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Fluorescent sensors for Ca^{2+} and Pb^{2+} based on binaphthyl derivatives

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ABSTRACT

Two novel binaphthyl compounds have been synthesized for the selective fluorescent recognition of Ca^{2+} or Pb^{2+}. By introducing different terminal groups to the receptor unit, the fluorescence signals of the receptors are significantly changed: **1** is fluorescence enhancement for Ca^{2+}, **2** is fluorescence quenching for Pb^{2+}. The binding properties for metal ions were examined by the absorption and fluorescence spectra. The fluorescence intensity enhancement was ascribed to the complex formation between Ca^{2+} and **1** which blocked the photo-induced electron transfer process.

1. Introduction

Calcium ion play an important role as a messenger in biological systems. Many physiological processes are triggered, regulated, or influenced by calcium ion [1,2]. Lead ion is the most toxic heavy metal ion causing adverse environmental and health problems. A wide variety of symptoms (which include memory loss, irritability, anemia, muscle paralysis, and mental retardation) have been attributed to lead poisoning, suggesting that Pb^{2+} affects multiple targets *in vivo* [3]. Therefore, it is important to explore new methods for analyzing Ca^{2+} and Pb^{2+} *in vitro* and *in vivo*. Because of the importance of ionic calcium and lead in biological processes, chemists have been interested in the design of chemosensors for the specific detection of them.

Design of chemosensors for the selective detection of a specific analyte is a topic of considerable interest, due to their wide ranging application in the broad areas of chemistry, biology and optics [4–11]. In optical sensors, the use of fluorescence as detecting method offers distinct advantages in terms of sensitivity, selectivity and response time. Fluorescent molecular sensors have attracted considerable interest because of their intrinsic sensitivity and selectivity [4,6,7]. They are also called fluoroionophores because they consist in a recognition moiety (ionophore) linked to a transducting moiety (fluorophore). The choice of the fluorophore is of major importance because it governs the recognition event into an optical signal owing to the change of its photophysical characteristics due to the perturbation by the bound cation of various photoinduced processes (electron transfer, energy transfer, charge transfer). The recognition moiety is responsible for the efficiency and selectivity of binding. Among ionophores for metal ions, binaphthyl-based ligands offer numerous advantages because of the rigidity of the complexing unit and tuneable strategies to obtain molecules with appropriate substituents. Although the beginning of the development can be traced back to the enantioselective quenching of binaphthyl-based fluorescence first reported about 30 years ago [12]. Only recently binaphthyl-based fluorescent sensors have been used for the monitoring of many chemical species [13–24].

In the early reports some fluorescent molecular sensors have been designed for Ca^{2+} or Pb^{2+}, which include derivatives of crown ether [25–28], calixarene [29–32], anthracene [33–35], squaraine [36–39] and so on. Despite having many sensors, chemists continue endeavoring to design new ones and to improve their sensitivity, selectivity and reliability in order to satisfy various needs. In connection with our developments of fluorescent sensors for metal ions [40,41], and studies about binaphthyl-based ligands for luminescent lanthanide complexes [42–44], we designed and synthesized two binaphthyl-based compounds in a novel but simple approach as fluorescent sensors for Ca^{2+} and Pb^{2+}, which show distinctly different optical properties. To best of our knowledge, much less attention has been paid to binaphthyl-based compounds as fluorescent sensors for meal ions. And to our surprise, only having different terminal groups, sensor **1** show selectivity and response toward Ca^{2+} with fluorescence enhancement, but sensor **2** is fluorescence quenching for Pb^{2+}.

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2. Experimental

2.1. Materials

All commercially available chemicals were of A.R. grade and all solvents used were purified by standard methods.

2.2. Methods

IR-spectra were measured on Nicolet Nexus 670 FT-IR using KBr pellets in the range of 400–4000 cm^{-1}. The ^1H NMR spectra were recorded on a Varian Mercury plus 300BB spectrometer in $CDCl_3$ or d_6-DMSO solution with TMS as internal standard. Fluorescence measurements were performed on a Hitachi F-4500 spectrophotometer equipped with quartz curettes of 1 cm path length. The excitation and emission slit widths were 5.0 nm. Absorption spectra were made on a Shimadzu UV-240 spectrophotometer. HRMS were determined on a Bruker Daltonics APEXII 47e FT-ICR spectrometer.

