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(S)-Binaphthalene-based fluorescence polymer sensors for direct and visual F^- detection

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ABSTRACT

(*S*)-binaphthalene-based polymer sensors **P-1** and **P-2** could be synthesized by the polymerization of 5,5′-((2,5-dibutoxy-1,4-phenylene)bis(ethyne-2,1-diyl))bis-(2-hydroxy-3-(piperidin-1-ylmethyl) benzaldehyde (**M-1**) with (*S*)-2,2′-dimethoxy-(1,1′-binaphthalene)- 6,6′-diamine (**M-2**) and (*S*)-2,2′-dimethoxy-(1,1′-binaphthalene)-3,3′-diamine (**M-3**) *via* nucleophilic addition–elimination reaction. The fluorescence emission of **P-1** and **P-2** could exhibit gradual enhancement behavior upon the addition of F^-. Compared with other anions, such as Cl^-, Br^-, I^-, HSO_4^-, ClO_4^-, $H_2PO_4^-$, AcO^-, and NO_3^-, F^- could lead to the most remarkable fluorescence enhancement as high as 3.67-fold for **P-1** and 12.85-fold for **P-2**, respectively. More importantly, both two polymer sensors **P-1** and **P-2** can display a bright yellow fluorescence color change for F^- under a commercially available UV lamp, which could be clearly observed by the naked eyes for direct visual detection of F^-.

1. Introduction

In the past few years much attention has been paid to the detection of fluoride anion (F^-) since F^-, as the smallest anion, is of great importance for health and environmental issues [1–7]. Fluoride could be easily absorbed by human body and slowly excreted. As a result, in the appropriate concentration ranges, F^- plays an essential role in preventing dental decay and treating with osteoporosis. But in excess, F^- can cause fluorosis and other bone related diseases [8]. Recently, there have been many reports on fluorescent chemosensors for the highly selective and sensitive detection of anions focused on either a hydrogen-bonding interactions [9–18], anion-π interactions [26–30], Lewis acid–base interactions [19–25] or a chemodosimeter approach [26–30].

Although there have been some reports on fluorescence sensors for F^- detection, most of them were focused on small organic molecules [31–34], only a few were polymer-based sensors [35–41]. Compared with small molecule sensors, the advantages of fluorescence polymer sensors for direct ion detection demonstrate that they can make use of the high sensitivity of conjugated polymers to electron density changes of the conjugated polymer backbone and to external structural perturbations when they can interact with analytes [42,43]. Swager reported that the delocalized π-electronic conjugated "molecular wire" polymer can tremendously amplify the fluorescence response signal in consequence of facile energy migration along the polymer backbone upon light excitations. As a consequence, the conjugated polymer-based sensor can lead to the greatly enhanced fluorescence response behavior relative to one of its monomer units [44,45].

Optically active binaphthalene derivatives, for example 2,2′-binaphthyladiamine (BINAM) and 2,2′-binaphthol (BINOL), are two of the most representative C_2-symmetric compounds. The skeletal structure of BINOL is a well-known building block, which can be systematically modified at a well-defined molecular level by the introduction of various functional groups at the 3,3′-, 5,5′-, and 6,6′-positions, leading to a variety of polybinaphthyl compounds with a stable chiral configuration, as well as high chiral induction and chiral discrimination for fluorescence sensors and asymmetric catalysis [46–53]. To the best of our knowledge, so far there has been no report on binaphthalene-based polymer as sensor for F^- recognition detection. In this paper, we designed two (*S*)-binaphthalene-based fluorescence polymers **P-1** and **P-2** as colorimetric chemosensors for direct and visual detection of F^- over other anions at low concentration.

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2. Experimental section

2.1. Materials and measurements

All solvents and reagents were commercially available and analytical reagent grade. THF and Et_3N were purified by distillation from sodium in the presence of benzophenone. NMR spectra were collected on a Bruker 300 spectrometer (300 MHz for ^1H NMR and 75 MHz for ^{13}C NMR) and reported as parts per million (ppm) from the internal standard TMS. Fluorescence spectra were obtained from a Perkim ls-55 spectrometer. The circular dichroism (CD) spectrum was determined with a Jasco J-810 spectropolarimeter. Specific rotation was determined with a Ruololph Research Analyfical Autopol I. C, H, and N of elemental analyses were performed on an Elementar Vario MICRO analyzer. Thermogravimetric analyses (TGA) were performed on a PerkinElmer Pyris-1 instrument under a N_2 atmosphere. Molecular weight was determined by gel permeation chromatography (GPC) with a Waters 244 HPLC pump, and THF was used as solvent relative to polystyrene standards.

