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SYNTHESIS AND OPTICAL PROPERTIES OF POLYARYLIMIDAZOLE DERIVATIVES CONTAINING NAPHTHYL GROUPS IN THE 4,5-POSITIONS FOR COATED AND NON-DOPED OLED MATERIALS

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Abstract – In this study, we describe the development of an imidazole emitting material, characterized by low concentration quenching via a bulky substituent group, for coated and non-doped OLEDs. Specifically, we synthesized novel 1,2,4,5-tetraarylimidazoles that have naphthyl groups replacing the aryl groups at the 4,5-positions and investigated their optical, concentration quenching, thermal, and coating properties.

Recently, emitting materials, which are characterized by low concentration quenching properties, used in non-doped organic light-emitting diodes (OLEDs) have attracted significant attention.[1] However, numerous conventional fluorescent dyes exhibit strong luminescence in solution but aggregation-caused quenching (ACQ) occurs in the solid state. The formation of delocalized excitons via intermolecular π–π stacking interactions may cause ACQ and consequently obstruct the application of fluorescent dyes in OLEDs.[2] For molecules that exhibit low concentration quenching characteristics, we must restrict multiple aryl rotor intramolecular rotations against the stator in the aggregate state.

Among these molecules, previous studies have observed and investigated polyaryl-substituted heterocycles based on pyrrole,[3] oxazole,[4] thiazole,[5] 1,3,4-oxadiazole,[6] 1,2,5-oxadiazole,[7] 1,2,5-thiadiazole,[8] and 1,2,4-triazole,[9] which are especially useful *N*-containing five-membered heterocyclic compounds to emitting materials in organic EL devices. On the other hand, imidazole derivatives in heterocyclic chemistry are characterized by specific properties, such as easy ring construction and numerous useful methods to introduce multiple aryl groups onto the imidazole ring; these characteristics have been reported in several OLED materials.[10] However, no studies exist on imidazole derivatives that have polycyclic-fused aromatic rings, which are introduced directly onto the 4,5-positions of an imidazole ring.

In this study, to develop the coatable imidazole dyes characterized by low concentration quenching, we synthesized and investigated the properties of 1,2,4,5-polyarylimidazoles that bear 1- and 2-naphthyl groups at the 4,5-positions (**1b**, **1c**) using the Davidson modification[11] of Radziszewski imidazole synthesis.[12]

