



Full Length Article

AIE activity, mechanochromism, acidchromism, and high-level anti-counterfeiting based on multifunctional tetraphenylvinyl imidazopyridine

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ABSTRACT

TPE-IP was designed and synthesized by assembling tetraphenylethene (TPE) and imidazopyridine (IP) units, with weak push-pull molecular structure and propeller-like conformation, which were confirmed by fluorescence emission in various solvents and theoretical calculations. TPE-IP showed aggregation-induced enhanced emission (AIEE) activity due to the suppressed molecular motions in aggregated states. Interestingly, TPE-IP exhibited dual-band fluorescence emission in various solvents, deriving from localized and intramolecular charge transfer states. By grinding and heating, TPE-IP presented reversible mechanochromism, accompanied by a transition between deep blue and green fluorescence. Moreover, TPE-IP displayed high-contrast acidchromism, but with different responses to HCl, CF₃COOH, and CH₃COOH fuming. Meanwhile, the reversible acidchromism could be completed by HCl/CH₃COOH and Et₃N fuming, but not for CF₃COOH and Et₃N fuming. At last but not least, TPE-IP has the potential to be applied in the fields of anti-counterfeiting and information encryption.

1. Introduction

Organic semiconductor materials have tunable photophysical properties and can function as an optical logic gate with multiple encoding features [1–3]. Among of them, mechanochromic (MC) materials have already attracted widespread interest from application and academic research due to enormous potential values in pressure sensors, anti-counterfeiting, erasable paper, and data storage systems [4,5]. By external mechanical forces, MC materials show stimulus responsive fluorescence variation, including emission wavelength and intensity [6, 7]. In 2001, Tang found aggregation-induced emission (AIE) phenomenon [8], which significantly accelerates the development process of MC materials. Currently, aggregation induced emission (AIE) active molecules have become the main source of MC materials [9]. Of note, tetraphenylethene (TPE) unit has been used to construct MC materials due to distorted molecular conformation and typical AIE activity [10].

With the rise of e-commerce, many brand merchants have entered e-commerce platforms to enjoy the dividends of online sales. However, counterfeit products online have become increasingly severe, damaging brand image and consumer interests. In fact, the battle of cat and mouse

between counterfeit products and anti-counterfeiting has been ongoing. As we all know that multi-stimulus responsive luminescent materials (light, heat, stress, acid/alkali, solvent vapor, oxygen, etc.) are conducive to increase the difficulty of trademark replication based on more monitoring parameters [11,12]. Even so, there are only very few MC materials simultaneously combines other stimulus responsive fluorescence conversion [13]. Furthermore, fluorescence dyes with acidchromism have been widely applied in health monitoring, environmental monitoring, food safety, and other fields [14,15]. Combining acid sensitive structure units with TPE skeleton, the new luminescent compounds will be endowed with AIE activity, mechanochromism and acidchromism characteristics [16], which contribute to expand the application range of fluorescent chromophores, enhance anti-counterfeiting ability of the materials [17–19].

Herein, we designed and synthesized a new multifunctional material, named as TPE-IP, by assembling TPE and imidazopyridine (IP) units (Fig. 1a). Compared with TPE unit, IP unit has stronger electron withdrawing ability, thereby forming twisted donor-acceptor (D-A) molecular configuration. IP unit can bond with active proton and enhance intramolecular charge transfer (ICT) effect [20], boosting the generation of

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acidchromism, while the twisted D-A molecular configuration easily collapses under external mechanical forces, leading to mechanochromism. As expected, TPE-IP shows AIE activity, reversible mechanochromism, and different responses and reversibility to HCl, CF₃COOH and CH₃COOH. Finally, anti-counterfeiting patterns are successfully constructed based on mechanochromism and acidchromism of TPE-IP [21].

