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ORIGINAL ARTICLE

Perylene Diimide Based Fluorescent Dyes for Selective Sensing of Nitroaromatic Compounds: Selective Sensing in Aqueous Medium Across Wide pH Range

P. S. Hariharan[1] · **J. Pitchaimani**[2] · **Vedichi Madhu**[2] · **Savarimuthu Philip Anthony**[1]

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Abstract Water soluble perylenediimide based fluorophore salt, N,N′-bis(ethelenetrimethyl ammoniumiodide)-perylene-3,4,9,10-tetracarboxylicbisimide (PDI-1), has been used for selective fluorescence sensing of picric acid (PA) and 4-nitroaniline (4-NA) in organic as well as aqueous medium across wide pH range (1.0 to 10.0). PDI-1 showed strong fluorescence in dimethylformamide (DMF) ($\Phi f=0.26$ (DMF) and moderate fluorescence in water. Addition of picric acid (PA) and 4-nitroaniline (4-NA) into PDI-1 in DMF/aqueous solution selectively quenches the fluorescence. The concentration dependent studies showed decrease of fluorescence linearly with increase of PA and 4-NA concentration. The interference studies demonstrate high selectivity for PA and 4-NA. Interestingly, PDI-1 showed selective fluorescence sensing of PA and 4-NA across wide pH range (1.0 to 10.0). Selective fluorescence sensing of PA and 4-NA has also been observed with trifluoroacetate (PDI-2), sulfate (PDI-3) salt of PDI-1 as well as octyl chain substituted PDI (PDI-4) without amine functionality. These studies suggest that PA and 4-NA might be having preferential interaction with PDI aromatic core and quenches the fluorescence. Thus PDI based dyes have been used for selective fluorescent sensing of explosive NACs for the first time to the best our knowledge.

Keywords Perylene diimide · Fluorescent sensor · Explosive sensor · Picric acid sensor

Introduction

Developing fluorescent chemosensors have attracted significant attention over the years because they not only help for environmental screening of potentially hazardous chemicals but also serve as diagnostic tool for medicinal field and mechanistic tool for understanding biochemical process [1–4]. The simple operation, real time monitoring, fast response and high sensitivity further enhanced the interest in the field [5]. Particularly, small organic fluorophore molecules are highly preferred since they often synthesized via straightforward reaction, and importantly their solubility and structures can be tuned chemically to enhance the sensing and selectivity [6]. Several fluorescent sensors for selective sensing of heavy metal cations (Hg^{2+}, Zn^{2+}, Cd^{2+} and Mg^{2+}) and anions such as F^{-} and cyanide have been designed and synthesized [7–12].

Nitroaromatic compounds (NACs) such as nitrobenzene (NB), 4-nitroaniline (4-NA), trinitrotoluene (TNT) and picric acid (PA) are extensively used for several applications that include dyes, insecticides and explosives [13–15]. NB used as industrial solvents whereas TNT is commonly used explosive chemical. PA has also been widely used as explosives and on small scale it used as antiseptic and for the treatment of burns [16]. Further PA has finds application in dye industries, pharmaceuticals and chemical laboratories [17]. However, contamination of NACs in water and soils are very serious especially chemically sensitive animals and plants [18, 19]. It is noted that some of the NACs like PA and nitrobenzoic

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✉ Vedichi Madhu
madhu@karunya.edu

✉ Savarimuthu Philip Anthony
philip@biotech.sastra.edu

[1] School of Chemical & Biotechnology, SASTRA University, Thanjavur 613401, Tamil Nadu, India

[2] School of Science and Humanities, Department of Chemistry, Karunya University, Coimbatore 641 114, Tamil Nadu, India

acids are highly water soluble. For example, exposure of PA to humans can cause respiratory problems, damage liver, and bladder tumor [20–23]. Therefore, it is of great importance for detection and quantification of these toxic explosive NACs that contaminate the environment or ecosystem. Various techniques including gas chromatography, electrochemical methods, solid-phase micro-extraction-liquid chromatography, ion mobility spectrometry, Raman spectroscopy and fluorescence spectroscopy have been adopted for the detection of nitro explosives [24–28]. Of these methods, fluorescence is one of the preferred methods over others because of its high sensitivity, selectivity, fast response and easy sample preparation [1–6]. Fluorescent probes based on pyrene, polycyclic aromatic derivatives, triphenylamine derivatives, conjugated polymers, porphyrin derivatives and Rhodamine derivatives have been investigated for selective sensing of nitroaromatics [29–32]. Pang et al. recently reported dansyl modified squaric dye for selective sensing of PA [29]. BODIPY based fluorescent chemosensor was developed by Ravikanth et al. [33]. Nevertheless still it is a great challenge to develop fluorescent sensor particularly water soluble fluorescent probes.

