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Supplementary Information

Bay-substituted perylene diimide based donor-acceptor type copolymers: design, synthesis, optical and energy storage behaviours

Lalmohan Das ^a, Puspendu Das ^a, Sk Mustak Ahamed ^a, Ayan Datta^a, Arun Kumar Pal ^b, Ayan Datta ^b, and Sudip Malik^a*

^a School of Applied & Interdisciplinary Sciences (SAIS), Indian Association for the Cultivation of Science, 2A and 2B Raja S. C. Mullick Road, Jadavpur, Kolkata-700032, India. Email: psusm2@iacs.res.in

^b School of Chemical Sciences (SCS), Indian Association for the Cultivation of Science, 2A and 2B Raja S. C. Mullick Road, Jadavpur, Kolkata-700032, India.

Table of content

1.	General informational about materials and measurement	S2-S3
2.	Materials synthesis	.S3-S6
3.	Spectroscopic characterization	S7-S17
4.	Physical characterization of polymers	S18-S20
5.	Morphology study of copolymers	
6.	Electrochemical study	S23-S28
7.	Comparison Table	S29-S31
8.	Reference	S32

1.General information:

1.1 Materials:

3,4,9,10-perylenetetracarboxylic dianhydride, 2-ethylhexylamine, liquid bromine, 4formylboronic acid, rubeanic acid (dithiooxamide), Pd (PPh₃)₄, Pd (dppf)Cl₂, 2,1,3benzothiadiazole-4,7-bis (boronic acid pinacol ester), benzene-1,4-diboronic acid, diphenylamine, N-bromo succinimide (NBS), bis-pinacolato diborane, potassium carbonate (K₂CO₃) and potassium acetate (KOAC) were purchased from Tokyo chemical industry (TCI) India. The solvents like dichloromethane (DCM), petroleum ether, chloroform (CHCl₃), methanol (MeOH), tetrahydrofuran (THF), toluene, acetonitrile, acetone, hexane, ethanol (EtOH), dimethyl formamide (DMF), were purchased from Merck. All the solvents except THF and toluene were used after distillation. THF was used after drying under nitrogen with sodium metal and benzophenone. DMF was dried using distillation. The CDCl₃ (Aldrich) was also used as received. For spectroscopic measurement the HPLC grade of solvents were used. The column chromatography was performed on silica (silica gel 100-120 mesh).

1.2 Measurement and techniques:

The ¹H and ¹³C NMR spectra were performed on 400 MHz Bruker DPX spectrometer using CDCl₃ as solvent and TMS as standard reference with chemical shift given in parts per million (ppm) and solid-state ¹³C cross-polarization/magic angle spinning nuclear magnetic resonance (CP/MAS NMR) was performed with 600 MHz Bruker DPX spectrometer. The MALDI-TOF mass spectrometry was studied with Bruker Ultra flextreme (Bruker Daltonics Pvt. Ltd) in THF solvent using dithranol matrix. The Powder X-ray diffraction (PXRD) experiment was performed in Bruker D-8 Advanced SWAX, Germany diffractometer using Ni-filtered Cu K α ($\lambda = 1.5406$ Å) radiation in the small angle region (0.5°-10°) and (2°-50°) were recorded at a rate of $(2\theta) = 2^{\circ}$ per min. FEG-TEM image was recorded by JEM-2100F. FT-IR spectra was recorded on a FTIR instrument (Perkin Elmer, FTIR Spectrometer, S3 Spectrometer Two). Thermogravimetric analysis (TGA) was performed with PerkinElmer Thermogravimetric Analyzer (TGA 4000) with heating rate 10°C/min. under nitrogen atmosphere. DSC was done by Perkin-Elmer DSC 8000. The UV-vis spectroscopy was performed by Agilent Cary 5000 UV-vis NIR spectrophotometer. Emission studies (photoluminescence) in dispersed state and solid state were performed with a Horiba Jobin Yvon Fluoromax 3 spectrometer at corresponding excitation wavelength with slits 2/2. Fluorescence average lifetimes (t_{ava}) were measured through Time-Correlated Single Photon Counting (TCSPC) with Horiba Jobin Yvon Fluoromax 3 spectrometer with magic angle with 475 nm laser probe in dispersed state. Absolute quantum yield in dispersed state was done using integrated sphere on Edinburgh Instruments (Model: FLS1000-DD-STM). All electrochemical studies such as cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS) and galvanostatic charge/ discharge (GCD) were performed by using a CHI6087E electrochemical workstation (CH Instrument, USA).

