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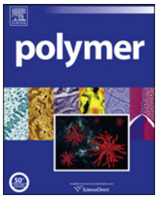

A highly selective and sensitive fluorescence chemosensor based on optically active polybinaphthyls for Hg^{2+}

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ABSTRACT

The chiral polymer was synthesized by the polymerization of 4,7-diethynylbenzo[2,1,3]-thiadiazole (**M-1**) with (*R*)-6,6′-dibutyl-3,3′-diiodo-2,2′-bis(diethylaminoethoxy)-1,1′-binaphthyl (***R*-M-1**) *via* Pd-catalyzed Sonogashira reaction. The chiral polymer has orange fluorescence due to the extended π-electronic structure between binaphthyl unit and benzo[2,1,3]thiadiazole (**BT**) group *via* ethynyl bridge. The responsive optical properties of the polymer on various metal ions were investigated by fluorescence spectra. The fluorescence of the chiral polymer can produce the pronounced enhancement as high as 1.8-fold upon addition of 1:2 molar ratio of Hg^{2+}. Compared with other cations, such as K^{+}, Mg^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Ag^{+}, Cd^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+} and Fe^{3+}, Hg^{2+} can produce the pronounced fluorescence response of the polymer. The result indicates this kind of chiral polybinaphthyls incorporating diethylamino and benzo[2,1,3]thiadiazole (**BT**) moieties as receptors exhibits highly sensitive and selective behavior for Hg^{2+} detection.

1. Introduction

Mercury and its derivatives which have inherently high toxicity are widespread global pollutants and they have caused serious environmental and health problems [1,2]. Therefore, the detection of mercury has received increasing attention in recent years. Many fluorescence chemosensors for mercury detection have been reported, but most of them are based on small molecules [3,4] and polymer-based chemosensors are very few [5–8]. An advantage of fluorescent conjugated polymers over small molecules is that signal amplification occurs from electronic communication along the polymer backbone. Swager et al. reported that the delocalizable π-electronic conjugated “molecular wire” polymer can greatly amplify the fluorescence responsive change due to facile energy migration along the polymer backbone upon light excitations [9]. As a result, a single conjugated polymer provides enhanced optical response relative to one of its monomer units, and conjugated polymers can be used as the optical platforms in highly sensitive chemical and biological sensors [10–14]. Recently, Li and co-workers reported a novel D-π-A alternative copolymer of carbazole and thieno[3,4b]pyrazine which showed relatively high sensitivity and selectivity for mercury ion by taking advantage of the amplified fluorescence quenching ability of conjugated polymers [15].

Optically active 2,2′-binaphthol (BINOL) and its derivatives are one of the most important C_2 symmetric compounds and have often been used as starting materials for the preparation of conjugated polymers that have a main-chain chiral configuration. These rigid and regular chiral binaphthyl-based polymers could lead to the multifunctional properties, such as fluorescence chemosensors for sensitive detection of metal ions or chiral molecules recognition [16–18], asymmetric catalysts [19–21], and optoelectronic materials [22,23] by controlling the chiral organic groups and linker structure at a well-defined molecular level.

Some probes for Hg^{2+} are often based on the coordination of multiple nitrogen atoms with the metal ion or strong Hg–S binding [3], herein, the chiral polybinaphthyls incorporating diethylamino units and benzo[2,1,3]thiadiazole (**BT**) group was designed and synthesized by Sonogashira reaction. Recently, Huang and co-workers reported poly(*p*-phenyleneethynylene) containing **BT** unit used as a highly Hg^{2+}-selective chemosensor, and this kind of polymer showed fluorescence quenching effect on Hg^{2+} [24]. Most of fluorescence chemosensors including small molecules and conjugated polymers on Hg^{2+} exhibit fluorescence quenching effect [3]. Although a few small molecular sensors work in “turn-on” mode [25–28], to our knowledge, very few examples based on polymer chemosensors on Hg^{2+} show a fluorescence enhancement result [5,8]. Since fluorescence quenching is not only disadvantageous for

