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Article

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A Water Soluble Naphthalimide-Based Chemosensor for Fluorescent Detection CN^- in Pure Water and Its Application in Practical Samples

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By rationally introducing aromatic carboxyl functionalized 1,8-naphthalimide, a water soluble fluorescent chemosensor (DA) was successfully synthesized. It could selectively and sensitively detect CN^- in pure water via an intramolecular charge transfer to twisted intramolecular charge transfer (ICT-TICT) state change mechanism. The detection limit of the chemosensor DA to CN^- was 1.38×10^{-8} mol L^{-1} which was lower than 1.9 µmol L^{-1} (set by the World Health Organization (WHO)). Notably, DA displayed rapid response (about 1 s) for recognizing CN^- in pure water. Furthermore, DA could be applied to monitoring CN^- in tap water. Meanwhile, we prepared the test strips based on DA, which could rapidly and efficiently detect CN^- in water.

Keywords: water-soluble chemosensor, fluorescent, 1,8-naphthalimide derivative, CN^- recognition

Introduction

Cyanide (CN^-) salts are extensively used in industry, such as electric plating, gold mining, plastic production, and other areas.[1] However, cyanide is greatly toxic to mammals with even a small amount of this species, leading to emesis, unconsciousness, and finally to death.[2] Therefore, cyanide concentrations in drinking water must be lower than 1.9 µmol L^{-1}.[3] In this regard, the reasonable design and synthesis of effective sensors to selectively detect cyanide anion at the environmental and biological levels have received a lot of attention.[4]

Fluorescence sensors for specific anions play a fundamental role in medical, environmental and biological applications, what is more, it is one of the best tools with high sensitivity, rapid response and easy performance, much interest has been focused on the design and synthesis of fluorescent chemosensors for anions.[5]

To date, much effort has been drawn to develop the fluorescent chemosensors for CN^-. However, most of them worked properly in a high percentage of organic solvent media for the detection of cyanide ions.[6] As we know, the use of organic solvents usually damages the normal function of biomolecules, thus, good water solubility are essential for practical use of the chemosensors in biochemical research.[7] Although some of them could work in pure water,[8] they did not display enough sensitivity for the maximum allowable level (1.9 µmol L^{-1}) of CN^- in drinking water. Thus, it is highly challenging to develop new soluble fluorescent chemosensors for sensing CN^- with high sensitivity and excellent selectivity in 100% water solutions.

1,8-Naphthalimide derivatives are an excellent environmentally sensitive fluorophore compounds, which is extensively applied to various fields of science and technology.[9] As a consequence of their strong blue fluorescence and good photostability, 1,8-naphthalimide derivatives are often used for fluorescence sensors, switchers and ion probes.[10] However, the application of naphthalimide sensors was limited due to poor solubility. We all know that carboxyl not only has a remarkable coordination ability,[11] but also has an excellent hydrophilicity.

With these considerations in mind and based on our research interest in ions recognition,[12] we designed and synthesized a novel, low-cost and water soluble chemosensor (DA) for fluorescent detecting cyanide ions by rationally introducing aromatic carboxyl functionalized

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1,8-naphthalimide. As expected, chemosensor DA displays high sensitivity and rapid response (about 1 s) for recognizing CN^- in pure water system. Compared with most of the previously reported fluorescent chemosensors for CN^- and naphthalimide sensors,[13] the advantage of DA is that it could work in 100% water solutions. It is worth noting that compared with recently reported aromatic carboxyl functionalized 1,8-naphthalimide chemosensors, DA can be directly dissolved in pure water without changing COOH into COONa.