Perylenediimides (PDI) are highly coloured organic dyes with excellent optical properties such as high molar extinction coefficients, fluorescence quantum yields and light harvesting properties and outstanding thermal, chemical and photochemical stability that made them as potential materials for various optoelectronic devices [34–37]. The excellent fluorescence properties of PDIs have been utilized to develop fluorescent probes for some metal cations and anions [38–46]. Nonetheless, the growth of PDIs as fluorescent probes for sensing applications are relatively slow compared to Rhodamine and coumarine based dyes that could be due to poor water solubility. Moreover, the strong fluorescent PDIs have never been used as fluorescent probe for detecting explosive NACs. In this manuscript, we report the selective fluorescence sensing of picric acid (PA) and 4-nitroaniline (4-NA) using PDI fluorophore salt, *N, N′*-bis(ethelene trimethylammoniumiodide)-perylene-3,4,9,10-tetracarboxylicbisimide (PDI-1) in organic as well as aqueous medium across wide pH range (1.0 to 10.0). Addition of PA and 4-NA selectively quenches the fluorescence of PDI-1. The concentration dependent studies suggest that PDI-1 can detect PA and 4-NA up to 1 μM. The interference studies demonstrated high selectivity of PDI-1 for PA and 4-NA and other NACs did not have significant influence. Interestingly, PDI-1 showed strong fluorescence and selective fluorescence sensing of PA and 4-NA across wide pH range (1.0 to 10.0). To get the insight on the fluorescence sensing, trifluoroacetate (PDI-2), sulfate (PDI-3) and octyl chain substituted PDI (PDI-4) with amine functionality have also explored for fluorescence sensing of NACs which also showed selective sensing of PA and 4-NA. These studies indicate that there might be selective and preferred interaction of PDI aromatic core with PA/4-NA. Thus, for the first times to the best our knowledge, PDI fluorescent dyes have been used for selective sensing of NACs in water and organic solvents.

Experimental Section

Perylenetetracarboxylic-3,4,9,10-bisanhydride and 2-N,N′-dimethylaminoethylenediamine were received from Sigma-Aldrich and used as without further purification. N,N′-bis(dimethylaminoethyl)-perylene-3,4,9,10-tetracarboxylic bis imide (PDI-1a), N,N′-bis(ethelenetrimethylammoniumiodide)-perylene-3,4,9,10-tetracarboxylicbisimide (PDI-1) and N,N′-bis(octyl)-perylene-3,4,9,10-tetracarboxylicbisimide was synthesized according to the reported procedure [47]. FT- IR spectra for the synthesized compound was measured and compared with the reported literature to confirm the formation of PDI compounds. Absorption and fluorescence spectra were recorded using Perking Elmer Lambda 1050 and Jasco fluorescence spectrometer-FP-8200 instruments. FT-IR spectra were measured using a Shimadzu IRAffinity-1S spectrophotometer with KBr pellets.

Synthesis of N,N′-bis(Dimethylaminoethyl)-Perylene-3,4,9,10-Tetracarboxylicbisimide (PDI-1a) [47]

In atypical procedure, perylenetetracarboxylic-3,4,9,10-bisanhydride (2 g, 5 mmol), quinoline 15 ml, and 2-N,N′-dimethylaminoethylenediamine (20 mmol) and dicyclohexylcarbodiimide (0.7 g, 3.4 mmol) were refluxed with stirring in an inert atmosphere at 230–240 °C for 3–4 h. After cooling to room temperature, the reaction mixture was poured into 200 ml of ethanol. The resultant precipitate was filtered and dried. The yield was 80 %. IR: ν (cm-1)=3016, 2958, 1699, 1652, 1592, 1571, 1508, 1443, 1270.

Synthesis of N,N′-bis(Ethelenetrimethylammoniumiodide)-Perylene-3,4,9,10-Tetra-Carboxylicbisimide (PDI-1)

Methyl iodide (10 mmol) was added into toluene (80 ml) solution of PDI-1a (2.5 mmol) and refluxed with vigorous stirring for 90 min. After cooling to room temperature, the precipitate was filtered, washed with ether and dried. The yield was 88 %. IR: ν (cm-1)=3485, 3010, 2957, 1700, 1656, 1590, 1576, 1560, 1501, 1441, 1402, 1268.

