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A fluorescence turn-on chemosensor for hydrogen sulfate anion based on quinoline and naphthalimide

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ABSTRACT

A new fluorescence turn-on chemosensor **1** based on quinoline and naphthalimide was prepared and its anion sensing toward various anions behavior was explored in this paper. Sensor **1** exhibited a highly selective fluorescent response toward HSO_4^- with an 8-fold fluorescence intensity enhancement in the presence of 10 equiv. of HSO_4^- in DMSO-H_2O (1/1, *v*/*v*) solution. The sensor also displayed high sensitivity to hydrogen sulfate and the detection limit was calculated to be 7.79×10^{-7} M. The sensing mechanism has been suggested to proceed *via* a hydrolysis process of the Schiff base group. The hydrolysis product has been isolated and further identified by ^{1}H NMR and MS.

1. Introduction

The design and development of new chemosensors for biologically and environmentally important anions have attracted considerable attention [1–11]. Among various important anions, hydrogen sulfate (HSO_4^-) is of particular interest owing to its established role in biological system and industrial areas. This amphiphilic anion eventually dissociates at high pH to generate toxic sulfate anion (SO_4^{2-}), causing irritation of skin and eyes and even respiratory paralysis [12–13]. Therefore, reliable and efficient ways of detecting and sensing the HSO_4^- anion are necessary.

In recent years, many efforts have been devoted to designing and developing various sensors for HSO_4^- anions detection [14–23]. Among those chemosensors, fluorescence chemosensors become increasingly popular due to their high sensitivity. In terms of sensitivity, the "Off-On" fluorescent sensors are favored over "On-Off" fluorescent sensors. However, designing and developing novel fluorescence turn-on receptors for anion detection is still a challenging issue since anions in general act as fluorescence quenchers [24–27].

Up to now, many of the reported chemosensors based on the hydrogen bonding (O—H or N—H) mechanism between the receptors with HSO_4^- anion were designed, which have displayed poor selectivity in aqueous media [28–31]. Chemosenors based on Schiff bases for HSO_4^- anion have proven attractive because they could easily hydrolyze in the presence of HSO_4^- in aqueous solution. However, the examples of HSO_4^- selective "turn-on" chemosensors based on a Schiff base platform *via* a hydrolysis mechanism are still scarce [32–34].

Herein, we report a new turn-on type chemosensor **1** based on Schiff base, which was synthesized by the simple linkage of 8-hydroxy-2-quinolinecarbaldehyde **2** and *N*-amido-4-butyl-1,8-naphthalimide **4** (Scheme 1). More importantly, sensor **1** not only displayed obvious fluorescence enhancement in the presence of HSO_4^- anion but also exhibited high selectivity and sensitivity to hydrogen sulfate anion over other competitive anions in DMSO-H_2O solution.

2. Experimental

2.1. Materials and apparatus

All reagents were purchased from commercial suppliers and were used without further purification. Double distilled water was used throughout the experiment. The solution of anions was prepared from the form of tetrabutylammonium (TBA) salts. Fluorescence detections were performed on a RF5301 fluorescence spectrometer. The ^{1}H NMR spectroscopy study was conducted with a Varian INOVA-400 spectrometer using tetramethylsilane (TMS; $\delta = 0$ ppm) as an internal standard. MS were carried out on LCMS-QP2010 (Shimadzu).

2.2. Synthesis

2.2.1. Synthesis of 8-hydroxy-2-quinolinecarbaldehyde (**2**) [35]

8-hydroxy-2-methylquinoline (2.00 g, 12.5 mmol), selenium dioxide (1.74 g, 15.6 mmol) and 50 mL of 1,4-dioxane were mixed and

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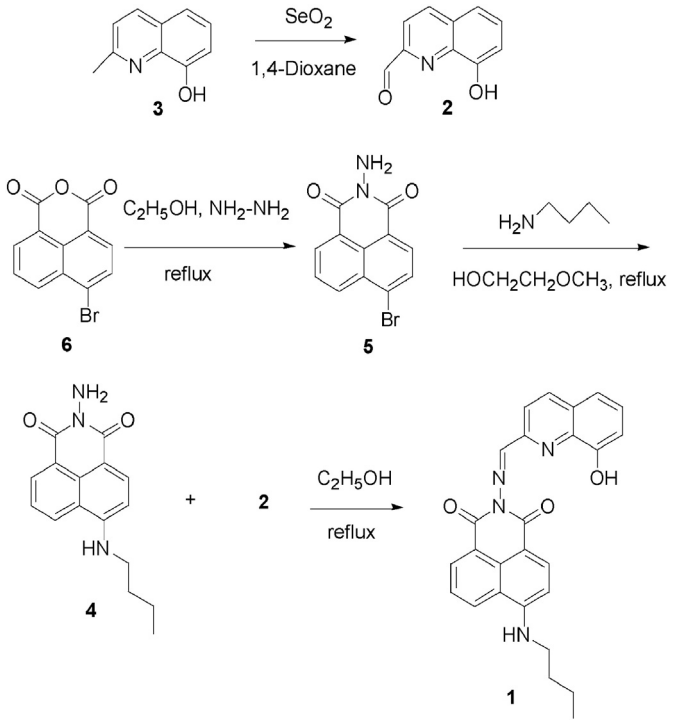


