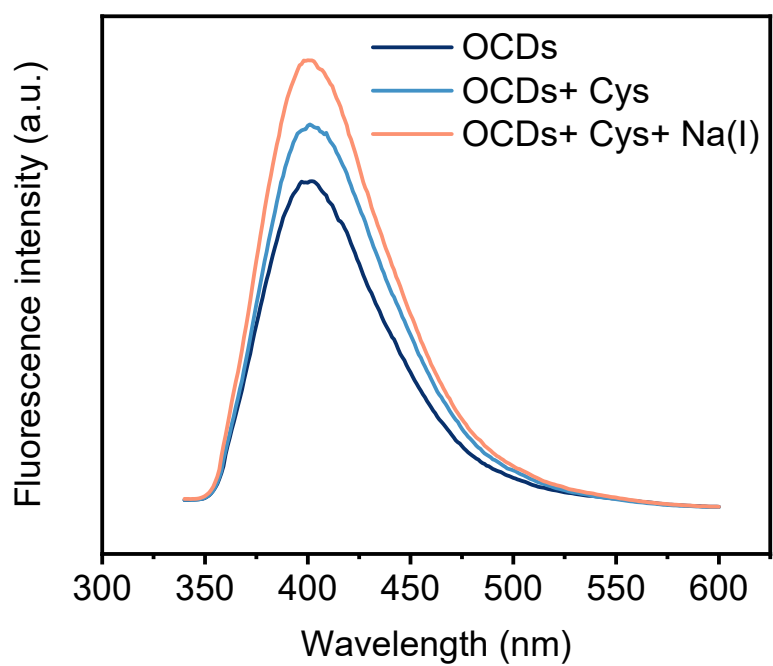


Fig. S40 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Na(II).

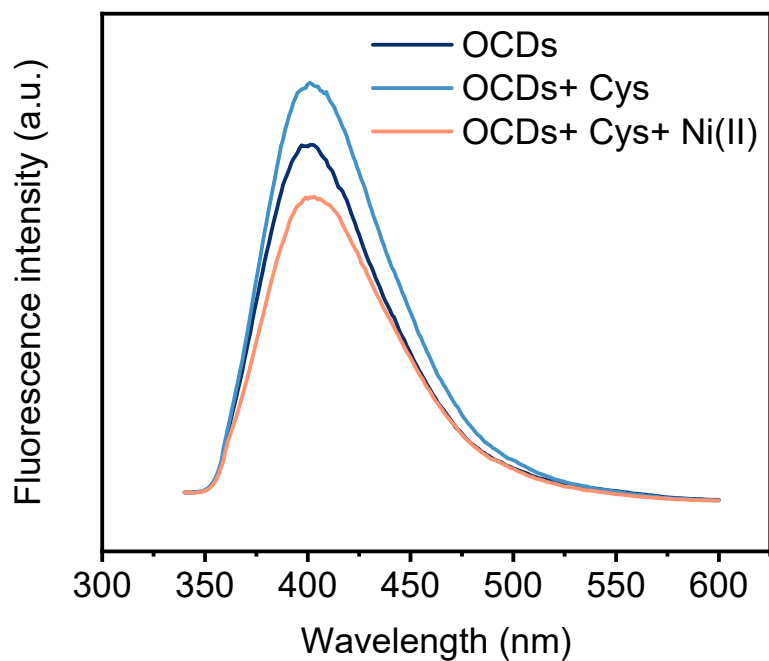


Fig. S41 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Ni(II).

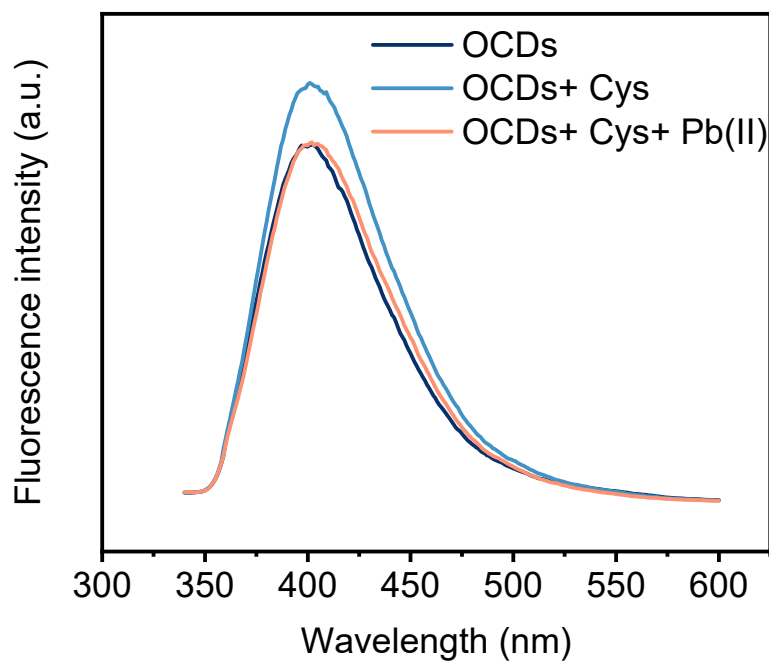


Fig. S42 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Pb(II).

