INTRODUCTION

Semiconducting polymers offer distinct advantages over their inorganic counterparts including the following: light weight, improved mechanical flexibility, large active layers, and can be manufactured using low-cost solution processing. It is hypothesized that conjugated polymers will fulfill a larger number of functions providing large-scale industrial manufacture of organic electronics becomes feasible. In actuality, the commercial success of organic light-emitting diodes (OLEDs) should serve as a platform to promote the efficacy of organic optoelectronic devices. Aside from OLEDs, the electrical, magnetic, and optical properties of conjugated polymers makes them promising candidates for organic photovoltaic devices, organic field-effect transistors, electrochromic devices, and stable electronic memories on flexible circuit boards. Furthermore, they have proven to be suitable candidates for gas and humidity sensing and bio-sensing, which can be attributed to their sensitivity to biological, chemical, and physical perturbations.

The attractiveness of conjugated polymers lies in the facile tunability of their optical, electronic, and morphological properties. Previous literature has proven that copolymerising an electron-rich donor monomer with an electron-deficient acceptor unit in a so-called donor-π-acceptor (D-π-A) arrangement is an effective method of tuning these properties. The alternation of donor and acceptor units results in hybridization of the lowest unoccupied molecular orbital (LUMO) of the acceptor and highest occupied molecular orbital (HOMO) of the donor, resulting in a decreased band gap. Moreover, it is believed that the D-π-A arrangement promotes intramolecular charge transfer along the polymer backbone, which can enhance charge transfer along the polymer backbone, which can enhance carrier mobility. This D-π-A methodology has been used to produce a range of conjugated chromophores that absorb light over the whole solar spectrum.

Literature reports have speculated that the highly planar and symmetrical structure of thieno[3,4-c]pyrrole-4,6-dione (TPD) repeat units along polymer chains could improve electron delocalization thereby enhancing interchain interactions and increasing their hole mobility. Furthermore, the nitrogen of the imide functionality can be substituted with solubilizing groups, facilitating solution processing of the final polymer. Finally, the electron-withdrawing nature of the TPD moiety allows it, once polymerized with alternate electron donor units, to form the highly desirable D-π-A arrangement. There is currently a large body of work covering the use of TPD in optoelectronic devices, mainly in photovoltaic devices and organic field-effect transistors. TPD-based copolymers have displayed efficiencies up to 8% when fabricated into a bulk heterojunction solar cell using PC70BM as the acceptor. Additionally, TPD-containing copolymers have displayed high hole mobilities (1.29 cm²/Vs) when fabricated into field-effect transistors.

2,6-Linked anthracene units are finding use in D-π-A conjugated polymers. Iraqi and co-workers presented the...
preparation of D-π-A polymers with alternating 2,6-linked anthracene units with aryloxy substituents at their 9,10-positions and various benzothiadiazole alternate repeat units. Bulk heterojunction solar cells fabricated from these polymers displayed efficiencies ranging from 1.93% to 4.17% when blended with PC70BM.

Copolymers comprising alternating benzo[1,2-b:4,5-b']dithiophene units and electron-donating units have been reported in literature. However, to the best of our knowledge, nobody has reported any alternating TPD-anthracene copolymers. In the present contribution, we report the synthesis of three alternating copolymers comprising TPD with varying substituents as electron-accepting moieties and triisopropylsilylacetylene-functionalized anthracene as electron-donor moieties. The preparation of polymers PTATPD(O), PTATPD(DMO), and PTATPD(BP) (Fig. 1) is presented along with a study of their optical, electrochemical, thermal, and structural properties.

