Talanta 97 (2012) 343–348

Contents lists available at SciVerse ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

A simple sensitive colorimetric/fluorometric probe for iodide

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ARTICLE INFO

Article history:
Received 25 October 2011
Received in revised form
10 April 2012
Accepted 21 April 2012
Available online 28 April 2012

Keywords:
Iodide
Sensor
Carbazole
Colorimetry
Fluorometry

ABSTRACT

A simple fluorene oligomer 7, 7′-bis(9, 9-bis(6-(9H-carbazol-9-yl)hexyl)-9H-fluoren-2-yl)-9,9,9′,9′-tetrakis(6-(9H-carbazol-9-yl)hexyl)-9H, 9′H-2, 2′-bifluorene (**OCZ**) with peripheral carbazole side chains is synthesized and used to detect iodide (I^-) as colorimetric/fluorometric probe. It shows high fluorescence quenching sensitivity and selectivity towards I^-. When **OCZ** acted as the colorimetric probe, the color change is observed with naked eyes at a very low concentration of 0.1 mM, which is among the best reported performances. The good sensitivity and selectivity may be due to the wide range of electron-rich carbazole groups.

1. Introduction

The fluorescent molecules present the unique advantages of high detection sensitivity and operation simplicity. Hereby they provide a number of potential applications in environmental monitoring, industrial process control and biomedical diagnostics [1,2]. Especially, they can be used to detect trace iodide (I^-) and metal ions [3–5], which play important roles in environment and living systems. It is well known that among the biologically important anions, iodide is of particular importance due to its essential role in thyroid gland [6]. Thus the detection of I^- has attracted the interest of many researchers in recent years. Compared with other sensor methods, such as UV spectrophotometry, ion-selective electrode, cyclic voltammetry and so on [7,8], the fluorescent molecules exhibit high selectivity and are receiving more and more attentions [9]. Though considerable efforts have been made to synthesize different fluorescent chemosensory systems [10], it is still necessary to develop the simple sensitive fluorescent molecules in order to realize practical application. In this competition, carbazole receives less attention than other nitrogen-containing materials. Till now, the reported sensor materials which contain the carbazole group were mainly applied in the electrochemistry detection field [11]. In the few reports of the carbazole group used as fluorescent chemosensors, the carbazole usually appeared in the backbone of the copolymers or used as hybrid materials [12,13]. For example, Valiyaveettil et al. reported the carbazole-containing conjugated copolymers acted as a colorimetric/fluorometric sensor for iodide anion [14]. Li et al. reported an alternative fluorescent copolymer of carbazole to detect Mercury [15]. Kim et al. reported the carbazole-based multi-nitrogen-containing molecule could be used as chromofluorogenic sensor [16].

On the other hand, colorimetric sensors are of great significance due to their capability to detect analyses by naked eyes without any other instruments [17]. Therefore, developing colorimetric sensor materials is also an interesting challenge. In this paper, we report a simple fluorene oligomer 7,7′-bis(9, 9-bis(6-(9H-carbazol-9-yl)hexyl)-9H-fluoren-2-yl)-9,9,9′ 9′-tetrakis(6-(9H-carbazol-9-yl)hexyl)-9H, 9′H-2,2′-bifluorene (**OCZ**) with peripheral carbazole side chains, which can detect I^- by dual-channel assays of colorimetry and fluorometry with high sensitivity and selectivity.

This fluorene oligomer **OCZ** has a simple structure of four fluorene units as the backbone and eight alkyl-linked peripheral carbazole groups as the side chains. The carbazole units were seldom linked as side chains and used as chemosensory materials. However, carbazole as side chain may have two advantages when it is used in chemosensor: improving the solubility and decreasing the steric hindrance when it reacts with analyte. Owing to these factors, **OCZ** could be a promising candidate of the chemosensor material.

2. Experiments

2.1. Chemicals and instruments

All reagents, unless otherwise specified, were obtained from Aldrich, Acros and TCI Chemical Co. and used as received.

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http://dx.doi.org/10.1016/j.talanta.2012.04.043

The solvents toluene and *N*,*N*-dimethylformamide (DMF) were dried and purified by distillation. All other solvents were further purified before use. All manipulations involving air-sensitive reagents were performed under an atmosphere of dry nitrogen. Column chromatography was performed using silica gel (200–300 mesh).

The ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker AVANCZ 500 spectrometer at 500 MHz and 125 MHz, respectively, using deuterated chloroform ($CDCl_3$) as the solvent at 298 K, and tetramethylsilane (TMS) as the internal standard. Ultraviolet–visible (UV–vis) absorption spectra were recorded on a model UV-3100 spectrophotometer. Fluorescence spectra were performed using a model RF-5301PC spectrophotometer. Differential scanning calorimetry (DSC) analysis was determined using a NETSCH (DSC-204) instrument at a heating rate of 10 °C/min under nitrogen flushing. Thermal gravimetric analysis (TGA) was determined using a Pyris1 TGA thermal analysis system at heating rate of 20 °C/min under nitrogen.

2.2. *Synthesis*

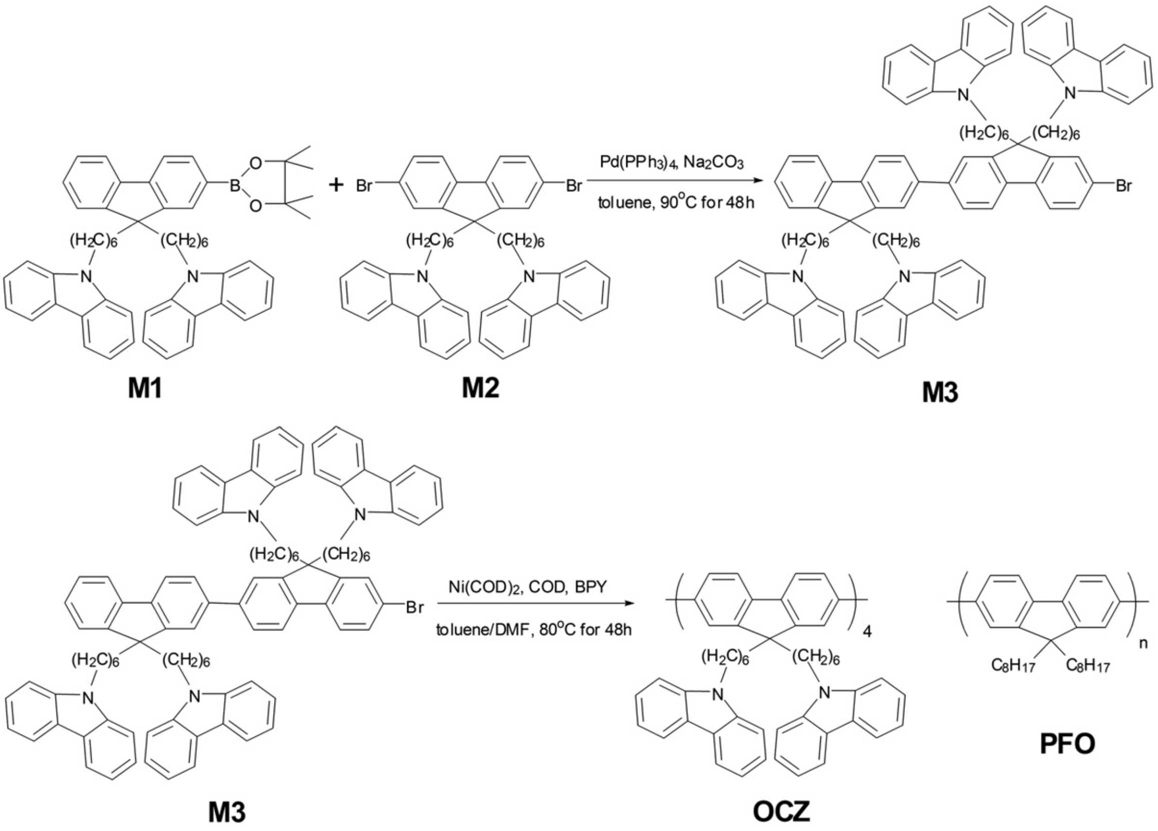
The synthesis of **OCZ** was readily prepared in three steps as shown in Scheme 1. First, the intermediate compounds M1 and M2 were obtained according to the literatures [18,19], then through the Suzuki reaction, the dimeric fluorene M3 was achieved. At last, **OCZ** was prepared from M3 by the Yamamoto reaction. The chemical structures of the intermediates and **OCZ** were characterized by ^{1}H-NMR, ^{13}C-NMR, mass spectrometry and elemental analysis.