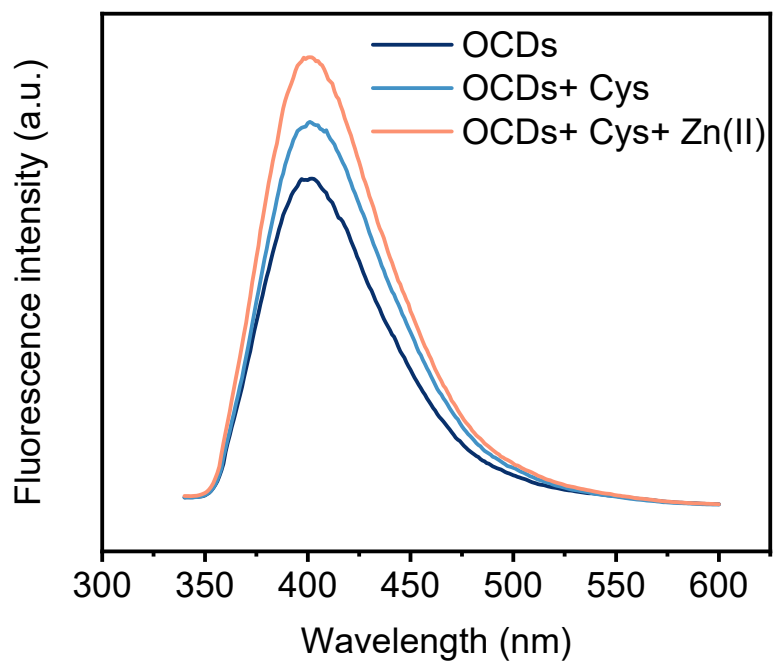


Fig. S43 Comparison of fluorescence spectra of OCDs, OCDs + cysteine and OCDs + cysteine + Zn(II).

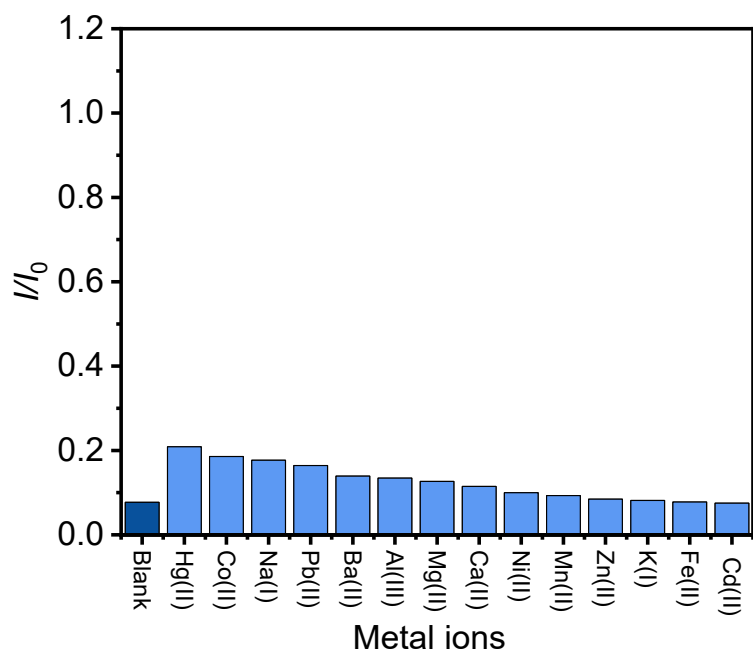


Fig. S44 The comparison of relative fluorescence intensities of the mixtures of OCD, cysteine (0.3 mM) and Fe(III) (0.1 mM) with different metal ions.

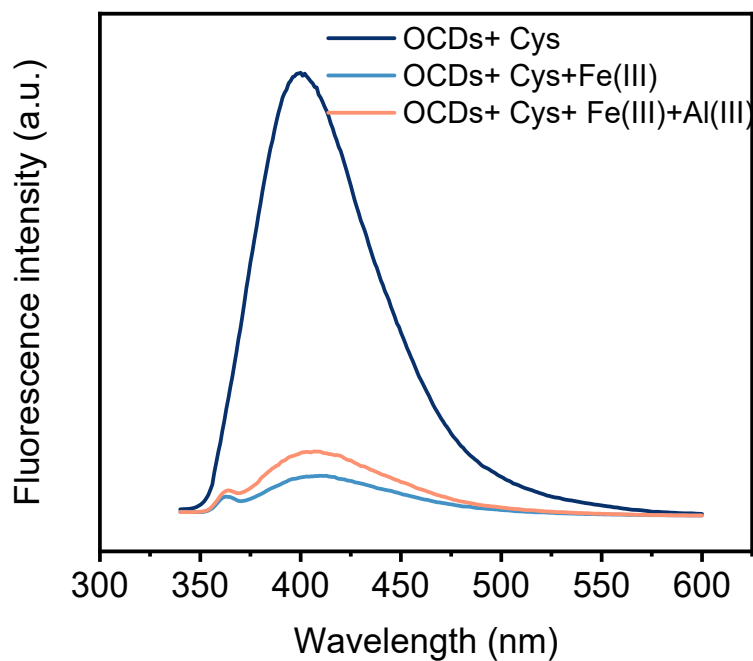


Fig. S45 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Al(III).

