

A New Piezochromic Fluorescence and Aggregation-Induced Emission Compound Containing Tetraphenylethylene and Triphenylamine Moieties with Morphology-alterable Property

Xie Zhou · Hai-yin Li · Zhen-guo Chi · Bing-jia Xu ·
Xi-qi Zhang · Yi Zhang · Si-wei Liu · Jia-rui Xu

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Abstract A novel piezochromic fluorescent (PCF) compound with aggregation-induced emission (AIE) effect and morphology-alterable emission property was developed. The amorphous and crystalline aggregates were obtained, and their spectroscopic properties and morphological structures were reversibly and repeatedly exhibited upon pressing (fuming) or annealing. The piezochromic fluorescent nature was generated through crystalline-amorphous phase transformation. It was proposed that AIE compounds existing a twisted propeller-shaped conformation will exhibit PCF activity. The common relationship between AIE and PCF established will guide researchers in identifying and synthesizing more piezochromic fluorescent materials.

Keywords Piezochromic fluorescence · Morphology-alterable emission · Phase transformation · Tetraphenylethylene · Aggregation-induced emission

X. Zhou
School of Pharmaceutical Sciences, Sun Yat-sen University,
Guangzhou 510275, China

H.-y. Li · Z.-g. Chi (✉) · B.-j. Xu · X.-q. Zhang · Y. Zhang ·
S.-w. Liu · J.-r. Xu (✉)
PCFM Lab, DSAPM Lab, KLGHEI of Environment and Energy
Chemistry, FCM Institute, State Key Laboratory of Optoelectronic
Materials and Technologies, School of Chemistry and Chemical
Engineering, Sun Yat-sen University,
Guangzhou 510275, China
e-mail: chizhg@mail.sysu.edu.cn
e-mail: xjr@mail.sysu.edu.cn

Introduction

The modification or alteration of molecular chemical structures is the most common approach for controlling fluorescence properties. However, for the dynamic control of solid state fluorescence with high efficiency, reversibility, and reproducibility, effective materials are quite limited because most chemical reactions in the solid state involve insufficient conversion and irreversible reactions, or result in loss of fluorescence capability [1, 2]. To overcome this problem, a very attractive approach is to control the solid fluorescence properties dynamically by altering the mode of solid state molecular stacking without changing the chemical structure of the constituting molecules. If the fluorescent color change of a material is based on a pressure-dependent mode of molecular stacking, it is called piezochromic fluorescent material. Many piezochromic materials based on the change of absorption characteristics under pressure have been reported [3–5]. As fluorescence can be detected with high sensitivity, materials exhibiting piezochromic fluorescence have a wide variety of applications such as optical recording and strain- or pressure-sensing systems. However, piezochromic fluorescent materials are exceedingly rare [6–10]. Recently, a lot of fluorescent compounds with AIE effect have been synthesized in our laboratory, and it has been very interesting to find that some of the AIE compounds are piezochromic fluorescent materials [11–15]. AIE materials are an important class of anti-aggregation-caused-quenching materials first reported by Tang in 2001 [16] and have attracted considerable research attention because of their potential

application in various fields such as organic light-emitting devices and chemosensors [17–27]. To the best of our knowledge, except our laboratory, there are only two research groups reported their studies on piezochromic fluorescent material with AIE effect [28, 29]. In this article, we present a new PCF-AIE compound (TPA-TPE) as an example. It is found that the piezochromic fluorescent nature is generated through crystalline-amorphous phase transformation. It was proposed that AIE compounds existing a twisted propeller-shaped conformation will exhibit PCF activity.

Experimental

Materials and Methods

Diphenylmethane, 4,4'-dibromobenzophenone, *n*-butyllithium in hexane (2.2 M), tetrakis(triphenylphosphine) palladium(0), tetrabutyl ammonium bromide (TBAB), and *p*-toluenesulphonic acid purchased from Alfa Aesar were used as received. 4-(diphenylamino)phenylboronic acid was obtained from Zhenjiang Haitong Chemical Industry Co., Ltd. (China). All other reagents and solvents were purchased as analytical grade from Guangzhou Dongzheng Company (China) and used without further purification.

