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# A pillar-layered Cd(II) metal-organic framework for selective detection of organic explosives

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#### ABSTRACT

The detection of explosives is crucial for homeland security, environmental cleaning, and military issues. As a new class of porous materials, metal-organic frameworks (MOFs) are promising platforms for the detection of organic explosives. In this work, a new pillar-layered Cd(II) MOF,  $[CdL_{0.5}dpe_{0.5}]\cdot 2H_2O$  (**BUT-202**,  $H_4L = 4,8$ -disulfonaphthalene-2,6-dicarboxylic acid, dpe = 1,2-bis(4-pyridyl)ethylene), was synthesized and characterized by single-crystal X-ray diffraction, powder X-ray diffraction, thermogravimetric analysis, infrared spectroscopy, and elemental analysis. **BUT-202** has good fluorescent properties, which can be selectively quenched by trace amounts of 2,4,6-trinitrophenol (TNP) in DMF with low detection limit of 0.2  $\mu$ M.



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#### **KEYWORDS**

Metal-organic framework; selective detection; organic explosive; fluorescent quenching

#### 1. Introduction

Selective and sensitive detection of highly explosive and explosive-like substances has become a serious issue concerning national security and environmental protection [1–5]. Among explosive substances, 2,4,6-trinitrophenol (TNP) is commonly used in dyes, fireworks, matches, glass, and leather industries because of its explosive power [6, 7]. In addition, TNP has also been recognized as a toxic pollutant. Thus, selective detection of TNP is very important for tracing buried explosives and environmental monitoring [8–11].

Detection of highly explosive substances including TNP is mainly based on trained canines [12] and modern analytical techniques such as gas chromatography coupled with mass spectrometry (GC–MC) [13], surface-enhanced Raman spectroscopy [14], mass spectrometry

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(MS) [15], thermal neutron analysis [16], electrochemical procedures [17], and ion mobility spectroscopy (IMS) [18, 19]. However, all these methods are time consuming, expensive, and require complex equipment and trained personnel [20]. Therefore, there is an urgent need for researchers to develop portable, reliable, and inexpensive methods/technologies for the detection of highly explosive substances.

Compared with traditional analytical techniques, fluorescence sensing is a promising technology in the detection of organic explosives owing to its advantages of low cost, electronic tunability, easy portability, and operability, etc. [21, 22]. The challenge of developing this technology rests with selection of fluorescent materials, which should clearly and solely respond to the checked molecules. As a new class of porous materials, metal-organic frameworks (MOFs), constructed from organic ligands and metal ions or clusters through coordination bonds, have been considered as good platforms for detection/sensing applications because of their advantages of high surface area, design ability of the framework, strong interactions between the framework and guest molecules as well as excellent optical properties [23–26]. As efficient fluorescent sensors, MOFs have been utilized for detecting metal ions [27–29], DNA [30, 31], volatile organic compounds (VOCs) [32–35], energetic compounds [36], etc. Till now, several MOFs based on Zn/Cd have been reported for detection of nitrobased explosives and show good performance [37–41], however, the selective detection of single explosive nitroaromatic compound in the mixture of nitroaromatic compounds is still challenging.

In this work, a pillar-layered Cd(II) MOF,  $[CdL_{0.5}dpe_{0.5}]\cdot 2H_2O$  (**BUT-202**, BUT = Beijing University of Technology), was synthesized through the reaction of light emitting ligand acid, 4,8-disulfonaphthalene-2,6-dicarboxylic acid (H<sub>4</sub>L), and Cd(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O in the presence of 1,2-bis (4-pyridyl) ethylene as additional ligand. The fluorescence of **BUT-202** can be selectively quenched by trace amounts of TNP in DMF with low detection limit of 0.2  $\mu$ M.

#### 2. Experimental

#### 2.1. Materials and general methods

All reagents and solvents (AR grade) were purchased from commercial sources and used without purification. FT–IR data were acquired on a SHIMADZU IR Affinity-1 instrument. TGA data were obtained on a TGA-50 (SHIMADZU) thermogravimetric analyzer with a heating rate of 10 °C min<sup>-1</sup> under air. Powder X-ray diffraction (PXRD) patterns were recorded on a Ragiku Smartlab3 Diffractometer using Cu-K $\alpha$  radiation ( $\lambda = 1.541874$  Å) at room temperature. Simulation of the PXRD pattern was performed by the single-crystal data and diffraction-crystal module of the Mercury program. The fluorescence data were collected on a F-4600 fluorescence spectrometer. Elemental microanalyses (EA) were performed by a Vario Macro cube Elementar. UV–Vis spectra were obtained with a UV-2600 spectrophotometer from 250 to 800 nm at room temperature.

