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RESEARCH ARTICLE

CHARACTERIZATION OF FE3+ SELECTIVE FLUORESCENCE PROBES BASED ON PYRENE DERIVATIVES

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ABSTRACT

A Fe^{3+}-selective fluorescent probe derived from pyrene derivatives was synthesized and characterized. The fluorescence intensity of the probe at 397 nm was enhanced with the addition of Fe^{3+}. In the range of 1-9μM, there was an obvious linear correlation between the fluorescent intensity and the concentration of Fe^{3+}. The UV-vis spectra also indicated that the binding of between the probe and Fe^{3+}.

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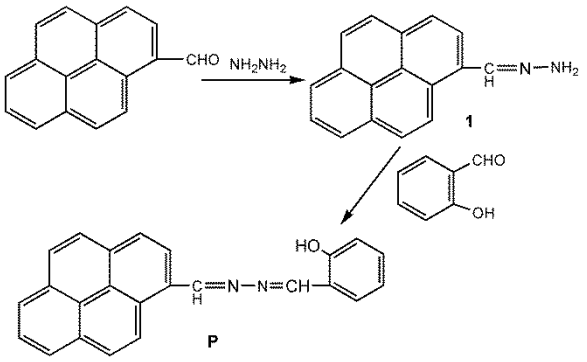
INTRODUCTION

Metal ions, as indispensable substances in human survival, development, and life activities, are essential trace elements for the human body. When excessive metal ions enter the human body through respiratory and dietary pathways, they can cause harm to the human body. At the same time, their enrichment in the environment can also cause environmental pollution and ecological damage (Zhuang, 2021). Among them, Fe^{3+} is one of the important components of hemoglobin. Low Fe^{3+} content can lead to the inability of red blood cells in the bone marrow to synthesize hemoglobin and myoglobin normally, reduce enzyme activity, and reduce the body's immunity, intelligence, and anti infection ability, making it prone to diseases such as iron deficiency anemia and immune deficiency (Lang, 2023); Excessive Fe^{3+} can lead to diseases such as osteoporosis and tissue and organ dysfunction (Wang, 2022). Zn^{2+} is an essential trace element in the human body, second only to Fe^{3+} in content. Zn^{2+} deficiency can lead to developmental delay and neurological diseases; Excessive consumption can lead to a series of physiological dysfunction in the body's immune system (Kowada, 2020 and Mondal, 2019). The presence of a large number of metal ions in the environment can cause serious pollution, so an efficient and sensitive method is needed to detect metal ions in the environment and organisms. These methods need to be easy to operate and can provide fast and accurate results. Fluorescence probe detection method has excellent characteristics such as wide source, easy modification, high sensitivity, high selectivity, convenient operation, and fast response, and has achieved high popularity in detecting metal ions in the environment and ecology (Li, 2003; Makarishcheva, 2022; Parts, 2021; Nugraha, 2020; Nie, 2018; Kashyap, 2019; Sun, 2018 and Sun, 2023). Developing and exploring fluorescent probes with better performance has gradually become a current research hotspot, therefore designing and synthesizing a fluorescent probe with high selectivity and sensitivity in aqueous solution is of great significance. Based on the excellent performance and wide application prospect of pyrene derivatives (Yeldir, 2022; Gou, 2022 and Yu, 2022), this paper showed a study in which pyrene formaldehyde and hydrazine hydrate were used to synthesize a fluorescent probe which displayed selectivity for Fe^{3+}, and the detailed study was also carried out in this paper.

EXPERIMENTAL SECTION

Reagents and Instruments: Anhydrous ethanol, pyrene formaldehyde, 98% salicylaldehyde, 85% hydrazine hydrate dimethyl sulfoxide, disodium ethylenediaminetetraacetate (EDTA), 4-hydroxyethyl piperazine ethanesulfonic acid (HEPES), Ethyl acetate. Before using the reagents, no special treatment was performed. All reagents were analytical pure. UV-vis spectra were carried on a Hitachi U-2910 spectrophotometric. Fluorescent spectra were recorded using a Hitachi F-4600spectrofluometer.

Synthesis of P: Synthesis route of P was shown in Scheme 1.



Scheme 1. Synthesis route of P

Synthesis of compound 1: In a 250 mL round bottom flask, 0.40 g of pyrene formaldehyde and 16 mL of hydrazine hydrate, an appropriate amount of 50 mL anhydrous ethanol were added. The reaction was heated and refluxed for 4 h, and then cooled to room temperature. The yellow solid product obtained by suction filtration and storeed from light. Synthesis of compound P: 80 mg of compound, 145 μL salicylaldehyde (slightly excessive) and an appropriateamount of 50 mL anhydrous ethanol were added into a round bottomed flask. The reaction was heated and refluxed for 6 h, and then cooled to room temperature. Yellow solid P obtained by suction filtration and storeed from light.

