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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF Al^{3+} -SELECTIVE FLUORESCENT PROBE BASED ON PYRENE DERIVATIVE

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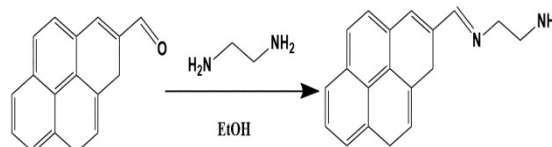
ABSTRACT

In this work, a pyrene derivative P was synthesized and characterized as an Al^{3+} -selective fluorescent probe. The addition of Al^{3+} to the solution of P in ethanol caused an obvious fluorescent enhancement at 399 nm.

INTRODUCTION

As a non-essential element of human body, Al is one of the most abundant metals on the earth (Gao, 2019). As an alloy component, Al has favorable properties such as light weight, corrosion resistance, high strength and high ductility (Kaur, 2020), and is widely used in modern life in the manufacturing of cookware, water purification, conductive materials, building materials, textile, paper making and pharmaceutical industry (Anu, 2021; Gul, 2020; Kshirsagar, 2020; Tian, 2019). But too much Al is harmful to humans and other organisms (Dhineshkumar, 2020; Yin, 2022; Liang, 2019; Ali, 2022). Therefore, timely detection of Al^{3+} concentrations in the environment and human body is of great significance. Many methods for the detection of Al^{3+} have been studied. Traditional methods include atomic fluorescence spectrometry, atomic absorption spectrometry, inductively coupled plasma, *et al.* (2016). However, these methods have many disadvantages, such as complicated sample preparation, expensive instruments, and long time consumption. In contrast, fluorescence probe method solves the disadvantages of traditional analysis methods, and has the advantages of high sensitivity, low cost, simple operation and short response time (Zhang, 2022; Wu, 2020, Das, 2021). Among many fluorescent probe luminescent groups, pyrene and its derivatives, as a typical dense aromatic hydrocarbon, have a relatively simple structure and are connected by four benzene rings. Because of its unique fluorescence, stable chemical properties, long fluorescence lifetime and high quantum yield, pyrene derivatives have a good prospect for the development of fluorophore synthesis

probes (Long, 2019, Chao, 2021, Yu, 2022). In summary, this study intends to construct Schiff base structure with pyrene-1-formaldehyde as the central structural unit, and design and synthesize fluorescent probes with specific recognition of Al^{3+} (Scheme 1).



Scheme 1. The synthesis route of P

EXPERIMENTAL SECTION

Synthesis of probe P: Under reflux, 0.4998 g (2.1mmol) of pyrene-1-carbaldehyde was added to a 100mL three necked bottle containing 0.7 mL (10.5 mmol) of ethylenediamine and 30 mL of ethanol, the mixture reacted for 6 h and then cooled to room temperature. The white powdery solid (0.3384 g) so obtained was filtered off and used directly.

Test conditions: The excitation wavelength of fluorescence measurement was 340 nm, the slits of excitation and emission wavelength were 10 nm, respectively. The concentration of probe P

was 10 μM in DMSO. The concentration of metal ions was 100 μM and the test medium was ethanol.

RESULTS AND DISCUSSION

Selectivity measurement: The probe P (10 μM) and different 100 μM metal ions (K^+ , Ca^{2+} , Na^+ , Mg^{2+} , Al^{3+} , Zn^{2+} , Ni^{2+} , Ba^{2+} , Pb^{2+} , Cu^{2+} , Hg^{2+} , Ag^+ , Cd^{2+} and Co^{2+}) were added to the ethanol (Figure 1). As it can be seen that the fluorescence intensity at 399 nm was significantly greater when probe P was combined with Al^{3+} than that of probe P bounded to other tested metal ions. Therefore, we can conclude that probe P was an Al^{3+} selective fluorescent probe.

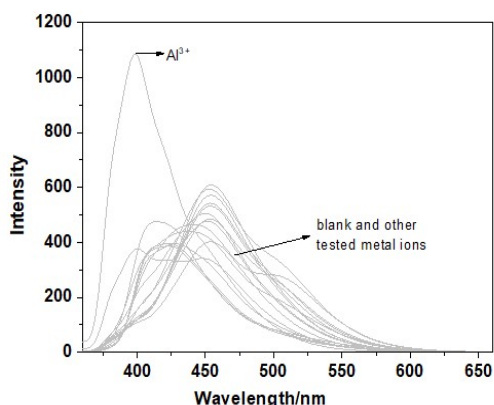


Figure 1. Selectivity test of probe P (10 μM) for different metal ions (100 μM) in ethanol

Sensitivity measurement: For probe P, not only qualitative analysis but also quantitative analysis should be carried out to clarify its detection range for Al^{3+} . Therefore, the fluorescence intensity of probe P (10 μM) was determined when there were different concentrations of Al^{3+} . It can be seen from the experimental data that the fluorescence intensity at 399 nm, 425 nm and 452 nm changed regularly with the increase of Al^{3+} , and when the concentration of Al^{3+} was between 0.05 and 0.1 μM , the system had a linear relationship at 425 nm with the correlation coefficient $R^2=0.9965$, and the linear equation was $F/F_0=0.9527+1.4599 [Al^{3+}]$; When the concentration of Al^{3+} was in the range of 0.4 ~ 9.2 μM , the system had a linear relationship at 452 nm with the correlation coefficient $R^2=0.9976$, and the linear equation was $F/F_0=1.2014+0.0618 [Al^{3+}]$. When the concentration of Al^{3+} was in the range of 9.1~10 μM , the system had a linear relationship at 399 nm with the correlation coefficient $R^2=0.9973$, and the linear equation was $F/F_0=0.9375 [Al^{3+}]-7.8423$.