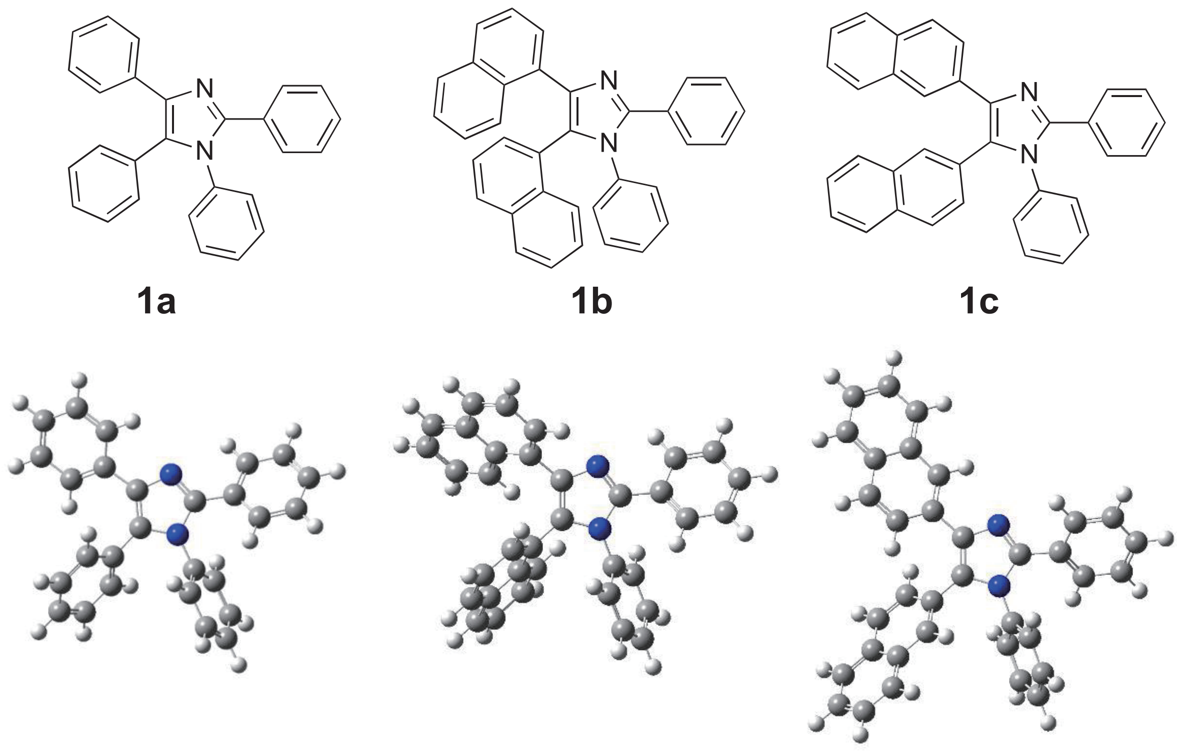
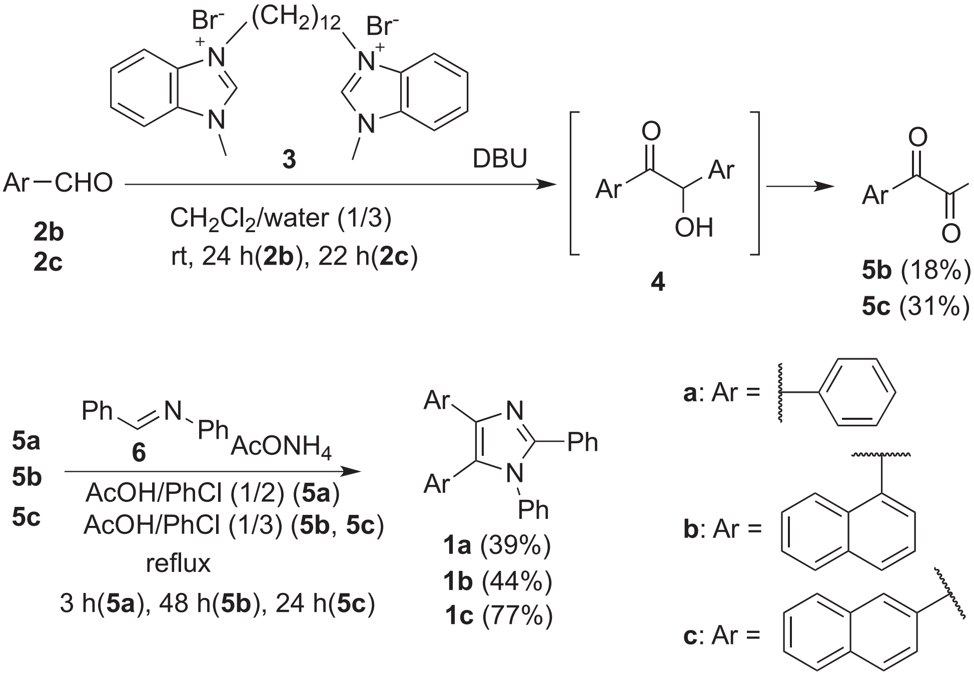


Figure 1. Chemical structures and optimized structures of 1,2,4,5-tetraarylimidazoles **1a-1c** calculated with Gaussian 09W DFT (B3LYP/6-31G(d))

As previously reported,[13] we synthesized benzimidazolium salt **3** from 1-methylbenzimidazole and 1,12-dibromododecane in acetonitrile. In a two-layer medium (dichloromethane/water = 1/3), precursor **5** was inducted via naphthoin **4** produced by an aldehyde **2** benzoin condensation reaction using catalytic *N*-heterocyclic carbene, which was generated from **3** in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU).[14] We obtained 4,5-dinaphthylimidazoles **1a**, **1b**, and **1c** in moderate yields using a cyclization reaction between precursor **5** and Schiff base **6** in the presence of ammonium acetate (Figure 1, Scheme 1).