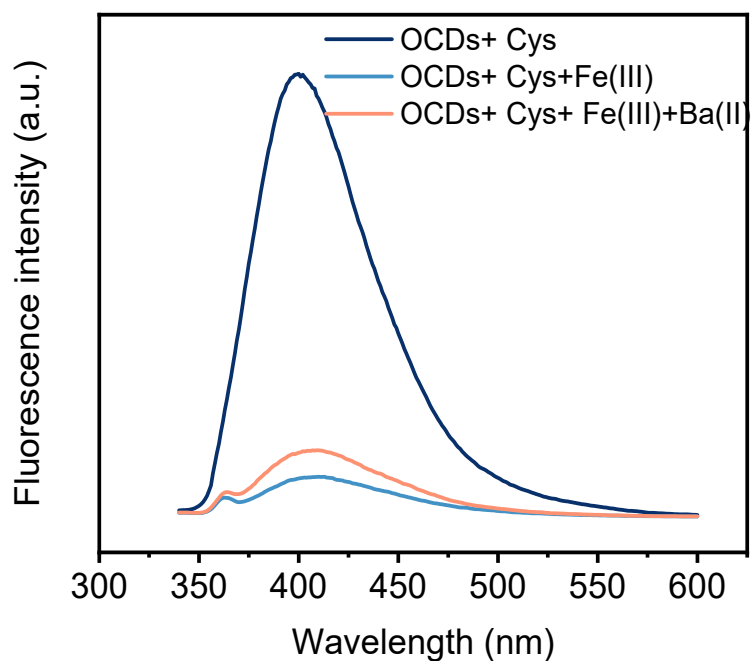


Fig. S46 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Ba(II).

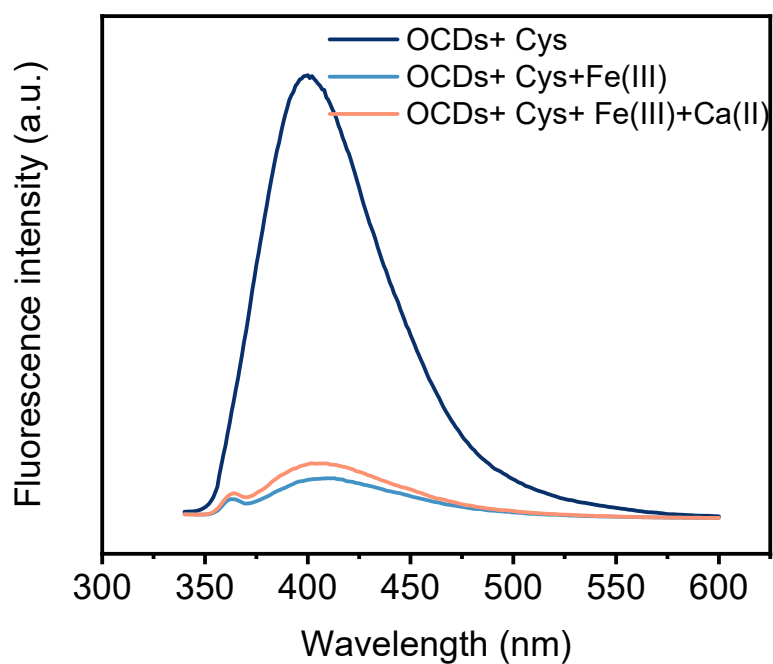


Fig. S47 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Ca(II).

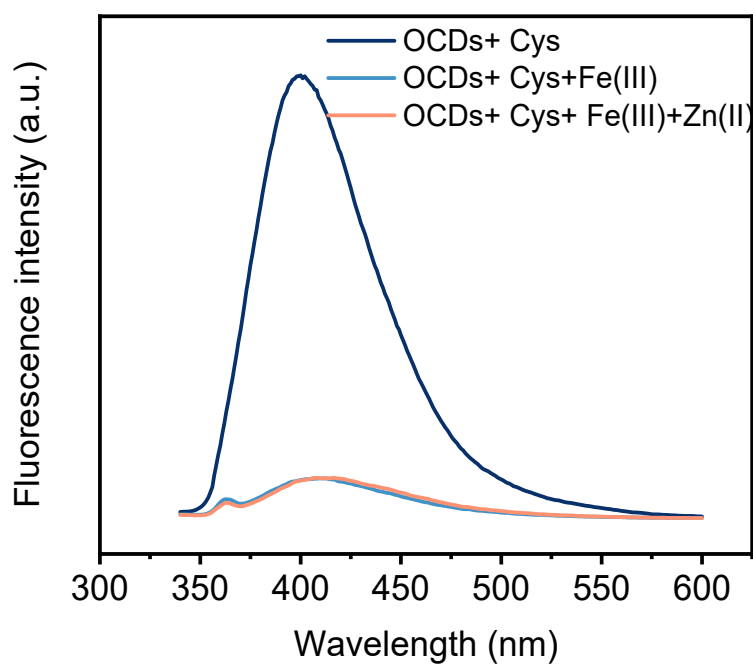


Fig. S48 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Zn(II).

