A novel triphenylacrylonitrile based AIEgen for high contrast mechanochromism and bicolor electroluminescence

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A novel thermally stable and aggregation-induced emission (AIE) active compound, 2,2’-(((1,1’-biphenyl)-4,4’-diylibis(phenylazanediyl))bis(4,1-phenylene))bis(3,3-diphenylacrylonitrile) (BP2TPAN) was synthesized through a C–N coupling reaction between 2-(4-bromophenyl)-3,3-diphenylacrylonitrile (Br-TPAN) and N,N’-diphenyl-1,4-phenylenediamine, under mild conditions using Pd(OAc)2 and P(–Bu)3 as a catalyst. The BP2TPAN was characterized by nuclear magnetic resonance spectroscopy, high resolution mass spectrometry and elemental analysis. The thermal analysis showed that the glass transition and decomposition temperatures (5% weight loss) are 96 and 414 °C, respectively. The fluorescent emission peaks changes at 540 and 580 nm upon grinding were attributed to a transformation from crystal to amorphous occurring by altering the condensed state. The photoluminescence quantum yield and fluorescence lifetime of the as prepared and ground samples were 74.3 and 8.4%, 3.4 and 5.1 ns, respectively. The difference of the luminous efficiency of before and after grinding samples indicates BP2TPAN has a high contrast more importantly, both doped and nondoped OLED devices emit different color, the doped one is highly efficient and its Lmax, CEmax, PEmax and EQE are up to 15 070 cd m⁻², 11.0 cd A⁻¹, 7.5 lm W⁻¹, and 3.1%, respectively.

1 Introduction

Organic luminescent materials which show dynamically switchable solid-state emission in respect to mechanical stimuli such as shearing and grinding, have attracted increasing interest owing to their academic importance and promising applications in optical storage, mechanical sensors, displacement or deformation detectors, security paper, optical memory applications in optical storage, mechanical sensors, displacement or deformation detectors, security paper, optical memory devices, etc. AIE active molecules are highly fluorescent in solutions, but become highly emissive in aggregated states. The AIE active molecules are highly fluorescent in the solid state, which is an essential requirement for mechanochromic materials. The mechano luminescence phenomenon has been explained as an alteration of molecular packing modes, such as crystal to amorphous form transformation, formation of excimers/ exciplex, or planarization under the action of forces. Over the past few years, many novel twisted conformations AIE active mechanochromic materials have been developed by Chi, Park, Tian, Tang et al. group, and the relationship between the molecular structure and the mechanochromic luminescence has been evaluated. However, a few studies on AIE mechanochromic materials with its high thermal stability and high contrast have been reported. On the other hand, AIE compounds are ideal alternatives used for OLED applications. So far, however, many AIE compounds are mainly fabricated OLED devices with only one electroluminescent color.

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However, a few studies reported OLED devices with two or more electroluminescence emitting color based AIE compounds.\textsuperscript{24,25}

Herein, a novel highly twisted conformation compound BP2TPAN with good thermal stability, containing a triphenylacrylonitrile (TPAN), as depicted in Scheme 1, was successfully synthesized by C-N coupling between 2-(4-bromophenyl)-3,3-diphenylacrylonitrile (Br-TPAN) and N,N’-diphenyl-1,4-phenylenediamine (Fig. S1\textsuperscript{†}).

BP2TPAN is AIE-active, and its photoluminescence quantum yield (PLQY) of as prepared samples ($\Phi_{\text{PLQY}}$) up to 74.3%. BP2TPAN solids also exhibit obvious mechanochromism behavior; upon grinding, the emission maxima of the as prepared powders were changed from 540 to 580 nm. It’s also showed good thermal stabilities, with degradation temperature ($T_d$, at which a sample loses 5% weight) and glass transition temperature ($T_g$) being 414 and 96 °C, respectively. More importantly, the doped and nondoped OLED devices show different emission color, which is rarely reported.

2 Experimental

2.1 Materials

Sodium tert-butoxide, sodium hydride (60 wt% in mineral oil), palladium diacetate, N,N’-diphenylbenzidine, were obtained from TCI (Shanghai) Development Co., Ltd. Benzophenone and 4-(4-methylphenyl)acetonitrile were obtained from JSK chemical Scientific Ltd. Tri-tetrt-butylphosphine (0.49 M in toluene) was purchased from Puyang Huicheng Electronic Material Co., Ltd. Benzophenone, 15 mmol (603 mg) of NaH (60 wt% in mineral oil), and 50 mL of toluene under nitrogen immediately prior to used. THF was distilled from sodium benzophenone under nitrogen before used. The commercially available reagents were used without further purification.

