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Multifunctional properties of a star-shaped triphenylaminebenzene-1,3,5-tricarbohydrazide fluorescent molecule containing multiple flexible chains

Xin Feng,[‡] Yating Chen,[‡] Yunxiang Lei, Yunbing Zhou, Wenxia Gao,* Miaochang Liu, Xiaobo Huang* and Huayue Wu

A star-shaped triphenylamine-benzene-1,3,5-tricarbohydrazide molecule with a twisted molecular conformation was found to display amazing multifunctional optical properties. The design of peripheral triphenylamine units and central benzene connected with hydrazide groups leads to the formation of rare polymorphic property in the presence of multiple flexible chains. Two polymorphs with different fluorescence colors exhibited bathochromic mechanfluorochromic activities with high contrast due to a crystalline-to-amorphous transition.

Organic mechanofluorochromic (MFC) materials, which can exhibit the changes of solid-state fluorescence colors and intensities under the stimulation of external pressure, have recently attracted considerable attention owing to the wide applications in the fields of chemosensors, rewritable optical media, memory devices, and security systems.¹ On the view of macroscopic, the MFC activities are often attributed to morphological changes.² Therefore, the crystallization ability of the candidate compounds is very important because only by obtaining the original sample with crystalline state can it be possible to realize the transition from a crystalline state to an amorphous state or from one crystalline state to another. Especially, the realization of the latter depends to a great extent on the polymorphic property of the compounds, which is reported to exist in one-third of organic compounds, but its discovery strongly depends on experimental experiences rather than prediction.³ Up to now, most of organic compounds with MFC properties have been reported to focus on rigid and twisted molecular structures,⁴ and flexible chains are confirmed to be used to regulate their packing modes.⁵ However, flexible chain is a double-edged sword because the increase of its number will easily lead to the disorder of organic molecules and greatly reduce their crystallization abilities, which are not advantageous to the generation of the compounds with MFC and/or polymorphic properties. Therefore, although some

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organic MFC-active compounds containing multiple flexible amide chains have been reported, ⁶ to the best of our knowledge, this kind of compounds with both mechanofluorochromism and polymorphism are not reported, which is still challenging and worthy of further study.

In this work, a star-shaped triphenylamine-benzene-1,3,5tricarbohydrazide molecule (TBTCH, Fig. 1A) was designed and synthesized, in which the peripheral rigid triphenylamine (TPA) groups and the central benzene ring are connected with amide groups and C=N bonds. Our design ideas are as follows: (1) the rigid structures in the center and the periphery can weaken the disorder caused by a large number of flexible chains and keep the target compound with appropriate crystallization ability; (2) the TPA unit has propeller-like conformation and strong electron-donating ability, which is beneficial to obtain aggregation-induced emission (AIE), strong solid-state emission, and thus possible MFC activity; (3) the flexible chains containing hydrazide groups can be used to regulate the molecular packing modes by intermolecular N-H...O, N-H...N, or C-H...O hydrogen bonds, and the varied balance of the non-covalent interactions between molecules is advantageous to the generation of polymorphism, moreover, the existence of numerous hydrogen bonds also provides the basis for the formation of organic gel between fluorescent molecules.⁷ As expected, **TBTCH** exhibits interesting multifunctional optical properties containing both polymorphism and mechanofluorochromism.

The target compound **TBTCH** was prepared from a simple twostep procedure using commercially available trimethyl benzene-1,3,5-tricarboxylate as the starting material according to Scheme S1 (ESI⁺), and the detailed experimental methods are provided in the supporting information. Considering the strong electron-donating ability of TPA unit, **TBTCH** could be thought as a D- π -A-type molecule with the electron cloud flowing from the periphery to the center. The absorption and fluorescence spectra in various organic solvents with the increasing polarity, such as toluene, chloroform, dichloromethane (DCM), dimethyl sulfoxide (DMSO), acetonitrile (MeCN), and methanol, were investigated (Fig. S1 and Table S1, ESI⁺). The maximum absorption wavelength of this compound in different solvents was in the range of 373-385 nm, indicating that the polarity of the solvent had little influence on the absorption

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wavelengths. However, the fluorescence spectrum exhibited an obvious bathochromic shift from 442 nm in toluene to 518 nm in methanol with a fluorescence color change from blue to yellow. This result revealed that TBTCH displayed obvious solvatochromic property. Based on the Lippert-Mataga equation,⁸ the effect of solvent polarity on the photophysical properties was investigated. The absorption maxima and emission maxima of TBTCH and solvent polarity parameter in different solvents are collected in Table S1 (ESI⁺) and the Stokes shift (Δv) against orientation polarizability (Δf) was conducted. As depicted in Fig. S2 (ESI⁺), Δv has a positive linear correlation with Δf (R = 0.95), which revealed that the dipole moments of the excited states of TBTCH were larger than those of the ground states. Therefore, the solvatochromic properties of TBTCH should be attributed that the solvent molecules with large polarity effectively reduce the energy of excited states and enhance the intramolecular charge transfer (ICT) effect.⁹

