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A colorimetric and fluorescence sensor based on biphenolic-dansyl derivative for specific fluoride ion detection

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ABSTRACT

2'-(2-((4-(methylthio)phenyl)amino)-2-oxoethoxy)-[1,1'-biphenyl]-2-yl 5(dimethyl amino)naphthalene-1-sulphonate (1) was synthesised and used as a F⁻ ion sensor. The selectivity of sensor 1 with various anions (F⁻, Cl⁻, Br⁻, CH₃COO⁻, C₆H₅COO⁻ and H₂PO₄⁻) in DMSO was evaluated by ¹H NMR, UV-visible and fluorescence study. For ¹H NMR, an amide proton of sensor 1 disappeared due to the deprotonation reaction upon addition of F⁻ ion. On the contrary, the proton signal of HF₂⁻ emerged at 16.11 ppm. Upon addition of F⁻ ion, sensor 1 showed a new absorption band at 300 nm and the solution turned colourless. For fluorescence response, sensor 1 exhibited the enhancement of emission intensity at 434 nm with slight blue shift when titrated with F⁻ ion. These results suggest that sensor 1 possessed high sensitivity and selectivity towards F⁻ ion detection. In addition, the sensor 1 test strip coated on TLC plates and filter papers were demonstrated for specific detection F⁻ ion.



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1. Introduction

Nowadays, anions play an important roles in many applications, such as catalytic, biological, environmental and industrial processes. The selectivity and sensitivity of anion sensors depend on the size, shape, charge and basicity strength of the anions [1–5]. The molecule sensors for anion detection were developed with different functional groups, including amide, urea, thiourea and guanidinium. The interactions between sensors and anions are hydrogen bonding, anion-pi and electrostatic forces. Among

various anions, fluoride ion is one of the most necessary anions because of its role in the dental care and treatment for osteoporosis [6–8]. On the other hand, the excessive accumulation of fluoride ion in the human body can lead to severe disorders and diseases [9–11]. Hence, numerous methods, such as chromatography and electrophoresis, have been developed to monitor fluoride ion in biological and environmental samples [2,10]. However, these

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instrumental techniques are quite complicated, timeconsuming and require expensive tools.

In the challenge to fluoride ion detection, the fluorescence sensor has been widely used as an alternative tool for anion recognition. Many conditions for fluoride ion sensing modulate through UV-visible absorption and fluorescence spectral change due to molecular aggregation, transformation and translocation [7–10]. The dansyl fluorophore could provide high fluorescent and quantum yield with high sensitivity when responding to the specific anion [11]. Recently, several studies have reported on the synthesis of novel fluorescent dansylbased sensors for anion detection. Miao et al. [11] prepared a sensor based on a C-linked peptidocalix-4 arene functionalised with four l-alanine and dansyl units, which possessed high selectivity to fluoride ion. Chen et al. [12] reported a new fluorescent chemosensor of tetra-sulphonamide derivative bearing two dansyl groups that exhibited strong binding interaction with fluoride ion through the deprotonation complexation.

Herein, we report the synthesis of a new sensor, 2'-(2-((4-(methylthio)phenyl) amino)-2-oxoethoxy)-[1,1'biphenyl]-2-yl 5-(dimethylamino)naphthalene-1-sulphonate (**1**), containing a 4-(methylthio) aniline derivative as a binding site and dansyl moiety as a fluorophore. The sensor **1** was characterised by ¹H NMR, ¹³C-NMR, ESI-MS, FT-IR and elemental analysis. In addition, the complexation study of sensor **1** with F^- ion was investigated by ¹H- NMR, UV-visible and fluorescence spectroscopy. Moreover, the sensor **1** coated on TLC (Thin Layer Chromatography) plate and filter paper for F^- ion detection was also investigated.

