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Polyhedral oligosilsesquioxane tethered tetraphenylethylene as turn-on fluorescent sensor for fluoride ions detection



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ARTICLE INFO

Keywords: Aggregation-induced emission Luminescence Polyhedral oligosilsesquioxane Chemosensor Selectivity

ABSTRACT

A novel tetraphenylethylene derivative tethered with polyhedral oligosilsesquioxane nanocages (TPE-POSS) was designed and synthesized as turn-on fluorescent sensor for detection of F⁻. The aggregation-induced emission (AIE) property, self-assembly behavior and detection mechanism of the complex were then elucidated by UV-vis absorption spectra, photoluminescence spectra and dynamic light scattering experiments. The results reveal that POSS nanocages can collapse due to the Si–O bond cleavage with the present of F⁻. The POSS nanocages collapse then trigger the AIE activity of TPE luminogens, endowing TPE-POSS with excellent turn-on detecting ability, high selectivity and sensitivity to F⁻, with a detection limit as low as 1.66×10^{-7} M.

1. Introduction

Anions widely exist in nature and play a vital role in many fields such as environmental science, clinical medicine, biochemical process and chemical [1,2]. Among various anions, fluoride ion (F^-) is of great interest due to its important function for human health and the environment [3–5]. The presence of fluoride in low levels has proven to be critical for the prevention of dental caries and osteoporosis treatment. However, overexposure to fluoride can result in many diseases, such as kidney disorders, metabolic disorders and skeletal fluorosis. For these reasons, it is of great significance to develop a simple and precise detecting method for the fluoride levels.

Up to now, several analytical techniques for detecting fluoride ion have been reported, such as ion-selective electrode [6], colorimetry [7], ion chromatography and so on [8–10]. Nevertheless, those methods usually suffer from some intrinsic disadvantages, including tedious sample preparations, sophisticated instruments and time-consuming operations. By contrast, fluorescence detection method is emerging as an alternate due to its advantages such as simple operation, low cost, high selectivity and sensitivity. So far, numerous fluorescent sensors for the detection of fluoride ions have been developed [11–22].

According to the recognition mechanism, a myriad of fluoride ion

fluorescence sensors can be divided into deprotonation type and reactive type [23–25]. Furthermore, many F⁻ fluorescent probes based on the mechanism of deprotonation show poor selectivity due to they can easily interfered by basic anions such as OH⁻, H₂PO₄ [23]. To avoid the shortcoming, fluoride ion fluorescence sensors can be designed based on the mechanism of the strong affinity between silicon and fluoride. For example, polyhedral oligosilsesquioxane (POSS) substituted perylene diimide (PDI) derivative shows selective response to fluoride ion due to the fluoride-triggered self-quenching of PDI [26]. However, such fluorescent F⁻ sensor is based on the fluorescence quenching response. The turn-off mode is easily interfered by photobleaching or fluorescence sensors, which is also an inherent shortcoming of most fluorescence sensors.

As is well known, the conventional luminophores exhibit strong emission in dilute solutions, but emission becomes weaker or even quenched at high concentration or in an aggregated state. The aggregation caused quenching (ACQ) effect of many luminogens creats obstacles, which ultimately limits its application. In 2001, Tang's group put forward the fascinating concept of aggregation-induced emission (AIE) materials, which exhibit strongly emission and high quantum yields at high concentration or in the aggregate state [27–31]. Especially, as a typical AIE molecule, tetraphenylethylene (TPE) has the

https://doi.org/10.1016/j.dyepig.2021.109491

Received 2 February 2021; Received in revised form 20 May 2021; Accepted 20 May 2021 Available online 24 May 2021 0143-7208/© 2021 Elsevier Ltd. All rights reserved.

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Scheme 1. Synthetic routes of TPE-POSS.

advantages of high solid luminescent efficiency, high yield and easy functionalization [32–35]. Due to the distinct advantages, TPE derivatives have been widely used in organic semicodunctors, organic light-emitting diodes, ion recognition and sensing [36–38]. However, there are few reports about TPE-based probe for fluoride ion [39]. In particular, to the best of our knowledge, the fabrication of TPE-based probe with turn-on mode has still not been reported.