2.3. Synthesis: compounds *1* and *2*

Compounds **1** and **2** were readily prepared according to Scheme 1. We introduced two different terminal groups into the 1, 1′-binaphthyl framework to construct the fluorescent receptors. All of the compounds were characterized by ^1H NMR, ^{13}C NMR, IR and HRMS. They were all obtained in high yield and were soluble in common organic solvents such as $CHCl_3$, MeOH and DMSO.

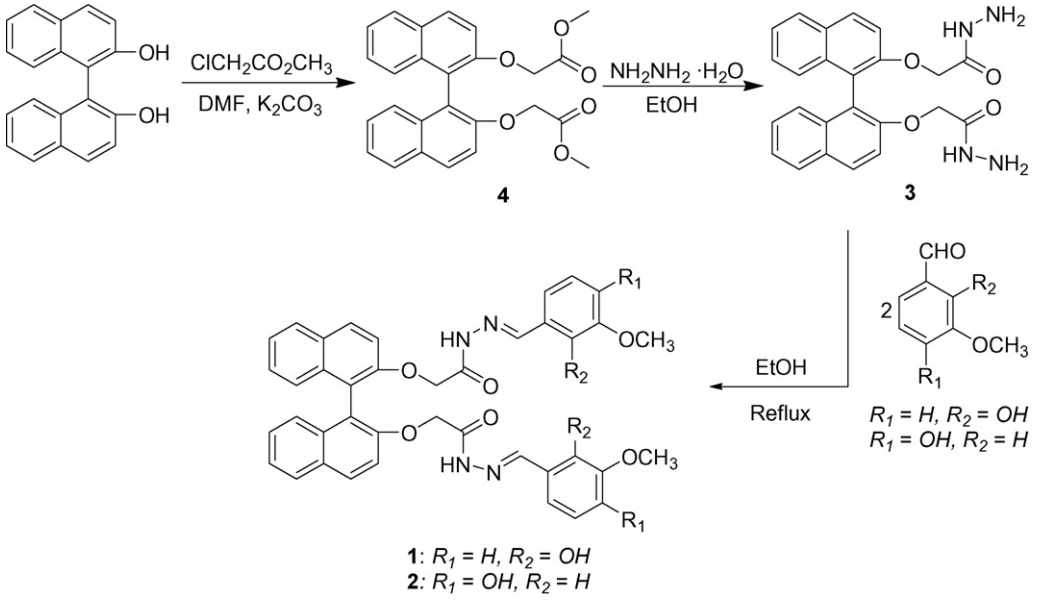
2.4. Syntheses: compounds *1*–*4*

Anhydrous K_2CO_3 (9.5 g, 70.98 mmol) was added into the 10 mL DMF solution of 1, 1′-bi-2-naphthol (5 g, 17.48 mmol) at 80 °C. After two hours, a solution of methyl chloroacetate (4 g, 36.89 mmol) in 10 mL DMF was added dropwise to the mixture and maintained at 100 °C for 4 h. When cooled, 60 ml distilled water was poured and the turbid solution was extracted by 40 mL chloroform three times. Organic phase combined was washed with water and dried with anhydrous Na_2SO_4. Solvent removed, the crude residue was purified by flash column chromatography on silica gel (Petroleum ether/AcOEt = 5:1 to 3:1) to afford the compound **4**; Yield: 65%. ^1H NMR (300 MHz, $CDCl_3$): δ 7.96 (d, J = 9.0 Hz, 2H), 7.88 (d, J = 7.8 Hz, 2H), 7.39–7.34 (m, 4H), 7.25 (d, J = 8.7 Hz, 2H), 7.20 (d, J = 8.7 Hz, 2H), 4.57 (s, 2H), 4.56 (s, 2H), 3.64 (s, 3H), 3.64 (s, 3H) ppm. IR (KBr film): ν_{max} 2953, 1755, 1621, 1592, 1508, 1434, 1377, 1333, 1288, 1213, 1149, 1107, 1091, 1001, 819, 749 cm^{-1}. The hydrazine hydrate (1.5 g, 1.4 mL) was added to **4** (5 g, 11.63 mmol) in dry ethanol (10 mL) at 80 °C. Then the mixture was allowed to reflux for 4 h to afford 3 as a white solid; Yield: 91%. ^1H NMR (300 MHz, d_6-DMSO): δ 8.73 (s, 2H, –NH–), 8.05 (d, J = 9.0 Hz, 2H), 7.94 (d, J = 8.1 Hz, 2H), 7.51 (d, J = 8.7 Hz, 2H), 7.34 (t, J = 7.2 Hz, 2H), 7.22 (t, J = 8.4 Hz, 2H), 6.91 (d, J = 8.7 Hz, 2H), 4.58 (d, J = 14.7 Hz, 2H), 4.44 (d, J = 14.7 Hz, 2H), 4.24 (brs, 4H, $-NH_2$) ppm. IR (KBr film): ν_{max} 3428, 3274, 3051, 2921, 1661, 1622, 1594, 1532, 1509, 1264, 1219, 1147, 1086, 985, 802, 737 cm^{-1}.