2. Materials and methods

2.1 General

¹H- and ¹³C-NMR spectra were collected on a Bruker Avance 3 HD 400 MHz Nuclear Magnetic Resonance (NMR) spectrometer. Elemental analysis (CHNS/O) was performed on a Thermo ScientificTMFLASH 2000. Fluorescence spectra were recorded using an Agilent 8000 spectrophotometer. Mass spectra were obtained on an Agilent 1100 Series LC/MSD Trap spectrometer. All reagents were purchased from Sigma-Aldrich and used without further purification.

2.2 Synthesis of sensor 1

The synthesis of **sensor 1** is shown in Scheme 1. 2-chloro-*N*-(4-(methylthio) phenyl)acetamide (compound **S1**) was prepared by following the literature [13].



Scheme 1. Synthesis pathway of sensor 1.

2.2.1. 2-((2'-hydroxy-[1,1'-biphenyl]-2-yl)oxy)-N-(4-(metylthio)phenyl)acetamide (compound S2)

2,2-biphenol (1.07 g; 5.75 mmol) and potassium carbonate (1.19 g; 8.61 mmol) were mixed and stirred in CH_3CN (20 mL). The compound **S1** (1.86 g; 7.81 mmol) in CH₃CN was slowly added to the solution. The mixture was stirred and refluxed at 150°C under N₂ atmosphere for 10 h. The reaction mixture was poured into water and extracted with CH₂Cl₂ (three times). The organic layer was purified by column chromatography using 9:1 CH₂Cl₂/EtOAc as an eluent, affording a light yellow solid (0.30 g, 15%). M. p. 174–176°C. ¹H NMR (400 MHz, CDCl₃): δ 2.10 (s, 3H), 4.64 (s, 2 H), 6.93 (m, 2 H), 7.02 (m, 2 H), 7.10 (m, 1H), 7.20 (m, 3H), 7.31 (m, 4 H), 8.10 (d, 1H), 9.02 (s, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 166.5, 154.5, 153.3, 137.0, 132.8, 132.2, 131.3, 130.0, 129.6, 128.5, 127.2, 127.0, 125.4, 123.3, 121.7, 123.0, 116.9, 116.6, 113.0, 68.2, 18.7. FT-IR (KBr, cm^{-1}) v = 3314 (N-H), 3160 (O-H), 2926 (C-H), 1653 (C = O), 1530 (C = C), 1276 (C-O) Element Analysis for C₂₁H₁₉NO₃S (%): C 69.02, H 5.24, N 3.83 found C 69.11, H 5.18, N 3.84. MS: m/z calculated for C₂₁H₁₉NO₃S+Na⁺ ([M+ Na⁺]), 388.10; found, 388.0961.

2.2.2 Sensor 1

Compound S2 (0.30 g; 0.77 mmol), dansyl chloride (0.17 g; 0.63 mmol) and potassium carbonate (0.14 g; 1.01 mmol) were combined and refluxed at 150°C in CH₃CN (15 mL). After refluxing under N₂ atmosphere for 8 h, the reaction mixture was poured into water and extracted with CH₂Cl₂ (three times). The organic layer was dried with anhydrous Na2SO4. The resulting product was purified by column chromatography using 9:1 CH₂Cl₂/EtOAc as an eluent, affording a yellow solid of sensor 1 (0.10 g, 21.28%). ¹H NMR, ¹³C NMR and ESI-MS spectra are shown in Figure S1, S2 and S3. M.p. 138-140°C ¹H NMR (400 MHz, DMSOd6): δ 2.44 (s, 3H), 2.84 (s, 6 H), 4.45 (s, 2 H), 6.62 (t, 2 H), 6.80 (d, 1H), 7.00 (t, 1H), 7.22 (m, 4 H), 7.42 (m, 7 H), 7.86 (t, 2 H), 8.46 (d, 1H), 9.38 (s, 1H). ¹³C NMR (100 MHz, DMSO): δ 165.9, 153.5, 147.5, 134.9, 134.0, 132.5, 132.4, 132.0, 131.7, 131.4, 130.3, 129.8, 129.6, 129.2, 128.8, 128.0, 127.5, 125.8, 123.7, 123.1, 121.7, 120.3, 119.7, 115.7, 113.2, 111.4, 67.2, 60.5, 45.7, 29.8, 21.1, 16.8, 14.2 FT-IR (KBr, cm⁻¹) v 3395 (N-H), 2917 (C-H), 1693 (C = O), 1530 (C = C), 1368 (S = O), 1286 (C-O), 858 (S-O). Element analysis for C₃₃H₃₀N₂O₅S₂ (%): C 66.22, H 5.34, N 4.44 found C 66.34, H 5.07, N 4.66. MS: m/z calcd for C₃₃H₃₀N₂O₅S₂-H⁺ ([M-H⁺]), 599.17; found, 599.1632.