Scheme 1

The optical and thermal properties of the imidazole derivative **1** are listed in Table **1**. The maximum absorption and excitation wavelengths were longer in the order of **1a**, **1b**, **1c**, which depends on the difference in conjugation length of **1**. On the other hand, The fluorescence of **1b** was observed at a longer wavelength than that of **1c**. It was because that π-extended system showed smaller Stokes shift. Also, the fluorescence wavelength in the solid state shifted to a shorter wavelength compared with the solution state. Changes in the excited state may have caused molecule aggregation.[15]

Table 1. Optical and thermal properties of imidazole derivatives

	Optical properties					Thermal propeties		
Imidazoles	Absorption[a]		Excitaion[b] Solution	Emission[b] Solution	Solid	T_m[c]	T_g[c]	T_d[d]
	λ_{max} (nm)	ε (M^{-1} cm^{-1})	λ_{max} (nm)	λ_{max} (nm)	λ_{max} (nm)	(°C)	(°C)	(°C)
1a	288	20,282	288	388	384	219	-	255
1b	295	26,814	300	425	410	230	88	311
1c	310	23,773	314	412	398	170	86	294

[a] Absorption spectra were measured in CH_2Cl_2 (1.0 x 10^{-5} M).
[b] Excitaion and emission spectra were measured in CH_2Cl_2 (1.0 x 10^{-7} M). Emission spectra were obtaind from λ_{max} observed in the UV/Vis spectra.
[c] Melting point (T_m) and Glass-transition point (T_g) were measured by DSC at heating rate of 1 °C/min under N_2.
[d] Decompositon point (T_d) were measured by TG at heating rate of 10 °C/min under N_2. T_d was the temperature when the mass decreased by 5%.

For the thermal properties, the melting (T_m) and decomposition point (T_d) was nearly equivalent to general OLED materials. The glass transition temperature (T_g), which is necessary for coated OLED materials, appeared for **1b** and **1c**, but we did not observe it in **1a**. The naphthyl group at the 4,5-positions in the imidazole increased the substituent twist at the 1,4,5-positions in the imidazole ring, which produced the amorphous properties of **1b** and **1c** compared with **1a**.[16]

