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Synthesis and Characterization of Thermally-Stable and Soluble Perylene Bisimide

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N,N'-Didodecyl-1,7-di(4-*tert*-butylphenoxy)perylene-3,4,9,10-tetracarboxylic acid bisimide (**4a**) and N,N'-didodecyl-1,6,7,12-tetra(4-*tert*-butylphenoxy)- perylene-3,4,9,10-tetracarboxylic acid bisimide (**4b**) have been synthesized derived from 1,7-dibromo- and 1,6,7,12-tetrabromo-perylene tetracarboxylic acid bisanhydride. Because these perylene bisimides exhibit good solubility, the structures of **4a** and **4b** are characterized by FT-IR, ^{1}H and ^{13}C NMR and mass spectroscopy. The results show an agreement with the proposed structures. The optical properties of perylene bisimides have been determined by UV/vis and fluorescence spectroscopy. It has been shown that the displacement of bromines by 4-*tert*-butylphenoxy groups causes the significant red shift and the increase in the number of 4-*tert*-butylphenoxy groups determines the red shift location. Due to electron inductive effect which was caused by 4-*tert*-butylphenoxy groups, the LUMO of **4a** and **4b** are 3.89 and 3.86 eV, respectively. TG measurements show that **4a** and **4b** possess good thermal stability with high decomposition temperatures (> 400 ºC).

Key Words: Perylene bisimide, Synthesis, Solubility, Optical property, Cyclic voltammetry, Thermal stability.

INTRODUCTION

Perylene bisimide (PBI) dyes have received a great deal of attention owing to their outstanding intense luminescence, high chemical stability and thermal stability which are favourable for application as functional materials[1,2]. In the past decade, more and more interesting investigation has been focused on the modification at the terminal and bay position of perylene bisimide structures through high-yield synthetical routes to amend the solubility and optical properties of products[3-10].

In the perylene bisimides family, di- and tetra-substituted perylene bisimides have played important roles in the synthesis of bay-functionalized perylene dyes[1,4,9,11-15]. In recent years, a multitude of perylene bisimides has been synthesized by nucleophilic substitution of the corresponding 1,7-dibromo- and 1,6,7,12-tetrabromo-substituted perylene bisimides to achieve promising properties, which are attractive for application

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in fields such as light harvesting arrays, solar cells, field effect transistor, light-emitting diodes, *etc*. In the case of dibrominated perylene bisimides, the exchange of the bromines is straightforward, nucleophiles such as carbon, aryl, cyano, phenoxy and nitrogen could be easily reacted with the perylene core to form novel difunctionalized perylene bisimide derivatives[16-22]. On the other hand, few reports are available in literature on the exchange of the bromines of tetrabromo-substituted perylene bisimides. Recently, Zhu's group have prepared tetra-substituted perylene bisimides *via* the Suzuki coupling reaction of tetrabrominated perylene bisimide[23]. More recently, Hill *et al.*[24] also reported that four 4-*tert*-butylphenoxy groups have been successfully introduced to the 1-,6-,7- and 12-positions of the perylene bisimide backbone. Wang and co-workers[25,26] have also prepared conjugated di(perylene bisimide) and tri(perylene bisimides) from tetrabrominated perylene bisimides.

In this paper, we designed and synthesized N,N'-didodecyl-1,7-di(4-*tert*-butylphenoxy)perylene-3,4,9,10-tetracarboxylic acid bisimide and N,N'-didodecyl-1,6,7,12-tetra(4-*tert*-butylphenoxy)perylene-3,4,9,10-tetracarboxylic acid bisimide derived from 1,7-dibromo- and 1,6,7,12-tetrabromo-perylene tetracarboxylic acid bisanhydride. The properties of the fluorescent perylene bisimides were detected by UV-vis, fluorescence spectrometry, cyclic voltammetry and TG measurement, which to be contrast. The phenomena indicated that the resulted functional materials may be useful in light-emitting diodes and solar cells.

EXPERIMENTAL

N-Methyl-2-pyrrolidone (NMP) was purified with distillation under reduced pressure. 4-*tert*-Butylphenol was purified by recrystallization from petroleum ether. All other chemicals and solvents were purchased without further purification.

