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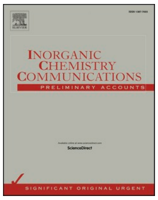

Two coordination polymers based on *p-tert*-butylcalix[4]arene as efficient luminescent sensor for Fe^{3+} and MnO_4^- ions

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ABSTRACT

We herein report two coordination polymers $[Cd_2L(H_2O)_4]\cdot CH_3OH\cdot DMF$ (**1**) and $[Mn_2L(H_2O)_3(CH_3O)]\cdot(NH_2CH_3)_2\cdot 3CH_3OH\cdot H_2O$ (**2**) assembled with a novel *p*-tertbutylcalix[4]arene ligand (H_4L = tetrakis [(2-biphenylcarboxyl)oxy]-*p*-tertbutylcalix[4]arene). Isostructural coordination polymers **1** and **2** feature layer structures, which are further linked by hydrogen bonds to generate supramolecular double layers. The luminescent character of **1** in different ions and anions were explored. The quenching to Fe^{3+} and MnO_4^- in water makes **1** into a promising luminescent sensor.

1. Introduction

Calix[4]arene have been used as excellent buildings in the assemble of coordination polymers (CPs) because their upper or lower rims can be easily modified by substituents [1,2]. Up to now, a variety of functional groups, such as pyrazolyl, 2-(1H-pyrazol-3-yl)pyridine and acetamido have been incorporated into calix[4]arenes [3–5]. *P*-tert-butylcalix[4]arenes, as an important branch of calix[4]arenes, is popular in organic synthesis because of its variable cavity and easy adjustment by functional groups [6–8]. Nevertheless, only very limited *p*-tert-butylcalix[4]arenes-based CPs have been reported because most of them have poor solubility and crystallinity [9]. Recently, the research on the CPs of modified *p*-tert-butylcalix[4]arenes of our group has led to several CPs through the introduction of hydrophilicpyridyl group [10,11]. To promote the research, we designed a new tetrakis[(2-biphenylcarboxyl)oxy]-*p*-tertbutylcalix[4]arenes ligand (H_4L, Scheme 1). The ligand features four carboxylic acids, which may be favourable for the synthesis of CPs.

Chemical sensing has attracted a great deal of attention in the fields of separation, food safety, environmental protection and medical science [12–15]. In this regard, luminescent-CPs based on quenching effects have achieved interests because of their short response time and easy operation [16–18]. Fe^{3+} ion is one of the most abundant transition metal in cellular systems, and plays an indispensable role in living organisms [19,20]. MnO_4^- ion, for its excellent oxidation ability, is widely used in laboratory, manufacturing and daily life [21,22]. It is necessary to detected them selectively and sensitively.

In this paper, we constructed two CPs based on H_4L, namely $[Cd_2L(H_2O)_4]\cdot CH_3OH\cdot DMF$ (**1**) and $[Mn_2L(H_2O)_3(CH_3O)]\cdot(NH_2CH_3)_2\cdot 3CH_3OH\cdot H_2O$ (**2**). **1** and **2** exhibit unique double layer structures. Notably, **1** shows a strong emission. **1** could also luminescent detects Fe^{3+} and MnO_4^- with high selectivity and sensitivity.

2. Synthesis of H_4L

The intermediate *p*-tert-butylcalix[4]arenes was prepared according to the literature methods [23]. *P*-tert-butylcalix[4]arenes (3.85 mmol) and NaH (129 mmol) were suspended in dried DMF and stirred for 30 min [24]. Then 2-(4-bromomethylphenyl)benzoic acid methyl ester (15.8 mmol) was added, and the mixture was placed in a water bath (50 °C) for 48 h. After the reaction was completed, a little methanol was added to the flask until no bubbles were produced. The solvent was removed by rotary evaporation, then put sufficient water into flask. Next, removed insoluble materials by vacuum filtration. After the filtrate was transferred to the beaker, hydrochloric acid (12 mol) was added into beaker to adjust pH = 1. The solid was filtered and dried at 80 °C. The product was recrystallized by methanol. Finally, white solid H_4L was obtained in a yield of 55.5% (4.24 g). IR (cm^{-1}): 3743(s), 3426(m), 3064(m), 3032(m), 2955(w), 2905(w), 2865(w), 2656(s), 2547(s), 1912 (s), 1695(w), 1600(m), 1482(w), 1401(m), 1366(m), 1294(w), 1253 (m), 1193(m), 993(m), 937(s), 877(m), 821(m), 762(w), 646(s), 571(s) (Fig. S1).