Compounds **1** and **2** were prepared according to the literature [45].

Compound **1**: yield: 92%. ^1H NMR (300 MHz, d_6-DMSO): δ 11.57 (s, 1H, –N=CH–), 11.48 (s, 1H, –N=CH–), 11.29 (s, 1H, –N=C–OH), 10.89 (s, 1H, –N=C–OH), 10.69 (s, 1H, –NH–), 10.59 (s, 1H, –NH–), 9.36 (s, 2H, –OH), 8.33–8.27 (m, 2H), 8.15–8.01 (m, 2H), 7.99–7.93 (m, 2H), 7.60 (d, J = 8.1 Hz, 1H), 7.54–7.46 (m, 1H), 7.40–7.20 (m, 4H), 7.14–6.95 (m, 4H), 6.85–6.74 (m, 2H), 5.27–5.08 (m, 2H), 4.82–4.62 (m, 2H), 3.80 (s, 6H) ppm. ^{13}C NMR (75 MHz, d_6-DMSO): δ 169.4, 164.5, 164.3, 153.9, 153.6, 148.2, 148.0, 147.1 (2C), 146.0, 141.4, 133.5, 133.4, 129.6, 129.4, 128.9, 128.1, 126.6, 125.2, 124.9, 124.7, 124.1, 123.6, 120.7, 120.5, 120.1, 119.2, 119.1, 118.8, 118.7, 118.0, 117.8, 116.4, 115.7, 113.9, 112.9, 68.4, 67.6, 56.2, 55.8 ppm. IR (KBr film): ν_{max} 3319, 3217, 3056, 2932, 2836, 1691, 1612, 1584, 1530, 1508, 1465, 1362, 1327, 1255, 1219, 1149, 1079, 946, 809, 778, 733 cm^{-1}. HRMS (ESI) m/z obsd 699.2443($[M+H]^+$, calcd 699.2449 for $C_{40}H_{35}N_4O_8$).

Compound **2**: yield: 90%. ^1H NMR (300 MHz, d_6-DMSO): δ 11.41 (s, 1H, –N=CH–), 11.29 (s, 1H, –N=CH–), 10.78 (s, 1H, –NH–), 10.18 (s, 1H, –NH–), 9.54–9.44 (m, 2H, –OH), 8.07–8.06 (m, 1H), 8.01–7.89 (m, 2H), 7.84–7.81 (m, 1H), 7.57–7.49 (m, 2H), 7.44–7.08 (m, 6H), 7.06–6.95 (m, 4H), 6.80–6.73 (m, 2H), 5.27–5.00 (m, 2H), 4.76–4.55 (m, 2H), 3.78 (s, 3H), 3.75 (s, 3H) ppm. ^{13}C NMR (75 MHz, d_6-DMSO): δ 170.0, 169.7, 164.7, 164.5, 154.8, 154.5, 149.8, 149.5, 149.4, 149.0, 145.1 (2C), 134.0 (2C), 130.3, 130.0, 129.9, 129.6, 129.4, 128.7, 127.3, 126.1, 125.8, 125.4, 125.2, 124.6, 124.2, 123.0, 122.2, 120.4, 119.8, 118.5, 116.5, 116.1, 110.0, 109.5, 68.8, 68.2, 56.2, 56.1 ppm. IR



Scheme 1. The synthesis of receptors **1** and **2**.

(KBr film): ν_{max} 3412, 3318, 3054, 2928, 1671, 1593, 1512, 1462, 1430, 1383, 1272, 1210, 1084, 814, 749 cm^{-1}. HRMS (ESI) *m/z* obsd 699.2441([M+H]$^+$, calcd 699.2449 for $C_{40}H_{35}N_4O_8$).