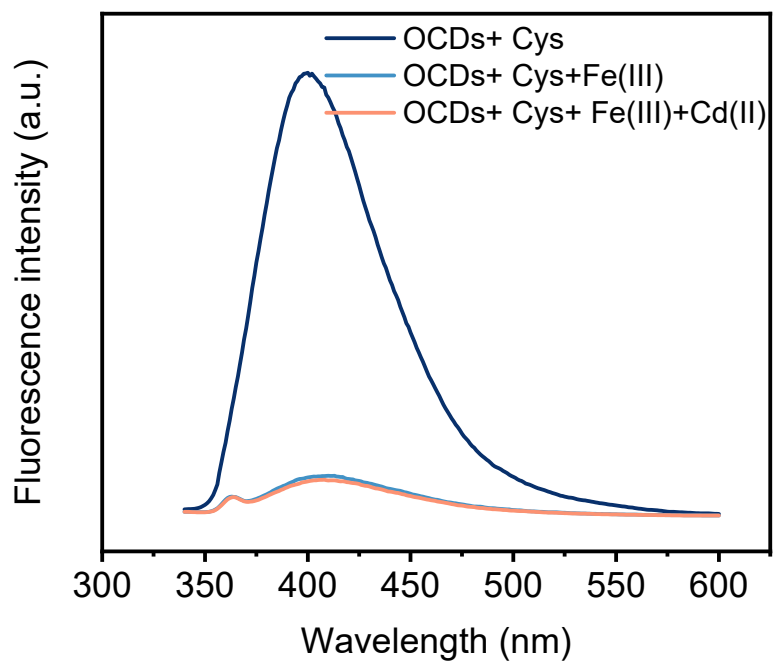


Fig. S49 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Cd(II).

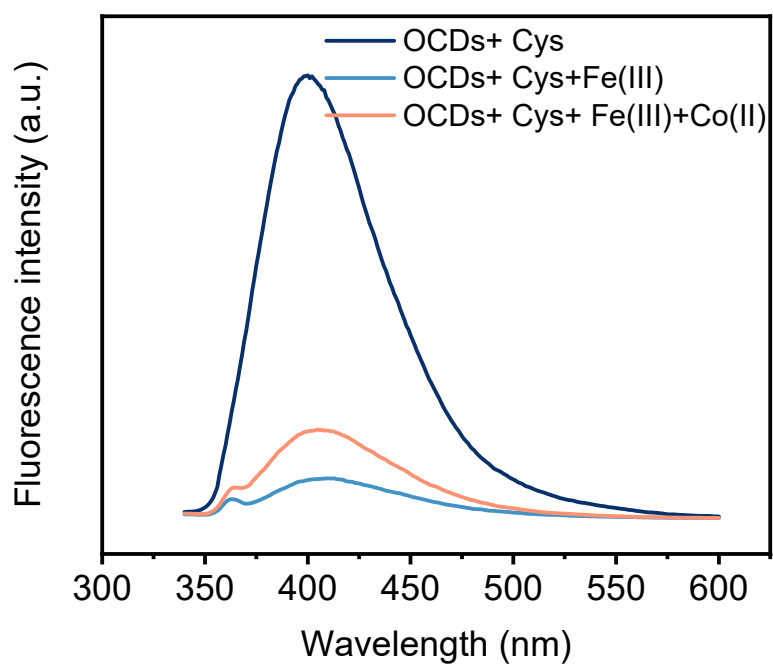


Fig. S50 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Co(II).

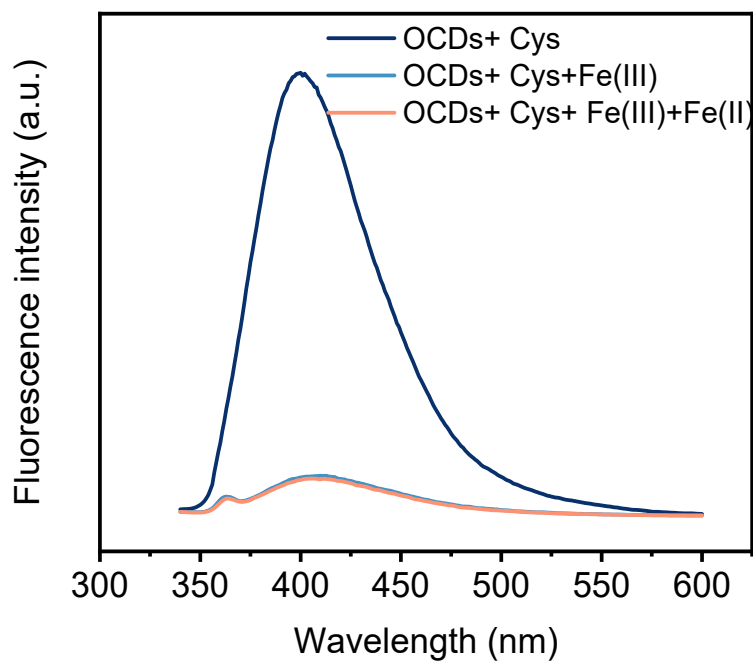


Fig. S51 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Fe(II).

