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Swallow-Tailed Alkyl and Linear Alkoxy-Substituted Dibenzocoronene Tetracarboxdiimide Derivatives: Synthesis, Photophysical Properties, and Thermotropic Behaviors

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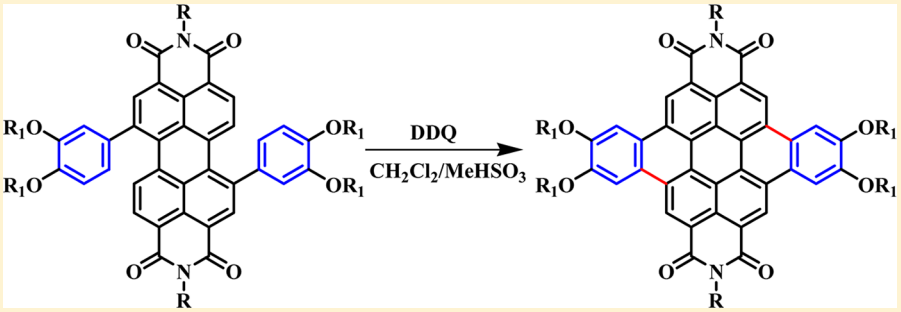
Supporting Information



ABSTRACT: A series of dibenzocoronene tetracarboxdiimide derivatives decorated with alkyl swallow-tail and alkoxy moieties were synthesized, and their structures were characterized. 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) as an effective oxidant was first used in the benzannulation of perylene diimides with the almost quantitative yield. The thermotropic behavior was investigated using differential scanning calorimetry (DSC) and polarization optical microscopy (POM). The introduction of alkyl swallow-tail and alkoxy substituents facilitates thermotropic liquid crystalline behavior. The branching site of alkyl swallow-tail units at the α position and the longer alkoxy chains played a similar role in lowering the mesophase transition as well as isotropization transition temperatures. The UV−vis absorption spectra of all compounds appeared as absorption in 425−600 nm region, and POM images of certain compounds exhibited characteristic columnar hexagonal (Col_h) packing and readily self-assembled into a homeotropic alignment toward the substrate.

INTRODUCTION

Discotic liquid crystals (DLCs), comprising a planar rigid aromatic core and flexible aliphatic side chains, are attractive candidates for applications in organic electronic and optoelectronic devices[1−12] as they readily self-organize into ordered columnar supramolecular architectures arising from strong interactions between the aromatic cores and nanophase separation.[4,13−15] This column arrangement provides a one-dimensional high-speed pathway for charge migration. The charge carrier mobility is enhanced owing to large intermolecular π-orbital overlaps in the columnar phase.[16−18] Until now, DLCs possessing large aromatic cores such as hexabenzocoronene and phtalocyanine derivatives have exhibited high carrier mobility in excess of 0.1 $cm^2\ v^{-1}\ s^{-1}$.[19−21]

In the past few years, self-assembly of liquid crystalline perylene diimides (PDIs) has attracted considerable attention in organic electronics[1,22−26] due to their favorable combination of liquid crystalline, photophsical, semiconducting, and photoconducting properties.[27−38] Generally, the simple approach to inducing liquid crystallinity of PDIs[28,36,39−41] is merging various swallow-tail moieties as N-substituents for weakening $\pi-\pi$ stacking interactions of the perylene core. Very recently, Funahashi[41] reported on the thermotropic liquid crystalline PDIs with swallow-tailed oligosiloxane chains showing high electron mobility and solubility. Futuremore, liquid crystalline core-extended PDI derivatives[42−44] can be prepared in different shapes from disklike coronene tetracarboxdiimides (CDIs) to elongated terrylene and quaterrylene tetracarboxdiimides, and such liquid crystalline derivatives perform significant functional properties as a result of different spectral absorption characters.[43,44] More important is that the management of the molecular structure presents the opportunity to control the spontaneous self-alignment such as elongated perylene tetracarboxdiimides tend to edge-on arrangement on a surface while disklike coronene tetracarboxdiimides to face-on. Therefore, enlargement of the aromatic core is supposed to achieve a more effective $\pi-\pi$ interaction resulting in highly ordered supramolecular structures and higher charge carrier mobilities; for example, coronenemonoimide derivatives reported showed a large intracolumnar charge carrier mobility of ca. 0.2 $cm^2\ v^{-1}\ s^{-1}$ in the discotic mesophase.[44]

Disk-shaped dibenzocoronene tetracarboxdiimes (dibenzo-CDIs) reported in the literature[45−47,49] have a more extended

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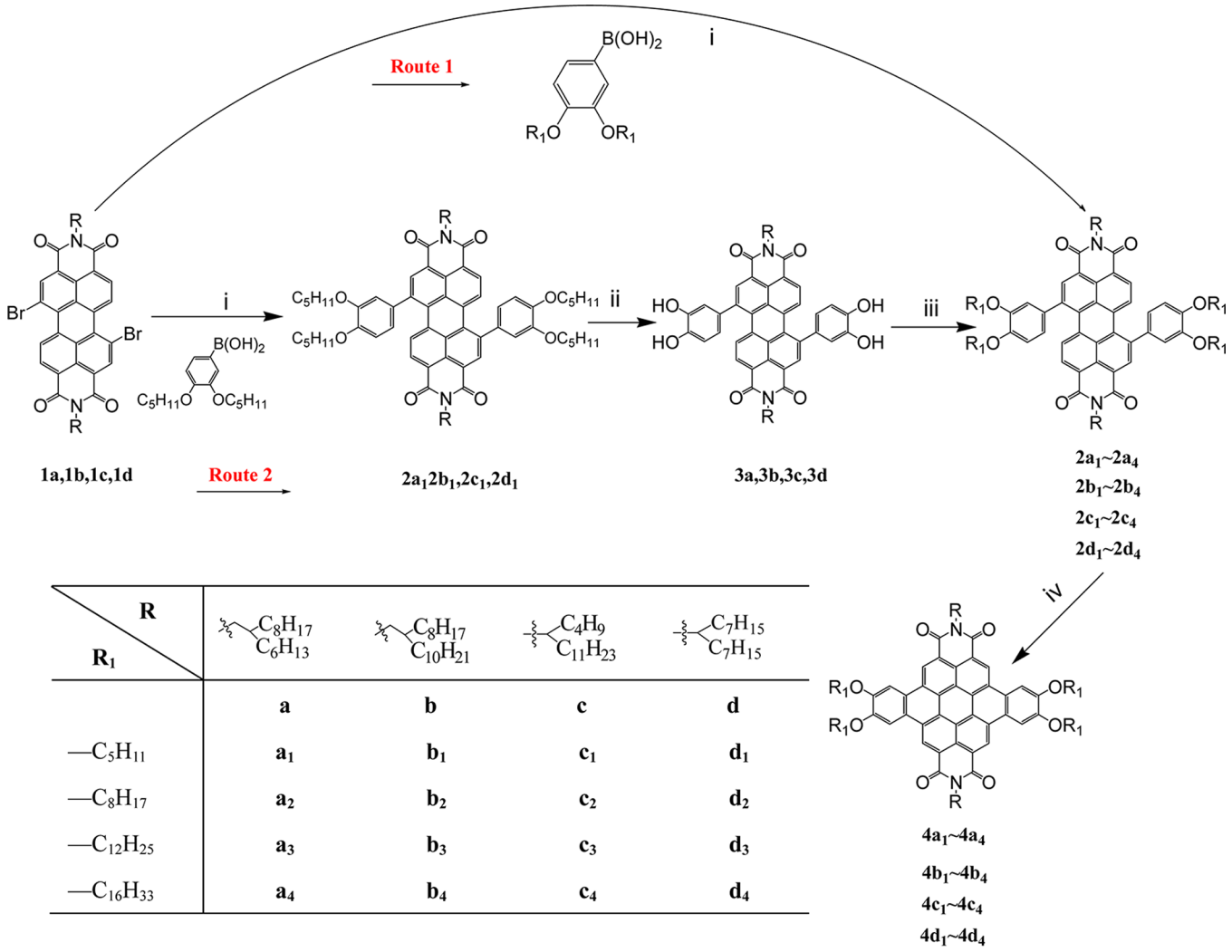
Scheme 1. Synthetic Routes of Dibenzo-CDIs 4[a]



R / R1	a	b	c	d
—C5H11	a1	b1	c1	d1
—C8H17	a2	b2	c2	d2
—C12H25	a3	b3	c3	d3
—C16H33	a4	b4	c4	d4

[a]Reagents and conditions: (i) K_2CO_3, $Pd(PPh_3)_4$, THF, 80 °C, 24 h; (ii) BBr_3, dry CH_2Cl_2, −78 °C, 12 h; (iii) R_1Br, K_2CO_3, dry C_2H_5OH, acetone, 80 °C, 12 h; (iv) DDQ, dry CH_2Cl_2, 0 °C, 6 h.

planar π-system than CDIs, but to the best of our knowledge, there is no liquid crystalline dibenzocoronene tetracarboxdiime derivatives published up to date. The aim of this research here is to provide new liquid crystalline derivatives with different spectral properties and give a systematic investigation of thermotropic liquid crystalline behaviors of them. A basic question here is how to effectively weaken the strong degree of crystallinity, which is intrinsic in dibenzo-CDIs, using a proper synthetic strategy. Here, we report our successful method of alkyl swallow-tail moieties as N-substituents and alkoxy chains in core-extended phenyl substituents inducing liquid crystalline phases to accomplish this goal. It could be expected that the thermotropic behavior of dibenzo-CDIs may be fine-tuned when decorated with diverse structures and length of swallow-tailed N-side chains having different spatial dimensions as well as a variety of alkoxy chains. In addition, the incorporation of these flexible side chains not only controls over the dibenzo-CDI derivatives thermotropic behavior but also induces high solubility, both of which are essential for facile processing.[50−56]

For this purpose, we present two facile and effective synthetic strategies of a series of dibenzo-CDIs decorated with four types of swallow-tailed alkyl and linear alkoxy chains. Remarkably, DDQ as an efficient oxidant was first employed in the benzannulation of PDIs' reaction with almost quantitative yield. According to the literature, our synthetic methods and exclusive products were first reported. By introduction of swallow-tailed alkyl and alkoxy groups, a significant reduction in the isotropization temperature was observed. The thermotropic behavior was investigated using TGA, DSC, and POM.