RESULTS AND DISCUSSION

Polymer synthesis

The synthetic route for the preparation of the three TPD monomers is outlined in Scheme 1. It is perhaps worth mentioning that M2 and M3 were obtained in slightly lower yields, relative to M1. This was attributed to the additional steric hindrance associated with 3,7-dimethyloctan-1-amine and 4-butylaniline. The structures of PTATPD(O), PTATPD(DMO), and PTATPD(BP) are outlined in Fig. 1. Suzuki polycondensation of M1 with M4, M2 with M4, and M3 with M4 yielded PTATPD(O), PTATPD(DMO), and PTATPD(BP), respectively. Pd(OAc)2 and tri(o-toly)phosphine were used as the catalyst and sodium hydrogen carbonate as the base. All polymerizations were left for 24 hr with large quantities of bright red precipitate forming. The polymers were fractionated via Soxhlet extraction using methanol, acetone, hexane, and toluene. The toluene fractions were collected, reduced in vacuo, and precipitated in methanol. Subsequent studies were conducted on the toluene fractions only. The chemical structures of PTATPD(O), PTATPD(DMO), and PTATPD(BP) were confirmed via 1H nuclear magnetic resonance (NMR) (supplementary information) and elemental analysis. The number-average molecular weight (Mn) and weight-average molecular weight (Mw) were estimated using gel permeation chromatography (GPC) using 1,2,4-trichlorobenzene as the eluent at 140°C (Table 1). Substituting n-octyl chains in PTATPD(O) for dimethyloctyl chains in PTATPD(DMO) on the TPD moiety results in a significantly higher Mw. It is speculated that the branched alkyl chain disrupts intermolecular interactions in solution, increasing the solubility of the resulting polymer. Thus, the final polymer product is able to achieve a higher Mw before precipitating out of solution. PTATPD(BP) displays a higher Mw (97,700 Da) relative to PTATPD(O) (74,600 Da). However, the Mn of PTATPD(BP), and PTATPD(O) are 25,900 and 27,300 Da, respectively. Thus, PTATPD(BP) has a significantly wider distribution of molecular weights, relative to PTATPD(O).

Figure 1. Structures of PTATPD(O), PTATPD(DMO), and PTATPD(BP).

Scheme 1. Synthetic route towards the TPD monomers M1, M2, and M3.
Optical properties

The ultraviolet–visible absorption properties of the polymers were investigated in solution and film state (Fig. 2). The results are summarized in Table 1. All polymers display an intense transition band at 383 nm in solution, corresponding to a π-π* transition. This band is red-shifted to 388, 390, and 389 nm for PTATPD(O), PTATPD(DMO), and PTATPD(BP), respectively, in films states. PTATPD(O), PTATPD(DMO), and PTATPD(BP) display a second, less intense transition at 529, 533, and 532 nm in solution, respectively. This is red-shifted to 548, 553, and 550 nm for PTATPD(O), PTATPD(DMO), and PTATPD(BP) in film states, respectively. This transition corresponds to intramolecular charge transfer between the TPD-acceptor unit and the anthracene-donor moiety. The small bathochromic shift (∼20 nm) that is observed from solution to films indicates that the polymers adopt a similar conformation in both states. The optical band gaps, as estimated from the onset of absorption wavelengths, are 2.16, 2.14, and 2.12 eV for PTATPD(O), PTATPD(DMO), and PTATPD(BP), respectively.

The results indicate that the optical properties of the polymers are not significantly affected by the molecular weight or the nature of the substituent attached to the TPD moiety. It is speculated that there is a large torsion angle between the anthracene moiety and the TPD unit, arising from the intramolecular repulsion between the bulky TIPS group and functionalized-imide on the TPD. Consequently, the planarity of the polymer is disrupted. Thus, orbital overlap between non-coplanar aromatics is poor, leading to localization of the π-electron wave functions and a decreased electronic conjugation. Therefore, the true effects the different substituents have on the optical properties are not revealed. It is hypothesized that the lack of a regular, planar structure will result in an amorphous polymer.

Najari and co-workers synthesized a series of thiено[3,4-c]pyrrole-4,6-dione-alt-2,7-carbazole polymers. The lowest optical band gap reported by the group was 1.97 eV. They speculated that the alkyl chains twist the polymer backbone decreasing the effective conjugation length resulting in a wider optical band gap.[38]

The homopolymer, regioregular poly(3-hexylthiophene-2,5-diyl) (P3HT), displays a reduced optical band gap of 1.9 eV and a larger bathochromic shift from solution to film.[39,40] It is hypothesized that the localization of the π-electron wavefunction, decreased electronic conjugation, and amorphous nature of the polymers synthesized in this report are responsible for the wider optical band gaps, relative to P3HT. Furthermore, it is known that the solar harvesting of P3HT is restricted by its mismatch with the solar spectrum.[38,40] The optical band gaps of PTATPD(O), PTATPD(DMO), and PTATPD(BP) are wider than that of P3HT. Thus, these polymers are not optimized with respect to the maximum photon flux of the solar spectrum.

The photoluminescence (PL) emission spectra of the polymers in solution and solid films are illustrated in Fig. 2. The PL spectra of all polymers in chloroform solution and thin films were excited at incident wavelengths of their absorption λmax. In solution, non-coplanar aromatics is poor, leading to localization of the π-electron wave functions and a decreased electronic conjugation. Therefore, the true effects the different substituents have on the optical properties are not revealed. It is hypothesized that the lack of a regular, planar structure will result in an amorphous polymer.