2.2. Synthesis of compound M-2

A mixture of the nitro precursor **1** (0.61 g, 1 mmol) dissolved in DMF (10 mL) and 10% Pd/C (0.04 g) was stirred at ambient temperature under a 1 bar atm of hydrogen overnight. The reaction mixture was filtered through a pad of Celite, and concentrated under reduced pressure. The product was purified by column chromatography on silica gel column chromatography (EtOAc/petroleum ether, 1:5 v/v) to afford the desired product (0.49 g, 1.43 mmol) in 95% yield and characterized by ^1H NMR (300 MHz, $CDCl_3$): 8.90 (d, $J = 2.4$ Hz, 2H), 8.24 (d, $J = 9.3$ Hz, 2H), 8.24 (dd, $J = 9.5$ Hz, $J = 2.4$ Hz, 2H), 7.63 (d, $J = 9.0$ Hz, 2H), 7.15 (d, $J = 9.6$ Hz, 2H), 3.86 (s, 6H).

2.3. Synthesis of compound M-3

(*S*)-2,2′-Dimethoxy-1,1′-binaphthalene (4.21 g, 13.4 mmol, 1.0 equiv.) and N,N,N′,N′- tetramethylethylenediamine (TMEDA, 8.00 mL, 53.4 mmol, 4.0 equiv.) were dissolved in Et_2O (300 mL), and the solution was cooled to 0 °C. *n*-BuLi (14.7 mL, 2.5 m in *n*-hexane, 36.7 mmol, 2.75 equiv.) was added dropwise and the resulting mixture was stirred for 2 h at room temperature. The reaction mixture was cooled to 0 °C and TsN_3 (7.90 g, 40.1 mmol, 3.0 equiv., Ts = 4-toluenesulfonyl) was added dropwise. The resulting mixture was stirred for 1 h at 0 °C and for a further 12 h at room temperature. The reaction was quenched with water (50 mL) and extracted with EtOAc (3 × 100 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The crude product **2** was directly used for the next product. A suspension of $LiAlH_4$ (40 mmol, 1.5 g in 10 mL Et_2O) was cooled to 0 °C, a solution of the crude product 2 in Et_2O (25 mL) was added dropwise into the suspension solution, and the resulting mixture was stirred for 12 h at room temperature. Water (1.5 mL), NaOH (15%, 1.5 mL), and water (4.5 mL) were added to the reaction mixture sequentially at 0 °C, and the mixture was stirred for 0.5 h. Filtration through diatomaceous earth filter aid and flash column chromatography on silica gel (EtOAc/petroleum ether, 1:2 v/v) afforded the desired product (2.45 g, 7.10 mmol) as a slightly brown solid in 53% yield. ^1H NMR (300 MHz, $CDCl_3$): $d = 7.65$ (d, $J = 8.2$ Hz, 2H), 7.29 (d, $J = 6.8$ Hz, 2H), 7.21 (s, 2H), 7.10–7.00 (m, 4H), 3.37 (s, 6H).

2.4. Synthesis of polymer P-1

Monomer **M-1** (75.8 mg, 0.22 mmol) and **M-2** (0.13 g, 0.22 mmol) were dissolved in 10 mL toluene. The mixture solution was stirred at 80 °C for 48 h. MeOH (20 mL) was added to precipitate the yellow polymer. The resulting polymer was filtered, washed several times with MeOH, and dried under vacuum (0.13 g, 66% yield). $[\alpha]_D^{25} = -4.0$ ($c = 0.10$, THF); ^1H NMR (300 MHz, $CDCl_3$): $d = 8.80$ (s, 2H), 8.06–7.03 (m, 2H),7.78–7.71 (m, 2H), 7.60 (s, 2H), 7.56–7.51 (m, 4H), 7.21–7.18 (m, 2H), 7.02–6.99 (m, 4H), 4.04 (m, 6H), 3.82 (m, 4H), 3.75 (s, 4H), 3.70 (s, 2H), 2.57 (br s, 10H), 1.87 (m, 6H), 1.69–1.29 (m, 24H), 0.86 (m, 6H); GPC: $M_w = 16950$, $M_n = 5930$, PDI = 2.86; elemental analysis calcd (%) for $C_{58}H_{52}N_4O_6$: C 77.24, H 5.77, N 6.22; found: C 77.34, H 6.05, N 6.18.