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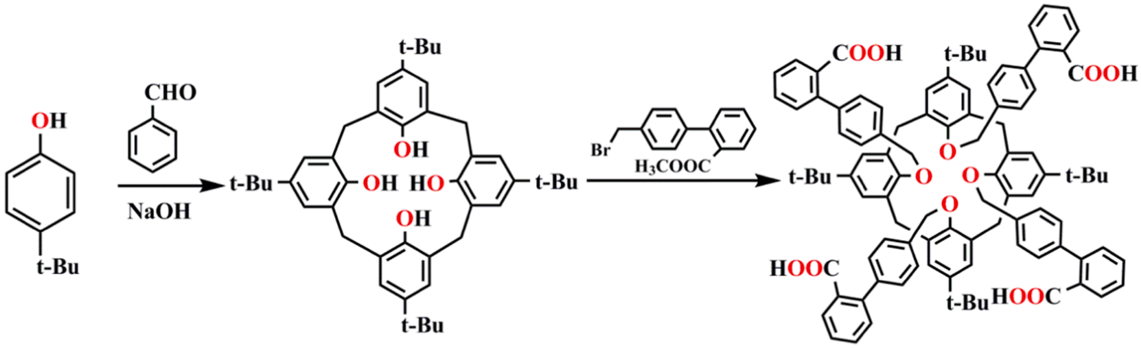

Scheme 1. Synthetic procedure of H_4L.