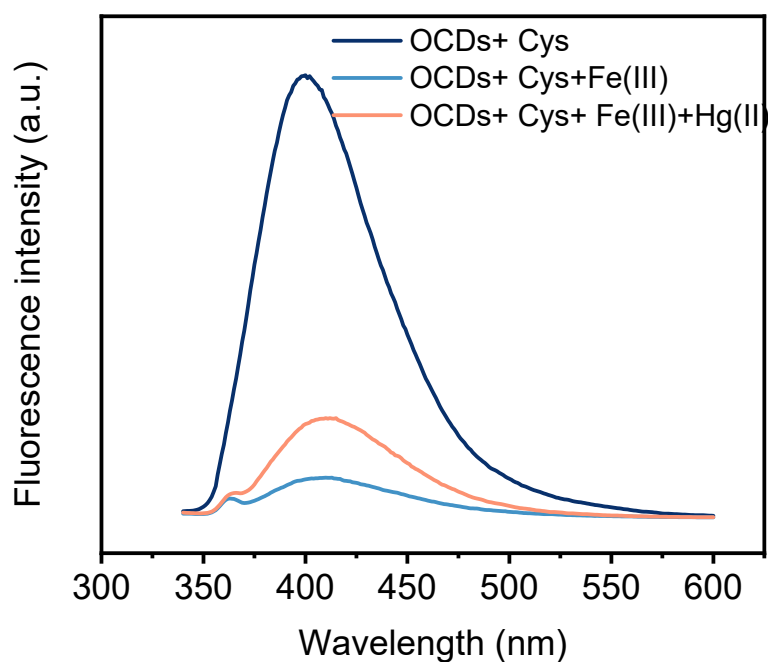


Fig. S52 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Hg(II).

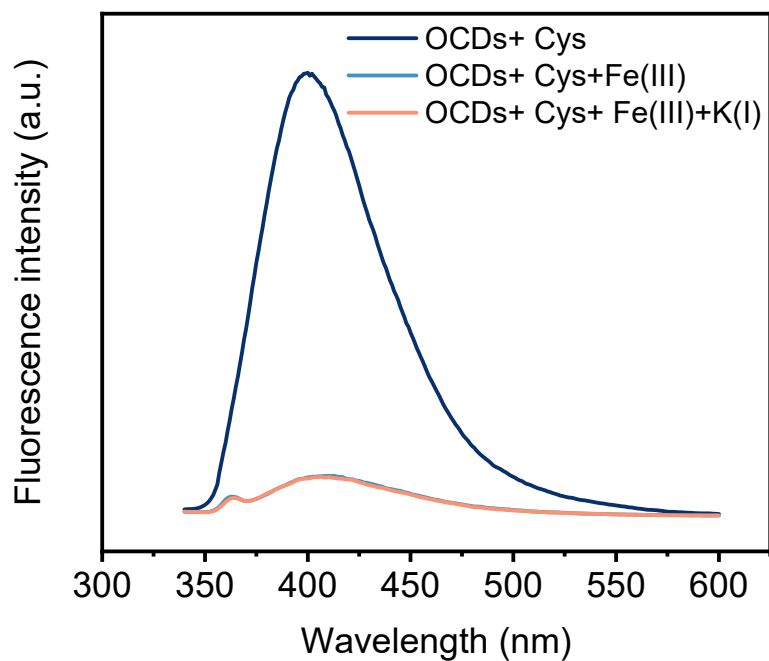


Fig. S53 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + K(I).

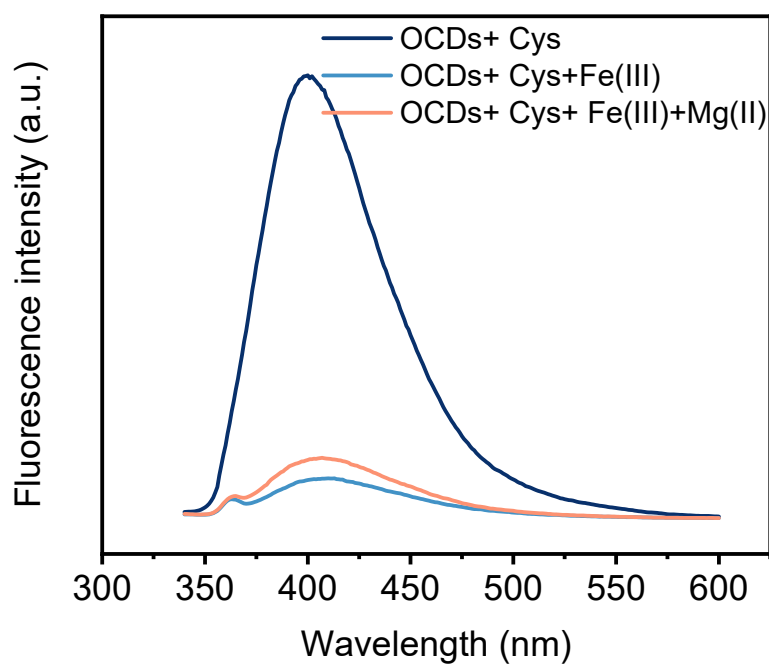


Fig. S54 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Mg(II).