2. Results and discussion

2.1. Synthesis, photophysical properties in solutions, and aggregation-induced enhanced emission (AIEE)

As shown in Fig. 1a, TPE-IP was prepared by adopting the previous synthetic method, further characterized, and confirmed by NMR, HR-MS, and X-ray single crystal diffraction. The crystal TPE-IP (CCDC 2346137) is a monoclinic system, with the space group C 1 2/c 1 [a = 53.805(2) Å, b = 5.7435(2) Å and c = 36.5060(13) Å; $\alpha = 90^\circ$, $\beta = 122.486(2)^\circ$ and $\gamma = 90^\circ$]. By intermolecular hydrogen bonding and weak interactions such as C–H...C, C–H...H, C...C and C–H...N, TPE-IP maintains propeller-like molecular conformation, with dihedral angles of 83.99°, 64.17° and 86.78° between rings B–C, C–D, and D–E in sequence, but rings A–B presents planarized configuration, with a dihedral angle of 3.05° (Fig. 1b and c). Two adjacent imidazopyridine units adopt side to face arrangement mode, without intermolecular π – π stacking in crystals (Fig. 1d).

TPE-IP shows a wide absorption band covering 300–410 nm in various solvents (10^{−5} mol/L), and its absorption maxima remain basically constant with increasing solvent polarity, indicating a tiny dipole moment in the ground state (Fig. 2a). Interestingly, TPE-IP presents two obvious emission peaks at 405 nm and 525 nm in fluorescence spectra, which should come from localized state (LE) and intramolecular charge transfer state (ICT) emission respectively [22] (Fig. 2b). In various solvents, fluorescence emission maxima of TPE-IP also show tiny shifts, illustrating that the dipole moment of the excited state is also small. In toluene, DCM and THF, the LE state exhibits stronger emission than the ICT state, but with an opposite result in DMSO, which should be attributed to the enhanced solvent polarity. Beyond expectation, the ICT state exhibits stronger emission than the LE state in n-hexane. To avoid

molecular aggregation, n-hexane solution of TPE-IP was diluted to 10^{−6} mol/L from 10^{−5} mol/L, with higher emission intensity for the LE state than ICT state after dilution, confirming the influence of molecular aggregation (Fig. 2c). By using THF and H₂O as benign and poor solvents respectively, the aggregation-induced activity of TPE-IP was investigated. When the water fraction (f_w) reaches 99%, the emission intensity of TPE-IP shows significant enhancement, outlining an obvious aggregation-induced enhanced emission (AIEE) scene (Fig. 2d and e). In contrast, TPE-IP still gives stable emission maxima in the aggregated state compared to various solutions, which should be attributed to the absence of intermolecular π – π stacking interactions [23]. The optimized ground state geometry and vibrational frequencies of TPE-IP were calculated using wB97X-D3(BJ)/def2-SVP theory level. Then, single point energy was computed at wB97M-was computed at wB97M – V/def2-TZVP level. As shown in Fig. 2f, the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) energy levels of TPE-IP were −7.529 eV and 0.326 eV respectively. The electron cloud density of HOMO orbital of TPE-IP was distributed on TPE and imidazole units, but the electron cloud density of LUMO orbital of TPE-IP shifts more towards electron-withdrawing benzimidazole unit. The diversity in electron cloud densities demonstrates ICT effect of TPE-IP.

2.2. Mechanochromism and acidchromism

By slow diffusion of n-hexane into DCM solution of TPE-IP, light brown crystals were obtained under natural light, emitting bright blue fluorescence under 365 nm UV irradiation, with an emission peak at 450 nm (Fig. 3a). By grinding, emission maxima of TPE-IP showed bathochromic-shifts of 23 nm, with green fluorescence under 365 nm UV radiation, indicating obvious mechanochromism. The absolute fluorescence quantum yields (Φ_F) of TPE-IP were investigated by an integrating sphere before/after grinding, which indicated grinding resulted in obvious falling Φ_F from 0.19 to 0.09. In crystalline state, there are numerous intermolecular interactions, which are disrupted by grinding, leading to the increasing molecular motion, and decreasing Φ_F . By heating, TPE-IP showed reversible mechanochromism, reemitting blue fluorescence under 365 nm UV irradiation, whose emission spectra have good overlap with that of crystal TPE-IP (Fig. 3a and b). XRD spectra