2.2.1. *The synthesis of M3*

2.0 g (2.53 mmol) M1, 3.12 g (3.79 mmol) M2 and 0.21 g (0.19 mmol) $Pd(PPh_3)_4$ were dissolved in freshly distilled toluene (9 mL) in the round bottom flask. Ethanol (2 mL) and sodium carbonate 2 M in water (6 mL) were added. After removal of oxygen, the reaction system was stirred at 90 °C and refluxed for 24 h. After cooling to room temperature, the mixture was extracted several times with chloroform/water. The organic phase was collected and dried with anhydrous magnesium sulphate. After filtration and solvent evaporation, the residue was purified by chromatography using petroleum ether/dichloromethane as the eluent to afford a white solid 2.18 g (yield: 61%). ^{1}H-NMR (500 MHz, $CDCl_3$): $\delta = 8.04$ (d, $J = 7.7$ Hz, 8 H), 7.71 (m, 3H), 7.56 (m, 5H), 7.46 (d, $J = 8.1$ Hz, 1H), 7.36 (m, 10H), 7.25 (m, 10H), 7.17 (t, $J = 7.4$ Hz, 8H), 4.10 (dd, $J = 16.7$, 7.4 Hz, 8H), 1.78 (m, 16H), 0.97 (d, $J = 96.8$ Hz, 17H), 0.62 (m, 8H). ^{13}C-NMR (125 MHz, $CDCl_3$): $\delta = 152.81$, 151.20, 150.79, 150.52, 140.92, 140.55, 140.34, 140.14, 139.79, 139.24, 130.18, 127.20, 126.97, 126.42, 126.19, 126.11, 125.52, 122.83, 122.76, 121.19, 121.09, 120.28, 120.15, 120.05, 119.86, 118.66, 108.56, 55.36, 55.01, 42.82, 40.25, 29.63, 28.70, 26.78, 23.62.

2.2.2. *The synthesis of* **OCZ**

0.66 g (2.4 mmol) bis(1,5-cyclooctadiene)nickel, 0.27 mL (2.45 mmol) 1,5-cyclooctadiene and 0.375 g (2.4 mmol) 2,2′-bipyridine were dissolved in dry toluene (8 mL) and dry DMF (8 mL) in a Schlenk flask within a glove box. The mixture was heated at 60 °C with stirring under nitrogen for 20 min to generate the catalyst, and a solution of 1.41 g (1.0 mmol) M3 in dry toluene (15 mL) was added. The reaction was heated at 80 °C for 48 h. The mixture was then poured into a mixture of methanol and concentrated hydrochloric acid (1:1) and stirred for 4 h. The precipitated solid was filtrated and purified by chromatography using petroleum ether/dichloromethane as the eluent to afford a white solid 0.7 g (yield: 53%). The obtained **OCZ** was soluble in common solvents, such as tetrahydrofuran (THF), toluene, dichloromethane,



Scheme 1. The synthetic route of **OCZ** and the chemical structure of **PFO**.

chloroform and so on. ^{1}H-NMR (500 MHz, $CDCl_3$): δ=8.05 (t, J=8.0 Hz, 8H), 8.00 (d, J=7.7 Hz, 8H), 7.77 (d, J=7.9 Hz, 4H), 7.73 (dd, J=12.4, 7.7 Hz, 4H), 7.61 (m, 12H), 7.38 (m, 8H), 7.31 (dd, J=11.3, 4.1 Hz, 9H), 7.24 (m, 14H), 7.18 (dd, J=7.6, 4.5 Hz, 13H), 7.13 (dd, J=15.9, 8.7 Hz, 10H), 4.10 (t, J=7.2 Hz, 8H), 4.03 (t, J=7.2 Hz, 8H), 2.00 (s, 8H), 1.93 (m, 8H), 1.60 (m, 16H), 1.06 (d, J=6.2 Hz, 32H), 0.70 (s, 17H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ=151.86, 151.63, 150.95, 141.13, 140.74, 140.51, 127.59, 127.39, 126.76, 126.62, 125.93, 123.26, 123.16, 121.60, 121.52, 120.70, 120.27, 119.07, 108.98, 55.64, 55.44, 43.23, 43.16, 40.84, 40.73, 30.09, 29.11, 27.20, 24.20, 24.05. MALDI-TOF-MS: m/z 2653.63 [M]+ 2653.8. Anal. Calcd. for $C_{196}H_{186}N_8$: C, 88.71; H, 7.06; N, 4.22. Found: C, 88.60; H, 7.11; N, 4.28.

2.3. *The fluorometric and colorimetric detection*

UV–vis absorption spectra and fluorescence spectra were utilized to characterize the fluorometric and colorimetric responses of the **OCZ** solution toward I^- and some other anions. All fluorescence detections were carried out in the homogeneous **OCZ** THF solution with a concentration of 1.0×10^{-6} M. The anions were potassium salts, viz. F^-, Cl^-, Br^-, I^-, NO_3^-, AcO^- and CO_3^{2-}, which were dissolved in deionized water with a concentration of 0.1 M.