2.5. Binding studies

The studies on the binding properties of **1** and **2** were carried out in methanol. The perchlorate salts solution (1.0×10^{-2} M) were prepared by dissolving the desired amount of perchlorate salts in dry methanol. Association constants were calculated by means of a linear curve fitting with Origin 7.0 (Origin-Lab Corporation).

3. Results and discussion

3.1. Complexation studies with Ca^{2+} and Pb^{2+}

Fig. 1 shows the fluorescence titration of **1** with Ca^{2+} in methanol. Addition of a 20-fold Ca^{2+} results in a 3 times enhancement of fluorescence intensity with respect to the metal ion-free state. The free acceptor **1** in the methanol possessed a fluorescence peak at 445 nm with over 70 nm red shifts of 1, 1′-bi-2-naphthol, at 372 nm. This indicates an efficient intramolecular energy transfer

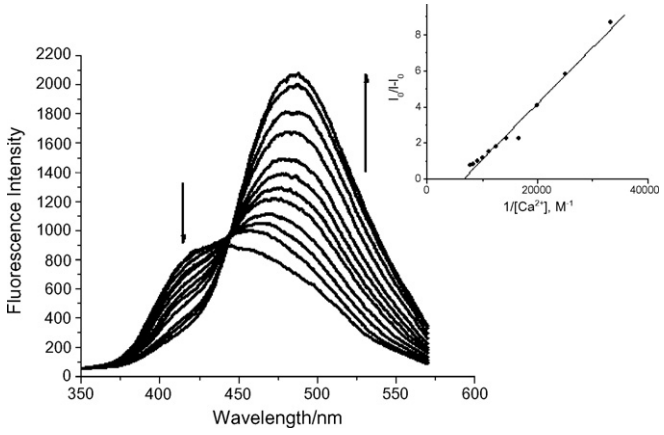


Fig. 1. Fluorescence titration of **1** (1.0×10^{-5} M) in the presence of different concentrations of Ca^{2+} in CH_3OH, λ_{ex} = 297 nm. Inset shows Benesi–Hilderbrand plot of **1** with Ca^{2+} at 488 nm.

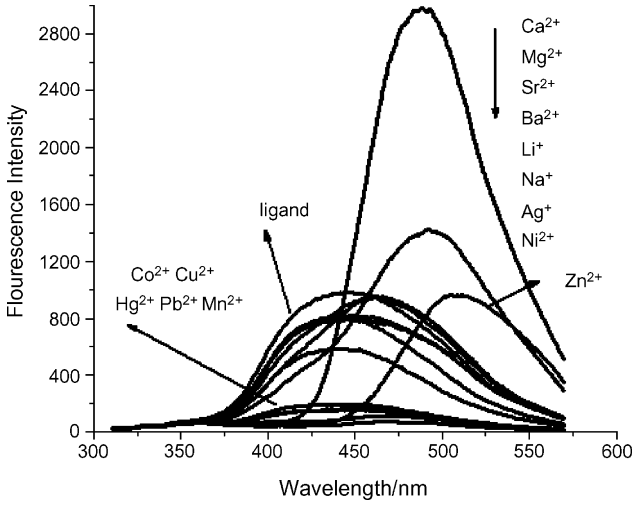


Fig. 2. Fluorescence spectrum of **1** (1.0×10^{-5} M) in methanol in the presence of several metal ions ([M] = 1.0×10^{-5} M (Co, Cu, Ni, Ag, Hg, Pb, Mn) ([M] = 5.0×10^{-5} M (Li, Na, Mg, Ca, Sr, Ba, Zn)). λ_{ex} = 297 nm.

