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Article

# Porous Europium Metal-Organic Frameworks as Highly Sensitive Bi-Functional Sensor for Isoprocarb and Levofloxacin

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## Abstract

The development of highly sensitive fluorescence sensing materials has attracted much attention in recent years. In this study, a new two-dimensional porous europium metal-organic frameworks (EuMOFs) have been obtained. Studies have shown that EuMOFs is a stable, fast response, and highly sensitive fluorescence sensor for isoprocarb and levofloxacin (Lvx), which are closely related to food safety and human health. The limits of detection (LOD) for isoprocarb and Lvx are as low as 1.0 and 0.5 nM, respectively, which were much lower than the national standards (GB 28260-2011 for isoprocarb is 2.583  $\mu$ M). EuMOFs can also achieve strong anti-interference detection of isoprocarb in apple peel and rice extract solution, and Lvx in real urine, with excellent detection stability in 0.01~9.0 nM. The recovery rates for isoprocarb and Lvx in real samples are in 99.12%~101.25%.

**Keywords:** lanthanide MOFs; fluorescence; sensor; isoprocarb; levofloxacin

## 1. Introduction

Isoprocarb can inhibit acetylcholinesterase, so they are widely used to kill insects. The use of pesticides improves crop yield and the preservation of agricultural products, but their residues also cause food and environmental pollution. The use of pesticides in cereals has been widely reported, and the trend of their use is expected to increase substantially in the coming decades, which is a serious concern. Isoprocarb that remains in plants, vegetables and fruits, can inhibit the activity of the human central and peripheral nervous system and lead to the accumulation of the neurotransmitter acetylcholine in the body, leading to acute poisoning. In addition, long-term intake of food with pesticide residue levels exceeding the recommended standards can lead to chronic poisoning, endocrine interference, reproductive impact, accelerate dementia, increase the risk of cancer [1]. The World Health Organization and the Food and Agriculture Organization of the United Nations have classified isoprocarb as a high-hazard pesticide. Therefore, the detection and quantitative analysis of isoprocarb is highly needed.

Levofloxacin (Lvx), as one of the fluoroquinolones, is a typical broad-spectrum antibiotics, which is widely used in the treatment of various bacterial infections in animals and humans, pharmaceutical, food, environment and other fields. Improper use of Lvx may cause adverse symptoms such as heart disease, central nervous system reactions, respiratory system damage, gastrointestinal damage, immune dysfunction, impaired kidney function, and lead to serious multi-antibiotic resistance, threatening global public health [2]. And excessive use of Lvx is not completely metabolized in the human body and will exist in the urine. The content of various substances in urine is usually used as an effective indicator for monitoring toxic substances in the human body and plays

an important role in health assessment. Therefore, the development of reliable and sensitive methods to detect Lv<sub>x</sub> is a current health monitoring need.

Instrument methods for the detection of isoprocarb have high sensitivity, accuracy and repeatability, such as high performance liquid chromatography, gas chromatography, and spectrophotometry [3–5]. The reported methods such as high-performance liquid chromatography, enzyme-linked immunosorbent assay, electrochemical analysis, spectrophotometry, chemiluminescence, capillary electrophoresis and iodine determination have been applied to the detection of Lv<sub>x</sub> [6,7]. However, these above methods for the sensing of isoprocarb and Lv<sub>x</sub> have strict requirements for the pretreatment and handling of samples. The instrument method has some disadvantages such as long time, non-portability, expensive equipment, and complicated operation process, so it is difficult to meet the requirements for the monitoring of isoprocarb or Lv<sub>x</sub> in real-time field detection.

Metal compounds have wide application in the field of optics [8–16], electronics [17–19], magnetism, catalysis [20–31], gas adsorption and separation [32–35], bio-applications [36–43], and so on [44,45]. As one kind of metal compounds, lanthanide compounds have the unique fluorescence characteristics of large Stokes shift, long fluorescence lifetime, sharp emission band, etc. [46–54], and have great application potential in luminescent sensors [55–62]. Lanthanide complexes have the unique characteristics of rapid response, high sensitivity and real-time monitoring ability, which overcome the shortcomings of traditional methods and provide an effective technology and application prospect for molecular detection [63,64]. Based on the above shortcomings in detection efficiency, a dual fluorescence sensing system for isoprocarb and Lv<sub>x</sub> was established.

In summary, a new LnMOFs have been obtained and characterized in detail, and their fluorescence properties have been studied in detail. Interestingly, **EuMOFs** is a stable, fast, highly sensitive bifunctional sensor for isoprocarb and Lv<sub>x</sub>. Based on the study of fluorescence lifetime, UV-visible absorption and density functional theory (DFT), the sensing mechanisms were discussed. As an efficient pesticide responder, **EuMOFs** provides a new detection platform for isoprocarb residues in crops, and has great potential application value in food safety and human health. The developed fluorescence sensor can also detect Lv<sub>x</sub> in human urine effectively and quickly, which has extraordinary significance for human health and ecological environment.

## 2. Materials and Methods

### 2.1. Synthesis of EuMOFs

The ligand of 2,2-dihydroxyacetic acid (C<sub>2</sub>H<sub>4</sub>O<sub>4</sub>, 8.51 mg, 0.092 mmol) and 1.0 mL H<sub>2</sub>O were mixed evenly by ultrasonic treatment in a 10.0 mL flask for 5.0 min, and was adjusted to pH=5 with NaOH. 10.2 mg Eu (NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O (0.023 mmol) and 1.0 mL methanol were mixed in the second flask and sonicated for 5.0 min. The above two solutions were mixed evenly with ultrasonic treatment for 6.0 min, and then reacted for 3 days in 80 °C oven to obtain colorless massive crystals, high quality single-crystal were selected for single-crystal X-ray analysis.