The specific I^- detection steps were as follows: 3.0 mL **OCZ** solution was put into the standard quartz cell, then the potassium salts aqueous solutions containing different anions were added and the photoluminescence (PL) spectra were measured after a strong stirring, and then adding more and more potassium salts and recording the PL spectra repeatedly. During the anions detection experiments, the cumulative adding of potassium salts solution were 3, 6, 9, 12, 15, 18, 21, 27, 33, 39, 45 and 50 μL, respectively. Ignoring the volume changes caused by adding potassium salts solution, the related anion concentrations were 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.9, 1.1, 1.3, 1.5 and 1.7 mM, respectively. The detections were carried out at the room temperature. The UV–vis absorption spectra were measured under the same conditions. The color changes of the **OCZ** solution upon the addition of I^- were observed after adding the anion samples immediately. But due to the individual differences, the slightly deviation may exist in the determination of the color change by naked eyes.

3. Results and discussion

3.1. *Thermal properties*

The thermal properties of **OCZ** were characterized by thermogravimetric analyzer (TGA) and differential scanning calorimetry (DSC). And the curves are given in Fig. 1. During the DSC measurement, as shown in Fig. 1(a), the plot shows a glass transition temperature (Tg) at 94 °C for **OCZ**, and no crystallization or phase change is observed upon heating beyond Tg. For an amorphous material, the Tg is the critical temperature, at which the material changes its behavior from a hard and relatively brittle state into a molten or rubber-like state. Thus the result of DSC shows the physical state of **OCZ** is very stable at the usual detection temperature. In the TGA measurements, as shown in Fig. 1(b), **OCZ** exhibits excellent thermal stability with Td (the degradation temperature at 5% weight loss under nitrogen atmosphere) up to 422 °C. The results indicate that it is stable enough for the application as a chemosensory material.

3.2. *Optical properties*

The optical properties of **OCZ** were investigated by absorption and photoluminescence (PL) spectra and shown in Fig. 2. As can be seen, the absorption spectrum of **OCZ** in THF solution displays three main absorption peaks at 262, 292 and 366 nm, which are assigned to the peripheral carbazole units and the fluorene backbone, respectively. The PL spectrum shows the PL maximum at 408 nm with a shoulder at 430 nm, which is a typical fluorene emission. The fluorescence efficiency of **OCZ** in solution was determined to be 98% by using quinine sulfate (0.1 M, H_2SO_4 solution) as standard. And the high fluorescence efficiency is one of the essential features of a promising fluorescent sensing material.

3.3. *Anion detection*

The sensing properties of **OCZ** toward iodide were studied as well as other common anions. The **OCZ** solution was diluted to a concentration of 1.0×10^{-6} M. The anions were potassium salts, viz. F^-, Cl^-, Br^-, I^-, NO_3^-, AcO^- and CO_3^{2-}, with a concentration of 0.1 M. These anions are very common and have the significant influences on the environment and human physiology, especially the halogen elements [20]. But the similar chemical properties of

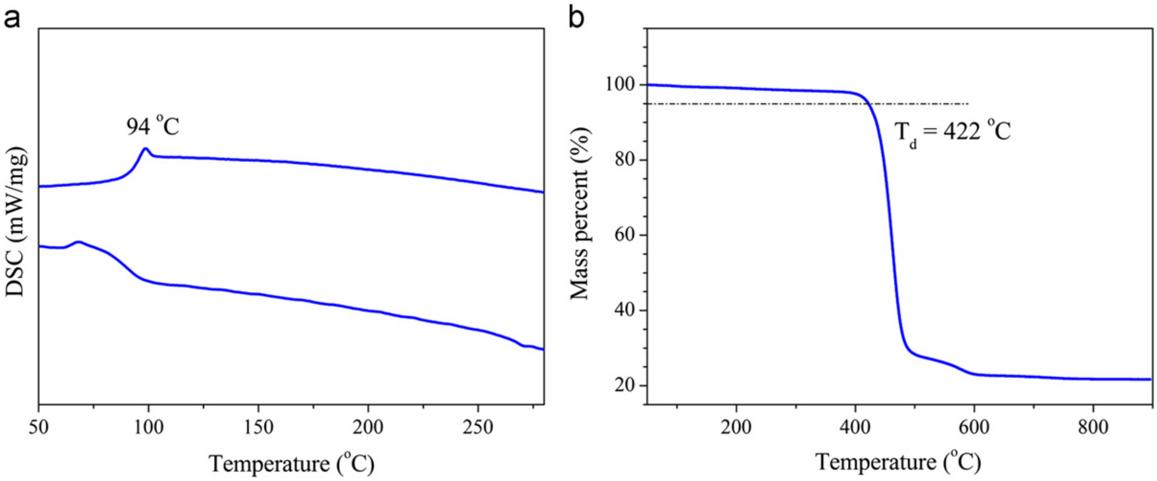


Fig. 1. The DSC (a) and TGA (b) curves of **OCZ**.

