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A Novel Cyanostilbene-based Fluorescent Probe for Rapid Detection of Aniline

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Abstract: Aniline sensing is of great societal implications, as aniline is a crucial chemical raw material but with high toxicity, processing with huge impacts on environment safety and human health. Herein, a novel cyanostilbene derivative (CN-DBE) was designed and synthesized as selective fluorescent probe material for aniline detection. Its aggregation-induced emission property, sensing property and detection mechanism were elucidated by photoluminescence spectra and numerical simulation. The results reveal that CN-DBE possesses high selectivity, quantitative and rapid detection ability to aniline due to the electron transfer mechanism. Moreover, the CN-DBE compound can also enable the fabrication of test strips, which provide a cheap and simple way to detect aniline leakage.

Key words: fluorescence sensor; cyanostilbene; aggregation-induced emission; aniline; rapid detection; photo-induced electron transfer

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用于快速检测苯胺的新型氰基苯乙烯荧光探针

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摘要: 苯胺是一种使用广泛但具有高毒性的化学原料,对环境安全和人类健康有巨大的潜在威胁。因此,对苯胺的检测具有重要的应用价值。本文设计并合成了一种可对苯胺进行选择性检测的氰基二苯乙烯衍生物(CN-DBE)荧光探针材料。通过光致发光光谱和数值模拟等方法对CN-DBE的聚集诱导发光性质、传感性能和检测机理进行了详细研究。结果表明,基于光诱导电子转移机理,CN-DBE可实现对苯胺的高选择性、定量、快速检测。此外,利用浸渍法制备了相应的CN-DBE试纸,可实现对苯胺的可视化检测,提供了一种廉价且简单的苯胺泄漏检测方法。

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关　键　词：荧光传感器；氰基二苯乙烯；聚集诱导发光；苯胺；快速检测；光诱导电子转移

1 Introduction

As a crucial chemical raw material, aniline is widely used in dye industry, pesticide production and rubber auxiliaries[1-4]. Yet, aniline is a highly toxic chemical, which can cause harmful effects to human body and environment[5-6]. The leakage of aniline can cause methemoglobinemia and damage to organs such as liver, kidney and skin through direct contact and breathing[7-8]. Therefore, aniline detection is of great societal implications for public health and environment safety. In particular, aniline in large amount will leak into the water environment when safety accidents occur in industrial production and transportation. Rapid and visible detection of abrupt and massive aniline-leakage in water media is vitally required for assessing and controlling the damage.

So far, the traditional methods for detection of aniline include high performance liquid chromatography[9], gas chromatography-mass spectrometry[10], spectral analysis[11] and cyclic voltammetry[12] which applied by the difference of optical properties, polarity and other chemical properties. Nonetheless, these methods require special instruments which is labor-intensive, time consuming and expensive, and are not readily available in most cases[13]. Compared with traditional approaches, fluorescent probe is emerging as an alternate due to its advantages such as simple operation, low cost, high selectivity and sensitivity[14-19]. However, most of them are affected by the phenomenon of aggregation caused quenching (ACQ), which limits the application of probes in many fields[20-23].

Different from traditional luminescent materials, a new class of luminogens with aggregation-induced emission (AIE) effect which have higher luminous efficiency in the aggregated state was proposed by Tang's group[24-27]. Among them, cyanostilbene is widely used due to its excellent fluorescent properties including high photostability and excellent fluorescence quantum yields as well as a controllable emission wavelength[28-30]. Cyanostilbene and its derivatives have already been applied into detection sensors, near-infrared biological imaging, solar cells and anti-counterfeiting materials[31-36]. However, the detection of aniline in aqueous medium by AIE materials is relatively rare. It is of great interest to know whether it is possible to fabricate cyanostilbene-based fluorescent sensor for rapid detection of aniline for industrial safety.

Herein, we designed and synthesized a cyanostilbene derivative which can be serving as a fluorescent sensor for aniline. The photoinduced charge transfer between cyanostilbene and aniline can trigger the fluorescence quenching of cyanostilbene, enabling visible detection to aniline. The selectivity, quantitative and rapid detection to aniline have been characterized in detail, which proves it is a valuable method to detect aniline in aqueous media. In addition, the test papers prepared by soaking CN-DBE solution can achieve the visible detection immediately, which proves its potential in rapid detection of aniline leaks.

