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## A simple dual-channel sensor for detecting cyanide in water with high selectivity and sensitivity

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#### ABSTRACT

Taking advantage of the special nucleophilicity of cyanide, a new simple colorimetric chemosensor has been synthesised. This allows a deprotonation reaction to monitoring the cyanide. With the addition of CN<sup>-</sup> to the chemosensor aqueous solution, which could induce a change in the solution colour from yellowish to deep yellow, while no colour change could be observed in the presence of other hackneyed anions, by which CN<sup>-</sup> can be distinguished from other anions immediate with the naked eye. At the same time, a fluorescence quenching was implemented upon adding cyanide into the chemosensor aqueous solution. The absorption spectra detection limits of the chemosensor for cyanide was  $5.35 \times 10^{-8}$  M and the fluorescence spectra detection limit was  $2.63 \times 10^{-8}$  M. The cyanide test strips based on the chemosensor could serve as a convenient cyanide test kits. Furthermore, the chemosensor was successfully applied to detect cyanide in sprouting potatoes.

#### ARTICLE HISTORY

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#### KEYWORDS

Cyanide; high sensitivity; specific selectivity; test kits; sprouting potatoes

#### 1. Introduction

Because of the significance of anions in surroundings, development biological and the industrial processes, of optical chemosensors for anions has constantly been an area where widespread interest (1-6). The sensitive and selective discovery of cyanide is growing to a pressing undertaking, owing to the high toxicity of cyanide in department of biology, and also the sustainable environment issue undermined by its extensive use in industry (7-10). The cyanide of thereof salt are ubiguitous chemical substances found in earth's surface water not only from industrial waste but also from biological systems (11). The maximum permissible standard of CN<sup>-</sup> in potable water is only 1.9 mM in the light of the World Health Organization (WHO) (12). For the purpose of minimising the obstruction of additional anions, many nucleophilic addition reaction mechanisms in view of cyanide anion sensors have been already reported (13-22). The characteristic of its UV and fluorescence response for cyanide anion detection in purely organic phase limited its practical application under the state of the aqueous needed (23-28).

So far, a good deal of sensors for  $CN^-$  have been synthesised and found. However, a significant portion of these sensors for  $CN^-$  are dependent on hydrogen bonding motive, and consequence they typically show poor selectivity with respect to other anions (such as F<sup>-</sup> and CH<sub>3</sub>COO<sup>-</sup>) (29–31). In order to overcome this factor, a number of other testing methods for CN<sup>-</sup> have been designed and investigated by forthputting nucleophilic reaction of CN<sup>-</sup> (32–35). These methods, whatsoever, easily lead to worse selectivity in other popular anions. The rapidly developing area of reaction-based transducer has made progress in this field due to the unique features of CN<sup>-</sup> by means of wide variety of organic functional groups, including -C=O-, -C=N- and -C=C-(36–40). Here we report a new chemosensor **FC** based on deprotonation mechanism that exhibits sensitive and selective detection of CN<sup>-</sup> under mild conditions. The reaction-based system relies on cyanide-induced chemosensor utilise deprotonation reactions and electric charge transferring.

In view of the above, and as a part of our research interest in ions recognition (41–54), we attempt to synthesise a large number of nucleophilic reactions to acquire efficient optical chemosensor which can sense  $CN^-$  with categoric selectivity and high sensitivity. Till this moment, optical chemosensor detection of  $CN^-$  in aqueous phase still remains limited. Reasonable alternative strategies customarily use by some researchers for the detection of  $CN^-$  apply reaction-based pointers (55, 56). This typically provides improved selectivity regarding  $CN^-$  detection because of the nucleophilic character of it. With these considerations in mind, we report here a simple synthesised



body compound **FC** that exhibits a selective, colorimetric and response towards cyanide anion in aqueous.

#### 2. Experimental

#### 2.1. Instruments and reagents

All reagents for synthesis were of analytical grade, commercially and were used without further purification. All the anions were added in the form of tetrabutylammonium (TBA) salts which were purchased from Sigma-Aldrich Chemical, stored in a vacuum desiccators. Melting points were measured on an X-4 digital melting point apparatus and were uncorrected. UV-vis spectra were recorded on a Shimadzu UV-2550 spectrometer. Fluorescence spectra were recorded on a Shimadzu RF-5310. <sup>1</sup>H NMR spectra were recorded on a Mercury-600BB spectrometer at 600 MHz and <sup>13</sup>C NMR spectra were recorded on a Mercury-600BB spectrometer at 150 MHz with DMSO- $d_{\epsilon}$  as solvent. Chemical shifts are reported in ppm downfield from tetramethylsilane (TMS,  $\delta$  scale with solvent resonances as internal standards). Mass spectra were performed on a Bruker Esquire 3000 plus mass spectrometer (Bruker-Franzen Analytik GmbH Bremen, Germany) equipped with ESI interface and ion trap analyser.