Experimental

Materials and apparatus

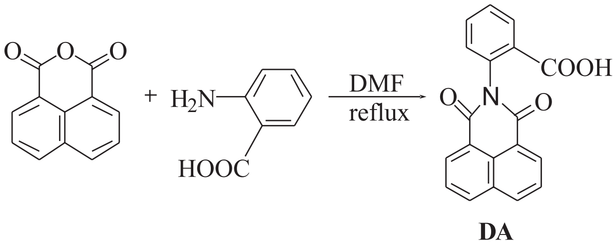
Fresh double-distilled water was used throughout the experiments. All other reagents and solvents (analytical grade) were commercially available and were used without further purification. ^{1}H nuclear magnetic resonance (NMR) spectra was recorded on an Agilent DD2 at 600 MHz spectra. ^{1}H chemical shifts are reported in ppm downfield from tetramethylsilane (TMS, δ scale with the solvent resonances as internal standards). Fluorescence spectra were recorded on a Shimadzu RF-5310 (Japan). Melting points were determined on an X-4 digital melting-point apparatus (Beijing Tech Instrument Co., Ltd., China) and were uncorrected. Mass spectra (MS) were recorded on a Bruker Esquire 3000 plus mass spectrometer (Bruker-Franzen Analytik GmbH, Bremen, Germany) equipped with ESI (electrospray ionization) interface and ion trap analyzer.

Synthesis of the chemosensor DA

Compound DA was synthesized by condensation reaction of anthranilic acid and 1,8-naphthalic anhydride (Scheme 1).[14] Anthranilic acid (0.55 g, 4.0 mmol) and 1,8-naphthalic anhydride (0.79 g, 4.0 mmol) were dissolved in absolute dimethylformamide (DMF, 20 mL) at 80 °C. The reaction mixture was stirred and refluxed for 7 h. The mixture was still clear liquid after cooling down to room temperature. White precipitate appeared after adding distilled water, through filtration, washed with ethanol and dried under vacuum, then obtained the product DA in 85% yield (mp > 300 °C).

Characterization data for DA

^{1}H NMR (600 MHz, DMSO-d_6) δ 12.85 (s, 1H, COOH), 8.49 (m, 4H, Nap-H), 8.08 (m, 1H, Ph-H), 7.89 (t, *J* 6 Hz, 2H, Nap-H), 7.75 (t, *J* 6 Hz, 1H, Ph-H), 7.60 (t, *J* 6.6 Hz, 1H, Ph-H), 7.50 (d, *J* 6 Hz, 1H, Ph-H). ESI-MS *m/z*: $[M + Na]^+$ calcd. for $C_{19}H_{11}NO_4Na$: 340.06; found: 340.01.



Scheme 1. Synthetic procedures of chemosensor DA.

General procedure for fluorescence spectra experiments

The stock solution of DA (0.2 mmol L^{-1}) in pure water (pH = 7.2) was prepared and stock at room temperature, which diluted to a concentration of 20 µmol L^{-1} as the determined solution. The aqueous solutions of anions (10 mmol L^{-1}) (F^-, Cl^-, Br^-, I^-, AcO^-, SO_4^-, $H_2PO_4^-$, ClO_4^-, CN^-, SCN^-, S^{2-} and OH^-) were prepared via sodium salts. The fluorescence spectra were obtained by excitation at 315 nm. The excitation and emission slit widths were both 5 nm.

General procedure for ^{1}H NMR experiments

For ^{1}H NMR titrations, sensor DA was prepared in DMSO-d_6 and NaCN was prepared in double-distilled water. First of all, only sensor DA in DMSO-d_6 was added into the NMR tube, and then CN^- anion was added at 0.1, 0.5, 1.0, 2.0 and 4.0 equiv. sequentially. All solutions were mixed directly in the NMR tube.

Practical samples analysis

For practical sample analysis, the tested water tap samples were prepared by adding known amounts of CN^-. The fluorescence spectrum of each sample was tested for three times, and the found concentration of CN^- was measured according to the developed calibration curve.

