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Chemical routes to materials

Solid-state photochromism and acidochromism multifunctional materials constructed by tetraphenylethene and spiropyran

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ABSTRACT

Solid-state stimuli-responsive materials are attracting increasing attention due to their broad prospects in the field of functional materials, but there are still some challenges. We design and synthesize two efficient solid-state stimuli-responsive materials SP-TPEm (m = 4, 6) based on TPE covalent connection with *N*-position of spiropyran through different lengths of alkyl spacers. Both of them show aggregation-induced emission behaviors. Efficient solid-state photoswitching between SP-TPEm and their photoisomer MC-TPEm is achieved. Both of the two films show great stimulus-responsive properties, and the characteristic emission peak is at around 460 nm for TPE and 601 nm for MC isomer. Therefore, SP-TPE4 is applied as an anticounterfeiting ink due to its great solid-state photochromic characteristics. In addition, the solid-state films of SP-TPEm are acidochromic responsive. Moreover, the surface wettability of the SP-TPEm films can be adjusted by UV light. Hence, we provide a new strategy for building solid-state multifunctional materials.

Introduction

Stimuli-responsive functional materials have attracted abundant attention for their wide applications in optical data storage, chemical sensors, displays, security ink, anticounterfeiting, bioimaging, optical shutters and photoswitchable molecular devices [1–5]. Spiropyran (SP) [6–12] is one of the most widely studied and most intensive organic photoswitches which can undergo reversible structural isomerization between the two forms of colorless closed-ring (SP) and colored open-ring merocyanine (MC). The common structure of spiropyran is composed of two aromatic rings: benzopyran and indole, connected by a sp^3 hybrid spiro-C atom. The two aromatic rings are orthogonal to each other, and the closed-ring spiropyran molecule is unconjugated and colorless. When the SP is excited by ultraviolet radiation, heat or force, the C-O bond is

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cleaved and transforms to conjugated ring-opening and colored MC state; under white light, the open-ring system returns to the closed SP state. However, most of the reported fluorescent switches with SP units work only in solution and very few were reported in the solid state. Harada [13] reported that low temperature even to 80 K could realize the solid-state photochromism for SP, which is owing to the reduction of the MC to SP isomerization rate. Nevertheless, too low temperature restricts its practical applications.

Therefore, constructing efficient solid-state fluorescent switches about SP derivatives are still challenging and meanwhile are very important for multidimensional applications. The main battle for the solid-state photochromism of spiropyran is their strong intermolecular accumulation. Its isomerization requires a large amount of free volume to complete the response from SP to MC form. Recently, Qingkai Qi [14] reported spiropyran derivatives with a popular AIE molecule DSA exhibited highly efficient solid-state luminescent switching and were applied in optical anticounterfeiting and super-resolution imaging. The highly twisted and three-dimensional structure of AIE group DSA could reduce intermolecular interaction and offer enough free volume which is beneficial for SP to MC isomerization. XingSu [15] reported a high-contrast fluorescent light switch based on tetraphenylethene with multi-emissions. Recently, Zhen Wu [16] reported a molecule which was composed of two spiropyran moieties with a twisted TPE group through the 5-position of spiropyran and exhibited photochromism, acidochromism in the solid state. However, it demonstrates poor fluorescence emission.

Combined with the previous reports, we found that introducing AIE moiety into SP can not only realize solid-state fluorescent switching by changing its molecular stacking modes, but also improve fluorescence emission in the aggregation state. Tetraphenylethene (TPE) and its derivatives have attracted much attention in recent years because of its unique aggregation-induced emission (AIE) characteristics [17–26].

In this article, we covalently linked TPE with two SPs at the *N*-position through spacers to obtain compounds of SP-TPEm($m = 4,6$); here, TPE is an efficient AIE luminescent group and has a large twisted structure, which can provide enough free space for SP to MC isomerization. In addition, we expect that the connection of TPE and SP at the *N*-position of SP can induce large volume which can prevent intermolecular accumulation and is favorable for isomerization.

Experimental section

Materials

Commonly used reagents were purchased directly only if explained. 2,3,3-Trimethylindolenine, 2-bromoethanol, 5-nitrosalicylaldehyde, 1,4-dibromobutane and 1,6-dibromohexane were obtained from Energy Chemical, and DMF was purified and directly used by common methods.

Instrumentation

The instrumentation involved in this paper is illustrated in the supporting information.