In this work, a novel fluorescent probe molecular (TPE-POSS) was designed and prepared by introducing four polyhedral oligosilsesquioxane (POSS) nanocages to the TPE luminogen. The synthetic route of TPE-POSS is shown in Scheme 1. The potential roles of the TPE core and the POSS substituents are fluorescent synthon and triggering synthon, respectively. First, TPE core is a typical AIE luminogen, the variation of its aggregation-state may lead to a drastic fluorescence change. Second, POSS nanocage is composed of Si–O bonds, its collapse triggered by F^- can induce the aggregation of TPE luminogens, endowing TPE-POSS with precise turn-on detecting function.

2. Experimental section

2.1. Materials and measurement

Boron tribromide (BBr₃) was purchased from Aladdin and p-Aminopropylisobutyl POSS (AM0265) was purchased from Hybrid Plastics. All the other chemicals were purchased from Heowns Biochemical Technology Co. Ltd. without further purification. All solvents were purchased from Tianjin Jiangtian Chemical Reagents Co. Ltd. and used as received.

¹H, ¹³C and ²⁹Si NMR spectra were recorded on a Bruker AVANCE III spectrometer at room temperature. Mass spectroscopy was done on a FT-ICR-MS Solarix 7T Agilent 6540 instrument. Fourier transform infrared (FTIR) spectra was recorded on a PerkinElmer FTIR-100. The photoluminescence spectra were obtained from a Hitachi FL-2500 luminescence spectrometer. UV–vis absorption spectra of solution samples were measured on an Agilent Technologies Cary 300 UV/Vis spectrophotometer. A Malvern NanoZS instrument equipped with a 4 mW He–Ne



Fig. 1. (a) Fluorescence spectra of TPE-POSS in different H₂O/THF mixtures from 0% to 90%. ([TPE-POSS] = 10 μ M; λ_{exc} = 340 nm). (b) Plot of *I*/*I*₀ versus water fraction of THF/H₂O mixture of TPE-POSS, where *I*₀ represents the fluorescence intensity in pure THF solution. Inset: Photographs of TPE-POSS in THF/water mixtures (*f*_w = 0, 90%) taken under the illumination of a UV lamp.

laser (633 nm) was used to perform dynamic light scattering (DLS) measurements.

2.2. Synthesis and characterization

The intermediates 1, 2, 3 and 4 were synthesized according to the literature [33].

Synthesis of TPE-POSS: The mixture of 4 (300 mg, 0.32 mmol), p-Aminopropylisobutyl POSS (1.69 g, 1.93 mmol) and hydroxybenzotriazole (HOBT) (337 mg, 2.49 mmol) was completely dissolved in anhydrous tetrahydrofuran (20 mL) and then cooled in ice bath. Afterward, diisopropylcarbodiimide (DIPC) (0.5 mL, 3.23 mmol) was added to the above solution with stirring. The reaction was moved to refluxing at 65 °C for 12 h. Then the mixture was filtered and evaporated to remove the solvent. The crude product was obtained by crystallization with dichloromethane. The crude product was purified by column chromatography (silica gel, petroleum ether/dichloromethane = 1:3, V/ V) to give a white solid (0.94 g, yield 67%). The compound was characterized by ¹H NMR, ¹³C NMR, HRMS spectrum and FT-IR (Fig. S1-4). ¹H NMR (400 MHz, CDCl₃) δ (TMS, ppm): 7.78 (d, 8H, J = 8.20 Hz, Ar-H), 7.49 (d, 8H, J = 8.20 Hz, Ar-H), 6.97 (d, 8H, J = 8.68 Hz, Ar-H), 6.74 (d, 8H, J = 8.76 Hz, Ar-H), 6.14 (t, 4H, J = 5.56 Hz, NH), 5.05 (s, 8H, CH₂), 3.47 (m, 8H, CH₂), 1.88 (m, 28H, CH₂), 1.73 (m, 8H, CH₂), 0.97 (m, 168H, CH₃), 0.70 (m, 8H, CH₂), 0.63 (m, 56H, CH₂). ¹³C NMR

(100 MHz, CDCl₃) δ (TMS, ppm): 167.11, 156.89, 140.49, 138.50, 137.24, 134.45, 132.64, 127.35, 127.09, 113.96, 69.23, 42.32, 25.71, 25.69, 23.90, 23.85, 23.08, 22.49, 22.46, 9.51. FT-IR (KBr pellet) $\nu_{\rm max}$ in cm $^{-1}$: 3348, 2955, 2927, 2871, 1645, 1608, 1540, 1505, 1464, 1402, 1367, 1330, 1292, 1107, 956, 837, 744, 569, 486, 429. HRMS m/z: Calculated for C₁₈₂H₃₂₀N₄O₅₆Si₃₂ [M]⁺: 4359.2540, found [M]⁺: 4359.1484.

