A Highly Selective Perylenediimide-Based Chemosensor: “Naked-Eye” Colorimetric and Fluorescent Turn-On Recognition for Al$^{3+}$

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A novel “turn-on” fluorescent probe (PCN) was designed, synthesized, and characterized with perylene tetracarboxylic disimide as the fluorophore and Schiff base subunit as the metal ion receptor. The probe demonstrated a considerable fluorescence enhancement in the presence of Al$^{3+}$ in DMF with high selectivity and sensitivity. Furthermore, the considerably “off–on” fluorescence response simultaneously led to the apparent color change from colorless to brilliant yellow, which could also be identified by naked eye easily. The sensing capability of PCN to Al$^{3+}$ was evaluated by the changes in ultraviolet–visible, fluorescence, Fourier transform–infrared, proton nuclear magnetic resonance, and high-resolution mass spectrometry spectroscopies. The linear concentration range for Al$^{3+}$ was 0–63 µM with a detection limit of 0.16 µM, which allowed for the quantitative determination of Al$^{3+}$.

Keywords: fluorescence, perylened derivatives, Al$^{3+}$, turn on, hydrolysis

INTRODUCTION

Aluminum is the third most prevalent metal in the lithosphere, and it is toxic to living organisms due to its potential neurotoxicity in excessive amounts (Frantzios et al., 2000; Abeywickrama et al., 2019). Excess Al$^{3+}$ will induce a wide range of diseases such as microcytic hypochromic anemia, osteoporosis, muscular atrophy, and Alzheimer’s disease (Cronan et al., 1986; Fasman, 1996; Nayak, 2002; Walton, 2007; Zhang et al., 2020). According to the recommendation of the World Health Organization, the tolerable value of average human intake of Al$^{3+}$ ions is around 3.0–10.0 mg/day (Valeur and Leray, 2000; Qin et al., 2014). Moreover, the environment may be polluted due to high level of Al$^{3+}$ in the ecosystem (Godbold et al., 1988; Sade et al., 2016; Ye et al., 2019a). Fluorescence techniques for detecting various ions have become an optimal choice due to their high sensitivity and selectivity (Suresh et al., 2018; Ye et al., 2019b; Bai et al., 2020; Wu et al., 2020; Zhao et al., 2020). In the past few years, various fluorescent chemosensors including coumarin, naphthalimide, pyrene, BODIPY, anthracene, and rhodamine were exploited for detection of Al$^{3+}$ (Samanta et al., 2014; Hossain et al., 2017; Kozlov et al., 2019; Prabhu et al., 2019; Roy et al., 2019; Li et al., 2020).

Perylene tetracarboxylic disimide derivatives (PDIs), as a representative of strong fluorescence and superior functional organic dyes, display exceptional optical, redox, and electrochemical properties and high thermal stability (Ahrens et al., 2003; Wurthner, 2004; Wasielewski, 2006; Xu et al., 2008; Luo and Chen, 2013; Gao et al., 2020). In addition to their industrial application as
pigment, many PDIs also exhibit unique spectroscopical, near-unity fluorescence quantum yields, and strong electron-deficient nature. Owing to the delocalization effect and rigid plane, PDIs are endowed with high fluorescence quantum yields and have been widely utilized as a chromophore (Miyake et al., 2006; Soh et al., 2006; He et al., 2007; Kirschning et al., 2007; Yan et al., 2009; Ruan et al., 2010; Nanashima et al., 2012; Zhang et al., 2013). Fluorescent chemosensors based on modified perylene dye have been used to monitor various ions. PDI-based 2-thiophenamine derivative was reported for the selective determination of Hg$^{2+}$ with a detection limit of 2.2 $\mu$M in DMF–H$_2$O (1:1, v/v; Malkondu and Erdemir, 2015). A new water-soluble fluorescent probe was given by the condensation polymerization between 2,2':6',2''-terpyridine-containing dibromide and perylene diimide-diamines, which was used to determine Fe$^{3+}$ and monitors the Fe$^{3+}$/Fe$^{2+}$ transition after the addition of a reducing agent such as ascorbic acid (Vc; Jin et al., 2016).