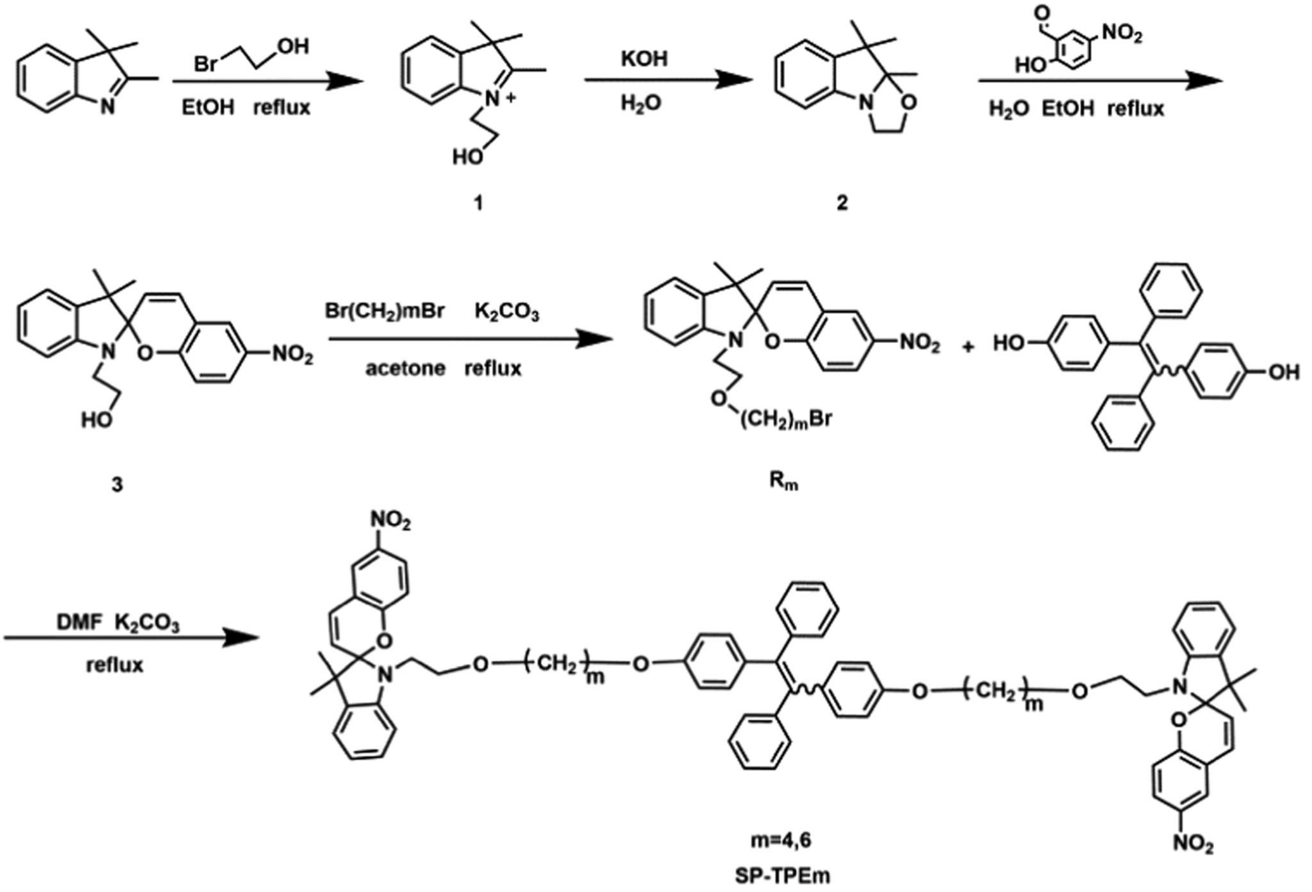
Synthesis

The synthetic route of SP-TPEm is shown in Scheme 1. The synthesis of SP-TPE4 and SP-TPE6 is similar, and the synthetic process and corresponding characterization of the intermediate compounds are illustrated in supporting information. Herein, we use R4 and SP-TPE4 as the example to illustrate the synthetic process. The wavy line of TPE represents the *cis* or *trans* structure. The detailed synthetic information is described as below.

1′-(2-(4-Bromobutoxy)ethyl)-3′,3′-dimethyl-6-nitrospiro[chromene-2,2′-indoline](compound R4)

The mixture of compound 3 (2.69 g, 7.6 mmol), 1,4-dibromobutane (2.49 g, 11.56 mmol), K_2CO_3(2.28 g 22.8 mmol) and 80 mL acetone were added into a 100 mL flask with three necks and then mixed and stirred at 60 °C overnight. When the reaction was accomplished, we refined the mixture by column chromatography and the eluent was ethyl acetate/petroleum ether (1:6, v/v); then, R4 was received as a yellow solid. Yield: 75%.

^{1}H NMR(400 MHz, $CDCl_3$), ppm: 8.35(d, 1H, $J = 2.8$ Hz, Ar–H), 8.15(dd, 1H, $J = 9.1$, 2.8 Hz, Ar–H), 7.20–7.09(m, 3H, Ar–H), 6.97–6.91(m, 2H, Ar–H), 6.82(d, 1H, $J = 7.8$ Hz,–C=CH–), 6.45(d, 1H,



Scheme 1 Synthetic route of the SP-TPEm.