Synthesis of N,N′-di(Octyl)-Perylene-3,4,9,10-Tetracarboxylicbisimide (PDI-4)

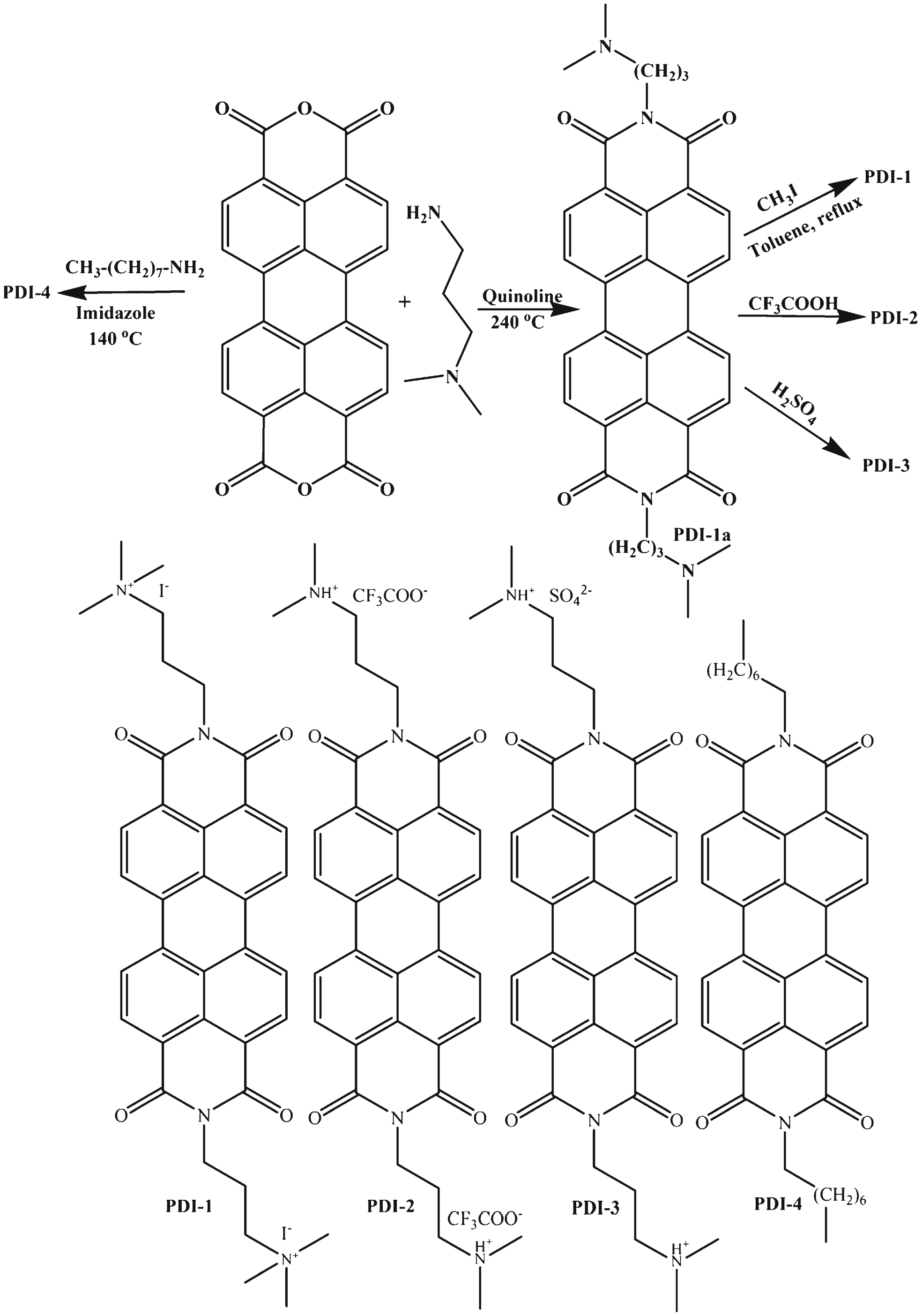
In a 50 mL round bottom flask, perylene-3,4,9,10-tetracarboxylic dianhydride, (0.784 g, 2 mmol), 1-

octylamine (0.516 g, 4 mmol) and imidazole (4 g) were added and refluxed at 140 °C for 3 h. The reaction mixture was cooled to room temperature and dispersed in 100 ml of ethanol followed by addition of 300 ml of 2 M HCl. The mixture was stirred overnight. The resulting dark-red solid was filtered off and washed thoroughly with distilled water followed by methanol. The collected solid was dried under vacuum. The yield was 75 %. IR: ν (cm-1)=3017, 2959, 1671, 1650, 1591, 1575, 1502, 1271.