RESULTS AND DISCUSSION

A series of dibenzo-CDI derivatives were efficiently synthesized, and the routes 1 and 2 are illustrated in Scheme 1. Both synthetic routes began from the corresponding 1,7-dibromoperylenetetracarboxdiimides **1**, which were obtained by imidization of 1,7-dibromoperylene bisanhydride[57−59] with four kinds of alkyl swallow-tail amines. However, the isomer 1,6-dibromoperylenetetracarboxdiimide derivative was also formed while bromination of perylene 3,4,9,10-bisanhydride led to 1,7-dibromoperylene bisanhydride. Thus, bis(3,4-alkoxyphenyl)perylenetetracarboxdiimide derivatives **2** contained the 1,7- and 1,6-isomers. The isomers **2** could be observed and characterized by ^{1}H NMR and ^{13}C NMR. Because of the symmetry of both isomers, an assignment of the

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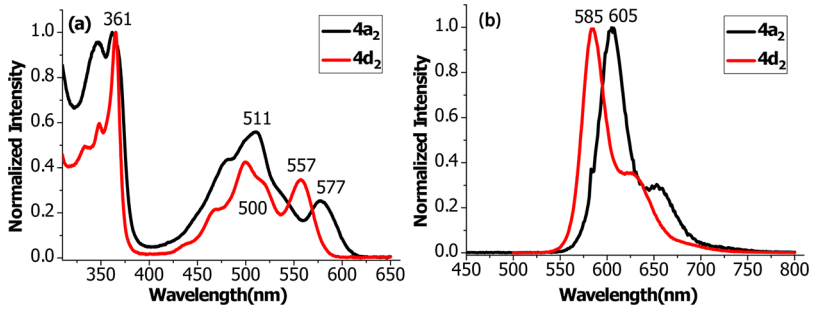

Figure 1. UV−vis spectra (a) and fluorescence emission spectra (b) of compound $\mathbf{4a}_2$ and $\mathbf{4d}_2$ in cloroform.

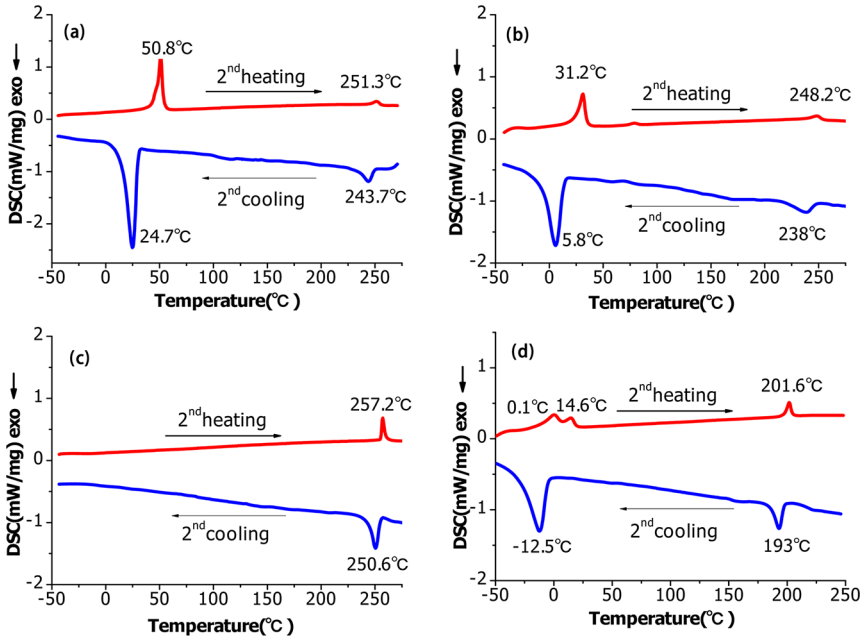

Figure 2. DSC curves of (a) compound $\mathbf{4a}_4$, (b) compound $\mathbf{4b}_4$, (c) compound $\mathbf{4d}_3$, and (d) compound $\mathbf{4d}_4$. The measurements were performed under a nitrogen atmosphere with heating and cooling rates of 10 K/min.

signals to the individual isomers was not possible. For symmetry reasons, even the mixtures were suitable to transfer the appropriate only products **4** in the last case of dehydrocyclization. In the benzannulation of PDIs' reaction, the DDQ/$MeHSO_3$ system[60] was effectively utilized for the oxidative C−C bond formations instead of most common oxidants such as $FeCl_3$, $MoCl_5$, or $SbCl_5$. In this case, we avoided problems such as chlorination of the phenyl entities and the large excess use of oxidants. The almost quantitative yield and ease of isolation of clean products are very advantageous.

In route 1, 1,7-dibromoperylenetetracarboxdiimide **1** reacted with 3,4-dialkoxyphenylboronic acid under Suzuki conditions to afford the corresponding PDI derivatives **2** in 55−60% yield. Then compounds **2** were dehydrocyclized with DDQ in dry dichloromethane at 0 °C with almost quantitatively dibenzo-CDI products **4**. For this method, the fatal problem is that different 3,4-dialkoxyphenylboronic acids must be synthesized in order to acquire matching compounds **4**.

The synthetic route 2 was carried out with the same procedure as the synthetic route 1 leading to preparation of 1,7-bis(3′,4′-dipentyloxyphenyl)perylene tetracarboxdiimide $\mathbf{2a}_1$, $\mathbf{2b}_1$, $\mathbf{2c}_1$, and $\mathbf{2d}_1$ first. Then compounds $\mathbf{2a}_1$, $\mathbf{2b}_1$, $\mathbf{2c}_1$, and $\mathbf{2d}_1$ in the presence of BBr_3 yielded the intermediate **3**. Then compounds **3** were etherified under Williamson conditions with four kinds of bromine alkyl leading to compounds **2** in around 85% yield. Finally, the benzannulation with DDQ under similar reaction conditions as route 1 afforded the products **4**.

Compared with synthetic route 1, synthetic route 2 reduced the number of synthesized compounds **2** with different alkoxy chains and avoided repeatedly preparation of various alkoxy-substituted 3,4-dialkoxyphenylboronic acids. Compounds **3** were easily modified with different alkyl groups in better yield. The yield of the last dehydrocyclization reaction was always high and over 90%. All synthesized dibenzo-CDI products **4** were red in the solid state and highly soluble in common organic solvents such as dichloromethane, chloroform, and THF. The above synthesized molecular structures were determined by the ^1H and ^{13}C NMR spectra, MALDI-TOF, and elemental analysis .

Photophysical Properties. UV−vis spectra and fluorescence emission spectra of dibenzo-CDI compounds **4** were recorded in chloroform. The spectra of compounds **4** with the same *N*-alkyl and different alkoxy groups were almost identical to each other. Figure 1 showed the representative absorption and emission spectra of compound $\mathbf{4a}_2$ and $\mathbf{4d}_2$. Both compounds showed absorption bands from 300 to 650 nm with two distinct absorption peaks in Figure 1a. According to

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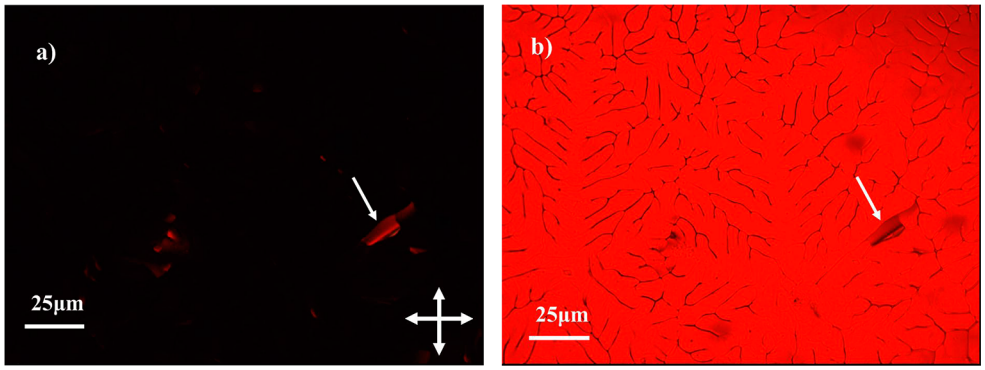

Figure 3. Optical microscopy images of compound $\mathbf{4b_4}$ cooled at 1 °C/min from the isotropic phase between two glass slides (a) 87 °C with and (b) without cross-polarizers (the arrows indicate defects in the homeotropic organization).