2.5. Synthesis of polymer P-2

P-2 could be obtained by the similar synthesis method as **P-1** *via* polymerization reaction of **M-1** (75.8 mg, 0.22 mmol) with **M-3** (0.13 g, 0.22 mmol) in 71% yield. $[\alpha]_D^{25} = +68.0$ ($c = 0.10$, THF); ^1H NMR (300 MHz, $CDCl_3$): $d = 8.99$ (s, 2H), 7.95–7.88 (m, 2H),7.74–7.67 (m, 2H), 7.60 (s, 2H), 7.47–7.39 (m, 2H), 7.25–7.20 (m, 4H), 7.06–6.99 (m, 4H), 4.07 (dd, $J = 6.32$, 11.34 Hz, 6H), 3.73 (d, $J = 10.89$ Hz, 4H), 3.64 (s, 4H), 3.56 (s, 2H), 2.57 (br s, 10H), 1.88 (m, 6H), 1.69–1.29 (m, 24H), 0.85 ppm (m, 6H); GPC: $M_w = 17890$, $M_n = 6400$, PDI = 2.80; elemental analysis calcd (%) for $C_{58}H_{52}N_4O_6$: C 77.24, H 5.77, N 6.22; found: C 77.07, H 6.00, N 6.34.

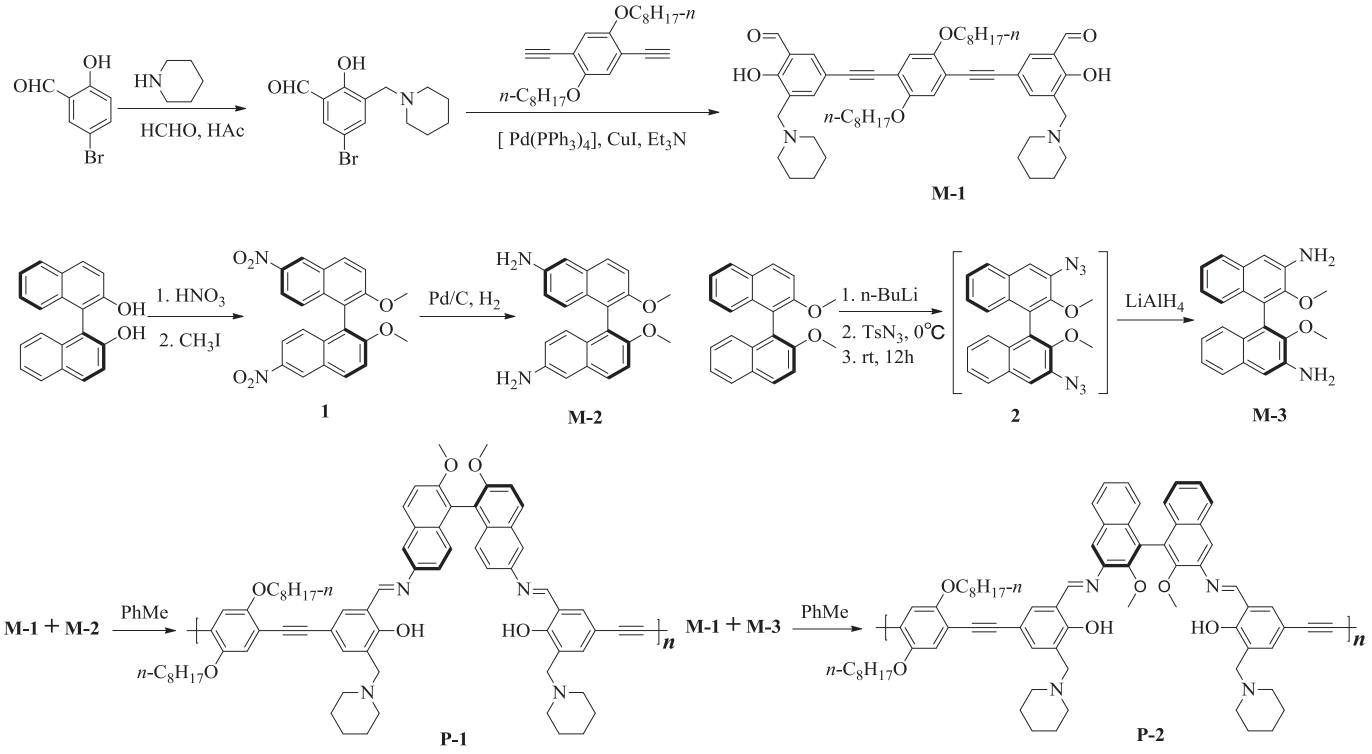
3. Results and discussion

3.1. Synthesis and structure feature of the polymers

The synthesis procedures of the chiral polymer sensors **P-1** and **P-2** are outlined in Scheme 1. Monomers 5,5′-((2,5-dibutoxy-1,4- phenylene)bis(ethyne-2,1-diyl))bis(2-hydroxy-3-(piperid-in-1-ylmethyl) benzaldehyde (**M-1**), (*S*)-2,2′-dimethoxy-(1,1′-binaphthalene)-6,6′-diamine (**M-2**), (*S*)-2,2′-dimethoxy-(1,1′-binaphthalene)-3,3′-diamine (**M-3**) and **P-2** could be synthesized according to the reported literatures [54–56]. **P-1** and **P-2** could be synthesized by the polymerization of **M-1** with **M-2** and **M-3** *via* nucleophilic addition–elimination reaction, respectively. These two polymers **P-1** and **P-2** were further purified by washing with MeOH and collected as yellow and red solids, respectively. **P-1** and **P-2** could be easily dissolved in common organic solvents (THF, toluene, CH_2Cl_2, $CHCl_3$) due to the nonplanarity of the twisted polymer chain backbone and the flexible *n*-octyl substituents. GPC results of **P-1** and **P-2** show moderate molecular weights. TGA of **P-1** and **P-2** were carried out under a N_2 atmosphere at a heating rate of 10 °C/min. TGA results show that both polymers have high thermal stability, with no weight loss before 350 °C, and tend to completely decompose at 700 °C, indicating that these two polymers can provide desirable thermal properties for practical applications as fluorescence sensors.