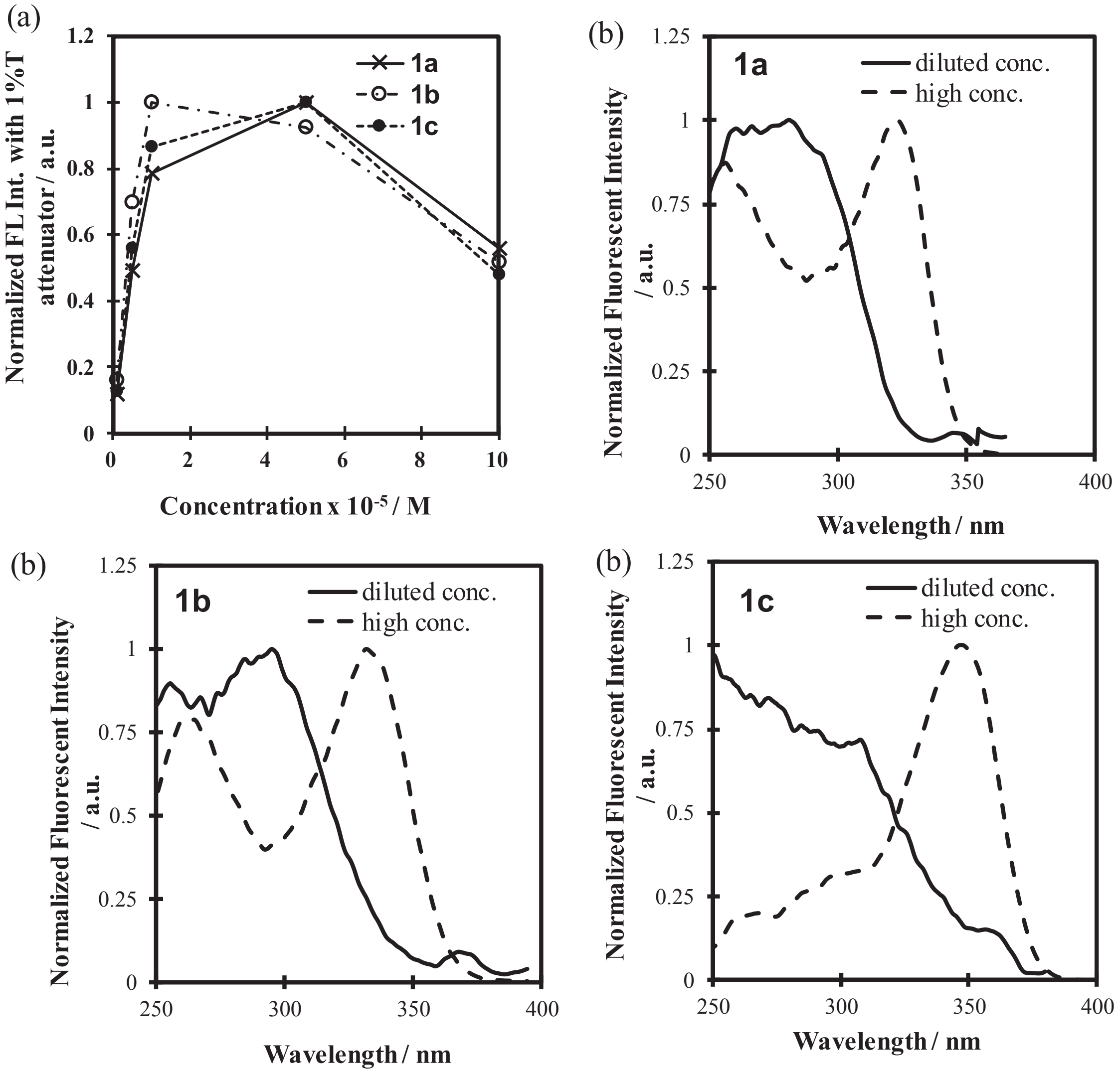


Figure 2. (a) Correlations between the concentration of **1** in CH_2Cl_2 and fluorescent intensity. We observed λ_{ex} at 288 nm (**1a**), 295 nm (**1b**), and 310 nm (**1c**). (b) Comparison between the excitation spectra of the diluted (1×10^{-8} M) and high concentration (1×10^{-4} M) compound in CH_2Cl_2. We observed λ_{em} at 388 nm (**1a**), 425 nm (**1b**), and 412 nm (**1c**).

We investigated the relationship between the concentration of **1** and the normalized fluorescence intensity using FL spectrometry with a 1% T attenuator on the emission detector (Figure 2a). Fluorescence quenching of **1** occurred from 1.0 to 5.0×10^{-5} M, and the fluorescent intensity decreased by half at

1.0×10^{-4} M. However, excitation spectra were completely different between the diluted and high concentration solution (Figure 2b). The decrease in fluorescence intensity at a high concentration suggests a possible internal shielding effect rather than concentration quenching.[17]

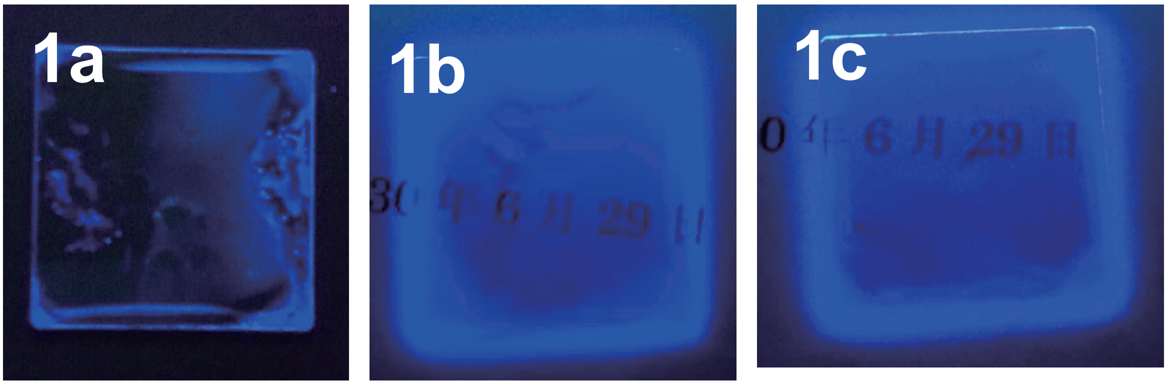


Figure 3. Emission of the **1** spin-coated thin film quartz plate (2 × 2 × 0.1 cm) exposed to UV (254 nm, 4 W) irradiation

Figure 3 displays the emission of the **1** spin-coated quartz plate (2 × 2 × 0.1 cm) exposed to UV (254 nm, 4 W) irradiation. Compounds **1b** and **1c** formed a thin film via spin coating but **1a** did not form this thin film. These compounds correlate with the T_g and crystallinity. The spin-coated substrates of **1b** and **1c** emitted fluorescence when exposed to UV irradiation and have potential as a material for OLEDs after exposure to the spin-coated method.