J = 16.0 Hz, –CH=C–), 4.17(m, 2H, Br–CH2–), 3.93 & 3.89(m, 2H, N–C–CH2–), 3.76 & 3.56(m, 2H, N–CH2–), 3.42(m, 2H, N–C–C–O–CH2–), 1.90(m, 4H, $–CH_2–$), 1.28(s, 3H, CH_3), 1.18 (s 3H, CH_3).

The synthesis of compound SP-TPE4

Compound R4 (1.98 g, 4.06 mmol), TPE (0.49 g, 1.135 mmol), K_2CO_3 (0.49 g, 1.135 mmol) and 50 mL of dry DMF were added into a 100 mL flask, and then, the reaction was stirred at 100 °C for more than 24 h. When the reaction was finished, we refined it with silica gel column chromatography and the eluent of ethyl acetate/petroleum ether (1:3, v/v) was used. Afterward, a yellow solid of SP-TPE4 was obtained with the yield of 45%.

^{1}H NMR(400 MHz, $CDCl_3$), ppm:8.35(d,1H,*J* = 2.8 Hz,Ar–H), 8.15(dd, 1H,*J* = 9.1, 2.8 Hz, Ar–H), 7.19–6.88(m, 12H, Ar–H), 6.80(d, 1H,*J* = 7.8 Hz, –C=CH–), 6.61(m, 2H, Ar-HTPE), 6.45(d, 1H,*J* = 16.0 Hz, –CH=C–), 4.17(m, 2H, TPE–O–CH2–), 3.97(m, 2H, N–C–CH2–), 3.78 & 3.70(m, 2H, N–CH2–), 3.65 & 3.43(m, 2H, N–CC–O–CH2–), 2.05–1.95(m, 4H, $–CH_2–$), 1.45(s, 3H, CH_3), 1.18 (s 3H, CH_3).

^{13}C NMR ($CDCl_3$) δ (ppm): 160.96 (SP C–O), 157.17 (TPE C–O), 150.51(SP C),144.13 (TPE C–C=C–), 141.23 (TPE–C=C–), 136.49, 136.39, 131.33,127.53, 126.16, 125.56, 113.50, 113.41(TPE C), 141.31, 139./57, 139.53, 132.53, 130.05, 127.63, 126.61, 125.67, 124.62, 123.14, 122.37, 121.70, 112.01, 111.20, 109.77(SP C),68.71(SP C–O CH_2), 66.93(N C-O CH_2), 63.61(TPE C-O CH_2), 50.10, 47.89, 28.45, 25.90(CH_2), 20.38(CH_3).

MALDI-TOF (*m*/*z*): Calcd for C74H72N4O10, 1177.38; Found 1177.35.

Results and discussion

Synthesis

TPEm were successfully synthesized by several steps according to Scheme 1. Compound 3 was prepared according to the literature [27], and Rm and TPEm were prepared by etherification reaction on the basis of literature [28]. The structure properties of the intermediates and targets were identified by ^{1}H NMR, ^{13}C NMR and MALDI TOF MS. Specific synthetic steps and characterization results are included in the supporting information (Figure S1).

AIE properties

Solutions with concentration of 10^{-5} mol/L were prepared with different ratios of THF/H_2O, and their AIE properties were analyzed by fluorescent spectroscopy. As shown in Fig. 1, the THF solution of SP-TPE4 exhibits a low emission at 410 nm and 487 nm. As the water content increased, the emission intensity became stronger. In particular, when the water content was more than 90%, the luminous intensity at 487 nm increased rapidly which is typical AIE behavior. SP-TPE6 exhibited similar AIE behavior with emission intensity doubled as shown in supporting information (Figure S2).

Photoswitching properties in solution

We investigated the photoswitching properties of the SP-TPE4 and SP-TPE6 in THF solution as shown in Fig. 2. Obviously, the maximum absorption bands at UV region were ascribed to the $\pi - \pi^*$ electronic transition of indoline and chromene units. When the solutions were irradiated by 365 nm LED lamp for 5 min, a new broad absorption band at around 590 nm for SP-TPE4 and 594 nm for SP-TPE6 appeared which were attributed to the open-ring form with extended conjugation, and the solution changed from colorless to deep blue. Accordingly, as shown in Fig. 2b and d, the emission bands centered at 503 nm for SP-TPE4 and around 497 nm for SP-TPE6 excited at 400 nm decreased after irradiation, which were ascribed to the emission of closed SP moiety. In addition, the new emission bands peaked at 711 nm (ex: 400 nm) for SP-TPE4 and 713 nm (ex: 400 nm) for SP-TPE6 emerged and gradually intensified after irradiation. These two new emission bands were assigned to the open-ring form MC isomers which possess longer conjugation length than their close-ring form SP isomers, therefore illustrated the photoswitching properties of SP-TPEm in solutions.