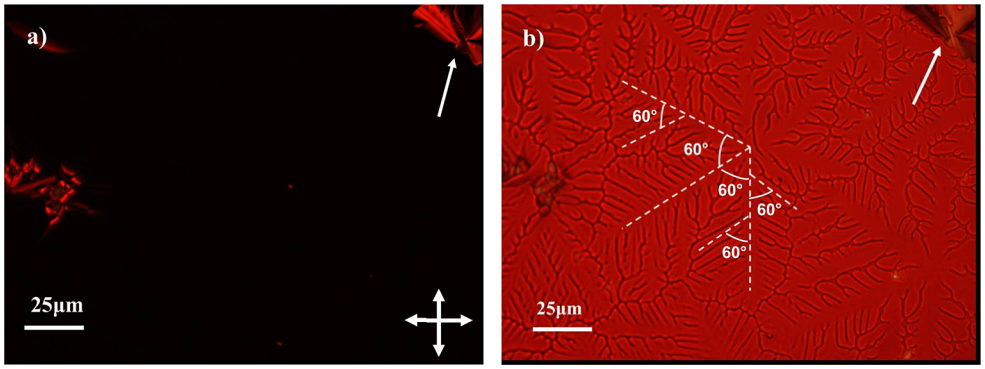

Figure 4. Optical microscopy images of compound $\mathbf{4d_4}$ cooled at 1 °C/min from the istropic phase between two glass slides (a) 195 °C with and (b) without coss-polarizers (dashed lines indicate the propagation direction of the dendritic structures) (the arrows indicate defects in the homeotropic organization).

the literature,[46,48] in the region of shorter wavelengths (up to 400 nm), a maximum absorbance of 361 nm in the spectrum of compounds $\mathbf{4a_2}$ and $\mathbf{4d_2}$ is correlated with a defined structure. The absorption for the four compounds displayed bathochromic shift, which was ascribed to introduction of electron-donating alkoxy groups. The bathochromic shift absorption showed differences in the long-wavelength region with different branching site positions of the *N*-alkyl substituents: compound $\mathbf{4a_2}$, with an *N*-alkyl substituent branching site at the α position, showed a strong peak at about 511 nm and one weak peak at about 577 nm; compound $\mathbf{4d_2}$ at the β position showed a strong peak at about 500 nm and one weak peak at about 557 nm. The fluorescence spectrum of compounds $\mathbf{4a_2}$, $\mathbf{4d_2}$ (Figure 2b) showed emission maxima difference about 20 nm.

Thermal Behaviors and Thermotropic Properties. Thermal behaviors and thermotropic properties of compounds **4** were investigated by TGA, DSC, and POM. TGA measurements revealed that all compounds **4** were thermally stable up to 300 °C with a 5% weight loss under nitrogen; see **S74** (Supporting Information). Through DSC and POM, the majority of compounds **4** did not evidently exhibit endothermic peaks in DSC curves and optical textures in POM images. So here only compounds $\mathbf{4a_4}$, $\mathbf{4b_4}$, $\mathbf{4d_3}$, and $\mathbf{4d_4}$ allowing a melting process and an investigation of their morphology from isotropization to crystallization were discussed in detail. It was observed that before reaching the decomposition temperature an expected isotropization transition of compound $\mathbf{4c_4}$ was not obtained because of its short butyl branched chain of N-alkyl substituent offering weak limit of the face-to-face $\pi-\pi$ stacking compared to compound $\mathbf{4d_4}$.

The DSC curves of compounds $\mathbf{4a_4}$, $\mathbf{4b_4}$, and $\mathbf{4d_4}$ showed two endothermic peaks on heating and two exothermic peaks on the cooling process, which indicated that they all exhibit an enantiotropic mesophase, while there was no obvious endothermic peak in the heating and cooling cycle in DSC curve of compoud $\mathbf{4d_3}$ possibly owing toa small enthalpy change between the crystalline (C_r) phase and a liquid crystalline (Col_h) phase (see Figure 2c). This indicated that the thermal behavior of compounds appears to have a strong dependence on the length of alkoxy side chains and the structures of branched alkyl chains. Compound $\mathbf{4d_4}$ showed the lowest isotropization temperature at 201.6 °C upon heating, while the others at about 250 °C (see Figure 2). Compared with compounds $\mathbf{4a_4}$ and $\mathbf{4b_4}$, the branching sites of N-alkyl substituents at the α position greatly lower the isotropization temperature than that at the β position for the large steric hindrance spacer requirement. For compounds $\mathbf{4d_3}$ and $\mathbf{4d_4}$ there was a decrease in clearing temperature with an increase of the alkoxy chain length; the longer alkyl chains at the *N*,*N*′-imide position played the same role in clearing the temperature as well as the melting temperature parallel with compounds $\mathbf{4a_4}$ and $\mathbf{4b_4}$.

The four dibenzo-CDI derivatives $\mathbf{4a_4}$, $\mathbf{4b_4}$, $\mathbf{4d_3}$, and $\mathbf{4d_4}$ were sandwiched between two glass slides and slowly cooled from the isotropic phase, and a small hysteresis of about 10−15 °C between DSC and POM measurement for the isotropization temperatures was observed because of higher heating rates. Four compounds $\mathbf{4a_4}$, $\mathbf{4b_4}$, $\mathbf{4d_3}$, and $\mathbf{4d_4}$ showed similar

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morphological behaviors. Representative optical microscopy images of $4b_4$ and $4d_4$ are presented in Figures 3 and 4, respectively.

In local areas, upon slowly cooling at rate 1 °C/min, dendritic structures with negligible distinction in relation to the disturbing organization were observed in nonpolarized light. But when cross-polarizers were added, the images became black with some small birefringence indicating that there existed homeotropic phase where the columnar axes were mostly perpendicular to the glass substrate. The uniform and symmetrical structure in disk-shaped molecules enjoyed a face-on manner and resulted in the homeotropic orientation as reported.[43,61,62] On the other hand, the introduction of ether groups probably enhanced the molecular affinity toward substrate and further promoted the homeotropic arrangement.[61] As displayed in Figure 4b, the dendritic textures formed with the angle between the branching almost 60°, which implied the hexagonal packing.

CONCLUSION

We designed and successfully synthesized a series of novel dibenzo-CDI derivatives with α- or β-branched alkyl and different alkoxy groups. DDQ was first employed in the PDI's core-extension reaction as the oxidant and the reaction could obtain almost quantitative yield. Introduction of alkyl and alkoxy resulted that the isotropic-phase temperatures of compounds **4** were largely lower, especially for compound $4d_4$ (~201 °C). The morphological behaviors of compounds $4a_4$, $4b_4$, $4d_3$, and $4d_4$ after crystallization from isotropic phase implied the homeotropic alignment toward surface, which further indicated that symmetrical disk-shaped compounds preferred a face-on arrangement upon cooling from the isotropization for the increase of molecular affinity toward surface by the influence of ether group.

EXPERIMENTAL SECTION

N,*N*′-Bis(swallow-tail alkyl)-1,7-dibromoperylene-3,4,9,10-tetracarboxydiimide **1a**, **1b**, **1c**, and **1d** were prepared following the literature[38] methods. All other chemicals were purchased from commercial source. Solvents were of analytical grade and were purified by the standard method if necessary.

Compounds $2a_1$, $2b_1$, $2c_1$, and $2d_1$. These compounds were prepared according to the general method. To a three-neck round-bottom flask under nitrogen atmosphere were dissolved *N*,*N*′-bis(swallow-tail alkyl)-1,7-dibromoperylene-3,4,9,10-tetracarboxydiimides **1a**, **1b**, **1c**, and **1d** and 3,4-dipentyloxyphenylboronic acid in THF. To this reaction mixture were added 1 M K_2CO_3 aqueous solution and catalyst $Pd(PPh_3)_4$ in sequence. The resulting suspension was stirred for 24 h at 80 °C under nitrogen atmosphere. The reaction mixture was allowed to cool to room temperature and extracted with dichloromethane. The collected organic layer was dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography, eluting with dichloromethane−petroleum ether.

***N*,*N*′-Bis(2-hexyldecyl)-1,7-bis(3,4-dipentyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide ($2a_1$).** The reaction of **1a** (1.94 g, 2 mmol), 3,4-dipentyloxyphenyl boronic acid (0.74 g, 2.5 mmol), and $Pd(PPh_3)_4$ (0.12 g, 0.1 mmol) in 45 mL of THF and 30 mL of 1 M K_2CO_3 aqueous solution under nitrogen atmosphere at 80 °C for 24 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1.5) as eluent produced compound $2a_1$ (1.54 g, 58%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.45 (s, 2H), 8.13(d, 2H), 7.77 (d, 2H), 7.04 (m, 6H), 4.02−4.15 (m, 12H), 1.80−1.93 (m, 10H), 1.24−1.56 (m, 62H), 0.82−0.99 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 163.5, 163.2, 150.5, 149.6, 140.5, 134.5, 134.0, 133.8, 131.0, 129.4, 128.5, 128.3, 126.4, 121.5, 121.3, 121.2, 114.4, 113.7, 69.5, 69.1, 44.8, 36.8, 31.8, 31.7, 30.1, 29.7, 29.6, 29.3, 29.0, 28.2, 26.5, 22.6, 22.5, 14.0. MALDI-TOF MS *m*/*z*: $[M + Na]^+$ calcd for $C_{88}H_{122}N_2O_8Na$ 1357.91, found 1358.1. Anal. Calcd for $C_{88}H_{122}N_2O_8$: C, 79.12; H, 9.20; N, 2.10. Found: C, 78.74; H, 9.23; N, 2.07.

***N*,*N*′-Bis(2-octyldodecyl)-1,7-bis-(3,4-dipentyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide ($2b_1$).** The reaction of **1b** (2.2 g, 2 mmol), 3,4-dipentyloxyphenyl boronic acid (0.74 g, 2.5 mmol), and $Pd(PPh_3)_4$ (0.12 g, 0.1 mmol) in 45 mL of THF and 30 mL of 1 M K_2CO_3 aqueous solution under nitrogen atmosphere at 80 °C for 24 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1.5) as eluent produced compound $2b_1$ (1.73 g, 60%) as dark purple solid: ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.60 (s, 2H), 8.17 (d, 2H), 7.90 (d, 2H), 6.99 (m, 6H), 4.05−4.16 (m, 12H), 1.76−1.92 (m, 10H), 1.21−1.58 (m, 78H), 0.82−0.99 (m, 24H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 163.5, 163.1, 150.5, 149.7, 140.5, 134.5, 134.0, 133.8, 131.0, 129.4, 128.5, 128.3, 126.4, 121.5, 121.3, 121.2, 114.4, 113.8, 69.5, 69.1, 44.8, 36.8, 31.9, 31.8, 30.1, 29.7, 29.6, 29.3, 29.0, 28.2, 26.6, 22.5, 14.0. MALDI-TOF MS *m*/*z*: $[M]^+$ calcd for $C_{96}H_{138}N_2O_8$ 1447.05, found 1448.2. Anal. Calcd for $C_{96}H_{138}N_2O_8$: C, 79.62; H, 9.61; N, 1.93. Found: C, 79.77; H, 9.38; N, 1.87.