Proton and carbon nuclear magnetic resonance ($^1\text{H-NMR}$ and $^{13}\text{C-NMR}$) spectra were measured on a Mercury-Plus 300 spectrometer [CDCl_3 , tetramethylsilane (TMS) as the internal standard]. High resolution mass spectra (HRMS) were measured on a Thermo MAT95XP-HRMS spectrometer. Elemental analyses (EA) were performed with an Elementar Vario EL elemental analyzer. Photoluminescence spectra (PL) were measured on a Shimadzu RF-5301pc spectrometer with a slit width of 1.5 nm for both excitation and emission. UV-vis absorption spectra (UV) were recorded on a Hitachi UV-vis spectrophotometer (U-3900). Differential scanning calorimetry (DSC) curves were obtained with a NETZSCH thermal analyzer (DSC 204 F1) at a heating rate of 10 °C/min under N_2 atmosphere. Pressed samples were prepared by pressing the crystalline samples obtained from dichloromethane/*n*-hexane solution (1:4, v/v) in an IR pellet press at 1,500 psi for 1 min. Annealing experiments were done on a hot-stage with automatic temperature control system. The water-DMF mixtures with different water fractions were prepared by slowly adding distilled water into the DMF solution of samples under ultrasound at room temperature. For example, a 70% water fraction mixture was prepared in a volumetric flask by adding 7 mL distilled water into 3 mL DMF solution of the sample. The concentrations of all samples were adjusted to 10 μM after adding distilled water.

Synthesis of Br-TPE

A 2.2 M solution of *n*-butyllithium in hexane (20.0 mmol, 9.1 mL) was added to a solution of diphenylmethane (3.36 g, 20.0 mmol) in anhydrous tetrahydrofuran (50 mL) at 0 °C under an argon atmosphere. After stirring for 1 h at that temperature, the bis(4-bromophenyl)methanone (5.44 g, 16.0 mmol) was added and the reaction mixture was stirred for 10 h allowing the temperature to rise gradually to room temperature. Then the reaction was quenched with an aqueous solution of ammonium chloride and the mixture was extracted with dichloromethane. The organic layer was evaporated after drying with anhydrous sodium sulfate and the resulting crude alcohol was dissolved in toluene (80 mL). The *p*-toluenesulphonic acid (0.68 g, 3.6 mmol) was added, and the mixture was refluxed overnight and cooled to room temperature. The mixture was evaporated and the crude product was purified by silica gel column chromatography using *n*-hexane as eluent to yield a light yellow powder (5.70 g, 73%). $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm): 6.82–6.90 (m, 4H), 6.95–7.05 (s, 4H), 7.06–7.16 (s, 6H), 7.18–7.27 (m, 4H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ (ppm): 120.93, 127.10, 128.10, 131.30, 133.20, 138.55, 142.38, 143.40; FT-IR (K.Br) ν (cm^{-1}): 3053, 3021, 2354, 1590, 1480, 1434, 1382, 1065, 1007, 826, 761, 696, 482; HRMS (EI), m/z : 490($[\text{M}]^+$, calcd for $\text{C}_{26}\text{H}_{18}\text{Br}_2$, 490); Anal. calcd for $\text{C}_{26}\text{H}_{18}\text{Br}_2$: C 63.70, H 3.70; found: C 63.65, H 3.72.