# **2.2.** Synthesis of $H_4L$

The ligand acid was synthesized according to the literature procedure with some modification [42, 43]. Fuming sulfuric acid (SO<sub>3</sub>, 20 wt%; 20 mL) was slowly added to a 100 mL threeneck flask containing 4 g (18.5 mmol) naphthalene-2,6-dicarboxylic acid under stirring. The reaction mixture was stirred at 120 °C for 12 h. After cooling to room temperature, the mixture was placed into 50 mL concentrated HCl solution and white crude product precipitated. The powder was filtered and washed with concentrated HCl solution three times (30 mL each time). Then pure product was dried at 80 °C in an oven. Yield 6.0 g (~86%) based on naph-thalene-2,6-dicarboxylic acid. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz)  $\delta$  = 9.61 (s, 2H), 8.50 (s, 2H). FT–IR (KBr pellet, cm<sup>-1</sup>): 1686(s), 1592(w), 1421(s), 1295(s), 1251(s), 1188(s), 1043(s), 916(w), 803(w), 765(w), 734(w), and 607(m).

# **2.3.** Preparation of $[CdL_{0.5}dpe_{0.5}] \cdot 2H_2O$ (BUT-202)

A mixture of  $Cd(NO_3)_2$ ·4H<sub>2</sub>O (10 mg, 0.03 mmol), H<sub>4</sub>L (13 mg, 0.035 mmol), dpe (3 mg, 0.019 mmol), *N*,*N*-dimethylacetamide (DMA) (1.6 mL), and 0.8 mL H<sub>2</sub>O were added to a 5 mL vial and sealed. The vial was then heated in an oven at 80 °C for 48 h. After cooling to room temperature, the resulting light yellow crystals were harvested by filtration and washed with DMA and acetone, and then dried in air. FT–IR (KBr, cm<sup>-1</sup>): 1695(w), 1610(s), 1560(m), 1400(m), 1359(m), 1249(m), 1174(s), 1023(m), 980(w), 838(m), 787(w), 594(s), and 552(m). Elemental analysis calculated (%): C, 33.82; H, 2.58; S, 7.517; N, 3.29. Found: C, 34.43; H, 2.66; S, 7.427; N, 3.44.

# 2.4. Single-crystal X-ray diffraction

The crystal data of **BUT-202** were collected on a Rigaku Supernova CCD diffractometer equipped with a graphite-monochromatic enhanced Cu K $\alpha$  radiation ( $\lambda = 1.54184$  Å) at 100 K. The data-sets were corrected by empirical absorption correction using spherical harmonics, implemented in the *SCALE3 ABSPACK* scaling algorithm [44]. The structure of **BUT-202** was solved by direct methods and refined by full-matrix least-squares on  $F^2$  with anisotropic displacement using the *SHELXTL* software package [45]. Non-hydrogen atoms were refined with anisotropic displacement parameters during the final cycles. Hydrogens of the ligands were calculated in ideal positions with isotropic displacement parameters. Details of the structural refinements are listed in Table S1 of the Supporting Information and CIF file, and some of the bond lengths and bond angle data are summarized in Table S2 of the Supporting Information.

#### 2.5. Fluorescence measurement

#### 2.5.1. Fluorescence titration experiment

In a typical experiment, 2 mg of finely ground **BUT-202** was weighed and added to a cuvette containing 2.5 mL of DMF under stirring. The fluorescence upon excitation at 307 nm of **BUT-202** suspension was measured *in situ* after incremental addition of freshly prepared analytic solutions (1 mM, 20 µL addition each time). The mixed solution was stirred at a constant rate to maintain its homogeneity. All the experiments were performed in triplicate, and consistent results were reported (Figure 2 and Figure S6 of the Supporting Information).