***N*,*N*′-Bis(1-butylpentadecyl)-1,7-bis(3,4-dipentyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide ($2c_1$).** The reaction of **1c** (1.94 g, 2 mmol), 3,4-dipentyloxyphenyl boronic acid (0.74 g, 2.5 mmol), $Pd(PPh_3)_4$ (0.12 g, 0.1 mmol) in 45 mL of THF and 30 mL of 1 M K_2CO_3 aqueous solution under nitrogen atmosphere at 80 °C for 24 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound $2c_1$ (1.52 g, 57%) as dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.63 (d, 2H), 8.17 (m, 2H), 7.93 (m, 2H), 7.05 (m, 6H), 5.15 (s, 2H), 4.07−4.10 (m, 8H), 1.78−2.24 (m, 16H), 1.19−1.55 (m, 60H), 0.82−0.99 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.8, 164.6, 163.7, 163.5, 150.3, 149.8, 149.2, 148.4, 140.9, 135.8, 135.0, 134.6, 134.3, 132.4, 129.8, 129.7, 129.3, 129.1, 127.7, 122.6, 122.2, 121.9, 121.6, 121.5, 119.2, 114.5, 114.4, 114.1, 113.1, 69.6, 69.5, 69.4, 69.1, 54.5, 32.3, 32.0, 31.8, 29.6, 29.5, 29.3, 29.1, 29.0, 28.9, 28.8, 28.2, 28.1, 26.9, 22.6, 22.5, 22.4, 14.0. MALDI-TOF MS *m*/*z*: $[M]^+$ calcd for $C_{88}H_{122}N_2O_8$ 1334.92, found 1335.4. Anal. Calcd for $C_{88}H_{122}N_2O_8$: C, 79.12; H, 9.20; N, 2.10. Found: C, 79.25; H, 9.28; N, 2.06.

***N*,*N*′-Bis(1-heptyloctyl)-1,7-bis(3,4-dipentyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide ($2d_1$).** The reaction of **1d** (1.93 g, 2 mmol), 3,4-dipentyloxyphenyl boronic acid (0.74 g, 2.5 mmol), and $Pd(PPh_3)_4$ (0.12 g, 0.1 mmol) in 45 mL of THF and 30 mL of 1 M K_2CO_3 aqueous solution under nitrogen atmosphere at 80 °C for 24 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound $2d_1$ (1.44 g, 55%) as dark purple solid. ^{1}H NMR ($CDCl_3$, 600 MHz): δ ppm 8.64 (d, 2H), 8.17 (m, 2H), 7.93 (m, 2H), 7.06 (m, 6H), 5.15 (s, 2H), 3.97−4.09 (m, 8H), 1.80−2.23 (m, 16H), 1.20−1.55 (m, 56H), 0.79−0.99 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.7, 164.6, 163.7, 163.5, 150.4, 149.9, 149.7, 142.0, 140.9, 135.7, 135.0, 134.6, 132.6, 132.3, 129.7, 129.5, 129.2, 128.7, 128.4, 127.7, 127.0, 122.6, 122.2, 121.9, 121.6, 121.3, 114.6, 114.4, 69.5, 69.1, 54.5, 32.3, 31.7, 29.6, 29.5, 29.2, 28.9, 28.8, 28.2, 28.1, 26.9, 22.5, 22.4, 14.0. MALDI-TOF MS *m*/*z*: $[M]^+$ calcd for $C_{86}H_{118}N_2O_8$ 1329.88, found 1329.9. Anal. Calcd for $C_{86}H_{118}N_2O_8$: C, 78.98; H, 9.09; N, 2.14. Found: C, 78.72; H, 8.82; N, 2.18.

Compounds 3a, 3b, 3c, and 3d. These compounds were prepared according to the general method. To a solution of compounds $2a_1$, $2b_1$, $2c_1$, and $2d_1$ in dry dichloromethane at −78 °C was added dropwise BBr_3. The stirred reaction mixture was allowed to warm to room temperature. After 12 h, it was poured into ice, the product was extracted with ethyl acetate, and the organic extracts were dried and filtered. The crude product was purified by silica gel column chromatography using acetone−petroleum ether as eluent.

***N*,*N*′-Bis(2-hexyldecyl)-1,7-bis-(3,4-dihydroxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (3a).** The reaction of $2a_1$ (5.34 g, 4 mmol) and BBr_3 (4.4 g, 4.4 × 4 mmol) in 100 mL dry

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dichloromethane at −78 °C for 12 h followed by column chromatography purification on silica gel using acetone−petroleum ether (v/v = 1:3) as eluent produced compound **3a** (4.20 g, 90%) as a dark blue solid. ^{1}H NMR (acetone-d_6, 400 MHz): δ ppm 8.32 (s, 2H), 8.12 (d, 2H), 8.03 (s, 2H), 7.72 (d, 2H), 6.90 (m, 6H), 4.14 (d, 2H), 2.91 (m, 4H), 2.05 (m, 4H), 1.24−1.47 (m, 44H), 0.79−0.95 (m, 12H). ^{13}C NMR (acetone-d_6, 75 MHz): δ ppm 163.2, 162.8, 146.5, 146.0, 140.4, 134.0, 133.7, 131.3, 131.1, 130.3, 129.8, 128.4, 128.3, 127.1, 126.1, 121.0, 120.6, 116.8, 115.5, 44.4, 36.3, 31.7, 31.6, 29.9, 29.5, 29.4, 29.3, 29.1, 28.9, 28.7, 28.5, 28.4, 26.3, 22.4, 13.5. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{68}H_{82}N_2O_8$ 1504.61, found 1055.1. Anal. Calcd for $C_{68}H_{82}N_2O_8$: C, 77.39; H, 7.83; N, 2.65. Found: C, 77.44; H, 8.10; N, 2.58.

***N,N′*-Bis(2-octyldodecyl)-1,7-bis-(3,4-dihydroxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (3b).** The reaction of **2b$_1$** (5.80 g, 4 mmol) and BBr_3 (4.4 g, 4.4 × 4 mmol) in 100 mL of dry dichloromethane at −78 °C for 12 h followed by column chromatography purification on silica gel using acetone−petroleum ether (v/v = 1:3) as eluent produced compound **3b** (4.20 g, 90%) as a dark blue solid. ^{1}H NMR (acetone-d_6, 400 MHz): δ ppm 8.29 (s, 2H), 8.13 (d, 2H), 8.03 (s, 2H), 7.72 (d, 2H), 6.90 (m, 4H), 6.71 (s, 2H), 4.14 (d, 2H), 2.94 (m, 4H), 2.04 (m, 4H), 1.28−1.43 (m, 60H), 0.79−0.99 (m, 12H). ^{13}C NMR (acetone-d_6, 75 MHz): δ ppm 163.1, 162.8, 146.5, 146.1, 140.4, 134.0, 133.7, 133.6, 131.1, 129.7, 128.7, 128.4, 128.2, 126.1, 121.5, 116.8, 115.5, 64.9, 50.6, 44.4, 41.3, 36.3, 31.7, 31.6, 29.8, 29.7, 29.4, 29.1, 28.9, 28.6, 28.4, 28.1, 26.8, 26.2, 24.7, 22.4, 18.9, 13.4. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{76}H_{98}N_2O_8$ 1166.73, found 1167.3. Anal. Calcd for $C_{76}H_{98}N_2O_8$: C, 78.18; H, 8.46; N, 2.40. Found: C, 77.98; H, 8.38; N, 2.38.

***N,N′*-Bis(1-butylpentadecyl)-1,7-bis(3,4-dihydroxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (3c).** The reaction of **2c$_1$** (5.34 g, 4 mmol) and BBr_3 (4.4 g, 4.4 × 4 mmol) in 100 mL of dry dichloromethane at −78 °C for 12 h followed by column chromatography purification on silica gel using acetone−petroleum ether (v/v = 1:3) as eluent produced compound **3c** (3.92 g, 93%) as a dark blue solid. ^{1}H NMR (acetone-d_6, 400 MHz): δ ppm 8.43 (s, 4H), 8.18 (m, 4H), 8.01 (s, 2H), 7.05 (m, 6H), 5.17 (d, 2H), 2.89 (m, 6H), 2.31 (m, 2H), 1.84 (m, 2H), 1.18−1.33 (m, 42H), 0.80−0.95 (m, 12H). ^{13}C NMR (acetone-d_6, 75 MHz): δ ppm 144.2, 143.6, 138.6, 132.5, 131.7, 129.7, 127.3, 126.8, 125.0, 118.2, 114.5, 113.4, 51.4, 29.6, 29.4, 29.3, 27.3, 27.0, 26.7, 26.5, 26.2, 26.0, 25.7, 24.2, 19.9,11.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{68}H_{82}N_2O_8$ 1504.61, found 1505.1. Anal. Calcd for $C_{68}H_{82}N_2O_8$: C, 77.39; H, 7.83; N, 2.65. Found: C, 77.66; H, 7.76; N, 2.58.