Table 1. A summary of the GPC, UV–vis absorption, and photoluminescence data for PTATPD(O), PTATPD(DMO), and PTATPD(BP)

| Polymer | Mw (Da) | PDI | λmax (nm) | ε (M⁻¹ cm⁻¹) | λmax (nm) | Eg,opt (eV) | λmax (nm) | λmax (nm) | Stokes shift (nm) |
|---------|---------|-----|-----------|--------------|-----------|------------|-----------|-----------|------------------|
| PTATPD(O) | 74,600 | 2.73 | 383, 529 | 62,500 | 388, 548 | 2.16 | 545 | 617 | 69 |
| PTATPD(DMO) | 106,700 | 2.61 | 383, 533 | 61,300 | 390, 553 | 2.14 | 544 | 619 | 66 |
| PTATPD(BP) | 97,700 | 3.77 | 383, 552 | 62,000 | 389, 550 | 2.12 | 552 | 629 | 79 |

aMeasurements conducted on the toluene fraction of the polymer using DRI detection.
bAbsorption coefficient measured at λmax = 383 nm in chloroform solution.
cOptical energy gap determined from the onset of the absorption band in thin film.
dStokes shift determined from film studies.

GPC, gel permeation chromatography; UV–vis, ultraviolet–visible; DRI, differential refractive index.

Figure 2. Normalized absorption spectra (full line) and photoluminescence spectra (dashed line) of PTATPD(O), PTATPD(DMO), and PTATPD(BP) in: (a) chloroform solution and (b) thin films. This figure is available in colour online at wileyonlinelibrary.com/journal/pat
PTATPD(O), PTATPD(DMO), and PTATPD(BP) exhibit emission maxima at 545, 544, and 552 nm, respectively. Interestingly, all polymers display a shoulder peak in their solution PL spectra. We tentatively attribute this to an unresolved vibrational-electronic transition. The emission maxima are red-shifted to 617, 619, and 629 nm for PTATPD(O), PTATPD(DMO), and PTATPD(BP), respectively, in solid state. The small Stokes shift of 69, 66, and 79 nm, respectively, indicates that there is a small energy difference between the ground state and excited states of the polymers.

Electrochemical properties

Cyclic voltammetry was used to characterize the frontier energy levels of the three polymers. The cyclic voltammograms on drop cast films in tetrabutylammonium perchlorate acetonitrile solutions of PTATPD(BP), PTATPD(O), and PTATPD(DMO) display irreversible oxidation peaks (Fig. 3). It is hypothesized that oxidation of the polymer yields a radical cation state localized on the imide moiety, which undergoes an irreversible chemical transformation. In contrast, the reduction peaks of all polymers are quasi-reversible. The energy levels of all polymers were estimated from the onset of the oxidation and reduction potentials. PTATPD(BP), PTATPD(O), and PTATPD(DMO) display the same HOMO level (−5.9 eV). The results indicate that incorporation of different substituents on the imide functionality does not alter the HOMO level of the resulting polymer. However, it does have a slight influence on the LUMO level of the resulting polymer. PTATPD(DMO) displays the highest LUMO level (−3.3 eV). In comparison, PTATPD(BP) and PTATPD(O) display a lower LUMO level (−3.4 eV). The results suggest that attaching different substituents to the imide functionality can subtly perturb the electron density on the main polymer chain.

The electrochemical band gap of the polymers is significantly larger than their corresponding optical band gap. It is believed that the additional energy is the result of the strong Coulomb attraction of excitons, which needs to be overcome in order to generate free charge carriers.

The results obtained suggest that the thermal properties of the conjugated polymers are influenced by the size of the pendant group attached to the imide functionality, the larger the group the lower the $T_d$ and $T_g$. PTATPD(DMO) and PTATPD(BP) display lower $T_d$ and $T_g$ temperatures relative to PTATPD(O). We tentatively hypothesize that the larger groups create “free volume” within the polymer by increasing separation between polymer chains, allowing the polymer to reorganize more easily within solid state resulting in lower $T_d$ and $T_g$ temperatures.