Results and Discussion

PDI derivative of dimethylamine (PDI-1a) is highly insoluble in most of the solvent and showed very weak fluorescence in aqueous solution (0.06 M NaOH and 0.05 M detergent (SDS) [47]. However, conversion dimethyl amine to trimethyl ammonium iodide salt (PDI-1, Scheme 1) increased the solubility in different solvent (DMF, DMSO) including water and exhibited strong fluorescence. PDI-1 showed fluorescence

Scheme 1 Synthetic scheme and molecular structure of PDI fluorophores (PDI-1 to PDI-4)



quantum yield of 0.26 (DMF) compared to fluorescein. PDI-1 has also showed good fluorescence in water. Fluorescence sensing of PDI-1 was explored by adding NACs such as (2-nitroanilline (2-NA), 3-nitroanilline (3-NA), 2-nitroanilline (4-NA), 2, 4-dinitroanilline (2,4-DNT), trinitrotoluene (TNT), 4-nitrobenzoic acid (4-NBA), nitrobenzene (NB), picric acid (PA) and 4-nitrophenol (4-NP)) and monitoring the fluorescence change. PDI-1 in DMF showed fluorescence at 545 nm and a small hump at 580 nm. Interestingly, addition of PA and 4-NA into DMF solution of PDI-1 strongly reduces the fluorescence intensity both at 545 and 580 nm (Fig. 1). The fluorescence intensity of PDI-1 was reduced by 4–6 times by addition of PA and 4-NA. Other NACs did not significantly quench the PDI-1 fluorescence. Excitation spectra were recorded to get the insight on the selective fluorescence quenching of PDI-1 by PA and 4-NA (Fig. S1). The excitation spectrum of PDI-1 showed peak at 370, 465, 490 and 520 nm. Addition of PA and 4-NA leads to selective disappearance of peak at 370 nm. Nevertheless, other peaks are observed clearly. Other NACs addition did not show any change in the excitation spectra of PDI-1. These studies clearly suggest that PA and 4-NA might selectively interact with PDI-1 aromatic core. The concentration dependent studies of PDI-1 for PA and 4-NA showed linear decrease of fluorescence intensity (Fig. 2). This study indicate that PDI can detect both PA and 4-NA easily up to 1.0 μM. Interference studies were performed to explore the influence of the other NACs on the detection of PA and 4-NA by PDI-1 (Fig. 3). The interference studies confirmed high selectivity and other NACs had little influence on the sensing of PA and 4-NA.

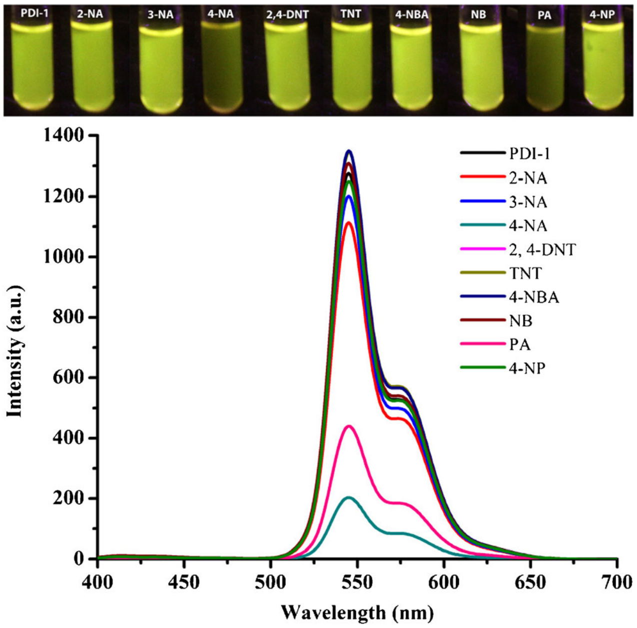


Fig. 1 Digital images and fluorescence spectra of PDI-1 with different NACs in DMF