The absorption and fluorescence spectra of TBTCH in tetrahydrofuran (THF)-water mixture were investigated the possible AIE property. When water volume fraction (f_w) was at 0-50%, the maximum absorption wavelengths of the mixtures did not change significantly and the fluorescence molecules should exist in free form (Fig. S3a, ESI⁺). Correspondingly, the emission wavelengths of the mixtures gradually red-shifted and the intensity decreased gradually (Fig. 1B), which should be ascribed to the increase of the polarity of the mixtures.¹⁰ However, when $f_w \ge 60\%$, the maximum absorption wavelengths of the mixtures obviously became larger, and the absorption spectra became broad and upward-shifted, which should be caused by the Mie scattering effect, revealing the formation of aggregates.¹¹ Correspondingly, the emission wavelengths of the mixtures decreased gradually, and the intensity increased gradually and reached the maximum value at f_w = 90%, which was about 7-fold higher than that in pure THF solvent (Fig. S3b, ESI⁺). The emission behaviors of the mixtures at f_w = 60-90% should be dominated by the AIE effect by inhibiting the intramolecular rotations and thus eliminating the non-radiative energy loss.¹²

Furthermore, the gelation behavior of TBTCH was examined in organic solvents. When the chloroform solution of TBTCH was heated at 60°C and then cooled to room temperature, the gelator could be obtained, which had excellent gelation ability with very low critical gelation concentration of 0.27 wt%. The gelator showed yellow color in appearance and emitted cyan fluorescence (Fig. 1C), and turned into the solution after being heated, revealing good thermo-reversibility. The sol-gel and gel-sol transitions were the results of the assembly of three-dimensional network and the disruption of non-covalent interactions within the gel network, respectively. The assembly of three-dimensional network could be observed in the scanning electron microscopy (SEM) image after the gelator was dried (Fig. 1C), which was driven by the hydrophobic interactions as well as hydrogen bonds and π - π stacking interactions to immobilize a large volume of organic solvent in the organogel.¹³ The fluorescence of the gelator increased along with the increased gelator concentration, and the intensity began to decrease once the concentration was higher than 25 μ g/mL (Fig. S4a, ESI⁺). The decrease of fluorescence of the gelator demonstrated the formation of π - π stacking interaction.¹⁴ The ¹H NMR spectra of the gelator showed the gradual high field



Fig. 1 (A) Chemical structure of **TBTCH**. (B) Fluorescence spectra of **TBTCH** in THF-water mixtures with different f_w at a concentration of 1×10^{-5} mol/L. Insets: Inset: fluorescence photos at $f_w = 0\%$, 50%, and 90% under 365 nm irradiation. (C) Image of gel under natural light (a) and 365 nm light (b), and SEM image of xerogel (c). (D) Fluorescence spectra of **TBTCH**-*c* and **TBTCH**-*g* under 365 nm irradiation.

shift of protons in aromatic rings with increasing the concentration of gelator (Fig. S4b, ESI⁺), which further indicated the formation of intermolecular π - π stacking interactions.¹⁵ Additionally, the hydrogen-bond interactions seemed to play a key role in the threedimensional self-assembly of the gelator because numerous hydrogen bonds were observed in the aggregated state of the compound due to the presence of amino groups and carbonyl groups in the chemical structure (see below).⁷

In particular, even if the presence of multiple flexible hydrazide groups, two kinds of single crystals **TBTCH-***c* and **TBTCH-***g* could be successfully obtained from a slow evaporation of the DMSO/chloroform/methanol (v/v/v = 1:5:5) mixture and the DMSO/THF (v/v = 1:2) mixture, respectively. **TBTCH-***c* emitted cyan fluorescence at 477 nm with fluorescence quantum efficiency (Φ_F) of 45.3%, while **TBTCH-***g* emitted green fluorescence at 498 nm with a Φ_F value of 29.2% (Fig. 1D and Table S2, ESI⁺). This result indicated that **TBTCH-***g* had polymorphic property. Compared with **TBTCH-***g*, **TBTCH-***c* showed a higher Φ_F value, which was attributed that **TBTCH-***c* had a larger radiative rate constant ($k_{\rm f} = 1.73 \times 10^8 \, {\rm s}^{-1}$) and smaller non-radiative rate constant ($k_{\rm nr} = 2.09 \times 10^8 \, {\rm s}^{-1}$) than **TBTCH-***g* ($k_{\rm f} = 1.64 \times 10^8 \, {\rm s}^{-1}$ and $k_{\rm nr} = 3.98 \times 10^8 \, {\rm s}^{-1}$) (Table S2, ESI⁺).