**[Eu(OA)<sub>1.5</sub>·3H<sub>2</sub>O]<sub>n</sub> (EuMOFs):** Yield: 35.6%, based on Eu<sup>3+</sup>. Anal. Calcd (%): C, 12.51, H, 2.10. Found (%): C, 13.05; H, 2.46. FT-IR (Figure S1) (KBr pellet, cm<sup>-1</sup>): 3449(s), 2828(w), 2356(w), 2339(w), 2027(w), 1636(s), 1586(w), 1387(m), 1351(m), 1269(w), 1082(s), 998(m), 769(m), 565(s), 468(w).

### 2.2. Preparation of Sensing Solution

Configuration of the **EuMOFs** solution: As synthesized **EuMOFs** was ground into a white powder and made into a transparent and homogeneous solution using DMF as the solvent (1.0×10<sup>-3</sup> M, calculation by the molar of Eu<sup>3+</sup>) for later use.

Configuration of sensing solution: 50  $\mu\text{L}$  EuMOFs stock solution ( $1.0 \times 10^{-3}$  M) was dissolved in 4.9 mL of deionized water, and 50  $\mu\text{L}$  of tested species water solution ( $1.0 \times 10^{-3}$  M) mixed thoroughly before the fluorescence measurement. In the blank sample, 50  $\mu\text{L}$  of tested species solution was replaced by 50  $\mu\text{L}$  of deionized water. Real sample of tap water was collected from the water tap in our laboratory. Real sample of lake water was gathered from the Yao Lake near our university. The urine samples were provided by the researchers of this study, and the serum was purchased from Innochem. The real samples were filtered with 0.45  $\mu\text{m}$  membrane filters prior to testing.

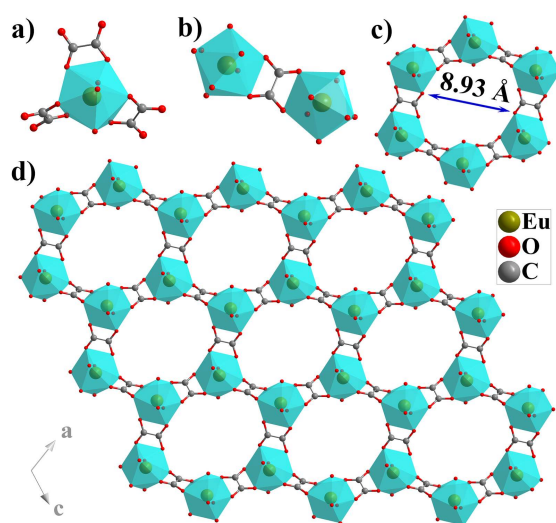
### 2.3. Computational Details

Quantum chemical studies were performed using density functional theory (DFT) implemented in GAUSSIAN 16 package [65,66]. Geometry optimization and single point energies are calculated at B3LYP functional with 6-31G(d) basis sets for other elements [67–71].

## 3. Results and Discussion

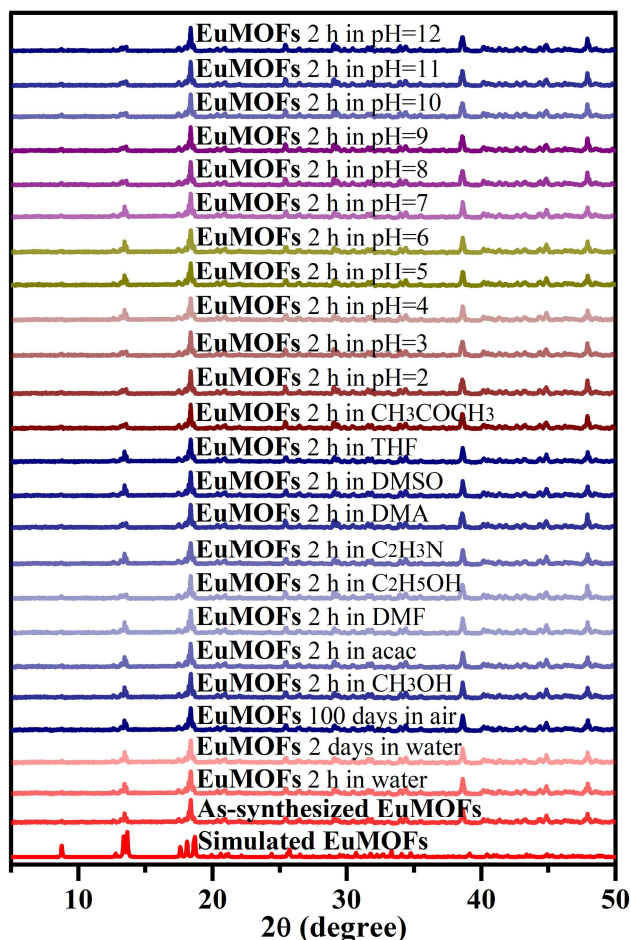
### 3.1. Crystal Structure Analysis

Single-crystal X-ray diffraction analyses reveal that EuMOFs, crystallizes in a triclinic space group  $P21/c$  and EuMOFs are in a Monoclinic space group  $P21/c$ . The crystallography data and structural refinement parameters of EuMOFs are listed in Table S1. EuMOFs crystallizes in the monoclinic  $P21/c$  space group, with  $a = 11.0633(7)$   $\text{\AA}$ ,  $b = 9.5274(4)$   $\text{\AA}$ ,  $c = 10.0760(8)$   $\text{\AA}$ ,  $\alpha = 90^\circ$ ,  $\beta = 114.423(8)^\circ$ ,  $\gamma = 90^\circ$ ,  $V = 967.02(12)$   $\text{\AA}^3$ ,  $Z = 4$ . Each SBU contains one  $\text{Eu}^{3+}$ , one and half deprotonated oxalic acid that decomposition from 2,2-dihydroxyacetic acid, three coordination  $\text{H}_2\text{O}$  to form a neutral unit. The lanthanide ion is coordinated by 9 O to form polyhedral structure, where six O from the ligand of OA, and other three O are from coordinated  $\text{H}_2\text{O}$  (Figure 1a). The ligand of OA adopts a chelating coordination pattern (Figure S2). Two polyhedral structures are connected to form a dinuclear cluster structure (Figure 1b), the nine coordinated O around the metal center are arranged in a single truncated quadrangular prism structure (Figure S3). Three dinuclear clusters are connected in the  $ac$  plane to form a single-hole structure (Figure 1c), and the hole has a diameter of 8.93  $\text{\AA}$ . It further connects in the  $oa$  and  $oc$  directions through coordination bond to form a 2D porous MOFs structure (Figure 1d). The water contact angles of EuMOFs (Figure S4) sheet is in  $77.6$ – $78.8^\circ$ , and lower than  $90^\circ$ , which is due to the fact that water easily forms hydrogen bonds with uncoordinated O. The coordination bond lengths are in normal range of 2.2981–2.588  $\text{\AA}$  for coordination compounds (Table S2) [72–74].