Powder X-ray diffraction

Powder x-ray diffraction patterns of polymers PTATPD(O), PTATPD(DMO) and PTATPD(BP) were obtained to investigate the molecular organization of polymers in solid state (Fig. 5). The X-ray diffraction (XRD) patterns of all polymers display a single broad diffuse feature, which is consistent with the random scatter of an amorphous solid. Furthermore, the lack of a peak

![Figure 3](https://wileyonlinelibrary.com/journal/pat)  
**Figure 3.** Cyclic voltammograms of PTATPD(O), PTATPD(DMO), and PTATPD(BP). This figure is available in colour online at wileyonlinelibrary.com/journal/pat

![Figure 4](https://wileyonlinelibrary.com/journal/pat)  
**Figure 4.** (a) Thermogravimetric analysis and (b) differential scanning calorimetry plots of PTATPD(O), PTATPD(DMO), and PTATPD(BP) with a heating rate of 10°C min$^{-1}$ under an inert atmosphere of nitrogen. This figure is available in colour online at wileyonlinelibrary.com/journal/pat
It is speculated that the intramolecular repulsion between solubilizing groups within the polymer reduces the planarity and decreases the electronic conjugation. Thus, changing the pendant-group has little impact on the optical properties of the resulting polymer.

**EXPERIMENTAL**

All materials were purchased from commercial suppliers and used as received, unless otherwise stated. Toluene was dried and distilled over sodium under an inert argon atmosphere. Acetonitrile was dried and distilled over phosphorous pentoxide under an inert argon atmosphere, then stored over molecular sieves (3 Å). 1,3-Dibromo-5-(n-octyl)-4H-thieno[3,4-c]pyrrole-4,6-(5H)-dione (M1)\[41\], 1,3-dibromo-5-(3,7-dimethyloctyl)-4H-thieno[3,4-c]pyrrole-4,6(5H)-dione (M2)\[42\] and 2,6-dibromo-9,10-bis(3-trisopropylsilylacetylene)anthracene\[43\] were prepared according to literature procedures.

**Measurements**

\(^1\)H and \(^13\)C NMR spectra were recorded on a Bruker AV 400 (400 MHz) using chloroform-d (CDCl\(_3\)) or acetone-d\(_3\) as the solvent. \(^1\)H NMR spectra of the polymers were recorded on a Bruker Avance III HD 500 (Bruker, Coventry, United Kingdom) (500 MHz) spectrometer at 100°C using 1,2-dideutrotetrachloroethane (C\(_2\)D\(_2\)Cl\(_4\)) as the solvent. Coupling constants are given in Hertz (Hz). Carbon, hydrogen, nitrogen, and sulfur elemental analysis was performed on a Perkin Elmer 2400 series CHNS/O analyzer (Perkin Elmer, Buckinghamshire, United Kingdom). Analysis of halides was undertaken using the Schöniger flask combustion method. GPC analysis was conducted on polymer solutions using 1,2,4-trichlorobenzene as the eluent. Polymer samples were spiked with toluene as a reference. GPC curves were obtained using a Visocet GPCmax VE2001 GPC solvent/sample module and a Waters 410 Differential Refractometer (Waters, Hertfordshire, United Kingdom), which was calibrated using a series of narrow polystyrene standards (Polymer Laboratories). Thermogravimetric analyses (TGAs) were obtained using a Perkin Elmer TGA-1 Thermogravimetric Analyzer (Perkin Elmer, Buckinghamshire, United Kingdom). Analysis of halides was undertaken using the Schöniger flask combustion method. GPC analysis was conducted on polymer solutions using 1,2,4-trichlorobenzene at 140°C as the eluent. Polymer samples were spiked with toluene as a reference. GPC curves were obtained using a Visocet GPCmax VE2001 GPC solvent/sample module and a Waters 410 Differential Refractometer (Waters, Hertfordshire, United Kingdom), which was calibrated using a series of narrow polystyrene standards (Polymer Laboratories). Thermogravimetric analyses (TGAs) were obtained using a Perkin Elmer TGA-1 Thermogravimetric Analyzer (Perkin Elmer, Buckinghamshire, United Kingdom) at a scan rate of 10°C min\(^{-1}\) under an inert nitrogen atmosphere. DSCs were obtained using a Perkin-Elmer Pyris 1DSC (Perkin Elmer, Buckinghamshire, United Kingdom) in the temperature range 50–220°C. Powder X-ray diffraction samples were recorded on a Bruker D8 advance diffractometer (Bruker, Coventry, United Kingdom) with a CuKa radiation source (1.5418 Å, rated as 1.6 kW). The scanning angle was conducted over the range 2–40°. Ultraviolet–visible absorption spectra were recorded using a Hitachi U-2100 Double Beam UV/Visible Spectrophotometer (Hitachi, Berckshire, United Kingdom). Polymer solutions were made using chloroform and measured using quartz cuvettes (path length = 1 × 10\(^{-2}\) m). Thin films, used for absorption spectra, were prepared by drop-casting solutions onto quartz plates using 1 mg cm\(^{-3}\) polymer solutions that were prepared with chloroform. Photoluminescence spectra were recorded on a Horiba FluoroMax 4 spectrometer (Horiba, Middlesex, United Kingdom). Polymer solutions were made using chloroform and measured using quartz cuvettes (path length = 1 × 10\(^{-2}\) m). Thin films were prepared by drop-casting solutions onto quartz plates using 5 mg cm\(^{-3}\) polymer solutions that were prepared with chlorobenzene. Cyclic voltammograms were recorded using a Princeton Applied
1,3-Dibromo-5-(4-butylophenyl)thieno[3,4-c]pyrrole-2,6-dione (M3)