Scheme 1. The synthetic route of sensor **1**.

stirred in a 100 mL round bottom flask. The resulting solution was refluxed for 24 h. The reaction was monitored until completion by TLC method. The reaction mixture was then filtered off and the selenium metal was washed with dichloromethane. The combined filtrates were evaporated off under reduced pressure. The crude product was purified by column chromatography on silica gel to give pure **2** as a yellow needle crystal (1.30 g, 59.9% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.17 (s, 1H), 8.28 (d, 1H, J = 8.3 Hz), 8.11 (s, 1H), 8.01 (d, 1H, J = 8.3 Hz), 7.58 (t, 1H, J = 7.7 Hz), 7.39 (d, 1H, J = 7.9 Hz), 7.24 (d, 1H, J = 8.0 Hz).

*2.2.2. Synthesis of N-amido-4-bromine-1,8-naphthalimide (**5**) [36]*

1,8-naphthalimide (2.77 g, 10 mmol) was dissolved in absolute ethanol (50 mL). An excess of hydrazine hydrate (85% *w*/*w*, 1.18 g, 20 ml) was added. After refluxing for 4 h, the mixture was cooled and the precipitate was filtered and recrystallized from ethanol to give **5** as a yellow solid (2.48 g, 84.9% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.68 (d, 1H, J = 7.3 Hz), 8.60 (d, 1H, J = 8.3 Hz), 8.43 (d, 1H, J = 7.8 Hz), 8.05 (d, 1H, J = 7.9 Hz), 7.86 (t, 1H, J = 7.9 Hz), 5.52 (s, 2H).

*2.2.3. Synthesis of N-amido-4-butyl-1,8-naphthalimide (**4**) [37]*

Compound **5** (1.46 g, 5 mmol) and 10 mL *n*-butylamine were added into 30 mL of methoxyethanol and refluxed for 3 h. After cooled to room temperature, the mixture was poured into 100 mL water. The precipitate was filtered, dried in vacuum to give **4** as a yellow solid (1.35 g, 95.6% yield). ^{1}H NMR(400 MHz, $CDCl_3$): δ 8.57 (d, 1H, J = 6.8 Hz), 8.44 (d, 1H, J = 8.8 Hz), 8.10 (d, 1H, J = 8.2 Hz), 7.61 (t, 1H, J = 7.9 Hz), 6.69 (d, 1H, J = 8.5 Hz), 5.51 (s, 2H), 5.38 (s, 1H), 3.43–3.38 (m, 2H), 1.84–1.76 (m, 2H), 1.56–1.51 (m, 2H), 1.02 (t, 3H, J = 7.3 Hz).

*2.2.4. Synthesis of sensor **1** [37]*

Compound **2** (0.69 g, 3.9 mmol) was dissolved in 30 mL hot ethanol, then compound **4** (1.00 g, 3.5 mmol) was added to the solution. The mixture was refluxed for 3 h and the solid was precipitated. After filtration, the solid was collected, washed successively with ethanol and dried in vacuum to give compound **1** as an orange solid (1.40 g, 90.8% yield). M.p.: 138–140 °C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.16 (s, 1H), 8.90 (s, 1H), 8.78 (d, 1H, J = 7.2 Hz), 8.51 (d, 2H, J = 9.0 Hz), 8.32 (t, 2H, J = 7.0 Hz), 7.94–7.91 (m, 1H), 7.73 (t, 1H, J = 7.8 Hz), 7.57 (t, 1H, J = 7.9 Hz), 7.51 (d, 1H, J = 7.9 Hz), 7.19 (d, 1H, J = 7.1 Hz), 6.84 (d, 1H, J = 8.8 Hz), 3.43–3.35 (m, 2H), 1.74–1.67 (m, 2H), 1.49–1.40 (m, 2H), 0.96 (t, 3H, J = 7.3 Hz). ^{13}C NMR (150 MHz, DMSO-d_6): δ 169.4, 159.9, 159.3, 153.5, 150.6, 149.3, 137.8, 136.6, 134.4, 130.7, 129.2, 129.0, 128.5, 128.4, 123.8, 121.5, 119.7, 117.8, 117.4, 112.0, 106.6, 103.5, 42.1, 29.4, 19.4, 13.3 ppm. HRMS-ESI: Calcd. for $C_{26}H_{22}N_4O_3$ $[M + H]^+$: 439.1770. Found: 439.1761.

