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Mechanochromism and electroluminescence in positional isomers of tetraphenylethylene substituted phenanthroimidazoles

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Abstract

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The study of aggregation-induced emission (AIE) luminogens has gained momentum due to its remarkable luminogenic properties and applications in mechano-sensors and organic light emitting diodes (OLEDs). In this article we have studied three positional isomers (*ortho*, *meta*, and *para*) of phenanthroimidazoles **3a–3c** and explored their AIE, mechanochromic and electroluminescence behavior. The phenanthroimidazoles **3a–3c** were synthesized by the Suzuki cross-coupling reaction of (2-bromo/3-bromo/4-bromo)phenathroimidazoles **2a–2c** with 4-(1,2,2-triphenylvinyl)phenylboronic acid pinacol ester in good yields. The phenanthroimidazoles **3a–3c** exhibit strong AIE. The mechanochromic study reveals reversible good color contrast mechanochromism between blue and green color. The *ortho* (**3a**) and *meta* (**3b**) isomers exhibit 98 nm grinding induced spectral shift whereas *para*-isomer (**3c**) shows 43 nm. Moreover, the **3a–3c** were explored as a non-doped blue emitters in efficient organic light-emitting diodes. Among the three emitters, **3c** provided high quantum efficiency of 4.0% in a non-doped blue device.

Introduction

Luminescent organic compounds are widely used in organic light-emitting diodes chemo-sensors and mechano-sensors.¹⁻³ (OLEDs). bio-imaging. The bio-sensing. mechanochromic materials change their colors in response to external mechanical stimuli (grinding, rubbing or crushing). The solid state emission is one of the important requirements for mechanochromic and OLED materials.⁴ Recently mechanochromic materials have gained significant importance due to their potential applications in the field of mechano-sensors, optical recording, security papers and optoelectronic devices.⁵⁻⁹ However, conventional organic compounds suffer from notorious effect called aggregation caused quenching (ACQ) which decreases their luminescence efficiency.¹⁰⁻¹² The aggregation induced emission (AIE) is promising solution of ACQ effect.¹³⁻¹⁶ The AIE molecules are highly emissive in the solid state and successfully used in the OLEDs and mechanochromic material applications.¹⁷⁻¹⁹

The luminescence properties of compounds in the solid-state are dependent on the molecular arrangement as well as intermolecular interactions. The mode of the molecular packing and/or conformation of the compound would affect the molecular stacking mode and effective conjugation which alter the luminescent properties. Thus, controlling the mode of molecular packing is significant in tuning the photophysical properties.²⁰⁻²²

The research on OLEDs have been continuously paying attention in both academia and industry due of their potential applications in solid state lighting and flat panel display.²³⁻²⁵ The phenanthroimidazole is an aromatic heterocycle, containing phenanthrene ring fused with imidazole ring, is endowed with ambipolar nature.²⁶⁻²⁷ Phenanthroimidazole has been extensively

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used to construct efficient blue emitters with fine color purities.²⁸⁻²⁹ Efforts are made to increase the efficiency of the OLED devices made from phenanthroimidazole derivatives.³⁰

The tetraphenylethylene (TPE) is AIE active molecule and has been explored in developing OLED and mechanochromic materials.^{17-19, 31-32} Our group is involved in the design and synthesis of TPE based luminogens for mechanochromism and OLEDs.³³⁻³⁵ Recently, we have reported TPE substituted phenanthroimidazoles as efficient mechanochromic materials and luminogens for OLED.³⁶⁻³⁷ We were further interested to study the positional effect of TPE unit by synthesizing positional isomers (*ortho/meta/para*) of TPE substituted phenanthroimidazole. The positional isomers (*ortho/meta/para*) may have different extent of conjugation and planarity which will endow interesting effect on their photophysical and mechanochromic properties. In ortho-isomer there is possibility of more twisting due to steric hindrance between TPE and phenanthroimidazole, and in *meta*-isomer less conjugation due to *meta*-position effect whereas *para*-isomer has possibility of high planarity and conjugation. The results obtained here support our hypothesis and we are successful in improving mechanochromic properties of TPE substituted phenanthroimidazoles. The phenanthroimidazoles 3a-3c show AIE, reversible mechanochromism and highly efficient blue emission in non-doped OLED devices. The ortho (3a) and *meta* (3b) isomers exhibit good mechanochromic spectral shift of 98 nm while *para* (3c) isomer shows 43 nm. Additionally, the *para* isomer (3c) showed high quantum efficiency (QE) of 4.0% in the non-doped blue device.