**Figure 1.** (a) SBU structure of EuMOFs; (b) the dinuclear cluster structure; (c) single hole structure constructed by three dinuclear cluster; (d) 2D porous MOFs structure of EuMOFs. .

**Powder X-ray Diffraction (PXRD).** PXRD peaks of **EuMOFs** samples that soaked in pH = 2-12 solution, 9 kinds of organic solvents, water and exposed in the air for 2 h or longer time show that their diffraction peaks are consistent with the peaks of as-synthesized samples and their single crystal data (Figure 2), indicating highly stable characteristics of **EuMOFs**.



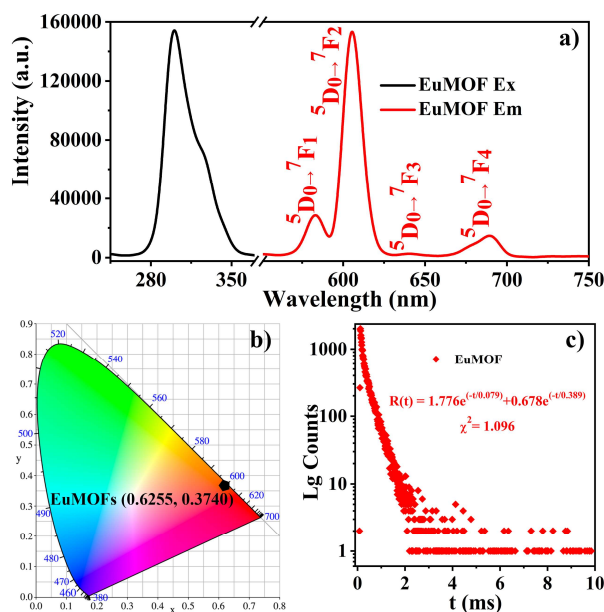
**Figure 2.** PXRD pattern of **EuMOFs** that immersed in pH = 2-12 solution, organic solvents, and soaked in water for 2 days and exposure in the atmosphere for 100 days.

### 3.2. Photophysical Studies

To study the photophysical properties of **EuMOFs**. The excitation and emission spectra, fluorescence lifetimes, and the fluorescence quantum yields of solid-state sample **EuMOFs** were measured at room temperature. As shown, wide excitation bands were observed in 210-350 nm, which were monitored at their maximum emission peak at 605 nm (Figure 3a). At the optimal excitation of 295 nm, **EuMOFs** emits four characteristic strong linear emission peaks in 550-750 nm, which are due to  $^5D_0 \rightarrow ^7F_1$ ,  $^5D_0 \rightarrow ^7F_2$ ,  $^5D_0 \rightarrow ^7F_3$ , and  $^5D_0 \rightarrow ^7F_4$  transitions at 582, 605, 641, and 689 nm, respectively (Figure 3a) [75], which is a pure red fluorescence (CIE: 0.6255, 0.3740; Figure 3b). The photofluorescence quantum yield of **EuMOFs** is a relative high value of 9.81%. The maximum emission (605 nm) of the  $^5D_0 \rightarrow ^7F_2$  transition was selected to monitor its fluorescence lifetime, which fits a double exponential function (Figure 3c) and shows a long lifetime of 0.305 ms.

The fluorescence intensities of the electric-dipole transitions  $^5D_0 \rightarrow ^7F_2$  is greater than the  $^5D_0 \rightarrow ^7F_1$  magnetic dipole transitions, indicating that  $\text{Eu}^{3+}$  is in an irreversible symmetrical position, which are in line with its single crystal structure. Single crystals of **EuMOFs** were selected and photographed

under natural light and 365 nm UV light, it was transparent (Figure S5a) and showed red fluorescence (Figure S5a'), respectively.

