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Short communication

Novel spiropyran derivative based reversible photo-driven colorimetric and fluorescent probes for recognizing Fe^{3+} , Cr^{3+} and Al^{3+} metal ions



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GRAPHICAL ABSTRACT



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ABSTRACT

A novel spiropyran derivative, 3-(5-methoxy-6'-nitro-3H-spiro[benzo[d]thiazole-2,2'-chromen]-3-yl)propane-1sulfonate (**MO-SP**) as a chemosensor for metal ions has been synthesized. The prepared **MO-SP** as a colorimetric and fluorescent sensor is sensitive to common trivalent metal ions (Al^{3+} , Cr^{3+} and Fe^{3+}). In addition, it can be used as a "naked eye" identification agent of Fe^{3+} , Cr^{3+} and Al^{3+} . When Fe^{3+} , Cr^{3+} or Al^{3+} was added, the color changed from orange to yellow, meanwhile, the absorbance at the maximum absorption wavelength decreased obviously, but it did not change significantly when other metal ions were added. The **MO-SP** is proved to be sensitive, selective, good linear-relationship and low detection limit for recognition of Fe^{3+} , Cr^{3+} and Al^{3+} . Moreover, the **MO-SP** also could be employed as molecular logic function.

1. Introduction

In human life, metal ions can be seen everywhere, but when the

content of some metal ions such as Fe^{3+} , Hg^{2+} and Cr^{3+} is high, it will affect the metabolism of cells and injury some organs which will eventually cause irreversible harm to our body [1–4]. In order to detect

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and measure metal ions, researchers have made great efforts to develop suitable chemical sensors [5–7]. These sensitivity and selectivity sensors play an important role in the field of biological, medicinal and environment researches.

Transition metals and heavy metal elements are abundant in nature, some of which play an exceedingly important role in life. For the past few years, domestic and foreign research enthusiasm for trivalent metal ions (M³⁺) is only increasing [8]. Chromium, one of the major pollutants for environment, was produced by steelmaking industry, fossil fuel combustion and paint industries, each of which delivers toxic ingredients into the soil and streams [9-11]. In addition, highly sensitive Cr^{3+} can enter the body then bind to DNA and has harmful effects on cell structure and cell composition [12]. Sorting the most abundant elements in the current crust by quality, aluminum is the third, so it is widely used in shipbuilding, aerospace equipment, corrosion resistant containers and indoor sound insulation board [13-16]. The heavy use of aluminum leads to increasing leakage into the environment, which can be dangerous both in humans, animals and plants [17]. For instance, health problems, such as neurological disorders, have also been linked to the accumulation of Al^{3+} in brain tissue [18]. Iron-one of the high content metals on Earth, plays the role of a catalyst to transport oxygen in oxido-reductase reactions [19]. Either the excess or deficiency may damage organisms and induce a variety of diseases such as renal failure, cell death, cancer and Alzheimer [20–23]. Considering the potential impact of M³⁺ on the environment and human survival, we need to find appropriate methods to detect them effectively.

Several methods for the determination and detection are reported such as atomic absorption spectrometry with electrothermal (graphite furnace) atomization (GF-AAS) [24–25] and inductively coupled plasma optical emission spectrometry (ICP-OES) [26]. The advantages of GF-AAS are high efficiency and good sensitivity, which can reach ppb level, on the contrary, its disadvantages are large background interference and time consumption. ICP-OES can measure the amount of elements in the sample but it is not economical and environmentally friendly. The analytical methods based on UV–Visible spectrophotometry and fluorescence spectroscopy are relatively real-time, inexpensive and convenient, so they are valued and widely used.

Spiropyrans are one of the important photochromic compounds that have attracted much interest from the viewpoints of both fundamental elucidation of photochemical reactions [27,28] and their potential application for optical devices and sensors [29-33]. The photochromism of spiropyrans usually results from photo-cleavage of the C-O bond under an irradiation, creating a ring-opened merocyanine form which displays broad absorption in the visible region and which can be converted back to the (colorless) ring-closed SP form by another irradiation. With the in-depth study of photochromic spiropyrans, researchers found that different external stimuli such as solvents, pH and ions have different effects on the performance of spiropyrans. For example, the choice of different solvents can cause reversible changes in the position and intensity of the UV-Vis absorption spectrum of the compound, which is called solvatochromism. In addition, spiropyran compounds complexed with some ions to form relatively stable blue-shift metal complexes, which are easily detected on the spectrum, and the stability

of different ion complexes can be distinguished by comparison of spectral behaviors, which is ionchromism [34–40].