Photochromic properties in the solid state

Further, we explored the absorption spectra of SP-TPEm in the solid state. The films of SP-TPE4 and SP-TPE6 were prepared by drop casting. First, the film

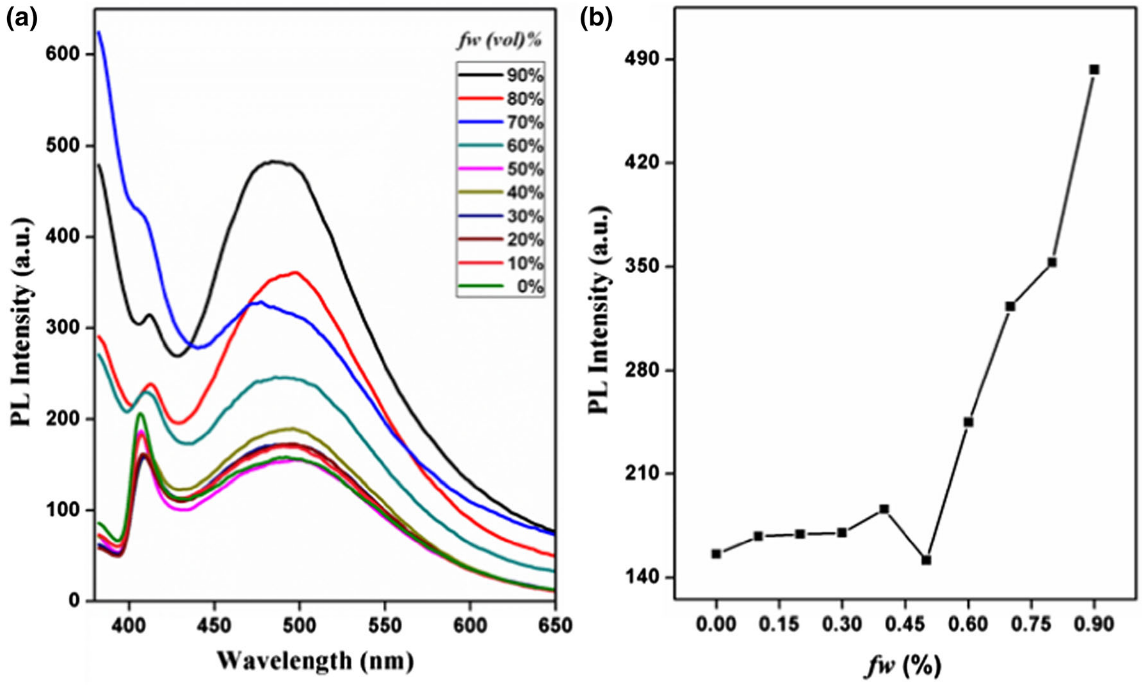


Figure 1 **a** Luminescent spectra of SP-TPE4 in THF/H_2O with different water contents. (λ_{ex} = 360 nm) **b** Emission intensity of SP-TPE4 related to water content at 487 nm.