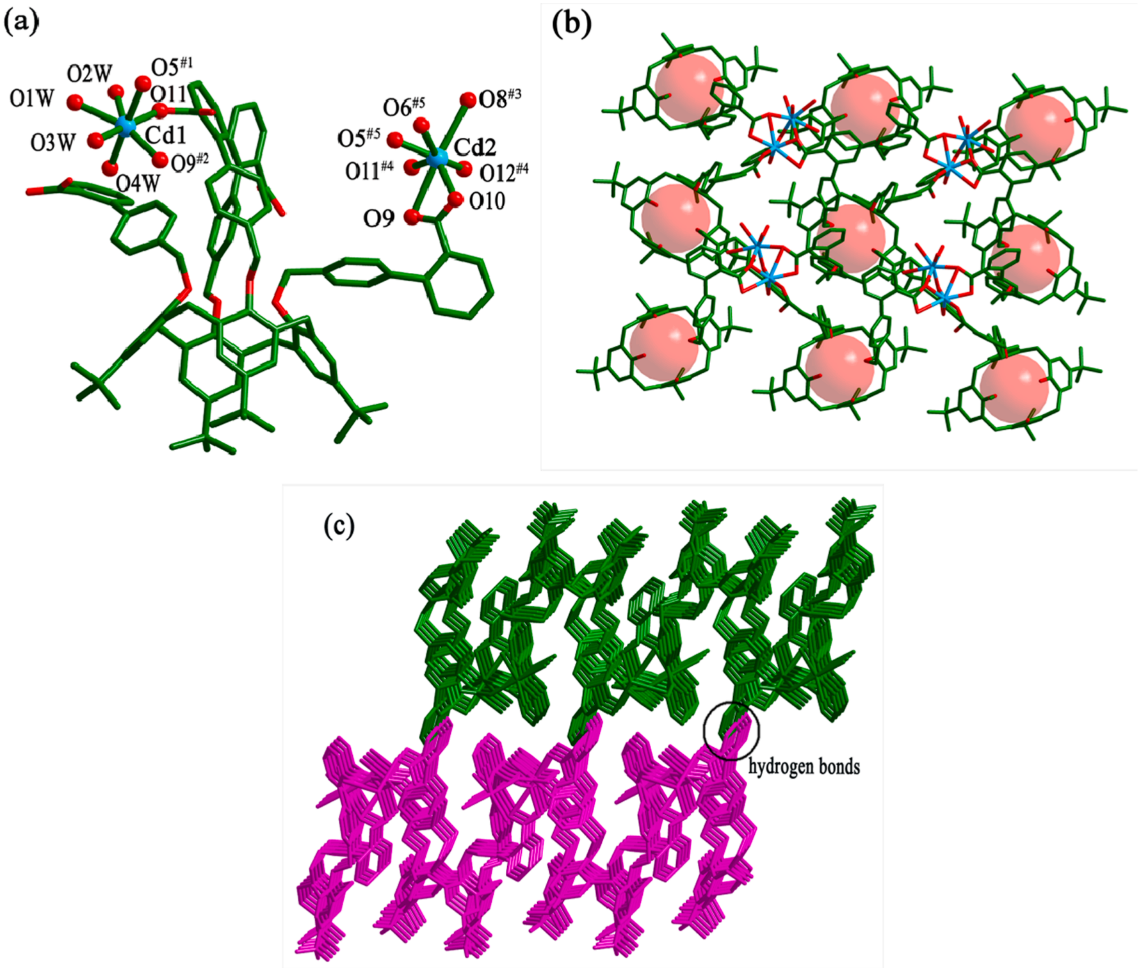

Fig. 1. (a) Coordination spheres of Cd^{2+} ions. (b) View of the layer (red balls represent the cavities of L^{4-} anions). (c) The supramolecular double layer. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3. Results and discussion

SQUEEZE instruction in PLATON was utilized and the disordered solvents were obtained through electron cloud density, TGA and elemental analysis (Fig. S2) [25]. The individual unit of **1** contains one L^{4-} anion, two Cd^{2+} ions, four coordinated water molecules, one free methanol and one free DMF molecule. Cd1 and Cd2 both show seven coordination modes, whereas their coordination environments are completely different (Fig. 1a). Cd1 ion is surrounded by three O atoms from three individual L^{4-} anions and four water molecules. Cd2 ion is coordinated by seven O atoms from four individual L^{4-} anions. Cd1 and Cd2 ions are held together via three carboxylate O atoms from three individual L^{4-} anions to afford a dimer. The conformation of L^{4-} anion is “cone”. The neighboring dimers are linked by L^{4-} anions to generate a layer (Fig. 1b). According to the extension direction of *t*-butyl groups, the layers could be described as plane-α and plane-β, respectively. As there exists hydrogen bonds between L^{4-}anions ($C69\cdots C69^{\#6} = 3.461$ Å, $\angle C69\text{-}H69\cdots C69^{\#6} = 125.12°$), a fascinating supramolecular double layer is generated (Fig. 1c).

The structure of **2** is isomorphous with **1**. Nevertheless, two major

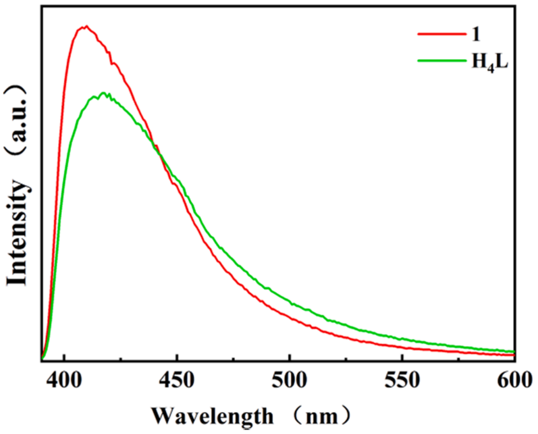

Fig. 2. The emission spectra of H_4L and **1**.

differences exist for the CPs (Fig. S3). Firstly, the coordination modes of metal ions have differed. Two Mn^{2+}cations in **2** show octahedral geometries, instead of the seven coordination modes. Mn1 is surrounded by three O atoms of three L^{4-} anions, one methanol, and one water molecule. Mn2 is coordinated by four O atoms of three individual L^{4-} anions and two water molecules. Secondly, the free molecules in **2** are different. There are one free $NH_2(CH_3)_2^+$ cation, one free water and three free methanol. The balance positive charge $NH_2(CH_3)_2^+$ came from the decomposed DMF [26].