In the present work, a novel spiropyran derivative, 3-(5-methoxy-6'nitro-3H-spiro[benzo[d]thiazole-2,2'-chromen]-3-yl)propane-1-sulfonate (**MO-SP**) as a chemosensor for metal ions has been synthesized. The as-prepared probe can effectively recognize the trivalent metal ions (Cr^{3+} , Al^{3+} and Fe^{3+}). The **MO-SP** chemosensor can provide a broad application prospect because of its good anti-interference capability, excellent reversibility and low detection limit.

2. Experimental section

2.1. Reagents and materials

All raw materials and solvents are purchased and used without further purification. Metal ion solution (1.0×10^{-2} M) was prepared with pure water and nitrate (Ba^{2+} , Pb^{2+} , Ni^{2+} , Al^{3+} , Mg^{2+} , Cr^{3+} , K^+ , Na^+ , Fe^{3+} , Zn^{2+} , Cu^{2+} , Ag^+ , Ca^{2+} , Cd^{2+} and Co^{2+}).

2.2. Synthesis of 3-(2-methyl-5-methoxybenzothiazole) propane-1-sulfonate

The synthetic route of 3-(2-methyl-5-methoxybenzothiazole) propane-1-sulfonate is shown in Scheme 1. A 250 mL circular bottom flask was taken and 2-methyl-5-methoxybenzothiazole (0.90 g, 0.005 mol) was measured and dissolved in toluene (20 mL). After stirring for 30 min, 1, 3-propanolactone (1.00 g, 0.008 mmol) was added. The reaction mixture was refluxed overnight. The precipitate produced was filtered at room temperature and washed with acetone and ethyl acetate and eventually dried to yield the white powder (84%), namely intermediate. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.25 (d, *J* = 9.0 Hz, 1H), 8.12 (s, 1H), 7.39 (d, *J* = 6.8 Hz, 1H), 4.92 (t, *J* = 8.2 Hz, 2H), 3.96 (s, 3H), 3.16 (s, 3H), 2.63 (t, *J* = 6.2 Hz, 2H), 2.22–2.09 (m, 2H).

2.3. Synthesis of 3-(5-methoxy-6'-nitro-3H-spiro[benzo[d]thiazole-2,2'chromen]- 3-yl)propane-1-sulfonate (**MO-SP**)

The synthetic route of **MO-SP** is shown in Scheme 2. The above intermediate product (0.30 g, 0.001 mol) was dissolved in ethanol (20 mL) and then piperidine (0.09 g, 0.001 mol) was slowly added. After constant stirring for 1 h, 5-nitro salicylaldehyde (0.16 g, 0.001 mol) was put in, the solution was refluxed for 18 h to produce a red solution. The mixture was purified by column chromatography (dichloromethane: methanol = 50:1) and obtained the red powder (41%), namely **MO-SP**. ¹H NMR (300 MHz, DMSO- d_6) δ 8.59–8.48 (m, 2H), 8.17–8.08 (m, 2H), 7.98 (d, J = 2.4 Hz, 1H), 7.84 (dd, J = 9.7, 3.1 Hz, 1H), 7.28 (dd, J = 9.0, 2.3 Hz, 1H), 6.31 (d, J = 9.7 Hz, 1H), 4.87 (t, J = 7.9 Hz, 2H), 3.95 (s, 3H), 2.68 (t, J = 6.3 Hz, 2H), 2.16 (q, J = 8.2, 6.5 Hz, 2H). ¹³C NMR (75 MHz, DMSO- d_6) δ 179.42, 173.74, 161.12, 148.88, 143.26, 132.23, 132.03, 128.33, 124.91, 123.94, 121.29, 119.25, 117.27, 109.49, 100.24, 63.25, 56.93, 47.82, 24.64.



Scheme 1. Synthetic route of intermediate.



Scheme 2. Synthetic route of MO-SP.



