Preparation of \( \text{Eu}_{0.075}\text{Tb}_{0.925} \)-Metal Organic Framework as a Fluorescent Probe and Application in the Detection of \( \text{Fe}^{3+} \) and \( \text{Cr}_2\text{O}_7^{2−} \)

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Abstract: Luminescent Ln-MOFs (\( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \)) were successfully synthesised through the solvothermal reaction of \( \text{Tb}((\text{NO}_3)_{3−6}\text{H}_2\text{O}), \text{Eu}((\text{NO}_3)_{3−6}\text{H}_2\text{O}), \) and the ligand pyromellitic acid. The product was characterised by X-ray diffraction (XRD), TG analysis, EM, X-ray photoelectron spectroscopy (XPS), and luminescence properties, and results show that the synthesised material \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) has a selective ratio-based fluorescence response to \( \text{Fe}^{3+} \) or \( \text{Cr}_2\text{O}_7^{2−} \). On the basis of the internal filtering effect, the fluorescence detection experiment shows that as the concentration of \( \text{Fe}^{3+} \) or \( \text{Cr}_2\text{O}_7^{2−} \) increases, the intensity of the characteristic emission peak at 544 nm of \( \text{Eu}^{3+} \) decreases, and the intensity of the characteristic emission peak at 653 nm of \( \text{Eu}^{3+} \) increases in \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \). The fluorescence intensity ratio \( (I_{544}/I_{653}) \) has a good linear relationship with the target concentration. The detection linear range for \( \text{Fe}^{3+} \) or \( \text{Cr}_2\text{O}_7^{2−} \) is 10–100 \( \mu\text{M/L} \), and the detection limits are 2.71 \( \times 10^{-7} \) and 8.72 \( \times 10^{-7} \) M, respectively. Compared with the sensor material with a single fluorescence emission, the synthesised material has a higher anti-interference ability. The synthesised \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) can be used as a highly selective and recyclable sensing material for \( \text{Fe}^{3+} \) or \( \text{Cr}_2\text{O}_7^{2−} \). This material should be an excellent candidate for multifunctional sensors.

Keywords: Ln-MOFs; luminous sensing; \( \text{Fe}^{3+} \); \( \text{Cr}_2\text{O}_7^{2−} \)

1. Introduction

Heavy metals and inorganic anion pollutants in water pose hidden dangers to human health [1]. The United Nations Sustainable Development Goals set in September 2015 indicated that countries are expected to greatly improve human water quality by 2030. Thus, the detection of pollutants in water has become increasingly important. \( \text{Fe}^{3+} \) is one of the basic trace elements in humans. The lack or excess of this element can cause many physiological disorders, such as nausea, abdominal pain, anaemia, liver cirrhosis, and organ failure [2–4]. Salonen et al. [2] confirmed that elevated iron content is an important risk factor for acute myocardial infarction, Bijeh et al. [3] confirmed that the increased risk of cardiovascular disease is related to elevated iron content, and Jehn et al. [4] confirmed that elevated iron could lead to abnormal baseline metabolism. In addition, \( \text{Cr}_2\text{O}_7^{2−} \) is an important oxidant in laboratories and industry [5], and it is highly carcinogenic in the environment and harmful to the ecology, environment, and biological system [6–9]. Mansi et al. [6] confirmed that it is the second most abundant inorganic groundwater pollutant due to its wide application in many industrial fields, such as electroplating chrome, dyes, and leather tanning. Costa [7] confirmed that it is mutagenic and carcinogenic to organisms’ sexual function. Therefore, the selective sensing of \( \text{Fe}^{3+} \) and \( \text{Cr}_2\text{O}_7^{2−} \) in water quality has attracted growing attention from scholars. Many methods are used for the determination of \( \text{Fe}^{3+} \) and \( \text{Cr}_2\text{O}_7^{2−} \), such as atomic emission spectrometry, atomic absorption spectrometry, inductively coupled plasma mass spectrometry, electrochemical methods, and ion chromatography. However, these methods are complicated to operate,
costly, and have a long detection time. Therefore, developing a simple and efficient method to determine Fe\(^{3+}\) and Cr\(^{3+}\) is of practical significance. Fluorescence sensing technology can meet the requirements of new analysis and detection technology due to its high sensitivity, fast analysis speed, strong selectivity, simple operation, and low experimental cost. In recent years, it has received extensive attention [10].