Solid state luminescent properties of **1** and H_4L at RT were tested (Figs. 2 and S4). H_4L shows an emission at 432 nm (λ_{ex} = 350 nm) [27,28]. For **1**, the maximum emission spectrum appeared at 409 nm (λ_{ex} = 350 nm), which may be attributed to the emission of the L^{4-} anions.

As **1** exhibits a strong luminescent emission, the investigation of metal ions detection was carried out. We spread the crystals evenly on a glass plate, and put the glass plate into the aqueous solution of 0.01 mol·L^{-1} MCl_x (M = Na^+, Cd^{2+}, Co^{2+}, Cu^{2+}, Ni^{2+}, Mn^{2+}, Zn^{2+}, Mg^{2+} or Fe^{3+}) to make fully combined M@**1** [29], respectively. As shown in Fig. 3, compared with blank water, the intensities of Na^+, Cu^{2+} and Cd^{2+}

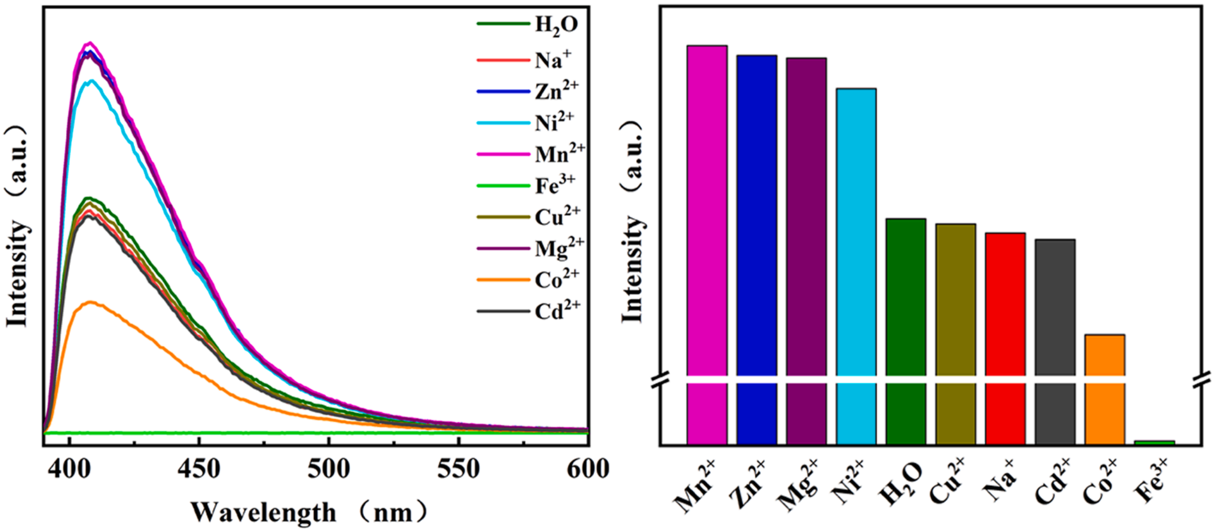

Fig. 3. Emission intensities in different metal ion solutions.