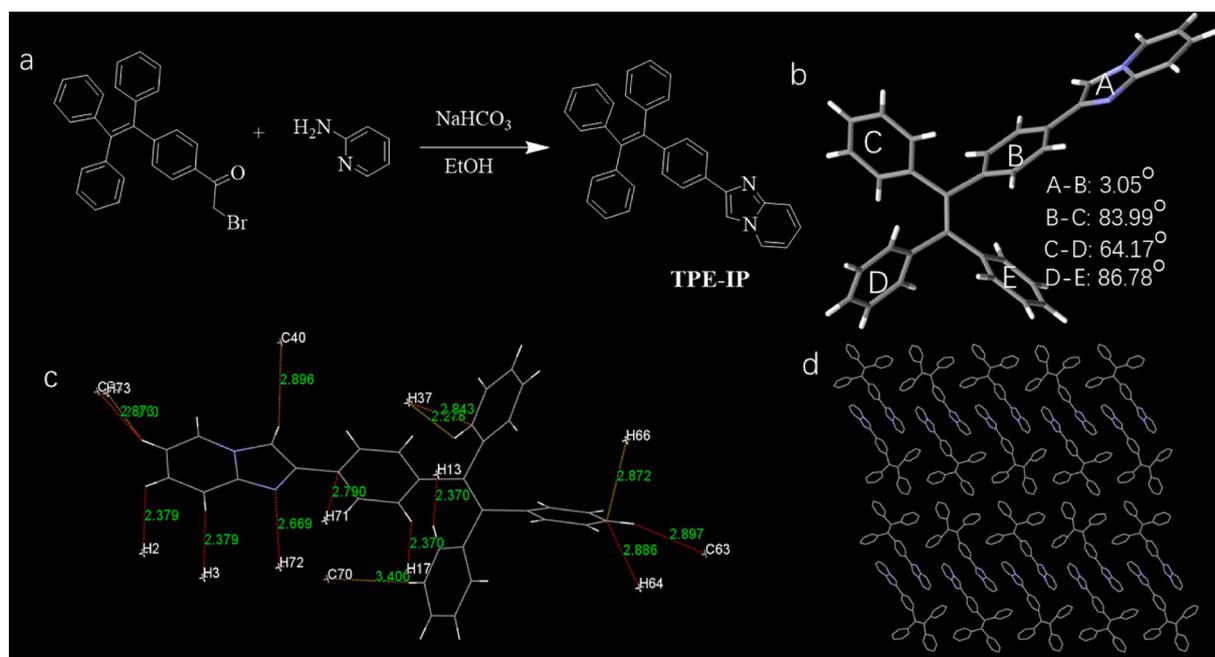


Fig. 1. (a) The synthesis route of TPE-IP. (b) Molecular conformation, (c) intermolecular interactions and (d) stacking patterns neglecting hydrogen atoms of TPE-IP.