Ln-MOFs materials refer to the self-assembly connection of metal ions and organic ligands by coordination bonds to form network complexes. Ln-MOFs materials have outstanding luminescence characteristics; that is, they have the advantages of large Stokes shift, high quantum yield and luminescence intensity, narrow emission spectrum range, flexible coordination mode, and long luminescence life. MOFs fluorescent probes are commonly used as sensors [11–27]. Hna et al. [26] synthesised Ce-MOF to detect Fe\(^{3+}\), and Gai et al. [27] synthesised dual-sensor Eu-MOF to detect Fe\(^{3+}\) and Cr\(^{2+}\). The ratio fluorescence probe is based on measuring the ratio of the fluorescence intensity of two independent fluorescence emission peaks for quantitative analysis, which can effectively reduce the influence of excitation light, environment, and probe concentration changes, and greatly improve the accuracy of the method. At present, the usual design method of the Ln-MOFs ratio probe is to select two kinds of Ln\(^{3+}\) to synthesise by different molar ratios [28–30] or to combine Ln-MOFs with one or two substances with different fluorescence emission wavelengths, including carbon dots (CDs), quantum dots, and fluorescent dyes [31–35]. Zhang et al. [30] used two different molar ratios of Tb and Eu as the metal centre. 2,2′-bipyridine-6,6′-dicarboxylate acid (H\(_2\)bpdc) is a ligand to synthesise Eu\(_{0.6059}\)Tb\(_{0.3941}\)ZMOF, which can realise the selective detection of haemolysed phosphate (lysophosphatidic acid or LPA) in human plasma. Xu et al. [31] reported that CDs with strong fluorescence activity and Eu\(^{3+}\) were encapsulated in MOF-253, and the dual-emission ratio probe Eu\(^{3+}\)/CDs@MOF-253 was synthesised to detect Hg\(^{2+}\). Therefore, the development of ratio fluorescent probe Ln-MOFs to detect Fe\(^{3+}\) and Cr\(^{2+}\) has great application prospects.

The selective fluorescence detection of Fe\(^{3+}\) and Cr\(^{2+}\) using ratio fluorescent probe Ln-MOFs is rarely reported in the literature. In this paper, luminescent Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF was successfully synthesised by the solvothermal reaction of Tb(NO\(_3\))\(_3\)·6H\(_2\)O, Eu(NO\(_3\))\(_3\)·6H\(_2\)O, and ligand pyromellitic acid. Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF was comprehensively characterised by XRD, thermogravimetric analysis (TG), elemental analysis, Fourier transform infrared spectroscopy (FTIR), transmission electron microscope (TEM), scanning electron microscope (SEM), and XPS. Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF has excellent stability in aqueous solution, and it can detect Fe\(^{3+}\) and Cr\(^{2+}\) in aqueous solution by dual-emission ratio fluorescence sensing, which provides a new idea for the fluorescence detection of Fe\(^{3+}\) and Cr\(^{2+}\).

2. Materials and Methods

Commercially available reagents and solvents were used. XRD characterisation was performed to determine the regular arrangement of atoms or ions in the Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF, which is one of the commonly used methods to explore the structure of matter. An elemental analyser was used for elemental analysis. FTIR was used to scan and analyse the range of 4000–400 cm\(^{-1}\) to determine the functional groups and chemical bonds of Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF. The thermal stability of Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF was analysed by TG, which was performed under N\(_2\) protection. TEM and SEM were used to observe the specific morphology of Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF. The FL/FS900 fluorescence spectrometer was used to record the steady-state luminescence performance of Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF. XPS and UV spectrophotometers were used to investigate the reaction mechanism.