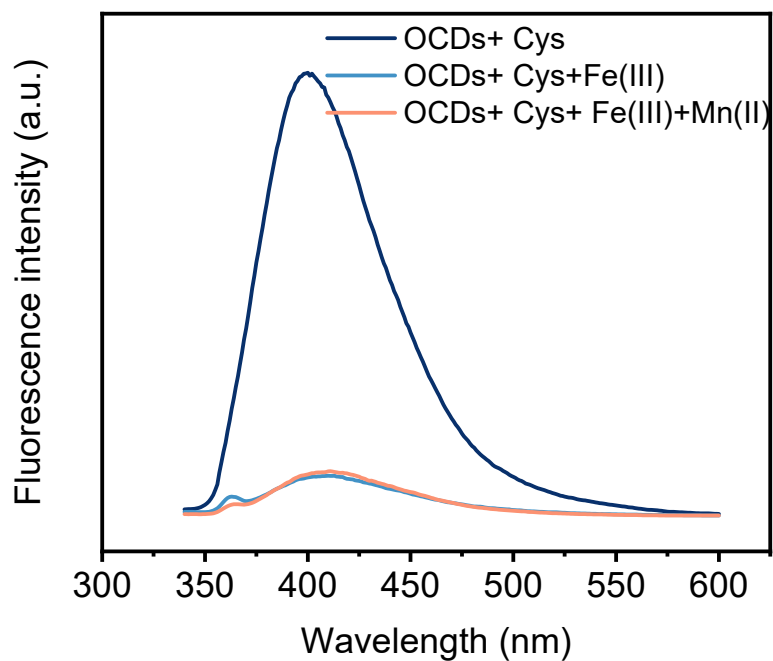


Fig. S55 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Mn(II).

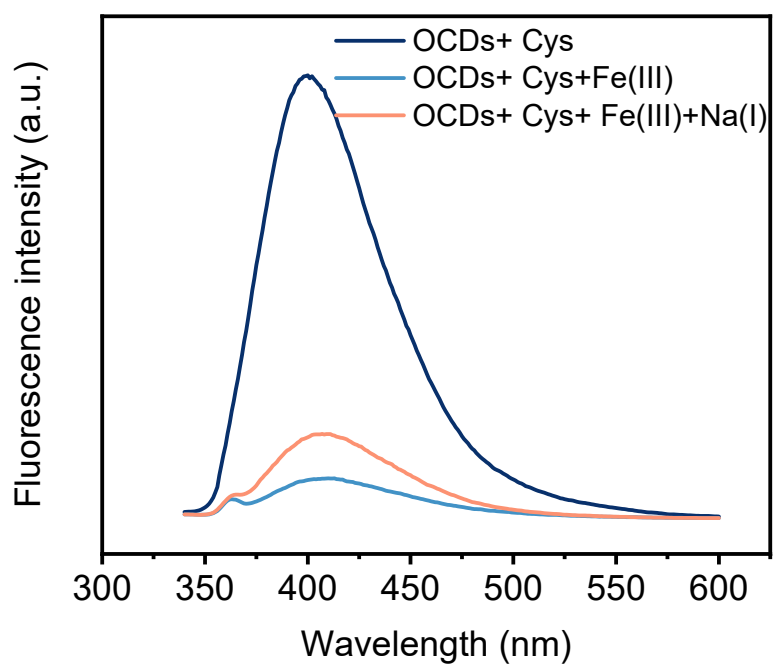


Fig. S56 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Na(I).

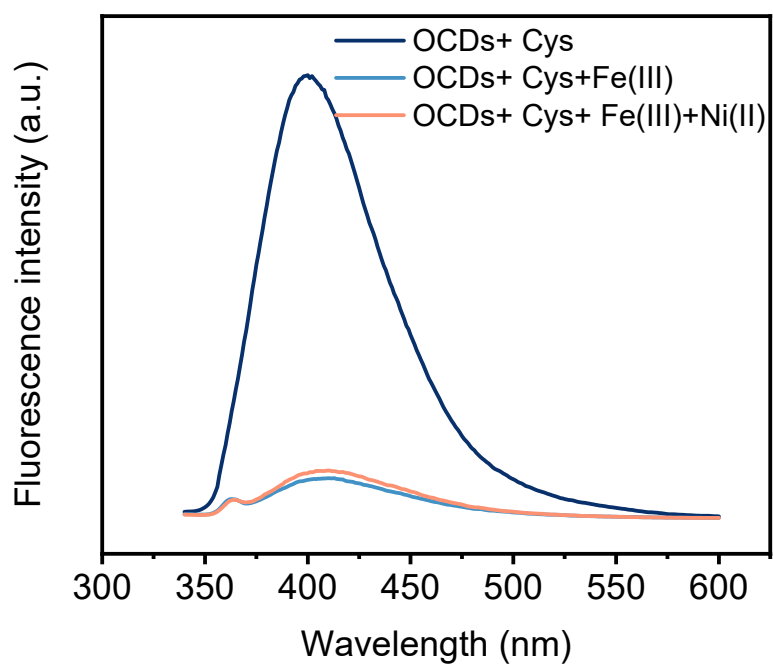


Fig. S57 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Ni(II).