2 Experiment

2.1 Apparatus

^{1}H NMR spectrum was recorded on a Bruker AVANCE Ⅲ spectrometer from Switzerland with deuterated chloroform ($CDCl_3$) as solvent and tetramethylsilane (TMS) as internal standard at room temperature. All the photoluminescence (PL) spectra were recorded on Hitachi FL-2500 fluorescence spectrophotometer from Japan.

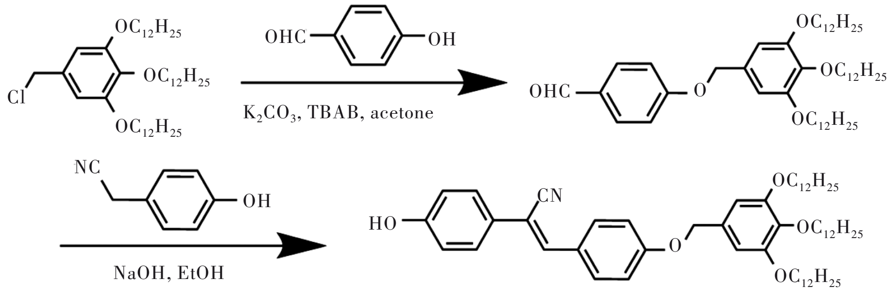
2.2 Chemicals

Potassium carbonate (98%, Tianjin, Heowns), 4-hydroxybenzaldehyde (98%, Tianjin, Heowns), 4-hydroxyphenylacetonitrile (98%, Tianjin, Heowns), anhydrous magnesium sulfate (98%, Shanghai, Energy Chemical), tetrabutylammonium bromide (TBAB, 97%, Tianjin Heowns), sodium hydroxide (98%, Shanghai, Energy Chemical) were used as received. 3,4,5-tris(dodecyloxy)benzyl chloride was synthesized according to the method in the literature[37]. The water in the experiments was purchased from

Hangzhou Wahaha Group Co., Ltd. All the other solvents were purchased from Jiangtian Chemical Reagents Co. Ltd. and used without further purification unless otherwise specified.

2.3 Synthesis of CN-DBE

The synthetic route of CN-DBE is shown in Scheme 1. With the addition of K_2CO_3 and TBAB, the intermediates could be synthesized by Williamson ether synthesis. Then add 3, 4, 5-tris(dodecyloxy) benzyl-p-benzaldehyde ether(0.76 g, 1 mmol), 4-hydroxyphenylacetonitrile(0.20 g, 1.5 mmol), and sodium hydroxide (0.12 g, 3 mmol) into a 250 mL round bottom flask, followed by the addition of tetrahydrofuran for 5 mL. After it was completely dissolved, 25 mL of anhydrous ethanol was added with the reaction heated and refluxed for a total of 24 h. After the reaction was completed, the mixture was cooled to room temperature, and dilute hydrochloric acid in certain amount was added to the system before adjusting the pH to weak acidity. Orange solid in a large amount was precipitated in the flask, and then filtered and washed. Following the process in which the orange solid was separated by column chromatography using dichloromethane/petroleum ether(1/1) as eluent, the yellow compound CN-DBE was obtained in 70% yield.



Scheme 1　Synthesis routes of CN-DBE

3 Results and Discussion

3.1 Characterization and Aggregation-induced Emission Property

To prove the successful preparation of CN-DBE, ^{1}H NMR spectrum was conducted to confirm the chemical structure of CN-DBE(Fig. 1). To investigate the thermostability of CN-DBE, the thermogravimetric analysis(TGA) experiment was conducted from 25 ℃ to 800 ℃ at a heating rate of 10 ℃/min. The 5% decomposition temperature of CN-DBE is 275 ℃, indicating the good thermostability of CN-DBE(Fig. 2).