3.2. Optical properties

The UV-vis absorption spectra of polymer sensors **P-1** and **P-2** towards various anions were conducted in THF (1×10^{-5} mol/L) at room temperature. All anions are in the form of tetrabutylammonium (TBA) salts. As shown in Fig. 1a, **P-1** exhibits a maximal absorption at 377 nm and two other absorption bands situated around 284 and 322 nm. The sharpest absorbance peak at 377 nm



Scheme 1. Synthesis of monomers and polymers.

can be attributed to the effective $\pi-\pi^*$ conjugated segments of the polymer main chain backbone. Upon the addition of F^-, the absorbance peaks at 322 and 377 nm appear an obvious reduction, meanwhile, two new peaks in the wavelength region at 425 and 479 nm show gradual enhancement with a bathochromic shift of 48 nm and 102 nm, respectively. And there is an isosbestic point centered at 403 nm, indicating an unusual interaction between F^- and **P-1** [7,15,16]. As to polymer **P-2**, it exhibits a maximal absorption at 373 nm and two other absorption bands around 282 and 321 nm. The strongest absorbance peak shows the obvious decrease upon the addition of F^-, but a new absorption peak at 421 nm turns on gradual increase with one clear isosbestic point at 397 nm. The solution color changes of **P-1** and **P-2** could be clearly observed from colorless to yellow by the naked eyes (Fig. 1, inset). Colorimetric sensing **P-1** and **P-2** can exhibit the ratiometric absorption changes with the obvious red shifts for only F^- anion, which indicates that F^- as the maximal electronnegativity element can affect the stable intramolecular hydrogen bond structure between the imine and hydroxyl of phenol in the main chain backbone of polymer sensors, thus promote p-π conjugation effect from imine groups and lead to the extended π-electronic structure in the new species with stronger push–pull electron *via* electrostatic interactions [57,58]. In contrast, almost no absorption spectra or visual color change of **P-1** and **P-2** could be detected for other anions as shown in Fig. 2 except F^-.