#### 2.5.2. Selective detection experiment

Similar to that in fluorescence titration experiment, 2 mg of finely ground **BUT-202** was added to a cuvette containing 2.5 mL of DMF under stirring. Fluorescence of the obtained

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suspension was recorded. Then, 1 mM DMF solutions of 3-NP (or 2,4-DNT, 2,6-DNT, 3-NT, 2-NT, NB, DMNB) and 1 mM DMF solutions of TNP were alternatively introduced (twice for each) into the suspension in such a sequence: 3-NP (20  $\mu$ L), 3-NP (20  $\mu$ L), TNP (20  $\mu$ L), and 3-NP (20  $\mu$ L). The process was repeated until the total volume of added analytic solutions reached 320  $\mu$ L. After each addition, the fluorescence of the suspension was monitored (Figure S9 of the Supporting Information).

# 3. Results and discussion

# 3.1. Synthesis and general characterizations of BUT-202

Solvothermal reaction of H<sub>4</sub>L with Cd(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O in the presence of dpe in DMA-water system yielded block-shaped single crystals of [CdL<sub>0.5</sub>dpe<sub>0.5</sub>]·2H<sub>2</sub>O (**BUT-202**). The phase purity of bulk samples of **BUT-202** was determined by PXRD. As shown in Figure S1 of the Supporting Information, the experimental PXRD pattern matches well with the diffractogram simulated from single-crystal data. The thermogravimetric analysis (TGA) plots of **BUT-202** are shown in Figure S2 of the Supporting Information. There are two distinct weight losses before 200 °C, with weight losses of 4.3 and 4.2%, respectively, which are caused by the sequential loss of coordinated water. After a clear platform, the main skeleton decomposes. The thermal stability of **BUT-202** is to about 450 °C. In addition, as shown in Figure S4 of the Supporting Information, slight blue shifts of the characteristic bands of the carbonyl group in **BUT-202** compared with that of their corresponding acid ligands could be observed, illustrating the metal coordination of carboxylate groups in the ligands.

# 3.2. Structure of BUT-202

Single-crystal X-ray diffraction data show that **BUT-202** crystallizes in the monoclinic  $P2_1/n$  space group. The asymmetric unit of **BUT-202** consists of one Cd(II), half a L<sup>4–</sup> ligand, half a dpe ligand, and two lattice water molecules. Each Cd(II) is six-coordinate by two carboxylic oxygens (O1 and O2) from one L<sup>4–</sup> ligand, sulfonic O5, N1 from one dpe ligand and O6 and O7 from two water molecules (Figure 1(a)). The Cd–O bond distances vary from 2.2262(16) to 2.5188(15) Å, while the Cd–N1 bond length is 2.2403(18) Å, which are all similar to values of reported MOFs [46, 47]. The L<sup>4–</sup> is tetradentate, with two carboxylic groups with bidentate chelate modes and two monodentate sulfinic groups. The Cd(II) ions are connected by L<sup>4–</sup> ligands to form a 2-D sheet structure (Figure 1(b)), which is further connected by dpe ligands via the Cd–N connection to generate a 3-D pillar-layered framework (Figure 1(c)). Each L<sup>4–</sup> links four Cd(II) ions, and accordingly, the L<sup>4–</sup> can be regarded as a four-connected node. Each Cd(II) links two L<sup>4–</sup> ligands and one dpe ligand, so the Cd(II) atom is a three-connected node. Thus, the 3-D structure of **BUT-202** can be simplified as a 3,4-coordinated bi-nodal net with



Scheme 1. Synthetic procedure of  $H_4L$  ligand acid.

the point symbol  $\{8\cdot10^2\}_2\{8^4\cdot10^2\}$ , which corresponds to the 3,4T48 topology (Figure 1). Similar topology has been observed in  $\{[Cd(btbb)_{0.5}(btec)_{0.5}(H_2O)]\cdot2H_2O\}_n$  [48].