***N,N′*-Bis(1-heptyloctyl)-1,7-bis(3,4-dihydroxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (3d).** The reaction of **2d$_1$** (5.23 g, 4 mmol) and BBr_3 (4.4 g, 4.4 × 4 mmol) in 100 mL of dry dichloromethane at −78 °C for 12 h followed by column chromatography purification on silica gel using acetone−petroleum ether (v/v = 1:3) as eluent produced compound **3d** (3.73 g, 91%) as a dark blue solid. ^{1}H NMR (acetone-d_6, 400 MHz): δ ppm 8.40 (s, 4H), 8.11 (m, 4H), 7.95 (s, 2H), 7.01 (m, 4H), 6.94 (s, 2H), 5.18 (d, 2H), 2.89 (m, 6H), 2.66 (m, 2H), 1.88 (m, 2H), 1.21−1.32 (m, 38H), 0.79−0.95 (m, 12H). ^{13}C NMR (acetone-d_6, 75 MHz): δ ppm 146.6, 146.0, 140.9, 134.0, 131.0, 129.6, 129.6, 128.7, 127.3, 120.5, 116.9, 115.7, 64.9, 53.9, 32.5, 32.0, 31.7, 31.6, 30.4, 29.7, 29.4, 29.3, 29.1, 29.0, 28.9, 28.4, 28.1, 26.7, 24.0, 22.4, 22.3, 19.1, 18.9, 13.4, 13.0. MALDI-TOF MS m/z: $[M]^+$ alcd for $C_{66}H_{78}N_2O_8$ 1026.58, found 1027.1. Anal. Calcd for $C_{66}H_{78}N_2O_8$: C, 77.28; H, 7.74; N, 2.69. Found: C, 77.33; H, 7.60; N, 2.73.

Compounds 2a$_2$−2a$_4$, 2b$_2$−2b$_4$, 2c$_2$−2c$_4$, and 2d$_2$−2d$_4$. These compounds were prepared according to the general method. To a solution of compounds **3a−d** in dry ethanol and acetone-d_6 were added bromooctane and K_2CO_3. The mixture was refluxed at 80 °C for 12 h and then poured into crushed ice. The product was extracted with dichloromethane, and the organic extracts were dried and filtered. The crude product was purified by silica gel column chromatography using dichloromethane−petroleum ether as eluent.

***N,N′*-Bis(2-hexyldecyl)-1,7-bis(3,4-dioctyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (2a$_2$).** To a solution of compound **3a** (0.53 g, 0.5 mmol) in 20 mL of dry ethanol and 20 mL of acetone were added bromooctane (0.58 g, 3 mmol) and K_2CO_3(0.3 g, 2.2 mmol). The mixture was refluxed at 80 °C for 12 h and then poured into ice. The product was extracted with dichloromethane, and the organic extracts were dried and filtered. The crude product was purified by silica gel column chromatography using dichloromethane−petroleum ether (v/v = 1:1.5) as eluent, and compound **2a$_2$** (0.64 g, 85%) was obtained as a dark purple solid. ^{1}H NMR ($CDCl_3$, 600 MHz): δ ppm 8.49 (s, 2H), 8.13 (d, 2H), 7.79 (d, 2H), 7.04 (m, 6H), 4.08−4.14 (m, 12H), 1.88−1.91 (m,10H), 1.24−1.55 (m, 86H), 0.83−0.92 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 163.6, 163.3, 150.5, 149.7, 140.6, 134.7, 134.1, 131.1, 129.5, 128.7, 128.5, 126.6, 121.6, 121.4, 121.2, 114.5, 113.9, 69.5, 69.1, 44.7, 36.7, 31.9, 31.8, 31.7, 30.1, 29.7, 29.6, 29.5, 29.3, 26.5, 26.1, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{100}H_{146}N_2O_8$ 1053.11, found 1054.4. Anal. Calcd for $C_{100}H_{146}N_2O_8$: C, 79.85; H, 9.78; N, 1.86. Found: C, 79.53; H, 9.64; N, 1.83.

***N,N′*-Bis(2-hexyldecyl)-1,7-bis-(3,4-didodecyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (2a$_3$).** The reaction of **3a** (0.53 g, 0.5 mmol), bromododecane (0.75 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1.5) as eluent produced compound **2a$_3$** (0.72 g, 84%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.44 (s, 2H), 8.12 (d, 2H), 7.75 (d, 2H), 6.99 (m, 6H), 4.07−4.15 (m, 12H), 1.87−1.91 (m, 10H), 1.24−1.59 (m, 118H), 0.85−0.90 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 163.6, 163.5, 150.4, 149.8, 140.7, 134.9, 134.4, 134.2, 131.7, 129.6, 128.9, 126.9, 121.7, 121.4, 114.5, 114.0, 69.5, 69.2, 44.7, 36.7, 31.9, 31.8, 31.7, 30.0, 29.7, 29.6, 29.5, 29.3, 26.5, 26.1, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M + Na]^+$ calcd for $C_{116}H_{178}N_2O_8Na$ 1750.35, found 1751.5. Anal. Calcd for $C_{116}H_{178}N_2O_8$: C, 80.60; H, 10.38; N, 1.62. Found: C, 80.57; H, 10.20; N, 1.56.

***N,N′*-Bis(2-hexyldecyl)-1,7-bis-(3,4-dihexadecyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (2a$_4$).** The reaction of **3a** (0.53 g, 0.5 mmol), bromohexadecane (0.92 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1.5) as eluent produced compound **2a$_4$** (0.83 g, 85%) as dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.46 (s, 2H), 8.13 (d, 2H), 7.76 (d, 2H), 6.99 (m, 6H), 4.06−4.13 (m, 12H), 1.79−1.96 (m, 10H), 1.23−1.58 (m, 150H), 0.84−0.87 (m, 24H). ^{13}C NMR ($CDCl_3$, 125 Hz): δ ppm 163.9, 163.3, 150.4, 149.7, 140.6, 134.7, 134.1, 134.0, 131.3, 129.5, 128.6, 128.5, 126.6, 121.6, 121.4, 121.2, 114.5, 113.8, 69.5, 69.1, 44.7, 36.7, 31.9, 31.8, 31.7, 30.1, 29.7, 29.6, 29.5, 29.3, 26.5, 26.1, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{132}H_{210}N_2O_8$ 1951.61, found 1951.8. Anal. Calcd for $C_{132}H_{210}N_2O_8$: C, 81.17; H, 10.84; N, 1.43. Found: C, 81.07; H, 10.68; N, 1.38.

***N,N′*-Bis(2-octyldodecyl)-1,7-bis(3,4-dioctyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (2b$_2$).** The reaction of **3b** (0.74 g, 0.5 mmol), bromooctane (0.58 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane-petroleum ether (v/v = 1:1.5) as eluent produced compound **2b$_2$** (0.69 g, 85%) as dark purple solid. ^{1}H NMR ($CDCl_3$, 600 MHz): δ ppm 8.44 (s, 2H), 8.12 (d, 2H), 8.11 (d, 2H), 6.99 (m, 6H), 4.07−4.16 (m, 12H), 1.88−1.91 (m, 10H), 1.22−1.55 (m, 102H), 0.83−0.92 (m, 24H). ^{13}C NMR ($CDCl_3$, 125 Hz): δ ppm 163.6, 163.3, 150.5, 149.7, 140.6, 134.7, 134.1, 131.4, 129.5, 128.7, 128.6, 126.7, 121.6, 121.4, 121.3, 114.5, 113.9, 69.5, 69.1, 44.7, 36.7, 31.9, 31.8, 30.1, 29.6, 29.3, 29.2, 26.5, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M + Na]^+$ calcd for $C_{108}H_{162}N_2O_8Na$ 1638.22, found 1639.5. Anal. Calcd for $C_{108}H_{162}N_2O_8$: C, 80.25; H, 10.10; N, 1.73. Found: C, 80.06; H, 9.89; N, 1.67.

***N,N′*-Bis(2-octyldodecyl)-1,7-bis-(3,4-didodecyloxyphenyl)-perylene-3,4,9,10-tetracarboxydiimide (2b$_3$).** The reaction of **3b** (0.74 g, 0.5 mmol), bromododecane (0.75 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−

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petroleum ether (v/v = 1:1.5) as eluent produced compound **2b$_3$** (0.76 g, 84%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.41 (s, 2H), 8.11 (d, 2H), 7.71 (d, 2H), 6.91 (m, 6H), 4.06−4.15 (m, 12H), 1.79−1.96 (m, 10H), 1.25−1.53 (m, 134H), 0.84−0.89 (m, 24H). ^{13}C NMR ($CDCl_3$, 100 Hz): δ ppm 163.6, 163.4, 150.5, 149.8, 140.6, 134.8, 134.2, 131.5, 129.6, 128.8,126.8, 121.7, 121.5, 121.3, 114.5, 69.6, 69.2, 44.7, 36.7, 31.9, 31.8, 30.1, 29.7, 29.6, 29.5, 29.4, 29.3, 26.6, 26.5, 26.1, 26.0, 22.7, 22.6, 14.1. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{124}H_{194}N_2O_8$ 1839.48, found 1839.9. Anal. Calcd for $C_{124}H_{194}N_2O_8$: C, 80.90; H, 10.62; N, 1.52. Found: C, 80.78; H, 10.49; N, 1.54.