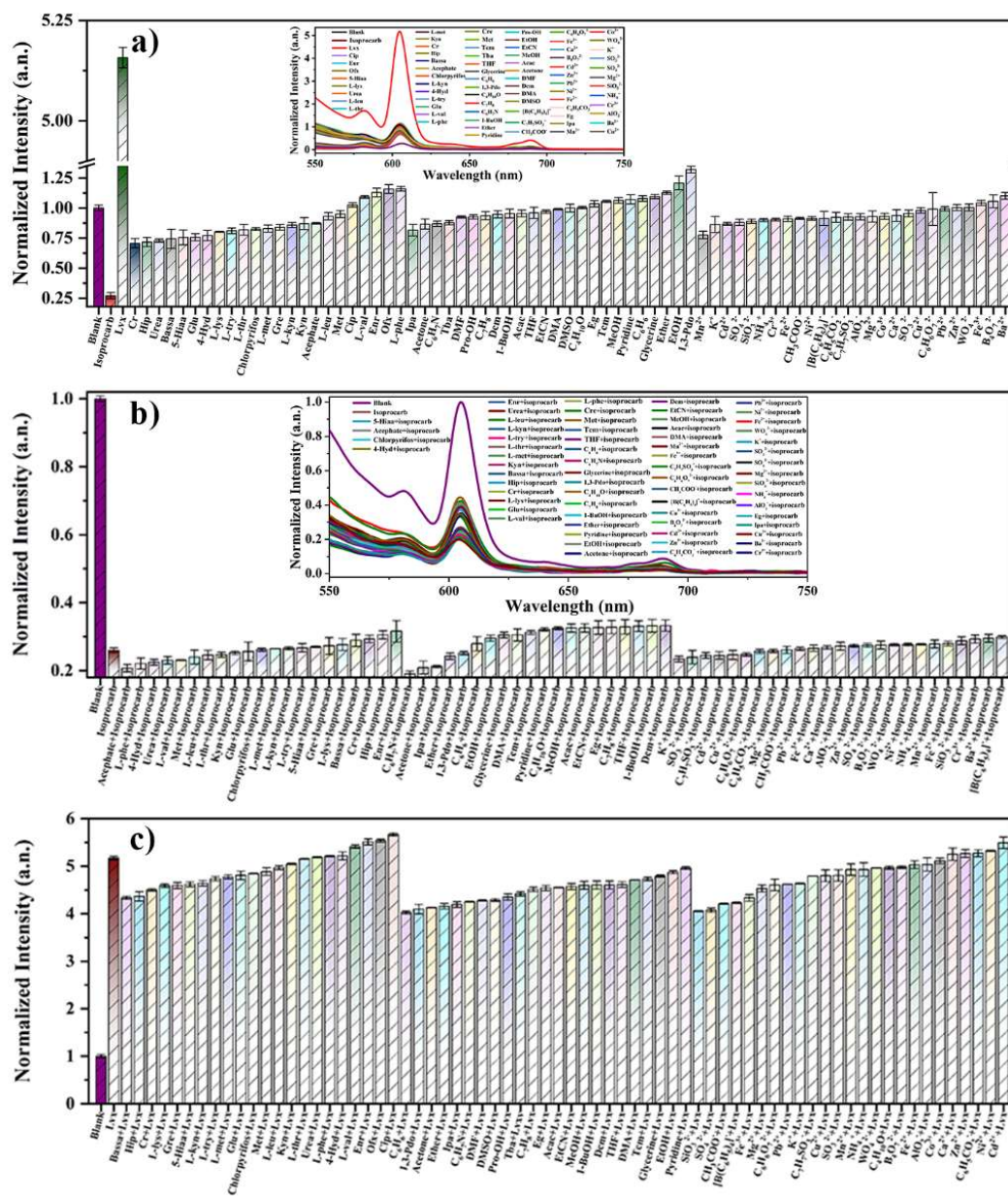


**Figure 3.** Solid-state photophysical studies for EuMOFs at room temperature: (a) excitation and emission spectra; (b) CIE coordinate graph; (c) fluorescence decays curve.

### 3.3. Sensing of Isoprocarb and Lvx

Isoprocarb is often residual in crops and foods, such as fruits and rice, which seriously endangers life and health. Lvx, a fluoroquinolone drug, is a broad-spectrum antibiotic, which is used more and more frequently, causing many diseases. Lvx has a strong ability to resist common biological decomposition, and the excessive use of Lvx is not completely metabolized in the human body and will exist in the urine. Therefore, there is an urgent need to explore ultra-sensitive and facile monitoring methods at the molecular level.

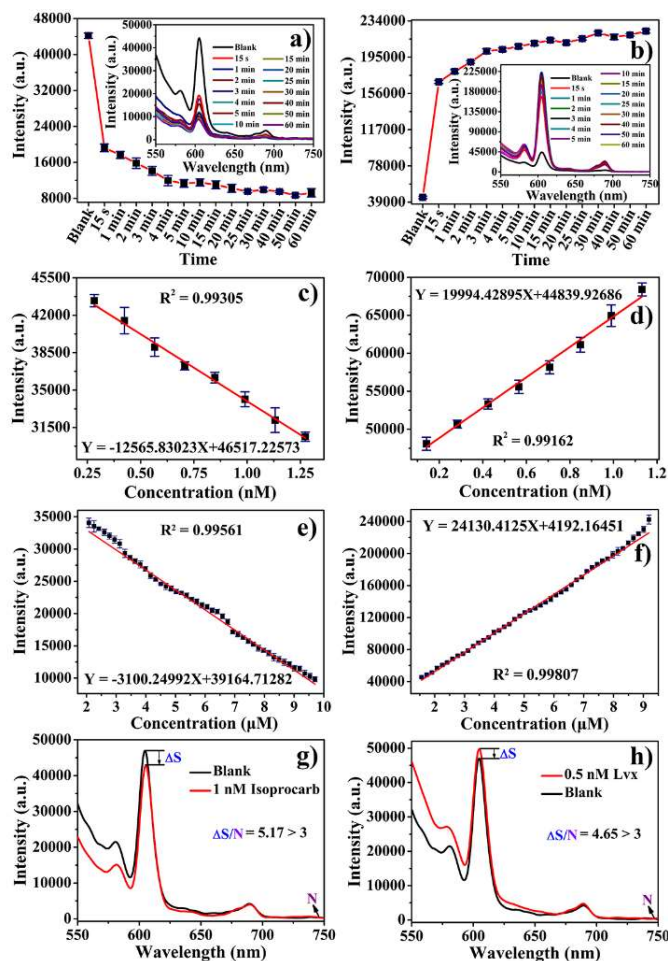
In the sensing, 15 kinds of cations of  $MCl_x$  or  $M(NO_3)_y$ , 11 kinds of anions of  $Na_mX$ , 24 kinds of organic solvents, 7 kinds of amino acids, 6 kinds of urea, 4 kinds of tumor markers, 4 kinds of floxacin drugs and 4 kinds of pesticides that listed in the supporting information (Section S1) were prepared into  $1.0 \times 10^{-3}$  M reserve solutions, respectively. 50  $\mu$ L of the above 75 substances solution was mixed with 50  $\mu$ L of  $1.0 \times 10^{-3}$  M EuMOFs reserve solution and 4.9 mL of deionized water, and then the mixed solution was placed at room temperature to react for 1.0 min. The luminescence histogram monitored at 605 nm ( $^5D_0 \rightarrow ^7F_2$  transition) shows that among the 75 substances, only isoprocarb significantly quenched the fluorescence of  $Eu^{3+}$ , Lvx significantly enhanced the fluorescence of  $Eu^{3+}$  (Figure 4a), while other species did not change the fluorescence intensity of the solution markedly, suggesting EuMOFs is a highly selective sensor for isoprocarb and Lvx.