2.2 Characterization

NMR spectrum was recorded on Bruker AMX-400 NMR spectrometer or Bruker AVANCE III HD600 600 MHz NMR instrument in deuterium solvent at room temperature. High-resolution mass spectrometry (HRMS) was performed on J&K scientific Ltd. Tri-tetrt-butylphosphine (0.49 M in toluene) was purchased from Puyang Huicheng Electronic Material Co., Ltd. Toluene was re-distilled under normal pressure from CaH\textsubscript{2} under nitrogen immediately prior to used. THF was distilled from sodium benzophenone under nitrogen before used. The commercially available reagents were used without further purification.

2.3 OLED fabrication

The device was fabricated by the following processes. First, ITO-coated glass substrates were cleaned successively using deionized water, acetone, and isopropanol in an ultrasonic bath and then dried in a drying cabinet followed by pretreatment with oxygen plasma. Then, the organic films of N,N,N’,N’-tetraakis(4-methoxy-phenyl)benzidine (MeO-TPD), 4,4’,4’’-tri(9-carbazoyl) triphenylamine (TCTA), AN2TPAN, 4,7-diphenyl-1,10-phenanthroline (Bphen), and 8-hydroxyquinolinolato-lithium (LiQ) were deposited by thermal evaporation under a base vacuum of about 10\textsuperscript{-6} Torr. Finally, aluminum (Al) metal was evaporated in another vacuum chamber without breaking the vacuum. The thicknesses of the films were determined by quartz thickness monitors. The active area of the EL device, defined by the overlap of the ITO and the cathode electrode, was 3 mm x 3 mm. Current density–voltage and current efficiency–current density characteristics was measured with a computer controlled Keithley 2400 Source Meter and BM-7A Luminance Colorimeter. The electroluminescence spectrum was measured by a Labsphere CDS-610. All measurements were carried out under air at room temperature without device encapsulation.

2.4 Synthesis

Synthesis of 2-(4-bromophenyl)-3,3-diphenylacrylonitrile (Br-TPAN). Into a 100 mL, two-necked, round bottom flask equipped with a condenser were placed 10 mmol (1.83 g) of benzophenone, 15 mmol (603 mg) of NaH (60 wt% in mineral oil), and 50 mL of toluene under nitrogen. The mixture was stirred at 80 °C for 10 min, then 11 mmol (2.17 g) of 4-bromobenzonitrile in 10 mL of toluene was added drop-wise over 20 min while maintaining at 80 °C. The reaction was lasted for another 10 h, cooling to room temperature, after solvent evaporation, 100 mL water and 50 ml chloroform respectively, were added. The organic layer was collected and washed with brine three times. The organic phase was then dried over anhydrous MgSO\textsubscript{4} and concentrated under vacuum. The crude product was purified by silica-gel column chromatography using petroleum ether/dichloromethane (DCM) as eluent (8/1–4/1). 3.05 g white solid was obtained in 84% yield. \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}), δ (TMS, ppm): 7.43 (m, 5H), 7.34 (d, 2H), 7.29 (m, 1H), 7.22 (t, 2H), 7.14 (d, 2H), 7.01 (d, 2H). \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}), δ (TMS, ppm): 158.41, 140.12, 138.72, 133.80, 131.70, 131.25, 130.67, 130.09, 129.87, 129.26, 128.49, 128.44, 122.55, 119.71, 119.71, 110.43.

Synthesis of BP2TPAN. Into a 50 mL two-necked round bottom flask were placed 1 mmol (336 mg) N,N’-diphenyl-1,4-
phenylenediamine, 2.5 mmol (900 mg) Br-TPAN, 3 mmol (288 mg) of t-BuONa, 10% mmol (22 mg) Pd(OAc)$_2$, 0.5 ml of P(t-Bu)$_3$ (0.45 M in toluene solution). The flask was evacuated under vacuum and flushed with dry nitrogen for three times. Then 30 ml fresh toluene was injected. Then the mixtures were heated to reflux under stirring for 36 h. Upon cooling to room temperature, the mixture was added 5 g silica (200–300 mesh), then, mixing uniform. Soxhlet extractor (250 mL) was placed 30 g silica (200–300 mesh), and the mixing mixture was added, the target compound BP2TPAN was extraction by 500 mL chloroform. Solvent was reduce evaporation to 100 mL, filtrate and filters wash with 20 ml chloroform three times, 621 mg yellow solid obtained in 69%. $^1$H NMR (600 MHz, THF-d$_8$, $\delta$) 7.58–7.39 (m, 14H, Ar-H), 7.32–7.02 (m, 26H, Ar-H), 6.98–6.88 (m, 4H, Ar-H), 6.68–6.58 (m, 2H, Ar-H); HRMS (MALDI, m/z): [M + H] calcd for C$_{288}$H$_{464}$N$_{4}$, 894.37225; found 894.28727 (calcd 894.37225). Elemental analysis calcd (%): C, 88.08%; H, 5.33%; N, 6.31%; found: C 88.08%, H 5.33%, N 6.31%.