***N,N′*-Bis(2-octyldodecyl)-1,7-bis-(3,4-dihexadecyloxyphenyl)perylene-3,4,9,10-tetracarboxydiimide (2b$_4$).** The reaction of **3b** (0.74 g, 0.5 mmol), bromohexadecane (0.92 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1.5) as eluent produced compound **2b$_4$** (0.88 g, 85%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.51 (s, 2H), 8.14 (d, 2H), 7.81 (d, 2H), 7.01 (m, 6H), 4.07−4.13 (m, 12H), 1.78−1.91 (m, 10H), 1.21−1.58 (m, 166H), 0.83−0.90 (m, 24H). ^{13}C NMR ($CDCl_3$, 125 Hz): δ ppm 163.5, 163.2, 150.6, 149.7, 140.5, 134.7, 134.1, 133.9,131.1, 129.4, 128.6, 128.4, 126.5, 121.5, 121.3, 121.2, 114.5, 69.5, 69.1, 44.8, 36.8, 31.9, 31.8, 31.7, 30.1, 29.7, 29.6, 29.5, 29.3, 26.6, 26.5, 26.1, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{140}H_{226}N_2O_8$ 2063.73, found 2064.0. Anal. Calcd for $C_{140}H_{226}N_2O_8$: C, 81.42; H, 11.03; N, 1.36. Found: C, 81.10; H, 11.10; N, 1.34.

***N,N′*-Bis(1-butylpentadecyl)-1,7-bis(3,4-dioctyloxyphenyl)perylene-3,4,9,10-tetracarboxydiimide (2c$_2$).** The reaction of **3c** (0.53 g, 0.5 mmol), bromooctane (0.58 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound **2c$_2$** (0.67 g, 87%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 300 MHz): δ ppm 8.63 (m, 2H), 8.18 (m, 2H), 7.94 (m, 2H), 7.07 (m, 6H), 5.19 (m, 2H), 3.97−4.14 (m, 8H), 1.79−2.24 (m, 16H), 1.21−1.44 (m, 82H), 0.82−0.91 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.8, 164.6, 163.7, 163.5, 150.4, 149.9, 149.7, 142.0, 140.9, 135.7, 135.0, 134.6, 134.4, 132.6, 132.4, 129.7, 129.5, 129.3, 128.7, 128.4, 127.7, 127.0, 122.7, 122.3, 121.9, 121.6, 121.3, 114.6, 114.5, 114.3, 69.6, 69.2, 54.6, 54.5, 54.4, 37.0, 34.0, 32.7, 32.3, 32.0, 31.8, 31.7, 31.4, 30.1, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 28.3, 26.9, 26.0, 25.9, 24.8, 22.6, 14.1, 14.0. MALDI-TOF MS m/z: $[M + Na]^+$ calcd for $C_{100}H_{146}N_2O_8Na$ 1526.1, found 1526.2. Anal. Calcd for $C_{100}H_{146}N_2O_8$: C, 79.85; H, 9.78; N, 1.86. Found: C, 79.62; H, 9.92; N, 1.79.

***N,N′*-Bis(1-butylpentadecyl)-1,7-bis-(3,4-didodecyloxyphenyl)perylene-3,4,9,10-tetracarboxydiimide (2c$_3$).** The reaction of **3c** (0.53 g, 0.5 mmol), bromododecane (0.75 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound **2c$_3$** (0.72 g, 84%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 600 MHz): δ ppm 8.63 (m, 2H), 8.17 (m, 2H), 7.93 (m, 2H), 7.06 (m, 6H), 5.20 (m, 2H), 3.92−4.09 (m, 8H), 1.78−2.23 (m, 16H), 1.20−1.55 (m, 116H), 0.83−0.90 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.8, 164.6, 163.7, 163.5, 150.4, 149.9, 149.7, 142.0, 140.9, 135.7, 135.0, 134.6, 134.6, 134.4, 132.9, 132.6, 132.3, 130.0, 129.7, 129.5, 129.3, 129.1, 128.7, 128.4, 127.7, 127.0, 122.6, 122.3, 121.9, 121.8, 121.6, 121.3, 114.6, 114.5, 114.3, 69.6, 69.2, 69.0, 54.6, 54.5, 54.4, 32.3, 32.0,31.9, 31.8, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 26.9, 26.1, 22.6, 14.1. MALDI-TOF MS m/z: $[M + Na]^+$ calcd for $C_{116}H_{178}N_2O_8Na$ 1750.35, found 1750.4. Anal. Calcd for $C_{116}H_{178}N_2O_8$: C, 80.60; H, 10.38; N, 1.62. Found: C, 80.29; H, 10.40; N, 1.60.

***N,N′*-Bis(1-butylpentadecyl)-1,7-bis(3,4-dihexadecyloxyphenyl)perylene-3,4,9,10-tetracarboxydiimide (2c$_4$).** The reaction of **3c** (0.53 g, 0.5 mmol), bromohexadecane (0.92 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound **2c$_4$** (0.83 g, 85%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.63 (m, 2H), 8.17 (m, 2H), 7.92 (m, 2H), 7.05 (m, 6H), 5.17 (m, 2H), 3.96−4.18 (m, 8H), 1.81−2.23 (m, 16H), 1.19−1.53 (m, 148H), 0.82−0.99 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.8, 164.6, 163.7, 163.5, 150.4, 149.9, 149.7, 142.0, 140.9, 135.7, 135.0, 134.6, 134.4, 132.6, 132.4, 129.7, 129.5, 129.3, 128.7, 128.4, 127.7, 127.0, 122.6, 122.3, 121.6, 121.3, 114.6, 114.5, 114.3, 69.6, 69.2, 54.6, 54.5, 54.4, 32.3, 32.0, 31.9, 31.8, 30.2, 29.6, 29.5, 29.4, 29.3, 29.2, 29.1, 26.9, 26.1, 26.0, 22.6, 22.4, 14.1, 14.0, 13.8. MALDI-TOF MS m/z: $[M + Na]^+$ calcd for $C_{132}H_{210}N_2O_8Na$ 1974.6, found 1975.5. Anal. Calcd for $C_{132}H_{210}N_2O_8$: C, 81.17; H, 10.84; N, 1.43. Found: C, 81.09; H, 10.55; N, 1.40;

***N,N′*-Bis(1-heptyloctyl)-1,7-bis(3,4-dioctyloxyphenyl)perylene-3,4,9,10-tetracarboxydiimide (2d$_2$).** The reaction of **3d** (0.53 g, 0.5 mmol), bromooctane (0.58 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound **2d$_2$** (0.63 g, 85%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 600 MHz): δ ppm 8.63 (m, 2H), 8.16 (m, 2H), 7.93 (m, 2H), 7.06 (m, 6H), 5.15 (s, 2H), 3.97−4.08 (m, 8H), 1.81−2.23 (m, 16H), 1.20−1.56 (m, 80H), 0.81−0.92 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.8, 164.6, 163.7, 163.5, 150.4, 149.9, 149.7, 142.0, 140.9, 135.7, 135.0, 134.6, 134.4, 132.6, 132.3, 129.8, 129.7, 129.5, 129.3, 129.1, 128.7, 128.4, 127.7, 127.0, 122.6, 122.3, 121.9, 121.6, 121.3, 114.6, 114.5, 114.3, 114.2, 69.6, 69.5, 69.4, 69.2, 54.7, 54.5, 54.4, 32.3, 31.9, 31.7, 31.5, 29.6, 29.5, 29.4, 29.3, 29.2, 26.9, 26.1, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M + Na]^+$ calcd for $C_{98}H_{142}N_2O_8Na$ 1498.07, found 1499.1. Anal. Calcd for $C_{98}H_{142}N_2O_8$: C, 79.74; H, 9.70; N, 1.90. Found: C, 79.78; H, 9.44; N, 1.92.

***N,N′*-Bis(1-heptyloctyl)-1,7-bis(3,4-didodecyloxyphenyl)perylene-3,4,9,10-tetracarboxydiimide (2d$_3$).** The reaction of **3d** (0.53 g, 0.5 mmol), bromododecane (0.75 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound **2d$_3$** (0.72 g, 85%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 600 MHz): δ ppm 8.63 (m, 2H), 8.17 (m, 2H), 7.93 (m, 2H), 6.94 (m, 6H), 5.15 (s, 2H), 3.97−4.08 (m, 8H), 1.80−2.25 (m, 16H), 1.21−1.56 (m, 112H), 0.81−0.90 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.8, 164.6, 163.7, 163.5, 150.4, 149.9, 149.7, 149.2, 148.4, 142.0, 140.9, 135.7, 135.0, 134.6, 134.4, 132.6, 132.4, 130.0, 129.9, 129.7, 129.5, 129.3, 129.1, 128.7, 128.4, 127.7, 127.0, 122.6, 122.3, 121.9, 121.6, 121.3, 119.3, 114.6, 114.5, 114.2, 113.2, 69.6, 69.4, 69.2, 54.6, 54.5, 54.4, 32.3, 31.8, 31.7, 31.5, 29.6, 29.5, 29.4, 29.3, 29.2, 26.9, 26.1, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M + Na]^+$ calcd for $C_{114}H_{174}N_2O_8Na$ 1722.32, found 1723.5. Anal. Calcd for $C_{114}H_{174}N_2O_8$: C, 80.51; H, 10.31; N, 1.65. Found: C, 80.39; H, 10.42; N, 1.67.

***N,N′*-Bis(1-heptyloctyl)-1,7-bis(3,4-dihexadecyloxyphenyl)perylene-3,4,9,10-tetracarboxydiimide (2d$_4$).** The reaction of **3d** (0.53 g, 0.5 mmol), bromohexadecane (0.92 g, 3 mmol), and K_2CO_3 (0.3 g, 2.2 mmol) at 80 °C for 12 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:2) as eluent produced compound **2d$_4$** (0.82 g, 85%) as a dark purple solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 8.58 (d, 2H), 8.14 (m, 2H), 7.90 (m, 2H), 7.06 (m, 6H), 5.14 (s, 2H), 3.96−4.07 (m, 8H), 1.81−2.25 (m, 16H), 1.20−1.55 (m, 142H), 0.80−0.89 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.8, 164.6, 163.7, 163.5, 150.4, 149.9, 149.7, 142.0, 140.9, 135.7, 135.0, 134.6, 132.6, 132.3, 129.9, 129.7, 129.5, 129.3, 129.1, 128.7, 128.4, 127.7, 127.0, 122.6, 122.3, 121.9, 121.6, 121.3, 114.6, 114.5, 114.3, 69.6, 69.2,54.5, 32.3, 31.9, 31.7, 29.7, 29.6, 29.5, 29.4, 29.3, 29.2, 26.9, 26.7, 26.1, 26.0, 22.6, 14.0. MALDI-TOF MS m/z: $[M]^+$ Calcd for $C_{130}H_{206}N_2O_8$ 1923.58, found 1923.8. Anal. Calcd for $C_{130}H_{206}N_2O_8$: C, 81.11; H, 10.79; N, 1.46. Found: C, 80.89; H, 10.59; N, 1.44.