Synthesis of Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF: Product preparation was the first step. Eu\(_{0.075}\)Tb\(_{0.925}\)-MOF with Tb and Eu were prepared as the metal centre, and pyromellitic acid was prepared as the organic ligand as follows: Dissolved Tb(NO\(_3\))\(_3\)·6H\(_2\)O + Eu(NO\(_3\))\(_3\)·6H\(_2\)O (0.2 mmol), pyromellitic acid (0.2 mmol), DMF (8 mL), distilled water (4 mL), and CH\(_3\)CH\(_2\)OH (4 mL) were transferred to an autoclave (volume: 25 mL). The product was then sealed and heated in a 120 °C vacuum drying oven for 48 h and gradually cooled to ambient temperature. After the autoclave was opened, the product was collected after centrifugation, washed
thoroughly with DMF and ethanol, paralleled three times, and dried. Thus, the target product \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) was obtained.

3. Results and Discussion

3.1. XRD Characterisation

\( \text{Eu-MOF}, \text{Tb-MOF}, \) and \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) combined with lanthanide nitrate and pyromellitic acid were prepared by the solvothermal method. Figure 1 shows the XRD patterns of Ln-MOFs. As shown in the figure, the 20 diffraction angle peak positions of the simulated XRD pattern and the synthesised samples \( \text{Eu-MOF}, \text{Tb-MOF}, \) and \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) are the same, and there are sharp peaks at the diffraction angles from 9 to 10. At the same time, the diffraction peaks 9 to 10 of the crystal synthesised by Silva et al. [36] are basically the same, indicating that the synthesised \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) has high purity and good crystallinity [36–39].

![Figure 1. (a) XRD patterns of Ln-MOFs. (b) Enlarged version.](image)

3.2. TG Analysis

Figure 2 shows the TG analysis results of Ln-MOFs. The weight loss of Ln-MOFs is mainly divided into two stages. Before 340 °C, \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) has good thermal stability.

![Figure 2. TG of Ln-MOFs.](image)

3.3. FTIR Analysis

Figure 3 shows the FTIR spectrum of Ln-MOFs. Compared with the FTIR spectrum of pyromellitic acid, the main characteristic peaks in the FTIR spectrum of \( \text{Eu}_{0.075}\text{Tb}_{0.925}\text{-MOF} \) are similar to those of pyromellitic acid, but the C=O stretching vibration peak disappeared at 1720 cm\(^{-1}\) in the original pyromellitic acid spectrum (significantly weakened), thereby indicating that the carboxyl oxygen is coordinated with Tb and Eu atoms in the ligand.
Figure 3 shows the FTIR spectrum of Ln-MOFs. Compared with the FTIR spectrum of Benzene-1,2,4,5-tetracarboxylic acid and Ln-MOFs, the main characteristic peaks in the FTIR spectrum of Eu$_{0.075}$Tb$_{0.925}$-MOF are similar to those of pyromellitic acid, but the C=O stretching vibration peak disappeared at 1720 cm$^{-1}$ in the original pyromellitic acid spectrum (significantly weakened), thereby indicating that the carboxyl oxygen is coordinated with Tb and Eu atoms in the ligand.

3.4. Elemental Analysis and XPS

A comparison of elemental (Table 1) and XPS (Figure 4) analyses shows that, corresponding to the content of the element, the distribution ratio of Eu to Tb in Eu$_{0.075}$Tb$_{0.925}$-MOF is 0.075:0.925. The specific loadings of Tb(NO$_3$)$_3$ and Eu(NO$_3$)$_3$ are 42.74% and 3.46% respectively, and the cooling rate is 0.017 K/s.