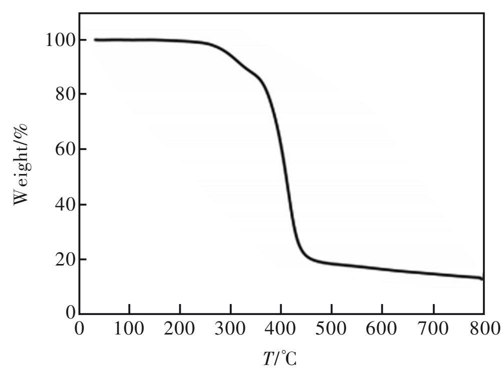


Fig.2　TGA curve of CN-DBE under the rate of 10 ℃/min

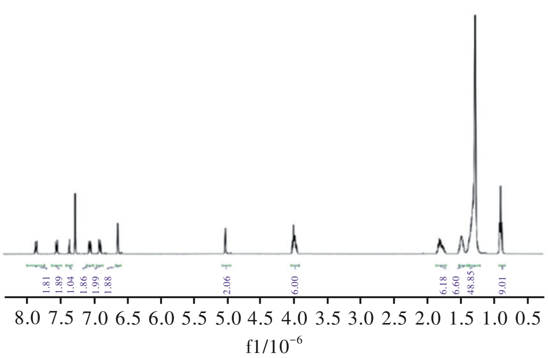


Fig.1　^{1}H NMR of CN-DBE in $CDCl_3$

The photophysical property of CN-DBE was investigated in THF/H_2O mixture with THF as a good solvent and water as a poor solvent. As displayed in Fig. 3(a), CN-DBE shows weak emission in pure THF due to the dispersed state of molecules caused by its good solubility in THF. With the increase of water content, the PL intensity showed apparent enhancement due to formation of aggregates. Compared with the CN-DBE molecules in pure THF, the fluorescence intensity of CN-DBE in THF/H_2O mixture with

f_w = 95% was boosted with 7.5 folds (Fig. 3(b)). Moreover, the emission wavelength was red-shifted from 453 nm to 480 nm, which is attributed to the aggregation-induced conformational planarization[22]. These results showed that CN-DBE is a typical AIE material.

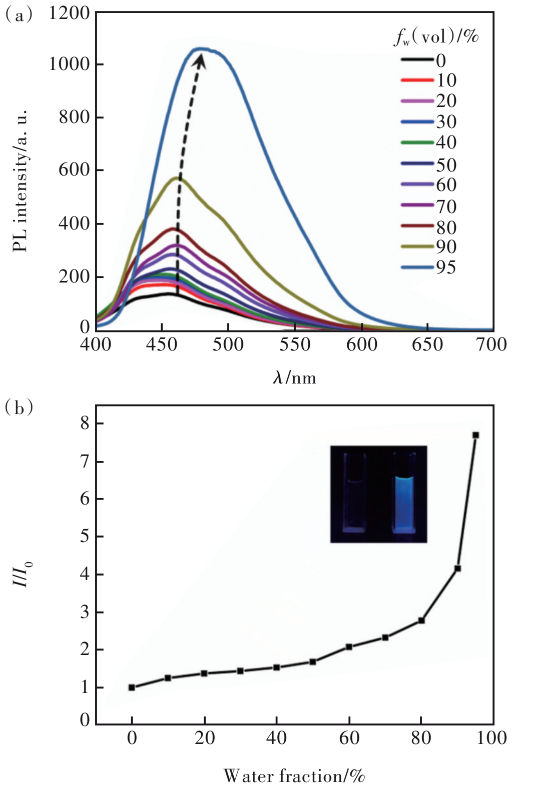


Fig.3　(a) Fluorescence spectra of CN-DBE in different H_2O/THF mixtures from f_w = 0% to 95%. ([CN-DBE] = 10 μmol·L^{-1}, λ_{ex} = 350 nm). (b) Plot of I/I_0 *versus* water fraction of H_2O/THF mixtures of CN-DBE, where I_0 represents the fluorescence intensity in pure THF solution. Inset: photographs of CN-DBE in THF/water mixtures (f_w = 0, 95%) taken under the illumination of a UV lamp (365 nm).

3.2　Aniline Detection

The intriguing AIE effect of CN-DBE prompts us to explore its potential application as fluorescence sensor for detecting aniline. Fluorescence spectroscopy experiments were conducted to quantify aniline induced fluorescence intensity changes of CN-DBE. As displayed in Fig. 4(a), the fluorescence intensity decreased gradually with the amounts of aniline (0.01–0.1 mol·L^{-1}) were added into CN-DBE solution (10 μmol·L^{-1}), exhibiting turn-off response to aniline in water. Moreover, it can be observed from Fig. 4(b) that the intensity of I/I_0 and aniline concentration possesses a good linear relationship within the concentration. According to the definition by IUPAC ($C_L = K \cdot S_b/m$), the detection limit was calculated to be 1.1 × 10^{-3} mol·L^{-1}.