Compounds 4a$_1$−4d$_4$. These compounds were prepared according to the general method. To a solution of compounds **4a$_1$**−**4d$_4$** in dry dichloromethane and methanesulfonic acid (v/v = 9:1) was added DDQ at 0 °C.The stirred reaction mixture was allowed to warm to room temperature. After 6 h, it was poured into ice, the product was

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extracted with dichloromethane, and the organic extracts were dried and filtered. The residue was purified by silica gel column chromatography, eluting with dichloromethane−petroleum ether.

***N,N′*-Bis(2-hexyldecyl)-5,6:11,12-bis(3,4-dipentyloxybenzo)-coronene-2,3,8,9-tetracarboxydiimide ($4a_1$).** The reaction of $2a_1$ (0.66 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v =1:1) as eluent produced compound $4a_1$ (0.61g, 92%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.76 (s, 4H), 8.44 (s, 4H), 4.87 (m, 4H), 4.58 (m, 4H), 4.27 (d, 4H), 2.27−2.30 (m, 8H), 1.70−1.93 (m, 18H), 1.14−1.55 (m, 60H), 0.78−0.819 (m, 12H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.5, 151.0, 126.1, 124.3, 123.9, 121.1, 121.0, 120.9, 120.1, 104.8, 69.2, 44.6, 36.8, 31.9, 31.8, 30.1, 29.8, 29.6, 29.4, 29.3, 28.8, 26.6, 22.9, 22.6, 22.5, 14.2, 14.0, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{88}H_{118}N_2O_8$ 1330.89, found 1331.5. Anal. Calcd for $C_{88}H_{118}N_2O_8$: C, 79.36; H, 8.93; N, 2.10. Found: C, 79.13; H, 8.82; N, 2.07.

***N,N′*-Bis(2-hexyldecyl)-5,6:11,12-bis(3,4-dioctyloxybenzo)-coronene-2,3,8,9-tetracarboxydiimide ($4a_2$).** The reaction of $2a_2$ (0.75 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4a_2$ (0.69 g, 92%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.77 (s, 4H), 8.45 (s, 4H), 4.88 (m, 4H), 4.57 (m, 4H), 4.26 (d, 4H), 2.25−2.29 (m, 8H), 1.64−1.95 (m, 18H), 1.15−1.57 (m,72H), 0.81−1.02 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.5, 151.0, 126.1, 124.3, 123.9, 121.1, 120.9, 120.1, 104.8, 69.2, 44.6, 36.8, 32.0, 31.9, 31.8, 30.2, 29.9, 29.8, 29.6, 29.5, 29.3, 26.7, 26.6, 22.8, 22.6, 22.5, 14.2, 14.0, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{100}H_{142}N_2O_8$ 1499.08, found 1500.3. Anal. Calcd for $C_{100}H_{142}N_2O_8$: C, 80.06; H, 9.54; N, 1.87. Found: C, 79.81; H, 9.41; N, 1.84.

***N,N′*-Bis(2-hexyldecyl)-5,6:11,12-bis(3,4-didodecyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4a_3$).** The reaction of $2a_3$ (0.86 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4a_3$ (0.79 g, 92%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.77 (s, 4H), 8.45 (s, 4H), 4.88 (m, 4H), 4.56 (m, 4H), 4.26 (d, 4H), 2.25−2.29 (m, 8H), 1.65−1.97 (m, 18H), 1.15−1.54 (m, 104H), 0.72−0.91 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.6, 151.0, 126.1, 124.3, 123.9, 121.1, 120.9, 120.1, 104.8, 69.2, 44.6, 36.8, 32.0, 31.9, 31.8, 30.2, 29.9, 29.8, 29.6, 29.5, 29.3, 26.6, 22.9, 22.6, 22.5, 14.1, 14.0, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{116}H_{173}N_2O_8$ 1722.32, found 1723.7. Anal. Calcd for $C_{116}H_{173}N_2O_8$: C, 80.83; H, 10.12; N, 1.63. Found: C, 80.77; H, 9.98; N, 1.59.

***N,N′*-Bis(2-hexyldecyl)-5,6:11,12-bis(3,4-dihexadecyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4a_4$).** The reaction of $2a_4$ (0.98 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4a_4$ (0.90 g, 91%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.77 (s, 4H), 8.45 (s, 4H), 4.87 (m, 4H), 4.56 (m, 4H), 4.25 (d, 4H), 2.25−2.28 (m, 8H), 1.63−1.95 (m, 18H), 1.14−1.54 (m,136H), 0.79−0.88 (m, 24H). ^{13}C NMR ($CDCl_3$,100 Hz): δ ppm 164.5, 151.0, 126.0, 124.5, 123.9, 120.9, 120.0, 104.8, 69.3, 36.9, 32.0, 31.9, 30.3, 30.0, 29.9, 29.8, 29.7, 29.4, 26.8, 22.7, 22.6, 14.1. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{132}H_{206}N_2O_8$ 1947.58, found 1947.9. Anal. Calcd for $C_{132}H_{206}N_2O_8$: C, 81.34; H, 10.65; N, 1.44. Found: C, 81.27; H, 10.46; N, 1.42.

***N,N′*-Bis(2-octyldodecyl)-5,6:11,12-bis(3,4-dipentyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4b_1$).** The reaction of $2b_1$ (0.72 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4b_1$ (0.66g, 93%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.78 (s, 4H), 8.46 (s, 4H), 4.88 (m, 4H), 4.58 (m, 4H), 4.27 (d, 4H), 2.25−2.28 (m, 8H), 1.70−1.95 (m, 18H), 1.14−1.57 (m, 64H), 0.73−1.11 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.6, 151.0, 126.1, 124.4, 123.9, 121.1, 121.0, 120.1, 104.8, 69.2, 36.8, 31.9, 31.8, 31.7, 30.1, 29.6, 29.5, 29.3, 29.2,28.8, 26.7, 22.9, 22.5, 14.2, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{96}H_{134}N_2O_8$ 1443.01, found 1443.3. Anal. Calcd for $C_{96}H_{134}N_2O_8$: 79.84; H, 9.35; N, 1.94. Found: 79.72; H, 9.12; N, 1.89.

***N,N′*-Bis(2-octyldodecyl)-5,6:11,12-bis(3,4-dioctyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4b_2$).** The reaction of $2b_2$ (0.81 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4b_2$ (0.74 g, 92%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.78 (s, 4H), 8.46 (s, 4H), 4.88 (m, 4H), 4.57 (m, 4H), 4.26 (d, 4H), 2.25−2.29 (m, 8H), 1.63−1.96 (m, 18H), 1.12−1.56 (m, 88H), 0.73−1.08 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.6, 151.0, 126.1, 124.4, 123.9, 121.1, 120.9, 120.1, 104.8, 69.2, 36.8, 32.0, 31.9, 31.8, 30.2, 29.9, 29.8, 29.7, 29.6, 29.5, 29.3, 26.7, 22.8, 22.5, 14.2, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{108}H_{158}N_2O_8$ 1611.2, found 1612.4. Anal. Calcd for $C_{108}H_{158}N_2O_8$: C, 80.45; H, 9.88; N, 1.74. Found: C, 80.27; H, 9.72; N, 1.70.

***N,N′*-Bis(2-octyldodecyl)-5,6:11,12-bis(3,4-didodecyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4b_3$).** The reaction of $2b_3$ (0.92 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4b_3$ (0.84 g, 91%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.78 (s, 4H), 8.45 (s, 4H), 4.88 (m, 4H), 4.57 (m, 4H), 4.25 (d, 4H), 2.25−2.27 (m, 8H), 1.65−1.96 (m, 18H), 1.15−1.54 (m,120H), 0.73−1.08 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 164.6, 151.0, 126.1, 124.3, 123.9, 121.1, 120.9, 120.1, 104.8, 69.2, 44.6, 36.8, 32.0, 31.8, 30.2, 29.9, 29.8, 29.7, 29.6, 29.5, 29.3, 29.2, 26.7, 22.7, 22.5, 14.1, 14.0, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{124}H_{190}N_2O_8$ 1835.45, found 1837.3. Anal. Calcd for $C_{124}H_{190}N_2O_8$: C, 81.08; H, 10.43; N, 1.53. Found: C, 79.92; H, 10.21; N, 1.48.

***N,N′*-Bis(2-octyldodecyl)-5,6:11,12-bis(3,4-dihexadecyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4b_4$).** The reaction of $2b_4$ (1.03 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4b_4$ (0.96 g, 93%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.77 (s, 4H), 8.45 (s, 4H), 4.88 (m, 4H), 4.57 (m, 4H), 4.25 (d, 4H), 2.25−2.28 (m, 8H), 1.63−1.96 (m, 18H), 1.12−1.59 (m,152H), 0.73−1.08 (m, 24H). ^{13}C NMR ($CDCl_3$, 100 Hz): δ ppm 164.5,151.0, 126.1, 124.3, 123.9, 121.0, 120.1, 104.8, 69.3, 44.6, 36.9, 31.9, 30.2, 29.9, 29.8, 29.7, 29.4, 26.7, 22.7, 22.6, 14.1, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{140}H_{222}N_2O_8$ 2059.7, found 2059.9. Anal. Calcd for $C_{140}H_{222}N_2O_8$: C, 81.58; H, 10.86; N, 1.36. Found: C, 81.27; H, 10.93; N, 1.33.