The fluorescence responses of the polymers **P-1** and **P-2** towards various anions were investigated in THF (1×10^{-5} mol/L) by using the same excitation wavelength 367 nm. As is evident from Fig. 3a, the chiral polymer **P-1** shows a fluorescence emission at 471 nm. Upon the addition of F^-, the emission shows the gradual decrease, and a new peak at 580 nm arises and shows gradual enhancement with a large bathochromic shift of 109 nm. The maximal fluorescence enhancement can reach as high as 3.67-fold at 100 equiv. of F^-. In view of **P-2** (Fig. 4), it emits fluorescence

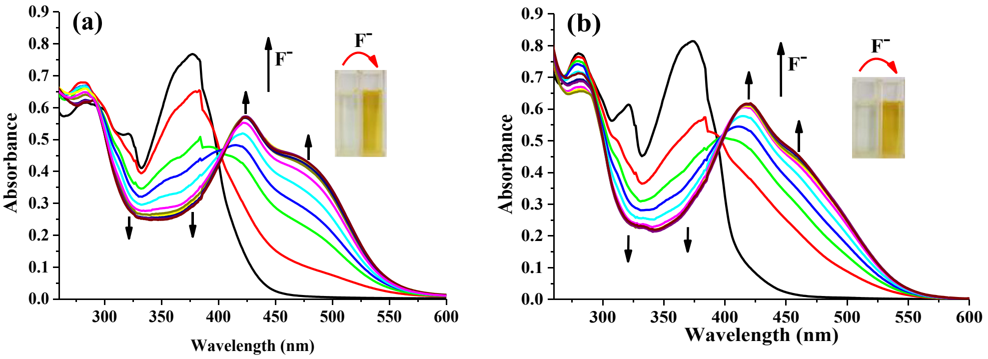


Fig. 1. UV-vis spectra of (a) **P-1** (1×10^{-5} mol/L in THF, inset: color changes upon the addition of F^-) and (b) **P-2** (1×10^{-5} mol/L in THF, inset: color changes upon the addition of F^-) in the presence of F^- (0, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100 equiv.).

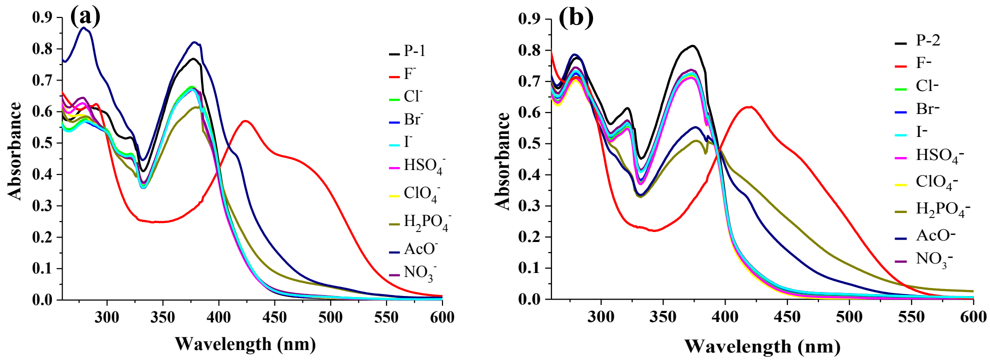


Fig. 2. UV-vis spectra of (a) **P-1** (1×10^{-5} mol/L in THF) and (b) **P-2** (1×10^{-5} mol/L in THF) in the presence of 100 equiv. of various anions.