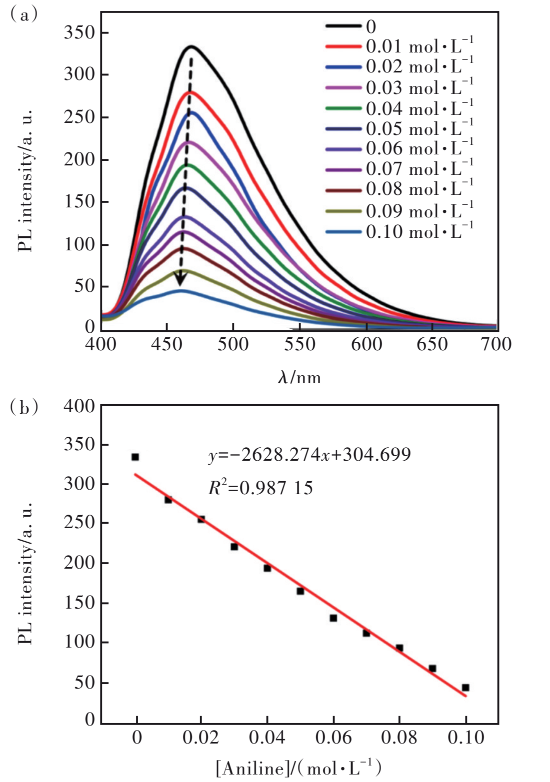


Fig.4　(a) Changes in the emission spectra of CN-DBE (10 μmol·L^{-1}) with different concentrations of aniline in the mixture of THF/water (v:v = 1:4). (b) Plot of the fluorescence intensity of CN-DBE with different concentrations of aniline.

In order to evaluate the specific detection of aniline by CN-DBE sensor, we compared the fluorescence responses of CN-DBE to aniline with other amine compounds under the same test condition, including diisopropylamine, cyclohexylamine, triethylamine, n-butylamine, dicyclohexylamine, benzylamine, diethylamine. As can be seen from Fig. 5(a), after adding diisopropylamine, cyclohexylamine, triethylamine, n-butylamine, dicyclohexylamine, benzylamine and diethylamine, the fluorescence intensity of the system did not decrease drastically, which is different from the obvious turn-off response when aniline was added. The optical images shown in Fig. 5(b) indicate that CN-DBE has fluorescence quenching effect only after adding aniline. The above results illustrate that CN-DBE has excellent specificity detection performance for aniline.

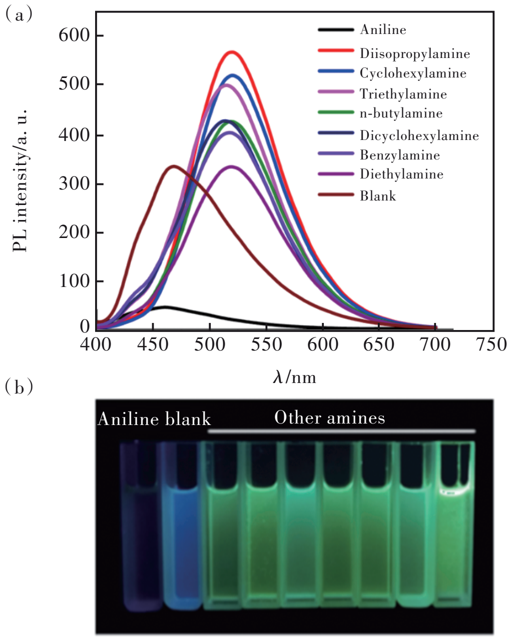


Fig.5 (a) Emission spectra changes of CN-DBE upon the addition of amines. λ_{ex}=350 nm. (b) The optical image of CN-DBE with the addition of amines under UV irradiation.

The interference of ions generally exists when detecting aniline in the aqueous medium. Therefore, to evaluate the anti-interference ability of CN-DBE probe, we compared the detection ability of CN-DBE for aniline in the presence of various ions. As shown in the Fig. 6, with the addition of other interfering substances, the fluorescence response of CN-DBE to aniline was almost unaffected. All these results suggest that CN-DBE can act as a selective fluorescent sensor for aniline with anti-interference ability in aqueous media. Each experiment was repeated three times to make sure the accuracy of result.

In order to investigate the response time of the CN-DBE on aniline detection, the intensity changes of PL spectra were detected by the addition of 0.04 mol·L^{-1} aniline, and the results are shown in Fig. 7. After the addition of aniline, CN-DBE exhibited fluorescence quenching immediately, and the fluorescence intensity dropped to the minimum value after 5 s when it was first measured. It proves that CN-DBE has a very short response time and can be used for immediate detection of aniline in aqueous media.