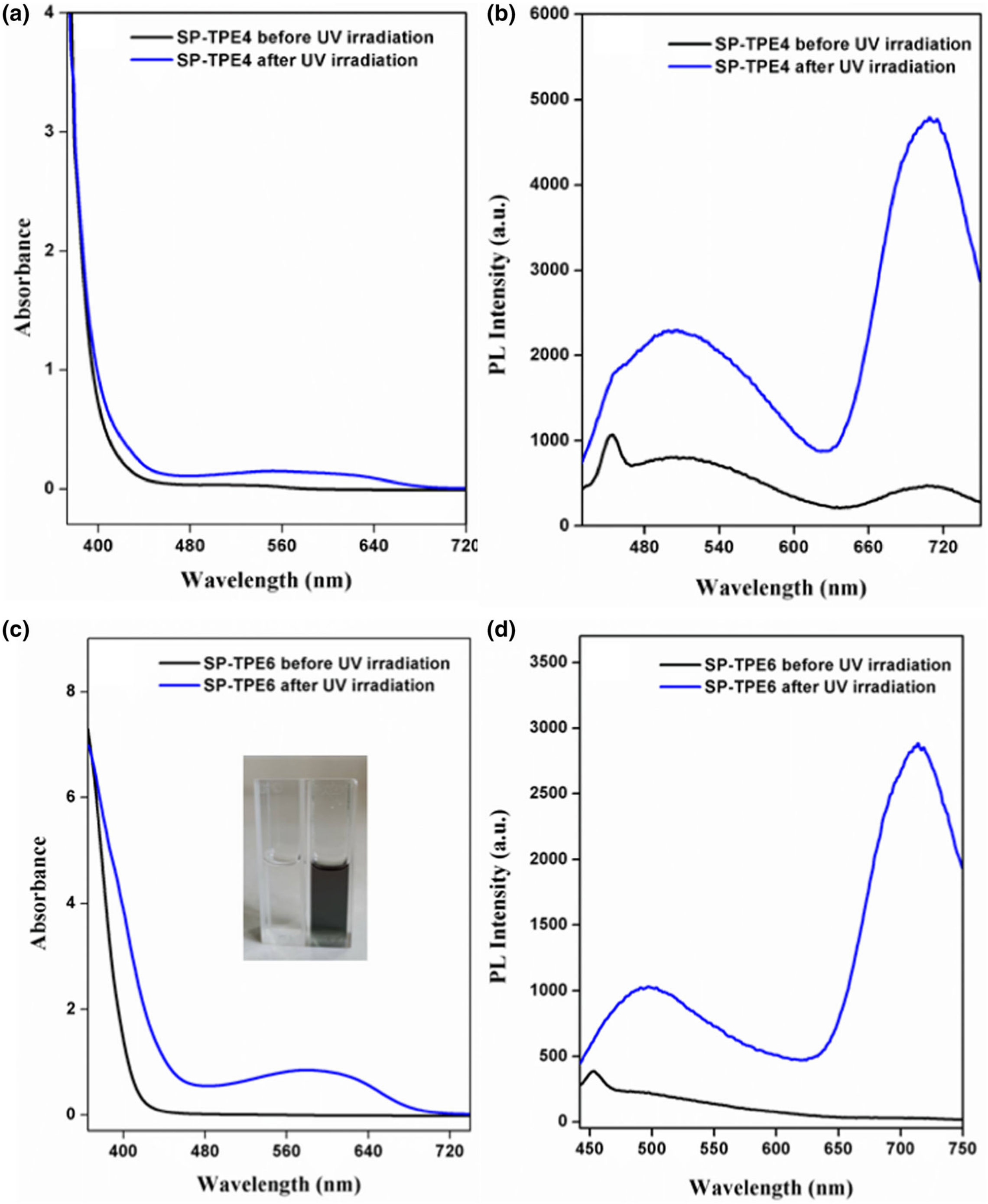


Figure 2 **a** UV−Vis absorption spectra for SP-TPE4, **b** PL spectra for SP-TPE4 (ex: 400 nm), **c** UV−Vis absorption spectra for SP-TPE6, **d** PL spectra for SP-TPE6 (ex: 400 nm).

absorption spectra of SP-TPE4 and SP-TPE6 were investigated as shown in Fig. 3a and c. Obviously, the broad absorption at UV region for SP-TPE$_4$ was ascribed to the $\pi - \pi^*$ electronic transition of indoline, chromene and TPE groups and decreased after UV irradiation. When the films were irradiated by 365 nm LED lamp for 5 min, a new absorption band at 569 nm for SP-TPE4 and 573 nm for SP-TPE6 appeared which were attributed to the open-ring MC form with extended π-conjugation. Second, the film fluorescent spectra of SP-TPEm were investigated as shown in Fig. 3b. Obviously, the emission of the TPE was at around 460 nm, and the emission bands at 601 nm for ring-opening form of SP-TPE4 increased after UV irradiation. In addition, the emission of SP-TPE6 in the solid state was also detected and exhibited similar properties with SP-TPE4 as shown in Fig. 3d. Therefore, both of the two compounds show great stimulus-responsive characteristics. In addition, the powder of SP-TPE4 was irradiated by a handheld LED lamp at 365 nm as shown in Fig. 3f. After the UV irradiation, the color of SP-TPE4 powder changed from light orange to brownish red, which exhibited efficient transformation from SP-TPE4 to MC-TPE4. In addition, the transformation exhibited good stability. The color of SP-TPE4 in film state could be maintained for more than three days and hardly fade in the dark. We investigated the molecular stacking structure by the X-ray diffraction (XRD) analysis as shown in Fig. 3e. In the wide-angle area, the diffraction peaks are broad and locate at $2\theta = 19.94$ for SP-TPE4 and 21.01 for SP-TPE6, corresponding to d-spacings of 4.45 and 4.22 Å, respectively, which indicate that both of the solid powders are amorphous and the molecules are loosely accumulated which is beneficial to the transformation from SP to MC.

We conducted chemical calculations using B3LYP/6-31G(d) basis set to explore the mechanism. From the optimized structure of SP-TPE4 (Fig. 3g), we can see that although there are alkyl spacers, the molecule still adopts a highly nonplanar molecular structure. In addition, because TPE and SP are connected by the *N*-position of SP, both of them demonstrate large space volume and are beneficial for the photoisomerization of SP.

In view of the good solid-state photochromic properties of SP-TPEm, we applied SP-TPE4 to anticounterfeit ink as shown in Fig. 3f. Therefore, we printed a word of XTU onto a filter paper and then the SP-TPE4 solution was used as ink and dropped into the word. After solvent evaporation and an entire drying at room temperature, we observed colorless word of XTU on the paper. Then, the printed filter paper was exposed to a 365 nm handheld lamp and immediately the color changed to orange. This color change can be easily distinguished by the human eye. In addition, the orange color after irradiation can maintain more than 3 days in the dark room and can be restored to its initial state under visible light conditions. It is thus clear that SP-TPE4 is a good anticounterfeiting material.