**Figure 4.** (a) Histogram of normalized fluorescence intensities at 605 nm of **EuMOFs** reacting with 75 substances, insert: full spectra; (b) histogram of fluorescence intensities of **EuMOFs** reacted to isoprocarb at the presence of 10 equiv. anti-interference species, insert: full spectra; (c) histogram of fluorescence intensities of **EuMOFs** reacted to Lvx at the presence of 10 equiv. anti-interference species. .

In real environments, the sensing results susceptible to be disturbed by other substances. To investigate whether the detection of isoprocarb and Lvx would be interfered by other substances, the competition experiment was performed. 4.4 mL deionized water was mixed with 50  $\mu\text{L}$  **EuMOFs** stock solution ( $1.0 \times 10^{-3}$  M), 50  $\mu\text{L}$  isoprocarb or Lvx solution ( $1.0 \times 10^{-3}$  M,  $\text{H}_2\text{O}$ ) and 500  $\mu\text{L}$  competitive substance ( $1.0 \times 10^{-3}$  M  $\text{H}_2\text{O}$ ). The fluorescence measurement of **EuMOFs** reacting with isoprocarb or Lvx in the presence of competing substances were determined by the above method. It shows that isoprocarb shows obvious fluorescence quenching at the presence of 10 equiv. anti-interference substances (Section S2, Figure 4b), Lvx shows obvious fluorescence enhancing in the presence of 10 equiv. anti-interference substances (Section S3, Figure 4c, Figure S6), revealing high anti-interference of the sensor.

In addition, the reaction time of **EuMOFs** towards isoprocarb and Lvx were evaluated. The fluorescence intensity was monitored at 605 nm ( ${}^5D_0 \rightarrow {}^7F_2$  transition), the dotted line shows that **EuMOFs** sensing isoprocarb (Figure 5a) and Lvx (Figure 5b) show fast response time of 15 s, and the sensing signal is stable within 1.0 hour, suggesting that **EuMOFs** is a highly stable sensor. In order to further understand the sensing characteristics of **EuMOFs**, the relationships between the fluorescence intensity of the sensor and the concentration of isoprocarb and Lvx were studied. It was found that the fluorescence intensity (605 nm) had excellent linear relationships with the concentration of isoprocarb and Lvx in 0.01-1.6 nM, which have linear equations of  $Y = -12565.83023X + 46517.22573$ ,  $R^2 = 0.99305$  for isoprocarb (Figure 5c), and  $Y = 19994.42895X + 44839.92686$ ,  $R^2 = 0.99162$  for Lvx (Figure 5d). In a wide concentration range of 0.01-10.0  $\mu\text{M}$  isoprocarb and Lvx concentration can be determined by fluorescence measurement as well, which satisfies the equation of  $Y = -3100.24992X + 39164.71282$ ,  $R^2 = 0.99561$  for isoprocarb (Figure 5e),  $Y = 24130.4125X + 4192.16451$ ,  $R^2 = 0.99807$  for Lvx (Figure 5f). When  $1.0 \times 10^{-9}$  M isoprocarb reacts with  $1.0 \times 10^{-8}$  M **EuMOFs**, the ratio of signal to noise (S/N) is 5.17 (Figure 5g), revealing its LOD is as low as 1.0 nM, which is lower than the LOD value of the national standard (GB 28260-2011 for isoprocarb is 2.583  $\mu\text{M}$ ), indicating that **EuMOFs** is a highly sensitive probe for isoprocarb. When  $5.0 \times 10^{-10}$  M Lvx reacted with  $1.0 \times 10^{-9}$  M **EuMOFs**, the ratio of signal to noise (S/N) was 4.65, revealing its LOD is as low as 0.5 nM (Lv, Figure 5h), which is lower than the LOD value of some other sensitive fluorescent probes (Table 1), indicating that **EuMOFs** is a highly sensitive probe for Lv. The recovery rate of added samples in the sensing of isoprocarb and Lv is between 97.86%-101.48% under various conditions in Table 2.



**Figure 5.** The dot plot of the fluorescence intensity at 605 nm of **EuMOFs** reacted with isoprocarb (a) and Lv (b) at various reaction time, insert: full emission profiles; the linearity of fluorescence intensity versus the

concentration of isoprocarb (c) and Lvx (d) that monitored at 605 nm; plot of fluorescence intensity (605 nm) of **EuMOFs** reacting with 0.01-10.0  $\mu\text{M}$  isoprocarb (e) and Lvx (f); LOD measurement for sensing isoprocarb (g) and Lvx (h).