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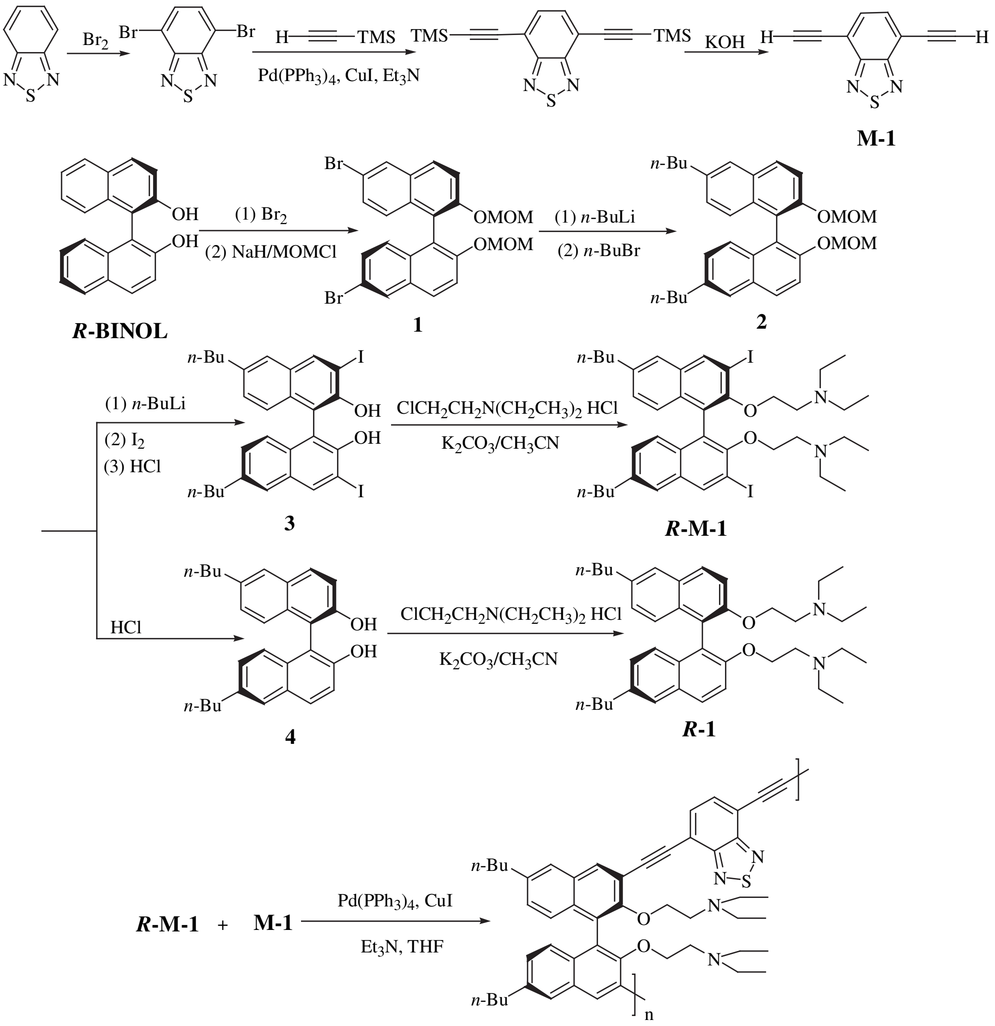

Scheme 1. Synthesis procedures of **M-1**, **R-M-1, R-1** and the chiral polymer.

a high signal output upon complexation, but also hampers temporal separation of spectrally similar complexes with time-resolved fluorometry, design and synthesis of chemsensors with obvious fluorescence enhancement response for mercury detection is still a considerable challenge [29,30]. In this paper, the obtained chiral polybinaphthyls exhibited an obvious fluorescence enhancement upon addition of Hg^{2+}. Compared with other cations, such as K^+, Mg^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Ag^+, Cd^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+} and Fe^{3+}, Hg^{2+} can produce pronounced fluorescence response of the chiral polymer.