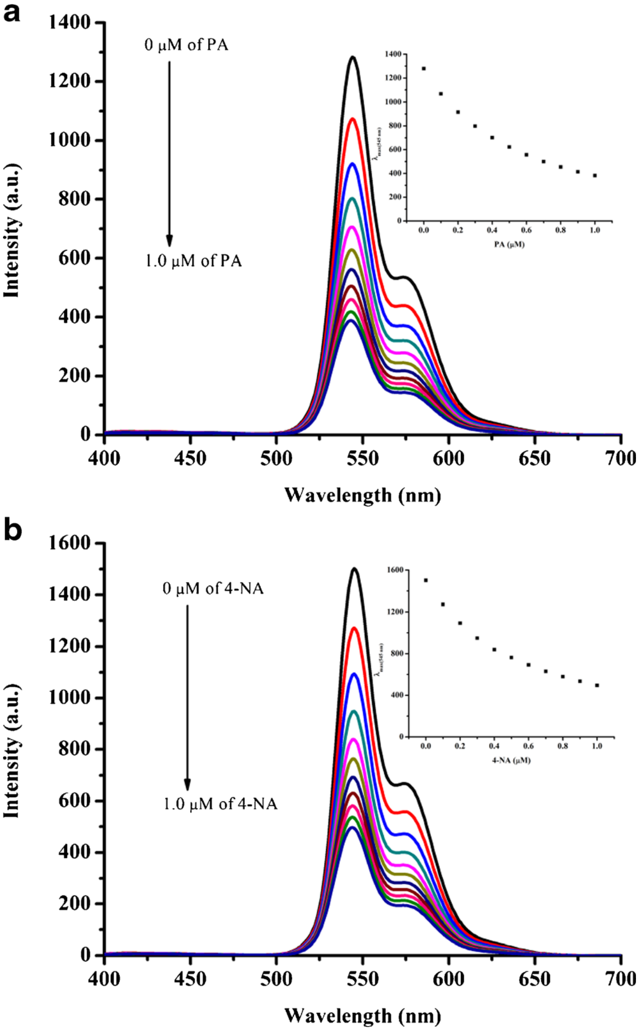


Fig. 2 The change of PDI-1 fluorescence intensity with concentration of PA (**a**) and 4-NA (**b**) in DMF

The fabrication of salt form of PDI makes it soluble in water. Importantly, PDI-1 in water also showed good fluorescence. PA is highly water soluble, hence it would be interesting to develop PA sensor in aqueous medium. PDI-1 in water exhibited fluorescence λ_{max} at 548 nm and a small hump at 580 nm. Addition of PA and 4-NA into aqueous solution of PDI-1 also selectively quenched the fluorescence (Fig. 4). As observed in DMF, other NACs addition did not show any significant influence on the PDI-1 fluorescence. In aqueous medium, PDI-1 fluorescence intensity was quenched by 10 times with addition of PA whereas 4-NA addition reduces fluorescence intensity by 6 times. PDI excitation spectra along with different NACs were recorded to get the insight on the selective fluorescence quenching of PDI-1 by PA and 4-NA (Fig. S2). The excitation spectrum of PDI-1 in water showed peak at 370, 490 and 540 nm. As observed in DMF, addition of PA and 4-NA leads to selective disappearance of peak at 370 nm. It is noted that apart from complete disappearance of 370 nm peak, PA addition reduces the intensity at 490 and 540 nm. However, addition of other