3. Results and discussion

3.1 Design of the sensor 1 structure

The structure of sensor **1** mainly consisting of a biphenolic skeleton connecting with dansyl and 2-chloro-*N*-(4-(methylthio)phenyl)acetamide derivatives was a rigid one that yielded a good optical signal change upon binding to anions. For the purpose of binding mechanism, an acidic proton of the –NH amide group was deprotonated by strong basic anion. Deprotonating of sensor **1** may induce the delocalised electrons within the molecules, resulting in a significant change of colorimetric and fluorescence emission signals.

For the synthesis of sensor 1, there were three main steps as shown in Scheme 1. 4-thiomethyl aniline and chloroacetyl chloride were used as starting compounds. The mechanism of compound S1 is nucleophilic substitution. It was found that the compound **S1** has a sharp singlet proton peak of CH₂ group as a characteristic peak. Then, compound S2 was obtained by the condensation reaction of compound **S1** and biphenol by using K_2CO_3 as a base. The percent yield was quite low because the compound **S2** was a minor product. For ¹H NMR spectrum, the methylene proton peak shifted to 4.64 ppm and aromatic protons shited towards downfield, indicating a compound S2 formation. Lastly, the coupling reaction of compound S2 with dansyl chloride was refluxed at 150°C in CH₃CN solvent to obtain sensor 1 as a major product. The asymmetric structure of sensor 1 consists of 4-methythio aniline as a binding site of anion and dansyl moiety as a signalling unit.

3.2 Anion sensing properties of sensor 1 using ¹H NMR studies

¹H NMR spectra of sensor **1** (5 × 10⁻³ M) with different anions (F⁻, Cl⁻, Br⁻, H₂PO₄⁻, CH₃COO⁻ and C₆H₅COO⁻) were studied in DMSO-d₆ as shown in Figure 1. In the case of fluoride ion, the NH peak of sensor **1** disappeared via deprotonation reaction [14–16]. Meanwhile, the triplet peak of HF₂⁻ species was found at 16.11 ppm as shown in Figure S4. In addition, the CH₂ proton peak shifted towards downfield from 4.45 to 4.59 ppm due to a decrease in electron density. The aromatic protons of sensor **1** also shifted upfield and downfield due to the anisotropic effect. In the case of other anions (Cl⁻, Br⁻, H₂ PO₄⁻, CH₃COO⁻ and C₆H₅COO⁻), the NH peak of sensor **1** was moved slightly downfield due to weak hydrogen bonding. The chemical shifts of aromatic protons



Figure 1. ¹H NMR spectra of sensor 1 (A) in DMSO- d_6 (5 × 10⁻³ M) upon addition of 0.02 M of F⁻ (B), Cl⁻ (C), Br⁻ (D), H₂PO₄⁻ (E), CH₃ COO⁻ (F), C₆H₅COO⁻ (G).