2. Experimental part

2.1. Materials

All solvents and reagents were commercially available and analytical-reagent-grade. (*R*)-2,2′-Binaphthol and benzo[2,1,3]thiadiazole were purchased from Aldrich and directly used without purification. THF and Et_3N were purified by distillation from sodium in the presence of benzophenone. CH_3CN were distilled from P_2O_5. 4,7-Dibromobenzothiadiazole [31], (*R*)-6,6′-dibromo-2,2′-bis(methoxymethoxy)-1,1′-binaphthyl (**1**) [32], (*R*)-6,6′-dibutyl-2,2′-bis(methoxymethoxy)-1,1′-binaphthyl (**2**) and (*R*)-6,6′-dibutyl-3,3′-diiodo-2,2′-binaphthol (**3**), (*R*)-6,6′-dibutyl-2,2′-binaphthol (**4**) [33] were prepared for the synthesis of monomers according to reported literatures (Scheme 1).

2.2. Measurements

NMR spectra were obtained using a 300-Bruker 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. FT-IR spectra were taken on a Nexus 870 FT-IR spectrometer. Fluorescence spectra were obtained from an RF-5301PC spectrometer. Specific rotation was determined with a Ruololph Research Analyfical Autopol I. MS was determined on a Micromass GCT. C, H, N and S of elemental analyses were performed on an Elementar Vario MICRO analyzer. Molecular weight was determined by GPC with Waters-244 HPLC pump and THF was used as solvent and relative to polystyrene standards.

2.3. Preparation of ***M-1*** (Scheme 1)

Compound **M-1** was prepared according to a reported method [31]. A mixture of 4,7-dibromobenzo-thiadiazole (2.21 g, 7.5 mmol), $Pd(PPh_3)_4$ (175 mg, 0.15 mmol), CuI (55 mg, 0.30 mmol) and trimethylsilyl acetylene (3.0 mL, 22.5 mmol) was dissolved in 80 mL Et_3N. The reaction mixture was stirred at 75 °C for 5 h under a N_2

atmosphere. The solution was then cooled to room temperature and the solvent was removed under reduced pressure, the residue was extracted with CH_2Cl_2. The organic layer was washed with cool water (25 mL × 3) and aqueous 1.2 mol L^{-1} HCl (30 mL), dried over anhydrous Na_2SO_4, and evaporated in vacuum to dryness. The residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) (10:1, v/v) to give 4,7-bis(trimethylsilylethynyl)-2,1,3-benzothiadiazole (460 mg, 98%) as a pale yellow powder. ^1H NMR ($CDCl_3$, 300 MHz): δ 0.35 (s, 18H), 7.72(s, 2H). A mixture of 4,7-bis(-trimethylsilylethynyl)-2,1,3-benzothiadiazole (900 mg, 2.7 mmol) in 1 mol L^{-1} KOH methanol solution (75 mL) was stirred at room temperature for 1 h. The completion of the reaction was determined by TLC. The reaction mixture was poured into water (25 mL) and extracted with $CHCl_3$ (3 × 35 mL). The combined organic layers were evaporated in vacuo to dryness after dried over anhydrous Na_2SO_4 to give **M-1** (435 mg, 2.36 mmol) as yellow powder. ^1H NMR (300 MHz, $CDCl_3$): δ 3.70 (s, 2H), 7.76 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 158.4, 132.2, 115.1, 82.3, 81.4. TOF-MS (40:800): *m*/*z*: 183.9 [M^+].