^{1}H NMR spectra were recorded on BRUKER 300 MHz or 600 MHz spectrometers in $CDCl_3$ and chemical shifts were obtained relative to tetramethyl silane (TMS). Fourier transform infrared (FT-IR) spectra were recorded on NICOLET-6700. Low and high resolution mass spectra were recorded on Finnigan MAT95S spectrometer for EI MS and Bruker Daltonics BIFLEX III for MALDI-TOF MS. UV/vis spectra were performed on a Shimadzu 3150 spectrophotometer at 25 ºC and fluorescent spectra were carried out on a PE LS55 fluorescence spectrophotometer at 25 ºC. Cyclic voltammograms (CV) were recorded on GAMRY reference 600. thermogravimetric analyses (TGA) were performed by a TA-SDT Q600 thermogravimetric analyzer at a heating rate of 10 °C/min under nitrogen atmosphere.

General procedure

Synthesis of 1,7-dibromoperylene-3,4,9,10-tetracarboxylic acid bisanhydride (2a): A mixture of perylene-3,4,9,10-tetracarboxylic acid bisanhydride **1** (10 g, 25.5 mmol), 110 mL of 98 wt % sulphuric acid and 20 mL fuming sulphuric acid were stirred at room temperature for 12 h, then I_2 (0.25 g, 0.96 mmol) was added. Subsequently, the mixture was heated to 85 ºC and the reaction mixture was heated for 14 h at 85 °C. The reaction was then cooled to room temperature and the excess bromine was removed by a gentle stream of N_2 gas and 21 mL water was added slowly. The resulting red powder was filtered through G4 funnel and washed with a large amount of water until the precipitate bacame neutral. The precipitate was dried in a vacuum overnight at 120 °C to give 12.9 g of a red powder, the yield is 91.4 %.

Synthesis of 1,6,7,12-tetrabromoperylene-3,4,9,10-tetracarboxylic acid bisanhydride (2b): A mixture of perylene-3,4,9,10-tetracarboxylic acid bisanhydride **1** (10 g, 25.5 mmol), 110 mL of 98 wt % sulphuric acid and 20 mL fuming sulphuric acid were stirred at room temperature for 12 h. The mixture was then heated to 65 °C and I_2 (0.24 g, 0.9 mmol) was added. Subsequently, the reaction temperature was heated to 85 °C and Br_2 (31.2 g, 194 mmol) was added dropwise over 12 h. Then the mixture was heated to 110 °C and more Br_2 (12.48 g, 77.6 mmol) was added dropwise over 4 h. The reaction mixture was heated for additional 12 h at 110 °C. After the reaction was cooled to room temperature, the excess bromine was removed by a gentle stream of N_2 gas and 21 mL water was added slowly. The resulting red powder was filtered through G4 funnel and washed with a large amount of water until the precipitate bacame neutral. The precipitate was dried in a vacuum overnight at 120 °C to give 15.1 g of a red powder, the yield is 83.8 %.

Synthesis of N,N'-didodecyl-1,7-dibromoperylene-3,4,9,10-tetracarboxylic acid bisimide (3a): A mixture of **2a** (0.95 g, 1.72 mmol) and dodecylamine (0.94 g, 5.07 mmol) in 10 mL propionic acid was heated to refluxing for 12 h under N_2. The resulting mixture was cooled and poured into water, filtrated and washed with water until the filtrate reached neutrality. The crude solid was dried at 60 °C in a vacuum. The product was purified by column chromatography (dichloromethane/petroleum ether = 3:1) to obtain deep red solid (0.56 mg), the yield is 37 %. m.p. 152-153 °C. Data for **3a**: FT-IR (KBr, ν, cm^{-1}): 3058, 2952, 2918, 2850, 1697 (C=O), 1655 (C=O), 1596, 1462, 1435, 1395, 1337, 1238, 1157, 1095, 1022, 860, 813, 748, 723, 682, 537; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 9.46 (t, 2H), 8.91 (d, 2H), 8.69 (q, 2H), 4.20 (t, 4H), 1.75 (t, 4H), 1.26-1.46 (m, 36H), 0.892 (s, 6H); MS (EI): m/z = 884.