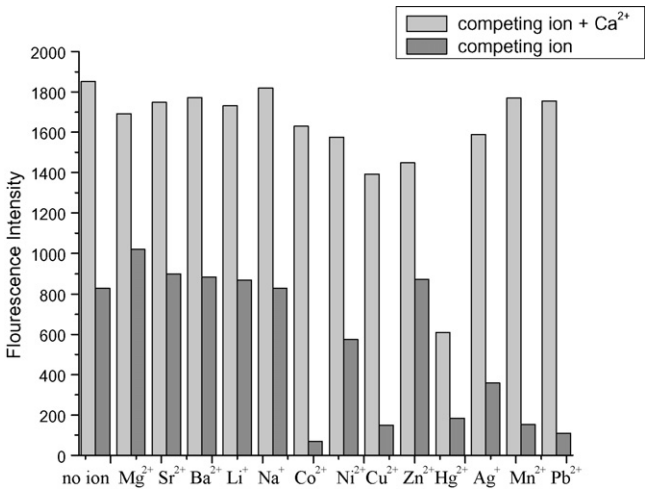


Fig. 3. The fluorescence intensity change profile of **1** (1.0×10^{-5} M in methanol) in the presence of selected metal ions. Excitation is at 297 nm, and emission is monitored at 488 nm.

from the phenyl rings to the naphthyl core [15,21]. Addition of calcium ions to a methanol solution of **1** led to a bathochromic shift of the main fluorescence band to 488 nm, which exhibits 43 nm red-shift. Further addition of Ca^{2+} led to an increase in intensity of the 488 nm band and a decrease in the intensity of the 445 nm peak. The change was marked by the presence of an isosbestic point at 448 nm. The large bathochromic shift for **1**-Ca^{2+} can be attributed to the strong stabilization of internal charge-transfer (ICT) state by the complexation of calcium with the hydroxy and the nitrogen atom of –N=CH– groups via chelation.

To obtain an excellent chemosensor, high selectivity is a matter of necessity. In the present work, studies of selective coordination of cations of **1** by means of fluorescence spectroscopy were then extended to related main group, transition and heavy metal ions (Li^+, Na^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+}, Hg^{2+} and Ag^+). Only the addition of Ca^{2+} resulted in a prominent fluorescent enhancement in fluorescence (Fig. 2). The selectivity of **1** for Ca^{2+} over other metal ions was investigated by the competition experiments. Fig. 3 shows the fluorescence intensity of **1** in the presence of selected metal ions. It is obvious that **1** has a

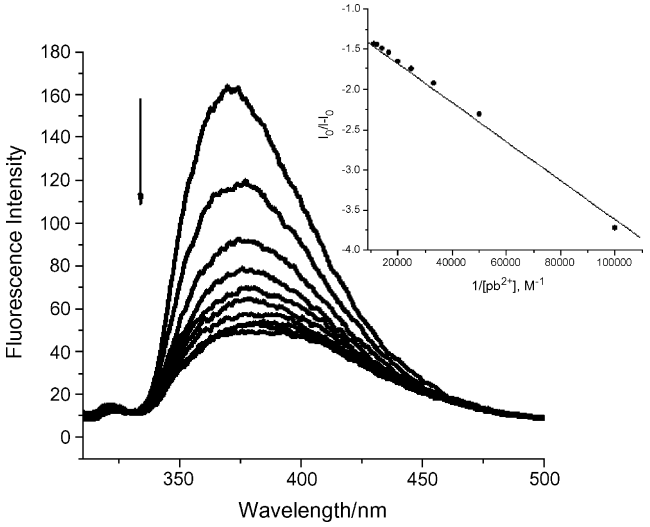


Fig. 4. Fluorescence titration of **2** (2.0×10^{-5} M) in the presence of different concentrations of Pb^{2+} in CH_3OH, λ_{ex} = 291 nm. Inset shows Benesi–Hilderbrand plot of **2** with Pb^{2+} at 371 nm.

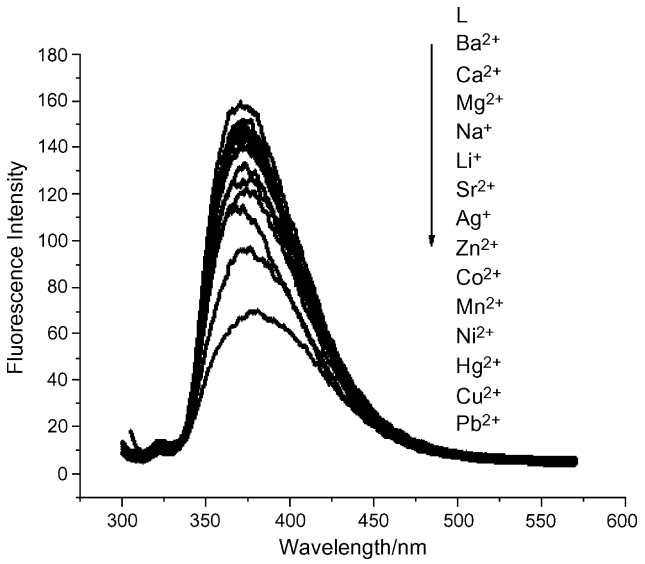


Fig. 5. Fluorescence spectrum of **2** (2.0×10^{-5} M) in methanol in the presence of several metal ions ([M] = 4.0×10^{-5} M). λ_{ex} = 291 nm.