Table 1. Element analysis table.

| Ln-MOFs       | C   | H   | N   | O   | Eu/Tb |
|---------------|-----|-----|-----|-----|-------|
| Eu-MOF        | 30.37% | 1.87% | 1.87% | 26.64% | 39.25% |
| Tb-MOF        | 22.01% | 1.84% | 1.13% | 26.11% | 48.91% |
| Eu$_{0.075}$Tb$_{0.925}$-MOF | 23.53% | 1.85% | 1.56% | 26.85% | 46.21% |

Figure 4. XPS of Eu$_{0.075}$Tb$_{0.925}$-MOF before and after Fe$^{3+}$ addition: (a) Tb 3d, (b) Eu 3d, and (c) O 1s.

3.5. EM Characterisation

Figure 5 shows the TEM and SEM images of Eu$_{0.075}$Tb$_{0.925}$-MOF, which indicate that the prepared Eu$_{0.075}$Tb$_{0.925}$-MOF has a regular external morphology, a nanocolumn shape, and a diameter of about 500 nm.
3.6. Adsorption Characteristics of Eu$_{0.075}$Tb$_{0.925}$-MOF

Figure 6 shows the N$_2$ adsorption desorption isotherms of Eu$_{0.075}$Tb$_{0.925}$-MOF. The adsorption capacity increases slowly with the increase of pressure at the middle–high-pressure stage, indicating that Eu$_{0.075}$Tb$_{0.925}$-MOF is a porous material with an average pore size of 3.38 nm, a BJH average pore diameter of 20.99 nm, and a BET specific surface area of 12.9542 m$^2$/g.

Figure 6. The N$_2$ adsorption desorption isotherms of Eu$_{0.075}$Tb$_{0.925}$-MOF.

3.7. Photoluminescence Characteristics

Figure 7a shows the fluorescence emission spectrum of Eu$_{0.075}$Tb$_{0.925}$-MOF measured at ambient temperature. The figure shows that Eu$_{0.075}$Tb$_{0.925}$-MOF exhibits characteristic transitions of Tb$^{3+}$ and Eu$^{3+}$ under the excitation of 310 nm light, located at 544 and 653 nm respectively, showing the same intensity of fluorescence emission. This finding indicates that the ligand can effectively transfer energy to Tb$^{3+}$ and Eu$^{3+}$ at the same time [40–44].

As shown in the CIE diagram in Figure 7c,d, Eu-MOF shows red fluorescence, and Tb-MOF shows green fluorescence. When Eu$^{3+}$ and Tb$^{3+}$ synthesise Eu$_{0.075}$Tb$_{0.925}$-MOF at a ratio of 0.075:0.925, Eu$_{0.075}$Tb$_{0.925}$-MOF shows the intermediate colour of the two, which is a yellow-green fluorescence sensitive to the human eye. This material has potential application as a luminescent material and a light-sensitive material for naked-eye detection [45].
standard deviation of repeated detection of the original solution, and $S$ is the slope of the trend of Eu $0.075Tb^{0.925}$-MOF is yellow green–yellow–orange–red with the increase in Fe $^{3+}$ concentration. This material is expected to achieve naked-eye detection of Fe$^{3+}$.

The limit of detection (LOD) of Fe$^{3+}$ is evaluated by the equation $3S_b/S$, where $S_b$ is the standard deviation of repeated detection of the original solution, and $S$ is the slope of the linear fit. The LOD is calculated as $2.71 \times 10^{-7}$ M. Figure 8c shows that the colour change trend of Eu$_{0.075}$Tb$_{0.925}$-MOF is yellow green–yellow–orange–red with the increase in Fe$^{3+}$ concentration. This material is expected to achieve naked-eye detection of Fe$^{3+}$.

The prepared Eu$_{0.075}$Tb$_{0.925}$-MOF was subjected to a cyclic application experiment, and KNO$_3$ solution was used to wash the used materials. Figure 8d,e shows that the ratio of the luminous intensity of the material and the XRD did not change considerably, even after five cycles. Eu$_{0.075}$Tb$_{0.925}$-MOF is very stable in the sensing experiment.

Figure 7. (a) Emission spectra of Eu$_{0.075}$Tb$_{0.925}$-MOF, (b) CIE of Eu$_{0.075}$Tb$_{0.925}$-MOF, (c) CIE of Eu-MOF, and (d) CIE of Tb-MOF.