#### 2.2. Synthesis of the chemosensor FC

Compound FC can be readily prepared by a simple reaction between 3-hydroxy-2-naphthoic acid and 4-(diethylamino) salicylaldehyde (Scheme 1). 3-hydroxy-2-naphthoic acid (0.94 g, 5 mmol) and 98% H<sub>2</sub>SO<sub>4</sub> (30 µL, 10 mol%) was reflux in 40 mL methanol for 8 h at 65 °C to give 2-hydroxy-3-naphthoate. Soon afterwards, 2-hydroxy-3-naphthoate (2.02 g, 10 mmol) and hydrazine monohydrate (0.5 g, 10 mmol) were mixed in 30 mL ethanol stirred under reflux for 24 h at 80 °C. After cooling to room temperature, poured into 200 mL distilled water, the yellow precipitate was filtered, washed three times with hot absolute ethanol, and then got the compound 3-hydroxy-2-naphthohydrazide. Then 3-hydroxy-2-naphthohydrazide (1.01 g, 5 mmol), 4-diethylaminosalicylaldehyde (0.97 g, 5 mmol) and catalytic amount of acetic acid (AcOH) were combined in absolute ethanol (30 mL). The solution was stirred under reflux at 80 °C for 36 h. After cooling to room temperature, the yellow precipitate was filtered, washed three times with hot absolute ethanol, then recrystallised with EtOH to give a yellow powder product FC (1.45 g) in 73.4% yield (m.p. 262–265 °C). <sup>1</sup>H NMR (600 MHz, DMSO-*d<sub>c</sub>*) δ 11.95 (s, 1H), 11.36 (s, 1H), 10.07 (d, J = 132.3 Hz, 1H), 8.48 (d, J = 7.3 Hz, 2H), 7.90 (t, J = 16.6 Hz, 1H), 7.76 (t, J = 13.5 Hz, 1H), 7.51 (d, J = 27.2, 19.7 Hz, 1H), 7.38 (t, J = 7.4 Hz, 1H), 7.30 (d, J = 33.8 Hz, 1H), 7.24 (t, J = 12.6 Hz, 1H), 6.31 (d, J = 8.7 Hz, 1H), 6.16 (s, 1H), 3.43–3.32 (m, 4H), 1.08 (t, J = 60.8, 6.7 Hz,



**Figure 1.** (Colour online) (a) UV–vis spectra of **FC** ( $c = 2 \times 10^{-5}$  M) after addition of 50 equiv. of various anions. (b) UV–vis colour changes of **FC** ( $c = 2 \times 10^{-5}$  M) after addition of 50 equiv. of various anions ( $c = 1 \times 10^{-2}$  M).

6H). <sup>13</sup>C NMR (151 MHz, DMSO-*d*<sub>6</sub>) δ 160.23 (s), 154.89 (s), 150.91 (d, *J* = 25.3 Hz), 136.29 (s), 132.10 (s), 130.29 (s), 129.07 (s), 128.64 (s), 127.16 (s), 126.29 (s), 124.21 (s), 120.04 (s), 111.03 (s), 106.78 (s), 104.23 (s), 97.88 (s), 44.27 (s), 13.00 (s). IR (KBr, cm<sup>-1</sup>) *v*: 3416 (–OH), 1631 (C=O), 1496 (CH=N). ESI-MS *m/z*: (M+H<sup>+</sup>) Calcd for C<sub>22</sub>H<sub>24</sub>N<sub>3</sub>O<sub>3</sub> 378.1812; Found 378.14.

#### 2.3. General procedure for UV-vis experiments

All the UV–vis experiments were recorded on a Shimadzu UV-2550 spectrometer after the addition of tetrabutylammonium salts in DMSO/H<sub>2</sub>O (2:8, v/v) solution. While keeping the ligand concentration constant ( $2.0 \times 10^{-5}$  M) in all experiments. Solutions of anion were prepared from the perchlorate salts of F<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, l<sup>-</sup>, AcO<sup>-</sup>, H<sub>2</sub>PO<sub>4</sub><sup>-</sup>, HSO<sub>4</sub><sup>-</sup>, ClO<sub>4</sub><sup>-</sup>, CN<sup>-</sup> and SCN<sup>-</sup>.