Upon grinding using mortar and pestle, green-emitting **TBTCH-***g* showed a 30 nm red shift in the emission spectrum from 498 nm to 528 nm, and transformed into yellow-emitting ground sample with a decrease of $\Phi_{\rm F}$ from 29.2% to 15.5%, revealing the MFC property (Fig. 2A, Fig. 2C and Table S2, ESI⁺). The X-ray powder diffraction (XRD) curves showed the crystalline structure was destroyed upon grinding because the strong and sharp diffraction peaks disappeared completely (Fig. S5a, ESI⁺). Furthermore, according to the SEM experiments, large size columnar crystals of **TBTCH-***g* were obviously damaged under the grinding treatment (Fig. S6, ESI⁺). Thus, the solid-state fluorescence color change was ascribed to the transition from the crystalline state to the amorphous state. When the ground sample was fumed using ethyl acetate (EA) vapor, the resultant fumed sample exhibited a 22 nm blue shift in the emission spectrum and emitted yellow-green fluorescence at $\lambda_{\rm em} = 506$ nm

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with a $\Phi_{\rm F}$ value of 27.3%. For the fumed sample, considering that only several broad and weak diffraction peaks (ϑ = 5.4°, 11.5° and 16.3°) reappeared and the XRD curve could not completely recover to that of the original sample (Fig. S5a, ESI⁺), it should be the mixtures of crystalline and amorphous states. In view of TBTCH-c, after being ground, the resultant sample emitted yellow-green fluorescence at λ_{em} = 512 nm with a Φ_{F} value of 28.3% (Fig. 2A, Fig. 2D and Table S2, ESI⁺). However, the solid-state fluorescence color of the ground sample could not recover to the original state by the common fumigation method. It should be pointed out that although the ground sample of TBTCH-c was very similar to the fumed sample of TBTCH-g in the fluorescence colors and spectra, this sample was completely amorphous according to the XRD curve (Fig. S5b, ESI⁺). By heating the TBTCH-c sample at 217°C for 1 h and then cooling to room temperature, the resultant annealed sample was consistent with the fumed sample of TBTCH-g (Fig. 2A, Fig. 2C and Table S2, ESI⁺). The conversion between **TBTCH-***g* and **TBTCH-***c* could only be achieved by the recrystallization process under the corresponding conditions. Considering that the reversibility of the MFC activity of the TBTCH-g sample, it could be developed as a rewritable optical medium (Fig. 2B). First, the TBTCH-g solid was laid on the filter paper coated with commercial solid glue, and then a hollow pattern model of animal was placed on the surface of TBTCH-g. The hollow part was ground with a metal spatula, and then a yellow animal picture was presented in the green background under the 365 nm irradiation, which had a high contrast in color. After being fumed by EA vapor, the animal picture disappeared.

Because the photophysical properties and MFC activities of the fluorescent molecules are often related to their crystalline structures,¹⁶ we first investigate the molecular conformations of **TBTCH-***g* and **TBTCH-***c* (Fig. S7, ESI⁺). For **TBTCH-***g*, the dihedral angles between the central phenyl ring (A) and the peripheral phenyl rings B, C, and D are 17.55°, 34.98°, and 35.95°, while the corresponding dihedral angles in **TBTCH-***c* are 18.02°, 35.16°, and 36.05°, respectively, which indicates that **TBTCH-***c* has a more twisted molecular conformation than **TBTCH-***g*. We further



Fig. 2 (A) Fluorescence images of the solid samples of TBTCH under 365 nm irradiation: (a) TBTCH-g; (b) the ground sample of TBTCH-g; (c) the fumed sample of TBTCH-g; (d) TBTCH-c; (e) the ground sample of TBTCH-c. (B) A rewritable optical medium based on the surface of TBTCH-g coated on filter paper upon grinding (write) and fuming (erase). (C) Fluorescence spectra of TBTCH-g upon different conditions. (D) Fluorescence spectra of TBTCH-c upon different conditions.