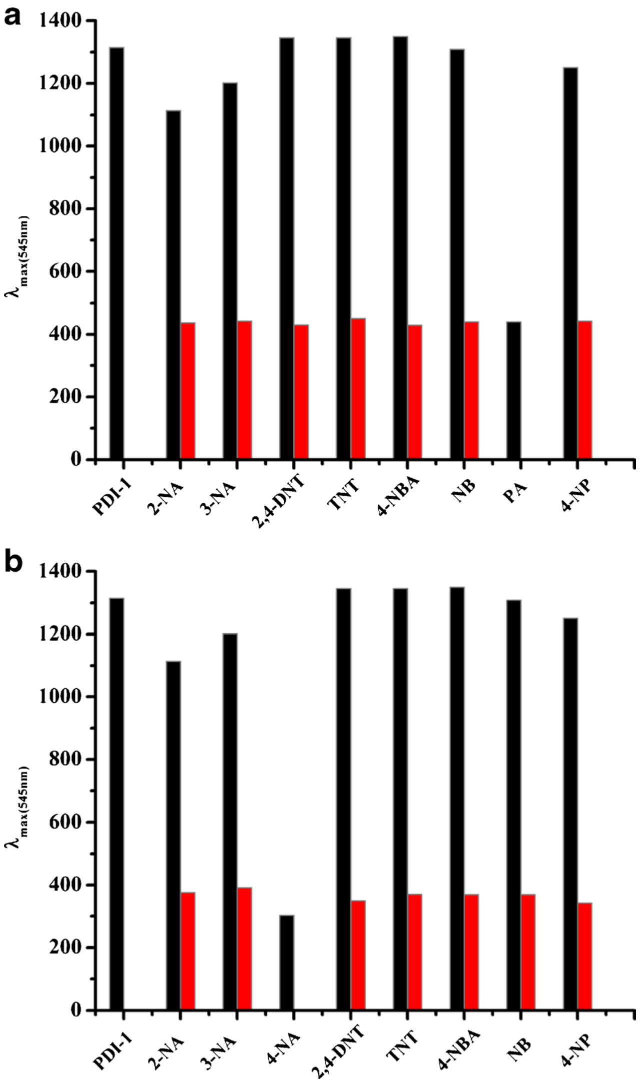


Fig. 3 Selectivity studies of PDI-1 for (**a**) PA nd (**b**) 4-NA (10^{-6} M) in presence of other NACs (10^{-3} M) in DMF

NACs did not show any significant change in the PDI-1 excitation spectra. These studies again indicate that PA and 4-NA might selectively interact with PDI-1 aromatic core. In water, PA might have stronger interaction with PDI-1 relative to 4-NA. The concentration dependent studies of PDI-1 for PA and 4-NA were performed to observe the change of fluorescence with concentration and calculate the limit of detection. PDI-1 fluorescence intensity was linearly decreased with increased addition of PA/4-NA from 0.1 to 1.0 μM (Fig. S3). It is noted that 4-NA addition quenches the PDI-1 fluorescence relatively stronger in DMF whereas PA quenches strongly in water. The interference studies of PDI-1 indicate the high selectivity for PA and 4-NA (Fig. 5). The interference studies suggest that other NACs did not have significant influence on the sensing of PA and 4-NA.

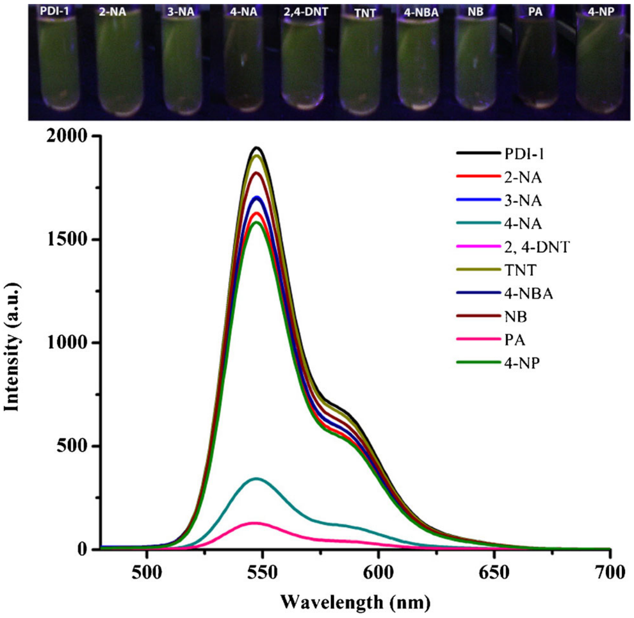


Fig. 4 Digital images and fluorescence spectra of PDI-1 with different NACs in water

Developing fluorescence chemosensor that can selectively detect NACs across wide pH range are highly interesting and challenging since most of the fluorophore exhibit strong fluorescence either at neutral/acidic/basic medium only. This might be due to the change of electronic structure upon addition/removable of proton by the change of pH. PDI-1 is soluble in water across wide pH range (1.0 to 10.0). Interestingly, it exhibit relatively steady fluorescence at pH 1.0 to 10.0 (Fig. S4). It is noted that PDI-1 showed slightly stronger fluorescence at lower pH compared to basic medium. The above fluorescence sensing experiment in water has performed at neutral *pH*. Interestingly, addition of PA and 4-NA into aqueous solution of PDI-1 at pH 1.0, 3.0, 8.0 and 10.0 showed selective fluorescence quenching (Fig. 6 and S5). However, other NACs did not show significant influence on the PDI-1 fluorescence. The fluorescence intensity reduction was nearly same at all pH solution. Thus, this study demonstrates that PDI-1 can be used for selective sensing of PA and 4-NA across wide pH range.