2.3. General procedure for fluorescence experiment

The solution of sensor **1** (5.0×10^{-6} mol L^{-1}) in DMSO-H_2O (1/1, *v*/*v*) was prepared and stored in dry atmosphere. The solution was used for all spectroscopic studies after appropriate dilution. The solution of 5.0×10^{-5} mol L^{-1} TBA salts of the respective anions (F^-, Cl^-, Br^-, I^-, SO_4^{2-}, ClO_4^-, AcO^-, $H_2PO_4^-$, CN^-, CO_3^{2-}, HCO_3^-, SO_3^{2-}, HSO_3^-, S^{2-}, NO_3^-, PO_4^-, SCN^- and HSO_4^-) was prepared in double distilled water. The fluorescence spectra were obtained by excitation at 467 nm and emission at 542 nm. The excitation and emission slit widths were both set at 3 nm.

3. Results and discussion

3.1. Selectivity studies

The selectivity properties of sensor **1** for anions (F^-, Cl^-, Br^-, I^-, SO_4^{2-}, ClO_4^-, AcO^-, $H_2PO_4^-$, CN^-, CO_3^{2-}, HCO_3^-, SO_3^{2-}, HSO_3^-, S^{2-}, NO_3^-, PO_4^-, SCN^- and HSO_4^-) were studied using tetrabutylammonium as a counter in DMSO-H_2O (1/1, *v*/*v*) solution. The absorption spectra of sensor **1** was conducted with the common anions (10 equiv.), the results show that the UV–vis absorbance has no change in the presence of different anions (Fig. 1).

Besides, the fluorescent spectrum of sensor **1** was characterized by a strong emission at 542 nm. Among the common anions (10 equiv.), the fluorescence were greatly enhanced only in the presence of HSO_4^- (Fig. 2). The results suggest that sensor **1** has a high selectivity for the hydrogen sulfate anion over the common anions.

3.2. Fluorescence titration experiment

To gain deeper insight into the fluorescent properties, the fluorescence titration of sensor **1** toward HSO_4^- was investigated. Upon the addition of various equivalents of HSO_4^-, fluorescence intensity gradually

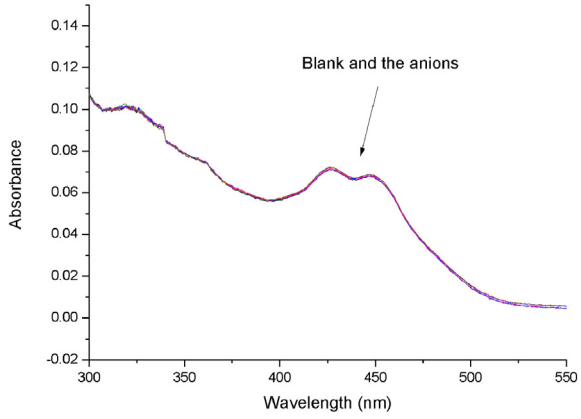


Fig. 1. The absorption spectra of sensor **1** (5.0 μM) in the presence of 10 eq. of different anions (as TBA salts) in DMSO-H_2O (1/1, *v*/*v*) solution.

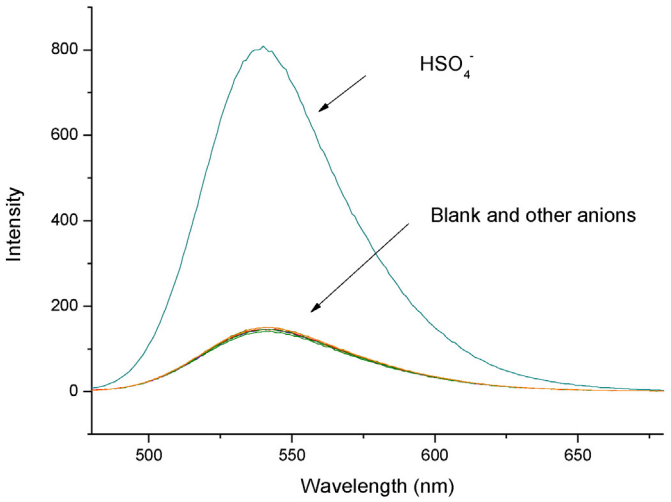


Fig. 2. Fluorescent emission spectra of sensor **1** (5.0 μM) in the presence of 10 eq. of different anions (as TBA salts) in DMSO-H_2O (1/1, *v*/v) solution. The excitation wavelength was 467 nm.