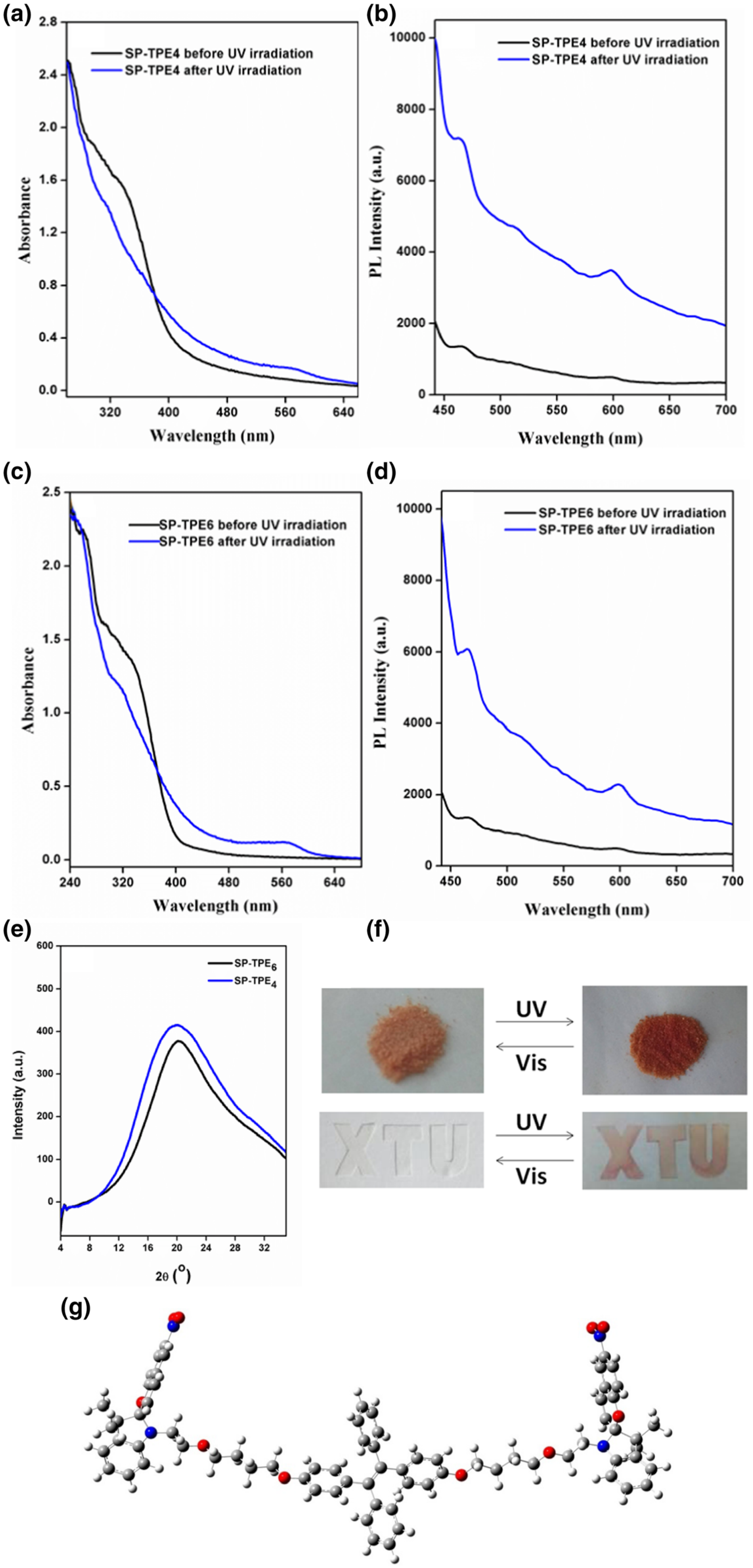
Response to acid and amine vapor

It has been reported [27, 29] that SP was acidochromic, when acid was added, the SP protonated and produced a yellow MCH^+ species. Therefore, we used the same method to make the printed filter paper of XTU and then it was exposed to acid vapor, and the color of XTU changed from colorless to yellow. Accordingly, this process was reversible under amine vapor as shown in Fig. 4a. In addition, we prepared a THF solution of SP-TPE4 with concentration of 10^{-4} mol/L and dropped it to the filter paper without word. When the solvent was evaporated, we fixed it on the beaker containing nitric acid (HNO_3) or hydrochloric acid (HCl). Then, the beaker was heated and the nitric acid volatilized, and the filter paper changed from white to yellow immediately. On the other hand, the gaseous NH_3 could make the filter paper return to white. Therefore, we could see that the solid-state SP-TPE4 was responsive to acid and amine vapor as shown in Fig. 4b. The schematic diagram as shown in Fig. 4e demonstrated the invertible protonation and deprotonation process of SP-TPE4 under the condition of acid and amine vapor. In order to further prove the process, the FTIR was carried out as shown in Fig. 4d, and a new broad stretching vibration peak at 3200–3700 cm^{-1} which was ascribed to the formation of hydroxyl groups appeared under acid vapor. Besides, the characteristic peak at 3200–3700 cm^{-1} recovered almost under amine vapor. In addition, we investigated the absorption spectra of acidochromism of SP-TPE4 in the film state as shown in Fig. 4c, and a new absorption band at around 420 nm appeared after acid vapor treated and recovered quickly after amine vapor treated.