2. Materials synthesis:

2.1 Synthesis of N, N'-Bis (2-ethylhexyl) perylene-3,4,9,10-tetracarboxylic diimide¹

(1)



Fig. S1 Synthesis of perylene diimide.

3,4,9,10-perylenetetracarboxylic dianhydride (2 g, 5.1 mmol) was taken in a two neck round bottom flask and degassed with nitrogen (N₂) 30 mL of dry dimethyl formamide (DMF) was added to the reaction mixture. Then 10 mL (50 mmol) of 2-ethylhexylamine was injected to it and refluxed at 150°C. After 8h, reaction mixture was allowed to stand for cooling to room temperature. It was poured into 100 mL (1N) HCl solution and stirred for 6 h. The precipitate was filtered by vacuum filtration and washed with 200 mL of 5% NaOH solution. The red precipitate was dried at vacuum the purification of crude product was done by column chromatography (CHCl₃: Hexane (1:1) as an eluent). Finally, a red colored pure product was obtained (2.5 g (yield = 80%)

¹**H NMR (400 MHz, CDCl₃) δ (ppm):** 8.7 (d, 4H), 8.65 (d, 4H), 4.18-4.11 (m, 4H), 1.97 (br, 2H), 1.33-0.88 (br, 28H).

¹³C NMR (400 MHz, CDCl₃) δ (ppm): 163.5, 134.4, 131.3, 129.1, 126.1, 123.1, 122.8, 44.2, 37.9, 30.8, 28.7, 24.1, 22.9, 14.1, 10.6

MALDI-TOF: Molecular weight = 614.79 (calculated), found = 617.23 (M+3)

2.2 1,7-diBromination of N, N'-Bis (2-ethylhexyl) perylene-3,4,9,10-tetracarboxylic diimide² (2):

400 mg (0.68 mmol) of **compound 1** was taken in a 250 mL two neck round bottom flask and degassed carefully with nitrogen three times. 20 mL of dry DCM was added to reaction mixture and stirred at room temperature. Then 2.6 mL (46.2 mmol) of liquid bromine was added dropwise to reaction mixture. Then the reaction mixture was refluxed at 70°C for 12 h. The reaction mixture was cooled and poured into 200 mL of saturated sodium thiosulphate pentahydrate solution to consume the excess bromine from the reaction mixture. The mixture was extracted with DCM and the combined organic phase was washed with water followed by brine solution. Organic phase was collected over the anhydrous Na₂SO₄ and concentrated by reduced pressure. The residue was purified by column chromatography (eluent: PET: DCM = 1:1). The first part was collected which is the mixture of regioisomer **1,7**-dibromo and **1,6**-dibromo of compound 1a. The regioisomerically pure **1,7**-dibromo of compound 1a (compound 2) (300 mg, yield 56%) was obtained by recrystallisation in CHCl₃: MeOH (v/v 5:1).

¹H NMR (400 MHz, CDCl₃) δ (ppm):9.48 (d, 2H), 8.91 (s, 2H), 8.69 (d, 2H), 4.18-4.11 (m, 4H), 1.95 (br, 2H), 1.37-0.90 (br, 28H)
¹³CNMR (400 MHz, CDCl₃) δ (ppm): 163.5, 163.1, 138.4, 133.2, 130.4, 129.6, 127.4, 123.6, 123.2, 121.2
MALDI-TOF: Molecular weight = 772.58 (calculated), Found = 771.15(M-1)

2.3 1,7-di (4-aldehyde phenyl) of N, N'-Bis (2-ethylhexyl) perylene-3,4,9,10tetracarboxylic diimide³ (3):

150 mg (0.19 mmol) of compound **2**, 142.4 mg (0.95 mmol) of 4-formylboronic acid, 330 mg (2.4mmol) of K₂CO₃ and 50 mg of Pd (PPh₃)₄ were taken in a 250 ml two neck round bottom flask and inert carefully with nitrogen. 20 ml dry THF and 2 ml H₂O were added to reaction mixture and refluxed at 70°C for one day. Then the reaction mixture was cooled and extracted with DCM. The combined organic phase was washed with water followed by brine solution. Then the solution was dried over Na₂SO₄ and concentrated by reduced pressure. The residue was purified by column chromatography (eluent: PET: DCM = 1:1) to get red color product (**3**) (140 mg yield 77%).