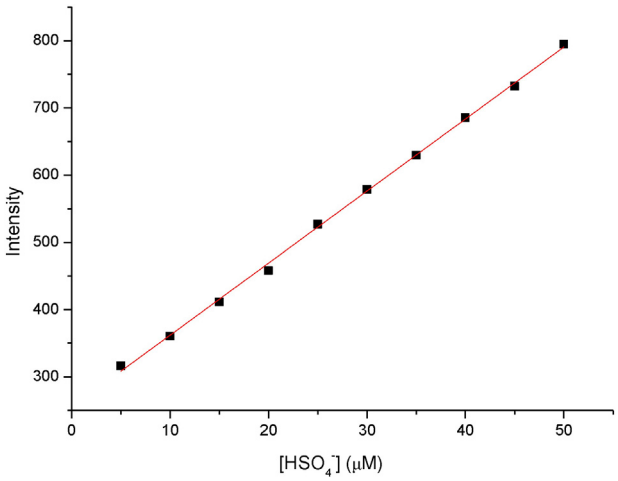


Fig. 4. Fluorescence intensity of **1** *vs* HSO_4^- concentrations. [**1**] = 5 μM. The concentrations of HSO_4^- in the range of 5 μM to 50 μM.

increased. When the concentration of hydrogen sulfate anions increased to 10 equiv., the fluorescence intensity reached maximum with 8-fold enhancement (Fig. 3). Fig. 3 (inset) also shows that there was a good linearity between the fluorescence intensity (542 nm) and concentrations of HSO_4^- in the range from 5 μM to 50 μM, indicating that sensor **1** can detect quantitatively relevant concentrations of HSO_4^-. The linear equation was found to be $y = 10.72\times + 254.62$ ($R^2 = 0.9986$) (Fig. 4), where *y* is the fluorescence intensity at 542 nm measured at a given HSO_4^- concentration and *x* represents the concentration of HSO_4^- added. Therefore, the detection limit (DL) of fluorescent spectra of sensor **1** for HSO_4^- was 7.79×10^{-7} M according to the Stern-Volmer plot [38]:

$$DL = 3\sigma/S = 7.79 \times 10^{-7}\ M$$

Where σ is the standard deviation of the blank solution; *S* is the slope of the calibration curve. The detection limit was sufficiently low to detect a submicromolar concentration of HSO_4^-, which indicates this chemosensor may have a high potential for the determination of hydrogen sulfate anion in biological and environmental science. Moreover, the detection limit of the sensor **1** is compared with representative reported sensors (Table 1).

3.3. Interference experiments

It is a matter of necessity for a good chemosensor to have high selectivity. To further certify the excellent selectivity of sensor **1**, the interference experiments were also carried out by the addition of 10 equiv. of anions such as F^-, Cl^-, Br^-, I^-, SO_4^{2-}, ClO_4^-, AcO^-, $H_2PO_4^-$, CN^-, CO_3^{2-}, HCO_3^-, SO_3^{2-}, HSO_3^-, S^{2-}, NO_3^-, PO_4^- and SCN^- to a DMSO-H_2O (1/1, *v*/v)

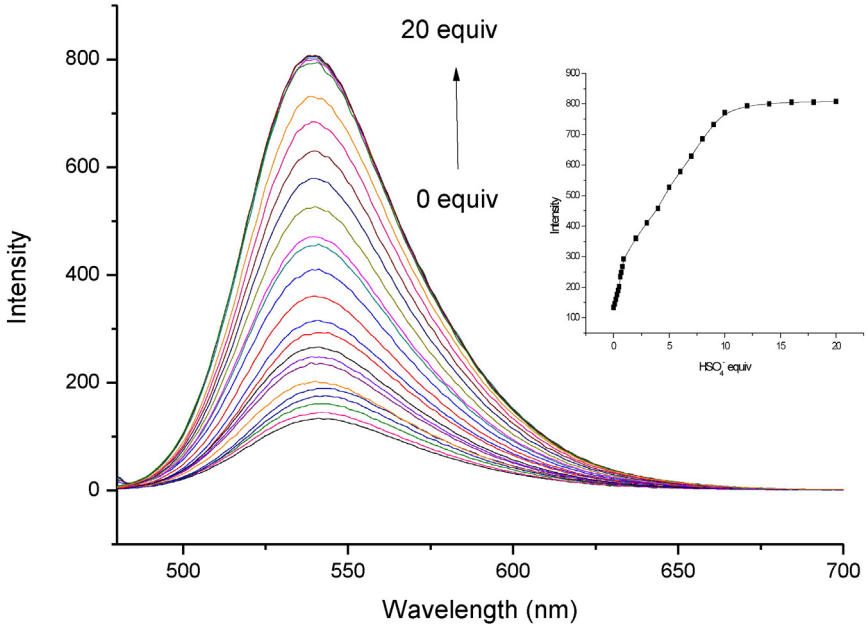


Fig. 3. Fluorescent emission spectra of sensor **1** (5.0 μM) in the presence of different equiv. of HSO_4^- in DMSO-H_2O (1/1, *v*/v) solution. The excitation wavelength was 467 nm.