Synthesis of TPA-TPE

Br-TPE (0.245 g, 0.5 mmol) and 4-(diphenylamino)phenylboronic acid (0.289 g, 1.0 mmol) in toluene (15 mL), 2 M aqueous K_2CO_3 solution (1.5 mL) and 5 drops of TBAB were added. The mixture was stirred for 40 min under an argon atmosphere at room temperature. Then the $\text{Pd}(\text{PPh}_3)_4$ catalyst (catalytic amount) was added and the reaction mixture was stirred at 80 °C for 16 h. After cooling to room temperature, the product was concentrated and purified by silica gel column chromatography with CH_2Cl_2 : *n*-hexane (v: v, 1: 4) to afford white powder (0.340 g, yield 83%). $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm): 6.98–7.17 (m, 29H), 7.21–7.30 (m, 9H), 7.30–7.36 (d, 4H), 7.42–7.49 (d, 4H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ (ppm): 123.15, 124.25, 124.60, 125.95, 126.65, 127.60, 127.95, 129.40, 131.60, 132.05, 138.45, 140.40, 141.30, 142.65, 144.05, 147.80; FT-IR (KBr) ν (cm^{-1}): 3033, 2361, 1590, 1486, 1318, 1279, 813, 748, 696, 508; HRMS (EI), m/z : 818 ($[\text{M}]^+$, calcd for $\text{C}_{62}\text{H}_{46}\text{N}_2$, 818); Anal. calcd for $\text{C}_{62}\text{H}_{46}\text{N}_2$: C 90.92, H 5.66, N 3.42; found: C 90.89, H 5.63, N 3.47.

Results and Discussion

Synthesis

The synthetic route for the desired compound TPA-TPE is illustrated in Scheme 1. Suzuki coupling of 4,4'-(2,2-diphenylethene-1,1-diyl)bis(bromobenzene) (Br-TPE) with 4-(diphenylamino)phenylboronic acid provided the target compound with 83% yield. The synthesis of Br-TPE started with diphenylmethane and used two steps. The hydroxy intermediate was not purified further and could be used directly in the next step. After the dehydration reaction of hydroxy intermediate in the presence of *p*-toluenesulphonic acid, the bromide intermediate Br-TPE was obtained. The molecular structures of the target compound and intermediate were confirmed by nuclear magnetic resonance ($^1\text{H-NMR}$, $^{13}\text{C-NMR}$), Fourier transform infrared spectroscopy, elementary analysis, and high resolution mass spectrometry.

Morphology-Alterable and PCF Properties

To investigate morphology-alterable emission and piezochromic fluorescent properties, we carried out a series of experiments as schematically illustrated in Fig. 1.

When the solutions of the compound in different solvents were evaporated to dryness with a vacuum evaporator, two different aggregates were obtained: amorphous aggregate from dichloromethane and crystalline aggregate from dichloromethane/*n*-hexane mixed solvent (1:4, v/v).

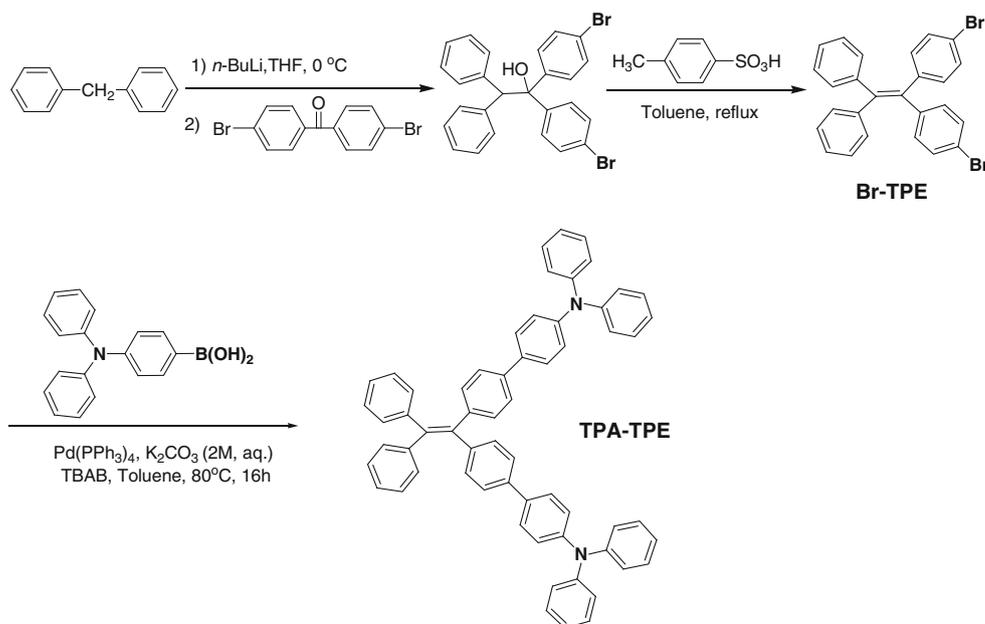
Figures 2, 3 and 4 show wide-angle X-ray diffractograms (WAXD), photoluminescence (PL) spectra and