To study the effect of anions in PDI-1, trifluoro acetate and sulfate salt of PDI was prepared in-situ from PDI-1a and fluorescence sensing was investigated. Trifluoro acetate (PDI-2) salt was prepared by adding trifluoroacetic acid (2 mole ratio) into PDI-1a (1 mole) powder mixed in DMF. Similarly, sulfate (PDI-3) salt was prepared by adding sulfuric acid (1 mole ratio) into PDI-1a powder stirred in DMF. In both cases, the insoluble PDI-1a was converted to soluble form that produced clear solution. Similar to PDI-1, both PDI-2 and PDI-3 showed strong fluorescence in DMF. PDI-2 showed fluorescence λ_{max} at 543 nm. Addition of PA and 4-NA selectively quenches the fluorescence of PDI-2 (Fig. S6). As observed with PDI-1, other NACs did not show any significant fluorescence quenching. The concentration dependent studies of

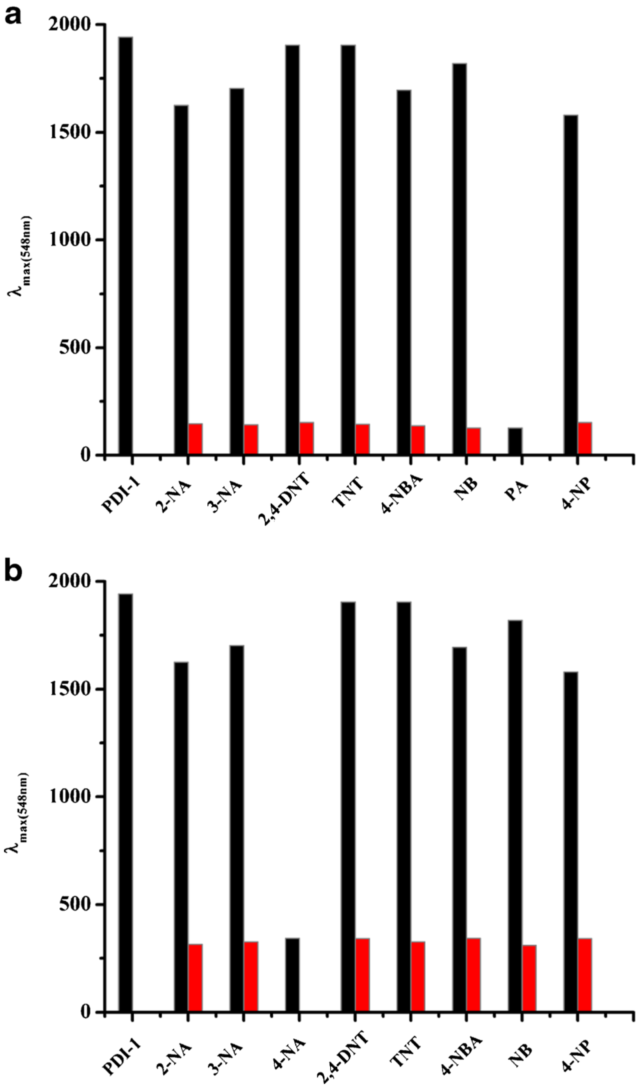


Fig. 5 Selectivity studies of PDI-1 for (**a**) PA nd (**b**) 4-NA (10^{-6} M) in presence of other NACs (10^{-3} M) in water