2.3. Stock solution preparation

The TPE-POSS was dissolved in THF solution with a concentration of 1×10^{-4} mol/L to prepare a stock solution. Moverover, the THF solutions (4 $\times 10^{-4}$ mol/L) of different anions like Cl⁻, Br⁻, I⁻, HSO₄, OH⁻, ClO₄, H₂PO₄, Ac⁻ and F⁻ were prepared. Different anion solutions were added to the probe stock solution. The working solution of the sensor and anions were diluting to the test concentration to execute the fluorescence spectrum test, and then the changes in fluorescence intensity were observed.

3. Results and discussion

3.1. Aggregation-induced emission properties

Regarding the AIE features of TPE derivatives, the photophysical



Fig. 2. (a) Changes in the emission spectra of TPE-POSS (10 μ M) in THF with different concentrations of fluoride ions (0–5 equiv.). (b) Plot of the fluorescence intensity (I/I₀) of TPE-POSS at 488 nm with different concentrations of fluoride ions. Inset: photographs of TPE-POSS in the absence and presence of F⁻ under UV light.



Fig. 3. Emission spectra changes of TPE-POSS upon the addition of various anions. $\lambda_{exc}=340$ nm. Inset: the optical images of TPE-POSS with the addition of various anions under UV irradiation. [TPE-POSS] = 10 μM ; [anions] = 40 $\mu M.$

properties of TPE-POSS were investigated in a mixture of THF/H2O with different water fractions (f_w) , in which THF is a good solvent and water is a poor solvent. As displayed in Fig. S5, the absorption of TPE-POSS increased dramatically with the addition of water, which is attributed to the scattering effect of in-situ-generated nanoaggregates [33]. Paralleling with the absorption changes, the emission intensity was gradually enhanced with obvious blue shift. As can be seen in Fig. 1a, when the water fraction (f_w) was less than 20%, the solution showed extremely weak emission because the molecule was in the dispersed state. With the water content increased, the fluorescence intensity showed significant enhancement. For instance, compared with the TPE-POSS sample in pure THF, the PL intensity of TPE-POSS in THF/H₂O mixture with $f_w =$ 90% was boosted with 19 folds whereas the emission wavelength was blue-shifted from 488 nm to 462 nm (Fig. 1b). The obvious blue shift of the fluorescence spectrum is due to the formation of aggregates. The internal movement of the molecule was restricted, which caused the molecule to a more distorted conformation, leading to a decrease in the conjugation length of the molecule and a blue shift in the spectrum [40]. All of these phenomena showed that TPE-POSS is a typical AIE material, which can also be seen from the inset photographs in Fig. 1b. Moreover, it was worth noting that there is almost no overlap between the fluorescence and the absorption spectra. This large Stokes shift helps the detection system, which can reduce the interference of excitation light scattering on fluorescence detection and improve the detection sensitivity.

3.2. Sensing mechanism studies of TPE-POSS to F⁻

The intriguing AIE effect and Si–O components of TPE-POSS prompt us to explore its potential application as fluorescence sensor for detecting fluoride ions. Fluorescence titration experiments were conducted to quantify the fluoride ions induced fluorescence intensity changes of TPE-POSS, using n-Bu₄NF as the fluoride source. As displayed in Fig. 2a and b, with the gradual addition of F⁻ (0–50 μ M) to TPE-POSS (10 μ M) solution, the emission intensity at 488 nm increased gradually and further stabilized. It can be observed a 7.5-fold turn-on ratio when 40 μ M F⁻ was added. The inset in Fig. 2b shows clearly the change of fluorescent color after adding fluoride ions under the ultraviolet lamp. The increase in fluorescence intensity at 488 nm had a good linear relationship (R² = 0.99) with the F⁻ concentration in the range of 5–35 μ M (Fig. 2b). According to the definition by IUPAC (L_D = KS_b/m) [41], the detection limit was calculated to be 1.66 \times 10⁻⁷ M. Moreover, the



Fig. 4. Emission intensity of TPE-POSS in the mixture of F^- and other anions. [TPE-POSS] = 10 $\mu M,$ [anions] = 40 $\mu M.$

TPE-POSS sensor has a good response speed to F⁻ (Fig. S6). It should be noted that TPE-POSS is not soluble in water, but it shows turn-on response to F⁻ in the THF-H₂O mixed solution (v:v = 1:1) (Fig. S7). According to the above results, the TPE-POSS sensor exhibits turn-on fluorescence response to fluoride ions, which avoids the shortcomings of turn-off sensors that are easily interfered by external factors.