Table 1
The comparison of the sensor **1** with other representative reported chemosensors for hydrogen sulfate anion.

Number	Fluorophore	Detection methods	DL (M)	Mechanism	References
1	Azobenzene	Uv-vis absorption	2.00×10^{-6}	Hydrogen bond	[14]
2	Naphthalene	Uv-vis absorption Fluorescence	2.91×10^{-6}	Hydrogen bond	[24]
3	Triphenylamine	Uv-vis absorption Fluorescence	1.00×10^{-8}	Hydrogen bond	[39]
4	Diketopyrrolopyrrole	Uv-vis absorption Flurescence	6.45×10^{-9}	Hydrolysis	[32]
5	4-Amino-7-nitro-2,1,3-benzoxadiazole	Uv-vis absorption Fluorescence	2.40×10^{-7}	Hydrolysis	[33]
6	BODIPY	Uv-vis absorption Fluorescence	6.45×10^{-8}	Hydrolysis	[23]
7	Naphthalimide	Fluorescence	7.79×10^{-7}	Hydrolysis	This work

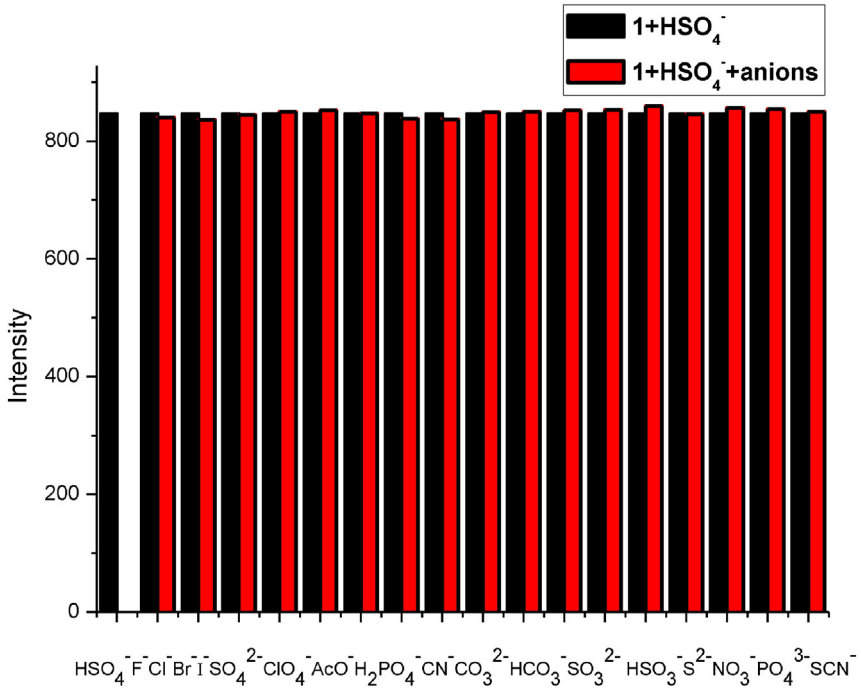


Fig. 5. Results of the interference experiments in the presence of 10 eq. of different anions in DMSO-H_2O (1/1, *v*/v) solution. The excitation wavelength was 467 nm.

solution of sensor **1** in the presence of 10 equiv. of HSO_4^-. As shown in Fig. 5, all the competitive anions had no obvious interference with the detection of HSO_4^-, indicating that the sensor **1** displayed an excellent selectivity toward HSO_4^-.