3.8. Fluorescence Sensing of Fe$^{3+}$

Gao and Ma [46,47] prepared Tb-MOF and used it for sensitive fluorescence sensing of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$. On this basis, this paper designs a ratio fluorescent probe, Eu$_{0.075}$Tb$_{0.925}$-MOF, for the fluorescence sensing of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ to improve the measurement accuracy and expand the linear range of the test. To determine the fluorescence performance of Eu$_{0.075}$Tb$_{0.925}$-MOF to Fe$^{3+}$, the fluorescence response of Eu$_{0.075}$Tb$_{0.925}$-MOF to Fe$^{3+}$ was investigated, and the results are shown in Figure 8.

Figure 8a shows that with the increase of the Fe$^{3+}$ concentration, the characteristic emission peak intensity of Tb$^{3+}$ decreases at 544 nm, and the characteristic emission peak intensity of Eu$^{3+}$ increases at 653 nm. The intensity at I$_{Eu} = 653$ nm and I$_{Tb} = 544$ nm is used to calculate the intensity change I$_0/I$, where I$_0$ (I$_{Eu0}/I_{Tb0}$) is the initial fluorescence intensity before fluorescence, and I (I$_{Eu}/I_{Tb}$) is the fluorescence intensity in the presence of Fe$^{3+}$. Figure 8b shows that I$_0/I$ and Fe$^{3+}$ present a linear relationship in the concentration range of 10–100 µM/L, and the linear regression equation is:

$$I_0/I = 0.71 - 7948.64x. \quad (1)$$

The limit of detection (LOD) of Fe$^{3+}$ is evaluated by the equation $3S_b/S$, where $S_b$ is the standard deviation of repeated detection of the original solution, and $S$ is the slope of the linear fit. The LOD is calculated as $2.71 \times 10^{-7}$ M. Figure 8c shows that the colour change trend of Eu$_{0.075}$Tb$_{0.925}$-MOF is yellow green–yellow–orange–red with the increase in Fe$^{3+}$ concentration. This material is expected to achieve naked-eye detection of Fe$^{3+}$.
The prepared Eu0.075Tb0.925-MOF was subjected to a cyclic application experiment, and KNO3 solution was used to wash the used materials. Figure 8d,e shows that the ratio of the luminous intensity of the material and the XRD did not change considerably, even after five cycles. Eu0.075Tb0.925-MOF is very stable in the sensing experiment.

The fluorescence sensing selectivity of Eu0.075Tb0.925-MOF to Fe3+ was investigated through the anti-interference experiment. The Eu0.075Tb0.925-MOF sample was immersed in NaX solution (Mg2+, K+, Pb2+, Al3+, Na+, Cd2+, Mn2+, Zn2+, Ni2+, Fe2+, Cu2+, Hg2+) at a concentration of 1 × 10⁻⁴ M. The results are shown in Figure 8f. Except for Fe3+, the luminous intensity ratio of Eu0.075Tb0.925-MOF exhibits almost no change after the addition of metal ions. However, when the same amount of Fe3+ was added to the Mg2+, K+, Pb2+, Al3+, Na+, Cd2+, Mn2+, Zn2+, Ni2+, Fe2+, Cu2+, and Hg2+ solution containing Eu0.075Tb0.925-MOF, the luminous intensity ratio of I_{Eu}/I_{Tb} was significantly higher. This result shows that the sensing ability of Eu0.075Tb0.925-MOF on Fe3+ will not be interfered with by the presence of other metal ions. Therefore, Eu0.075Tb0.925-MOF has a high selectivity for Fe3+ in an aqueous solution.

