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Colorimetric and highly selective "turn-on" fluorescent anion chemosensors with excited state proton transfer

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Abstract

2-(3-Hydroxy-naphthalen-2-yl)-benzo[*d*][1,3]oxazin-4-one (HNBO), 3-hydroxy-naphthalene-2-carboxylic acid (2-heptylcarbamoyl-phenyl)-amide (HNAHPA) and 2-[2-(2-hydroxy-phenyl)-benzooxazol-6-ylmethylene]-malononitrile (HBODC) are shown to be fluorescent and colorimetric anion chemosensors with high selectivity for F^- over Cl^-, in the low analyte concentration range of 10^{-6} to 10^{-4} M.

Keywords: Colorimetric anionsenor; Excited state proton transfer; Binding constant

1. Introduction

Anions play an important role in a wide range of chemical and biological processes, and considerable research attention has been focused on the design of host molecules that can selectively recognize and sense anion species through visible, electrochemical and optical responses [1,2]. Among the biologically important anions, fluoride is of particular interest owing to its established role in preventing dental caries [3]. The use of fluoride anions in the treatment of osteoporosis [4] is also being explored extensively, yet can lead to fluorosis [5], a type of fluoride toxicity that generally manifests itself clinically as increases in bone density.

This diversity of functions, both beneficial and otherwise, means that the problem of fluoride anion detection is of considerable current interest.

Colorimetric anion sensors have attracted much attention because they enable the low cost detection of analytes with the naked eye, without resorting to the use of expensive instruments [6,7]. On the other hand, the most desirable feature of an anion sensor based on fluorescence is a highly selective and sensitive response to applied perturbations consisting of a dramatic change in emission color and intensity. For this purpose, many fluorescent anion sensors have been developed on the basis of a variety of signaling mechanisms, such as competitive binding [8], photoinduced electron transfer (PET) [9], metal to ligand charge transfer (MLCT) [10], excimer/exciplex [11], intra-molecular charge transfer (ICT) [12] and excited state proton transfer (ESPT) [13].

It is surprising that, although the phenomenon of inter- and intra-molecular excited state proton transfer has been well documented [14], it has only rarely been exploited in anion sensing. Moreover, simple and easily synthesized electroneutral fluorescent and colorimetric anion sensors remain rare [15]. Presently, there is a strong demand for anion sensors with improved specificity, in particular in their selectivity for F^-, AcO^- and $H_2PO_4^-$ in the presence of Cl^-. In this paper, we have synthesized and investigated the potential use of the simple, neutral and easy to prepare ESPT compounds, 2-(3-hydroxy-naphthalen-2-yl)-benzo[*d*][1,3]oxazin-4-one (HNBO), 3-hydroxy-naphthalene-2-carboxylic acid (2-heptylcarbamoyl-phenyl)-amide (HNAHPA) and 2-[2-(2-hydroxy-phenyl)-benzooxazol-6-ylmethylene]-malononitrile (HBODC) [14b], as the colorimetric and/or fluorescent probes for fluoride ions with high fluoride/chloride selectivity. Among these molecules, best performance was obtained with HNAHPA in this work whose association constants (K_a) for the complexation with F^- and Cl^- were 7.58×10^5 and 8.17×10^3 M^{-1}, respectively, giving rise to the K_aF/K_aCl value of 93 (vide infra). It is also noteworthy that the association constant of HNAHPA with F^- (K_aF^-) is about nine times higher than that of the most recently reported best performance anion chemosensor

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based on ESPT, 2,5-bis(2-hydroxyphenyl)-1,3,4-oxadiazole, (K_aF/K_aCl = 86) [13c]. These results clearly demonstrate that HNAHPA exhibits significantly improved sensitivity as well as the better selectivity compared to those of previously reported materials [13]. Molecular structural aspects responsible for the enhanced anion sensing performance are discussed in this work.