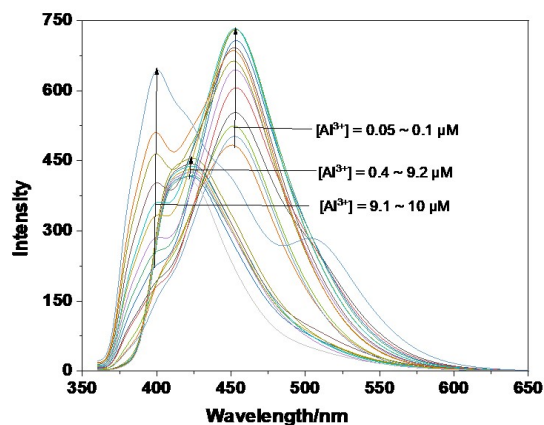


Figure 2. Fluorescence response of P (10 μM) with various concentration of Al^{3+} (0-10 μM) in ethanol

Competition: When probe P (10 μM) recognized Al^{3+} (100 μM), other concomitant metal ions had little effect on the signal (Figure 3). Therefore, probe P still displayed a good selectivity for Al^{3+} , which meant that the probe can be used in complex media, which laid a foundation for the practical application in the later stage.

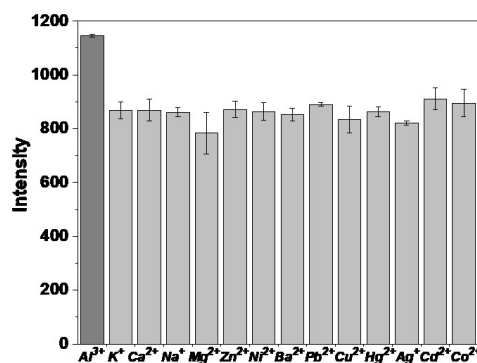


Figure 3. Spectral response of 10 μM probe P to 100 μM Al^{3+} upon addition of other metal ions (100 μM) in ethanol

Binding mode: According to Job's plot, the total concentration of P and Al^{3+} was guaranteed to be 10 μM . The experimental results showed that when the concentration ratio of $[P]/[Al^{3+}]$ was 1:1, the fluorescence intensity at 399 nm was the highest, which indicated that the formation of 1:1 stoichiometry between P and Al^{3+} (Figure 4).

Reversibility: To understand the stability of the formed complex, the chemical reversibility behavior of the binding of P and Al^{3+} in the ethanol was analyzed (Figure 5). According to the experimental data, when adding 10 μM Al^{3+} , the fluorescence intensity at 399 nm was enhanced obviously. And when EDTA was added, the fluorescence peak was shifted and the more EDTA was added, the more the fluorescence intensity was reduced. The reason may be that EDTA can combine with Al^{3+} to form a chelate compound. It can also be seen that when Al^{3+} was added again, fluorescence intensity at 399 nm was recovered. Therefore, we can assume that the reversibility of the probe P was satisfactory.

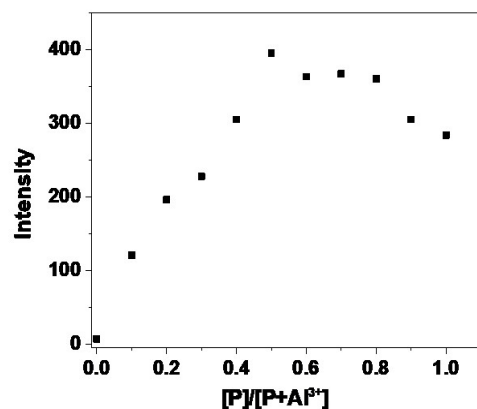


Figure 4. Determination of binding ratio of P- Al^{3+} by Job's plot method

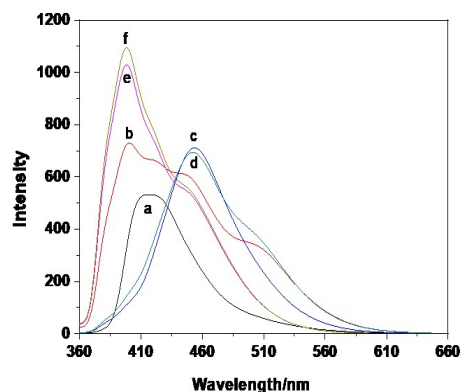


Figure 5. The reversibility of probe P in ethanol. a: 10 μM P; b: 10 μM P + 10 μM Al^{3+} ; c: 10 μM P + 10 μM Al^{3+} + 10 μM EDTA; d: 10 μM P + 10 μM Al^{3+} + 50 μM EDTA; e: 10 μM P + 10 μM Al^{3+} + 50 μM EDTA + 50 μM Al^{3+} ; f: 10 μM P + 10 μM Al^{3+} + 50 μM EDTA + 100 μM Al^{3+}

CONCLUSION

An Al³⁺-selective fluorescent probe was successfully constructed, which displayed good selectivity and sensitivity to Al³⁺ over other common metal ions. This study will promote the development of fluorescent probes in the future.

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