¹**H NMR (400 MHz, CDCl₃) δ (ppm):**10.15 (s, 2H), 8.64 (s, 2H), 8.18 (d, 2H), 8.08 (d, 4H), 7.78-7.26 (br, 6H), 4.17-4.09 (m, 4H), 1.93 (br, 2H), 1.37-0.88 (br, 28H).

¹³CNMR (400 MHz, CDCl₃) δ (ppm): 191.3, 163.5, 163.4, 148.0, 139.6, 136.1, 134.8, 132.6, 131.4, 130.7, 129.9, 129.0, 122.6, 122.3, 44.3, 37.9, 30.7, 28.7, 24.0, 23.0, 14.1, 10.6 MALDI-TOF: M. W=823.0, found=820.79(M-2)

2.4 Synthesis procedure of Polymer, TzTz-PDI:

50 mg (0.06 mmol) of **compound 3** and 20 mg (0.16 mmol) of Rubeanic acid were taken in a two neck round bottom flask and degassed with inert N₂. 20 ml dry DMF was added to reaction mixture and refluxed at 150° C for two days. After completion the reaction mixture was filtered and washed with 50 mL methanol. Further washing was continued by Soxhlet extraction in methanol, acetone, and hexane. The brown pure product (20 mg) was obtained after drying in vacuum.

2.5 Synthesis procedure of Polymer, Benz-PDI:

300 mg (0.388 mmol) of **compound 2** and 128 mg (0.775 mmol) benzene 1,4-diboronic acid, 300 mg (3.086 mmol) K₂CO₃ and 22.28 mg (0.019 mmol) of Pd (PPh₃)₄ were taken in a two neck round bottom flask. The entire setup was made inert by passing N₂. 30 mL dry THF and 2 mL H₂O were added to reaction mixture and refluxed at 70°C for 3 days. Then it was cooled to room temperature and poured into cold methanol. The resultant precipitated was filtered by vacuum filtration and washed with excess methanol, hot water and acetone successively. To remove the oligomers Soxhlet extraction was done with methanol, hexane and acetone. The red-colored pure product (100 mg) was dried into vacuum.

2.6 Synthesis procedure of polymer, Btz-PDI⁴:

200 mg (0.259 mmol) of **compound 2**, 352 mg (0.9065 mmol) of 2,1,3-benzothiadiazole-4,7-bis (boronic acid pinacol ester), 332.88 mg (2.40 mmol) K_2CO_3 and 15 mg (0.013 mmol) Pd (PPh₃)₄ were taken in a two neck RB. After that same procedure as previous polymer (**Benz-PDI**).

2.7 Synthesis of Bis(4-bromophenyl) amine (4):



500 mg (2.95 mmol) of diphenylamine was taken in a two neck round bottom flask and was inert with N₂. 10 mL of dry DMF added to it. The NBS solution (1.15 g (6.5 mmol) of N-bromo succinimide (NBS) was mixed with 10 mL of dry DMF) was added dropwise to the reaction mixture at 0°C and stirred for 3 h. Then, the entire reaction mixture was stirred at 60° C for 8 h. Aliquot was cooled and poured into 200mL of ice cooled water. White precipitate was filtered by vacuum filtration and dried into vacuum oven at 120°C. Further purification was done by column chromatography (stationary phase: silica gel; eluent: PET: DCM = 1:1) to get 675 mg (yield 70%) white crystal.

¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.37-7.35 (d, 4H), 6.93-6.91 (d, 4H), 5.65 (s, 1H)
 ¹³CNMR (400 MHz, CDCl₃) δ (ppm): 141.75, 132.35, 119.55, 113.42
 HR-MS (ESI): Molecular weight = 427.02 found=427.86

2.8 Synthesis of bis (4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) phenyl) amine⁵
(5):



In a two neck round bottom flask, 500 mg (1.52 mmol) of **compound 4**, 1.158 g (4.56 mmol) of bis-pinacolato diborane, 179.01 mg (1.824 mmol) of potassium acetate (KOAC) and 44.5 mg (0.0608 mmol) of Pd(dppf)Cl₂ were taken. The entire system was purged with N₂ gas for 15 mins. 60 mL of dry 1,4-dioxane was added to it and heated up to 110° C for 16 h. After cooled at room temperature reaction mixture was extracted with DCM and brine solution. The combined organic phase was passed over Na₂SO₄ and concentrated by reduce pressure. Further purification of the residue was done by column chromatography on silica gel and eluent (PET: DCM=1:1) to give 550 mg (yield 84%) white crystal **compound 5**.

¹H NMR (400 MHz, CDCl₃) δ (ppm):7.73-7.71 (d, 4H), 7.09-7.04 (d, 4H), 1.33 (br, 24H)

¹³CNMR (400MHz, CDCl3) δ (ppm): 145.04, 136.38, 116.82, 83.70, 24.91.

MALDI-TOF: Molecular weight = 421.15 (calculated), found=421.50 (M)⁺

2.9 Synthesis procedure of polymer, NH-PDI:

200 mg (0.25 mmol) of **compound 2**, 327 mg (0.77 mmol) of intermediate **5**, 332.8 mg (2.40 mmol) of K_2CO_3 and 15 mg (0.013 mmol) of Pd (PPh₃)₄ were taken in two neck RB. Next, same procedure was followed as **Benz-PDI** finally 110 mg black **NH-PDI** was obtained.

3. Spectroscopic characterization of compound:



Fig. S2 ¹H NMR of compound 1 in CDCl₃.





Fig. S4 MALDI-ToF mass of compound 1 using dithranol matrix and CHCl₃.



Fig. S5 ¹H NMR of compound 2 in CDCl₃.



Fig. S6 ¹³C NMR of compound 2 in CDCl₃.



Fig. S7 MALDI-ToF mass of compound 2 using dithranol matrix and CHCl₃.



Fig. S8 ¹H NMR of compound 3 in CDCl₃.



Fig. S9 ¹³C NMR of compound 3 in CDCl₃.



Fig. S10 MALDI-ToF mass of compound 3 with dithranol matrix.



Fig. S11 ¹H NMR of compound 4 in CDCl₃.



Fig. S12 ¹³C NMR of compound 4 in CDCl₃.



Fig. S13 HRMS mass of compound 4.



Fig. S14 ¹H NMR of compound **5** in CDCl₃.





Fig. S16 MALDI-ToF mass of compound 5 with dithranol matrix.





Fig. S17 Solid state ¹³C NMR of (a) Benz-PDI, (b) TzTz-PDI. c) GPC trace of Benz-PDI. All the copolymers are low soluble in chlorinated solvent likes CHCl₃, DCM, and chlorobenzene. Among all the polymers, Benz-PDI is more soluble in chlorobenzene. Gel permeation chromatography (GPC) analysis of Benz-PDI was performed on an Agilent 1260 Infinity II system equipped with MIXED-C column using polystyrene as internal standard and chlorobenzene as eluent at 35 °C.

Result: Mw =8485, PDI =1.62



4. Physical characterization of polymers.

Fig. S18 Differential scanning calorimetry (DSC) curve of a) Benz-PDI, b) Btz-PDI, c) TzTz-PDI, and d) NH-PDI.





Fig. S19 a) The optimized molecular structures of the four complexes along with the dihedral angle between PDI and different aromatic linker. b) Calculated transition density difference plots of the investigated systems. The yellow color refers the density loss and green color refers the density gain. The isovalues are of $\pm 0.0004au$.