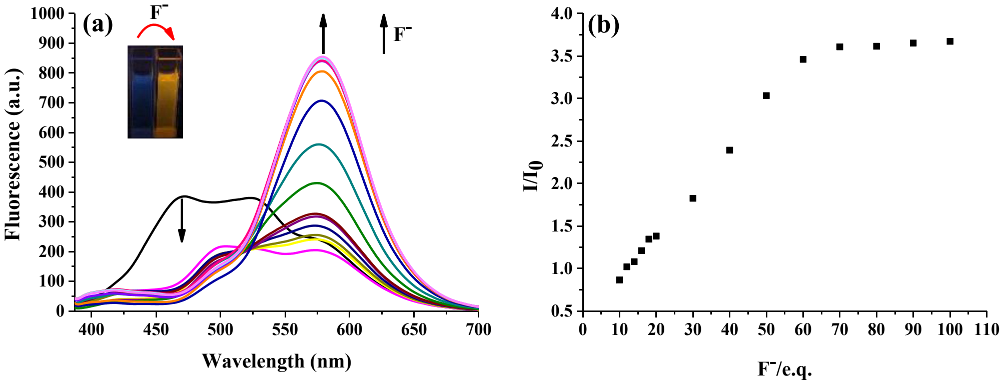


Fig. 3. Fluorescence spectra of (a) **P-1** (1×10^{-5} mol/L in THF, $\lambda_{ex} = 367$ nm) and relative fluorescence intensity values (I/I_0) for (b) **P-1** (1×10^{-5} mol/L in THF) in the presence of F^- (0, 10, 12, 14, 16, 18, 20, 30, 40, 50, 60, 70, 80, 90, 100 equiv.). Inset: emission observed from solution of **P-1** and **P-1**/F^- under UV lamp ($\lambda_{ex} = 365$ nm).

centered at 469 nm. But the original fluorescent emission peak completely disappeared with the molar ratio increase of F^- from 1:0 to 1:30 equiv., meanwhile two new fluorescence emissions centered at 506 nm and 595 nm come up. On further increasing the concentration of F^-, the fluorescence emission at 506 nm no longer increases, but the fluorescence emission centered at 595 nm still gradually increases with a bathochromic shift of 109 nm. It was clearly observed that the fluorescence enhancement response of **P-2** at 595 nm can reach as high as 12.85-fold in the presence of 100 equiv. of F^-. The large red shift together with the obvious fluorescence enhancement behaviors of polymers **P-1** and **P-2** only towards F^- could be attributed to the excited-state intermolecular proton transfer (ICT) through the strong intermolecular hydrogen-bonding interactions between hydroxyl of phenol and F^- [59–61]. Most interestingly, the color of the solutions of polymers **P-1** and **P-2** change from fresh blue to bright yellow after the addition of F^- under a commercially available UV lamp ($\lambda_{ex} = 365$ nm), which can be clearly observed by the naked eyes. The stoichiometry for F^- with **P-1** or **P-2** could be determined on the basis of the Benesi-Hildebrand expression: $1/(F{-}F_0) = 1/\{K(F_{max} - F_0)[F^-]^n\} + 1/(F_{max} - F_0)$, where F_0 and F are the fluorescence intensities in the absence and presence of F^-, respectively. Herein, F_{max} is the maximum fluorescence intensity in the presence of F^-, and K is the associated constant [7,62–64]. Plotting the $1/(F - F_0)$ quantity

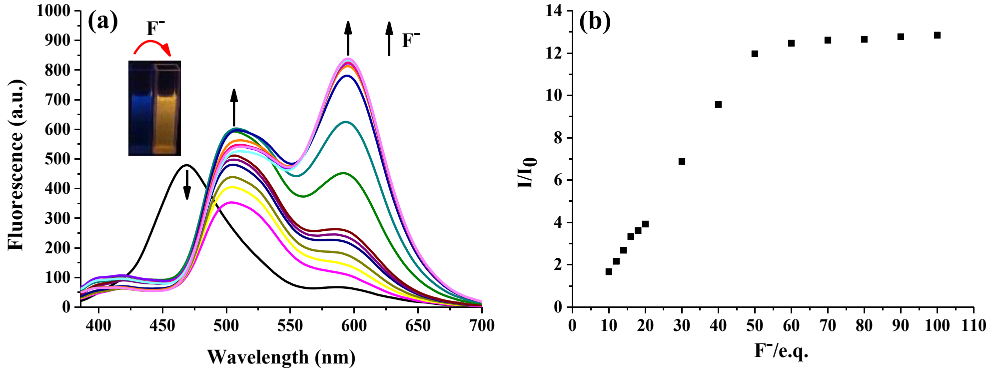


Fig. 4. Fluorescence spectra of (a) **P-2** (1×10^{-5} mol/L in THF, $\lambda_{ex} = 367$ nm) and relative fluorescence intensity values (I/I_0) for (b) **P-2** (1×10^{-5} mol/L in THF) in the presence of F^- (0, 10, 12, 14, 16, 18, 20, 30, 40, 50, 60, 70, 80, 90, 100 equiv.). Inset: emission observed from solution of **P-2** and **P-2**/F^- under UV lamp ($\lambda_{ex} = 365$ nm).