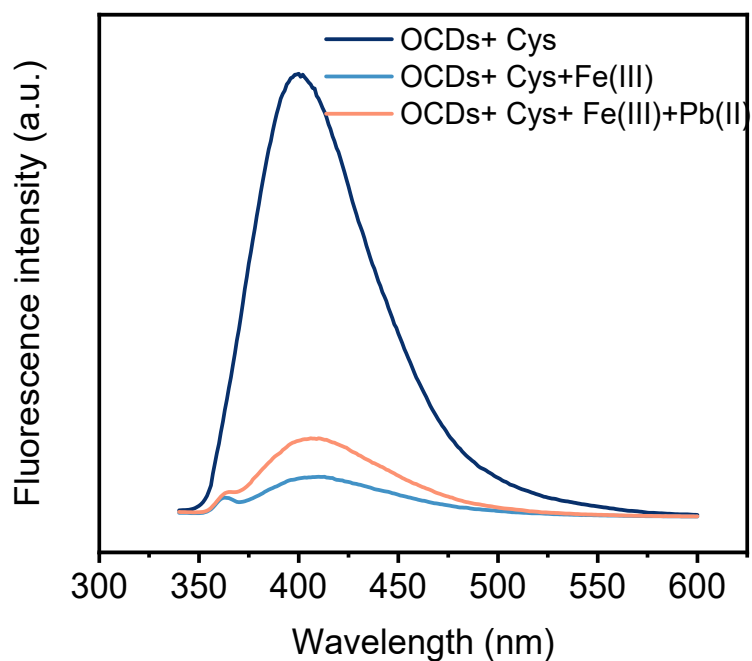


Fig. S58 Comparison of fluorescence spectra of OCDs + cysteine, OCDs + cysteine + Fe(III) and OCDs + cysteine + Fe(III) + Pb(II).

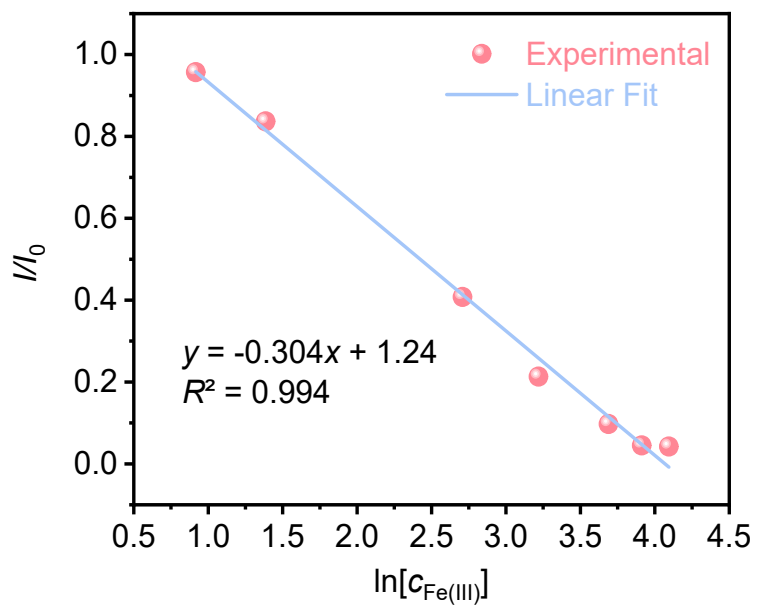


Fig. S59 The linear relationship between $\ln[C_{Fe(III)}]$ and fluorescence intensity in real samples.

Table S1. Comparison of reaction conditions for preparing carbon dots (bottom-up) that can specifically detect Fe³⁺.

Materials	Precursor	Synthesis Methods	Reaction condition	References
BNCs	1,1'-binaphthyl-2,2' - diamine, citric acid and toluene	Hydrothermal	180°C, 12 hours	Sens. Actuators B Chem. 2022, 360, 131645.
G-CDs	citric acid and melamine	Solvothermal	180°C, 8 hours	Sens. Actuators B Chem. 2021, 342, 129963.
NCQDs	Tartaric acid and L-arginine	Solvothermal	180°C, 12 hours	Microchem. J. 2020, 158, 105142.
S-doped C-dots	sodium citrate and sodium thiosulfate	Hydrothermal	200°C, 6 hours	J. Mater. Chem. A 2015, 3 (2), 542-546.
C-dots	4-Chlorophenol	Solvothermal	185°C, 10 hours	Spectrochim. Acta. A Mol. Biomol. Spectrosc. 2020, 226, 117594.
OCDs	Oxytetracycline	Hydrothermal	180°C, 1 hour	This work

Table S2. Performance comparison of the as-prepared OCDs with other reported fluorescent switching-off CDs on Fe(III) detection, which are listed according to the order of sensitivity decreasing (The numbers in the table correspond to the numbers in Fig. 4.c).

Number	Materials	Precursors	Method	Linear range (μM)	LOD (μM)	Sensitivity based on QE (μM^{-1})	References
1	OCDs	Oxytetracycline	bottom-up	0.5–20	0.440	3.52×10^{-2}	This work.
2	P,Cl-CDs	Maltose, phosphoric and hydrochloric acids	bottom-up	0.1–8.0	0.060	5.67×10^{-2}	S1
3	La-CDs	Citric acid monohydrate, methylamine hydrochloride and lanthanum chloride heptahydrate	bottom-up	0.15–10	0.091	3.361×10^{-2}	S2
4	C-dots	4-Chlorophenol	bottom-up	0.6–25	0.36	2.86×10^{-2}	S3
5	N-CDs	citric acid and allylamine hydrochloride	bottom-up	0.05–30	0.0135	1.93×10^{-2}	S4
6	BNCDs	1,1'-binaphthyl-2,2'-diamine, citric acid and toluene	bottom-up	0–80	0.219	1.43×10^{-2}	S5
7	P95-CDs	Citric acid and phenylalanine	bottom-up	0–50	3.5	1.03×10^{-2}	S6
8	N-CDs	citric acid and benzoylurea	bottom-up	30–100	1.1	9.7×10^{-3}	S7
9	G-CDs	citric acid and melamine	bottom-up	0.5–50	0.1	7.7×10^{-3}	S8