differential scanning calorimetry (DSC) curves of the samples: (a) obtained from dichloromethane; (b) obtained from dichloromethane/*n*-hexane; (c) melt quenched to room temperature; (d) crystalline sample was fumed with dichloromethane for 12 h; (e) crystalline sample was pressed at 1,500 psi for 1 min; (f) fumed sample was annealed at 180 °C for 5 min; (g) pressed sample was annealed at 180 °C for 5 min.

The crystalline and amorphous nature of the samples was verified by their WAXD, where that of the former exhibited many sharp reflection peaks (Fig. 2, Curve b), and that of the latter gave only a weak, broad, and diffused peak (Fig. 3, Curve a). The amorphous aggregate was yellowish green in appearance, with a strong green emission at 497 nm (Fig. 4, Curve a), whereas the crystalline aggregate was white, with a strong blue emission at 450 nm (Fig. 3, Curve b). The DSC heating curve of the amorphous sample showed a cold-crystallization peak around 134 °C, except for a melting peak at around 190 °C (Fig. 4, Curve a). The sizes of the two peaks were very close, which means the sample is amorphous. The crystal corresponding to the melting peak was completely generated through cold-crystallization. However, the crystalline sample only showed a sharp melting peak at ~200 °C, with no cold-crystallization peak (Fig. 5, Curve b). If the amorphous sample is annealed at 180 °C for 5 min, it will revert to the crystalline state.

When the crystalline aggregate samples were treated in three different conditions, namely, (1) heated to the melted state then quenched by liquid nitrogen to room temperature, (2) fumed with dichloromethane for 12 h, and (3) pressed for 1 min by 1,500 psi, they all reverted to the amorphous

Scheme 1 Synthetic route of the compound



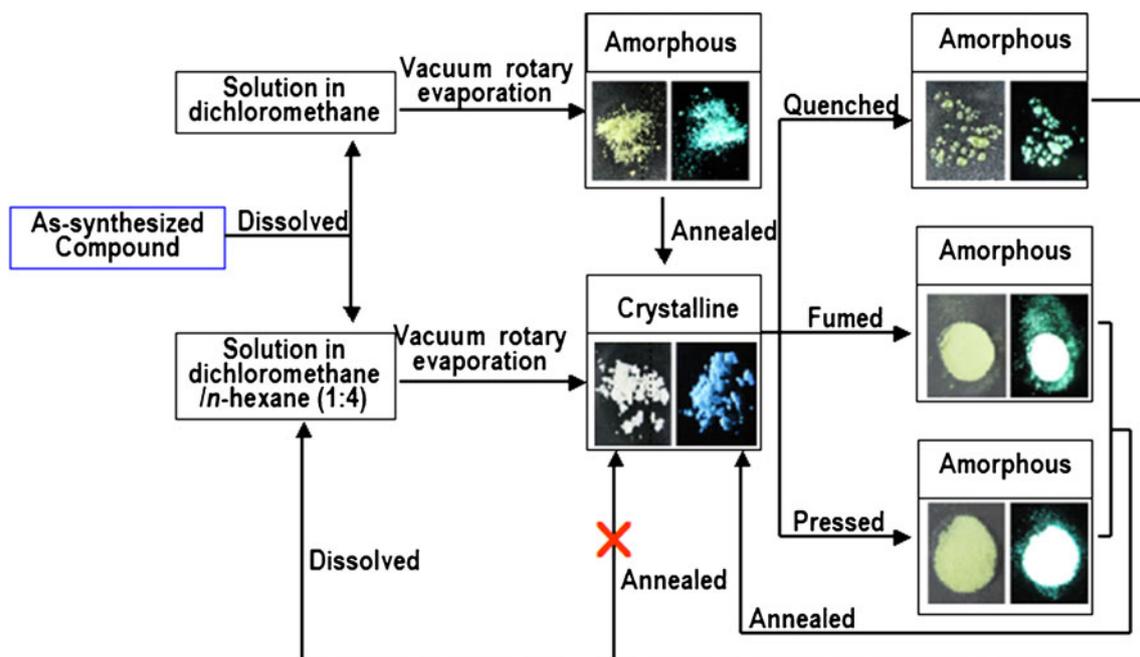


Fig. 1 A schematic representation of treatment procedures. The pictures in each group were taken at room temperature under (left) natural and (right) UV lights