Herein, a novel perylenebisimide-based fluorescent sensor for monitoring Al$^{3+}$ had been designed (Scheme 1; Erdemir and Kocyigit, 2018; Kashyap et al., 2019; Kumar et al., 2019; Erdemir et al., 2020). $N,N'$-bis[2-p-chlorobenzaldehyde]-ethyl perylene-3,4,9,10-tetracarboxylic diimide (PCN) was obtained via the polycondensation reaction of amino functionalized perylene-3,4,9,10-tetracarboxylic diimide and p-chlorobenzaldehyde (Scheme 2) and characterized by Fourier transform–infrared (FT-IR), proton nuclear magnetic resonance ($^1$H NMR), carbon-13 nuclear magnetic resonance ($^{13}$C NMR), and high-resolution mass spectrometry (HRMS) spectroscopies. It showed drastic fluorescence enhancement and obvious color change toward its binding of Al$^{3+}$ in DMF solution with excellent selectivity and sensitivity.

EXPERIMENTAL

Materials and Instruments

All chemicals and solvents were purchased from commercial providers and used without purifying. FT-IR spectra were measured using a Bruker ALPHA-T spectrometer (KBr, Bruker, Germany). The $^1$H NMR and $^{13}$C NMR spectra were recorded on a Bruker AVANCE 400 MHz system (Bruker, Germany) using CF$_3$COOD as the solvent. The HRMS was carried out on an FTMS Ultral Apex MS spectrometer (Bruker Daltonics Inc., USA). The ultraviolet–visible (UV-vis) spectra were gained on a UV-2550 ultraviolet spectrophotometer (Shimadzu, Japan). The fluorescence spectra were obtained through the PerkinElmer
LS55 fluorescence spectrometer (PerkinElmer, UK). All pH values were made with PHS-3C pH meter (Inesa, China).

**Synthesis of**

\[ \text{N,N'}-\text{bis(ethylenediamine)Perylene-3,4,9,10-Tetra-}
\text{carboxylic Bisimide (2)} \]

The \( \text{N,N'}-\text{bis(ethylenediamine)perylene-3,4,9,10-tetra-}
\text{carboxylic acid dianhydride} \) was refluxed with continuous stirring at 90°C. After refluxing for 24 h, the resulting mixture was treated with 0.2 M sodium acetate–acetic acid buffer solution (pH = 5.3, 50 mL). Then, 1M NaOH was added to the filtrate until pH = 13. The residue was filtered and washed with water and methanol, and dried to give compound 2. Yield: 46 %. m.p. > 300°C. All spectra of structural characterization of compounds are in the Supporting Information. FT-IR data in KBr (cm\(^{-1}\)): 3,360, 3,296 (\( \nu \) N-H), 2,933, 2,894, 2,841 (\( \nu \) C-H), and 1,674 (\( \nu \) C=O). \(^1\text{H NMR}\) [\( \delta \)H in parts per million (ppm) in CF\(_3\)COOD (TMS), 400 MHz]: 8.94–9.01 (m, 8H, Ar-H), 4.96 (br, 4H, -CH\(_2\)-), and 3.98 (br, 4H, -CH\(_2\)-). \(^{13}\text{C NMR}\) [\( \delta \)C in CF\(_3\)COOD (TMS), 100 MHz]: 166.38, 136.31, 133.09, 129.33, 126.33, 124.34, 121.56, 116.19, 115.15, 113.38, 112.34, 40.57, and 38.40.

**Synthesis of**

\[ \text{N,N'-bis([2-p-Chlorobenzaldehyde]-ethyl]Perylene-3,4,9,10-Tetra-}
\text{carboxylic Diimide} \]

The final product PCN was synthesized on the basis of the previous literatures (Malkondu and Erdemir, 2015; Fu et al.,...
Compound 2 (0.46 g, 1 mmol) was suspended first in MeOH/CHCl$_3$ (1:1, 160 mL) followed by adding 0.7 g, 5 mmol p-chlorobenzaldehyde, and then the mixture was stirred at 50°C for 48 h. After cooling to room temperature, the precipitated solid was obtained and washed with methanol. The residue was recrystallized from MeOH/CHCl$_3$ (v/v, 4:1) to obtain the dark-red solid. Yield: 84 %. m.p. 393°C.