Fig. 1. Absorption and color changes after exposure to dark, Vis 2 h and Vis 2 h + UV 2 h. Inset: Cycle curves at different irradiations at 500 nm.

2.4. Characterization

¹H NMR and ¹³C NMR spectra were recorded on Bruker Avance III 300 MHz or 400 MHz using tetramethylsilane (TMS) as an internal standard and DMSO as solvent. The IR spectra were performed on a Protégé 460 spectrometer using KBr disk. The fluorescence emission spectra were recorded on a F-280 spectrometer. The absorption spectra were measured by an UV-759 spectrophotometer.

3. Result and discussion

3.1. Photochromic behavior of MO-SP

Fig. 1 shows the photochromic performance of **MO-SP**. Before irradiation, the color of the **MO-SP** solution (4 \times 10⁻⁵ M, ethanol:

water = 1:1, V/V) was deep red and the absorption spectrum showed a prominent band at the visible region. While under visible condition, the solution of **MO-SP** changed to pale red with a reduced absorption at 500 nm and a slight red-shift. This result indicated that **MO-SP** can be transformed from its original open loop merocyanine (**MC**) structure to the closed loop **SP** structure under visible light irradiation. However, after 2 h of visible light exposure and 2 h of ultraviolet light exposure, the absorption can rise somewhat, but lower than that in the dark. The absorption peak of ultraviolet and visible light after cyclic irradiation at 500 nm showed that partial degradation occurred after the three cycles, but the reproducibility was good (inset of Fig. 1).

3.2. Metal-induced recognition of MO-SP

In order to study the recognition of metal ions by **MO-SP**, various cations such as Cd^{2+} , Mg^{2+} , Ni^{2+} , Ca^{2+} , Ca^{2+} , Co^{2+} , Fe^{3+} , Al^{3+} , Cr^{3+} , Pb^{2+} , Ag^+ , Na^+ , K^+ , Zn^{2+} and Ba^{2+} (1×10^{-2} M) were added to the aqueous solution of spiropyran derivative (4×10^{-5} M, ethanol:water = 1:1, V/V). Fig. 2 shows the absorption and fluorescence spectra of **MO-SP** after adding different cations. It was found that the absorption of $Fe^{3+}/Cr^{3+}/Al^{3+}$ -**MO-SP** at 500 nm decreased significantly while adding of other metal ions caused only negligible changes in absorption spectra (Fig. 2A). Similarly, the solution changed from orange to yellow when 0.05 mL of $Fe^{3+}/Cr^{3+}/Al^{3+}$ was added while the color of other metals did not change (Fig. S4). This result demonstrated that **MO-SP** could be used as a colorimetric chemosensor for M^{3+} ($Fe^{3+}/Cr^{3+}/Al^{3+}$). As is shown in Fig. 2B, the intensity of Al^{3+} varied the most, while that of Fe^{3+} and Cr^{3+} changed little. These results manifest that **MO-SP** has a superb identification for Fe^{3+} , Al^{3+} and Cr^{3+} .

The sensitivity of **MO-SP** was studied by titration experiment. Fig. 3 shows the effect of concentration of M^{3+} (Fe³⁺, Al³⁺ and Cr³⁺) on the absorption of **MO-SP** (Fig. 3A, B and C). Fig. 3a, b and c) displays the absorption wavelength diagram corresponding to each concentration at the absorption wavelength of 500 nm. It can be found that with increasing of the concentration of metal ion, the absorption at 500 nm



Fig. 2. Change of UV–Vis absorption (A) and fluorescent (B) of MO-SP (4×10^{-5} M, ethanol: water = 1:1, V/V) upon 0.05 mL addition of different metal ion solutions (Pb²⁺, Na⁺, Ni²⁺, Fe³⁺, Mg²⁺, K⁺, Cd²⁺, Zn²⁺, Ag⁺, Ca²⁺, Ba²⁺, Cu²⁺, Co²⁺, Cr³⁺ and Al³⁺).



Fig. 3. Absorption spectra of the **MO-SP** solution (4 × 10⁻⁵ M, ethanol: water = 1:1, V/V) with increasing amounts of M^{3+} (1 × 10⁻² M, in water). (A, a) Fe³⁺; (B, b) Cr³⁺; (C, c) Al³⁺.