2.4. Preparation of ***R*-*M*-*1*** (Scheme 1)

A mixture of compound **3** (0.20 g, 0.31 mmol), K_2CO_3 (0.30 g, 2.16 mmol), and 2-chlorotriethylamine hydrochloride (0.21 g, 1.23 mmol) was dissolved in 30 mL of acetone. The solution was refluxed overnight. The precipitate mixture was filtered away and the filtrate was rotary evaporated. The residue was poured into water and extracted with $CHCl_3$ (3 × 20 mL). The organic layer was washed with water and brine twice and then dried over anhydrous Na_2SO_4. After removal of solvent, the cruder product was purified by chromatography on silica gel with petroleum ether/acetone (15:1, v/v) as an eluent to afford a pale yellow liquid ***R*-M-1** in 69.2% yield (0.18 g). $[\alpha]_D^{25} = -10.0$ (*c* 0.76, THF). ^1H NMR (300 MHz, $CDCl_3$): δ 8.44 (s, 2H), 7.54 (s, 2H), 7.13 (dd, 2H, *J* = 8.7 Hz, 1.5 Hz), 7.45 (d, 2H, *J* = 8.7 Hz), 3.93–3.85 (m, 2H), 3.47–3.39 (m, 2H), 2.72 (t, 4H, *J* = 7.8 Hz), 2.52–2.44 (m, 2H), 2.33–2.24 (m, 2H), 2.12–2.04 (m, 8H), 1.69–1.59 (m, 4H), 1.44–1.29 (m, 4H), 0.94 (t, 9H, *J* = 7.2 Hz), 0.73 (t, 12H, *J* = 7.2 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 153.1, 140.5, 139.1, 132.5, 132.2, 128.8, 125.8, 125.7, 125.2, 92.4, 70.8, 51.2, 47.2, 35.5, 33.3, 22.4, 13.9, 11.7. FT-IR (KBr, cm^{-1}): 2960, 2928, 2858, 1567, 1430, 1376, 1345, 1226, 1032. MS (EI, *m*/*z*): 819 (0.1%), 721 (64%), 650 (5%), 100 (70%), 87 (100%), 72 (21%), 56 (29%), 44 (39%). Anal. calcd for $C_{40}H_{54}I_2N_2O_2$: C, 56.61; H, 6.41; N, 3.30. Found: C, 56.51; H, 6.38; N, 3.35.

2.5. Preparation of ***R*-*1*** (Scheme 1)

A mixture of compound **4** (0.50 g, 1.25 mmol), K_2CO_3 (1.21 g, 8.78 mmol), and 2-chlorotriethylamine hydrochloride (0.86 g, 5 mmol) was dissolved in 30 mL of acetone. The solution was refluxed overnight. The precipitate mixture was filtered away and the filtrate was rotary evaporated. The residue was poured into water and extracted with $CHCl_3$ (3 × 30 mL). The organic layer was washed with water and brine twice and then dried over anhydrous Na_2SO_4. After removal of solvent, the cruder product was purified by chromatography on silica gel with petroleum ether/acetone (10:1, v/v) as an eluent to afford a pale yellow liquid ***R*-1** in 75.5% yield (0.56 g). $[\alpha]_D^{25} = +7.0$ (*c* 0.68, THF). ^1H NMR (300 MHz, $CDCl_3$): δ 7.87 (d, 2H, *J* = 9.3 Hz), 7.62 (s, 2H), 7.38 (d, 2H, *J* = 9.0 Hz), 7.09 (s, 4H), 4.10–4.05 (m, 4H), 2.72 (t, 4H, *J* = 7.8 Hz), 2.57 (t, 4H, *J* = 6.3 Hz), 2.25 (q, 8H, *J* = 7.2 Hz), 1.68–1.63 (m, 4H), 1.44–1.35 (m, 4H), 0.94 (t, 6H, *J* = 7.2 Hz), 0.74 (t, 12H, *J* = 7.2 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 153.7, 138.1, 132.6, 129.7, 128.7, 127.9, 126.2, 125.4, 120.7, 115.5, 68.0, 51.2, 47.2, 35.5, 33.5, 22.4, 14.0, 11.2. FT-IR (KBr, cm^{-1}): 2958, 2927, 2858, 1595, 1500, 1465, 1265, 1245, 1091, 1062. MS (EI, *m*/*z*): 567 (0.1%), 497 (34%), 398 (38%), 355 (13%), 100 (24%), 87 (100%), 72 (17%), 44 (17%). Anal. calcd for $C_{40}H_{56}N_2O_2$: C, 80.49; H, 9.46; N, 4.69. Found: C, 80.40; H, 9.42; N, 4.72.