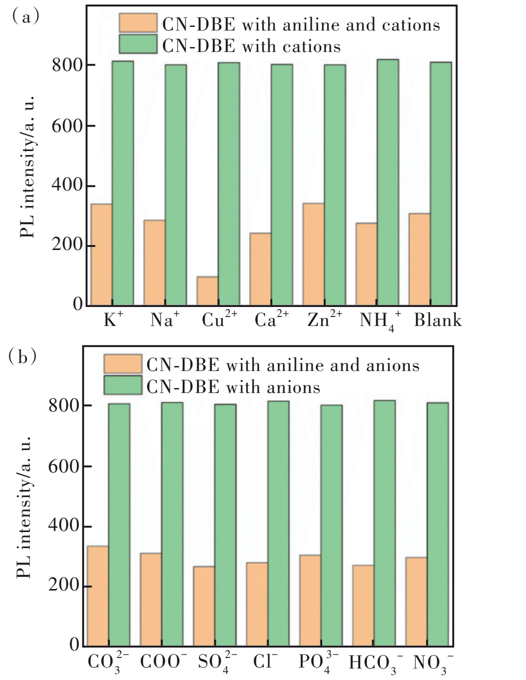


Fig.6 Emission intensity of CN-DBE in the mixture of aniline with cations (a) and anions (b). [CN-DBE] = 10 μmol·L^{-1}, [cations] and [anions] = 0.04 mol·L^{-1}.

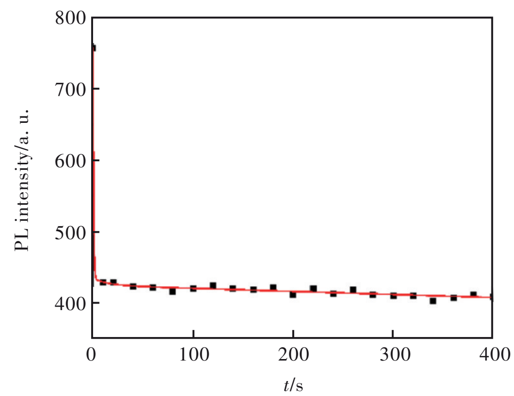


Fig.7 The response time and intensity changes of CN-DBE (10 μmol·L^{-1}) upon the addition of aniline (0.04 mol·L^{-1}) in the mixture of THF/water (v:v=1:4)

In the process of the practical detection of aniline, portability and immediacy are particularly important, so the potential application of CN-DBE in fabricating test paper was investigated. The paper strips were prepared by soaking the filter paper in CN-DBE solutions, and then dry them with aeration at room temperature. As shown in Fig. 8(a), the original PL emission of the test papers is bright blue under UV illumination. With addition of aniline, the fluorescence of test paper was effectively quenched (Fig. 8(b)). Moreover, the applicability of the CN-DBE test paper on real-world samples, such like

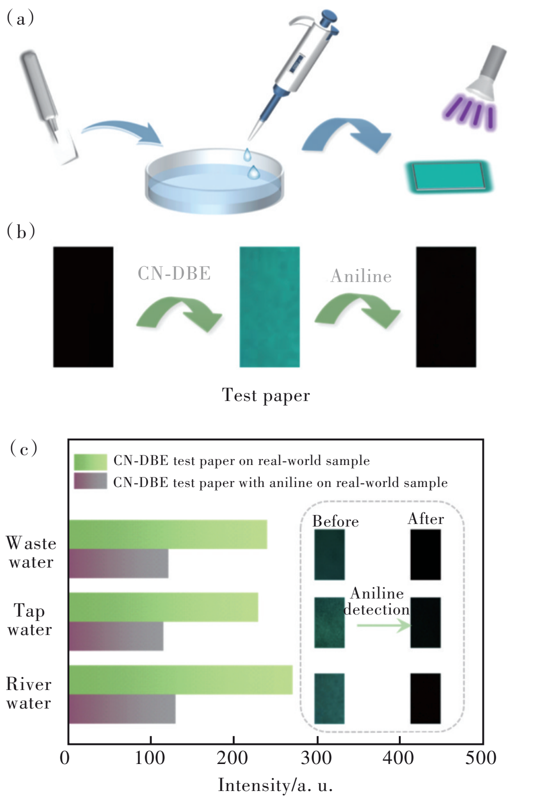


Fig.8 (a) The schematic diagram of preparing test papers. (b) Test papers under the illumination of a UV lamp. Left: pure test paper, middle: test paper with CN-DBE, right: test paper with CN-DBE after dropping aniline solution. (c) The applicability of the CN-DBE test paper on real-world samples.

wastewater, tap water and river water was further investigated. As shown in Fig. 8(c), the detection effect of CN-DBE is not affected by the complex matrix. The excellent response time and test strip portability make CN-DBE have great application potential in aniline leak detection in chemical plants.