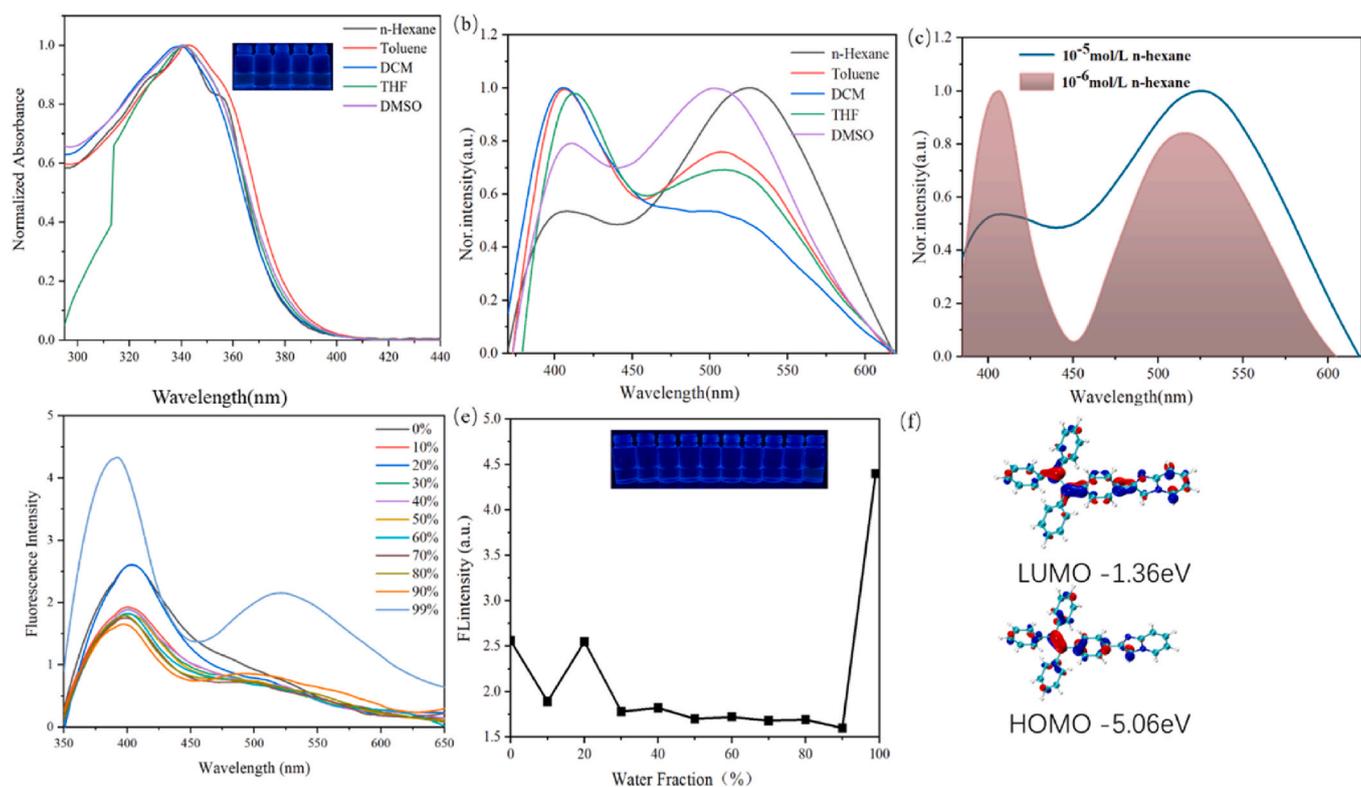


Fig. 2. (a) Normalization absorption and (b) fluorescence spectra of TPE-IP in various solvents (solution concentration: 10^{-5} mol/L). (c) Fluorescence spectra of TPE-IP in n-hexane (solution concentration: 10^{-5} mol/L and 10^{-6} mol/L). (d) Fluorescence emission spectra of TPE-IP in THF- H_2O solution with different water fractions (solution concentration: 10^{-5} mol/L). (e) Photos of the relationship between the fluorescence emission peak intensity/wavelength of TPE-IP (solution concentration: 10^{-5} mol/L. Inset: Photographs in THF- H_2O solution with different fractions of water under 365 nm UV illumination.). (f) The electron-cloud density profiles and energy level diagram for HOMO and LUMO orbitals of TPE-IP calculated by Gaussian 16 W program.

indicated crystals TPE-IP had strong diffraction signals, which transformed into a wide diffraction signal after grinding. Thereby, the mechanochromism of TPE-IP could be attributed to the phase transition from crystalline to amorphous state. After heating, some diffraction peaks reappear, demonstrating regression of crystalline state. To further understand the mechanochromism, DSC of TPE-IP were subsequently investigated in different solid states. The scanning calorimetry (DSC) analyses indicated that crystal TPE-IP had two endothermic peaks at 43.3 °C and 389 °C respectively, corresponding to solvent removal and melting temperature in sequence, while ground TPE-IP gave three endothermic peaks at 32.5 °C, 265 °C, and 389 °C, without the exothermic transition peak demonstrating cold-crystallization transition. Therefore, the amorphous phase of ground samples should not be in a metastable state. The imidazopyridine unit can bond with active proton due to alkalinity, endowing TPE-IP with acidchromism properties. By fumigation with HCl, CF_3COOH , and CH_3COOH , fluorescence emission maxima of TPE-IP presented significant red shifts due to the enhanced electron withdrawing ability of protonated imidazopyridine unit, accompanied by color changes from dark blue to light blue or green [24]. Moreover, TPE-IP sample fumigated with HCl provided the maximum wavelength shift, follow by CH_3COOH and CF_3COOH , which was not consistent with the strength of acidity. As speculation, the intermolecular stacking and arrangement modes of protonated TPE-IP may change [25], leading to different wavelength shifts. More interestingly, TPE-IP showed reversible acidchromism by HCl and Et_3N , as well as CH_3COOH and Et_3N fumigating in sequence, but with irreversible acidchromism for CF_3COOH and Et_3N fumigating.