Therefore, the selectivity of TPE-POSS sensor to anions was studied. The PL spectra were done after adding tetrabutylammonium salts of Cl⁻, Br⁻, I⁻, HSO⁻₄, OH⁻, ClO⁻₄, H₂PO⁻₄, Ac⁻ and F⁻ to the THF solution of TPE-POSS. As can be seen in Fig. 3, the fluorescence spectra displayed an obvious distinction of fluoride ions from other anions. Moreover, the inset photographs in Fig. 3 show that only the solution with fluoride ion added has fluorescence turn-on response, and the appearance of green fluorescence can be detected easily by naked-eye. The visible color changes did not observed with the addition of other anions. The above phenomena show that the sensor molecule has excellent specific recognition performance for fluoride ions.

In order to effectively and specifically the sensing fluoride ions, it is an essential requirement that other coexisting anions do not interfere with it. The competition experiments were carried out by measuring the fluorescence intensity of the sensor TPE-POSS in the presence of the interfering anions and fluoride ion. As displayed in Fig. 4, the competitive experiments show that there no significant changes in the fluorescence response of TPE-POSS toward F^- with the present of other anions, which indicates that the sensor TPE-POSS can effectively detect fluoride ions even in the presence of other competing anions. Therefore, there is no doubt that TPE-POSS can be used as a potentially highly selective sensor for detecting F^- .

3.3. Sensing mechanism studies of TPE-POSS to F

In order to elucidate the sensing mechanism between TPE-POSS and fluoride ions. ²⁹Si NMR spectra and dynamic light scattering (DLS) measurements were performed. It was found that the peak attributed to the POSS nanocages disappeared after reacting with F⁻ (Fig. 5a), which demonstrated the structural destruction of POSS nanocage. And DLS measurements further showed that the average size of TPE-POSS (10 μ M) in the THF solution increased from 22 nm to 142 nm after 4 equiv. fluoride ions added (Fig. 5b). These results indicated that POSS nanoparticles collapse with the addition of F⁻, which led to the aggregation of TPE cores and further promoted the fluorescence enhancement. In the light of the above results, it can be reasonably inferred that the reaction mechanism of POSS collapse led to TPE-POSS with excellent sensing performance (Scheme 2).



Fig. 5. (a) 29 Si NMR spectra of TPE-POSS in CDCl₃ (upper) and the solute after the addition of 4 equiv. TBAF (below) in CDCl₃. (b) DLS size distribution of TPE-POSS (10 μ M) before and after adding F⁻ (40 μ M).



Scheme 2. Schematic illustration of the sensing mechanism of TPE-POSS toward F⁻.

4. Conclusions

In conclusion, a novel fluorescent sensor based on TPE derivative connected to POSS nanocages (TPE-POSS) was successfully designed and synthesized. The POSS substituents can collapse due to the Si–O bond cleavage with the present of F⁻. The POSS nanocages collapse then trigger the AIE activity of TPE luminogens, endowing TPE-POSS with excellent turn-on detecting ability, high selectivity and sensitivity, with a detection limit as low as 1.66×10^{-7} M. The strategy outlined in this work should be applicable to design other turn-on mode fluorescent sensor by POSS nanocages and AIE effect.

Author contribution statement

Yu-Ting Zeng: Conceptualization, Methodology, Validation, Investigation, Writing-Original Draft, Visualization. Si-Yu Gao: Investigation, Validation. Kaspars Traskovskis: Investigation. Baoxiang Gao: Investigation. Xiang-Kui Ren: Conceptualization, Investigation, Writing-Review & Editing, Supervision, Methodology, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (Grant No. 21875157 and 21304069), the open foundation of State Key Laboratory of Chemical Engineering (No. SKL-ChE-20B04) and the research fund program of Guangdong Provincial Key Laboratory of Functional and Intelligent Hybrid Materials and Devices (No. 2019B121203003).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dyepig.2021.109491.

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