**Table 1.** LOD and linear range comparison of different methods for isoprocarb and Lvx detection.

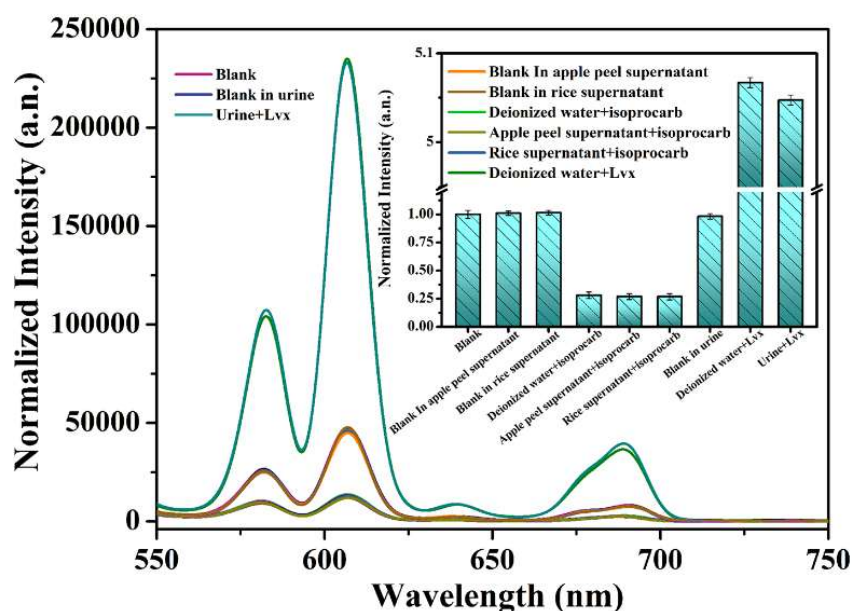
Method	Analyte	Linear range (nM)	LOD (nM)	References
DPV	Isoprocarb	100-100000	79	[76]
Amperometry	Isoprocarb	100-150000	8	[3]
Chronoamperometry	Isoprocarb	5-1000	160	[4]
Luminescent detection	Isoprocarb	300-5200	260	[1]
UV-Vis detector	Isoprocarb	1000-50000	300	[5]
Differential Pulse Voltammetry	Lvx	5000-1000000	1800	[7]
Luminescent detection	Lvx	5000-150000	850	[2]
Luminescent detection	Lvx	53-110000	16	[77]
Luminescent detection	Lvx	2500-5000	1260	[6]
Luminescent detection	Lvx	100-400	20	[78]
<b>EuMOFs</b>	Isoprocarb	0-9000	1	<b>This work</b>
<b>EuMOFs</b>	Lvx	0-10000	0.5	<b>This work</b>

**Table 2.** Recovery results for the determination of isoprocarb and Lvx in water, apple peel supernatant, rice supernatant, and Urine by **EuMOFs**.

Sample	Analyte	Added ( $\mu\text{M}$ )	<b>EuMOFs Recovery (%)</b>	Theoretical value ( $\mu\text{M}$ )
Water	Isoprocarb	2.253	98.35	2.424
		3.813	99.85	3.826
		5.372	100.32	5.349
		6.932	98.38	7.024
		8.492	99.45	8.515
Apple peel supernatant	Isoprocarb	2.253	100.12	2.239
		3.813	99.58	3.852
		5.372	99.28	5.428
		6.932	100.37	6.909
		8.492	99.39	8.520
Rice supernatant	Isoprocarb	2.253	100.87	2.174
		3.813	99.12	3.879
		5.372	100.78	5.325
		6.932	99.89	6.937
		8.492	100.57	8.476
		2.079	99.16	2.060
		3.640	101.48	3.696

Water	LvX	5.199	98.65	5.126
		6.759	100.38	6.785
		8.318	97.86	8.136
		2.079	99.61	2.067
		3.640	101.25	3.698
Urine	LvX	5.199	99.92	5.194
		6.759	100.66	6.810
		8.318	99.57	8.278

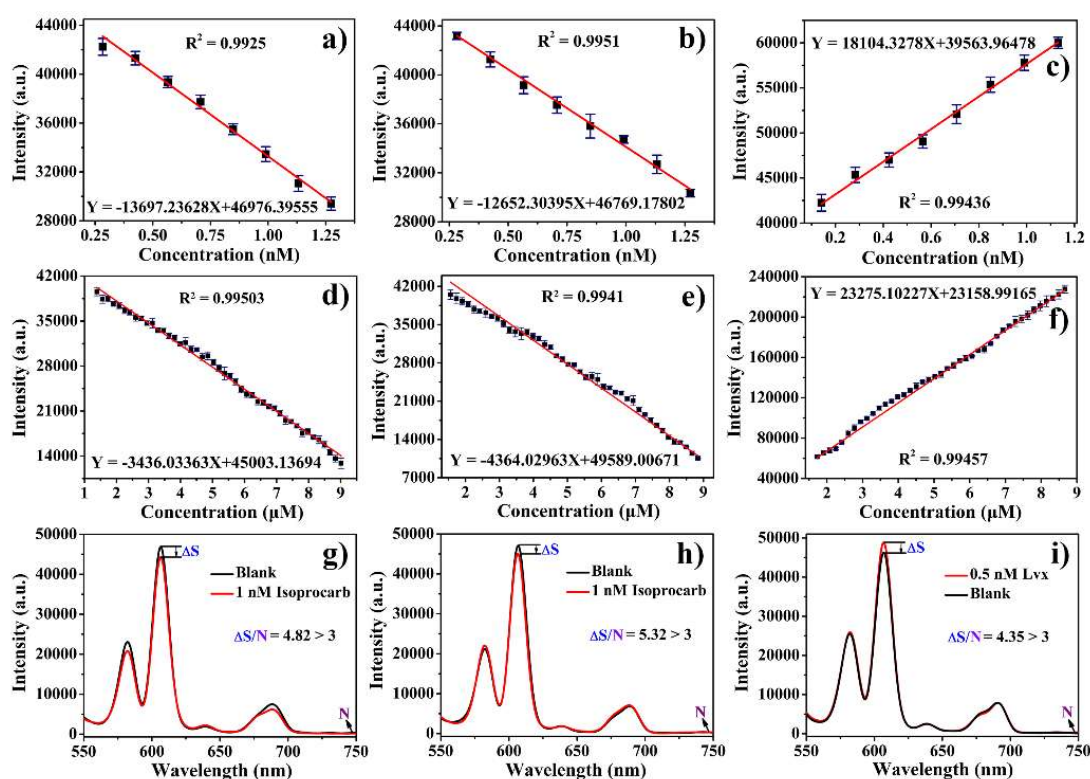
In addition, whether **EuMOFs** can be used as a luminescent probe for the detection of isoproc carb in actual sample of apple peel (Section S4) and rice supernatant (Section S5), and for the probe of LvX in urine (Section S6) were further investigated. 4.9 mL apple peel or rice supernatant mixed with 50  $\mu\text{L}$  **EuMOFs** solution ( $1.0 \times 10^{-3}$  M, DMF) and 50  $\mu\text{L}$   $1.0 \times 10^{-3}$  M isoproc carb, respectively. 4.9 mL urine solution mixed with 50  $\mu\text{L}$  stock **EuMOFs** solution ( $1.0 \times 10^{-3}$  M, DMF) and 50  $\mu\text{L}$   $1.0 \times 10^{-3}$  M LvX, respectively. It shows that **EuMOFs** reacted with isoproc carb in apple peel supernatant, rice supernatant showed obvious fluorescence quenching for isoproc carb, and LvX in urine showed obvious fluorescence quenching, confirming that **EuMOFs** has great potential application in the detection of isoproc carb in real samples of apple peel supernatant, rice supernatant, and LvX in real urine (Figure 6).