### 2.4. General procedure for fluorescence spectra experiments

All fluorescence spectra were recorded on a Shimadzu RF-5301 fluorescence spectrometer after the addition of tetrabutylammonium salts in DMSO/H<sub>2</sub>O (2:8, v/v) solution while keeping the ligand concentration constant



**Figure 2.** (Colour online) (a) Fluorescence spectra ( $\lambda_{ex} = 370$  nm) of **FC** ( $c = 2 \times 10^{-5}$  M) after addition of 50 equiv. of various anions. (b) Fluorescent colour changes of **FC** ( $c = 2 \times 10^{-5}$  M) after addition of 50 equiv. of various anions.

 $(2.5 \times 10^{-5} \text{ M})$  in all experiments. The excitation wavelength was 370 nm. Solutions of anion were prepared from the tetrabutylammonium salts of F<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>, AcO<sup>-</sup>, H<sub>2</sub>PO<sub>4</sub><sup>-</sup>, HSO<sub>4</sub><sup>-</sup>, ClO<sub>4</sub><sup>-</sup>, CN<sup>-</sup> and SCN<sup>-</sup>.

#### 3. Results and discussion

The synthesis process of chemosensor FC is shown in Scheme 1. The chemosensor FC has been characterised by <sup>1</sup>H NMR, <sup>13</sup>C NMR and ESI-MS (Figure S1–S3). The sensing abilities of FC towards various anions (F<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, l<sup>-</sup>, AcO<sup>-</sup>,  $H_2PO_4^-$ ,  $HSO_4^-$ ,  $CIO_4^-$ ,  $CN^-$  and  $SCN^-$ ) were investigated by UV-vis and fluorescence spectroscopy. When adding the DMSO solution of these anions (50 equiv.,  $c = 1 \times 10^{-2}$  M) to the solution of chemosensor FC ( $c = 2 \times 10^{-5}$  M, DMSO/H<sub>2</sub>O, 2:8) respectively, only the addition of CN<sup>-</sup> could induced a appear new peak at 396 nm instantly. Simultaneously (Figure 1a), a colour change from yellowish to dark yellow was observed by the naked eye (Figure 1b). In the corresponding UV-vis spectra, the UV absorption band in the 320–450 nm range showed an obvious strengthen and in good agreement with this colour change. We also carried out a series of host-guest recognition in fluorescence spectroscopy experiments. The recognition profiles of chemosensor FC towards various anions, and only the CN<sup>-</sup> added the fluorescence emission band in



**Figure 3.** (Colour online) (a) UV–vis spectra of **FC** ( $c = 20 \mu$ M) upon the addition of pure water solution of CN<sup>-</sup>. (Inset: UV–vis colour changes of **FC** ( $c = 2 \times 10^{-5}$  M) after addition of 50 equiv. of CN<sup>-</sup>). (b) Fluorescence spectra ( $\lambda_{ex} = 370$  nm) of **FC** ( $c = 20 \mu$ M) upon addition of CN<sup>-</sup>. (Inset: Fluorescent colour changes of **FC** ( $c = 2 \times 10^{-5}$  M) after addition of 50 equiv. of CN<sup>-</sup>.)

the 450–650 nm range showed an obvious decrease and none of these ions induced fluorescence quenching in the fluorescence spectra of the chemosensor (Figure 2a). The apparent fluorescence emission change could be distinguished by the naked eye (Figure 2b). All examined anions including more basic and nucleophilic ions such as F<sup>-</sup> and SCN<sup>-</sup> didn't cause any similar colour and spectra changes. These results suggested that chemosensor **FC** displayed an excellent selectivity for CN<sup>-</sup> than other anions.

The further investigate  $CN^-$  recognition properties of **FC** by UV–vis and fluorescence titration experiments. Figure 3 shows the family of absorption and emission spectra obtained over the course of the titration of chemosensor **FC** with the water solution of cyanide. In UV–vis spectra (Figure 3a), with the gradual addition of  $CN^-$ , the absorption in the range of 320–450 nm was significantly



**Figure 4.** (Colour online) (a) Ratiometric absorption and (b) fluorescence intensity changes of **FC** ( $c = 2 \times 10^{-5}$  M) upon addition of 50 equiv. of CN<sup>-</sup> ( $c = 1 \times 10^{-2}$  M) and 50 equiv. of various interference anions ( $c = 1 \times 10^{-2}$  M) in DMSO/H<sub>2</sub>O (2:8, v/v).