General Spectroscopic Methods: The stock solutions of P and metal ions (1.0 mM) were obtained by dissolving salts and P in deionized water and dimethylsulfoxide, respectively, and the testing solutions was freshly prepared before measurements by diluting the stock solutions.

RESULTS AND DISCUSSION

Selectivity Measurement: Fluorescent method was used for the selectivity measurement of the proposed P. Among the tested metal ions, such asHg^{2+}, K^{+}, Na^{+}, Ag^{+}, Mg^{2+}, Ca^{2+}, Cd^{2+}, Cu^{2+}, Co^{2+},Zn^{2+}, Pb^{2+},Fe^{3+},Ni^{2+}, Cu^{2+}, Al^{3+} andCr^{3+}. A specific fluorescence peak at 397 nm appeared with the addition of Fe^{3+}, after the addition of Zn^{2+}, the fluorescence peak showed an obvious redshift with peak appeared at 522 nm, Adding Cu^{2+}produced bimodal emission, and the fluorescence peak appeared at 397 nm and 450 nm, which was significantly different from other metal ions. This study focused on the performance of P in identifing Fe^{3+}.

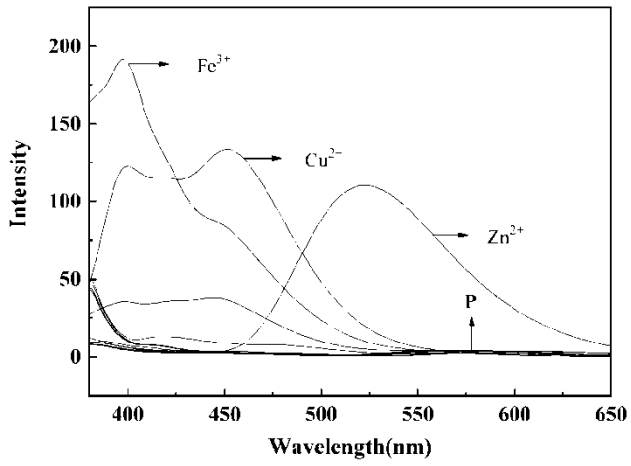


Fig. 1. Fluorescence spectra of P (10 μM) with different metal ions (100 μM) in ethanol

Titration experiment of P with Fe^{3+}: Fluorescence and UV-vis titration experiments were used to investigate the sensitivity of P to Fe^{3+}(Fig. 2 and Fig. 3). The results showed that with the increase of Fe^{3+}, the fluorescence intensity at 397 nm increased, showing a linear relationship in the range of 1 to 9 μM. The influence of different concentrations of Fe^{3+} on the absorption spectra of the probe P was also examined. It was found that isoelectric points appeared at 380 nm with the change of Fe^{3+} concentration. The absorbance at 285 nm enhanced with the increase of Fe^{3+} concentration.

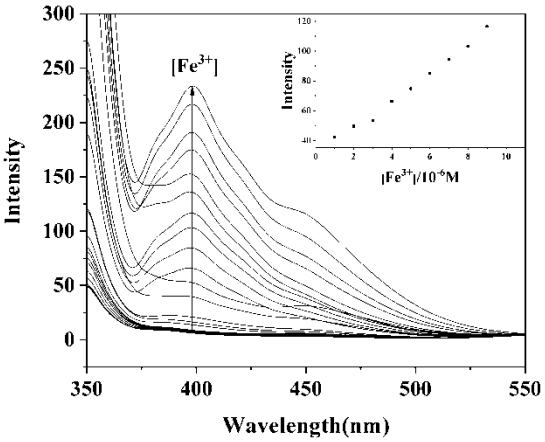
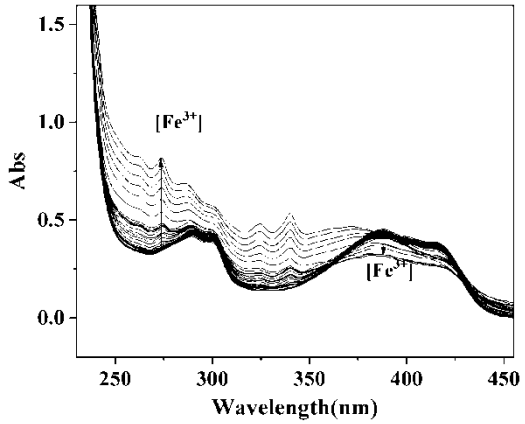