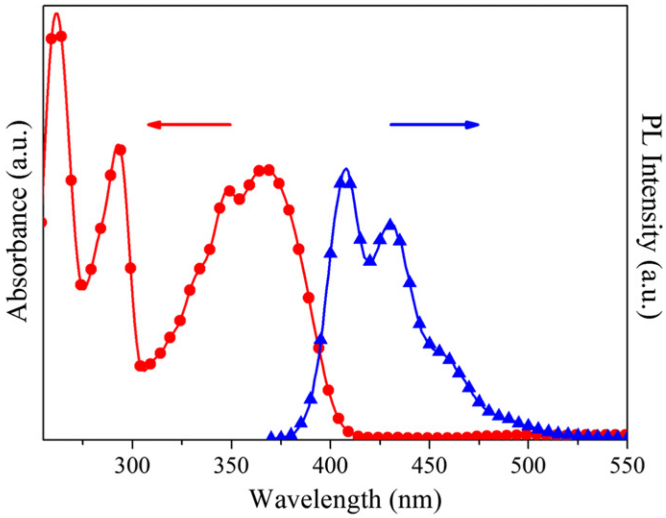


Fig. 2. The absorption (solid round) and PL (solid triangle) spectra of **OCZ** in THF solution.

these halogens would make them difficult to distinguish from each other. Thereby, the selective detection of halogens is of great practical significance. Interestingly, upon the addition of iodide salt, the initial colorless solution of **OCZ** changes to a yellow color rapidly. And the yellow color becomes more noticeable with the addition of I^-, which is shown in the inset of the Fig. 3(a). It should be noted that the color change is very sensitive. Even at a low concentration of 0.1 mM for I^-, the color change can be observed with naked eyes immediately. This colorimetric sensitivity of **OCZ** can be comparable with some selective iodide sensing organic compounds, such as the reports of Leclerc group and Huang group in which the detection limits are 0.45 mM and 0.25 mM, respectively [21,22]. Recently, Yang group published a simple core/shell nanoparticle as the highly selective iodide sensor with the detection limit of 6 μM [23]. Compared with this inorganic sensing material, **OCZ** has the advantage on the response time: **OCZ** shows the instant color change, while the color change of this inorganic nanomaterial could be recognized by naked eyes in 20 min. Therefore, taking both detection limits and response times into account, **OCZ** is still a promising material for iodide recognition.

The changes in the photophysical properties of **OCZ** upon adding I^- were observed in the absorption and PL spectra. The results are presented in Fig. 3. As can be seen, no new absorption bands appear but the absorption intensity gradually increases in the absorption spectra of **OCZ** upon the addition of I^- (see Fig. 3(a)). Meanwhile, I^- could quench the fluorescence of **OCZ**. Fig. 3(b) shows the influence of I^- on the PL spectra of **OCZ**. The emission decreases notably upon the addition of I^-. The fluorescence of **OCZ** could be quenched almost 100% by the addition of 0.3 mM I^-.

The inset of Fig. 3(b) is the titration curve of **OCZ** toward I^-. The fluorescent quenching coefficient Ksv is further calculated from the slope of the titration curve according to the modified Stern–Volmer equation,

$$\lg(I_0 - I)/I = \lg Ksv + n\lg(Q)$$

where I_0 and I are the fluorescence intensity in the absence and presence of I^-, respectively. Ksv is the quenching constant, Q is the concentration of I^-, and n is the ratio between iodide and **OCZ**. From the Stern–Volmer plot, the n and Ksv are calculated as 2 and 3×10^8, respectively. The Ksv is relatively higher among the reported anion sensing materials [24,25].