Figure 2. UV-visible absorption spectra of sensor 1 (1×10^{-4} M) in DMSO upon addition of F⁻ ion in the range of 0–0.13 M.

between sensor 1 and other anions. These results suggest that sensor 1 had high selectivity towards fluoride ion, which is ascribed to a small, strong basicity and high charge density of the F^- ion compared to other anions.

3.3 Anion sensing properties of sensor 1 using UV-visible studies

The sensing performance of sensor **1** towards anions was investigated by monitoring the spectral changes in

dimethyl sulfoxide (DMSO). As seen in Figure 2, two absorption bands were observed at 270 and 360 nm, respectively, for sensor **1**. Upon incremental addition of F^- ion, the absorbance at 270 and 360 nm was decreased and at the same time a new shoulder peak at 300 nm gradually evolved. This result suggests the presence of

a deprotonated form of sensor **1** [17–19] due to the binding interaction of sensor **1** against fluoride ion. For other anions (Cl⁻, Br⁻, H₂PO₄⁻, CH₃COO⁻ and C₆H₅COO⁻), the absorbance at 270 and 360 nm was slightly decreased and no shoulder peak was observed because of the weak hydrogen bonding interaction between



Figure 3. UV-visible absorption spectra of sensor **1** (1×10^{-4} M) in the presence of 0.13 M of various anions (F^- , CI^- , Br^- , $H_2PO_4^-$, CH_3 COO⁻, $C_6H_5COO^-$) in DMSO.



Figure 4. The absorbance at 300 nm for sensor 1 with various anions.



Figure 5. Colour change of sensor **1** (5×10^{-3} M) in DMSO- d_6 upon addition of 0.02 M of various anions: sensor **1** (A), F⁻ (B), Cl⁻ (C), Br⁻ (D), H₂PO₄^{-,} (E), CH₃COO⁻ (F) and C₆H₅COO⁻ (G).

sensor 1 and the anions. Figure 3 and Figure 4 show that sensor 1 gave a significant enhancement in absorbance at 300 nm upon addition of F^- ion, but sensor 1 with other anions exhibited the similar range in absorbance compared to pure sensor 1 in DMSO. Moreover, sensor 1 displayed a colour change from yellow to colourless with the presence of F^- ion as shown in Figure 5. On the contrary, the solution colour of sensor 1 remained yellow upon addition of other anions.

The selectivity study of sensor $\mathbf{1}$ to F^- ion under the competitive condition was further investigated. The result in Figure 6 shows that there was no significant change in absorbance at 300 nm when sensor $\mathbf{1}$ was

treated with fluoride ion and other competing anions. It is clearly seen that sensor **1** possessed high selectivity towards fluoride ion and its sensing performance was not interfered by other anions.

3.4 Anion sensing properties of sensor 1 using fluorescence studies

The sensing performance of sensor 1 against anions in DMSO were evaluated by fluorescence titration. The two fluorescence emissions of sensor 1 were observed at 440 and 564 nm. In the titration with F^- ion, the emission band at around 434 nm was enhanced with a notable blue shift; meanwhile, an emission band at 564 nm was



Figure 6. The absorbance at 300 nm of sensor $1(1 \times 10^{-4} \text{ M})$ in the presence of F⁻ and other competing anions in DMSO.



Figure 7. Fluorescence emission spectra of **sensor 1** (1×10^{-5} M) in DMSO upon addition of F⁻ ion in the range of 0–2.67 × 10^{-4} M.

decreased (Figure 7), suggesting the presence of a deprotonated form of sensor **1** [12,20,21]. Under UV light at 365 nm, sensor **1** with the presence of F^- ion displayed the colour change from orange-yellow to blue. In the case of other anions, the emission band at 440 nm slightly changed and, at 564 nm, was decreased in **Figure S5**. There was no colour change observed upon addition of other anions in Figure 8.