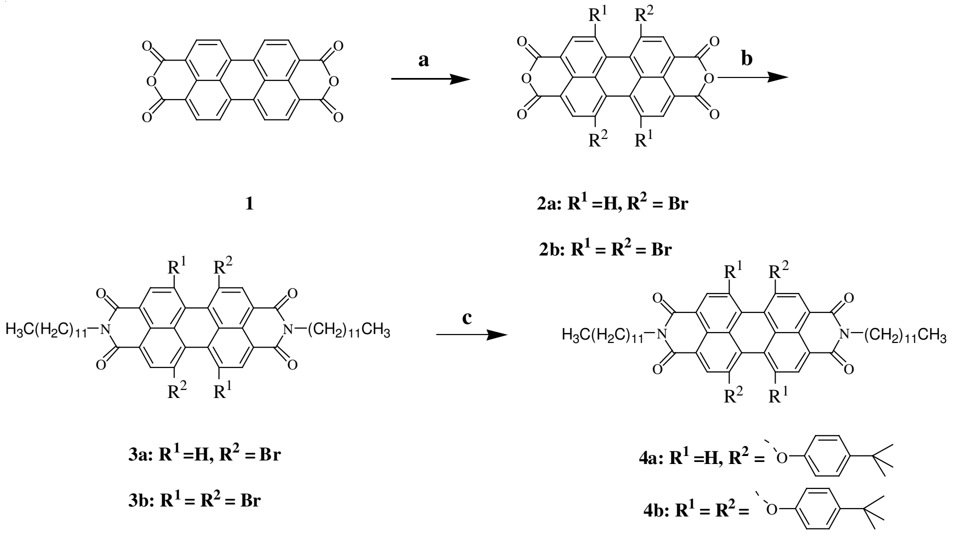
Synthesis of N,N'-didodecyl-1,6,7,12-tetrabromoperylene-3,4,9,10-tetracarboxylic acid bisimide (3b): 3b was synthesized by the reaction of **2b** (1.05 g, 1 mmol) and dodecylamine (0.94 g, 5.07 mmol) in 10 mL propionic acid using similar procedures as **3a**, the yield was 34 %. m.p. 220-221 °C. Data for **3b**: FT-IR (KBr, ν, cm^{-1}): 2954, 2923, 2653, 1704 (C=O), 1666 (C=O), 1584, 1460, 1434, 1387, 1362, 1281, 1233, 1161, 1094, 908, 803, 742, 647, 530, 483; ^{1}H NMR (300 MHz, $CDCl_3$), δ: 8.82 (s, 4H), 4.20 (t, 4H), 1.72 (t, 4H), 1.25~1.42 (m, 36H), 0.87 (t, 6H); ^{13}C NMR (300 MHz, $CDCl_3$): δ: 162.18, 136.09, 131.67, 131.44, 123.94, 122.74, 40.98, 31.92, 29.70, 29.64, 29.62, 29.60, 29.52, 29.34, 28.12, 27.07, 22.68, 14.10 ppm; MS (EI): m/z = 1042.

Synthesis of N,N'-didodecyl-1,7-di(4-*tert*-butylphenoxy)perylene-3,4,9,10-tetracarboxylic acid bisimide (4a): A mixture of compound **3a** (0.3 g, 0.34 mmol), 4-*tert*-butylphenol (0.16 g, 1.12 mmol) and K_2CO_3 (0.077 g, 0.558 mmol) in 10 mL NMP was heated to 80 ºC for 24 h under N_2. The resulting mixture was cooled and poured into water, filtrated and washed with water until the filtrate reached neutrality. The crude solid was dried at 60 ºC in a vacuum. The product was purified by column chromatography (dichloromethane/petroleum ether = 2:1) to obtain deep red solid (0.072 g), the yield is 18 %. m.p. 204-205 ºC. Data for **4a**: FT-IR (KBr, ν, cm^{-1}): 3058, 2956, 2924, 2857, 1698 (C=O), 1662 (C=O), 1596, 1506, 1462, 1435, 1408, 1334, 1264, 1221, 1177, 1047, 1018, 850, 811, 750, 558; ^1H NMR (300 MHz, $CDCl_3$) δ: 9.55 (d, 2H), 8.54 (d, 2H), 8.308 (s, 2H), 7.46 (t, 4H), 7.09 (d, 4H), 4.13 (t, 4H), 1.70 (m, 4H), 1.24-1.50 (m, 54H), 0.87 (t, 6H); MS (MALDI-TOF): m/z = 1022.9.