10	EfCDs	Acrylamide,EDTA and Ammonium persulfate	up bottom- up	0-50	0.0338	7×10^{-3}	S9
11	Carbon dots	L-glutamic acid and anhydrous ethylenediamine	bottom- up	8-80	3.8	3.58×10^{-3}	S10
12	HCDs	Citric acid, branched polyethyleneimine and potassium thiocyanate	bottom- up	1-150	0.052	3.3×10^{-3}	S11
13	NCQDs	Tartaric acid and L-arginine	bottom- up	0-70	0.50	3.13×10^{-3}	S12
14	CDs-1	p-Benzoquinone and EDA	bottom- up	25-200	22	2.7×10^{-3}	S13
15	Ph-gCNQDs	Citric acid, urea, dipotassium hydrogen phosphate and oleic acid.	bottom- up	0-250	25.18	2.54×10^{-3}	S14
16	B, N-CDs	Citric acid, tris base and boric acid	bottom- up	0-400	5.36	2.28×10^{-3}	S15
17	S-doped C-dots	sodium citrate and sodium thiosulfate	bottom- up	1-500	0.1	2.01×10^{-3}	S16
18	CDs	p-dihydroxybenzene and EDA	bottom- up	0-150	0.039	2×10^{-3}	S17
19	CDs	glycerol and DAMO	bottom- up	0.1-100	0.016	1.48×10^{-3}	S18
20	CQDs	Citric acid and 1-aminopropyl-3-methylimidazolium	bottom- up	0-300	13.68	1.27×10^{-3}	S19
21	E-CNDs	Ethylenediamine	bottom- up	0.5- 2000	0.018	4.76×10^{-4}	S20

22	CQDs	<i>Catharanthus roseus</i>	top-down	1–6	0.8	2.99×10^{-2}	S21
23	C-dots	<i>Curauá</i> -fiber	top-down	0–30	0.77	2.42×10^{-2}	S22
24	CDs	citric acid, polyvinylpyrrolidone and methionine	bottom-up	0-1000	0.00026	1.295×10^{-2}	S23
25	CQDs	Pear juice	top-down	0–50	2.28	1.22×10^{-2}	S24
26	HN-CDs	dwarf banana peel	top-down	5-25	0.66	9.1×10^{-3}	S25
27	N-CDs	<i>Magnolia liliiflora</i> flower	top-down	1–25	1.2	6.83×10^{-3}	S26
28	N-CDs	soluble starch and EDA	top-down	0-80	0.0849	6.2×10^{-3}	S27
29	N-CDs	natural bauhinia flflower	top-down	0-350	0.01	6.2×10^{-3}	S28
30	KCDs	kerosene fuel soot	top-down	0-150	2.25	6×10^{-3}	S29
31	CDs	Polymeric reverse osmosis (RO) membranes and H ₂ O ₂	top-down	0-100	2.97	5.3×10^{-3}	S30
32	NGQDs	chitosan and different acids	top-down	0-300	0.0667	4.5×10^{-3}	S31
33	CDs	Chloroplasts of spinach	top-down	1.0–100.0	0.3	3.78×10^{-3}	S32
34	CDs	Sweet <i>potato</i>	top-down	1–100	0.32	3.7×10^{-3}	S33
35	FNCDs	<i>Phyllanthus acidus</i> and aqueous ammonia	top-down	2-25	0.9	3.4×10^{-3}	S34
36	CDs	charcoal	top-down	50-250	72	3×10^{-3}	S35
37	S,N-doped C-dots	Distillers dried solubles	top-down	0.01–16	0.0032	2.72×10^{-3}	S36
38	N,S-CDs	chitosan and κ-carr	top-down	1-100	0.057	2.69×10^{-3}	S37
39	CNs	Pith of <i>tapioca</i>	top-down	20–200	26.5	2.59×10^{-3}	S38
40	CNDs	Olive solid wastes	top-down	0-50	1.4	2.4×10^{-3}	S39
41	CD-CP	<i>Chlorella pyrenoidosa</i>	top-down	1.6-200	0.00055	2.2×10^{-3}	S40
42	GQDs	alkali lignin	top-down	0-500	1.49	1.3×10^{-3}	S41

43	MCDs	Ganoderma spores	lucidum	top-down	0.0025- 0.1	0.0159	1.3×10^{-3}	S42
44	P-CQDs	pine wood		top-down	0-2000	0.3554	1.1×10^{-3}	S43
45	CB-CDs	<i>Cranberry</i> beans		top-down	30–600	9.55	9.65×10^{-4}	S44
46	M-GQDs	Miscanthus benzenesulfonic monosodium salt	p-amino- acid	top-down	0-1000	0.00141	2.94×10^{-4}	S45
47	CA-CFCDs	spent coffee grounds and citric acid		top-down	0-200	2.25	1.74×10^{-4}	S46

Table S3. Detection of Fe³⁺ in actual water samples.

Samples	Added (μM)	Measured (μM)	Recovery (%)	RSD (%)
	2.5	2.29	91.6	0.69
	4	3.50	87.5	0.52
Lake water	15	15.84	105.6	0.39
	40	47.22	118.1	1.63
	50	56.83	113.7	1.46
	60	57.35	95.6	1.80

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