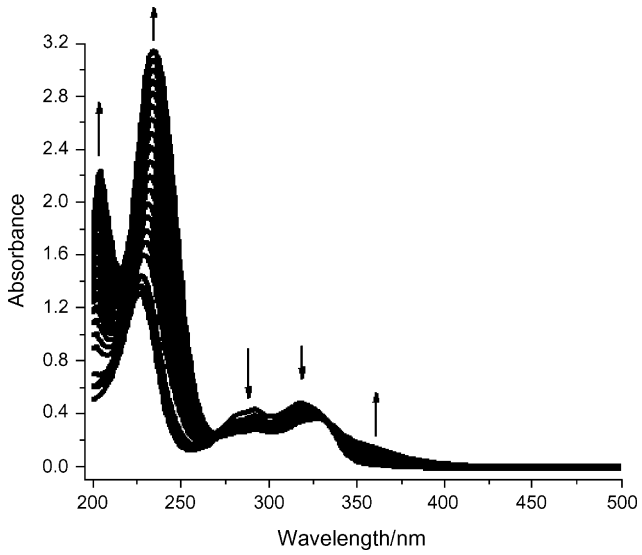


Fig. 6. UV–vis titration of **2** (1.0×10^{-5} M) in the presence of different concentrations of Pb^{2+} in CH_3OH.

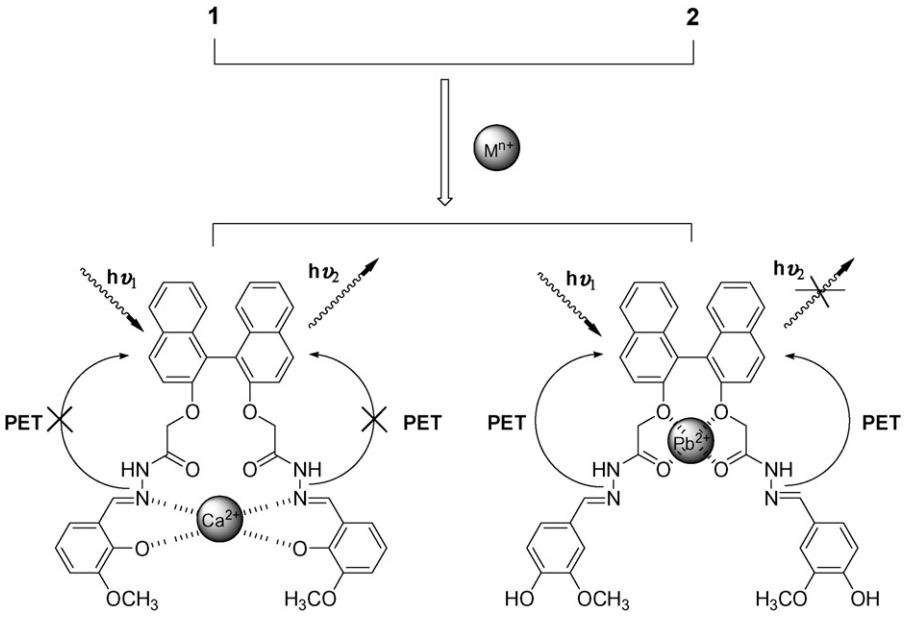
highly selective response to Ca^{2+}. On the basis of the photospectroscopic data, it seems that Li^+, Na^+, Ag^+, Sr^{2+}, Ba^{2+}, Ni^{2+} and Zn^{2+} behave like Mg^{2+}, whereas Mn^{2+}, Co^{2+}, Cu^{2+}, Hg^{2+} and Pb^{2+} act differently. The fluorescence spectra of **1**, with Li^+, Na^+, Ag^+, Sr^{2+}, Ba^{2+}, Ni^{2+}, Zn^{2+} and Mg^{2+} in methanol, cause emission increases along with red-shift. But no red-shift phenomenon are observed in the one of Mn^{2+}, Co^{2+}, Cu^{2+}, Hg^{2+} and Pb^{2+}, which probably shows that these metal ions bound in the same binding site with different affinities.