The TDDFT calculations show that in all cases, the absorption peaks are arisen mainly due to HOMO \rightarrow LUMO electronic transition. The absorption wavelengths for the Benz-PDI, Btz-PDI, TzTz-PDI and NH-PDI are calculated as 474 nm (f = 1.13), 472 nm (f =1.08), 482 nm (f = 0.95) and 521 nm (f = 0.63), respectively. The corresponding energy density plots are illustrated in Fig. S19b. Clearly, the impact of the increasing in donor strength from Benz-PDI to NH-PDI is notable. The HOMO-LUMO overlap integral (**S**_{pq}) for the investigated complexes are calculated as 0.84, 0.85, 0.69 and 0.52, respectively. A decrease in the **S**_{pq} value for the NH-PDI agrees with the condition for the charge-transfer phenomenon.



Fig. S20 a) Powder XRD of all copolymer. FEG-TEM image of b) Benz-PDI c) Btz-PDI.



Fig. S21 UV-Vis absorption spectra of Br₂-PDI in DCM.



Fig. S22 TCSPC of all polymers in chloroform solution.

5. Morphology study of copolymers:



Fig. S23 a) FESEM image of all copolymer films which are prepared from NMP solution and it was dried for 7 days in vacuum prior to observation.



Fig. S24 a) FEGTEM image of all copolymers and samples were prepared from the dispersed solution in isopropanol and it was cast on the carbon coated Cu grid. The mark part is revealing the stacking of sheets which support the FESEM observation of NH-PDI.

6.Electrochemical study.



Fig. S25 a) Schematic representation of electrode fabrication process b) graphical representation of three electrode and two electrode set-ups, respectively. c) Redox behaviour of NH-PDI copolymer, showing the formation of quinoid state.

6.2 Calculation of specific capacitance (C_{sp}), Energy density (E) and a power density (P_{max}):

The discharged specific capacitance (C F g^{-1}) was calculated from galvanostatic charge discharge (GCD) curves with the following equation 1.

$$C_{sp} = \frac{i \times \Delta t}{m \times \Delta V} \operatorname{F} g^{-1} \qquad (1)$$

Where, i is the current density, Δt (s) is the discharge time, m (g) is the mass of active materials deposited on working electrode and ΔV is the voltage window during discharge process.

The energy density (E, Wh Kg⁻¹) and power density (P_{max} , W Kg⁻¹) of all polymers were calculated from GCD curve by using the following equation 2,3.

$$E = \frac{C_{sp} \times (\Delta V)^2}{7.2} \operatorname{Wh} \operatorname{kg}^{-1} \qquad (2)$$

Where, C_{sp} is discharged specific capacitance and ΔV is the potential range.

$$P_{max} = \frac{E \times 3600}{\Delta t} \,\mathrm{W \, kg^{-1}} \tag{3}$$

Where, E is energy density and Δt (s) is the discharge time.

Copolymer	Specific capacitance (C _{sp}) (F g ⁻¹) at 0.5A g ⁻¹	Energy density(E) (Wh Kg ⁻¹)	Power density(P) (W Kg ⁻¹)
Benz-PDI	131	22	274.8
Btz-PDI	119	20	274.7
TzTz-PDI	113	19	275
NH-PDI	363	61	275

Table S1 Calculated value of Csp, E, and Pmax of all polymer in three electrode system.



Fig. S26 Change of peak current with scan rate of 10 mV s-1 to 100 mV s-1 of NH-PDI in three electrode system.



Fig. S27 a) Discharge capacitance (%) and coulombic efficiency (%) with number of cycles at current density 8 A g^{-1} of fabricated device of NH-PDI in three electrode system. b) Absorption spectra of NH-PDI (CHCl₃ solution) before and after 15000 cycles. FEG-TEM images of NH-PDI c) before and d) after 15000 cycles.



Fig. S28 Electrochemical Impedance spectra of all polymer in three electrode system.

Copolymer	Solution resistance (Ω)			
Benz-PDI	1.5			
Btz-PDI	2.5			
TzTz-PDI	6.7			
NH-PDI	3.5			

 Table S2 Solution resistance of copolymer in three electrode system.



Fig. S29 $v^{1/2}$ vs $i/v^{1/2}$ plot to determined k_1 (slope) and k_2 (intercept) value.