2. Experimental

2.1. Synthesis

2.1.1. 2-(3-Hydroxy-naphthalen-2-yl)-benzo[d][1,3]oxazin-4-one (HNBO)

7.3 g of Anthranilic acid (0.053 mol) and 10.0 g of 3-hydroxy 2-naphthoic acid (0.053 mol) were dissolved in 70 mL of pyridine and stirred for 30 min. Triphenyl phosphite (0.053 mol) 14 mL was added to this solution and subsequently heated at 100 °C for 4 h, and then the reaction mixture was poured into cold water and extracted with dichloromethane. The solution was dried with anhydrous magnesium sulfate. After removal of solvent, the crude product was purified by column chromatography on silica gel with ethyl acetate/*n*-hexane (volume ratio 1/3). The product was recrystallized from ethanol to give 6.0 g of pure HNBO (yield 39%). ^{1}H NMR ($CDCl_3$, 300 MHz) 12.05 (s, 1H), 8.73 (s, 1H), 8.29 (dd, 1H), 7.87 (t, 2H), 7.69 (t, 2H), 7.58 (m, 2H), 7.51 (d, 1H), 7.34 (t, 1H). *m/z* (MS-EI) calcd for $C_{18}H_{11}NO_3$, 289.28, found 289. Element analysis calcd for $C_{18}H_{11}NO_3$: C 74.73, H 3.83, N 4.84 and O 16.59 found C 74.76, H 3.88, N 4.80, O 16.61.

2.1.2. 3-Hydroxy-naphthalene-2-carboxylic acid (2-hepthylcarbamoyl-phenyl)-amide (HNAHPA)

One gram (3.46 mmol) of HNBO and 1.98 g of heptyl amine (16.5 mmol) were dissolved in 30 mL of pyridine. The solution was heated at reflux for 24 h. The reacted mixture was poured into cold water and neutralized with 1N HCl solution. The precipitate was collected by filtration. The crude product was purified by column chromatography on silica gel with ethyl acetate/*n*-hexane (volume ratio 1/10) and then recrystallized from ethanol to give 0.55 g of pure product (yield 40%). ^{1}H NMR ($CDCl_3$, 300 MHz), δ 12.67 (s, 1H), 11.81 (s, 1H), 8.68 (d, 1H), 8.38 (s, 1H), 7.95 (d, 1H), 7.70 (d, 1H), 7.55 (m, 3H), 7.36 (t, 2H), 7.21 (t, 1H), 6.28 (s, 1H), 3.51 (q, 2H), 1.65 (t, 2H), 1.37 (m, 4H), 1.28 (m, 4H) 0.86 (m, 3H). *m/z* (MS-EI) calcd for $C_{25}H_{28}N_2O_3$, 404.21, found 404. IR (KBr pellet, cm^{-1}) 3438 (ν_{OH}), 3307 (ν_{NH}), 1590 (νc_{CO}). Element analysis calcd for $C_{25}H_{28}N_2O_3$: C 74.23, H 6.98, N 6.93, O 11.87, found C 74.0, H 7.01, N 6.95, O 11.75.

2.1.3. 2-[2-(2-Hydroxy-phenyl)-benzooxazol-6-ylmethylene]-malononitrile (HBODC)

HBODC was prepared using a Knoevenagel condensation of the 6-carbaldehyde derivative of 2-(2′-hydroxyphenyl) benzoxazole (HBO) and malononitrile in the presence of piperidine according to the method reported earlier [14b]. The product was purified by silica gel column chromatography with eluent of dichloromethane/*n*-hexane (3/1) to afford HBODC (yield 53%). ^{1}H NMR ($CDCl_3$, ppm) 11.11 (s, 1H), 8.37 (d, J = 0.7 Hz, 1H), 8.06 (dd, J = 8.0, 1.4 Hz, 1H), 7.81–7.88 (m, 3H), 7.53 (t, J = 7.8 Hz, 1H), 7.16 (dd, J = 8.4, 0.7 Hz, 1H), 7.07 (t, J = 7.6 Hz, 1H); *m/z* (MS-EI) calcd for $C_{17}H_9N_3O_2$, 287.07, found 287. Anal. calcd for $C_{17}H_9N_3O_2$: C 71.08, H 3.16, N 14.63, found C 71.07, H 3.22, N 14.42.

3. Measurements

^{1}H NMR spectra were recorded on a JEOL JNM-LA300 (300 MHz) spectrometer in $CDCl_3$ solution. Molar masses of compounds were measured with JEOL, JMS-AX505WA by electron impart (EI) mode. Element contents of compounds were measured with EA1110 (CE Instrument, Italy). UV–vis absorption and fluorescence spectra were recorded using Shimadzu UV-1650PC and Shimadzu RF-500 spectrofluorimeter, respectively, with emission and excitation slit width of 3 nm each.

4. Results and discussion

In Fig. 1, schematic representations of the proposed anion binding and sensing modes of three new ESPT molecules are shown. Anion binding is supposed to stabilize the charge transfer (CT) excited state because of the resulting electrostatic interactions. The proton that becomes more acidic as a result of the localized negative charge on the bound F^- is to be transferred to the anion and the second emission channel is opened.