Fig. 2. Fluorescence response of P (10 μM) with various concentrations of Fe^{3+} (0.1-90.0 μM) in ethanol. Inset: linear relationship diagram of probe P and Fe^{3+}



Competitive effects of Fe^{3+} in the presence of metal ions: Further investigation was conducted on the competitive effects of common metal ions coexisting in P-Fe^{3+} system. The results were shown in Figure 4. Under the same conditions, the presence of Ca^{2+}, Na^{+}, K^{+}, Cr^{2+}, and Zn^{2+} plasma can all affect the fluorescence intensity of the probe to varying degrees. Therefore, it was determined that probe P still needed some improvement, laying the foundation for subsequent experimental analysis.

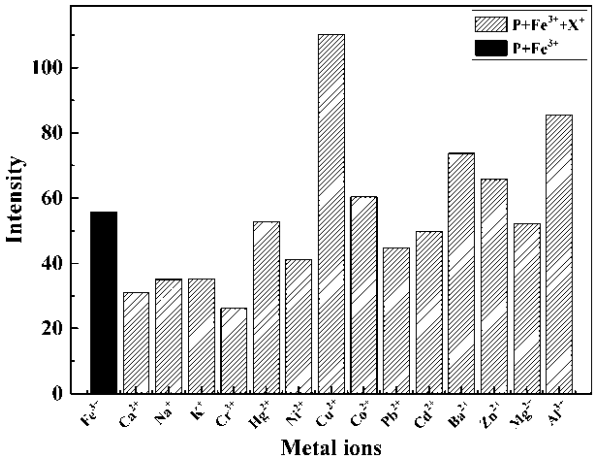


Fig. 4. Spectral response of probe P (10 μM) for identifying Fe^{3+} (10 μM) in the presence of different metal cation (50 μM) in ethanol

Reversible binding of P-Fe^{3+} system: As shown in Fig. 5, the reversibility of P-Fe^{3+} system was investigated. Only probe P had the little fluorescence intensity at 397 nm (Fig. 5a). After Fe^{3+} was added, the fluorescence intensity increased (Fig. 5b). After the addition of EDTA, the fluorescence intensity of the system was reduced due to the competition effect (Fig. 5c-d). When excessive Fe^{3+} was added to combine with the probe, the structural changes were induced and the fluorescence intensity at 397 nm was recovered and even enhanced (Fig. 5e-f), which proved that the probe had certain reversibility.

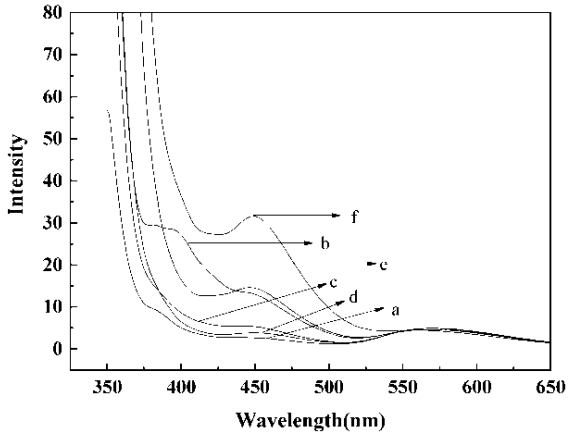


Fig. 5. Reversibility of P binding to Fe^{3+} inethanol: a. P (10 μM); b. P (10 μM) + Fe^{3+}(10 μM); c. P (10 μM) + Fe^{3+}(10 μM) + EDTA (10 μM); d. P (10 μM) + Fe^{3+}(10 μM) + EDTA (50 μM); e. P (10 μM) + Fe^{3+}(10 μM) + EDTA (50 μM) + Fe^{3+}(50 μM); f. P (10 μM) + Fe^{3+}(10 μM) + EDTA (50 μM) + Fe^{3+}(100 μM).

CONCLUSIONS

In summary, it can be seen that the probe had a clear recognition of Fe^{3+} at 397 nm. As the concentration of Fe^{3+} increased within the range of 1-9 μM, the fluorescence intensity at 397nm also increased linearly with a correlation coefficient of R^2=0.991. It was judged that probe P had certain anti-interference ability from other metal ions, and still needed to be improved for the recognition and detection of Fe^{3+} in the environment.

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