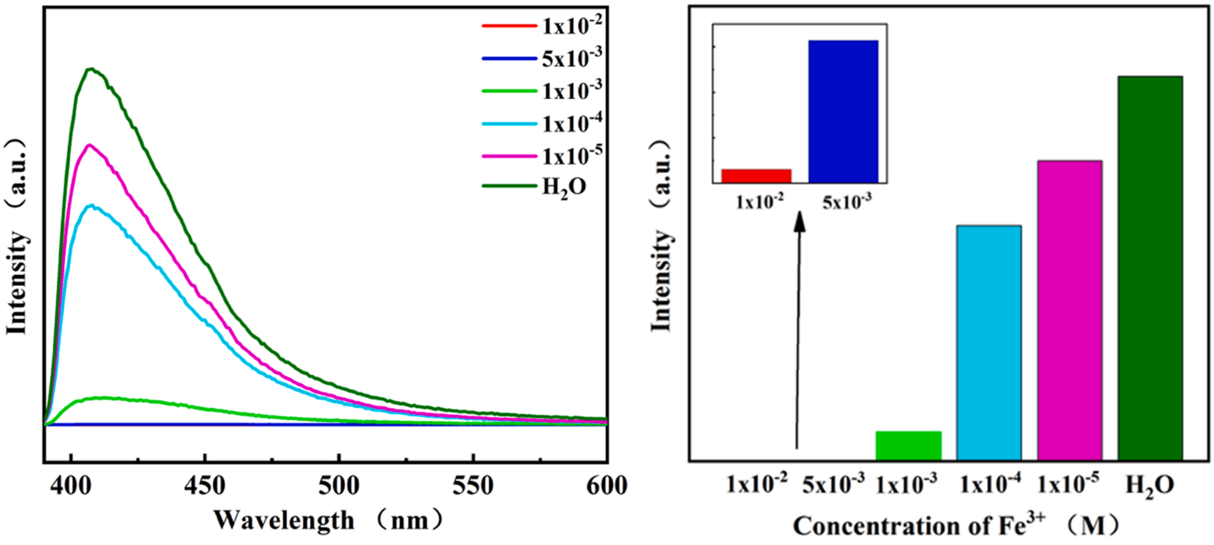

Fig. 4. Fe^{3+} concentration-dependent emission intensities of **1**.

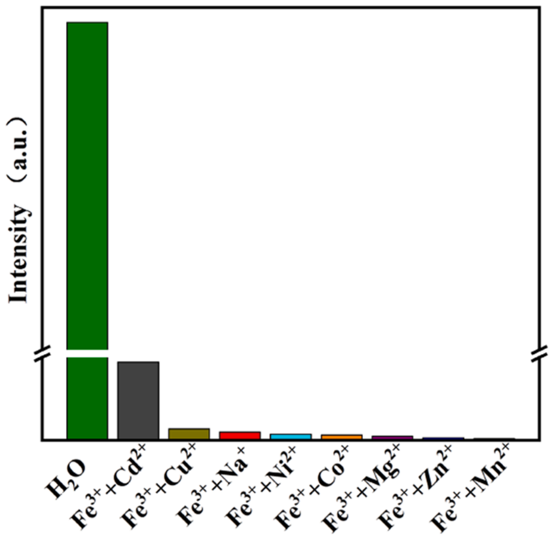

Fig. 5. The luminescent intensities of **1** in interfering cation solutions.

solutions were broadly unchanged after interaction with **1**. The intensities of Ni^{2+}, Mn^{2+}, Mg^{2+}, Zn^{2+} solutions were significantly increased. And the intensity of Co^{2+} solution has a slightly reduction. Notably, an obviously quenching was visible for the solution containing Fe^{3+} cations.

The quenching effect of Fe^{3+} ion for **1** was further investigated by studying the emissions of different Fe^{3+}@**1** concentrations [30]. As depicted in Fig. 4, with the increasing concentration of Fe^{3+}, the emission intensity of **1** gradually decreases. Obviously, the intensity almost disappeared when the concentration reaches 5×10^{-3} $mol \cdot L^{-1}$. The experimental results show that **1** is an efficient Fe^{3+} ion detection sensor [31]. The quenching value K_{sv} calculated by the Stern-Volmer equation is 1.22×10^4 M^{-1} (Fig. S5). The detection ability is comparable to those reported high efficiency CPs (Table S1) [32–34].