3.4. Response time

The effect of reaction time on the fluorescent spectra of the detecting system was studied. After adding 10 equiv. of HSO_4^- to the solution of

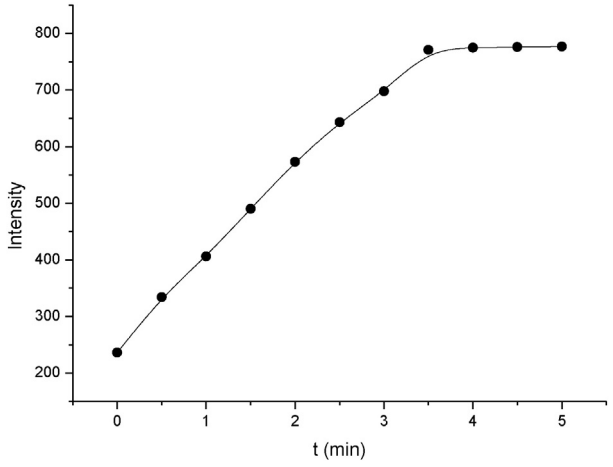


Fig. 6. Time course for the fluorescence intensity changes of sensor **1** in the presence of 10 eq. of HSO_4^- in DMSO-H_2O (1/1, *v*/v) solution. The excitation wavelength was 467 nm.

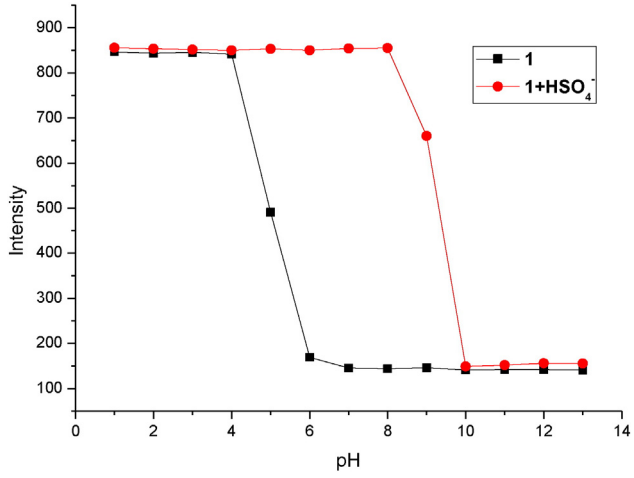


Fig. 7. The effect of pH on the fluorescence intensity changes of sensor **1** with and without HSO_4^- (10 equiv.).