Figure 8. (a) The emission spectra of Eu0.075Tb0.925-MOF dispersions with different Fe3+ concentrations under 310 nm excitation light. (b) Calibration line with Fe3+ (in the range of 10–100 µM/L), (c) CIE, (d) cycles of Eu0.075Tb0.925-MOF, (e) XRD pattern of Eu0.075Tb0.925-MOF after five cycles, and (f) I_{Eu}/I_{Tb} histogram of Eu0.075Tb0.925-MOF dispersion containing metallic cations.

The fluorescence sensing selectivity of Eu0.075Tb0.925-MOF to Fe3+ was investigated through the anti-interference experiment. The Eu0.075Tb0.925-MOF sample was immersed in NaX solution (Mg2+, K+, Pb2+, Al3+, Na+, Cd2+, Mn2+, Zn2+, Ni2+, Fe2+, Cu2+, Hg2+) at a concentration of 1 × 10⁻⁴ M. The results are shown in Figure 8f. Except for Fe3+, the luminous intensity ratio of Eu0.075Tb0.925-MOF exhibits almost no change after the addition of metal ions. However, when the same amount of Fe3+ was added to the Mg2+, K+, Pb2+, Al3+, Na+, Cd2+, Mn2+, Zn2+, Ni2+, Fe2+, Cu2+, and Hg2+ solution containing Eu0.075Tb0.925-MOF, the luminous intensity ratio of I_{Eu}/I_{Tb} was significantly higher. This result shows that the sensing ability of Eu0.075Tb0.925-MOF on Fe3+ will not be interfered with by the
presence of other metal ions. Therefore, Eu$_{0.075}$Tb$_{0.925}$-MOF has a high selectivity for Fe$^{3+}$ in an aqueous solution.

3.9. Fluorescence Sensing of Cr$_2$O$_7^{2-}$

To determine the fluorescence performance of Eu$_{0.075}$Tb$_{0.925}$-MOF to Cr$_2$O$_7^{2-}$, the fluorescence response of Eu$_{0.075}$Tb$_{0.925}$-MOF to Cr$_2$O$_7^{2-}$ was investigated, and the results are shown in Figure 9.

![Figure 9](image)

**Figure 9.** (a) The emission spectra of Eu$_{0.075}$Tb$_{0.925}$-MOF dispersions with different Cr$_2$O$_7^{2-}$ concentrations under 310 nm excitation light. (b) Calibration line with Cr$_2$O$_7^{2-}$ (in the range of 10–100 μM/L), (c) CIE, (d) cycles of Eu$_{0.075}$Tb$_{0.925}$-MOF, and (e) $I_{Eu}/I_{Tb}$ histogram of Eu$_{0.075}$Tb$_{0.925}$-MOF dispersion containing anions.

Figure 9a shows that with the increase in Cr$_2$O$_7^{2-}$ concentration, the characteristic emission peak intensity of Tb$^{3+}$ decreases at 544 nm, and the characteristic emission peak...
intensity of Eu$^{3+}$ increases at 653 nm. At the same time, $I_0/I$ and Cr$_2$O$_7^{2-}$ showed a linear correlation in the concentration range of 10–100 µM/L, the linear regression equation is:

$$I_0/I = 0.81 - 9660.83x,$$

and the LOD was $8.72 \times 10^{-7}$ M. The CIE diagram in Figure 9c shows that with the increase in Cr$_2$O$_7^{2-}$ concentration, the colour change trend of Eu$_{0.075}$Tb$_{0.925}$-MOF is yellow-green–yellow–orange–red, which is expected to realise the naked-eye detection of Cr$_2$O$_7^{2-}$.

A cyclic experiment was performed on Eu$_{0.075}$Tb$_{0.925}$-MOF. Figure 9d shows that the luminous intensity ratio of Eu$_{0.075}$Tb$_{0.925}$-MOF does not change much after five cycles. Eu$_{0.075}$Tb$_{0.925}$-MOF was very stable in the sensing experiment.