To study whether **1** has a selective recognition on Fe^{3+} ion, further experiments by mixing Fe^{3+} ions (0.01 M) with other metal ions (0.01 M) were performed [35]. As illustrated in Fig. 5, the reduction of emission was pronounced for all of the mixed solutions. That means the selectivity of **1** to Fe^{3+} ions is hardly affected by other metal ions. To probe the reusability of **1**, recycled experiments were carried out. After four cycles of experiments, **1** still has an obvious recognition effect to Fe^{3+} ions (Fig. S6).

The efficient quenching effect of **1** to Fe^{3+} impelled us to continuously explore the detection on trace amounts of anions. Using the same method, **1** was thoroughly contacted with 0.01 $mol \cdot L^{-1}K_nX$ ($X = I^-$, Br^-, $C_2O_4^{2-}$, Cl^-, $H_2PO_4^-$, HPO_4^{2-}, PO_4^{3-}, CNS^-, $S_2O_8^{2-}$, SO_4^{2-} and MnO_4^-) solution to form X@**1**. As shown in Fig. 6, different anions lead the luminescent intensity fluctuates to a certain extent. Of which an obvious recognition effect on MnO_4^- ion was observed [36].

Further research can discover a negative correlation between the fluorescence intensity of **1** and the concentration of MnO_4^- ions. Obviously, when the concentration of MnO_4^- reaches 10^{-2} $mol \cdot L^{-1}$, the fluorescence intensity of **1** tends to 0 (Fig. 7). Therefore, it is concluded that **1** is also an effective substance for the detection of MnO_4^- ion. As illustrated in Fig. S7, the quenching value K_{sv} is 1.06×10^4 M^{-1}. Comparing to the limited CPs reported for detecting MnO_4^- ion, **1** exhibits a more sensitive luminescent quenching effect (Table S2) [37,38].

In order to explore whether the quenching of MnO_4^- was disturbed by other anions, a series of mixing experiments were conducted [39]. We combined **1** with MnO_4^- and 0.01 M other anions (K_nX, $X = Br^-$, Cl^-, $H_2PO_4^-$, SO_4^{2-}, PO_4^{3-}, HPO_4^{2-} and $S_2O_8^{2-}$), respectively. As shown in Fig. 8, **1** still possesses obvious recognition effect on MnO_4^-. This phenomenon indicates that **1** has good anti-interference ability for the quenching of MnO_4^-. The recyclable tests verified that **1** could still shows a good recognition after four runs of sensing (Fig. S8) [40].

4. Conclusion

In short, we synthesized two CPs based on a novel *p*-tert-butylcalix[4]arenes ligand through the solvothermal method. Fascinating double layers are formed for the structures of **1** and **2**. Notably, **1** exhibits strong luminescent emission at RT. Systemic luminescent sensing study reveals that **1** displays high sensitively recognition effects and excellent cycling stabilities for Fe^{3+} ion and MnO_4^- anion. This work illustrates that *p*-tert-butylcalix[4]arenes-based CP has potential application in the field of multiple luminescent sensor.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