states. These results were also confirmed by WAXD, which only generated diffused peaks (Fig. 3, curves c, d, and e). The PL spectra of the obtained samples red-shifted to ~ 500 nm, which were very close to the amorphous sample obtained from the dichloromethane solution. The result obtained from treatment (3) indicates that the compound has significant piezochromic property.

The piezochromic behavior of the compound may be attributed to its twisted structure (Fig. 5). Reported AIE

compounds such as typical triphenylethylene, tetraphenylethylene, silole, and cyano distyrylbenzene derivatives have one common structural feature: multiple phenyl peripheries are linked to an olefinic core via rotatable C-C single bonds to form AIE moiety. The steric effect causes the AIE moieties or the molecules to take a twisted propeller-shaped conformation, making it difficult for them to assume a dense packing structure. Thus, the crystals have low lattice energy and are easily crashed by external pressure. This “crash” may be

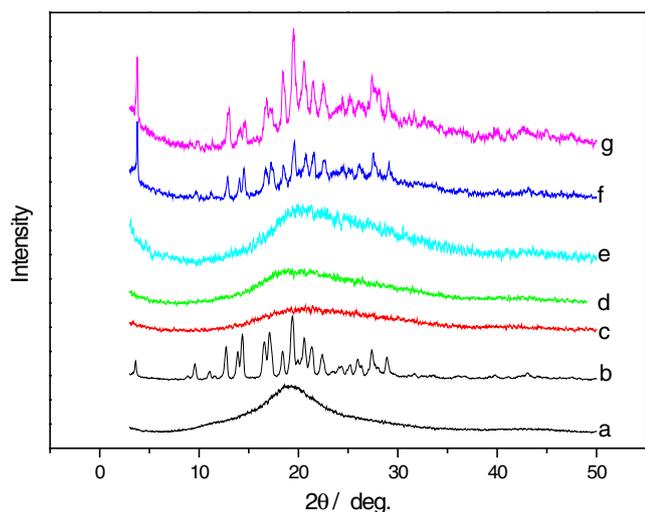


Fig. 2 WAXD patterns of the TPA-TPE samples obtained under different conditions

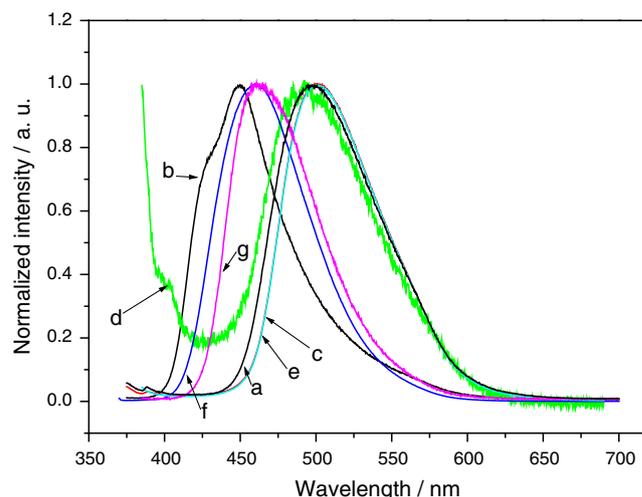


Fig. 3 PL spectra of the TPA-TPE samples obtained under different conditions

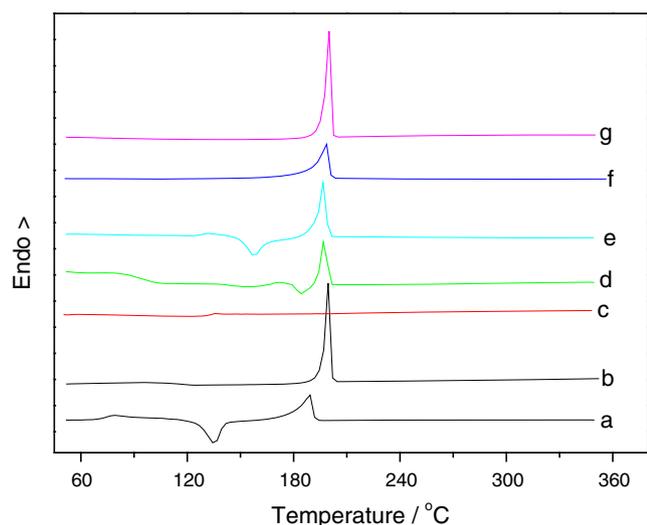


Fig. 4 DSC heating curves of the TPA-TPE samples obtained under different conditions