Hydrophilic and hydrophobic properties in the film

The SP-TPE4 THF solution was spin-coated onto the glass, and the contact angle of a water droplet was measured. As it can be seen from Fig. 5a, before irradiation the contact angle was 91.5° and changed to 81.2° (Fig. 5b) after irradiated by 365 nm LED lamp for 5 min. The decrease was due to the hydrophobic SP isomers converted to hydrophilic MC isomers after UV irradiation as exhibited in Fig. 5c.

Figure 3 The solid-state **a** absorption spectra, **b** PL spectra of SP-TPE4, **c** absorption spectra, **d** PL spectra of SP-TPE6, **e** XRD patterns of SP-TPEm films. **f** Color variation of SP-TPE4 powder and a filter paper with the letters of XTU used SP-TPE4 as the anticounterfeiting ink before and after UV irradiation. **g** Optimized molecular structures of SP-TPE4.



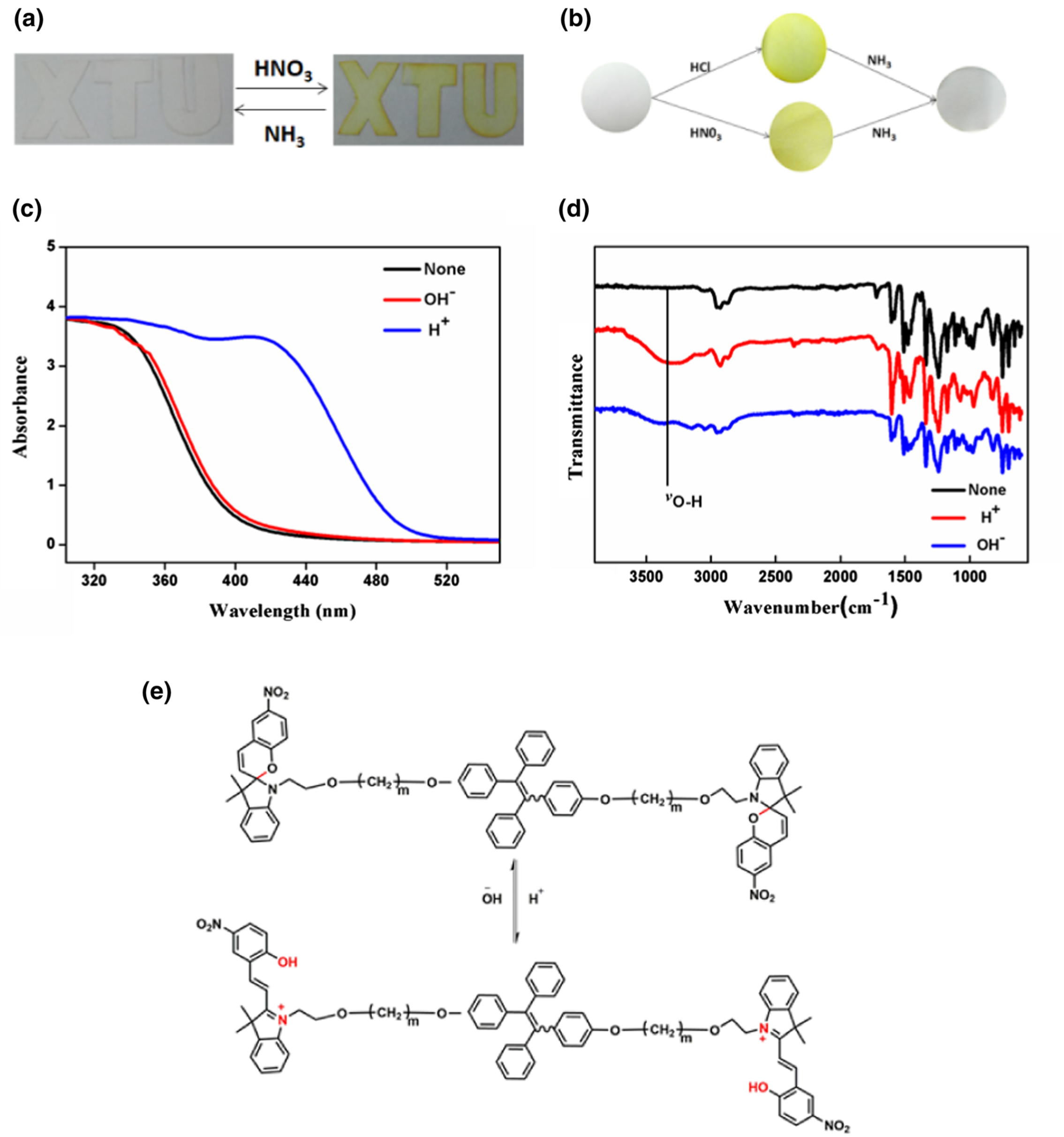


Figure 4 **a** Illustration of the acid/amine response of a filter paper printed with the letters of XTU and **b** a filter paper without letters. **c** The absorption spectra of SP-TPE4 in the film state under acid and amine vapor. **d** The corresponding FTIR spectra. **e** Illustration of the protonation and deprotonation process of SP-TPEm.