Synthesis of N,N'-didodecyl-1,6,7,12-tetra(4-*tert*-butylphenoxy)perylene-3,4,9,10-tetracarboxylic acid diimide (4b): 4b was synthesized by the reaction of **3b** (0.31 g, 0.3 mmol), 4-*tert*-butylphenol (0.30 g, 2 mmol) and K_2CO_3 (0.2 g, 1.5 mmol) in 10 mL NMP using similar procedures as **4a**, the yield was 22 %. m.p. 249-250 ºC. Data for **4b**: FT-IR (KBr, ν, cm^{-1}): 2959; 2924, 2854, 1695 (C=O), 1659 (C=O), 1588, 1504, 1463, 1436, 1410, 1356, 1289, 1218, 1174, 1137, 1111, 1020, 881, 838, 799, 726, 556; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.22 (s, 4H), 7.23 (d, 8H), 6.83 (d, 8H), 4.09 (t, 4H), 1.66 (m, 4H), 1.30-1.18 (m, 72H), 0.86 (t, 6H); ^{13}C NMR (600 MHz, $CDCl_3$): δ: 163.44, 155.95, 152.90, 147.29, 132.89, 126.65, 122.49, 120.49, 119.87, 119.46, 119.34, 40.67, 34.36, 31.89, 31.45, 29.70, 29.59, 29.51, 29.32, 28.11, 27.13, 22.66, 14.08 ppm; MS (MALDI-TOF): m/z: 1019.80296 (M^+).

RESULTS AND DISCUSSION

The synthesis of the fluorescent perylene bisimides **4a** and **4b** are outlined in **Scheme-I**. Würthner and co-workers reported that the tetrabromo product was difficult to afford in high yield due to contamination with di- and tri- brominated products[27]. Alternatively, Qiu *et al.*[23] and Fan *et al.*[28] reported that increased the reagent amount, elevated temperatures and prolonged reaction time led to a majority of the tetrabrominated product being formed. Utilizing the methods of Fan *et al.*[28] and Qiu *et al.*[23] compounds **2a** and **2b** were obtained after the steps of reaction with bromide in the presence of iodine in sulphuric acid. The brominated perylene bisanhydrides are insoluble in organic solvents, so the common characterizations are difficult to perform. A diverse library of bisimides has been obtained from perylene tetracarboxylic acid bisanhydride and a multitudes of aromatic and aliphatic amines *via* conventional imidization. More sterically demanding groups such as 2,6-diisopropylaniline, swallowtail alkyl chains, 3,4,5-trialkyl-substituted aniline groups or flexible tetraethylene glycol was incorporated to prevent aggregation and increase solubility[29]. Dodecylamine was chosen to heat with **2a** and **2b** in propionic acid to afford **3a** and **3b**, the yields are 37 and 34 %, respectively. Due to the existence of two dodecyl groups, **3a** and **3b** show good solubility in organic solvents, which provide convenience for purification and full characterizations. Further reaction with potassium carbonate and 4-*tert*-butylphenol in NMP lead to **4a** and **4b** in 18 and 22 % yield, respectively. Due to the presence of multi-substituted impurities, the overall yields are lower.