triggered by planarization of molecular conformation under external pressure (“pressing flat”) and should result in the generation of piezochromic fluorescent behavior. According to the hypothesis, we try pressing or grinding more than 30 AIE compounds synthesized in our laboratory, and as expected, almost all the compounds exhibit piezochromic fluorescent effect. We believe that the common relationship between AIE and PCF will guide researchers in identifying and synthesizing more piezochromic fluorescent materials.

The DSC heating curves of the three treated samples exhibited results which were considerably different from one another (Fig. 4, curves c, d, and e). The quenched sample exhibited only a glass transition at ~ 134 °C and no cold-crystallization peak; the fumed sample showed a cold-crystallization peak at around 184 °C followed by a melting peak at 197 °C; and the pressed sample showed a cold-

Fig. 5 The lowest-energy conformer optimized using the MM2 force field for TPA-TPE

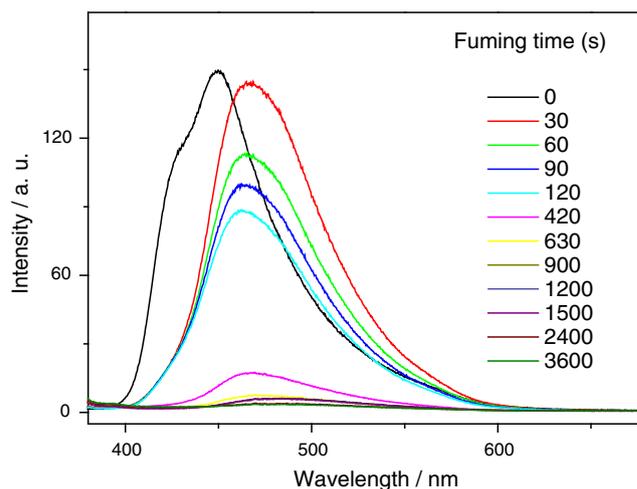
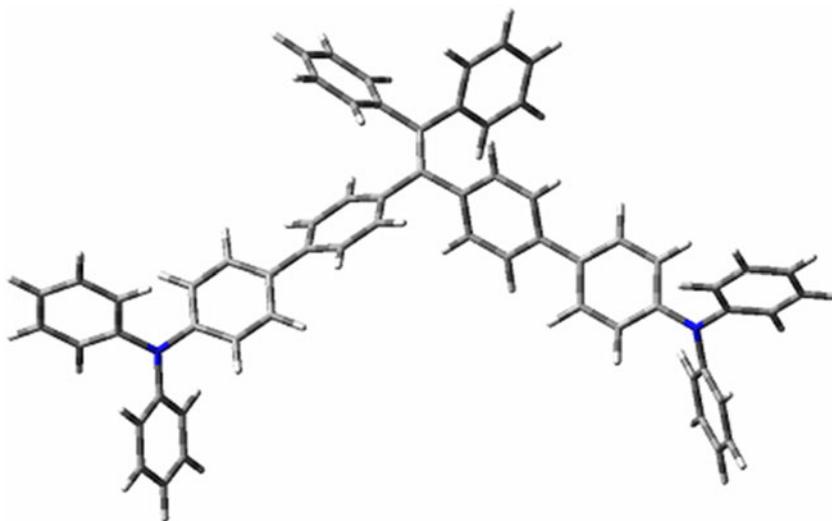


Fig. 6 PL spectra of the crystalline sample under fuming with dichloromethane

crystallization peak at around 157 °C and a melting peak at around 197 °C. The melting peak temperatures of the fumed and pressed samples were the same. However, the former exhibited a 27 °C higher cold-crystallization temperature than the pressed sample.