Conclusion

In short, we have successfully designed and synthesized two solid-state photoswitching materials SP-TPEm (*m* = 4, 6) based on spiropyran and TPE through alkyl spacers. Both of them show typical aggregation-induced emission behaviors. Effective optical switch between SP-TPEm and their photoisomer MC-TPEm is achieved after irradiated by 365 nm LED lamp both in solution and in the solid phase. Both of the films of SP-TPE4 and SP-TPE6 show great stimulus-responsive characteristics with loose molecular stacking and good emission. In addition, because of the great solid-state photochromic characteristics of SP-TPE4, it is applied as an anticounterfeiting ink. Moreover, the solid-state SP-TPEm are responsive to acid and amine. Moreover, the surface wettability of the SP-TPEm film can be regulated by UV irradiation. Therefore, we provide a new strategy to obtain solid-state multifunctional responsive materials.

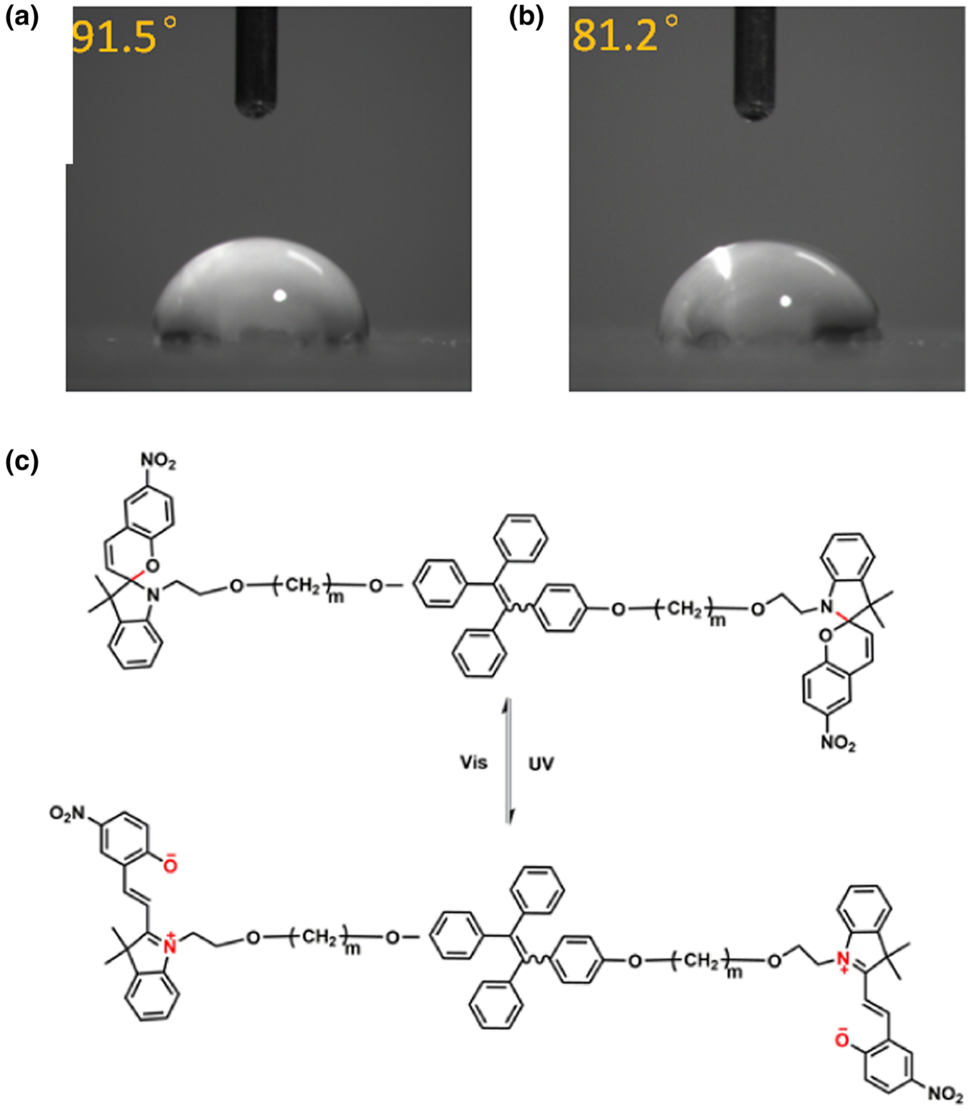


Figure 5 Illustration of the SP-TPE4 film contact angle **a** before and **b** after UV light illumination. **c** Schematic diagram of photoresponsive wettability.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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