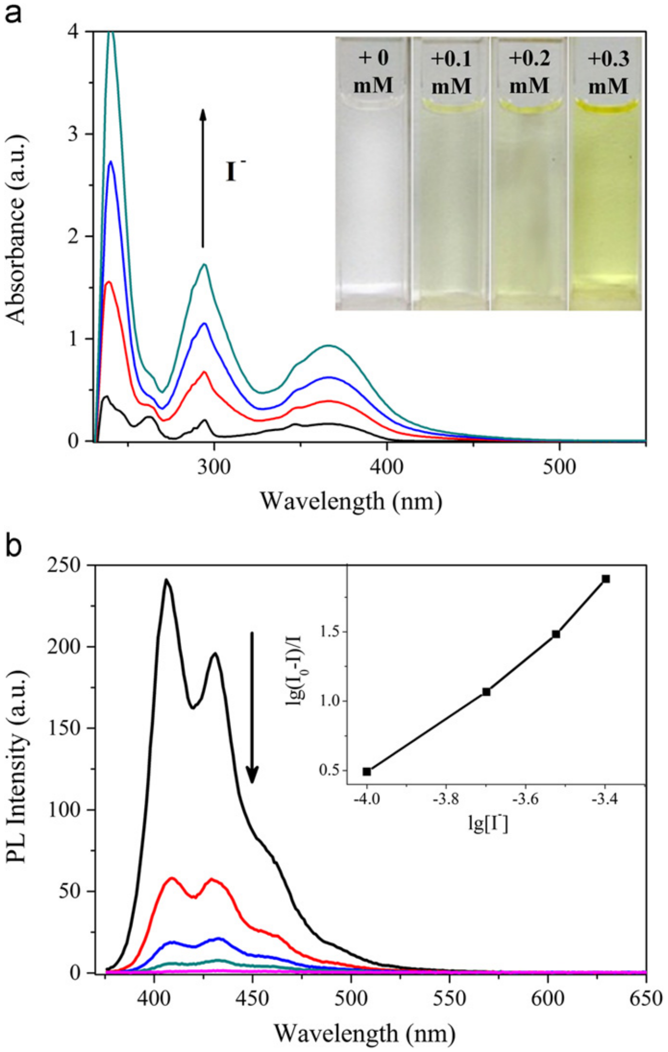


Fig. 3. (a) Variation of color, absorption response of **OCZ** upon the addition of I^- (the absorption curves from bottom to top correspond to the different adding volumes of I^- which are 0, 0.1, 0.2 and 0.3 mM, respectively); (b) PL spectra of **OCZ** (1.0×10^{-6} M) solution in THF after adding I^- (the concentration of I^- from top to bottom is 0, 0.1, 0.2, 0.3 and 0.4 mM, respectively). The inset is the Stern-Volmer plot.

For comparison, optical ion responsive properties of Poly(9,9-di-n-octylfluorenyl-2,7-diyl) (**PFO**) were also tested and the results are shown in Fig. 4. **PFO** is commercially available with the M_w of 54 k, and its chemical structure is shown in Scheme 1. Compared with **OCZ**, **PFO** has no carbazole side chains. The fluorescence emission spectra were measured by exciting the **PFO** solutions (in THF) at 389 nm. During the detection test, although **PFO** should have the stronger fluorescence quenching ability because of the long conjugation length, it showed very weak response to I^-.

According to the literatures, the mechanism of fluorescence quenching includes ground-state complexation, charge-transfer phenomena, electronic energy transfer, fluorescence resonance energy transfer (FRET), heavy atom effect, magnetic perturbations, etc [26–28]. In our case the "heavy-atom" effect is proposed, which is consistent with the reports of Valiyaveettil and Jang [14,29,30]. The heavy-atom effect is the enhancement of the rate of a spin-forbidden process by the presence of an atom with high atomic number, which is either part of, or external to, the excited molecular entity. Mechanistically, it responds to a spin–orbit coupling enhancement produced by a heavy atom [30].

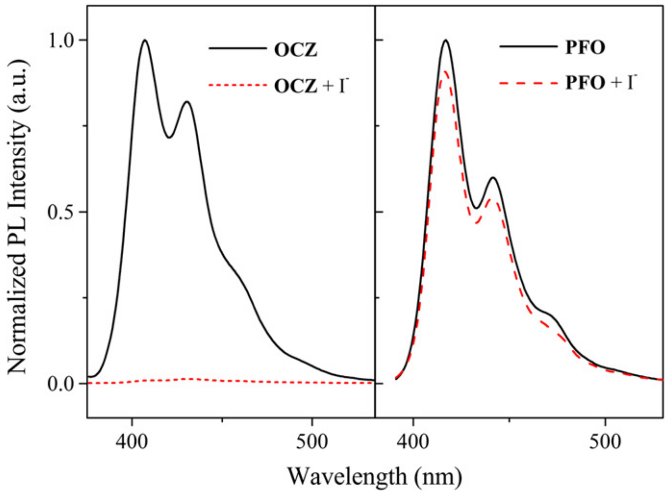


Fig. 4. The normalized PL response of **OCZ** (left) and **PFO** (right) upon addition of I^- (0.3 mM).