Fig. 4. Competitive selectivity study of M^{3+} -MO-SP complex in the presence of excess amounts of different metal ions (1 × 10⁻² M, in water). (A) Fe³⁺-MO-SP; (B) Cr³⁺-MO-SP; (C) Al³⁺-MO-SP.

decreased ratio-metrically and then gradually reached steady. This result suggested that there was an equilibrium relationship between **MO-SP** and M^{3+} (Fe³⁺/Cr³⁺/Al³⁺) complex. In order to prove the above conclusion, the fluorescence titration experiment was performed. As shown in Fig. S5, the fluorescence result of **MO-SP** is similarly in accordance with absorption of **MO-SP**.

3.3. Competitive selectivity for detecting M^{3+} (Fe³⁺, Al³⁺ and Cr³⁺) in the presence of other metal ions

The competitive selectivity of **MO-SP** towards M^{3+} was studied when the interference of various monovalent or divalent metal ions were added. The absorption spectra of **MO-SP** (4 × 10⁻⁵ M, ethanol:



Fig. 5. Linear relationship between the absorption of MO-SP at 500 nm and the concentration of M^{3+} (A) Fe^{3+} , (B) Cr^{3+} and (C) Al^{3+} .

water = 1:1, V/V) were used to investigate its sensitivity in aqueous solutions with different interfering ions $(1 \times 10^{-2} \text{ M})$ coexisting with Fe³⁺, Al³⁺ or Cr³⁺ (1 × 10⁻² M). It was found that after adding other metal ions, the absorbance of **MO-SP** did not change significantly (Fig. 4), indicating the established chemosensing process is completely unaffected by other metal ions. Moreover, as shown in Fig. S6, though there were some intensity fluctuations in the fluorescence spectra, it can also conclude the stability of **MO-SP** in the presence of other metal ions.

3.4. Detection limit and response time

Fig. 5A–C show the linear relationship between the absorption of **MO-SP** at 500 nm and the concentration of Fe³⁺ in the range of 0.1–2.1 μ M, Cr³⁺ in the range of 0.1–2.2 μ M, Al³⁺ in the range of 0.1–2.4 μ M, respectively. The detection limits of Fe³⁺, Cr³⁺ and Al³⁺ are calculated to be 4.56 μ M, 4.39 μ M and 4.89 μ M, respectively. This result indicated that **MO-SP** can be used as a highly selective recognition sensor of Fe³⁺, Cr³⁺ and Al³⁺.

It is acknowledged that the response time is one of the important factors to evaluate chemosensor performance. After 0.05 mL Fe³⁺/ Cr^{3+}/Al^{3+} was added to **MO-SP** (4 × 10⁻⁵ M) solution, the absorption at 500 nm gradually decreased in 0–400 s and changed almost stable after 400 s (Fig. 6). It can be found that the response time of **MO-SP** to Fe³⁺/ Cr^{3+}/Al^{3+} is short and the corresponding performance is good.



Fig. 6. Absorbance of MO-SP (4.0 \times 10⁻⁵ M) at 500 nm after adding M³⁺ (A) Fe³⁺, (B) Cr³⁺, and (C) Al³⁺ for a period of time.



Fig. 7. Change of absorbance of MO-SP when M^{3+} and EDTA are added alternately. (1: $Fe^{3+}/Cr^{3+}/Al^{3+}$ 2: EDTA).

3.5. Reversibility of MO-SP

Reversibility is also an effective indicator to evaluate the chemosensor performance of **MO-SP**. As can be seen from the Fig. 7, after the addition of M^{3+} (Fe³⁺/Cr³⁺/Al³⁺) and EDTA alternately, the absorption changes at 500 nm were observed. It can be found that Fe³⁺, Al³⁺ and Cr³⁺ can all be invertible three times. This result indicated that the sensor of MO-SP had great stability and repetition. The reason for the incomplete recovery of the reversible curve is that the number of molecules with reverse-photochromic ability gradually decreases or even completely loses its reversible property due to oxidation decomposition during the cyclic process of repeated discoloration and fading of photochromic materials.