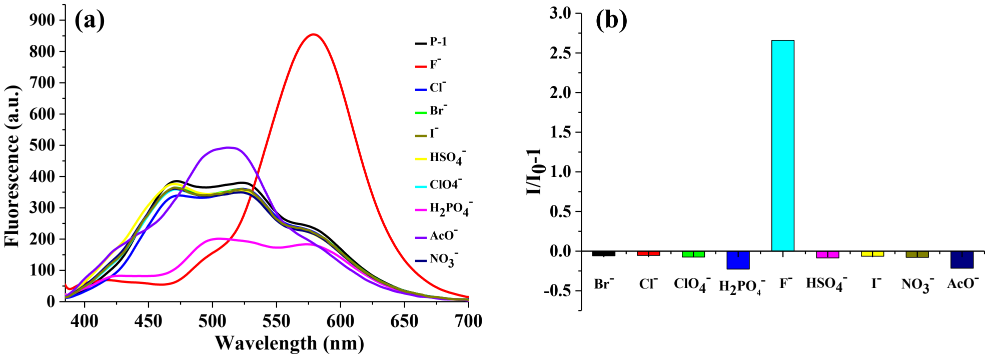


Fig. 5. The selectivity of the polymers towards F$^-$ and other anions. (a) Fluorescence spectra of **P-1** (1×10^{-5} mol/L in THF) and (b) Fluorescence enhancement values (I/I_0−1) of **P-1** (1×10^{-5} mol/L in THF, $\lambda_{em} = 580$ nm) in the presence of 100 equiv. anions.

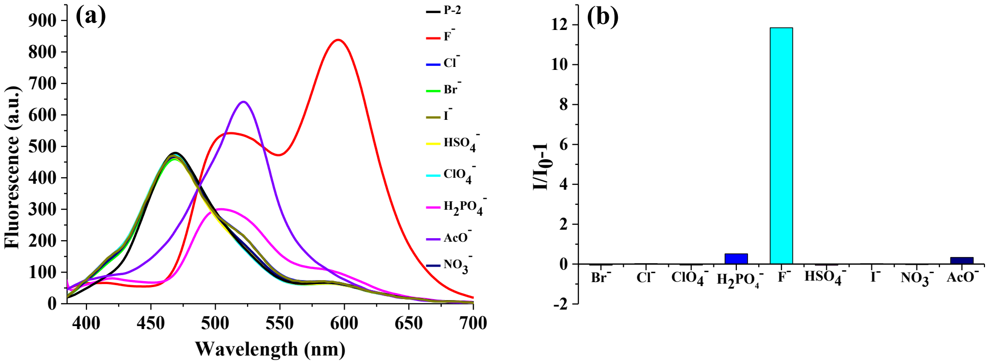


Fig. 6. The selectivity of the polymers towards F$^-$ and other anions. (a) Fluorescence spectra of **P-2** (1×10^{-5} mol/L in THF) and (b) Fluorescence enhancement values (I/I_0−1) of **P-2** (1×10^{-5} mol/L in THF, $\lambda_{em} = 595$ nm) in the presence of 100 equiv. anions.

against $[1/\mathrm{F}^-]^2$ ($n = 2$) by using the fluorescence intensity for **P-1** ($\lambda_{em} = 580$ nm) and **P-2** ($\lambda_{em} = 595$ nm) gave an excellent linear curve, indicating that there is a 1:2 binding mode between **P-1** or **P-2** and F$^-$. In addition, the associated constants calculated from the slope and intercept of the line are 2.35×10^6 M^{-2} for **P-1** and 5.24×10^6 M^{-2} for **P-2**, respectively (Figs. S5 and S6).