**RESULTS AND DISCUSSION**

**UV-Vis and Fluorescence Spectral Characteristics Studies**

The solvent effect of PCN has been studied through fluorescence measurement in different solvents (Supplementary Figure 8). The probe exhibits weak fluorescence properties in almost all the investigated solvents except EtOH. The compound exhibits weak yellow fluorescence emission with peaks from 541 nm (MeCN) to 558 nm (DMSO) in most solvents, but much no fluorescence in EtOH was observed. Based on the response mode of the fluorescent molecular probe’s "turn on" and "turn off," the solvent was selected for further study. The probe had failed to show the good selectivity toward various ions in DMSO and other solvents. The photophysical property of the fabricated fluorescent chemosensor in DMF was investigated. Free probe (PCN) demonstrated weak fluorescence at about 550 and 590 nm. To estimate the selectivity and sensitivity of PCN (10 µM), the UV-vis and fluorescence spectra of PCN toward different metal cations (such as Al$^{3+}$, Fe$^{3+}$, Cr$^{3+}$, Mg$^{2+}$, Pb$^{2+}$, Zn$^{2+}$, Hg$^{2+}$, Ca$^{2+}$, Cu$^{2+}$, Co$^{2+}$, Mn$^{2+}$, Sn$^{2+}$, Ni$^{2+}$, Ba$^{2+}$, Na$^{+}$, K$^+$, and Ag$^{+}$) have been investigated. UV-vis spectra of PCN were obtained in the existence of 5 equiv. of the tested cations. The absorption of PCN increased significantly in the presence of Al$^{3+}$ at 490 and 525 nm, and the colorless solution of PCN changed to yellow under natural light, indicating that the “naked eye” is visible (Figure 1). The fluorescence responses of PCN were measured in the presence of fivefold excess of various metal ions (Figure 2). Upon the addition of different cations (5 equiv.), only when added the Al$^{3+}$ into the solution can it induce a significant fluorescence enhancement at 550 and 590 nm. Moreover, a yellow-colored visual fluorescence change was observed after adding Al$^{3+}$ ion to the PCN solution. In contrast, most of the other metal ions, including some mono-,
and trivalent metal ions (Ag⁺, Na⁺, K⁺, Mg²⁺, Pb²⁺, Zn²⁺, Hg²⁺, Ca²⁺, Cu²⁺, Co²⁺, Mn²⁺, Sn²⁺, Ni²⁺, Ba²⁺, Cr³⁺, and Fe³⁺), were unresponsive to this system. The imine bond in PCN was hydrolyzed by the addition of Al³⁺ due to the Lewis acid character of Al³⁺, and compound 2 with strong fluorescent was released. It resulted to a prominent “light-on” yellow solution and fluorescence emission of PCN, which allowed for naked-eye detection of Al³⁺ under natural light and UV light of 365 nm. This mechanism was proven by FT-IR, ¹H NMR, and HRMS experiments. Therefore, PCN showed “off–on” response to Al³⁺ ions in the DMF solution. All these showed the good selectivity of PCN toward Al³⁺ over other cations.

FIGURE 3 | (A) UV-vis spectra recorded for probe PCN (10 µM) in DMF upon spectrometric titration with Al³⁺ (0–70 µM). (B) The relationship between the absorbance (525 nm) and the concentration of Al³⁺.

FIGURE 4 | (A) Fluorescence spectra recorded for probe PCN (10 µM) in DMF upon spectrometric titration with Al³⁺ (0–70 µM). (B) The relationship between the fluorescence intensity (550 nm) and the concentration of Al³⁺.