3 Results and discussion

3.1 Synthesis

BP2TPAN was synthesized according to the route shown in Fig. S1 (ESI†). First, Br-TPAN was synthesized according to the literature procedure through a Knoevenagel condensation reaction between 4-bromophenylacetonitrile and benzophenone. Then, the target molecule, desirable compounds of BP2TPAN, was obtained in good yields by Buchwald–Hartwig C–N cross-coupling reaction between Br-TPAN and N,N′-diphenyl-1,4-phenylenediamine, using Pd(OAc)$_2$ and (t-Bu)$_3$P as catalysts under mild conditions.

BP2TPAN was characterized by standard spectroscopic methods, from which satisfactory analysis data corresponding to its molecular structures was obtained (see Fig. S2–4 ESI† for detail). For example, thin layer chromatography (TLC) plates indicated high purity of the isolated compounds in 1 : 1 petroleum ether–chloroform solvent mixture, as only a single spot was obtained for each compound (Fig. S2, ESI†). HRMS (Fig. S4 ESI†) peak [M$^+$ at m/z] 894.28727 (calcd 894.37225) confirms the formation of expected adducts. Moreover, elemental analysis results of C 88.08%, H 5.33%, and N 6.31% are highly consistent with their theoretical values. Results altogether indicates that BP2TPAN was sufficiently pure.

3.2 AIE

Our previous work have confirmed that TPAN is a typical crystallization-induced emission luminogens, which could yielded new AIE activated compounds when combined with aniline, diphenylamine, triphenylamine and carbazole et al. It is therefore envisioned that BP2TPAN is also an AIEgen. BP2TPAN shows two absorption peaks at 340 and 394 nm in THF (Fig. S5, ESI†). The former is attributable to the π–π* transition, the latter corresponds to the intramolecular charge transfer (ICT) transition. To verify AIE activity of BP2TPAN, it was slightly solubilized in organic solvents (THF), no visible emission was observed. However, as prepared (Asp) solid powder, it emits strong yellow light at 540 nm with quantum efficiency of 74.3%, indicating that it is AIE-active. Emission spectra of BP2TPAN in THF and THF/water mixtures were also measured. Water was chosen because it is a typical nonsolvent for BP2TPAN, in which BP2TPAN molecules will be aggregated. When the water fraction ($f_w$) is $\leq 60$%, only weak signals were recorded, since the molecules were dissolved in the mixture. However, when $f_w$ is increased to 70%, the emission intensity was swiftly boosted because of the molecular aggregation, giving a strong yellow emission at 560 nm (Fig. 1). Further addition of water continuously strengthened the emission intensity. In the 10 : 90 THF–water mixture, the emission was remarkably enhanced compared to that in THF (Fig. 1). The AIE nature of BP2TPAPN also visible from the emission contrast of BP2TPNP in THF and 10 : 90 THF–water mixture (Fig. 1B). To quantitatively evaluate the AIE effect, PLQY of the luminogens in both solution ($\Phi_{sol}$) and as-prepared solid states were determined. The $\Phi_{sol}$ values of BP2TPAN in THF are as low as 0.1%, thus confirming that it is practically non-luminescent nature in solvents. However, the values for as prepared solids increased to 74.3%, indicating AIE characteristic.

In order to better understand the nature of AIE and the geometry of BP2TPAN, quantum chemical optimization of their energy levels based on DFT/B3LYP/6-31G(d) was conducted. The optimized geometries and HOMO/LUMO plots of BP2TPAN are illustrated in Fig. 2. The molecule adopt highly twisted propeller-like non-planar conformations, which are favorable
for active intramolecular rotations of multiple phenyls in solutions, thereby powerfully dissipating the excitons energy and making them non-emissive in solvents. When aggregated as suspensions, powders, thin solid films, on one hand, the intramolecular rotations are impeded; on the other hand, the propeller-like configurations prevent the formation of detrimental excimers or exciplexes, thereby generating boosted emissions. Notably, the dihedral angles of the adjacent phenyl rings (P1 and P2) is \( \approx 20^\circ \), the non-planar conformations of biphenyl (P1–P2) decreased conjugated degree. Moreover, the electron clouds of the LUMO levels for BP2TPAN are mainly located on the electron-accepting TPAN peripheries; however, those of HOMO levels are dominated by orbitals from the electron-donating triphenylamine units. Generally, such electron distribution imparts an intrinsic intramolecular charge transfer property to dye molecules. This intramolecular charge transfer might balances hole and electron mobility in OLED devices and improve device performance.