Similarly, the fluorescence sensing selectivity of Eu$_{0.075}$Tb$_{0.925}$-MOF to Cr$_2$O$_7^{2-}$ was investigated through the anti-interference experiment. Eu$_{0.075}$Tb$_{0.925}$-MOF was dispersed into a solution containing $\text{F}^-$, $\text{Cl}^-$, $\text{I}^-$, $\text{Br}^-$, $\text{NO}_3^-$, $\text{CrO}_4^{2-}$, $\text{SCN}^-$, $\text{IO}_3^-$, $\text{CO}_3^{2-}$, and Cr$_2$O$_7^{2-}$ with the same concentration. The results are shown in Figure 9e. Except for Cr$_2$O$_7^{2-}$, the luminous intensity ratio of Eu$_{0.075}$Tb$_{0.925}$-MOF is almost unchanged after the addition of anions. However, when the same amount of Cr$_2$O$_7^{2-}$ was added to the F$^-$, Cl$^-$, I$^-$, Br$^-$, NO$_3^-$, CrO$_4^{2-}$, SCN$^-$, IO$_3^-$, and CO$_3^{2-}$ solution containing Eu$_{0.075}$Tb$_{0.925}$-MOF, the luminous intensity ratio of Eu$_{0.075}$Tb$_{0.925}$-MOF has a high selectivity for Cr$_2$O$_7^{2-}$ in an aqueous solution.

### 3.10. Comparison with Other Sensors That Detect Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ Ions

Compared with the Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ detection methods used in other studies, as shown in Table 2, the prepared Eu$_{0.075}$Tb$_{0.925}$-MOF can reduce the effects of interference caused by excitation light, the environment, and probe concentration changes, and it has improved the detection accuracy relative to other methods.

**Table 2. Comparison of the reported methods for Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ using Ln-MOFs.**

| Ln-MOFs | Detect Ion | LOD (M) | Ratio Fluorescent Probe | Linear Range | References |
|---------|------------|---------|-------------------------|--------------|------------|
| Eu$_{0.075}$Tb$_{0.925}$-MOF | Fe$^{3+}$ | 2.71 $\times 10^{-7}$ | Dual emission | 10–100 µM | This work |
| Eu$_{0.075}$Tb$_{0.925}$-MOF | Cr$_2$O$_7^{2-}$ | 8.72 $\times 10^{-7}$ | | | |
| Eu-MOF: Tb-MOF [Eu/Tb, 4,4′-((5-carboxy-1,3-phenylene)bis(azanediyl))bis(carbonyl)] dibenzoic acid | Fe$^{3+}$ | 1 $\times 10^{-5}$ | Single emission | 0–1.0 mM | [47] |
| | Cr$_2$O$_7^{2-}$ | 8.94 $\times 10^{-5}$ | | | |
| Eu-MOF [Eu, 5-(2′,5′-dicarboxyphenyl) picolinic acid ligand] | Fe$^{3+}$ | 5.7 $\times 10^{-7}$ | Single emission | 0–50 µM | [48] |
| | Cr$_2$O$_7^{2-}$ | 4.2 $\times 10^{-7}$ | | | |
| Tb-MOF [Tb,H3BTB] | Fe$^{3+}$ | 1 $\times 10^{-5}$ | Single emission | - | [49] |
| Eu-MOF [Eu, 2-aminoterephthalic acid, 1,10-phenanthroline] | Fe$^{3+}$ | 4.5 $\times 10^{-5}$ | Single emission | 0–0.25 mM | [50] |
| Tb-MOF [Tb, 2-(2-carboxyphenoxy)terephthalic acid] | Fe$^{3+}$ | 2.0 $\times 10^{-4}$ | Single emission | $10^{-3}$–$10^{-5}$ M | [51] |
| Eu-MOF [Eu, 2-(3′,4′-dicarboxyphenoxo)isophthalic acid, 4,4′-bis(imidazolyl) biphenyl] | Fe$^{3+}$ | 1.32 $\times 10^{-5}$ | Single emission | 0–$10^{-5}$ M | [52] |