The amorphous samples obtained by fuming and pressing treatments, which can be reverted to the crystalline state by annealing at 180 °C for 5 min, exhibited good morphological reversibility. However, the amorphous sample obtained by melt quenching cannot be reverted to its crystalline state by the annealing method and has no cold-crystallization and melting transition peaks in heating DSC curve (Fig. 4 Curve c). The reversible behavior of the amorphous sample can be achieved by dissolving it in dichloromethane/n-hexane mixed solvent (1:4, v/v) and then evaporating the solution to dryness with a vacuum evaporator.

Figures 6 and 7 show the relationship of the PL spectra of the compound with dichloromethane fuming time. The PL peak intensity of the crystalline sample dramatically decreased within 420 s. Following this, the curve slowed down. The wavelength exhibited significant red-shifts from 450 to 493 nm. From 0 to 900 s, the wavelength quickly red-shifted with an increase in fuming time, and then it became flat. As the compound is AIE active, the permeation of a good solvent during fuming can loosen the packing of the molecules, leading to the increase in intramolecular rotations, the increase in non-emissive decay of excited state energy, and the decrease in the PL intensity.

To verify whether the chemical structure of TPA-TPE changed after heating, fuming, and pressing, $^1\text{H-NMR}$ experiments in CDCl_3 were conducted. Figure 8 shows that the $^1\text{H-NMR}$ spectra are the same, which indicates that the chemical structure did not change. This further implies that the different spectroscopic properties of TPA-TPE were caused by physical processes. In other words, the piezochromic fluorescent nature was generated through crystalline-amorphous phase transformation.

AIE Properties

To confirm whether a compound is AIE active, fluorescent behaviors are usually studied with a poor solvent added into a good one. The compound is insoluble in water, so increasing water fraction in the mixed solvent can change its existing form from a solution or well-dispersed state in pure DMF to aggregated particles in mixtures with high water content. The PL spectra of 10 μM TPA-TPE in water/DMF mixtures with different water contents are shown in Fig. 9. The figure shows that if the water fraction is less than 30%, the PL intensity is very weak. However, a dramatic enhancement in luminescence is observed for a water/DMF mixture with 30% water fraction. The PL