2.3. Anti-counterfeiting

Based on mechanochromism and acidchromism of TPE-IP, two

information encryptions were designed [26] (Fig. 5). Firstly, a piece of common filter paper was soaked and sonicated in n-hexane solution of TPE-IP. Then the filter paper was dried (80 °C) for 2 h in the oven, which emitted bright blue fluorescence under 365 nm UV lamp irradiation. Subsequently, “TPED” was written by a glass rod, with green fluorescence. Thereby “TPED” could be clearly presented due to high-contrast mechanochromism for TPE-IP. “TPED” was erased by heating, indicating potential application value in the field of reusable erasable paper. Taking advantage of the filter paper mentioned above, three letters “X”, “Y”, and “Z” were cut out, which were fumigated with hydrochloric acid and triethylamine in sequence. In sunlight, the three letters show the same color as common filter paper due to absorption spectra of TPE-IP mainly located on UV region below 400 nm (Fig. 2a). Under 365 nm UV lamp irradiation, color transitions of the three letters could be observed from blue to green and then to blue, which was attributed to reversible acidchromism of TPE-IP. To be emphasized, TPE-IP has great potential for enhancing information encryptions [27]. Furthermore, fluorescence spectra of the filter papers carrying TPE-IP were tested before/after grinding and fumigation with hydrochloric acid and triethylamine (Fig. 5c and d), whose emission maxima only showed shifts of 1–4 nm compared with that of solid samples of TPE-IP (Fig. 4c and d).

3. Conclusions

In conclusion, TPE-IP were prepared by assembling TPE and IP units. Crystal analysis and theoretical calculation display that TPE-IP show distorted molecular conformation and D-A molecular skeleton [28]. Owing to the distorted molecular conformation and the absence of intermolecular π - π stacking, TPE-IP presents AIEE and mechanochromic activity, with obvious color contrast between blue and green. More importantly, the mechanochromism can be reversibly tuned by grinding