The ability of HNBO, HNAHPA and HBODC to coordinate with F^-, AcO^-, $H_2PO_4^-$ and Cl^- as tetrabutylammonium salts was investigated using UV–vis absorption and fluorescence emission methods. The latter method was used to provide K_a values, and was complemented by the use of molar ratio analysis (see Supplementary material Figures S1d, S2f, S3f and Table S1); data consistent with the proposed 1:1 binding stoichiometry was found for most of the HNBO and HNAHPA cases except the 1:2 stoichiometry of HNAHPA with $H_2PO_4^-$. On the other hand, both 1:1 and 1:2 ratios were found for the interaction of HBODC with F^- and Cl^- ions, respectively. As summarized in Table 1, all our compounds clearly functioned as anion receptors. Remarkable changes in visual as well as the fluorescence emission colors were effected upon complexation with F^- ions in polar aprotic solvents such as dichloromethane (DCM) and dimethylsulfoxide (DMSO) as shown in Fig. 2. It must also be noted that these changes are virtually "turn-on" process with intensity ratios as high as 64, ~1120 and 41 times higher upon complexation with F^- ions for sensors HNBO, HNAHPA and HBODC, respectively. In fact, the solutions in all the systems change visibly to the naked eye from colorless to yellow upon the addition of fluoride ions, while colorless to cyan (HNBO) and green (HNAHPA and HBODC) when illuminated under 365 nm excitation as illustrated in Fig. 2. Interestingly, in both DCM and DMSO these changes are reversed upon the addition of methanol/water. Presumably, this is because water competes for F^- as a result of its hydrogen

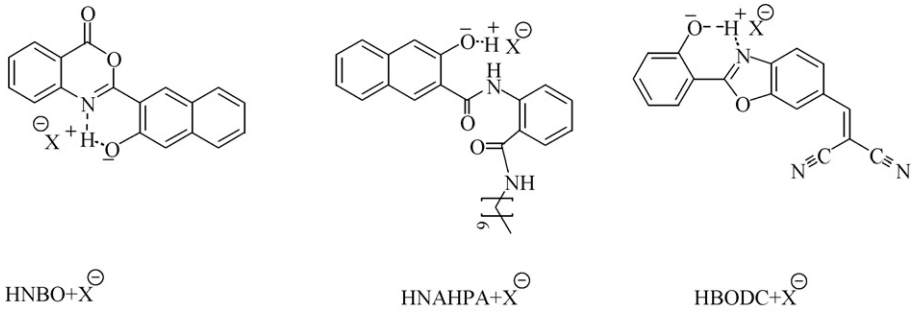

Fig. 1. Structures of the complexes of the anion sensors HNBO, HNAHPA and HBODC with X = (F^-, AcO^-, $H_2PO_4^-$ and Cl^-) ions.

bonding interactions with the phenolic OH hydrogen bond donating site. As a test of the proposed model of binding/sensing scheme shown in Fig. 1, the interaction of various anions with the *O*-methyl derivatives, of HNAHPA and HBODC namely 3-methoxy-naphthalene-2-carboxylic acid (2-hepylcarbamoyl-phenyl)-amide (MNAHPA) and 2-[2-(2-methoxy-phenyl)-benzooxazol-6-ylmethylene]-malononitrile (MBODC) were investigated. It was found that there is no change in color in these *O*-methyl derivatives, and also that there are no discernible changes in either their absorption or emission spectra, in the presence of F^-, AcO^-, $H_2PO_4^-$ or Cl^-, even in the presence of a large excess. It is therefore obvious that the anions bind to ESPT molecules through hydrogen bonding interactions via the phenolic OH groups of HNBO or HBODC, whereas cooperative hydrogen bonding interactions caused by both phenolic OH and anilide NH are likely to play an important role in HNAHPA. These interactions lead to an increase in the local concentration of the anion, and as a consequence, inter-molecular proton transfer in the excited state to the weakly basic anions occurs [13] with the formation of contact or solvent-separated ion pairs in polar aprotic solvents due to the enhanced acidity of the aromatic hydroxyl proton [14]; this conclusion is further supported by the similar spectral variations of the sensors in the presence of slightly basic (5×10^{-2} to 5×10^{-3} M) OH^- in methanol (see Supplementary material Figure S4).

The anions AcO^-, $H_2PO_4^-$ and Cl^- were found to induce similar variations in both the absorption and fluorescence spectra to the extent that depends on the anion's basicity; the higher the basicity the stronger the complex that results, which in turn brings about stronger color changes [16]. The trend in the selectivity of the anion-induced color changes was found to be $F^- > AcO^- > H_2PO_4^- > Cl^-$ as shown in Fig. 2.