enhanced, while the absorption in the range of 250– 320 nm markedly increased similarly. Concomitantly, with the gradual addition of CN<sup>-</sup> to chemosensor **FC**, a kind of gradually weakened phenomenon in the fluorescence intensity in 450–650 nm range was observed (Figure 3b). In other words, the alone chemosensor **FC** was emission as a yellow fluorescence, when it encountered CN<sup>-</sup>, it resulted in a dramatic fluorescence quenching of the fluorescence intensity. In the meantime, the detection limits of the **FC** for CN<sup>-</sup> calculated on the basis of  $3\sigma/S$  (Figures S4 and S5) were  $2.63 \times 10^{-8}$  M for fluorescence and  $5.35 \times 10^{-8}$  M for absorption spectra change, respectively, which are both far lower than the WHO guideline of CN<sup>-</sup> in drinking water (less than  $1.9 \times 10^{-6}$  M).

To make a thorough inquiry of the utility of chemosensor **FC** as an ion-selective chemosensor for CN<sup>-</sup>,



**Figure 5.** (Colour online) (a) Influence of pH on the UV–vis spectra of **FC** and  $CN^-$  in DMSO/H<sub>2</sub>O (v/v = 2:8). (b) Influence of pH on the fluorescence of **FC** and  $CN^-$  in DMSO/H<sub>2</sub>O (v/v = 2:8).

competitive experiments were carried out in the presence of 50 equiv. of  $CN^-$  and 50 equiv. of various other ions (F<sup>-</sup>,  $CI^-$ , Br<sup>-</sup>, I<sup>-</sup>, AcO<sup>-</sup>, H<sub>2</sub>PO<sub>4</sub><sup>-</sup>, HSO<sub>4</sub><sup>-</sup>, CIO<sub>4</sub><sup>-</sup> and SCN<sup>-</sup>) with the absorption and fluorescence spectra in the mixed solvent (DMSO/H<sub>2</sub>O, 2:8) (Figure 4). The results of these studies have revealed that these competing ions exerted no or little influence on the UV absorption (Figure 4a) and fluorescence emission spectra (Figure 4b) of chemosensor **FC** with CN<sup>-</sup>, which further indicated that **FC** has specific selectivity to CN<sup>-</sup>.

The selectivity of **FC** to  $CN^-$  was examined in absorption and fluorescence spectra in the mixed solvent (DMSO/H<sub>2</sub>O, 2:8) over a wide range of pH values. However, the UV-vis absorption and fluorescence emission of **FC** displayed a strong pH dependence, as illustrated by its absorption spectrum at 396 nm and fluorescence emission at 502 nm. An intense and stable absorption of **FC** in the pH range 5.0–14.0 period the binding of **FC** with  $CN^-$  occurred in UV-visible absorption spectrum and effectively between



Figure 6. (Colour online) The <sup>1</sup>H NMR titration and partial spectra of compound FC in DMSO-d<sub>6</sub> upon addition of varying amounts CN<sup>-</sup>.

pH from 4.0 to 14.0 in fluorescence emission spectrum (Figure 5). Thence the detection of  $CN^-$  can work well in the range of pH 5.0–14.0.

To gain better insight into the sensing mechanism (Scheme 2) of chemosensor **FC** to  $CN^-$ , the <sup>1</sup>H NMR titration, <sup>13</sup>C NMR spectra, IR spectrum and ESI-MS were carried out. In the <sup>1</sup>H NMR titration spectra (Figure 6), before the addition of cyanide anions, the <sup>1</sup>H NMR chemical shifts of the O-H<sub>a</sub>, O-H<sub>b</sub> and -NH<sub>c</sub> protons on **FC** were at  $\delta$  13.71, 10.01 and 10.07 ppm. With the addition  $CN^-$ , the hydroxy proton H<sub>a</sub> and H<sub>b</sub>, -NH– proton H<sub>c</sub> completely disappeared by the deprotonation. Upon the gradual addition of CN<sup>-</sup>, all aromatic protons exhibited an upfield shift on different



**Figure 7.** (Colour online) Left: colour change of test strips upon addition of  $CN^-$ . Right: the detection of test strips upon addition of  $CN^-$  under an UV lamp at 365 nm.