We synthesized 1,2,4,5-tetraarylimidazole derivatives (**1b**, **1c**) with a naphthyl group at the 4,5-positions in the imidazole ring. With the 4,5-position substituent effect, the absorption and fluorescence wavelengths shifted to longer wavelengths with an appearance of amorphous properties. Furthermore, imidazole derivatives were emitted even in the aggregated and solid state. These results suggest that these materials could possibly be used as coated and non-doped OLED materials. However, the low concentration quenching and T_g properties are insufficient.[17] Further research on imidazole-based novel organic light-emitting materials and their applications for OLED devices are currently in progress.

EXPERIMENTAL

General Methods and Materials. ^{1}H NMR spectra were recorded using a JEOL ECS-400 instrument. We report chemical shifts in parts per million (ppm) relative to a tetramethylsilane internal standard (0.0 ppm). We report ^{1}H NMR data as follows: chemical shifts (δ ppm), integration, multiplicity, and coupling constants (*J*, Hz). We report ^{13}C NMR data as follows: chemical shifts (δ ppm). Multiplicities are reported using the following abbreviations: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; and br, broad. We report only the strongest and/or structurally relevant IR peaks (cm^{-1}) using a PerkinElmer Spectrum One with a Universal ATR. All mass spectra were measured on a Bruker Autoflex II MALDI-TOF mass spectrometer. Column chromatography was performed using silica gel (BW-300; Fuji Silysia Chemical, Ltd.). UV-vis spectra were recorded using a Jasco V-670 UV-Vis Spectrophotometer. Fluorescent and excitation spectra were measured with a PerkinElmer LS55 Fluorescence Spectrometer. We analyzed differential scanning calorimetry and thermogravimetry using a Rigaku DSC8231 and TG8120, respectively. Elemental analyses were performed at A Rabbit Science Japan Co., Ltd. Commercially available reagents were used without further purification. Reactions were monitored with thin-layer chromatography that was performed on a 0.25 mm silica gel plate (60F$_{254}$) visualized via fluorescence when exposed to 254 and 356 nm irradiation.

1,2-Di(naphthalen-1-yl)ethane-1,2-dione (5b): Benzimidazolium salt **3** (1.779 g, 3 mmol) and water (30 mL) were added to a solution of 1-naphthaldehyde (4.782 g, 30 mmol) that was dissolved in CH_2Cl_2 (10 mL) and stirred for 1 h. After the addition of DBU (0.931 g, 6 mmol), the solution was stirred for 24 h at room temperature. CH_2Cl_2 (20 mL) was added to the reaction mixture and washed 5 times with water (50 mL). The organic phase was then dried over $MgSO_4$ and concentrated under reduced pressure. We then added MeOH and filtered the mixture to obtain a 1,2-di(naphthalen-1-yl)ethane-1,2-dione **5b** (0.445 g) residue. The filtrate was concentrated under reduced pressure; acetone was added and filtered to obtain **5b** (0.390 g) separately. Yellow powder **5b** (0.835 g, 18%) was recrystallized from EtOH to yield **5b** as yellow cubes (0.614 g). mp 191.0–193.0 °C (lit.[14] 192–194 °C). ATR-FT-IR 3051 (C—H), 1655 (C═O), 1568, 1506 (C═C), 762,693 cm^{-1} (C—H); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44–7.52 (2H, m, 6-H), 7.60–7.67 (2H, m, 7-H), 7.71–7.80 (2H, m, 3-H), 7.95 (2H, d, J = 7.6 Hz, 5-H), 7.99–8.05 (2H, m, 2-H), 8.15 (2H, d, J = 8.4 Hz, 4-H), 9.35 (2H, d, J = 8.8 Hz, 8-H); The HPLC (GL Science Inertsil® Diol, 5 μm, 4.6 mm × 250 mm, Hexane/$CHCl_3$ = 1/1, flow rate of 1 mL/min, 280 nm) retention time was 4.05 min.