2.6. Preparation of the chiral polymer (Scheme 1)

A mixture of ***R*-M-1** (163.6 mg, 0.19 mmol), **M-1** (35.5 mg, 0.19 mmol), 8 mol% $Pd(PPh_3)_4$ and 20 mol% CuI (11.0 mg, 0.038 mmol) was dissolved in the mixed solvents of 6 mL THF and 6 mL Et_3N. The solution was stirred at reflux for 3 day under N_2. The mixture was cooled to room temperature, and then was filtered through a short silica gel column in methanol (30 mL) to precipitate out the polymer. The resulting polymer was filtered and washed with methanol several times. Further purification could be conducted by dissolving the polymer in THF to precipitate in methanol again. The polymer was dried in vacuum to give 70.8 mg in 47.9% yield. $[\alpha]_D^{25} = -496.1$ (*c* 0.01, THF). $M_w = 5660$, $M_n = 2630$, PDI = 2.15. ^1H NMR (300 Hz, $CDCl_3$): δ 7.89–7.50 (m, 6H), 7.21–7.03 (m, 4H), 2.89–2.57 (m, 8H), 2.39–2.04 (m, 6H), 1.75–1.55 (m, 6H), 1.39–1.27 (m, 8H), 1.03–0.27 (m, 18H). FT-IR (KBr, cm^{-1}): 2956, 2926, 2855, 2199, 1494, 1435, 1375, 1350, 1260, 1096, 1028. Anal. calcd for $C_{50}H_{56}N_4O_2S$: C, 77.28; H, 7.26; N, 7.21; S, 4.13. Found: C, 74.37; H, 7.06; N, 6.99; S, 4.02.

2.7. Metal ion titration

Each metal ion titration experiment was started with a 3.0 mL polymer in THF solution with a known concentration (1.0×10^{-5} mol L^{-1}). Mercury perchlorate salt and other various metal salts (nitrate, 3.0×10^{-3} mol L^{-1}, CH_3CN) were used for the titration. Polymer–metal complexes were produced by adding aliquots of a solution of the selected metal salt to a THF solution of the chiral polymer. All kinds of measurements were monitored 1 h after addition of the metal salt to the polymer solutions.

3. Results and discussion

3.1. Syntheses and feature of the chiral polymer

The chemical structures of the monomers **M-1**, ***R*-M-1**, the model compound ***R*-1** and the chiral polymer are shown in Scheme 1. 4,7-Diethynylbenzo[2,1,3]thiadiazole (**M-1**) could be synthesized from benzo[2,1,3]thiadiazole by a 3-step reaction according to reported literature and needed to be kept in the dark at −4 °C before using [31]. The chiral monomer (*R*)-6,6′-dibutyl-3,3′-diiodo-2,2′-bis-(diethylaminoethoxy)-1,1′-binaphthyl (***R*-M-1**) and the model compound (*R*)-6,6′-dibutyl-2,2′-bis(diethylaminoethoxy)-1,1′-binaphthyl (***R*-1**) can be obtained by a 8-step and 6-step reaction from the starting material (*R*)-BINOL, respectively [13,17,33]. In this paper, a typical Sonogashira reaction condition was applied to the synthesis of the chiral polymer [34,35]. M_w, M_n and PDI of the polymer were determined by gel permeation chromatography using polystyrene standards in THF and the values of they are 5560, 2630 and 2.15, respectively. The GPC result of the chiral polymer shows the moderate molecular weight. The chiral polymer is an air stable solid with gray color and shows good solubility in common organic solvents, such as toluene, THF, $CHCl_3$, and CH_2Cl_2, which can be attributed to the nonplanarity of the twisted polymer backbone and the flexible tertiary amino and *n*-butyl group substitutents.

3.2. The selective and sensitive recognition of the chiral polymer on Hg^{2+}

Based on the fluorescence spectra of the chiral polymer, we can find that the polymer exhibits orange fluorescence at 563 nm due to the extended π-electronic structure [18,35,36]. The effects of the

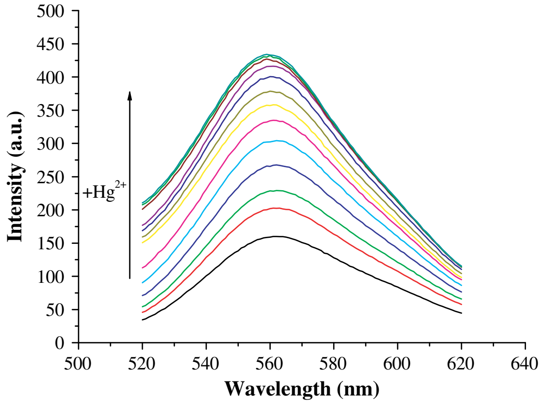

Fig. 1. Fluorescence spectra of the chiral polymer (1.0×10^{-5} mol L^{-1}) in THF with increasing amounts of Hg^{2+} (0, 0.2, 0.4, 0.6, 0.8, 1.0, 1.2, 1.4, 1.6, 1.8, 2.0, 3.0, 4.0×10^{-5} mol L^{-1}) ($\lambda_{ex} = 511$ nm).