***N,N′*-Bis(1-butylpentadecyl)-5,6:11,12-bis(3,4-dipentyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4c_1$).** The reaction of $2c_1$ (0.67 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4c_1$ (0.63 g, 94%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.64 (d, 4H), 8.26 (s, 4H), 5.26 (m, 2H), 4.61 (m, 8H), 2.27−2.62 (m, 16H), 1.14−1.80 (m, 60H), 0.76−1.03 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 165.3, 164.3, 151.0, 126.1, 123.9, 123.5, 121.6, 120.8, 120.6, 119.8, 105.0, 69.3, 55.1, 32.8, 32.5, 31.8, 29.9, 29.8, 29.7, 29.3, 29.0, 28.5, 28.3, 27.7, 22.9, 22.8, 22.5, 14.2, 14.1, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{88}H_{118}N_2O_8$ 1330.89, found 1331.5. Anal. Calcd for $C_{88}H_{118}N_2O_8$: C, 79.36; H, 8.93; N, 2.10. Found: C, 79.11; H, 9.12; N, 2.03.

***N,N′*-Bis(1-butylpentadecyl)-5,6:11,12-bis(3,4-dioctyloxy-benzo)coronene-2,3,8,9-tetracarboxydiimide ($4c_2$).** The reaction of $2c_2$ (0.75 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4c_2$ (0.71 g, 95%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.74 (d, 4H), 8.31 (s, 4H), 5.47 (m, 2H), 4.57 (m, 8H), 2.20−2.59 (m, 16H), 1.12−1.83 (m, 84H), 0.76−0.99 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 165.4, 164.4, 151.0, 126.3, 124.0,

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123.6, 121.7, 121.0, 120.9, 120.0, 105.0, 69.4, 55.1, 32.8, 32.5, 32.0, 31.8, 29.8, 29.7, 29.6, 29.5, 29.3, 27.7, 26.3, 22.9, 22.8, 22.5, 14.2, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{100}H_{142}N_2O_8$ 1499.08, found 1500.2. Anal. Calcd for $C_{100}H_{142}N_2O_8$: C, 80.06; H, 9.54; N, 1.87. Found: C, 79.98; H, 9.37; N, 1.83.

***N,N′*-Bis(1-butylpentadecyl)-5,6:11,12-bis(3,4-didodecyloxybenzo)coronene-2,3,8,9-tetracarboxydiimide ($4c_3$).** The reaction of $2c_3$ (0.86 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4c_3$ (0.79 g, 92%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.62 (d, 4H), 8.24 (s, 4H), 5.49 (m, 2H), 4.63 (m, 8H), 2.27−2.64 (m, 16H), 1.18−1.86 (m, 116H), 0.77−0.95 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 165.4, 164.5, 151.1, 126.4, 124.1, 121.8, 121.2, 121.0, 105.1, 69.4, 58.4, 55.1, 32.8, 32.5, 31.9, 31.8, 31.2, 29.9, 29.8, 29.6, 29.5, 29.3, 27.6, 26.3, 22.9, 22.7, 22.5, 18.4, 14.2, 14.1, 13.9. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{116}H_{173}N_2O_8$ 1722.32, found 1723.3. Anal. Calcd for $C_{116}H_{173}N_2O_8$: C, 80.83; H, 10.12; N, 1.63; Found: C, 80.75; H, 9.93; N, 1.66.

***N,N′*-Bis(1-butylpentadecyl)-5,6:11,12-bis(3,4-didodecyloxybenzo)coronene-2,3,8,9-tetracarboxydiimide ($4c_4$).** The reaction of $2c_4$ (0.97 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4d_4$ (0.93 g, 95%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.72 (d, 4H), 8.29 (s, 4H), 5.46 (m, 2H), 4.57 (m, 8H), 2.22−2.59 (m, 16H), 1.14−1.80 (m, 148H), 0.77−0.98 (m, 24H). ^{13}C NMR ($CDCl_3$, 100 Hz): δ ppm 165.2, 151.0, 126.0, 123.9, 121.5, 120.6, 119.8, 105.0, 69.4, 55.1, 32.8, 32.5, 31.9, 30.0, 29.9, 29.8, 29.4, 27.9, 26.4, 23.0, 22.7, 22.6, 14.3, 14.1, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{132}H_{206}N_2O_8$ 1947.58, found 1947.9. Anal. Calcd for $C_{132}H_{206}N_2O_8$: C, 81.34; H, 10.65; N, 1.44. Found: C, 81.23; H, 10.55; N, 1.43.

***N,N′*-Bis(1-heptyloctyl)-5,6:11,12-bis(3,4-dipentyloxybenzo)coronene-2,3,8,9-tetracarboxydiimide ($4d_1$).** The reaction of $2d_1$ (0.65 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4d_1$ (0.62 g, 95%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.79 (d, 4H, PerH), 8.34 (s, 4H, ArH), 5.47 (m, 2H, NH), 4.59 (m, 8H, OCH_3), 2.22−2.58 (m, 16H, CH_2), 1.14−1.79 (m, 56H, CH_2), 0.78−1.12 (m, 24H, CH_3). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 165.4, 164.4, 151.0, 126.4, 124.1, 123.7, 121.9, 121.2, 121.1, 120.8, 120.1, 105.0, 69.3, 58.4, 55.2, 32.8, 31.9, 29.8, 29.6, 29.4, 29.3, 28.5, 27.6, 22.7, 22.6, 18.3, 14.2, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{86}H_{114}N_2O_8$ 1302.86, found 1303.0. Anal. Calcd for $C_{86}H_{114}N_2O_8$: C, 79.22; H, 8.81; N, 2.15. Found: C, 79.08; H, 8.68; N, 2.17.

***N,N′*-Bis(1-heptyloctyl)-5,6:11,12-bis(3,4-dioctyloxybenzo)coronene-2,3,8,9-tetracarboxydiimide ($4d_2$).** The reaction of $2d_2$ (0.74 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4d_2$ (0.70 g, 95%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.67 (d, 4H), 8.33 (t, 4H), 5.48 (m, 2H), 4.61 (m, 8H), 2.24−2.60 (m, 16H), 1.13−1.81 (m, 80H), 0.79−1.00 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 165.3, 164.4, 151.0, 126.2 123.9, 123.5, 121.6, 120.8, 120.6, 119.9, 105.0, 69.4, 55.2, 32.9, 32.0, 29.9, 29.8, 29.7, 29.5, 27.8, 26.4, 22.8, 22.7, 14.2, 14.1. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{98}H_{138}N_2O_8$ 1471.05, found 1471.5. Anal. Calcd for $C_{98}H_{138}N_2O_8$: C, 79.95; H, 9.45; N, 1.90. Found: C, 79.78; H, 9.37; N, 1.87.

***N,N′*-Bis(1-heptyloctyl)-5,6:11,12-bis(3,4-didodecyloxybenzo)coronene-2,3,8,9-tetracarboxydiimide ($4d_3$).** The reaction of $2d_3$ (0.85 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4d_3$ (0.80 g, 94%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.78 (d, 4H), 8.33 (t, 4H), 5.47 (m, 2H), 4.59 (m, 8H), 2.22−2.59 (m, 16H), 1.30−1.79 (m, 112H), 0.77−0.91 (m, 24H). ^{13}C NMR ($CDCl_3$, 100 Hz): δ ppm 165.2, 164.4, 151.0, 126.0, 123.9, 123.4, 121.5, 120.7, 119.8, 105.0, 69.4, 55.3, 33.0, 32.0, 30.0, 29.9, 29.6, 29.5, 27.9, 26.4, 22.7, 14.1. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{114}H_{170}N_2O_8$ 1695.3, found 1695.5. Anal. Calcd for $C_{114}H_{170}N_2O_8$: C, 80.70; H, 10.10; N, 1.65. Found: C, 80.58; H, 9.97; N, 1.63.

***N,N′*-Bis(1-heptyloctyl)-5,6:11,12-bis(3,4-didodecyloxybenzo)coronene-2,3,8,9-tetracarboxydiimide ($4d_4$).** The reaction of $2d_4$ (0.96 g, 0.5 mmol) and DDQ (0.23 g, 1 mmol) at 0 °C for 6 h followed by column chromatography purification on silica gel using dichloromethane−petroleum ether (v/v = 1:1) as eluent produced compound $4d_4$ (0.91 g, 95%) as a red solid. ^{1}H NMR ($CDCl_3$, 400 MHz): δ ppm 9.83 (d, 4H), 8.37 (t, 4H), 5.48 (m, 2H), 4.56 (m, 8H), 2.20−2.58 (m, 16H), 1.25−1.61 (m, 144H), 0.81−0.88 (m, 24H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ ppm 165.4, 164.5, 151.1, 126.4, 124.1, 121.8, 121.1, 121.0, 120.1, 105.0, 69.4, 58.4, 55.2, 32.8, 31.9, 29.9, 29.8, 29.7, 29.4, 29.3, 27.7, 22.7, 22.6, 18.4, 14.0. MALDI-TOF MS m/z: $[M]^+$ calcd for $C_{130}H_{202}N_2O_8$ 1919.55, found 1920.7. Anal. Calcd for $C_{130}H_{202}N_2O_8$: C, 81.28; H, 10.60; N, 1.46. Found: C, 81.13; H, 10.37; N, 1.42.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

^{1}H NMR and ^{13}C NMR spectra of compound **2**−**4**; TGA curves of comoupoud **4**, the optical images of compound $4a_4$ and $4d_3$. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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