1,2-Di(naphthalen-2-yl)ethane-1,2-dione (5c): Benzimidazolium salt **3** (1.7779 g, 3 mmol) and water (30 mL) were added to a solution of 2-naphthaldehyde (4.781 g, 30 mmol) that was dissolved in CH_2Cl_2 (9 mL) and stirred for 1 h. After the addition of DBU (0.932 g, 6 mmol), the solution was stirred for 22 h at room temperature. CH_2Cl_2 (20 mL) was added to the reaction mixture and washed 5 times with water (50 mL). The organic phase was then dried over $MgSO_4$ and concentrated under reduced pressure. Then, EtOH was added, and the mixture was filtered to obtain a 1,2-di(naphthalen-2-yl)ethane-1,2-dione **5c** (0.281 g) residue. The filtrate was concentrated under reduced pressure, and the addition of MeOH and filtration was repeated 5 times to obtain **5c** (1.156 g) separately. Yellow **5c** powder was recrystallized from EtOH to obtain **5c** as yellow needles. mp 155.0–156.0 °C (lit.[18] 157–158 °C). ATR-FT-IR 3062 (C—H), 1658 (C═O), 1577, 1505 (C═C), 773, 714 cm^{-1} (C—H); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.58 (2H, m, 6-H), 7.58–7.67 (2H, m, 7-H), 7.83–7.94 (4H, m, 4,5-H), 7.98 (2H d, J = 8.8 Hz, 8-H), 8.15 (2H, dd, $J = 8.8$, 2.0 Hz, 3-H), 8.45 (2H, d, $J = 2.0$ Hz, 1-H); The HPLC (GL Science Inertsil® Diol, 5 μm, 4.6 mm × 250 mm, Hexane/$CHCl_3$ = 1/1, flow rate of 1 mL/min, 280 nm) retention time was 4.03 min.

1,2,4,5-Tetraphenyl-1*H*-imidazole (1a): Benzaldehyde (0.574 g, 5 mmol) and aniline (0.474 g, 5 mmol) in an acetic acid (2 mL)–chlorobenzene (4 mL) solution were refluxed for 1 h. Then, benzil (1.052 g, 5 mmol) and ammonium acetate (0.770 g, 10 mmol) were added and refluxed for another 2 h. MeOH was added to the solution and filtered to collect 1,2,4,5-tetraphenyl-1*H*-imidazole **1a** (0.731 g, 39%). Compound **1a** was recrystallized from hexane-CH_2Cl_2 to obtain **1a** as pale yellow needles. mp 214–216 °C (lit.[19] 216–217 °C). ATR-FT-IR 3050 (C─H), 1596 (C═C), 690 cm^{-1} (C─H); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.12–7.47 (16H, m, 2,3,4,5,6-H in 1-Ph, 3,4,5-H in 2-Ph, 2,3,4,5,6-H in 4-Ph, 2,3,4,5,6-H in 5-Ph), 7.49 (2H, dd, $J = 8.0$, 1.3 Hz, 2,6-H in 2-Ph); ^{13}C NMR (100.5 MHz, DMSO-d_6) δ 126.19 (2C), 126.30, 128.00 (4C), 128.10 (3C), 128.14, 128.26, 128.31 (2C), 128.59 (3C), 128.98 (2C), 130.22, 130.97 (2C), 131.12, 134.22, 136.48, 136.66, 145.83; MALDI-TOF-MS (dithranol) *m/z* 373 ([M+1]$^+$).

4,5-Di(naphthalen-1-yl)-1,2-diphenyl-1*H*-imidazole (1b): 5b (0.590 g, 1.9 mmol) and benzilideneanilne (0.352 g, 1.9 mmol) in a solution of acetic acid/chlorobenzene (2/1, 2.5 mL) was refluxed, and then, ammonium acetate (0.302 g, 3.8 mmol) was added. After the mixture was refluxed for 24 h, benzilideneanilne (0.150 g, 1.0 mmol) was added and the mixture was refluxed again for 24 h. MeOH was added to the mixture and filtered to obtain imidazole **1b** derivative (0.399 g, 44%) residues. The **1b** was recrystallized from EtOH to obtain **1b** as pale yellow cubes. mp 231–232 °C. ATR-FT-IR 3055 (C─H), 1592, 1494 (C═C), 768, 691 cm^{-1} (C─H); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.09–7.41 (13H, m, 2,3,4,5-H in 1-Ph, 3,4,5-H in 2-Ph, 3,6,7-H in 4-Napht, 3,6,7-H in 5-Napht), 7.41–7.55 (4H, m, 2,5-H in 4-Napht, 1,4-H in 5-Napht), 7.60 (1H, d, $J = 7.1$ Hz, 1-H in 1-Ph), 7.67–7.80 (4H, m, 2,5-H in 4-Napht, 2,5-H in 5-Napht), 7.85 (1H, d, $J = 8.0$ Hz, 8-H in 4-Napht), 8.57 (1H, d, $J = 8.0$ Hz, 8-H in 5-Napht); ^{13}C NMR (100.5 MHz, DMSO-d_6) δ 124.79, 124.82, 125.22, 125.55, 125.65, 125.70, 126.25, 126.63, 127.07, 127.18, 127.34, 127.74, 127.87, 127.96 (2C), 128.06 (3C), 128.16 (3C), 128.39, 128.76 (2C), 130.36, 130.63, 130.83, 131.75, 131.83, 132.50 (2C), 133.20, 136.80, 138.80, 145.98. MALDI-TOF-MS (dithranol) *m/z* 472 ([M]$^+$). The HPLC (GL Science Inertsil® Diol, 5 μm, 4.6 mm × 250 mm, Hexane/$CHCl_3$ = 1/1, flow rate of 1 mL/min, 280 nm) retention time was 5.36 min. *Anal.* Calcd for $C_{35}H_{24}N_2$: C, 88.95; H, 5.12; N, 5.93. Found: C, 89.00; H, 5.22; N, 5.78.