Synthesis

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The TPE substituted phenanthroimidazoles 3a-3c were synthesized by the Suzuki crosscoupling reaction of 4-(1,2,2-triphenylvinyl)phenylboronic acid pinacol ester with corresponding bromophenanthroimidazoles 2a-2c (ortho/meta/para) using Pd(PPh₃)₄ as catalyst which resulted 3a-3c in 51%, 65% and 67% yields, respectively (Scheme 1). The 9,10-phenanthroquinone 1 on condensation reaction with 4-aminobenzonitrile and respective bromobenzaldehydes (2bromobenzaldehyde for 2a, 3-bromobenzaldehyde for 2b, 4-bromobenzaldehyde for 2c) resulted 2a-2c in 70%, 86% and 76% yields, respectively. The positional isomers of phenanthroimidazoles 3a-3c were well characterized by ¹H NMR, ¹³C NMR, and high resolution mass spectrometry (HRMS).



Scheme 1: Synthetic route for the phenanthroimidazoles 3a-3c. (i) 4-aminobenzonitrile, 2bromobenzaldehyde (for 2a)/ 3-bromobenzaldehyde (for 2b)/ 4-bromobenzaldehyde (for 2c), acetic acid; (ii) 4-(1,2,2-triphenylvinyl)phenylboronic acid pinacol ester, K₂CO₃, Pd(PPh₃)₄, Toluene:Ethanol:Water (10.0 mL:4.0 mL:0.5 mL).

The thermal stability of organic materials is important for the device applications. To check the thermal stability of the phenanthroimidazoles 3a-3c, the thermogravimetric analysis (TGA) was performed (Fig. 1). The phenanthroimidazoles 3a-3c show excellent thermal stability and the thermal decomposition temperatures (T_d) corresponding to 5% weight loss under a nitrogen atmosphere are 383, 433 and 446 °C for 3a, 3b and 3c respectively (Table S1). The thermal stability of the phenanthroimidazoles 3a-3c follows the order 3c > 3b > 3a which reveals *para*-isomer has the highest thermal stability.



Fig. 1 Thermogravimetric analysis of phenanthroimidazoles **3a–3c** measured at a heating rate of 10 °C/ min under nitrogen atmosphere.

Photophysical properties

The phenanthroimidazoles 3a-3c are highly soluble in common organic solvents such as dichloromethane, tetrahydrofuran (THF), acetone, chloroform, etc., but they are insoluble in water and methanol due to hydrophobic aromatic rings. The non-rediative decay of the excited state energy through molecular rotations of phenyl rings make phenanthroimidazoles 3a-3c

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weakly emissive in THF solution. The phenanthroimidazoles **3a–3c** contains TPE unit and it is known for AIE active nature therefore we have studied their AIE properties. The AIE behavior was explored by preparing small aggregates of **3a–3c** molecules and studied by fluorescence spectroscopy. The aggregates were prepared in THF:water mixtures by gradual increase in the water percentage. The emission spectra do not show distinct change in emission wavelength and intensity, upto 80, 50 and 60% water fraction for **3a**, **3b** and **3c**, respectively. However, above 80, 50 and 60% water fraction for **3a**, **3b** and **3c** respectively, the enhancement in the emission intensity with red shift was observed (Fig. 2 and Fig. S1). This enhancement in the emission intensity may be due to suppressed free phenyl rings rotations due to the aggregate formation which reduces the non-radiative excited state energy loss. The weak fluorescence intensity of phenanthroimidazoles **3a–3c** in pure THF was enhanced in aggregated state (90% aqueous mixture) by 8, 50 and 18 folds, respectively (Fig. 2).

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Fig. 2 (A) Fluorescence spectra of (A) **3a**, and (B) **3b** in THF–water mixtures with different water fractions (10 μ M). (C) Plot of photoluminescence intensity (PL) vs. % of water fraction (f_w). Luminogen concentration: 10 μ M; intensity calculated at λ_{max} .