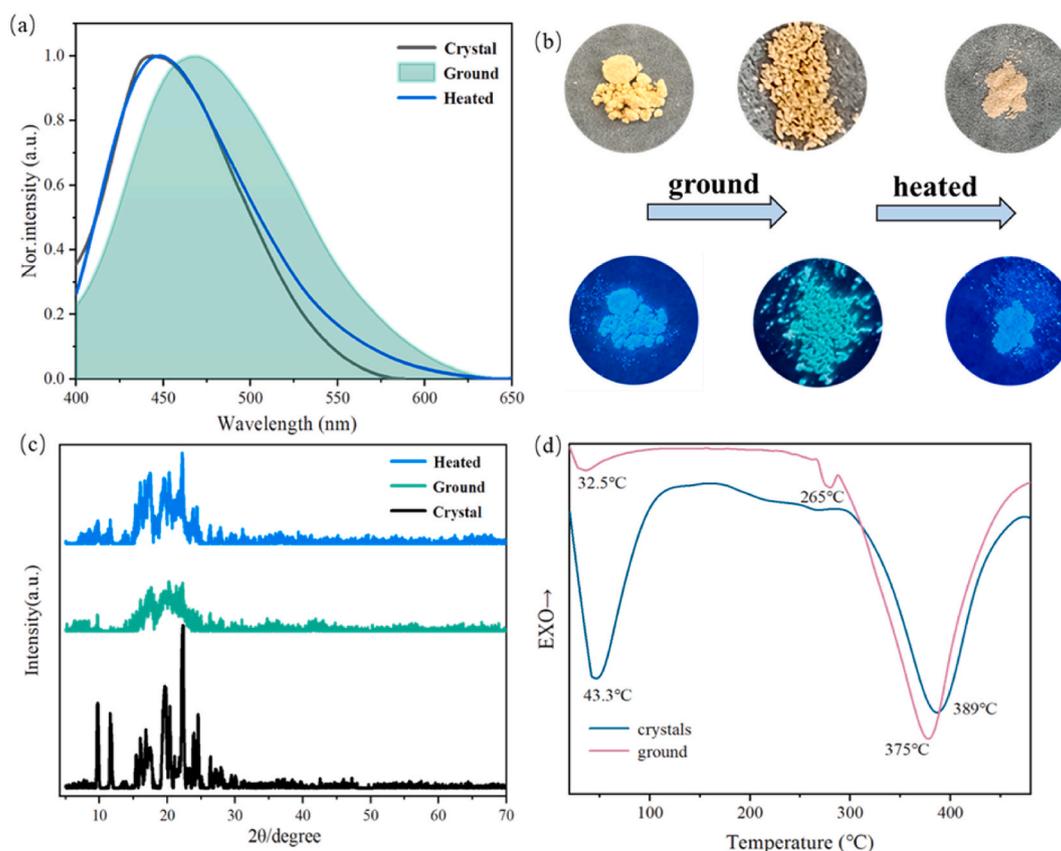


Fig. 3. (a) Fluorescence and; (b) UV and visible light images of TPE-IP before and after grinding and heating. (c) XRD spectra of TPE-IP before and after grinding and heating (d) DSC thermograms of crystal and ground TPE-IP samples.

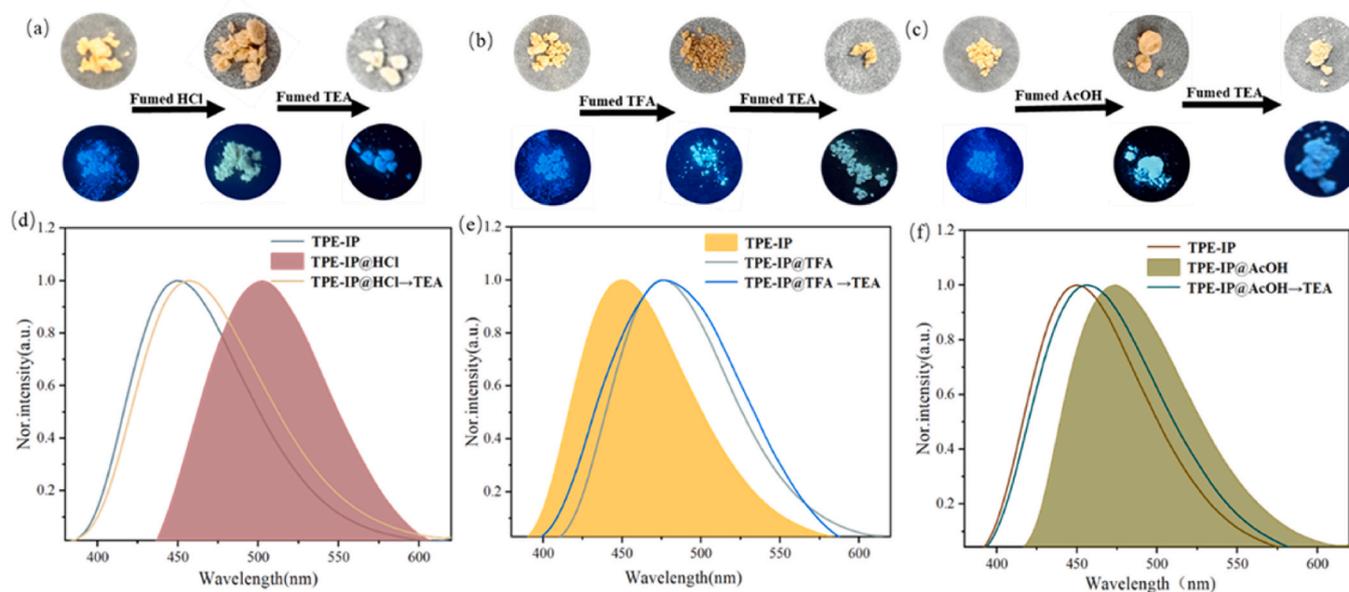


Fig. 4. (a) Under sunlight and 365 nm ultraviolet lamp, images of TPE-IP before and after fumigation with hydrochloric acid and triethylamine (b) under sunlight and 365 nm ultraviolet lamp, images of TPE-IP before and after fumigation with trifluoroacetic acid and triethylamine (c) under sunlight and 365 nm ultraviolet lamp, images of TPE-IP before and after fumigation with acetic acid and triethylamine (d) crystals, fluorescence spectra before and after fumigation with hydrochloric acid and triethylamine (e) crystals, fluorescence spectra before and after fumigation with trifluoroacetic acid and triethylamine (f) crystals, fluorescence spectra before and after fumigation with acetic acid and triethylamine.