**Figure 6.** Full spectrum of fluorescence of **EuMOFs** reacted with isoproc carb and LvX in real samples of apple peel supernatant, rice supernatant, and in urine, insert: fluorescence histogram.

It is found that the fluorescence intensity (605 nm) has excellent linear relationships with isoproc carb concentration in 0.01-1.6 nM in the real samples of apple peel supernatant, rice supernatant, and has excellent linear relationships with LvX concentration in urine, which have linear equations of  $Y = -13697.23628X + 46976.39555$ ,  $R^2 = 0.9925$  for isoproc carb in apple peel supernatant (Figure 7a),  $Y = -12652.30395X + 46769.17802$ ,  $R^2 = 0.9951$  for isoproc carb in rice supernatant (Figure 7b), and  $Y = 18104.3278X + 39563.96478$ ,  $R^2 = 0.99436$  for LvX in urine (Figure 7c). In a wide concentration range of 0.01-9.0  $\mu\text{M}$  in apple peel and rice preparation solution, isoproc carb concentration can be determined by fluorescence measurement as well, which satisfies the equation of  $Y = -3436.03363X + 45003.13694$ ,  $R^2 = 0.99503$  in apple peel supernatant (Figure 7d) and  $Y =$

4364.02963X+49589.00671,  $R^2=0.9941$  in rice supernatant (Figure 7e). In the concentration range of 0.01-9.0  $\mu\text{M}$  in urine, Lvx concentration can be determined by fluorescence measurement as well, which satisfies the equation of  $Y=23275.10227X+23158.99165$ ,  $R^2=0.99457$  (Figure 7f). When  $1.0 \times 10^{-9}$  M isoprocab reacted with  $1.0 \times 10^{-8}$  M EuMOFs in apple peel and rice supernatant respectively, the ratio of signal to noise (S/N) is 4.82 and 5.32, respectively, revealing its LOD is as low as 1.0 nM (Figure 7g, 7h), which is lower than the LOD value of the national standard (GB 28260-2011, isoprocab 2.583  $\mu\text{M}$ ), indicating that EuMOFs is a highly sensitive probe for isoprocab in apple peel and rice supernatant. And when  $5.0 \times 10^{-10}$  M Lvx reacts with  $1.0 \times 10^{-9}$  M EuMOFs in urine, the ratio of signal to noise (S/N) is 4.35, suggesting its LOD is as low as 0.5 nM (Figure 7i), which is lower than the LOD value of other fluorescent probes in Table 2, indicating EuMOFs is a highly sensitive probe for Lvx in real sample. The recovery rates of isoprocab and Lvx the above in real samples were between 99.12%-101.25% (Table 3), further confirmed the reliability and practicability of the sensing method of this work.



**Figure 7.** Plot of fluorescence intensity (605 nm) of EuMOFs reacting with isoprocab in real samples of apple peel supernatant (a), rice supernatant (b) and with Lvx in real samples of urine (c); the linearity of fluorescence intensity versus the concentration of 0.01-9.0  $\mu\text{M}$  isoprocab in apple peel supernatant (d), in rice supernatant (e) and 0.01-9.0  $\mu\text{M}$  Lvx in urine (f) at 605 nm; LOD measurement for sensing isoprocab in real samples of apple peel supernatant (g), rice supernatant (h) and Lvx in real samples of urine (i).

The results of sensing is superior than that reported methods listed in Table 1. Compared with other detection methods, the sensor developed in this work has the advantages of simple preparation, low LOD value, high sensitivity and wide linear range.

**Table 3.** Recovery results for the determination of isoprocab and Lvx in water, apple peel supernatant, rice supernatant, and Urine by EuMOFs.