Mechanochromism

The high solid state emission and possibility of mechanochromic behavior encouraged us to study the mechanochromic property. The mechanical stimuli responsive properties of phenanthroimidazoles 3a-3c were studied by emission spectroscopy. The emission spectra of

phenanthroimidazoles 3a-3c as synthesized, grinded and annealed solids are shown in Fig. 3 and Fig. S2. The phenanthroimidazoles 3a-3c show good color contrast reversible mechanochromic behavior between blue and green color. The pristine solids of phenanthroimidazoles 3a-3c show blue color emission and emission maxima at 450 nm for 3c whereas 3a and 3b show dual emissions at 370, 396 nm and 371, 394 nm respectively. Upon grinding using a mortar and pestle, the blue emitting pristine solids were converted to a yellowish green emitting solids and the emission peaks were red-shifted to 493, 494, and 493 nm for **3a**, **3b** and **3c** respectively (Fig. 3 and Fig. S2). The mechanochromic effect of **3a**, **3b** and **3c** shows spectral red shift of 98, 98 and 43 nm, respectively. The high spectral shift in 3a may be due to a highly twisted geometry possibly created by the steric hindrance between TPE and phenanthroimidazole. However, in case of *meta*-isomer (3b), less effective conjugation due to *meta*-position effect and high twisting results in high grinding induced spectral shift. The, *para*-isomer (3c) possibly has a high planarity and high effective conjugation which results in less grinding induced spectral shift. The changes in emission intensity during mechanochromic behavior were evaluated by calculating the fluorescence quantum yields in the solid state. The fluorescence quantum yields of pristine solids **3a**, **3b** and **3c** were 0.73, 0.54, and 0.73 respectively. The pristine solids decrease their fluorescence quantum yields upon grinding and were observed 0.69, 0.57, and 0.46 for **3a**, **3b** and **3c** respectively. The grinded solids can be reverted to their pristine form by annealing or fuming with solvent vapor. The grinded solid of 3c required annealing at 160 °C for 15 min whereas **3a** and **3b** required 240 °C annealing for 15 min. The fumigation with dichloromethane vapor for 2 min restored the original blue emission for all phenanthroimidazoles 3a-3c. The annealed samples of **3a** and **3b** show emission tails which may be due to the incomplete reversion of grinded solids to their pristine form.



Fig. 3 Emission spectra of (A) **3a**, and (B) **3b** as synthesized, grinded and annealed solids and photograph taken under 365 nm UV illuminations.

The powder XRD study of pristine, ground and fumed form of phenanthroimidazoles 3a-3c was performed for understanding the mechanochromic behavior. A sharp diffraction pattern of the pristine samples suggested its crystalline nature (Fig. 4 and Fig. S3). However, the ground samples exhibited a broad diffused pattern indicating its amorphous nature. The crystalline nature of the solid could be restored by annealing or fumigation, giving sharp peaks in the diffractogram as in pristine forms. The mechanochromic behavior of the phenanthroimidazole solids can be confirmed from the PXRD studies and is associated to the reversible morphological change from crystalline to amorphous form which further suggests the change in molecular arrangement due to grinding.

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Fig. 4 PXRD curves of (A) **3a**, and (B) **3b** as synthesized, grinded and annealed form. Device performances

The phenanthroimidazoles **3a-3c** were tested as non-doped emitters in the OLEDs based on the AIE characteristics. A device structure of confining holes and electrons in the emitting layer was adopted using 1,3-bis(N-carbazolyl)benzene (mCP) and diphenyl-4-triphenylsilylphenylphosphine oxide (TSPO1) carrier blocking layers to compare three emitters as non-doped emitters. Current density (**J**) and luminance (**L**) of the three devices measured by voltage scan method are presented in **Fig. 5**. The **J** of the **3a** and **3c** devices was quite similar, but the **J** of the **3b** device was relatively lower than that of other devices. The low **J** of the **3b** devices can be correlated with the single carrier current density of the three emitters (**Fig. 6**). The electron current density of **3a** and **3c** was much higher than that of **3b** as shown in the single carrier device data. As electrons are majority carriers in the devices by the electron deficient phenanthroimidazole and benzonitrile moieties, the electron density dominated the current

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density of the devices for **3a–3c**. Therefore, **3a** and **3c** showing high electron current density increased the current density of the devices. *Meta-* orientation of the two building blocks in **3b** hindered the hole and electron transport in the emitting layer.