### 3.3 Mechanochromic behavior

The remarkably twisted conformations and high PLQY of BP2TPAN make it potentially promising as mechanochromic luminogen with a high contrast. Upon grinding in an agate mortar with a pestle, the emission color of the as prepared solid changes from yellow to orange (Fig. 3A), with its emission maximum red shifted from 540 to 580 nm and the fluorescence lifetime values of the as prepared and ground amorphous solids of BP2TPAN are determined as 3.4 and 5.1 ns (Fig. 3D), respectively. The increased lifetime and decrease photoluminescence quantum yield of the ground sample suggested ground samples forms of excimers upon mechanical stimuli.

Although mechanochromic materials with switchable emission colors are reported in the literature, using external force to manipulate the fluorescent quantum yield is seldom reported. For BP2TPAN, PLQY of its the ground sample is lower than those as prepared samples, decrease of PLQY from 74.3% to 8.4% was observed. This result is consistent with the results from the naked eye observation, and indicating BP2TPAN has high contrast. This phenomenon may be attributed to crystals that readily break, trigger planarization of molecular conformation under external pressure, as well as the formation of excimers. Theoretically, excimer formation would promote fluorescence lifetime and decrease photoluminescence quantum yield. Herein the fluorescence lifetime values of the as prepared and ground amorphous solids of BP2TPAN are reported.

### 3.4 Thermal stability

Excellent thermal stability is highly desired for the device fabrication and optoelectronic applications of the molecular conjugates. Particularly, \( T_d \) of an organic luminophore is one of the most important factors that influence the device stability and lifetime. When device is heated above \( T_d \) of the organic luminophore, an irreversible failure can occur. Therefore, we checked the thermal property by measuring TGA and DSC of BP2TPAN. As shown in Fig. 4, BP2TPAN has high thermal stability with its \( T_d \) (defined as the temperature at which...
a sample loses its 5% weight) value 414 °C. DSC analysis reveals the \( T_g \) of BP2TPAN is 96 °C, thus suggesting their exceptional thermal stability. Such outstanding thermal properties and efficient solid-state emissions render its application in optoelectronic devices.

3.5 OLED

The high solid-state emission efficiency and good thermal properties of BP2TPAN prompted us to explore their potential application in OLED devices. We first fabricated multilayer nondoped OLEDs with a general device configuration of ITO/NPB (50 nm)/BP2TPAN (20 nm)/Bphen (40 nm)/LiF (1 nm)/Al (100 nm), where NPB and Bphen were chosen as hole- and electron-transporting layers, respectively. The electroluminescence (EL) spectrum, current density–voltage–luminance characteristics, current efficiency, power efficiency and external quantum efficiency of the devices are shown in Fig. 5 and S6† and summarized in Table 1. The nondoped devices (I), emit orange light with EL maxima \( (\lambda_{EL}) \) at 570 nm which are rather close to the PL emissions of their ground powders (580 nm). The device performance was moderate, with turn on voltages \( (V_{on}) \), maximum luminance \( (L_{max}) \), current efficiency \( (CE_{max}) \), power efficiency \( (PE_{max}) \), external quantum efficiency \( (EQE) \), and commission Internationale de L’Eclairage (CIE) being 4.8 V, 925 cd m\(^{-2}\), 2.9 cd A\(^{-1}\), 0.84 lm W\(^{-1}\), 1.1% and CIE (0.48, 0.51), respectively. Moreover, interestingly, when BP2TPAN doped into 2-methyl-9,10-di(2-naphthyl)anthracene [MADN] at a doping level of 3% (wt%) as an emitting layer, the device performance was dramatically improved by several times with
Table 1  OLED device performances of BP2TPAN$^a$

| Device | $V_{on}$, V | $\lambda_{max}$, nm | $L_{max}$, cd m$^{-2}$ | CE$\text{max}$, cd A$^{-1}$ | PE$\text{max}$, lm W$^{-1}$ | EQE, % | CIE, x, y |
|--------|-------------|---------------------|-------------------------|--------------------------|-----------------------------|--------|-----------|
| I      | 4.8         | 570                 | 925                     | 2.9                      | 0.84                        | 1.1    | 0.48, 0.51 |
| II     | 3.2         | 530                 | 15 070                  | 11.0                     | 7.5                         | 3.1    | 0.32, 0.55 |

$^a$ Device configuration: ITO/NPB (50 nm)/X (20 nm)/Bphen (40 nm)/LiF (1 nm)/Al (100 nm); X = BP2TPAN (device I), BP2TPAN (3%)·MADN (device II); abbreviations: $V_{on}$ = turn on voltages, $\lambda_{max}$ = EL peak, $L_{max}$ = maximal luminance, PE$\text{max}$ = maximal power efficiency, CE$\text{max}$ = maximal current efficiency, EQE = maximal quantum efficiency, CIE = Commission Internationale de l'Eclairage.

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