There was almost no change observed on the UV–vis spectra of receptor **1** upon addition of metal ions, the interaction between host and guest was only evaluated by fluorescent spectra.

The fluorescence titration of **2** with Pb^{2+} was carried out in methanol, which is shown in Fig. 4. Gradually increasing the concentration of Pb^{2+} caused the fluorescence emission intensities of **2** (2.0×10^{-5} M) at 371 nm to decrease remarkably. The fluorescence intensity was quenched more than 75% while the concentration of Pb^{2+} reached that 10 equivalents of **2**. The emission wavelength upon Pb^{2+} binding did not exhibit any detectable shift, which means that binding site between **2** and lead ion is different from **1** with Ca^{2+}.

We also study of selective coordination of cations of **2**. Addition of Li^+, Na^+, Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Hg^{2+} and Ag^+ led to slightly changes in the fluorescence of **2**. Only the addition of Pb^{2+} resulted in a prominent fluorescent quenching in fluorescence (Fig. 5).

The UV–vis spectrum of **2** show that the signals at the short wavelengths (<300 nm) increased significantly with Pb^{2+}, but much smaller changes were observed at the long wavelengths (Fig. 6). The absorption maximum wavelengths for **2**-Pb^{2+} are 234 nm. So the interaction between host and guest was only evaluated by fluorescent spectra.



Scheme 2. Proposed binding model of **1** with Ca^{2+} and **2** with Pb^{2+}.

The Job plot is extensively used to find the complexation mode in the host-guest interactions. The plots $I_0/(I-I_0)$ against $1/[M]$ according to the Benesi-Hildebrand equation [46,47]:

$$\frac{I_0}{I-I_0} = \frac{b}{a-b}\left\{\frac{1}{K[M]}+1\right\}$$

where I_0 is the fluorescence intensity of the sensor in the absence of the metal ion, I is the fluorescence intensity in the presence of the metal ion, $[M]$ is the concentration of the metal ion, and K is the association constant between the sensor and the metal ion. In the equation, a and b are constants. By the linear curve fitting results of the fluorescence intensity (**1** at 488 nm and **2** at 371 nm) of the interaction between **1** and Ca^{2+}, **2** and Pb^{2+}, the association constant for **1**-Ca^{2+} was estimated to be $3.9 \times 10^3\ M^{-1}$ and for **2**-Pb^{2+} was $5.0 \times 10^4\ M^{-1}$. The correlation coefficients of the non-linear curve fitting were all large ($R > 0.99$), which indicated that the 1:1 complex between **1** and Ca^{2+}, **2** and Pb^{2+} have been formed [14].

3.2. Binding model of the complexes

The photophysical properties revealed that 1:1 complexes were formed between **1** and **2** with metal ions. But the two sensors should form two different structure with metal ions, giving different fluorescence response. For the two sensors, their nitrogen lone pair electrons are positioned to quench the fluorescence of the binaphthyl units by an intramolecular photoinduced-electron-transfer (PET) process. In the presence of Ca^{2+}, the fluorescence enhancement of receptor **1** most likely arose from the calcium ion complex (**1**-Ca^{2+}) formation, in which the calcium ion bond with nitrogen atom of **1**, blocking the PET process. In contrast, the fluorecence quenching of receptor **2** most likely resulted from the lead ion complex (**2**-Pb^{2+}) formation, in which the lead ion is not bonding with nitrogen atom of **2**, increasing the electron transfer from receptor to the excited binaphthyl units, which in turn led to a more facile intramolecular PET process (Scheme 2).

4. Conclusion

We have designed and synthesized two binaphthyl-based fluorescent sensors for the selective recognition of Ca^{2+} or Pb^{2+}. We have demonstrated that by introducing different terminal groups to the receptor unit, because of the different structure of the complexes made the fluorescence signals of the receptors significantly different: **1** is fluorescence enhancement for Ca^{2+}, **2** is fluorescence quenching for Pb^{2+}. Using this strategy, the selective sensors will allow us to quickly identify the two metal ions.

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