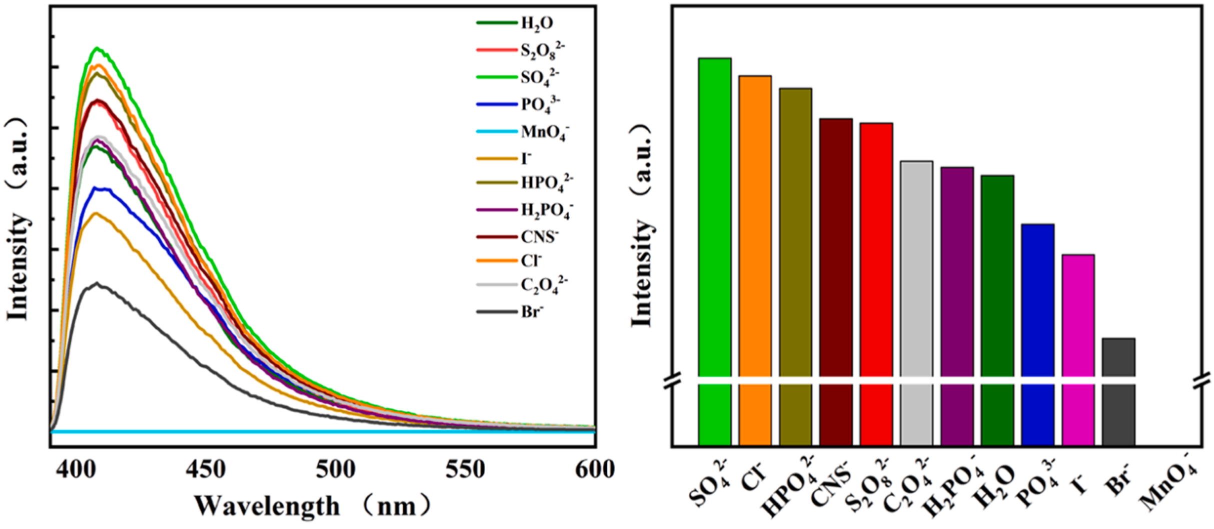

Fig. 6. Emission intensities of **1** in different anions.

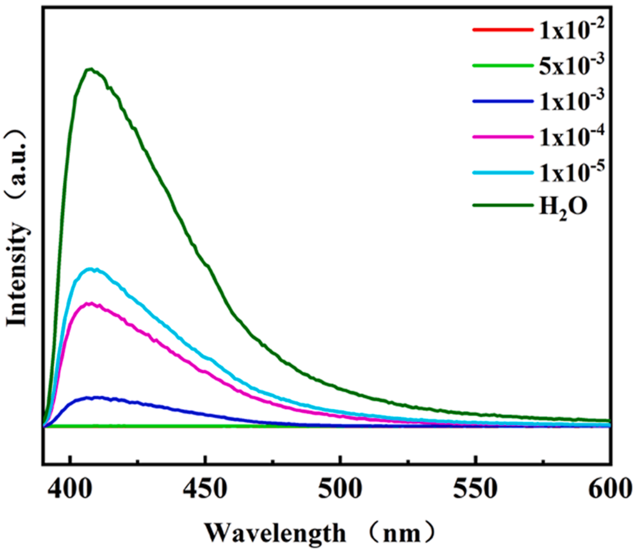

Fig. 7. MnO_4^- concentration-dependent emission intensities of **1**.

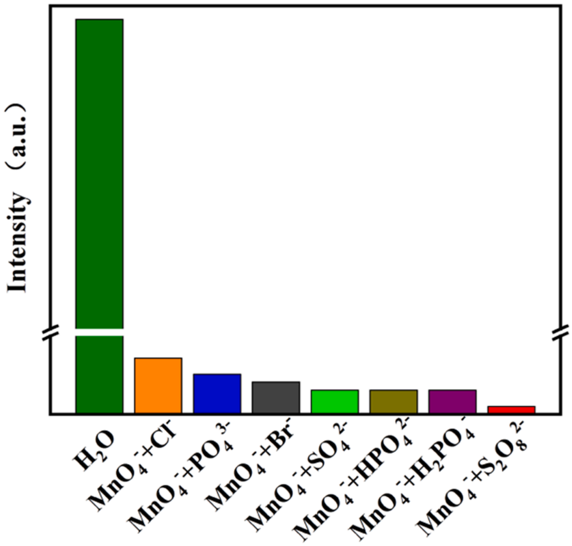

Fig. 8. The luminescent intensities of **1** in interfering anion solutions.

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Appendix A. Supplementary material

Crystallographic data for structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC reference number 2023849 and 2023850. Details on the materials, instruments, PXRD, IR spectra, TGA curves, UV/Vis spectra, Selected bond lengths and angles are list in supplementary content Supplementary data to this article can be found online at https://doi.org/10.1016/j.inoche.2020.108290.

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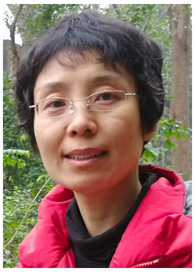

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