3.3 Sensing Mechanism of Aniline Detection

To clarify the detection mechanism, the quantum chemical calculation (B3LYP method and def2-tzvp basis set) was conducted by density functional theory. As shown in Fig. 9(a), the HOMO and LUMO state density was uniformly distributed at the whole aromatic unit of CN-DEBE, and the HOMO and LUMO energy levels are −5.42 eV and −1.75 eV. The HOMO energy level of aniline is higher than those of diisopropylamine, cyclohexylamine, triethylamine, n-butylamine, dicyclohexylamine, benzylamine and diethylamine. In particular, only the HOMO energy level of aniline is higher than CN-DBE, while the HOMO energy levels of other amine compounds are lower than CN-DBE. As shown in Fig. 9(b), the electrons in HOMO in CN-DBE tend to be excited to LUMO under illumination. Since the HOMO energy level of aniline is higher than that

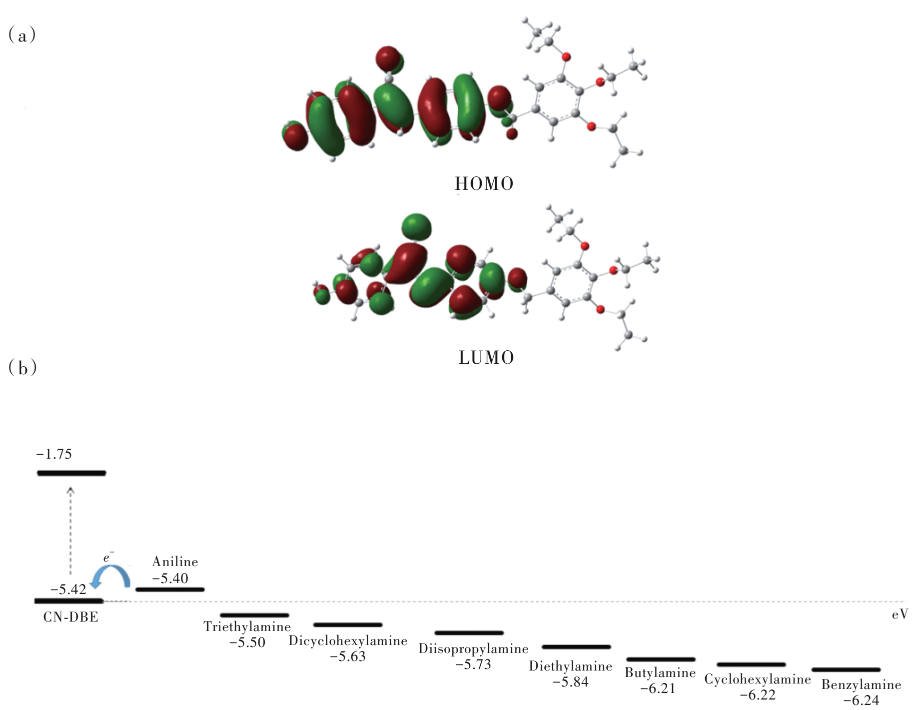


Fig.9 (a) Corresponding frontier orbital (HOMO and LUMO) distributions of CN-DBE. (b) The detection mechanism of aniline by CN-DBE.

of CN-DBE, the electrons on the HOMO of aniline will transfer to the HOMO of CN-DBE. Subsequently, the electrons of the LUMO in the CN-DBE will be transferred to the HOMO of aniline, resulting in the phenomenon of fluorescence quenching of the system. So it is speculated that the photoinduced electron transfer between CN-DEBE and aniline is the detection mechanism.

4 Conclusion

In summary, a cyanostilbene-based fluorescent sensor (CN-DBE) for aniline detection has been synthesized. The photoluminescence spectra reveal that CN-DBE is a typical AIE material. Due to the photoinduced electron transfer between CN-DBE and aniline, the fluorescence of CN-DBE can be quenched with the addition of aniline, enabling a visible turn-off detection effect. The experimental results indicate that CN-DBE possesses high selectivity and quantitative recognition to aniline. Particularly, the response time of CN-DBE to aniline is almost negligible, which proves its potential in rapid detection of aniline leakage.

Response Letter is available for this paper at: http://cjl.lightpublishing.cn/thesisDetails#10.37188/CJL.20220423.

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