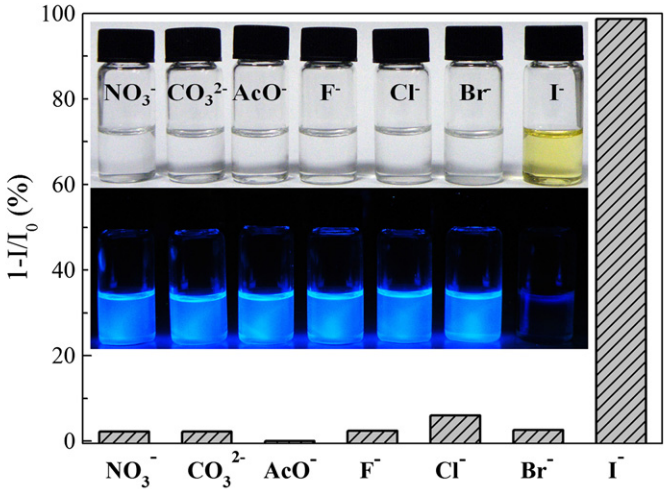


Fig. 5. PL response of **OCZ** upon addition of 0.3 mM different anions and the comparison photos of the color and fluorescence changes with different anions. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

In our case, the “heavy-atom” interaction between the excited state of the **OCZ** and the iodide leads to an enhancement of the spin–orbit coupling and the supervenient fluorescence quenching. Furthermore, the comparison between **OCZ** and **PFO** indicates that the introduction of carbazole groups is beneficial for the iodide detection. In other words, it concludes that the carbazole might help to capture I^- and improve the spin-orbit coupling efficiency. Moreover, a detailed study of the heavy-atom effect in the case of C_{70} and two different quenchers, iodide and bromobenzene, provided evidence for a quenching mechanism with the exponential distance dependence [31]. According to that, the peripheral carbazole groups would also play the role of expanding the effective sensing range of the fluorometric **OCZ** to I^- in solution, which might contribute to the high quenching efficiency.

Except I^-, **OCZ** presents nearly no response to other common anions. Fig. 5 shows the PL responses of **OCZ** upon the addition of 0.3 mM different anions and the comparison photos of the color and fluorescence changes with different anions. As can be seen, **OCZ** can detect I^- by dual-channel assays of colorimetry and fluorometry with high sensitivity.

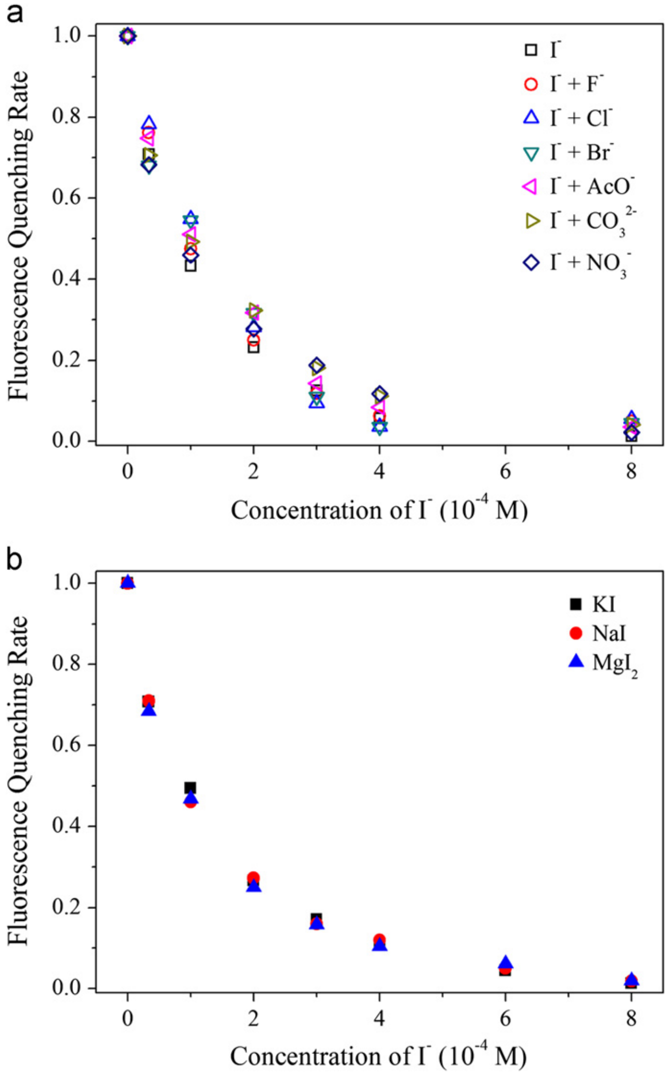


Fig. 6. The fluorescence quenching rate of **OCZ** in the presence of I^- and the mixtures mixed with the equal amount of other anions (a), and in the presence of different iodide salts (b).