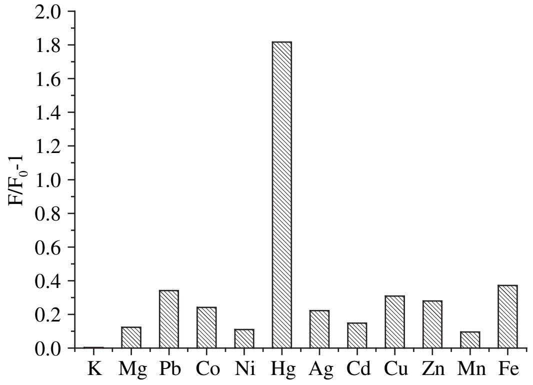

Fig. 3. Fluorescence enhancement degree of the polymer (1.0×10^{-5} mol L^{-1}) in the presence of various metal ions (each 2.0×10^{-5} mol L^{-1}).

fluorescence response behavior of the chiral polymer on Hg^{2+} have been investigated. Fig. 1 shows the fluorescence spectra of the polymer in THF solution (1.0×10^{-5} mol L^{-1}) upon the concentration molar ratio addition of Hg^{2+} (3.0×10^{-3} mol L^{-1}, CH_3CN) from 0.2 to 4. In regard to the fluorescence signal changes of the chiral polymer on Hg^{2+}, the fluorescence emission wavelengths of the Hg^{2+}–polymer complex do not show an obvious difference from the Hg^{2+}–free polymer. On the contrary, the fluorescence intensities of Hg^{2+} – containing polymer complex show gradual enhancement as high as 1.8-fold. It can also be found that the addition curve keeps nearly linear correlation with the concentration molar ratio of Hg^{2+} from 0.2 to 1.8 (Fig. 2). But the fluorescence intensity of Hg^{2+}–containing polymer complex appears no change upon the molar ratio addition of Hg^{2+} from 1:2 to 1:4. According to Fan and Jones's report, the sensing responses of the conjugated polymers incorporating different amino groups as receptors for Hg^{2+} can be attributed to the intramolecular photoinduced electron transfer (PET) between the conjugated polymer backbone and binding Hg^{2+} of different amino groups [5].

The effects of the fluorescence response behaviors of the monomer **M-1** and the model compound ***R*-1** on Hg^{2+} have been investigated in order to compare fluorescence changes of the polymer on the mercury detection with those of the corresponding small molecule sensors. The results indicate that Hg^{2+} causes a slight fluorescence quenching of **M-1**, which is contrary to that of the polymer (See Supporting Information, Fig. S1). In comparison with **M-1** and the polymer, ***R*-1** shows the more complicated fluorescence response behavior on Hg^{2+} (See Supporting Information, Fig. S2). Hg^{2+} causes the fluorescence quenching of ***R*-1** at a concentration from 0 to 1.0×10^{-5} mol L^{-1} and then a little fluorescence enhancement at a concentration range from 1.0×10^{-5} mol L^{-1} to 2.2×10^{-5} mol L^{-1}. Although **M-1**, ***R*-1** and the polymer show different fluorescence response behaviors on Hg^{2+}, it could be concluded that the resulting polymer exhibits the most sensitive detection than the two corresponding small molecules, which should be attributed to signal amplification from electronic communication within the conjugated polymer backbone.

The fluorescence response behaviors of the chiral polymer on other various metal ions were also examined under the same conditions as Hg^{2+} ion determination (Fig. 3). As a result, the fluorescence enhancement degree (F/F_0-1) of the chiral polymer is 0.37, 12.4, 34.2, 24.1, 11.0, 22.2, 14.8, 30.9, 28.0, 9.6 and 37.2% by K^+, Mg^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Ag^+, Cd^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+} and Fe^{3+} upon the 1:2 molar ratio addition of a metal salt solution, respectively. The results indicate the polymer exhibits excellent selectivity in sensing Hg^{2+}. For Hg^{2+} detection, one of the essential requirements is minor or no interference from other metal ions. The Hg^{2+} selective detection experiments of the chiral polymer in THF solution (1.0×10^{-5} mol L^{-1}) were conducted in the presence of the