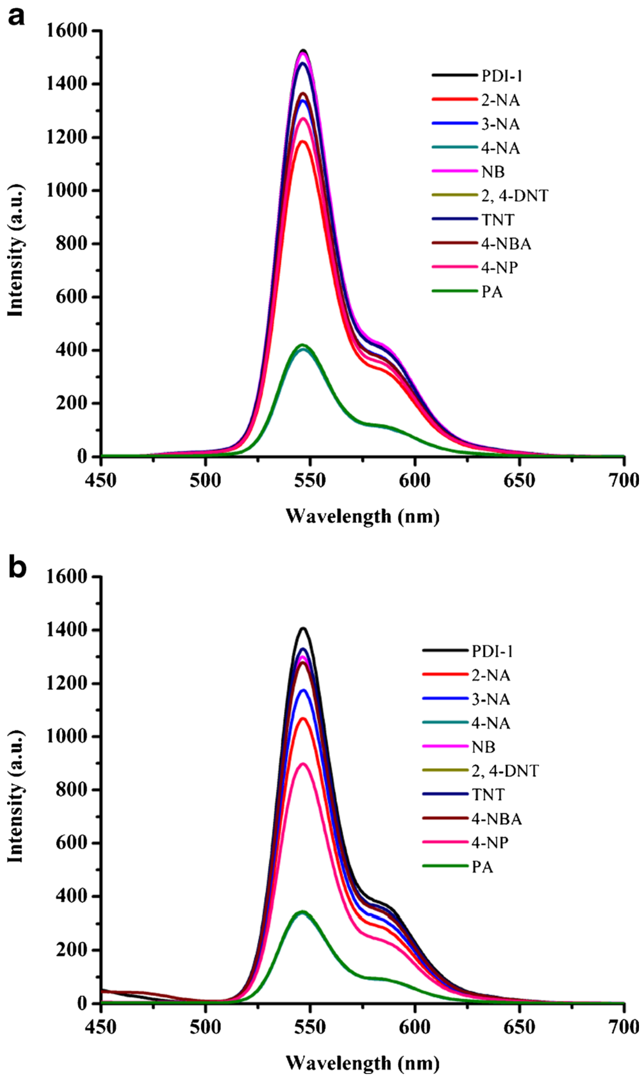


Fig. 6 Fluorescence spectra of PDI-1 with different NACs in water at pH (**a**) 1.0 and (**b**) 10.0

PDI-2 exhibited decrease of fluorescence intensity linearly with the concentration of PA and 4-NA (Fig. S7). PDI-2 also showed the detection limit of 1 μM for PA and 4-NA. The interference studies demonstrate the high selectivity for PA and 4-NA in presence of other NACs (Fig. S8). PDI-3 has also showed selective fluorescence quenching for PA and 4-NA only (Fig. S9). To get the insight on the fluorescence quenching of PDI salts, PDI derivative with alkyl group (PDI-4) has been prepared. Strong fluorescence was observed for PDI-4 when dissolved in tetrahydrofuran (THF). NACs sensing studies showed selective fluorescence quenching of PDI-4 by PA and 4-NA only. As observed with other PDIs, addition of other NACs did not show significant fluorescence quenching. These studies indicate that substitutional group at imide nitrogen whether it is hydrophilic amine salt functionality or hydrophobic alkyl chain did not show significant change in fluorescence sensing of PDI dyes. PDI-1 showed nearly same fluorescence intensity from acidic to basic medium. The fluorescence of PDI-1 was quenched by the addition of PA and 4-NA across wide pH range (1.0 to 10.0). This study suggests that selective NACs sensing of PDI-1 might not be due to the formation of supramolecular system via H-bond interactions. Since at basic condition, hydroxyl proton of PA would be deprotonated completely whereas at acidic medium it will be completely protonated that might modulate the H-bonding formation between PDI-1 and NACs. This further ruled-out any H-bonding interaction between the carbonyl group of PDI-1 and NACs. PDI-4 that has only alkyl groups without any ionic functionality has also showed similar selective fluorescence quenching for NACs. Hence PA and 4-NA might be forming selective electrostatic interaction with PDI aromatic core that quenches the fluorescence. The selective fluorescence sensing of PDI-1 for NACs in different water samples (tap, pond and ground water) demonstrate the practical applicability of PDI-1 for NACs sensing (Fig. S11).

Conclusion

In conclusion, highly selective fluorescence sensing of PA and 4-NA has been demonstrated using perylenediimide based fluorophore salts, (PDI-1 – PDI-3) and octyl chain attached PDI (PDI-4), in organic and aqueous medium. The strong fluorescence of PDI-1 in DMF was selectively quenched by the addition of PA and 4-NA. PDI-1 in water also showed selective fluorescence sensing of PA and 4-NA. The concentration dependent studies of PDI-1 showed decrease of fluorescence intensity linearly with increase of PA and 4-NA concentration. The detection limit of PA and 4-NA by PDI-1 is 1 μM. The interference studies confirmed the high selectivity of PDI-1 for PA and 4-NA. Interestingly, PDI-1 exhibited nearly same fluorescence intensity across wide pH range (1.0 to 10.0). Importantly, PDI-1 showed selective fluorescence sensing of PA and 4-NA at pH 1.0 to 10.0. The fluorescence sensing studies of trifluoroacetate (PDI-2), sulfate (PDI-3) salt and octyl chain attached derivative (PDI-4) has also showed selective fluorescence sensing of PA and 4-NA. These controlled studies suggest that there could be selective interaction between PDI core and PA/4-NA and substitutional group at imine position might not play significant role in sensing. Thus, water soluble highly fluorescent PDI dyes have been employed for selective sensing of explosive NACs both in organic and aqueous medium.

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