#### 3.3. Detection of organic explosives

#### 3.3.1. General fluorescence

Due to the excellent fluorescent emission of  $H_4L$ , **BUT-202** was explored for application in the detection of explosive nitroaromatic compounds based on fluorescent sensing. The solid state luminescent properties of **BUT-202**, free  $H_4L$ , and dpe were first checked at room temperature. As shown in Figure S4 of the Supporting Information, free  $H_4L$  and dpe exhibit fluorescent emissions at 395 and 369 nm upon excitations at 317 and 450 nm, respectively. Compared with the free ligands, **BUT-202** shows red-shifted emissions at 439 nm based on excitation at 317 nm. The fluorescent properties of **BUT-202** dispersed in different solvents of DMF, DMSO, acetone, methanol, 1,4-dioxane and acetonitrile, respectively, were examined (Figure S5 of the Supporting Information). Emission of **BUT-202** is strong in DMF, MeCN, and 1,4-dioxane, however, DMSO and acetone can quench the emission of **BUT-202**. Thus, the detection experiments were carried out in DMF.

#### 3.3.2. TNP selective detection based on fluorescent quenching

To explore the detection ability of **BUT-202** for a trace quantity of organic explosives, fluorescent quenching titrations were performed with sequential addition of 1 mM organic explosives DMF solution to the **BUT-202** DMF solution. For comparison, some other aromatics and aliphatic nitro compounds were also tested. Here, 11 analytes including TNP, 2,4-dinitrotoluene(2,4-DNT), 2,6-dinitrotoluene (2,6-DNT), 3-nitrotoluene (3-NT), 2-nitrotoluene



**Figure 1.** (a) Coordination environment of Cd(II) in **BUT-202**; (b) Two-dimensional (2-D) sheet generated by Cd(II) ions and  $L^{4-}$  ligands in **BUT-202**; (c) Schematic view of the 3-D framework built by 2-D Cd(II)/ $L^{4-}$  layers and dpe pillars in **BUT-202** (color scheme: Cd, green; N, blue; S, yellow; O, red; C, gray. Hydrogens on the ligands are omitted for clarity).



**Figure 2.** Effect on the emission spectra of **BUT-202** dispersed in DMF upon incremental addition of 1 mM of (a) TNP and (c) TO; SV plots of (b) TNP and (d) TO.

(2-NT), 4-nitrophenol (4-NP), 3-nitrophenol (3-NP), nitrobenzene (NB), 2,3-dimethyl-2,3-dinitrobutane (DMNB), Toluen (TO), and Chlorobenzene (CB) were checked (Figure 2 and Figure S6 of the Supporting Information). The fluorescence intensity of **BUT-202** is heavily dependent on the identities of the analyte added. TNP is the most effective quencher of all analytes and TO show negligible effect on its fluorescence. Figure S7 shows the percentage of fluorescent quenching in terms of adding a certain amount of different analytes at room temperature. Obviously, TNP gives the highest quenching efficiencies of 70% for **BUT-202** (Figure 2(a)). In addition, 4-NP also leads to relatively high quenching efficiency of 46%, whereas quenching efficiencies are low for the remaining analytes (Figure S6 of the Supporting Information). The quenching efficiencies of **BUT-202** for these analytes follow the order of TNP > 4-NP > NB > 2,4-DNT > 3-NT > 2-NT > 2,6-DNT > DMNB > CB > TO.

The fluorescent quenching efficiency can be quantitatively explained by the Stern–Volmer (SV) equation:  $(I_0/I) = K_{sv}[A] + 1$ , where  $I_0$  is the original fluorescent intensity, *I* indicates the fluorescent intensity in the presence of the analyte, [A] represents the concentration of the analyte (mM), and  $K_{sv}$  represents the quenching constant (M<sup>-1</sup>) [20]. The SV curve of TNP is linear at low concentration ( $R^2 = 0.9971$ ) and deviates linearly when it reaches a higher concentration; the other analytes gave linear SV plots (Figure 2 and Figure S8 of the Supporting Information). The nonlinear properties of the SV curve of TNP is likely due to self-adsorption or energy transfer process [49]. The K<sub>sv</sub> value of **BUT-202** toward TNP was calculated to be  $2.2 \times 10^4 \text{ M}^{-1}$ , which is comparable to that of some reported fluorescent sensors (Table S3 of the Supporting Information). Based on the K<sub>sv</sub> values and the standard deviations (S<sub>p</sub>) for

three repeated fluorescent measurements of blank solutions, the detection limits  $(3S_b/K_{sv})$  of **BUT-202** toward TNP was calculated to be 0.2  $\mu$ M.