3.6. Possible complexation mechanism of $MO-SP-M^{3+}$

Fig. 8A shows the infrared spectra of **MO-SP** in the presence and absence of M^{3+} . The characteristic absorption wavenumber of C-N bond at 1420 cm⁻¹ was shifted to 1384 cm⁻¹ after M^{3+} (Fe³⁺, Al³⁺ and Cr³⁺) was added. Similarly, the absorption wavenumber of C-S-C was shifted from 1240 cm⁻¹ to 1230 cm⁻¹, suggesting that C-S-C and C-N of MO-SP were involved in the recognition of M^{3+} . To further confirm this conclusion, ¹H NMR spectra of **MO-SP** in the presence and absence of M^{3+} were performed. In Fig. 8B, the proton (H_a, H_b) of **MO-SP** was 6.3 ppm and 4.8 ppm, while after adding M^{3+} (Fe³⁺, Al³⁺ and Cr³⁺), the H_a and H_b were offset to the lower field, which verified that N-M³⁺-S coordination occurred.

It is well known that the complex curve of chemosensor and the metal ion allows us to know their binding ratio, and the highest point of the complex curve corresponds to their binding ratio [41]. As seen from Fig. S7, Job's plot shows that the absorption of $Fe^{3+}/Cr^{3+}/Al^{3+}$ at 0.6/ 0.4/0.6 mol fraction absorbance reach maximum. These data indicate that the chemometrics of $Fe^{3+}/Cr^{3+}/Al^{3+}$ -**MO-SP** complexes are about 2:1,1:2 and 2:1, respectively. Therefore, the chemosensor mechanism between **MO-SP** and M^{3+} ($Fe^{3+}/Cr^{3+}/Al^{3+}$) can be proposed based on the analysis of Job's plot, IR and ¹H NMR (Scheme 3).

3.7. Application of MO-SP in molecular logic gate

In recent years, the application of molecular logic gates has become a hot topic [42,43]. The gate seems to be a simple switch, but can be used for the signal amplification and transduction, which is a very meaningful research. As seen from Fig. 9, $M3^+$ (Fe³⁺, Cr³⁺ and Al³⁺) and chelating agent EDTA were taken as the input signal, and the absorption change at 500 nm was taken as the output signal. The





Fig. 8. (A) FT-IR spectra of MO-SP and MO-SP $-M^{3+}$. (B) ¹H NMR spectra of MO-SP and MO-SP $-M^{3+}$.



Scheme 3. Possible complexation mechanism.

(a

	$M^{3+}(Fe^{3+}/Cr^{3+}/Al^{3+})$	EDTA	Absorbance at 500 nm
	0	0	0
	0	1	0
	1	0	1
l	1	1	0

Fig. 9. (a) Truth table, (b) INHIBIT logic circuit based on M³⁺ and EDTA as chemical inputs and absorbance mode as output.

occurrence of the absorbance at 500 nm depends on the complex of ${\bf MO}{\textbf{-SP}}$ and ${\rm M}^{3\, +},$ and the absorbance at 500 nm increased when EDTA was added to the solution. Only when M^{3+} (Fe³⁺, Cr³⁺ and Al³⁺) was used as input, the value of output for the absorbance at 500 nm is 1. While in other cases, the value is 0. Therefore, it is practical to apply the recognition of MO-SP to M³⁺ to circuit elements with memory and erase functions.

EDTA

4. Conclusions

In summary, a novel spiropyran derivative, 3-(5-methoxy-6'-nitro-3H-spiro[benzo[d]thiazole-2,2'-chromen]-3-yl)propane-1-sulfonate (MO-SP) as a chemosensor has been synthesized. The prepared MO-SP can be used as a colorimetric and fluorescent sensor to selectively detect common trivalent metal ions (Al3+, Cr3+ and Fe3+) in ethanol and water solutions. The chelated of MO-SP-M³⁺ can be converted to the original form of spiropyran by the irradiation and EDTA. Therefore, this chemosensor provided a reversible optical-driven and chemistry-driven method for M^{3+} detection. In addition, after the addition of M^{3+} (Fe³⁺, Cr^{3+} , Al^{3+}) and EDTA, the **MO-SP** can be used as the inhibition logic gate.

Declaration of Competing Interest

The authors declared that there is no conflict of interest.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https:// doi.org/10.1016/j.inoche.2020.107968.

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