Our ESPT molecules show higher binding affinity to and more efficient fluorescence enhancement with F^- than with other anions because of the higher charge density and smaller size of F^- anon, enabling stronger interaction with phenol or amide derivatives containing only a single hydrogen bonding donor group [13,17]. HNAHPA shows higher binding affinity and more efficient fluorescence enhancement with guest anions than HNBO and HBODC because the former binds anion in the ground state through hydrogen bonding interaction via both anilide NH and phenolic OH whereas the latter contains only a single phenolic OH group. In view of the acidity of protons deduced from the NMR chemical shift, the higher selectivity of HNAHPA is rationalized by the presence of more acidic phenolic OH ($\delta = 12.67$ ppm) in HNAHPA than HNBO ($\delta = 12.05$ ppm) and HBODC ($\delta = 11.11$ ppm) as well as the presence of additional anilide NH ($\delta = 11.81$ ppm). Indeed, HNAHPA has an affinity constant (K_a) with respect to F^- in dichloromethane ($\sim 7.58 \times 10^5\ M^{-1}$) that is an order of magnitude higher than those of HBODC ($K_a = 7.11 \times 10^4\ M^{-1}$) and HNBO ($K_a = 2.52 \times 10^4\ M^{-1}$). HNAHPA also exhibits remarkable selectivity for fluoride anions ($K_aF/K_aCl > 93$; $K_aAcO^-/K_aCl^- > 65$; $K_aH_2PO_4^-/K_aCl^- > 35$), as shown in Table 1. As for the HBODC, however, 2×10^{-5} M solution of it in DCM (see Supplementary material Figure S3) with ~0–10 equivalents of F^- and Cl^- ions formed both 1:1 and 1:2 complex with nearly equal association constant of $\sim 7.11 \times 10^4 / 7.06 \times 10^4\ M^{-1}$ and $\sim 1.41 \times 10^4 / 1.34 \times 10^4\ M^{-2}$, respectively. This causes much

Table 1
Association constants (K_a, M^{-1}) of the complexes with guest anions

Sensors	F^-	Cl^-	AcO^-	$H_2PO_4^-$
HNBO	2.52×10^4	ND	ND	ND
HNAHPA	7.58×10^5	8.17×10^3	5.32×10^5	2.89×10^5[a]
HBODC	7.11×10^4, 1.41×10^4[a]	7.06×10^4, 1.34×10^4[a]	6.83×10^3	5.85×10^4

Anions were added as their tetrabutylammonium salts and the concentrations of the ESPT molecules, 2×10^{-5} M in DCM were kept constant throughout the titrations. The association constants (all errors are ±5%) were determined from the best least-squares fit given the stoichiometry of each complex (1:1, or 1:1 and 1:2) with correlation coefficient ~0.980–0.999 determined by fluorimetric titration at room temperature. ND: not detected because the spectral changes were very small.

[a] Association constant of the 1:2 complex. For HNBO $\lambda_{max}(ex) = 360$ nm, $\lambda_{max}(em) = 432$ and 490 nm; for HNAHPA $\lambda_{max}(ex) = 384$ nm, $\lambda_{max}(em) = 430$, 570 and 522 nm; for HBODC $\lambda_{max}(ex) = 400$ nm, $\lambda_{max}(em) = 445$, 618 and 546 nm, in the absence and presence of fluoride ions, respectively.