The above results demonstrate that **BUT-202** has high quenching efficiencies toward TNP, but very poor toward 2,4-DNT, 2,6-DNT, 3-NT, 2-NT, NB, and DMNB. Motivated by these findings, we further checked the selective detection abilities of **BUT-202** toward TNP in the presence of these analytes. As is shown in Figure S9 of the Supporting Information, the emission intensity of **BUT-202** only shows slight changes in the presence of excess 3-NP (or 2,4-DNT, 2,6-DNT, 3-NT, 2-NT, NB, and DMNB); upon introducing TNP into the mixture of the above solution, the fluorescence was significantly quenched. This result reveals that the interference from 3-NP (or 2,4-DNT, 2,6-DNT, 3-NT, 2-NT, NB, and DMNB); or an be neglected, indicating the high quenching selectivity of **BUT-202** toward TNP. These results can be easily visualized by plotting the percentage fluorescence intensity versus volume of analyte added. As shown in Figure 3, the stepwise decrease in fluorescence of a higher concentration of 3-NP (or 2,4-DNT, 2,6-DNT, 3-NT, 2-NT, NB, and DMNB). The highly selective detection ability makes **BUT-202** a reliable sensing material for TNP.

To better understand the good selectivity of **BUT-202** toward TNP, the mechanism of fluorescence quenching was studied. Due to the limited pore size in **BUT-202**, the quenching process only happened on the surface. The detection of a fluorescent MOF toward organic explosives is commonly related to two processes, photoinduced electron transfer (PET) [50–52] and fluorescence resonance energy transfer (FRET) [53–55]. PET is a deactivation process involving an internal redox reaction between the excited state of the fluorophore and another species able to donate or to accept electrons. For a fluorescent MOF sensor, the conduction band (CB) lies at a higher energy level than the lowest unoccupied molecular orbitals (LUMOs) of an analyte, which leads to a driving force for the electron transfer from the MOF to the analyte, thus resulting in fluorescence quenching. Generally, the lower the LUMO energy levels of an analyte, the better quenching efficiency observed. The LUMO energy levels of the analytes, which are arranged in a descending energy order, are shown in Figure S10 of the Supporting Information. The LUMO energies are in good agreement with the maximum quenching efficiency observed for TNP, but the order of observed



Figure 3. Selective detection of TNP on BUT-202 in the presence of 2,4-DNT (or 2,6-DNT, 3-NT, 2-NT, NB, and DMNB) in DMF.

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quenching efficiency is not fully in accord with the LUMO energies of other nitroaromatics. These results indicate that PET is not the only mechanism for fluorescence quenching observed in these systems.

FRET is another reason for the observed fluorescent quenching. FRET occurs only when the emission spectrum of a fluorophore overlaps with the absorption spectrum of the acceptor. As shown in the UV–Vis absorption spectra of the analytes and **BUT-202** (Figure S11 of the Supporting Information), the absorption band of TNP has the greatest degree of overlap with the emission spectra of **BUT-202**, followed by 3-NP,4-NP, 3-NT, and 3,6-DNT. Clearly, the extent of the overlap is consistent with the quenching efficiencies for nitroaromatics as discussed above. As a result, the coexistence of PET and FRET makes TNP and 4-NP show higher photoluminescence quenching effect compared with other checked analytes.

# 4. Conclusion

A novel fluorescent MOF (**BUT-202**) was synthesized and characterized, containing a sulfonated carboxylate ligand  $H_4L$ , nitrogen containing auxiliary dpe and cadmium ion. The luminescence of **BUT-202** is caused by interaction of ligand and metal ions, and the fluorescence intensity of **BUT-202** is dependent on the kind of solvent, and the fluorescence intensity is largest in DMF. In addition, **BUT-202** can also selectively respond to explosive TNP in DMF even in the presence of other nitro compounds. The selectivity is controlled by electron and energy transfer mechanism and hydrogen bonding between TNP and the framework. The good selectivity of TNP has potential applications in explosive detection and selective fluorescent probes.

#### **Supplementary material**

Crystallographic data for the structure reported in this article has been deposited on the Cambridge Crystallographic Data Center (CCDC No. 1537643). The material can be obtained free of charge via https://www.ccdc.cam.ac.uk/deposit.

# **Disclosure statement**

No potential conflict of interest was reported by the authors.

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