Preparation of the test strips

The test strips were carried out by submersing filter papers in the pure water solution of DA (2.0×10^{-4} mol L^{-1}) and then dried in air. The CN^- water solution (2.0×10^{-4} mol L^{-1}) and competitive anions water solution were dropped on the test strips. After drying, the images were recorded by a digital camera under UV lamp.

Results and Discussion

We researched the fluorescent response of chemosensor DA to various anions in 100% water solutions, including

F^-, Cl^-, Br^-, I^-, AcO^-, SO_4^-, $H_2PO_4^-$, ClO_4^-, CN^-, SCN^-, S^{2-} and OH^-. As shown in Figure 1, the maximum fluorescent emission band of free DA appeared at 390 nm. When 5 equivalents of CN^- was added to the pure water solution of chemosensor DA, the fluorescence emission band at 390 nm significantly reduced. The obvious color change from bright blue to colorless could be distinguished by naked-eyes through UV lamp (Figure 1). To further evaluate the selectivity of chemosensor DA, the same experiments were also conducted using F^-, Cl^-, Br^-, I^-, AcO^-, SO_4^-, $H_2PO_4^-$, ClO_4^-, SCN^-, S^{2-} and OH^-. Whether in the fluorescent spectra or by naked-eyes through UV lamp, nearly no obvious fluorescence responses were observed with these anions (Figure 2), demonstrating DA has excellent fluorescent selectivity toward CN^- in pure water.

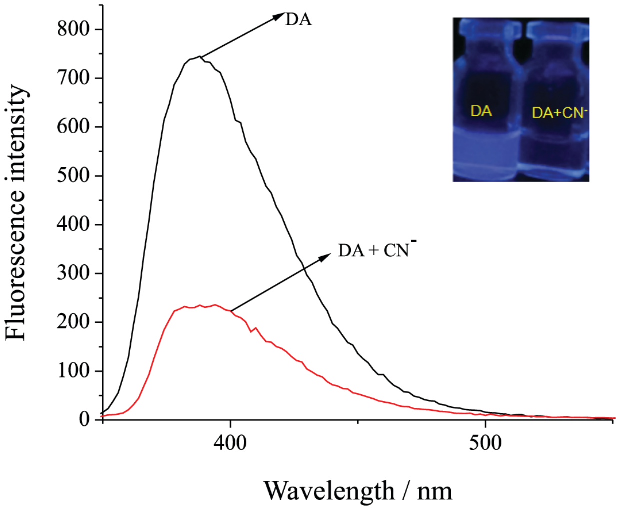


Figure 1. Fluorescence spectra of DA (20 µmol L^{-1}) in pure water media in the presence of CN^- (5 equiv.) (λex = 315 nm). Inset: color change of DA, DA + CN^- (5 equiv.) under UV lamp in pure water media.

Furthermore, the quantum yields of DA and DA + CN^- were calculated using the following equation 1:

$$Yu = (Ys \times Fu \times As) / (Fs \times Au) \quad (1)$$

where Yu is the quantum yield of DA and DA + CN^-; Ys is the quantum yield of reference compound (quinine sulfate, Ys = 0.55); Fu is the fluorescent integral of DA and DA + CN^-; Fs is the fluorescent integral of reference compound; Au is the absorbance of DA and DA + CN^-; As is the absorbance of reference compound. The quantum yield of DA and DA + CN^- were 0.4183 and 0.0594, respectively.

To get a quantitative idea of the sensing, the fluorescence titration experiment was further performed in pure water (Figure 3). Upon the addition of CN^-, the fluorescence emission intensity of chemosensor DA at 390 nm decreased gradually and reached to minimum value by 3.8 equiv. of CN^- (Figure 3a). The binding constant value Ka, which was evaluated by the Benesi-Hildebrand method, was found to be 6.08×10^5 mol^{-1} L (Figure 3b), and $pK_a = 4.17$. Furthermore, the detection limit (LOD) calculated according to the basis of 3σ/S (where σ is the standard deviation of the blank solution and S is the slope of the calibration curve) was 1.38×10^{-8} mol L^{-1} (Figure S3, Supplementary Information (SI) section), which is superior to other cyanide chemosensors (Table 1) and far below the World Health Organization (WHO) guideline of 1.9 µmol L^{-1}.[3]