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investigated the stacking arrangements and intermolecular interactions of these two compounds. As show hip Fig. 99 and Fig. S8a (ESI⁺), TBTCH-g and TBTCH-c both can be considered to be composed of the molecule pairs with reverse and dislocated stacking, and adopt very similar stacking arrangements. For TBTCHg, the two molecules in pair are linked by the C-H...O bonds (2.497 and 2.760 Å), N–H[…]O hydrogen bond (2.258 Å), and N–H[…]N hydrogen bond (2.590 Å) (Fig. 3b), and the adjacent molecules of different pairs are linked by the N-H...O hydrogen bond (2.184 Å) (Fig. 3c). Additionally, due to the large space structure of this molecule, the non-adjacent molecules can also be connected with each other by C–H[…]π (Ph) bonds (2.982, 3.311, and 3.495 Å) between triphenylamine molecules which are close in space (Fig. 3d). In view of **TBTCH-***c*, the molecules in the single crystal are also stabilized by the corresponding C–H^{$\cdot\cdot$}O and C–H^{$\cdot\cdot$} π bonds, and N– H...O and N-H...N hydrogen bonds (Figs. S8b-S8d, ESI+). The difference of the non-covalent interactions between TBTCH-g and TBTCH-c is that the distances of the former are generally larger than the corresponding distances of the latter, revealing that the latter has stronger non-covalent interactions and closer stacking. Another proof is that the crystal lattice of TBTCH-c had a slightly smaller volume and a slightly larger density (V = 2938.6 Å and d = 1.239Mg/m³) than that of **TBTCH-***g* (V = 2945.1 Å and d = 1.236 Mg/m³) (Table S3, ESI⁺). Considering that these two polymorphs have a nearly identical stacking pattern, compared with TBTCH-g, the blueshifted emission and higher $\Phi_{\rm F}$ value of **TBTCH-***c* should be attributed to the more twisted conformation and stronger noncovalent interactions.

Based on the results of the single-crystal structural analyses, the AIE activity and strong solid-state emission of **TBTCH** can be explained as follows. In the pure solvent, the intramolecular free rotations of phenyl rings around the carbon-carbon single bond enhance the non-radiative relaxation process of the isolated molecules and eliminate the emission. In the THF-water mixtures with high f_w value (60-90%), the non-covalent interactions caused by the aggregation effectively restrict the intramolecular rotations



Fig. 3 The stacking mode (a) and intermolecular interactions (b-d) in the crystals of TBTCH-g.

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and enhance the emissions. Moreover, the reverse and dislocated stacking arrangements are advantageous to avoid the formation of strong π - π stacking and thus lead to strong solid-state emission. More importantly, the crystalline structures of the polymorphs with weak non-covalent interactions are easily destroyed upon grinding, which results in the changes of morphology and thus generating MFC phenomena. It should be noted that although the ground samples of TBTCH-g and TBTCH-c are both amorphous states, they emit fluorescence with different colors and different quantum efficiencies. This result suggests that the red-shifted emissions of the polymorphs upon grinding should depend on the change of stacking mode caused by the destruction of crystalline structures rather than the planarization of the molecular conformation because the latter will result in consistent ground samples. Furthermore, the maximum absorption wavelengths of TBTCH-g and TBTCH-c before and after grinding remain unchanged (Fig. S9, ESI⁺), namely the effective conjugation is undisturbed, confirming the formation of the excimers.¹⁷ This further confirms that the change of stacking mode should play a more important role compared with that of molecular conformation because the latter will lead to the outstanding changes of the absorption wavelengths.¹⁸ The possible reasons for the bathochromic phenomenon in the MFC activities can be further explained as follows: upon grinding, the crystal structure is destroyed and the distances between molecules become smaller, which makes the overlap between benzene rings larger, resulting the formation of the excimers and thus the red-shifted emissions. Additionally, the fact that the ground samples of these two compounds could not restore to the corresponding original samples upon fuming or annealing should be attributed that these large amounts of noncovalent interactions caused by flexible chains and aromatic groups are difficult to recover completely.^{6b}

In conclusion, a star-shaped TBTCH with twisted conformation was designed and synthesized, in which the peripheral TPA units and the central benzene are connected with multiple hydrazide groups. TBTCH showed amazing multifunctional optical properties. The solvatochromic property was due to the ICT effect and the AIE property depends on the restriction of intramolecular rotation. This compound also exhibited excellent gelation ability in chloroform with good thermo-reversibility. Especially, TBTCH showed polymorphic property even if the presence of multiple flexible hydrazide groups. The difference emissions of TBTCH-g and TBTCHc were confirmed to be ascribed to different molecular conformations and non-covalent interactions. Furthermore, these two polymorphs displayed high contrast red-shifted MFC activities due to a crystalline-to-amorphous transition upon grinding. This work demonstrates the applicability of a rational strategy to design the multifunctional fluorescent material with polymorphic and MFC activities, even if it contains multiple flexible chains.

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Multifunctional properties of a star-shaped triphenylamine-benzene^{view Article Online} 1,3,5-tricarbohydrazide fluorescent molecule containing multiple flexible chains

Xin Feng, Yating Chen, Yunxiang Lei, Yunbing Zhou, Wenxia Gao, Miaochang Liu, Xiaobo Huang and Huayue Wu

A star-shaped triphenylamine-benzene-1,3,5-tricarbohydrazide molecule containing multiple flexible hydrazide chains displays amazing multifunctional optical properties including solvatochromism, gelation, aggregation-induced emission, polymorphism, and mechanochromism.