Scheme-I: Synthetic route of the fluorescent compounds **4a** and **4b**; (a) Br_2, H_2SO_4, I_2, 85 ºC or 110 ºC; (b) dodecylamine, refluxing, 12 h; (c) 4-*tert*-butylphenol, K_2CO_3, 80 ºC, 24 h

Solubility: The drawback of perylene bisimides is the inherent low solubility. Now there are two approaches to synthesize soluble PBIs. One way is to introduce substituents at N atoms of imide groups. Another is to attach electron-donating or electron-withdrawing groups directly to the perylene core. Primarily to enhance solubility, a dodecylamine group is appended at the terminal imide. The bay region is substituted with 4-*tert*-butylphenoxy groups, which also influence solubility due to more directly disrupt π-π interactions. The solubility of **4a** and **4b** were determined by the dissolution of 10 mg of solid PBI in 1 mL of organic solvent at room temperature (Table-1). **4a** and **4b** were highly soluble in conventional solvents, such as toluene, chloroform, dichloromethane and THF. While they were partially soluble in some organic solvents, such as NMP, acetone and DMF.

TABLE-1
SOLUBULITY OF PERYLENE BISIMIDES PBI **3** AND PBI **4** IN SOLVENTS

Toluene	THF	Chloroform	Dichloromethane	Acetone	NMP	DMF	DMSO
+ +	+ +	+ +	+ +	+ –	+ –	+ –	–
+ +	+ +	+ +	+ +	+ –	+ –	+ –	–

THF: tetrahydrofuran; DMF: N,N'-dimethyl formamide; DMSO: dimethyl sulphoxide + +: soluble; + –: partially soluble; –: insoluble.

Optical properties: The absorption spectra of perylene bisimide derivatives **3a**, **3b**, **4a** and **4b** are shown in Fig. 1. Comparatively, the spectrum of four compounds exhibits the similar trend of band shape, which shows two PBI core absorption bands with maximum absorption and along with one shoulder absorption (Table-2). Compare with **3a** and **3b**, the absorption bands of compounds **4a** and **4b** undergo a significant bathochromic shift. The longest absorption bands can be assigned to the electronic S_0 - S_1 transition (a transition dipole moment along the long molecular axis) undergo the red shift of the maximum absorption from 526 nm (**3a**)-548 nm (**4a**) and from 530 nm (**3b**)-585 nm (**4b**), respectively. Meanwhile, the lower absorption bands assigned to the electronic S_0 - S_2 transition (a transition dipole moment perpendicular to the long molecular axis) of compounds **4a** and **4b** locate at 441 and 454 nm, respectively. However, **3a** and **3b** are at 390 and 404 nm, respectively[1,16,30]. These results could be attributed to the electron-donating effect of 4-*tert*-butylphenolic groups and the steric twisting of the perylene core. Moreover, in comparison to compound **4a**, the increase in the number of 4-*tert*-butylphenoxy groups in **4b** results in 55 nm of bathochromic shifts of the longest absorption. This phenomena indicated that the loss of planarity and rigidity of the perylene bisimide chromophore influenced the absorption property.

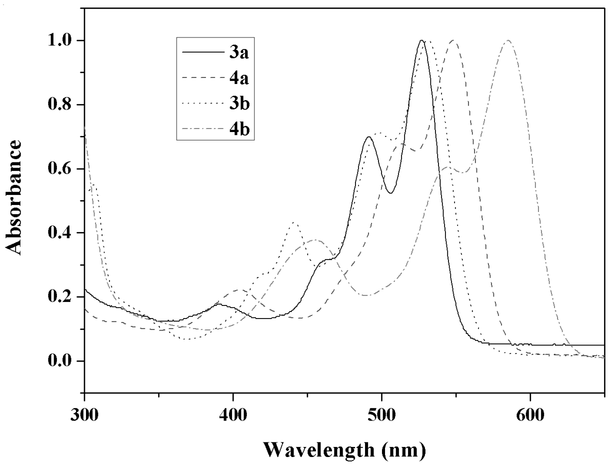


Fig. 1. Absorption spectra of compounds **3a**, **3b**, **4a** and **4b** in $CHCl_3$