and heating. XRD diffraction patterns confirmed that the mechano-chromism is attributed to the phase transformation between crystalline state and amorphous state. Interestingly, TPE-IP can response and

distinguish HCl, CF_3COOH and CH_3COOH , with the maximum wavelength shift for HCl fumigation, follow by CH_3COOH and CF_3COOH . Moreover, TPE-IP showed reversible acidchromism by HCl/ CH_3COOH

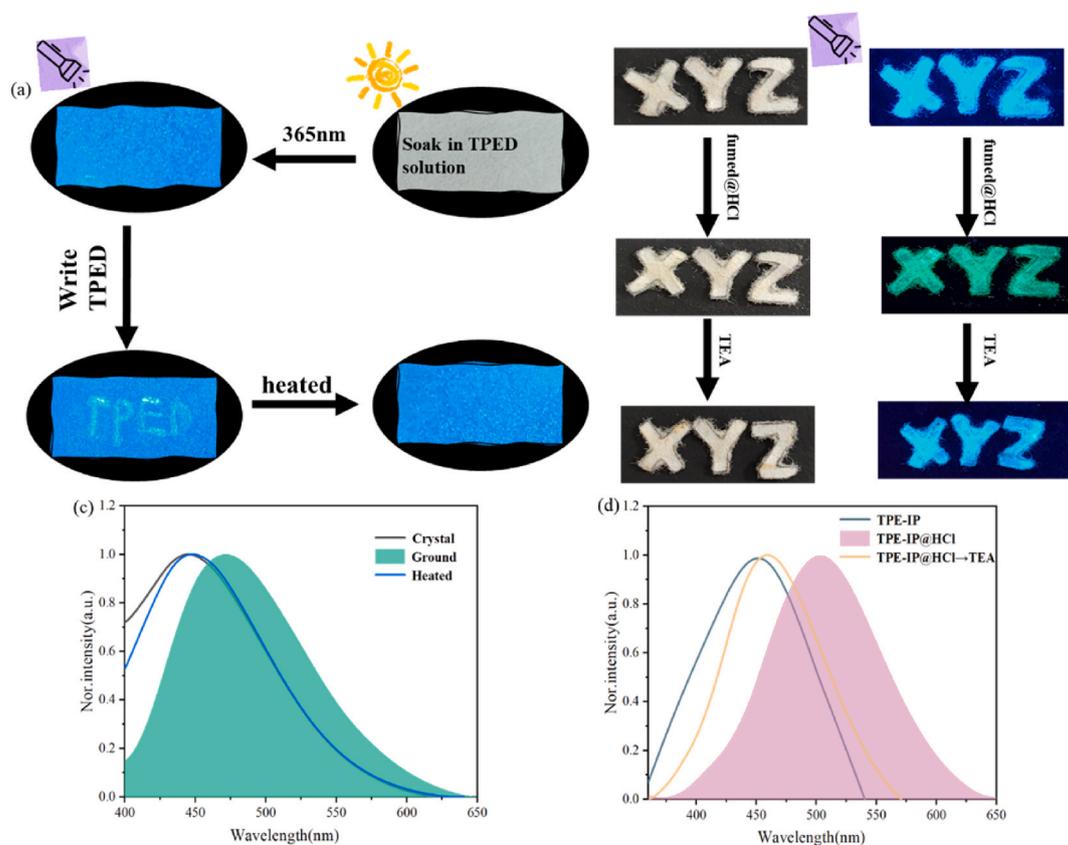


Fig. 5. (a) Mechanochromism and (b) acidichromism photographs of TPE-IP under 365 nm UV lamp before and after writing and erasing, as well as hydrochloric acid and triethylamine fumigation. (c) Fluorescence spectra of TPE-IP before and after grinding and heating. (d) Fluorescence spectra of TPE-IP before and after fumigation with hydrochloric acid and triethylamine.

and Et_3N fumigation, but not for CF_3COOH and Et_3N fumigation. Based on mechanochromism and acidichromism of TPE-IP, reusable erasable paper and anti-counterfeiting patterns are successfully constructed.

CRedit authorship contribution statement

Yuchen Hong: Writing – original draft, Validation. **Yupeng Zhao:** Data curation. **Yanjun Guo:** Supervision. **Yongtao Wang:** Writing – review & editing, Supervision, Conceptualization. **Lei Ma:** Data curation.

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.

Data availability

I have shared the link to my data.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jlumin.2024.120678>.

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