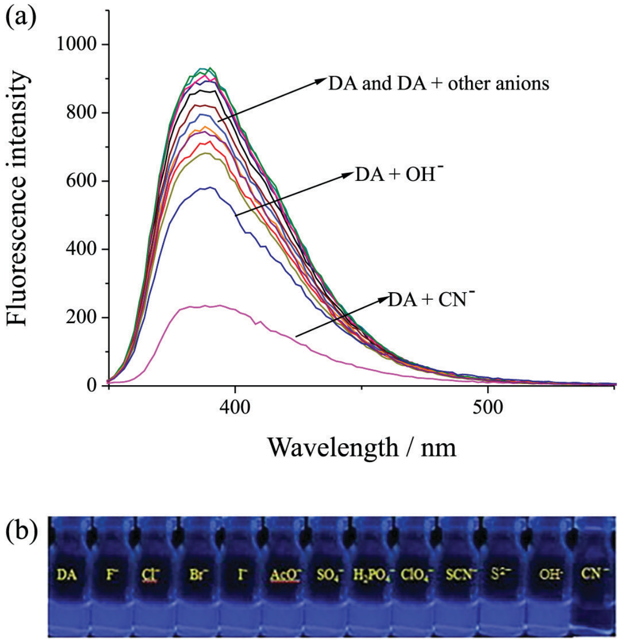


Figure 2. (a) Fluorescence spectra of DA (20 µmol L^{-1}) upon addition of 5 equiv. of various anions in pure water media (λex = 315 nm); (b) fluorescence responses of DA in the presence of 5 equiv. of different anions in pure water (λex = 315 nm).

The pH effects were measured to investigate whether the sensor is appropriate for the physiological detection (Figure 4). The detection of CN^- can operate well in the pH range of 4.0-8.0 in pure water, which confirmed that DA has also excellent sensing ability toward CN^- under physiological conditions.

We further examined the sensor's reactivity toward CN^- (Figure 5). While DA (2.0×10^{-5} mol L^{-1}) was treated with CN^- (5 equiv.) in pure water, the fluorescence intensity at 390 nm decreased rapidly and reached a plateau in about 1 s. The kinetic test result proved that our sensor could rapidly detect CN^- in pure water.

We further explore the selectivity of chemosensors DA for cyanide when DA was treated with other competitive anions, such as F^-, Cl^-, Br^-, I^-, AcO^-, SO_4^-, $H_2PO_4^-$, ClO_4^-, SCN^-, S^{2-} and OH^- (Figure 6). The fluorescence emission

Table 1. Sensors for cyanide determination

Sensor	Solvent	Quantum yield	Limit of detection / M	Reference
Bis(salamo)-type tetraoxime-based	$MeOH/H_2O$ = 1:1	–	8.91×10^{-7}	15
Pyrene-benzothiazol-based	$DMSO/H_2O$ = 1:1	0.23	2.8×10^{-7}	16
Naphthalene-based	H_2O	–	3.2×10^{-7}	17
Merocyanine-based	$EtOH/H_2O$ = 9:1	–	1.6×10^{-6}	18
Indanedione-based	THF/H_2O = 1:9	0.12	9.4×10^{-7}	19
Phenazine-based	$H_2O/DMSO$ = 7:3	–	1.4×10^{-6}	20
Boronic-based	H_2O/CH_3CN = 8:2	–	6.45×10^{-8}	21
Borondipyrromethene-based	$H_2O/EtOH$ = 1:1	0.015	5.9×10^{-8}	22
Rhodamine-B hydrazide-based	$H_2O/DMSO$ = 1:1	0.8916	1.59×10^{-7}	23
Our work	H_2O	0.4183	1.38×10^{-8}	