levels, which suggest the increase in the electron density in naphthalene ring and phenyl ring through charge delocalisation in the conjugated system. Meanwhile, in order to further illustrate the problem, we did a <sup>13</sup>C NMR characterisation of FC and CN<sup>-</sup>. When added 0.5equiv. CN<sup>-</sup> to **FC** (10 mg) in 5 mL DMSO- $d_{6'}$  the carbons of naphthalene ring and benzene ring both shifts to high-field can be clearly observed (Figure S6). This further illustrates that due to the deprotonation effect, causing an aromatic ring electron density increases, so that it moves to the high field. In addition, we also did the infrared spectroscopy experiments to prove mechanism. In the IR spectrum of FC, the stretching vibration absorption peaks of hydroxyl O-H and HC=N appeared in 3416 and 1496 cm<sup>-1</sup>, respectively. However, when FC met with CN<sup>-</sup>, the stretching vibration peaks of hydroxy (O-H) and HC=N absorption miraculously disappeared (Figure S7). This is clear proof of the FC encounter CN<sup>-</sup> occurred deprotonation effect. Moreover, from the ESI-MS data, we can also see an obvious peak at m/z 468.9601 assignable to [FC-3H<sup>+</sup> + 3Na<sup>+</sup> + HCN-H] (m/z called = 369) (Figure S8). This similarly supports the deprotonation phenomena of FC. These common phenomenon could be explained from the propose sensing mechanism show in Scheme 2.

Motivated by the favourable CN<sup>-</sup> recognition properties of chemosensor **FC**, we prepared test strips by immersing filter papers into the DMSO/H<sub>2</sub>O (2:8) solution of chemosensor **FC** ( $1 \times 10^{-3}$  M) and then dried them in air. The test



**Figure 8.** (Colour online) (a) UV–vis absorption for the chemosensor **FC** to detect cyanide in sprouting potato. Inset: colour changes observed for **FC** upon the addition of cyanide-containing and photograph of sprouting potato. (b) Fluorescence emission data for the chemosensor **FC** to detect cyanide in sprouting potato. Inset: colour changes observed for **FC** upon the addition of cyanide-containing under the UV lamp and photograph of sprouting potato.

strips could conveniently detect CN<sup>-</sup>, similar to that commonly used for the pH measurement. Upon the addition of CN<sup>-</sup> solutions to the test strips, the test strips show obvious colour change from grey to yellow, meanwhile, under the UV-lamp, the chartreuse fluorescence were disappear that can be clearly observed (Figure 7). Therefore, the test strips of **FC** have excellent application value in detection of CN<sup>-</sup>.



Scheme 2. (Colour online) A possible sensing mechanism of the chemosensor FC to CN<sup>-</sup>.

It is worth mentioning that we also investigated the practical utilities of the sensor to detect CN<sup>-</sup> in the sprouting potatoes. The sprouting potato (173 g) was first mashed before being soaked in water (230 mL) for 6 days until the extract became muddy. The mixture was filtered and the filtrate was washed with 80 mmol/L NaOH solution (120 mL) to get the cyanide-containing solution. As shown in Figure 8, upon the addition cyanide-containing solution into **FC**, an obvious colour change from colourless to yellow can be directly observed by the naked eyes (Figure 8a). And with imagine the unanimous yellow fluorescent disappear under the UV lamp confirming that **FC** is a promising CN<sup>-</sup> probe for practical applications (Figure 8b).

#### 4 Conclusions

In summary, we have research and development of a novel CN<sup>-</sup> chemosensor, which could through UV 'turn-on' and fluorescent quenching detect CN<sup>-</sup> with specific selectivity and high sensitivity. The recognition process based on cyanide induced a kind of deprotonation reaction mechanism. It's worth noting that the competitive anions did not afford any obvious interference response. The absorption spectra detection limits of the **FC** for CN<sup>-</sup> was  $5.35 \times 10^{-8}$  M and the fluorescence spectra detection limit was  $2.63 \times 10^{-8}$  M, which is far lower than the WHO guideline of CN<sup>-</sup> in



drinking water (less than  $1.9 \times 10^{-6}$  M). Moreover, test strips based on this chemosensor were fabricated, which could serve as practical colorimetric and fluorescence test kits to detect CN<sup>-</sup> for simple and fast measurement and the chemosensor **FC** is a good way to detect cyanide aqueous extracts of sprouting potatoes. The work shown here not only presents the fact of using recognition towards cyanide, but more importantly, it implicates the potential of life applications for colorimetric chemosensor towards other ions with deprotonated properties.

#### **Supplemental material**

Supplemental data for this article can be accessed online here: http://dx.doi.org/10.1080/10610278.2016.1151019

#### **Disclosure statement**

No potential conflict of interest was reported by the authors.

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