TABLE-2
DATA OF LONGEST ABSORPTION, SHOULDER ABSORPTION AND LOWER ABSORPTION OF **3a**, **3b**, **4a** AND **4b**

Compound	Longest absorption (nm)	Shoulder absorption (nm)	Lower absorption (nm)
3a	526	491	390
3b	548	512	404
4a	530	497	441
4b	585	542	454

Fig. 2 shows the normalized emission spectra of compounds **3a**, **3b**, **4a** and **4b** in $CHCl_3$. The maximum emission of **3a** and **3b** located at 545 and 564 nm, respectively. However, **4a** and **4b** are at 576 and 609 nm, respectively. As compared with perylene bisimides **3a** and **3b**, displacement of bromines by 4-*tert*-butylphenoxy groups causes the significant red shift of the emission band. In comparison to compound **4a**, the increase in the number of 4-*tert*-butylphenoxy groups in compound **4b** results in 33 nm of red shifts of the emission band. Because the 4-*tert*-butylphenoxy groups are electron-rich, it is not surprising that red shifts of the emission band of **4a** and **4b** occured. At the same time, appending 4-*tert*-butylphenoxy groups makes the loss of planarity and rigidity of the perylene core, which also contribute to red shifts.

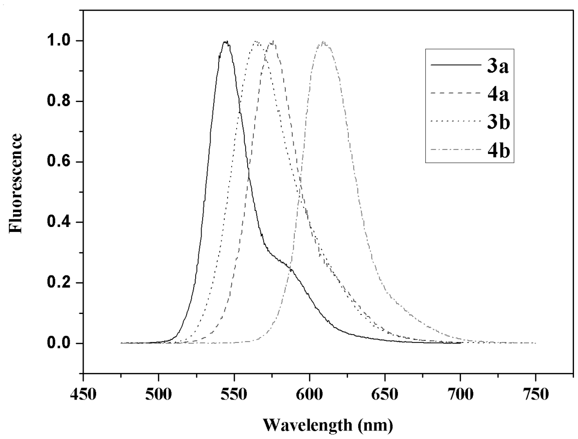


Fig. 2. Nomalized fluorescence emission spectra of compounds **3a**, **3b**, **4a** and **4b** in $CHCl_3$ [(1) compound **3a**, λ_{ex} = 518 nm; (2) compound **3b**, λ_{ex} = 550 nm; (3) compound **4a**, λ_{ex} = 532 nm; (4) compound **4b**, λ_{ex} = 600 nm)

Electrochemical behavior: Cyclic voltammetry (CV) is a useful tool to study reversible redox behaviour, electrochemical stability and to examine the HOMO and LUMO energy values of hole and electron transport materials. The cyclic voltammograms of compounds **4a** and **4b** are shown in Fig. 3 (Table-3). Eletrochemistry experiments were performed using an electrolyte solution of 0.1 M tetra-*n*-butylammonium hexafluorophosphate ($TBAPF_6$) and 0.1 mM compounds **4a** and **4b** in dichloromethane, which were carried out in a three-electrode cell consisting of a glassy carbon working electrode, a platinum counter electrode and a Ag/Ag^+ reference electrode, at a scan rate of 100 mV/s. From the Fig. 4, the half wave potentials ($E_{1/2}$) of compounds **4a** and **4b** *versus* Ag/Ag^+ were -0.77 and -0.80 V, respectively. Using $E_{[vacuum]} = E_{Ag/Ag^+} + 4.66$ V, a LUMO of 3.89 and 3.86 eV are calculated, respectively. The data indicated that introducing different number of 4-*tert*-butylphenoxy groups makes the LUMO level changed. At the same time, perylene bisimides represent a class of *n*-type organic semiconductor material, the lower the LUMO level is, the easier it is to obtain the electron. The LUMO level of **4b** is lower 0.03 eV than **4a**, so **4b** is good for injecting the electron. Because π-electron inductive effect of perylene core, because four 4-*tert*-butylphenoxy groups is stronger than two 4-*tert*-butylphenoxy groups, the LUMO level show significant influence.