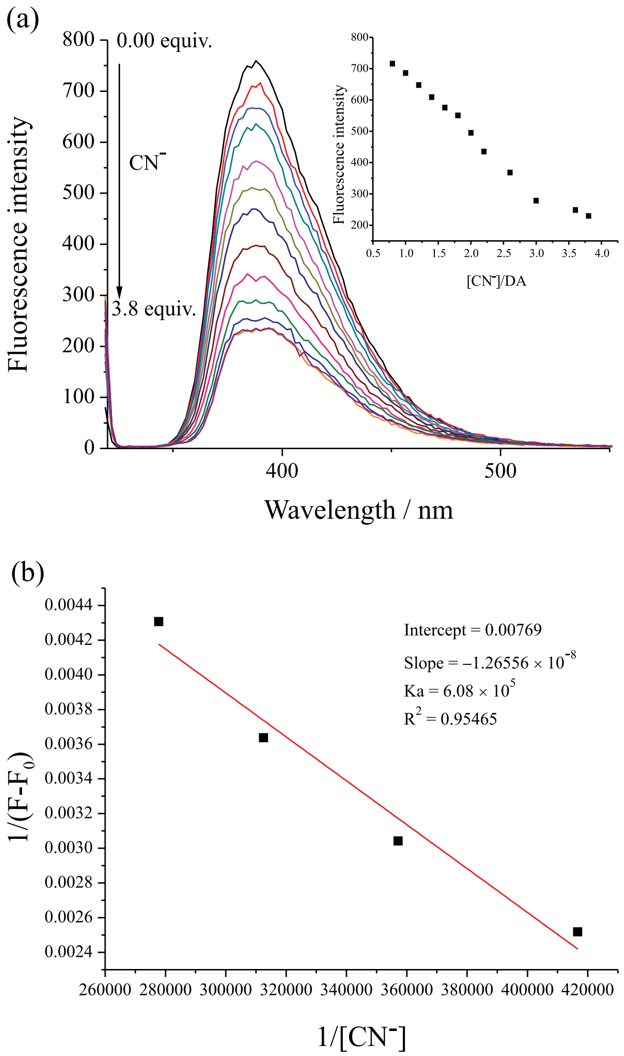


Figure 3. (a) Fluorescence spectra (λex = 315 nm) of DA (20 µmol L^{-1}) in the presence of different amounts of CN^-. Inset: a plot of fluorescence intensity at 390 nm *vs.* number of equivalents of CN^-; (b) Benesi-Hilderbrand plot of DA with CN^-.

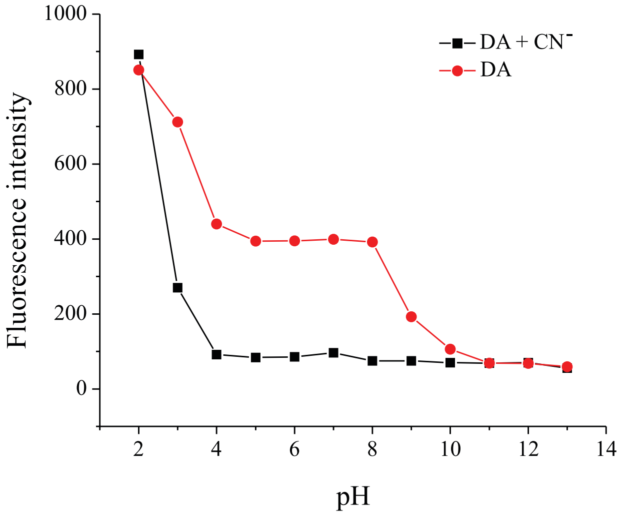


Figure 4. Emission intensity of DA (20 µmol L^{-1}) and DA + CN^- (50 equiv.) at different pH values. HEPES (4-(2-hydroxyerhyl) piperazine-1-erhanesulfonic acid) buffered water solution (1 mmol L^{-1}) (λex = 315 nm, λem = 390 nm).

intensity of chemosensor DA + CN^- was not affected by the presence of the other anions, especially F^- and AcO^-, which demonstrated that DA has specific selectivity for CN^-.