4,5-Di(naphthalen-2-yl)-1,2-diphenyl-1*H*-imidazole (1c): 5c (0.500 g, 1.69 mmol) and benzylideneanilne (0.444 g, 2.4 mmol) in a solution of acetic acid/chlorobenzene (2/1, 3 mL) were refluxed, and then, ammonium acetate (0.381 g, 4.8 mmol) was added. The mixture was refluxed for 24 h. CH_2Cl_2 (30 mL) was added to the reaction mixture and washed 8 times with water (100 mL). The organic phase was then dried over $MgSO_4$ and concentrated under reduced pressure. The solution was purified via column chromatography. Compound **5c** (35 mg) was recovered from the CH_2Cl_2 eluate, whereas **1c** as yellow powder (581 mg, 77%) was obtained from the EtOAc eluate. The powder was then recrystallized from EtOH yielding **1c** as pale yellow needles. mp 169.5–170 °C. ATR-FT-IR 3054 (C─H), 1598, 1495 (C═C), 818, 697 cm^{-1}(C─H); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.26–7.57 (16H, m, 2,3,4,5,6-H in 1-Ph, 3,4,5-H in 2-Ph, 1,3,6,7-H in 4-Napht, 1,3,6,7-H in 5-Napht), 7.65–7.93 (7H, m, 2,6-H in 2-Ph, 4,5,8-H in 4-Napht, 4,5-H in 5-Napht), 8.16 (1H, s, 8-H in 5-Napht); ^{13}C NMR (100.5 MHz, DMSO-d_6) δ 124.71, 124.79, 125.48, 126.06, 126.33, 126.64, 127.28, 127.31, 127.48, 127.64 (2C), 127.80 (2C), 128.04 (3C), 128.23 (3C), 128.32, 128.64 (2C), 129.04 (2C), 130.02, 130.40, 131.47, 131.66, 131.82, 132.18, 132.44, 132.90, 136.51, 137.05, 146.33; MALDI-TOF-MS (dithranol) *m/z* 472 ([M]$^+$). The HPLC (GL Science Inertsil® Diol, 5 μm, 4.6 mm × 250 mm, Hexane/$CHCl_3$ = 1/1, flow rate of 1 mL/min, 280 nm) retention time was 5.17 min. *Anal.* Calcd for $C_{35}H_{24}N_2$: C, 88.95; H, 5.12; N, 5.93. Found: C, 88.72; H, 5.15; N, 5.73.

Preparation of 1 spin-coated quartz plates. Quartz plates (2 × 2 × 0.1 cm) were irradiated with an ultrasonic wave in $CHCl_3$ for 10 min and then boiled for 10 min in 2-propanol. After air drying, they were cleaned for 10 min via a UV-O_3 cleaning system (Model 208 UV-O_3 Cleaning System, TECHNOVISION, INC.). Then, 2 drops of 1.0 wt% imidazole **1** toluene solution, which was filtered via a syringe filter (HLC-DISKR 13, 0.2 μm, hydrophobic, KANTO CHEMICAL CO., INC.), were placed onto a quartz plate and then immediately spin-coated. After reaching 1,000 rpm, the spin rates remained at 1,000 rpm for 10 s and were then stopped. After spin coating, the quartz plates were annealed on a hot plate at 180 °C for 10 min.

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