The association constant obtained from the nonlinear fitting was calculated to be $6.86 \times 10^5 \text{ M}^{-1}$ [22] as shown in **Figure S6**. The detection limit of sensors **1** with F^- ion measured by fluorescence titration at 564 nm as previously reported [23] was found to be 18 μ M. These results demonstrate that sensors **1** could detect F^- ion at low concentration level. Moreover, the sensing performance of sensor **1** in detecting F^- ion was compared to other molecular sensors summarised in Table 1.

3.5 Application for visual detection of fluoride ion on TLC plate

For the extensive applicability of sensor **1**, test strips were prepared by immersing sensor 1 (5×10^{-4} M) on the TLC plate and filter paper in DMSO. In Figure 9, it was found that sensor **1** gave the colorimetric response to F⁻ ion at the concentration of 0.05 M. The sensor **1** displayed a distinct change in colour on the TLC plate from green to blue and on the filter paper from brown to blue. The sensor **1**-coated on the TLC plate and filter paper can be used as an efficient and convenient tool for rapid detection of F⁻ ion.

4. Conclusion

The new sensor **1** based on a biphenolic-dansyl derivative has been achievably synthesised. The binding behaviour of sensor **1** in detecting F⁻ ion was predominantly evidenced by UV-visible, fluorescence and ¹H NMR techniques. For the visual detection, sensor **1** displayed the colour change from yellow to colourless under the



Figure 8. Fluorescence change of sensor **1** (5 × 10⁻³ M) in DMSO- d_6 upon addition of 0.02 M of various anions on excitation at 365 nm using UV lamp: blank (A), F⁻ (B), Cl⁻ (C), Br⁻ (D), H₂PO₄⁻ (E), CH₃COO⁻ (F) and C₆H₅COO⁻ (G).

Table 1.	Comparison	of selected	molecular	sensors with	sensor 1	for F ⁻	ion detection.
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			Detection limit		
No.	Name of sensors	Solvent medium	(µM)	Ref.	
1	1-(4-fluorophenyl)-3-((1-(2-hydroxyphenyl)-1H-1,2,3-triazol-4-yl)methyl)urea	DMSO	65.8	[24]	
2	N-(2-(benzothiazole-2-yl)phenyl)-4-nitrobenzamide	DMSO	58.0	[25]	
3	6,12-Diphenyl-5, 11-dihydroindolo[3,2- <i>b</i>]carbazole	CH₃CN	23.3	[26]	
4	sensor 1	DMSO	18.0	This work	



Figure 9. Fluorescence image (under 365 nm UV light) of sensor 1 (5×10^{-4} M) adsorbed on a TLC plate (a) and filter paper (b) with a spot of F⁻ ion solution on sensor 1: sensor 1(A), sensor 1 with 2.50×10^{-3} M of F⁻ (B), sensor 1 with 0.005 M of F⁻ (C), sensor 1 with 0.0075 M of F⁻ (D), sensor 1 with 0.01 M of F⁻ (E), sensor 1 with 0.025 M of F⁻ (F), sensor 1 with 0.05 M of F⁻ (G), sensor 1 with 0.10 M of F⁻ (H), sensor 1 with 0.25 M of F⁻ (I) and sensor 1 with 0.50 M of F⁻ (J).

naked eye and from orange-yellow to blue under UV light 365 nm upon addition of F⁻ ion. Moreover, the absorption spectrum of sensor 1 displayed a new shoulder peak at 300 nm, and also the emission spectrum showed a shift from 440 to 434 nm, which was ascribed to the presence of a deprotonated form of sensor 1. For ¹H NMR study, sensor 1 showed the disappearance of the amide proton signal due to the deprotonation reaction. This phenomenon, again, confirmed the existence of a deprotonated form. The association constant and limit of detection between sensor 1 and F⁻ ion were determined as 6.86×10^5 M⁻¹ and 18 µM, respectively. In addition, sensor 1-based test on the TLC plate and filter paper was utilised as a simple tool for F⁻ ion detection by the naked eye.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Conflicts of interest

The authors declare no conflict of interest.

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