Sample	Analyte	Added ( $\mu\text{M}$ )	EuMOFs Recovery (%)	Theoretical value ( $\mu\text{M}$ )
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		3.813	99.12	3.879
		5.372	100.78	5.325
		6.932	99.89	6.937
		8.492	100.57	8.476
Water	Lvx	2.079	99.16	2.060
		3.640	101.48	3.696
		5.199	98.65	5.126
		6.759	100.38	6.785
		8.318	97.86	8.136
Urine	Lvx	2.079	99.61	2.067
		3.640	101.25	3.698
		5.199	99.92	5.194
		6.759	100.66	6.810
		8.318	99.57	8.278

### 3.4. Sensing Mechanism

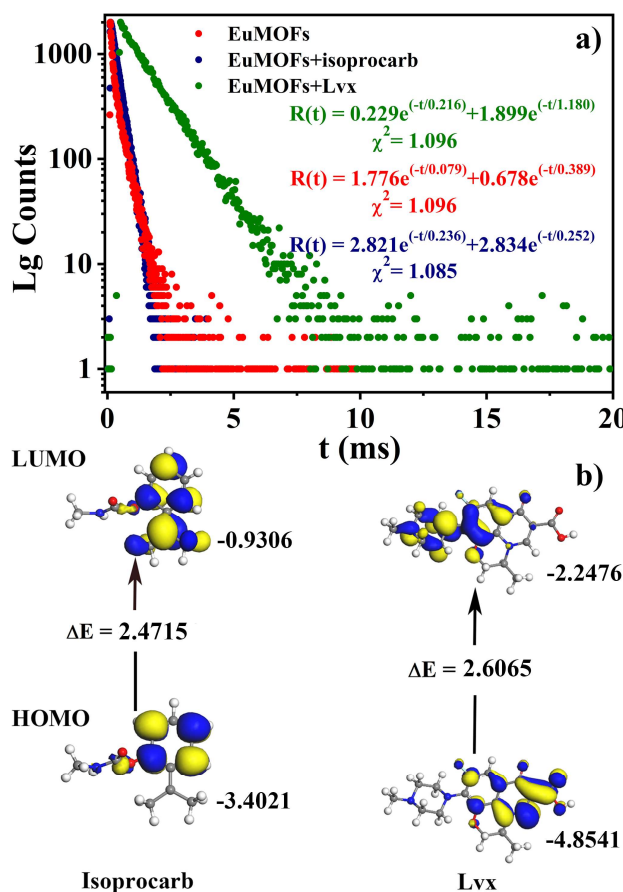
EuMOFs that reacted with  $1.0 \times 10^{-5}$  M isoprocab and Lvx were washed by  $3 \times 5.0$  mL water and measured by PXRD. The results confirmed the samples were in line with as-synthesized EuMOFs, and no peaks in line with isoprocab or Lvx appeared, confirming the structure of EuMOFs remain integrity after sensing (Figure S7). The fluorescence lifetimes of EuMOFs blank solution ( $1.0 \times 10^{-5}$  M) and EuMOFs solution ( $1.0 \times 10^{-5}$  M) reacted with isoprocab ( $1.0 \times 10^{-5}$  M) or Lvx ( $1.0 \times 10^{-5}$  M) are 0.281, 0.242, and 1.862 ms (Figure 8a), the photoluminescence quantum yields are 7.52%, 6.86% and 13.54%, respectively, revealing the lifetimes of the sensing samples for isoprocab are shorter than blank solution and for Lvx are longer than blank solution, and their fluorescence quantum yield are in line with their lifetimes, revealing the sensing of isoprocab is a dynamic quenching process and Lvx is a dynamic enhancing process.

The LUMO level of Lvx (-2.2476) is smaller than the LUMO level of isoprocab (-0.9306) (Figure 8b), and the LUMO level of Lvx is closer to the energy level of  $\text{Eu}^{3+}$ , which is easier to transfer energy

to  $\text{Eu}^{3+}$ , so the fluorescence is enhanced. The LUMO energy level of isoprocarb to  $\text{Eu}^{3+}$  is more suitable than  $\text{Eu}^{3+}$  than Lvx, so the fluorescence of sensing isoprocarb is decreased [79].

**Table 4.** HOMO and LUMO Energies for the isoprocarb, Lvx and  $\text{Eu}^{3+}$ .

Analytes	HOMO (eV)	LUMO (eV)	Band gap (eV)
Isoprocarb	-0.9306	-3.4021	2.4715
Lvx	-2.2476	-4.8541	2.6065



**Figure 8.** (a) Fluorescence lifetime decay curves of samples **EuMOFs**, **EuMOFs+isoprocarb** and **EuMOFs+Lvx**; (b) HOMO and LUMO of isoprocarb and Lvx.

#### 4. Conclusions

In summary, a porous lanthanide MOFs of **EuMOFs** has been obtained and their fluorescence properties have been studied in detail. Interestingly, **EuMOFs** was a bi-functional sensor for isoprocarb and Lvx, which showed excellent anti-interference ability, rapid response of 15 s, excellent linear relationship and wide linear range, LODs as low as 1.0 nM and 0.5 nM, respectively. In addition, **EuMOFs** has also been successfully applied to the detection of isoprocarb in apple peel and rice supernatant, and sensing of Lvx in real urine, which still has high selectivity for isoprocarb and Lvx in actual samples, excellent anti-interference ability, and excellent detection stability in the linear range of 0.01~9.0 nM. The recoveries for the two species sensing were in 99.12%~101.25%. The LOD for isoprocarb detection in apple peel and rice supernatant was as low as 1.0 nM, and for Lvx detection in urine was as low as 0.5 nM, with excellent reproducibility and recovery. The sensor has

a higher sensitivity for isoprocarb and LvX than previously reported fluorescence sensors or maximum residue limit levels (GB 28260-2011, isoprocarb 2.583  $\mu\text{M}$ ). More interestingly, it allows specific detection of isoprocarb residues in crop food samples and LvX in human urine. Based on the investigation of fluorescence lifetime and DFT, the sensing mechanisms were investigated. As an efficient pesticide response substance, EuMOFs provides a new detection platform for isoprocarb residues in crops, and has great potential application value in food safety and human health. The highly sensitive and quickly detection of LvX in human urine shows extraordinary significance for human health.

**Supplementary Materials:** The following supporting information can be downloaded at the website of this paper posted on Preprints.org.

**Author Contributions:** The work presented here was carried out as a collaboration among all authors. Conceptualization, methodology, and formal analysis, Y.Y., C.H., and C.Z.; writing—original draft preparation, Y.Y., N.S., and C.H.; writing—review and editing, N.S. and C.Z.; funding acquisition, C.Z. All authors have read and agreed to the published version of the manuscript.

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