FIGURE 5 | (A) The linear responses of fluorescence intensity (550 nm) with Al³⁺ concentrations. (B) Benesie–Hildebrand plot of PCN between PCN and Al³⁺ in DMF.
| Probes | Working media | LOD (µM) | Sensing mechanism | References |
|--------|---------------|----------|-------------------|------------|
| ![Image](image1.png) | EtOH:Tris (1:1 v/v) | 0.34 | PET | Shen et al., 2018 |
| ![Image](image2.png) | MeOH:H₂O (8:1 v/v) | 0.1 | ICT and CHEF | Fu et al., 2019 |
| ![Image](image3.png) | EtOH:H₂O (4:1 v/v) | 0.299 | CHEF | Gan et al., 2017 |
| ![Image](image4.png) | EtOH:H₂O (6:4 v/v) | 4.369 | hydrolysis | Zhang et al., 2018 |
| ![Image](image5.png) | MeOH:H₂O (9:1 v/v) | 4.39 | PET and CHEF | Roy et al., 2017 |
| ![Image](image6.png) | DMF:H₂O (4:1 v/v) | 0.39 | AIE | Wang et al., 2018 |
| ![Image](image7.png) | CH₃CN | 0.31 | ESIPT and C=N isomerization | Tajbakhsh et al., 2017 |
| ![Image](image8.png) | DMF | 0.16 | hydrolysis | This work |

LOD, limit of detection; ICT, internal charge transfer; PET, photoinduced electron transfer; CHEF, chelation-enhanced fluorescence; AIE, aggregation-induced emission; ESIPT, excited-state intramolecular proton transfer.
UV-Vis and Fluorescence Titration Experiments

The UV-vis and fluorescence titrations were measured by increasing the amount of Al^{3+} (0–70 µM) to PCN in DMF. The intensity of absorbance at 490 and 525 nm increased gradually after the addition of an increasing amount of Al^{3+} (Figure 3). The absorbance of PCN became steady at 525 nm with the Al^{3+} concentration being 63 µM. The change of UV-vis spectral could attribute to the binding affinity of PCN. As depicted in Figure 4, the independent PCN exhibited a weak
emission in DMF. However, a distinct increase in fluorescence emission was observed after Al$^{3+}$ was added and a plateau with the addition of 63 µM of Al$^{3+}$ was achieved at 550 nm. The weak fluorescence may correspond to the photoinduced electron transfer (PET) process resulted from the N atom of imine (C=N) to the luminescent perylene unit (Upadhyay et al., 2018; Fu et al., 2019b). With the increase of Al$^{3+}$, the PET effect of the sensor PCN is inhibited, and thereby the intense fluorescence of PDI units is restored. Also, it clearly indicates the structural change of PCN by the interaction of Al$^{3+}$ with PCN. As shown in Figure 5A, the fluorescence intensity showed a linear relationship ($R^2 = 0.9956$) with concentration of Al$^{3+}$ quantitatively. The detection limit of PCN for sensing Al$^{3+}$ was calculated to be 7.75 $\times$ 10$^{4}$ M$^{-1}$ in the DMF solution, as increased from the fluorescence titration curves of probe PCN with Al$^{3+}$ (Figure 5B). The comparison of probe PCN with other Al$^{3+}$ chemical sensors based on Schiff’s base was summarized in Table 1 with different sensing mechanisms (Gan et al., 2017; Roy et al., 2017; Tajbakhsh et al., 2017; Shen et al., 2018; Wang et al., 2018; Zhang et al., 2018; Fu et al., 2019). Compared with other sensors, the advantage of probe PCN was its lower detection limit, but its insolubility in water was its shortcoming, which might limit its application in biological and environmental chemistry to some extent.

### The Competition Experiments Studies

The fluorescence spectra of PCN were investigated to examine its selectivity in the existence of other metal ions. First, 10 equiv. of background metal ions (100 µM) was added to the solution of PCN (10 µM) to form a PCN/M$^{3+}$ system, and then, 5 equiv. of Al$^{3+}$ (50 µM) was added into the solution. As shown in Figure 6, Al$^{3+}$ detection by compound PCN was not influenced by the selected background metal cations. Therefore, the combined results clearly indicated that PCN exhibited remarkable Al$^{3+}$ signaling behavior and can function as a high selectivity and disturbance-free Al$^{3+}$ fluorescent probe even in the existence of most competing metal cations.
Sensing Mechanism of PCN for Al$^{3+}$