Fig. S30 Electrochemical Impedance spectra (EIS) of NH-PDI device in solid state.



Fig. S31 Picture showing individual potential of one device, series connection of five devices and illumination of LED (3.0V)

Chemical	Electr	Type of working electrode	Specific capacitance	Energy density (E)	Power	Cyclic stability @ current	% retention Csp after nth	Reference
structure	olyte	ciccitouc	(Csp)	uchisity (L)	(P _{max})	density A g ⁻¹	cycle	
		Electro polymerisation On ITO	241.2 F g ⁻¹ at 0.5 Ag ⁻¹	22.07 mWh cm ⁻³	17.4 W cm^{-3}			6
4Cz-4Cl-PBI								
PDI-Pyr	1 M H ₂ SO ₄	Two electrodes Graphite foil	192 F g ⁻¹ at 1 Ag ⁻¹	54 Wh kg-1	27 KW kg ⁻¹	4000 @ 3	88.13	7
	0.5 M H ₂ SO ₄	Three electrodes Graphite foil	124 F g ⁻¹ at 0.5 Ag ⁻¹	2 Wh kg-1	22 KW kg ⁻¹	5000 @ 5	100	8
P(PDI-r-BDT)	PC- LiClO ₄ gel	Three electrodes	113 F g ⁻¹ at 0.5 Ag ⁻¹	9.1 Wh kg ⁻¹	82 KW kg ⁻¹	4000 @ 2	100	9
	PMMA / TBAPF6/a cetonitrile gel	electro polymerisation	78.6 F g ⁻¹ at 0.5 Ag ⁻¹	8.95 Wh kg ⁻¹ At 1Ag ⁻¹	76.8 KW kg ⁻¹ At 100 Ag ⁻¹	1000 @ 10	79.6	10
PDI-4Cl-EDOT- 012								
BZ	5M NaCl	Three electrodes Carbon paper	$\begin{array}{c} 250 \pm 34 \; F \; g^{-1} \\ at \; 0.5 \; Ag^{-1} \end{array}$					11

7. Table-S3. Comparison table

	5M NaCl	Three electrodes Carbon paper	$\begin{array}{c} 292 \pm 47 \\ F \ g^{-1} \ at \\ 0.5 \ Ag^{-1} \end{array}$					11
NPH								
P-ANH	5M NaCl	Three electrodes Carbon paper	$\frac{147 \pm 18 \ F \ g^{-1}}{at \ 0.5 \ Ag^{-1}}$					11
	PMMA LiClO ₄ gel electrolyte	Two electrodes	84.2 F g ⁻¹ at 0.25 Ag ⁻¹	112.4 Wh kg ⁻¹	18,600 W kg ⁻¹	45000 @ 2	70	12
T2)/MWCNT								
	0.1M TBAPF ₆	Three electrodes Carbon cloth	196 F g⁻¹ at 1Ag ⁻¹			5000 @ 5	76	13
Figure 1 and the second	0.5 M H ₂ SO ₄	Three electrodes Graphite sheet	131 F g⁻¹ at 0.5Ag ⁻¹	22 Wh kg ⁻¹	274.8 W kg ⁻¹			This work
Btz-PDI	0.5 M H ₂ SO ₄	Three electrodes Graphite sheet	119 F g⁻¹ at 0.5Ag ⁻¹	20 Wh kg ⁻¹	274.7 W kg ⁻¹			This work

	0.5 H ₂ SO ₄	М	Three electrodes Graphite sheet	113 F g⁻¹ at 0.5Ag ⁻¹	19 Wh kg ⁻¹	274.7 W kg ⁻¹			This work
	0.5 M H ₂ SO ₄		Three electrodes Graphite sheet (working electrode)	363.4 F g⁻¹ at 0.5A g ⁻¹	61.1 Wh kg ⁻¹	275 W kg ⁻	15000 @ 8	80	This work
^é NH-PDI	PVA- H2SO4 (1:1)		Two electrodes Graphite sheet (working electrode)	134.2 F g⁻¹ at 0.5A g ⁻¹	22.2 Wh kg ⁻¹	274.8 W kg ⁻¹	20000 @ 8	69.1	This work

7. Reference

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