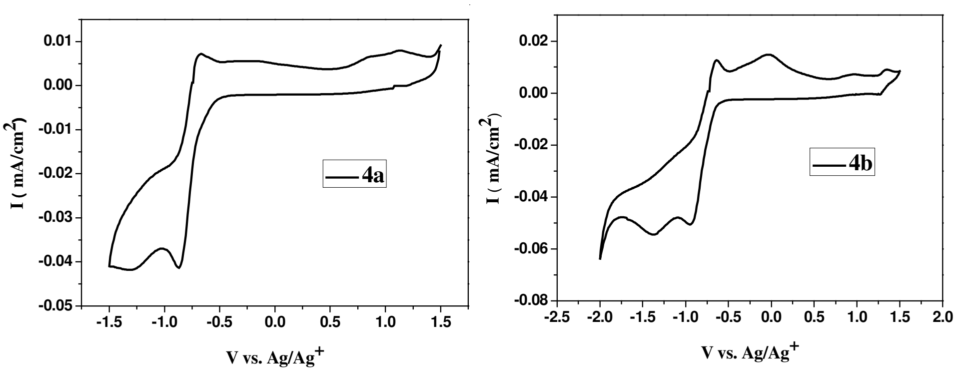


Fig. 3. Cyclic voltammetry of **4a** and **4b** realized in 0.1 M TBAPF$_6$-CH$_2$Cl$_2$, scan rate 100 mV/s

TABLE-3
LUMO OF COMPOUND **4a** AND **4b**

Compound	$E_{ox,1}$ (V)	$E_{red,1}$ (V)	$E_{1/2}$ (V)	LUMO (eV)
4a	-0.67	-0.87	-0.77	3.89
4b	-064	-0.95	-0.80	3.86

Thermal stability: It is found from Fig. 4 that all perylene bisimides possess excellent thermal stability. The initial decomposition temperature (the temperature where the weight loss exhibits 5 wt %) of compounds **3a** and **3b** are about 380 and 334 ºC, respectively. Whereas the same weight loss of **4a** and **4b** occurs at about 433 and 409 ºC, respectively. It is noteworthy that the thermal performances of **4a** and **4b** are similar.

Conclusion

A series of perylene bisimides have been synthesized derived from 1,7-dibromo- and 1,6,7,12-tetrabromo-perylene tetracarboxylic acid bisanhydride. The structures and properties of perylene bisimides are characterized by FT-IR, ^{1}H and ^{13}C NMR, MS, UV-vis, fluorescence spectroscopy and CV as well as TG measurement. Perylene bisimides exhibit good solubility, which could be highly soluble in conventional solvents, such as toluene, chloroform, dichloromethane and THF. UV-vis and fluorescence spectroscopy indicated that 4-*tert*-butylphenoxy groups in the bay region dramatically causes the red shift and the red shift location lies on the number of the substituent groups. Because of π-electron inductive effect of perylene core which

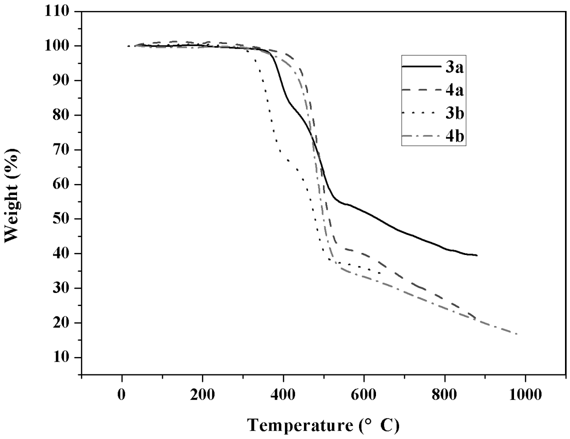


Fig. 4. Thermogravimetric analysis of compound **3a**, **3b**, **4a** and **4b**

was caused by four 4-*tert*-butylphenoxy groups, the LUMO level show significant influence and the LUMO of compounds **4a** and **4b** are 3.89 and 3.86 eV, respectively. TG measurement also shows that all perylene bisimides possess excellent thermal stability and the thermal performances are similar. The initial decomposition temperature of compounds **4a** and **4b** are about 433 and 409 ºC, respectively.

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