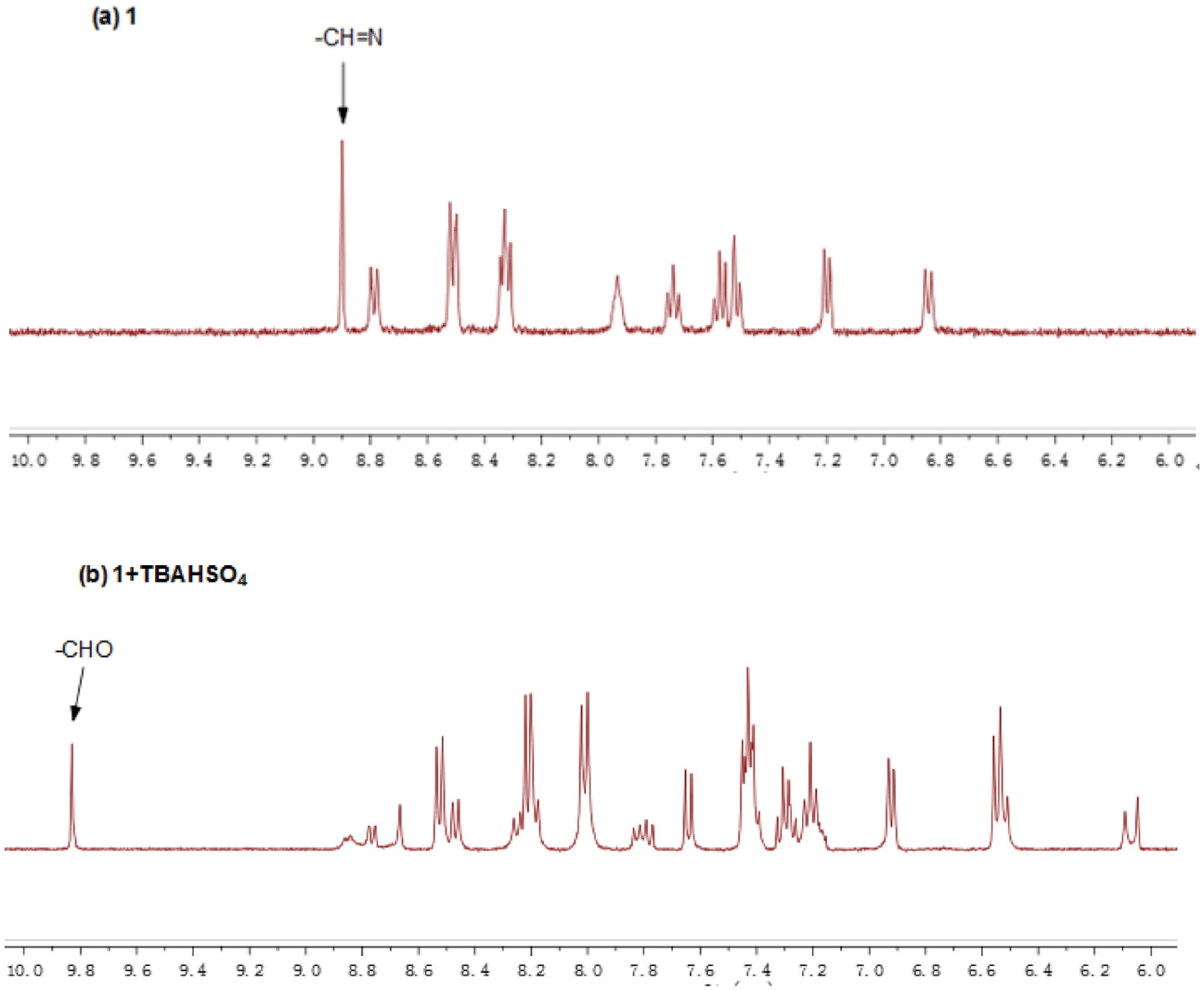


Fig. 8. Partial ^{1}H NMR spectra of sensor **1** in the presence of 0 eq. (a) and 10 eq. (b) of $TBAHSO_4$ in DMSO-d_6/D_2O (10:1, *v*/v).

sensor **1**, the fluorescent intensity at 542 nm of the system reached the maximum at 3.5 min and remained stable with increasing time (Fig. 6). Therefore, sensor **1** has a very short response time for HSO_4^-.

3.5. The effect of pH

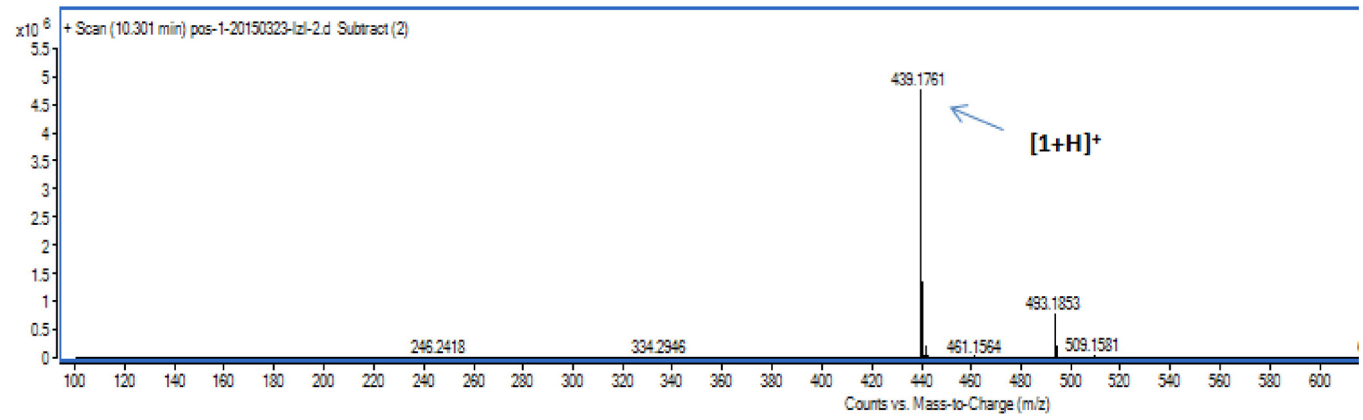
As pH value affects the sensor **1** and may change its fluorescence properties, we also evaluated the effect of pH on the fluorescence signal response to sensor **1** in DMSO-H_2O (1/1, *v*/v) solution. The fluorescence intensity of sensor **1** with HSO_4^- (10 equiv.) remained unaffected and no emission band shifts occurred over a pH range from 6 to 8 (Fig. 7). Therefore, sensor **1** could work over a pH span from 6 to 8 and has potential for biological applications.