The reversibility of the sensor is an extremely important aspect for the detection of the specific analyses, so we investigated the fluorescence behavior of chemosensor DA between CN^- and H^+ ions. The addition of CN^- ions to the solution of chemosensor DA induced fluorescence disappearing, with the addition of H^+, the fluorescence was turn on. This reversible process of "on-off-on" could be repeated at least three times with a little loss of fluorescent intensity (Figure 7).

On the basis of the spectral results, the recognition mechanism of chemosensor DA with CN^- is proposed as follows: before the addition of cyanide anions, the

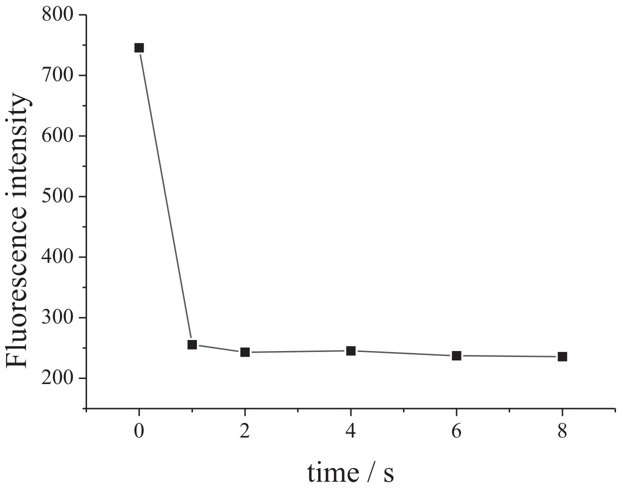


Figure 5. Time-dependent fluorescence intensity of DA (20 μmol L^{-1}) in the presence of 5 equiv. CN^- in pure water (λex = 315 nm, λem = 390 nm) was recorded after 0, 1, 2, 4, 6 and 8 s.

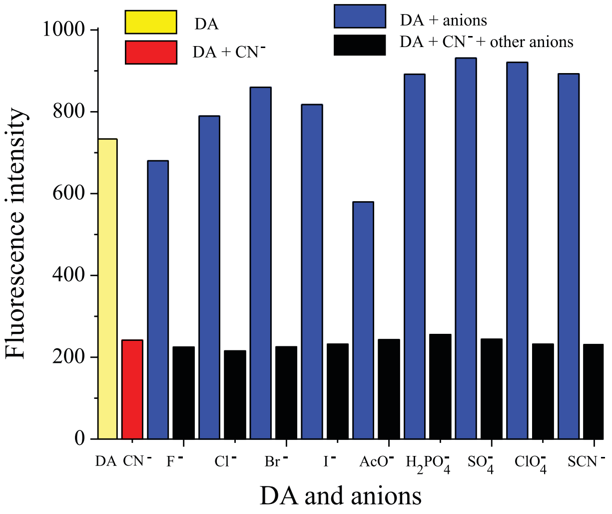


Figure 6. Emission intensity of DA (20 μmol L^{-1}) in the presence of 5 equiv. of other anions and DA + CN^- in the presence of 5 equiv. of other anions in pure water (λex = 315 nm, λem = 390 nm).

intramolecular rotation was restricted by the hydrogen bond. In this case, the sensor is in the intramolecular charge transfer (ICT) state and displays a very strong fluorescence. With the increasing addition of CN^-, the hydrogen bond is destroyed, the benzene ring of chemosensor DA could take place of an intramolecular rotation, and change to twisted ICT (TICT) state and show a weak fluorescence emission at 390 nm. With further adding H^+ to solution of DA + CN^-, the fluorescence intensity was recovered to an ICT state (Figure 8). Meanwhile, the ^1H NMR titration experiments were conducted to demonstrate the proposed recognition mechanism. As shown in Figure 9, upon the gradual addition of CN^-, the –COOH peak at 12.85 ppm disappeared, and the signal peak of the hydrogen atoms of the benzene ring and the naphthalene ring shifted upfield,