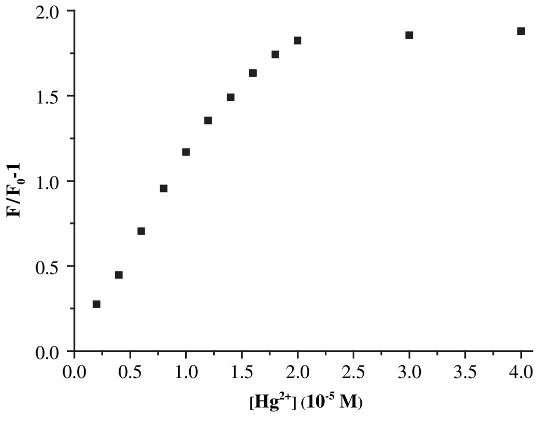

Fig. 2. Fluorescence enhancement values (F/F_0-1) of the chiral polymer *vs* the increasing concentration of Hg^{2+} from 1:0–1:4 molar ratios.

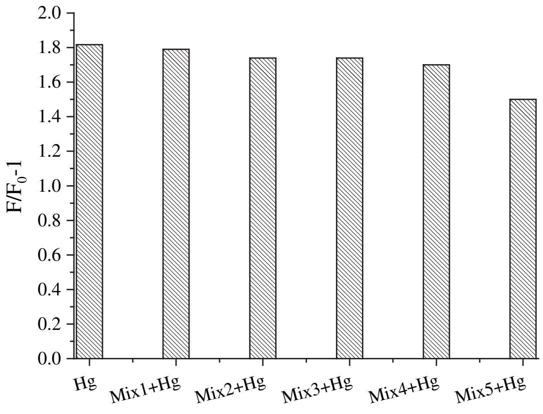

Fig. 4. Metal specificity: the concentration of **P-1** is 1.0×10^{-5} mol L^{-1} and Hg^{2+} is used at a concentration of 2.0×10^{-5} mol L^{-1}. Mix1: the mixture of Cu^{2+}, Ni^{2+} and Cd^{2+} (1.0×10^{-5} mol L^{-1}); Mix2: the mixture of Ag^{2+}, Zn^{2+} and Cu^{2+} (1.0×10^{-5} mol L^{-1}); Mix3, Mix4 and Mix5: the mixture of K^+, Mg^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Ag^+, Cd^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+} and Fe^{3+}, each 1.0, 2.0, 4.0×10^{-5} mol L^{-1}, respectively.

mixture of three chosen metal ions, such as Cu^{2+}, Ni^{2+}, Cd^{2+} or Ag^{+}, Zn^{2+}, Cu^{2+} (Fig. 4) at 1.0×10^{-5} mol L^{-1} in CH_3CN solution, respectively. Compared with that containing only Hg^{2+}, the fluorescence intensities of the chiral polymer do not appear obvious difference. Most importantly, while the fluorescence response was carried out by the Hg^{2+} titrations in presence of the mixed metal ions, such as the mixture of K^{+}, Mg^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Ag^{+}, Cd^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+} and Fe^{3+} at the concentration from 0 to 2.0×10^{-5} mol L^{-1}, no obvious interference was observed in the detection. But when the concentration of the mixed metal ions was added to a higher level, such as 4.0×10^{-5} mol L^{-1}, the coexisted ions showed interference in Hg^{2+} detection to some extent. The results indicate that the resulting chiral polymer can be used as Hg^{2+} selective probe which could hardly be affected by those coexisted ions in a particular range of the concentration.

4. Conclusion

A novel chiral polybinaphthyls chemosensor incorporating diethylamino and **BT** moieties as receptors was synthesized by Pd-catalyzed Sonogashira reaction. Compared with other cations, such as K^{+}, Mg^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Ag^{+}, Cd^{2+}, Cu^{2+}, Zn^{2+}, Mn^{2+} and Fe^{3+}, Hg^{2+} can produce pronounced fluorescence response of the polymer. This kind of chiral polymer exhibits excellent fluorescence response for Hg^{2+} detection without interference from other metal ions in a particular range of the concentration.

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Appendix. Supplementary data

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.polymer.2009.10.039.

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