3.6. Mechanism studies for hydrogen sulfate recognition

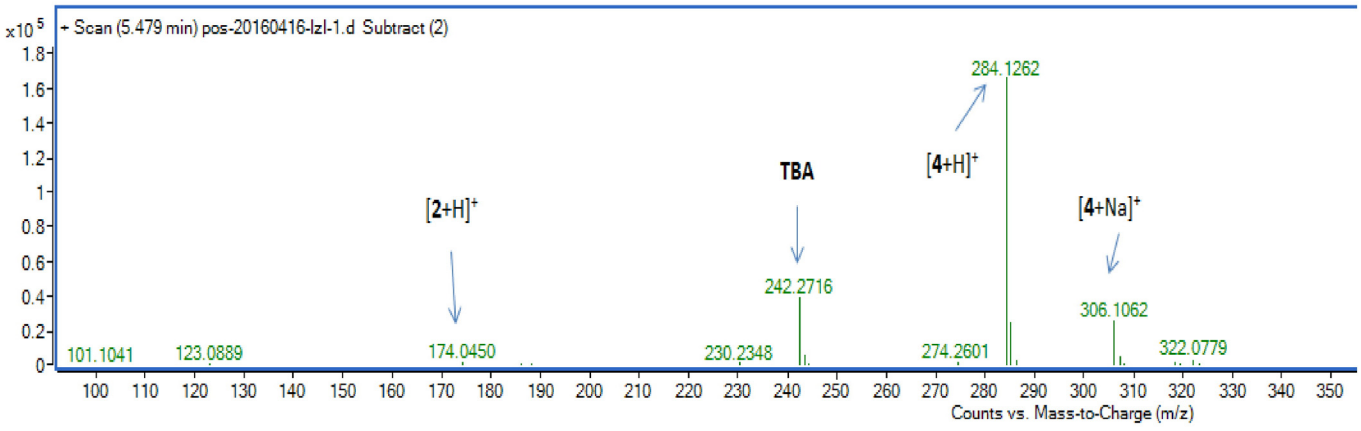
To better understand the detailed reaction pathway of sensor **1** with HSO_4^-, ^{1}H NMR experiment was carried out in DMSO-d_6-D_2O (10/1, *v*/*v*). As was shown in Fig. 8, ^{1}H NMR analysis of the product showed that free **1** had a singlet at δ 8.90 ppm which was assigned to the imino proton (–CH = N). After addition of 10 equiv. of HSO_4^-, the singlet at δ 8.90 ppm disappeared and a new singlet at δ 9.83 ppm corresponding to the aldehyde proton (–C*H*O) appeared. After separation, the products were identified by ^{1}H NMR as compound **2** and compound **4**. Besides, the high resolution mass spectra of **1** was conducted in the absence or presence of HSO_4^- anion (Fig. 9). When 10 equiv. HSO_4^- was added to the solution of sensor **1**, the peak at m/z = 439.1761 disappeared, and the new peak at m/z = 174.0450 and 284.1262 appeared, which corresponded to molecular ion peaks of the compound **2** (Calcd. for $C_{10}H_8NO_2$ $[M + H]^+$: 174.0555) and the compound **4** (Calcd. for $C_{16}H_{18}N_3O_2$ $[M + H]^+$: 284.1399) separately. By the ^{1}H NMR and the ESI-HRMS experiments, the fluorescence enhancement was caused by the HSO_4^--promoted hydrolysis of Schiff base, which leads to the cleavage of the imine bond to generate fluorescent amine and aldehyde (Scheme 2).

4. Conclusion

In summary, we have designed and synthesized a novel chemosensor for hydrogen sulfate anion detection based on naphthalimide and quinoline. Addition of hydrogen sulfate anion to a DMSO-H_2O solution of sensor **1** resulted in an obvious fluorescence enhancement in a short time, triggered by the hydrolysis of the C═N bond to form corresponding aldehyde and amine. The chemosensor showed fast response, good selectivity and high sensitivity toward hydrogen sulfate anion over other competing anions. This chemosensor has a



(a). 0 eq. of $TBAHSO_4$



(b). 10 eq. of $TBAHSO_4$.

Fig. 9. Mass spectrum of sensor **1** in the presence of 0 eq. (a) and 10 eq. (b) of $TBAHSO_4$.

high potential for the determination of hydrogen sulfate anion in biological and environmental science.

Acknowledgments

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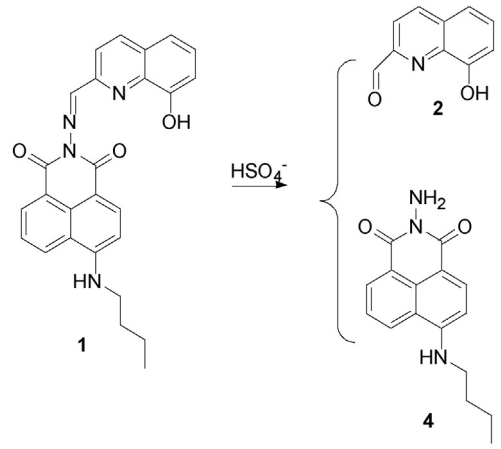

Scheme 2. The proposed sensing mechanism of sensor **1** for HSO_4^- anion in DMSO-H_2O (1/1, *v*/v) solution.

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