Herein, we further investigated the fluorescence response behaviors of the chiral polymers on other various anions including Cl$^-$, Br$^-$, I$^-$, HSO_4^-, ClO_4^-, $H_2PO_4^-$, AcO$^-$, and NO_3^-. In a set of comparable experiments, the fluorescence spectra of two polymers were performed in the absence and presence of 100 equiv. of anion by using 367 nm excitation wavelength. As can be seen from Figs. 5 and 6, the addition of F$^-$ to the solution of **P-1** or **P-2** can induce a remarkable fluorescence enhancement. By contrast, other anions have less influence on the fluorescence intensity of **P-1** and **P-2** under the same conditions. It was found that AcO$^-$ could lead to a little fluorescence enhancement for **P-1** at 491 nm (Fig. 5a) and for **P-2** at 522 nm (Fig. 6a), but the addition of Cl$^-$, Br$^-$, I$^-$, ClO_4^-, HSO_4^- and NO_3^- could cause slight fluorescence quenching for both **P-1** and **P-2** (Figs. 5 and 6). In addition, $H_2PO_4^-$ could limitedly quench the fluorescence emission with a bathochromic shift of 30 nm and 109 nm for **P-1**, and 37 nm for **P-2**, respectively. We also carried out the responses of the polymer **P-1** and **P-2** on $[Bu_4N]^+\cdot OH^-$ as a strong base. The obvious fluorescence enhancement responses of polymer sensors **P-1** and **P-2** on OH$^-$ was almost similar to F$^-$ (Fig. S7), which further revealed that stronger basicity of F$^-$ could play a very important role in the formation of intermolecular hydrogen bonds and hydroxyl deprotonation process of phenol.

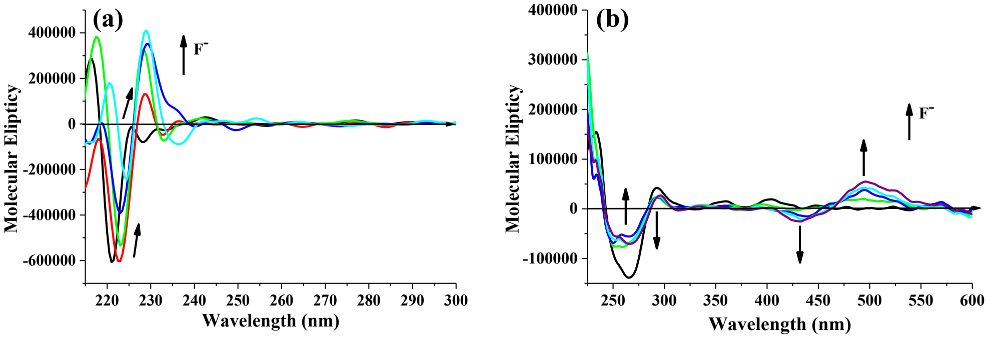


Fig. 7. CD spectra of (a) **P-1** (1×10^{-5} mol/L in THF) and (b) **P-2** (1×10^{-5} mol/L in THF) in the presence of F$^-$ (0, 25, 75, 100 equiv.).

The CD spectra of polymers **P-1** and **P-2** towards F⁻ were investigated in THF (1×10^{-5} mol/L). As is evident from Fig. 7, the chiral polymer **P-1** shows strong positive and negative Cotton effects situated at 216 nm and 221 nm arisen from the feature skeleton absorption of (*S*)-binaphthalene moieties in the polymer main chain. With the increasing amounts of F⁻, the signal at 221 nm gradually decreases with a 4 nm redshift; meanwhile the intensity of the signal at 226 nm shows a tremendous rise with a bathochromic shift of 3 nm. The conjugated chiral polymer **P-2** also has strong positive and negative Cotton effects at 233 nm and 264 nm, respectively. The positive Cotton effect at 403 nm can be regarded as the reflection of the extended conjugated structure in the repeating unit and the high rigidity of polymer backbone. Upon the addition of F⁻, the intensities of the signal centered at 265 nm and 293 nm gradually decrease, but two new signals at 431 nm and 498 nm appear and have a rising trend, which may be ascribed to the enlargement of dihedral angles of binaphthyl upon the formation of host–guest complexes [54,60].

4. Conclusion

Both two (*S*)-binaphthalene-type polymers **P-1** and **P-2** could exhibit selective and sensitive fluorescence enhancement response behavior towards F⁻ as high as 3.67-fold for **P-1** and 12.85-fold for **P-2**. More interestingly, these two chiral polymer colorimetric sensors can display bright yellow fluorescence color change for F⁻ under a commercially available UV lamp, which can be clearly observed by the naked eyes for the direct and visual detection of F⁻ at low concentrations.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.polymer.2014.09.017.

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