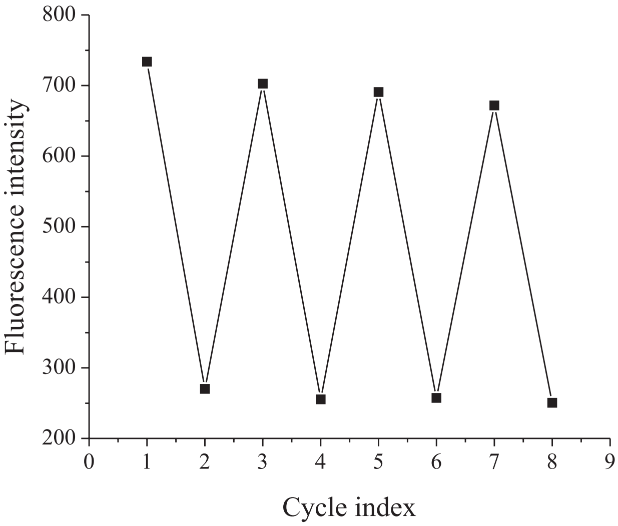


Figure 7. Fluorescence intensity of DA (20 μmol L^{-1}) upon the alternate addition of CN^- and H^+ in pure water.

indicating that the recognition of CN^- is a deprotonation process. In addition, we prepared the carboxylate salt of DA using sodium carbonate, and investigated its photophysical properties. The results show the fluorescence spectrum of DA^- and DA + CN^- have significant overlaps (Figure S4, SI section), and prove that the recognition of CN^- is a deprotonation process.

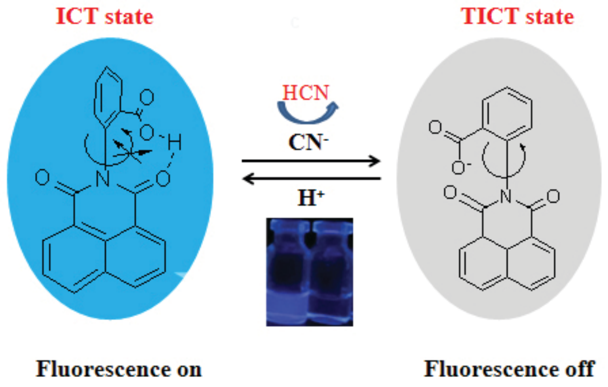


Figure 8. The proposed binding mechanism of DA for CN^- anion.

To study its practical applications, the chemosensor DA was used to measuring CN^- in tap water. By adding a known quantity of standard CN^- to tap water and calculating its recovery, the accuracy of the method was evaluated. The recoveries of different known quantities of CN^- added were gained from 96.6 to 103.6% with a favorable analytical precision (relative standard deviation (RSD) ≤ 5.0%), which indicated the chemosensor DA was able to determine CN^- in tap water. The results were presented in Table S1 (SI section).

Motivated by the favorable characteristic of chemosensor DA in solution, test strips were carried out