### 3.11. Mechanism Study

The mechanism of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ on Eu$_{0.075}$Tb$_{0.925}$-MOF fluorescence sensing is examined. Figure 10a shows that the UV absorption spectrum of Fe$^{3+}$ overlaps with the excitation spectrum of Eu$_{0.075}$Tb$_{0.925}$-MOF, which indicates that Fe$^{3+}$ and Eu$_{0.075}$Tb$_{0.925}$-MOF are competitively adsorbed. At the same time, Figure 4 shows that Fe$^{3+}$ is attached to the
surface of Eu$_{0.075}$Tb$_{0.925}$-MOF and that the interaction between Fe$^{3+}$ and the uncoordinated O atom in the ligand is weak. Eu$_{0.075}$Tb$_{0.925}$-MOF reduces the energy transfer from the ligand to Tb$^{3+}$, and Tb$^{3+}$ is quenched. As a result, the energy transfer from the ligand to Eu$^{3+}$ is increased, and the characteristic red fluorescence of Eu$^{3+}$ is displayed. Figure 10 shows that the UV absorption spectrum of Cr$_2$O$_7^{2-}$ overlaps with the excitation spectrum of Eu$_{0.075}$Tb$_{0.925}$-MOF, which indicates that Cr$_2$O$_7^{2-}$ and Eu$_{0.075}$Tb$_{0.925}$-MOF are competitively adsorbed. It will also cause the energy transfer from the ligand to Eu$^{3+}$ to increase and show the characteristic red fluorescence of Eu$^{3+}$.

![Figure 10](image)

**Figure 10.** (a) Fluorescence excitation spectra of Eu$_{0.075}$Tb$_{0.925}$-MOF and UV-Vis absorption spectra of Fe$^{3+}$. (b) Fluorescence excitation spectra of Eu$_{0.075}$Tb$_{0.925}$-MOF and UV-Vis absorption spectra of Cr$_2$O$_7^{2-}$. (c) The mechanism of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ on Eu$_{0.075}$Tb$_{0.925}$-MOF fluorescence sensing.

### 3.12. Application in Actual Water Sample Analysis

The ratio fluorescent probe Eu$_{0.075}$Tb$_{0.925}$-MOF was used for Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ in tap water. The results are shown in Table 3. The sample recovery rate is 101–114%, thereby showing that the established method has high accuracy and precision for the determination of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ content in actual samples.

| Sample          | Spiked (nM) | Found (nM) | Recovery (%) |
|-----------------|-------------|------------|--------------|
| Tap water (Fe$^{3+}$) |             |            |              |
| 20.0            | 22.1        | 110.5      |
| 40.0            | 45.7        | 114.3      |
| 60.0            | 61.6        | 102.7      |
| 800             | 88.7        | 110.9      |
| Tap water (Cr$_2$O$_7^{2-}$) |             |            |              |
| 20.0            | 20.9        | 104.5      |
| 40.0            | 41.3        | 103.3      |
| 60.0            | 60.9        | 101.5      |
| 80.0            | 80.8        | 101.0      |
4. Conclusions

The ratio fluorescent probe Eu$_{0.075}$Tb$_{0.925}$-MOF was synthesised in this experiment by using the solvothermal method and was used for Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ determination. Mainly on the basis of the internal filtering effect, the characteristic fluorescence emission peak intensity of Tb$^{3+}$ decreased, and the characteristic emission peak intensity of Eu$^{3+}$ increased on Eu$_{0.075}$Tb$_{0.925}$-MOF as the concentration of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$ increased. The ratio of the emission fluorescence intensity at the two wavelengths has a linear relationship with the target concentration, which realises the selective detection of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$. The linear detection range was 10–100 µM, and the LOD was 2.71 × 10$^{-7}$ and 8.72 × 10$^{-7}$ M, respectively. The synthesised material was used as a ratio fluorescent probe, which can effectively eliminate background fluorescence interference in the detection process and improve the detection accuracy. The trend of the fluorescence colour change of the synthesised material during the detection process indicates that the material is expected to realise naked-eye detection of Fe$^{3+}$ and Cr$_2$O$_7^{2-}$.

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