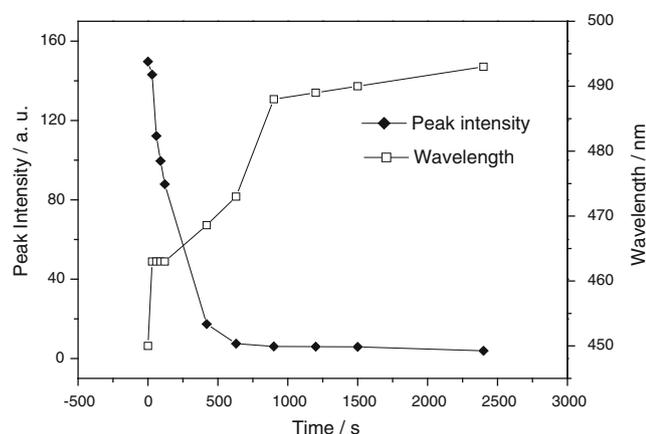


Fig. 7 PL peak intensity and wavelength versus fuming time with dichloromethane

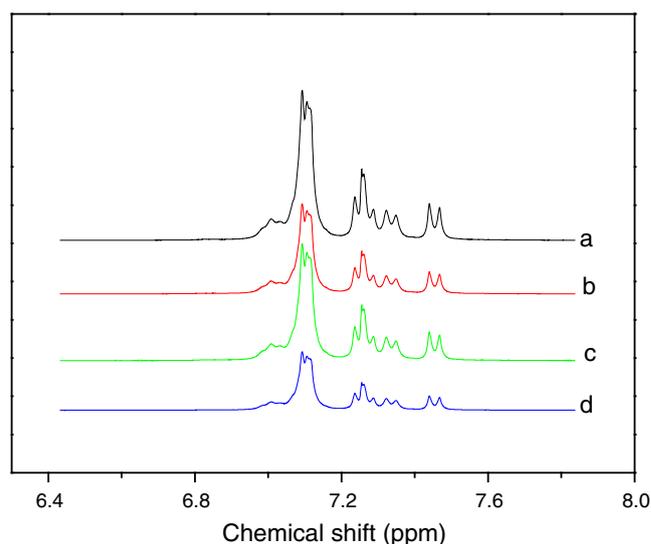


Fig. 8 $^1\text{H-NMR}$ of the TPA-TPE samples (in CDCl_3) **a** obtained from dichloromethane; **b** melt quenched to room temperature; **c** crystalline sample was fumed with dichloromethane for 12 h; and **d** crystalline sample was pressed at 1,500 psi for 1 min

intensity (a.u.) in pure DMF is only 3.6. However, in water/DMF mixture with 30% water fraction, the PL intensity is elevated to 186.9, which is about 52 times higher than that of the pure DMF solution. This increase in fluorescence intensity is attributed to an AIE effect caused by the formation of molecular aggregates, in which the restriction in intramolecular rotations leads to increased fluorescent emission. Emission images of TPA-TPE in pure DMF and 30% and 90% water fraction mixtures under 365 nm UV illumination are shown in Fig. 1 (inset). The compounds in 30% and 90% water fractions of water/DMF mixtures

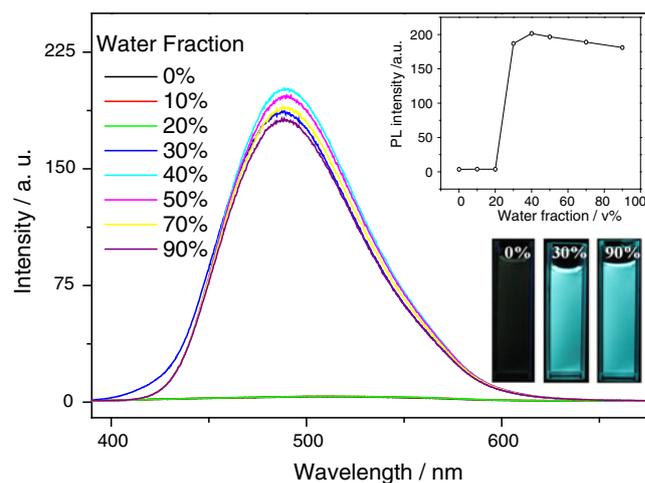


Fig. 9 PL spectra of the dilute solutions of TPA-TPE in water/DMF mixtures with different water fractions (10 μM ; excitation: 365 nm). The inset depicts changes in PL peak intensity (*up*) and emission images of TPA-TPE in pure DMF, and 30% and 90% water fraction mixtures under 365 nm UV illumination (10 μM) (*down*)

exhibit strong green emission. However, in pure DMF, the emission is very weak and virtually invisible. Thus, the compound has strong AIE activity. In other words, TPA-TPE has both aggregation-induced emission and piezochromic fluorescence characteristics and it has been called PAIE material in our laboratory.

Conclusions

A novel piezochromic compound with aggregation-induced emission effect and morphology-alterable emission property was developed. The amorphous and crystalline aggregates can be obtained by evaporation of different solutions. The emission properties of the novel piezochromic compound can reversibly and repeatedly switch the fluorescence upon pressing (fuming) or annealing due to crystalline-amorphous phase transformation. These excellent properties make the compound a promising candidate for stimuli-responsive, smart, and luminescent materials for information recording and light emitting device applications. It was proposed that AIE compounds existing a twisted propeller-shaped conformation will exhibit piezochromic fluorescent activity. The common structure-property relationship established will guide researchers in identifying and synthesizing more piezochromic fluorescent materials.

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