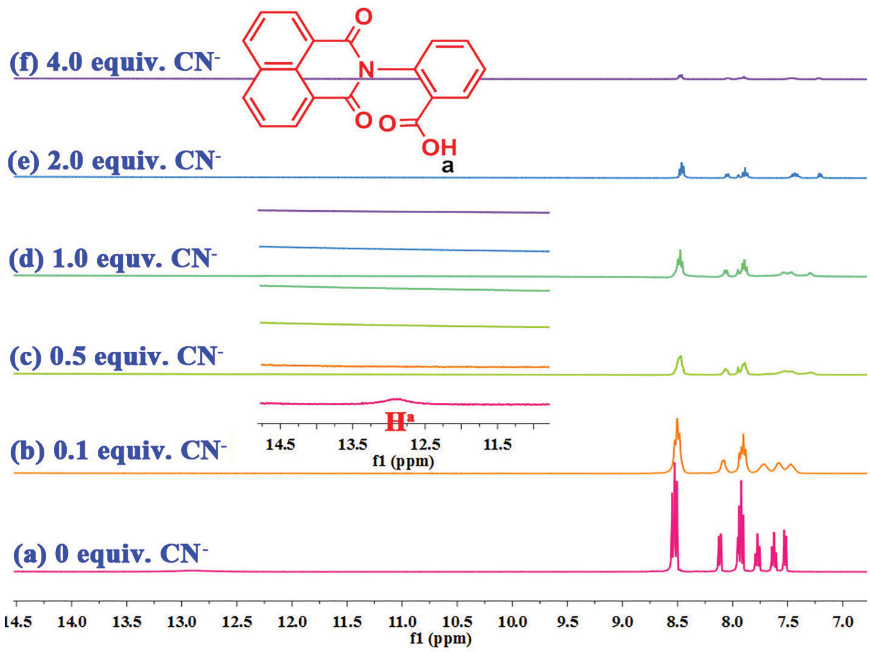


Figure 9. ^{1}H NMR spectra of free DA (600 MHz, DMSO-d_6) and in the presence of different amounts of CN^- (1 mol L^{-1}, H_2O).

by submersing filter papers in the pure water solution of DA (2.0×10^{-4} mol L^{-1}) and then dried in air. The test strips coated with DA were utilized to sense CN^- and other anions, similar to the pH test paper. As shown in Figure 10, when one drop of CN^- (2.0×10^{-4} mol L^{-1}) water solution was added on the test kits, the fluorescence turn off response can be observed by naked-eye under UV irradiation. The same procedures were done for competitive ions. However, potentially competitive ions had no influence on the detection of CN^- by the test strips. Hence, DA also possesses excellent selectivity for CN^- in test strips.

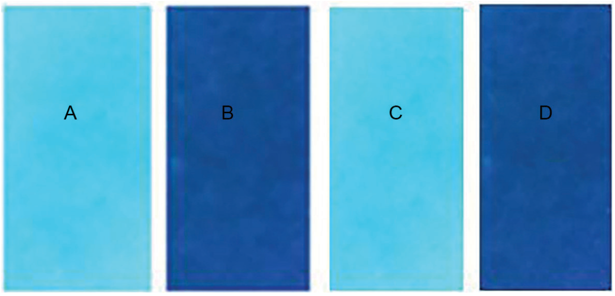


Figure 10. Photographs of DA on test strips (A) free DA; (B) after submersing in pure water solutions of DA + CN^-; (C) after submersing in pure water solutions of DA + other anions; (D) after submersing in pure water solutions of DA + CN^- + other anions using UV lamp at room temperature.

Conclusions

In summary, by rationally introducing aromatic carboxyl and 1,8-naphthalimide moieties, we designed a novel water soluble fluorescent chemosensor (DA). As expected, DA displays high sensitivity and rapid response (about 1 s) for recognizing CN^- in 100% water solutions via an ICT-TICT state change mechanism. The detection limits of the chemosensor DA toward CN^- was 1.38×10^{-8} mol L^{-1}, which was far below the WHO limit (1.9 µmol L^{-1}). Furthermore, the sensor DA was able to determine CN^- in tap water with good recovery. Meanwhile, we prepared the test strips based on DA, which could rapidly and efficiently detect CN^- in water.

Supplementary Information

Supplementary information is available free of charge at http://jbcs.sbq.org.br as PDF file.

Acknowledgments

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