To test the practical applicability of **OCZ** as an iodide selective sensor, the competition experiments were carried out in the presence of various concentrations of I^- mixed with the equal amount of other anions F^-, Cl^-, Br^-, AcO^-, CO_3^{2-} and NO_3^-. As shown in Fig. 6(a), although there are some small-scale fluctuations, the similar fluorescence quenching rates are observed with and without the other anions. The influence of cations of iodide salts to **OCZ** I^- detection is also studied. As shown in Fig. 6(b), we can find that varying the cation of the iodide salt has nearly no influence on the detection sensitivity of **OCZ**. These demonstrate that **OCZ** has a high selectivity for iodide.

In order to confirm the practicability of **OCZ** for analyzing the real world samples, the real water samples [32], including the deionized water, tap water, snow water, pond water and lake water of the South Lake at Changchun City, were collected and directly used in the I^- detection experiments. The pure **OCZ** solution is used as the reference; the PL responses of **OCZ** to different water samples (1% v/v) and the water samples spiked with 0.3 mM I^- are recorded and the results are displayed in Fig. 7. Compared with the first two columns in each group, the similar PL intensities imply that these real water samples have little interference in the performance of this fluorescence sensing system. But when added I^- into these blend systems, the PL

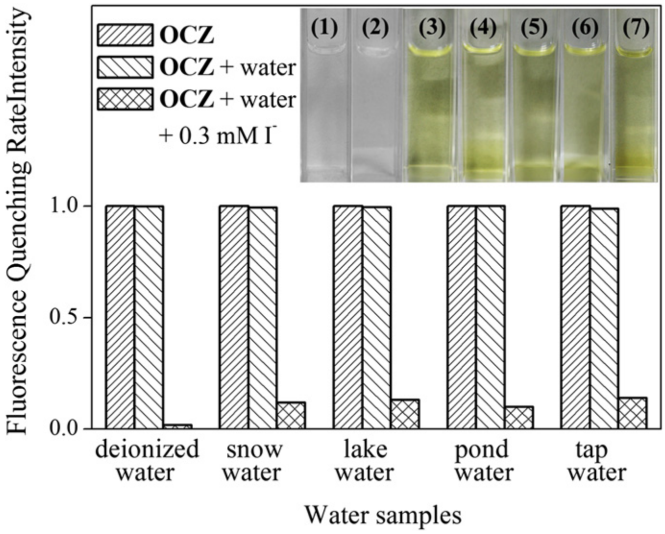


Fig. 7. The PL response of **OCZ** to different water samples (1% v/v) and the water samples spiked with 0.3 mM I^-. The inset pictures are the color changes of the mixed solutions. The samples from left to right are (1) **OCZ**, (2) **OCZ**+water sample (no obvious color change for all water samples), (3) **OCZ**+deionized water+I^-, (4) **OCZ**+snow water+I^-, (5) **OCZ**+lake water+I^-, (6) **OCZ**+pond water+I^- and (7) **OCZ**+tap water+I^-.

intensity of the mixture solution is significant decreased. Although the fluorescence quenching degrees are slightly lower than that of the pure **OCZ** solution which has been shown above, the changes of the PL intensities are almost all over 85%. Furthermore, the obvious color changes are observed upon the addition of I^- (see the inset pictures in Fig. 7) and it can be distinguished very easily. It is shows that **OCZ** is still sensitive and efficient in such complex systems. These results further reveals that this novel fluorometric/colorimetric probe material has great potential for the iodide detection in the real environmental samples.

4. Conclusions

In summary, we have developed a new type fluorene oligomer **OCZ** that could act as dual-channel assays of colorimetry and fluorometry with high sensitivity and selectivity to I^-. And the results indicate that the carbazole side chains play an important role in the detection of iodide. Based on this, **OCZ** is a promising material for chemosensory application with high performance and provides a new pathway for the molecular design for the chemosensory materials.

Acknowledgements

We are grateful to the financial support from National Science Foundation of China (grant numbers 20704015, 50973041), Ministry of Science and Technology of China (grant number 2009CB939701).

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