Fig. 5 Current density-voltage-luminance plot of non-doped 3a, 3b and 3c devices.



Fig. 6 Current density-voltage plots of hole only (a) and electron only (b) devices of non-doped3a, 3b and 3c.

QE of the non-doped devices was collected according to luminance in Fig. 7. The QE was relatively high in the **3a** device, but it was low in the **3b** device. Two main parameters, PL quantum yield and carrier balance, should be considered to interpret the QE data of the three emitters. The PL quantum yields of **3a**, **3b**, and **3c** compounds were 0.73, 0.54, and 0.73, respectively, indicating that **3a** and **3c** are potentially better than **3b** in terms of light-emitting efficiency of the emitters. As expected from the PL quantum yields, the maximum QE values of **3a**, **3b** and **3c** were 4.0, 2.4, and 3.2%, respectively. The relatively high QE of **3a** compared with that of **3c** is due to better carrier balance in the **3a** device in spite of similar PL quantum yields. In the case of **3a**, both hole transport and electron transport were better than those of **3b** and **3c**, which balanced carriers in the emitting layer and increased the QE. In the case of **3c**, the hole current density was too low, which decreased the QE of the **3c** device because of poor charge balance. Therefore, the order of QE of the non-doped device was **3a>3c>3b**.



Fig. 7 Quantum efficiency-luminance plot of non-doped 3a, 3b and 3c devices.

The non-doped OLEDs were all sky-blue devices and exhibited sky-blue emission as presented in Fig. 8. Peak wavelengths of the 3a, 3b, and 3c OLEDs were 480 nm, 483 nm, and

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482 nm, respectively, which corresponded to color coordinates of (0.199, 0.332), (0.212,0.372) and (0.215,0.313), proving sky-blue emission color of the fabricated devices. There was no large difference of the emission spectra because the same chromophore was used in the molecular structure. The electroluminescence emission maxima are close to the emission maxima in the amorphous solids which suggest the amorphous nature of the compounds in the OLED devices.



Fig. 8 Electroluminescence spectra of non-doped 3a, 3b and 3c devices.

Conclusion

In conclusion, we have synthesized mechanochromic and electroluminescent positional isomers of tetraphenylethylene substituted phenanthroimidazoles **3a–3c** by Pd-catalyzed Suzuki cross-coupling reaction. The phenanthroimidazoles **3a–3c** show good thermal stability and *para*-isomer exhibits highest thermal stability. The positional isomers **3a–3c** in the aggregated state show strong emission due to AIE compared to their emission in the solution state. The positional isomers **3a–3c** show reversible mechanochromism with good color contrast between blue and green color. The use of *ortho* and *meta* isomers is found to be good strategy than *para* isomer for

the development of mechanochromic materials and isomers 3a and 3b exhibit spectral shift of 98 nm whereas para isomer 3c exhibit spectral shift of 43 nm. Additionally, the 3a emitter show high QE of 4.0% in the non-doped blue device.

Experimental details

General methods

Chemicals were used as received unless otherwise indicated. All oxygen or moisture sensitive reactions were performed under argon atmosphere. ¹H NMR (400 MHz), and ¹³C NMR (100 MHz) spectra were recorded on the Bruker Advance (III) instrument by using CDCl₃. ¹H NMR chemical shifts are reported in parts per million (ppm) relative to the solvent residual peak (CDCl₃, 7.26 ppm). Multiplicities are given as: s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), dt (doublet of triplets), m (multiplet), and the coupling constants, *J*, are given in Hz. ¹³C NMR chemical shifts are reported relative to the solvent residual peak (CDCl₃, 7.0 ppm. Thermogravimetric analyses (TGA) were performed on the Metler Toledo Thermal Analysis system. The measurements were done at heating rate of 10 °C/minute and heated upto 800 °C. UV-visible absorption spectra were recorded on a Carry-100 Bio UV-visible Spectrophotometer. Emission spectra were taken in a fluoromax-4p fluorimeter from HoribaYovin (model: FM-100). The excitation and emission slits were 2/2 nm for the emission measurements. All of the measurements were done at 25 °C. HRMS were recorded on Brucker-Daltonics, micrOTOF-Q II mass spectrometer.