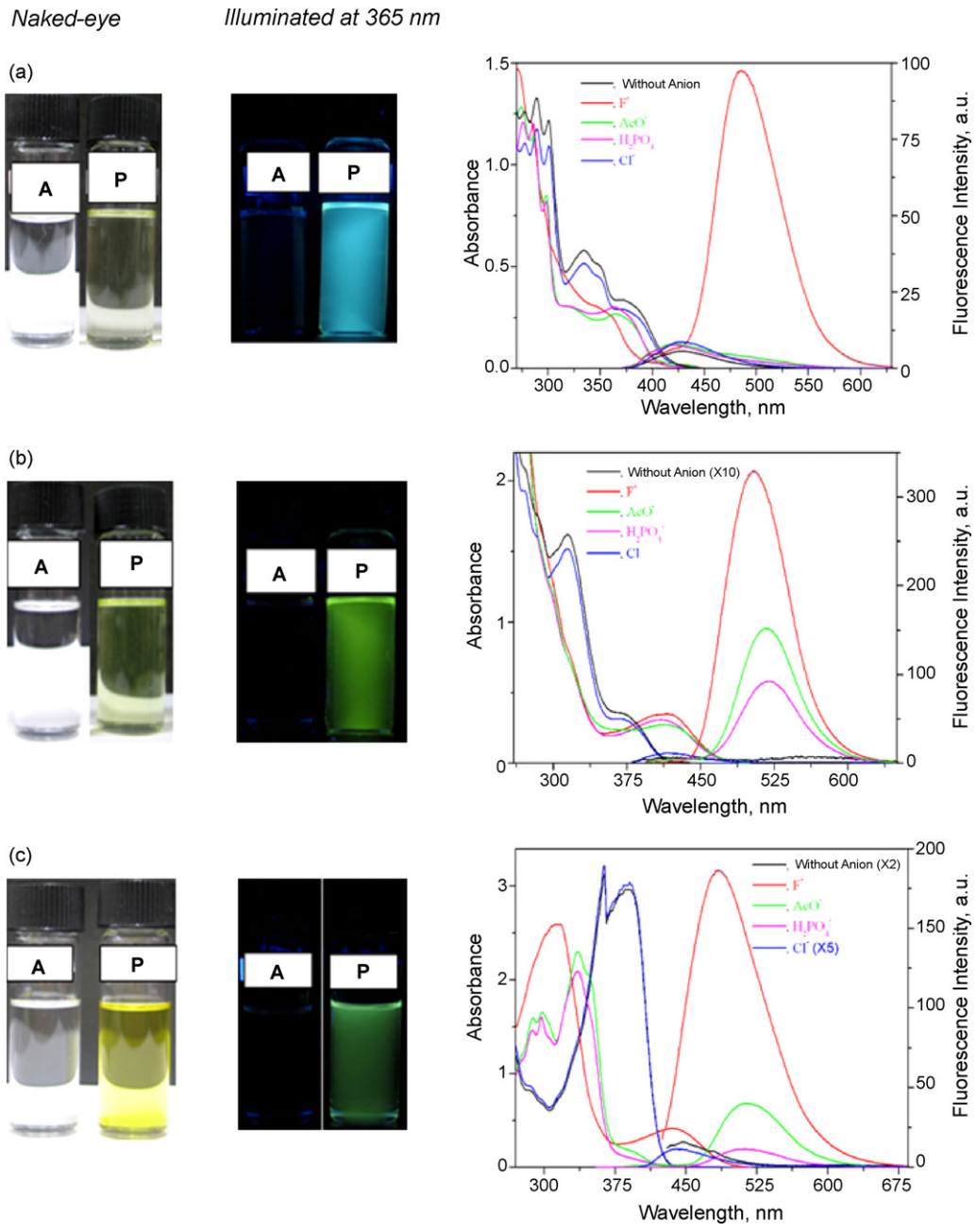

Fig. 2. (a–c) Photograph showing the color changes in HNBO, HNAHPA and HBODC in DCM (1×10^{-4} M) induced by the addition of 100 equivalents of fluoride ion under room lighting and illumination at $\lambda_{exc} = 365$ nm. The right panel shows the corresponding changes in the absorption and fluorescence spectra upon the addition of 100 equivalents of tetrabutylammonium fluoride, acetate, dihydrogen phosphate and chloride. The labels 'A' and 'P' denote the absence and presence of fluoride ions, respectively.

poorer selectivity of HBODC for F^-/Cl^- detection compared to HNAHPA and HNBO.

In conclusion, HNBO, HNAHPA and HBODC belong to a simple, hitherto unexplored class of anion receptors that operate on the basis of an excited state proton transfer signaling mechanism, which, allows for the sensitive and selective detection of fluoride anions under both visual (i.e., naked eye) and fluorescence emission conditions. Moreover, these molecules make it possible to detect the F^- ion quantitatively (see Supplementary material Table S1). Accordingly, we anticipate the use of these systems in various sensing applications as well as in other situations, such as anion transport and purification, for which the availability of cheap and easy-to-make anion receptors would be advantageous.

Supplementary material

UV-fluorescence spectra for both the absence and the presence of tetrabutylammonium fluoride (TBAF), chloride (TBAC), acetate (TBAAc) and phosphate (TBAP) in DCM. Selected fluorescence titration data and methods used to calculate the stoichiometries and stability constants of the complexes.

These materials are available free of charge via the Internet at http://www.elsevier.com/locate/jphotochemrev.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jphotochem.2007.04.028.

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