To elucidate the sensing mechanism, the IR, NMR, and HRMS spectra of PCN-Al$^{3+}$ were performed. The FT-IR spectroscopic analysis of PCN and PCN–Al$^{3+}$ complexes is shown in Figure 7. Compared with the FT-IR spectra of PCN, the peaks appeared at 3,386 and 3,323 cm$^{-1}$ in the FT-IR spectra after the addition of 5 equiv. of Al$^{3+}$, which is consistent with the amino peak of intermediate 2. The result suggested that intermediate 2 might be regenerated, which is consistent with the previous spectral analysis. The $^1$H NMR spectroscopic analysis of PCN and the reaction product of PCN with Al$^{3+}$ are shown in Figure 8. The addition of Al$^{3+}$ resulted in different peak profiles, the signal of aldime protons (H$_4$) at 8.99 ppm completely disappeared, and these peaks at 7.67–7.98 ppm corresponding to aromatic protons (H$_5$) also disappeared comparing with $^1$H NMR spectra, which also confirmed that probe PCN was hydrolyzed. Moreover, by comparing the HRMS spectra in Figure 9, it was found that the original peak at [M+H]$^+$ 721.1407 for free PCN disappeared, and a new peak at [M+H]$^+$ 477.1552 emerged after the addition of Al$^{3+}$. All results clearly delineated that Al$^{3+}$ induced the cleavage of imine. In order to get full insight into the mechanism, the fluorescence spectra of compound 2 and PCN in the absence and presence of Al$^{3+}$ were recorded separately in Figure 10. Upon the spectral changes of PCN induced by Al$^{3+}$, it was found that the spectral data were nearly identical with those of compound 2, which clearly confirmed the cleavage of the C=N of PCN in the presence of Al$^{3+}$(Scheme 3).

In order to understand the sensing mechanism in the probe PCN in the absence or presence of Al$^{3+}$ ion, density functional theory (DFT) quantum mechanical approach was performed. A Gaussian program (Frisch et al., 2009) was employed for DFT calculations at the B3LYP-D3BJ/def2-SV(P) level (Stephens...
et al., 1994; Weigend and Ahlrichs, 2005; Frisch et al., 2009; Grimme et al., 2010). The S1 state geometry was optimized, and the corresponding molecular orbitals were recorded by isosurfaces with the isovalue at 0.02. In the excite state of PCN, HOMO-1 (−6.62 eV) of the receptor unit is close to fluorophore HOMO (−6.16 eV) and located above the fluorophore HOMO-9 (−7.67 eV). Hence, the electron of the HOMO-1 will be transferred to fluorophore regime through the reductive PET mechanism (Maity et al., 2019; Dos Santos Carlos et al., 2020). Significantly, the fluorescent “off” state of PCN is observed. After Al^{3+} ion hydrolyzed the probe PCN, the HOMO-2 (−6.44 eV) energy levels of the primary amine are decreased than that of the
fluorophore HOMO-1 (−6.18 eV), as also observed. Hence, PET could not efficiently operate from the HOMO-2 of the primary amine to the fluorophore's HOMO-1 upon the removal of imine moiety, resulting in the fluorescent “on” state (Figure 11). Thus, the present calculation demonstrated that the electron donor imine leads to a highly efficient PET process.

CONCLUSION

In summary, a novel PDI-based Schiff base derivative PCN was synthesized and utilized as a fluorescent probe. PCN exhibited a selective turn-on response to Al$^{3+}$ over other coexisting competitive metal ions in DMF. DFT calculations showed that coordination of PCN to Al$^{3+}$ inhibits the PET process. The C=N of PCN was hydrolized by Al$^{3+}$, leading to the return to the intermediate compound, which resulted naked-eye visible color changes from colorless to yellow and nonfluorescent to yellow fluorescent. The detection limit was sufficiently low to determine the micromolar levels of Al$^{3+}$. This sensor is valuable for Al$^{3+}$ analysis in environmental samples.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/Supplementary Material, further inquiries can be directed to the corresponding author/s.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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