Synthesis:

Synthesis and characterization of phenanthroimidazoles 3a and 3b:

General procedure for phenanthroimidazoles 3a and 3b: $Pd(PPh_3)_4$ (0.004 mmol) was added to a well degassed solution of corresponding bromophenanthroimidazoles 2a and 2b (0.4 mmol), 4-(1,2,2-triphenylvinyl)phenylboronic acid pinacol ester (0.48 mmol), K₂CO₃ (1.2 mmol) in a mixture of toluene (12 mL)/ ethanol (4.0 mL)/ H₂O (1.0 mL). The resulting mixture was stirred at 80 °C for 24 h under argon atmosphere. After cooling, the mixture was evaporated to dryness and the residue subjected to column chromatography on silica (hexane-DCM 50:50 in vol.) to yield the desired phenanthroimidazoles 3a and 3b as colorless powders. The compounds were recrystallized from DCM:ethanol (8:2) mixtures.

3a: Yield: 51.0%. ¹H NMR (400 MHz, CDCl₃, 25 °C): δ 8.85 (d, 2H, *J*= 8 Hz), 8.77 (d, 1H, *J*=8 Hz), 7.68-7.79 (m, 3H), 7.58-7.62 (m, 1H), 7.41-7.47 (m, 2H), 7.31-7.38 (m, 3H), 7.22-7.25 (m, 1H), 7.00-7.07 (m, 7H), 6.88-6.96 (m, 6H), 6.80-6.83 (m, 2H), 6.70-6.74 (m, 4H), 6.48-6.65 (m, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃, 25 °C): δ 151.3, 143.6, 143.3, 143.1, 143.1, 141.4, 141.2, 140.8, 140.2, 137.8, 137.7, 132.6, 132.4, 131.2, 131.0, 130.9, 130.7, 130.4, 129.6, 129.4, 128.8, 128.4, 128.3, 127.8, 127.7, 127.6, 127.4, 127.3, 127.1, 126.6, 126.4, 126.2, 126.1, 125.8, 125.2, 124.3, 123.1, 122.9, 122.5, 120.7, 117.9, 112.3 ppm; HRMS (ESI): calcd. for C₅₄H₃₅N₃: 7 48.2723 (*M*+*Na*)⁺, found 748.2702.

3b: Yield: 65.0%. ¹H NMR (400 MHz, CDCl₃, 25 °C): δ 8.86 (d, 1H, *J*= 8 Hz), 8.80 (d, 1H, *J*=8 Hz), 8.72 (d, 1H, *J*= 8 Hz), 7.88-7.91 (m, 2H), 7.66-7.76 (m, 5H), 7.53-7.58 (m, 2H), 7.28-7.34 (m, 3H), 7.22 (d, 1H, *J*= 8 Hz), 7.03-7.16 (m, 18H) ppm; ¹³C NMR (100 MHz, CDCl₃, 25 °C): δ 149.9, 141.5, 137.6, 133.4, 132.8, 132.7, 131.9, 131.5, 129.5, 129.4, 128.4, 127.6, 127.1, 127.0, 126.7, 126.5, 126.0, 125.5, 124.7, 124.4, 123.2, 122.9, 122.3, 120.7, 117.7, 113.5 ppm; HRMS (ESI): calcd. for C₅₄H₃₅N₃: 7 48.2723 (*M*+*Na*)⁺, found 748.2708.

Device fabrication and measurements

The device structure of the **3a-3c** devices was as follows.

Device fabrication and device characterization were already reported in our previous work.³⁸ All emitters were vacuum deposited using a vacuum thermal evaporation process. Device structure of the non-doped devices was indium tin oxide (150 nm)/poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (60 nm)/4,4'-cyclohexylidenebis[N,N-bis(4-methylphenyl)aniline] (20 nm)/mCP (10 nm)/emitter (25 nm)/TSPO1 (5 nm)/2,2',2"-(1,3,5-benzinetriyl)-tris(1-phenyl-1-H-benzimidazole) (30 nm)/LiF (1.5 nm)/Al (200 nm).

Supplementary data

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Electronic supplementary information (ESI) available: NMR spectra and computational data for **3a** and **3b** and AIE and mechanochromic effect of **3c**.

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