THF (12 cm³) was added to a round bottom flask containing 4,6-dibromothieno[3,4-c]fur-an, 1,3-dione (1.50 g, 4.81 mmol) and 4-butylaniline (789 mg, 5.29 mmol). The mixture was degassed and heated to 50°C for 3 hr. Upon completion, the reaction mixture was cooled to room temperature. Thiouyl chloride (5 cm³) was added, and the reaction was stirred at 55°C for a further 4 hr. The reaction was precipitated into methanol (150 cm³). The product was filtered off and washed with methanol to give the title compound as white needles (1.73 g, 3.99 mmol, 81%).

1H NMR (400 MHz, CDCl3): ∂ (ppm) 7.31 (d, J = 8.56 Hz, 2H), 7.27 (d, J = 8.56 Hz, 2H), 2.67 (t, J = 7.76 Hz, 2H), 2.67 (m, 2H), 1.39 (H, J = 7.74 Hz, 2H), 0.96 (t, J = 7.32 Hz, 3H).

13C NMR (400 MHz, CDCl3): ∂ (ppm) 159.41, 143.54, 129.08, 129.00, 126.18, 113.92, 35.35, 33.36, 22.34, 13.90. El-MS (m/z): [M]+ calculated for C₁₈H₁₂Br₂NO₂S: C, 43.31; H, 2.96; N, 3.16; Br, 36.06; S, 3.87. Found: C, 76.31; H, 8.79; N, 1.39; S, 3.25.

2,6-Bis-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9,10-bis(triisopropylsilylacetylene)anthracene (M4)

A flask containing 2,6-dibromo-9,10-bis(trisopropylsilylacetylene)anthracene (3.00 g, 4.33 mmol), bis(pinacolato) diboron (1.83 g, 15.01 mmol), potassium acetate (2.52 g, 24.97 mmol), and Pd (dpdf)Cl₂ (0.2 g, 0.25 mmol) was placed under an argon atmosphere using standard Schlenk line techniques. Anhydrous THF (50 cm³) was added, degassed, and heated at 90°C for 48 hr. The mixture was cooled to room temperature, poured into water (50 cm³), and extracted with diethyl ether (3 × 100 cm³). The organic phases were combined, washed with H₂O (2 × 100 cm³), and dried (MgSO₄). The crude product was purified by dissolving in the minimum amount of hot ether and precipitated by dissolving in the minimum amount of hot ether and precipitated in vacuo and precipitated in methanol (500 cm³). The resulting precipitate was stirred overnight and the bright red solid collected by filtration (245 mg, 0.306 mmol, 81%). GPC: tolune fraction, M₉ = 27,300 g mol⁻¹, Mₘ = 74,600 g mol⁻¹, Polydispersity index, PDI = 2.73. 1H NMR (500 MHz, C₂D₂Cl₄): ∂ (ppm) 9.53 (s, 2H), 8.83 (d, J = 7.48 Hz, 2H), 8.44 (d, J = 7.63 Hz, 2H) 3.75 (br, 2H), 1.79 (br, 2H), 1.50–1.25 (m, 52H), 0.91 (s, 3H). Anal. Calculated for C₅₀H₆₁NO₂SSi₂: C, 75.04; H, 8.19; N, 1.75; S, 3.41; found: C, 75.08; H, 8.13; N, 1.66; S, 3.87.

Poly[9,10-bis(trisopropylsilylacetylene)anthracene-2,6-diyl-alt-5,3,7-dimethylocylthieno[3,4-c]pyrrole-4,6-diene] (PTATPD(DMO))

PTATPD(DMO) was synthesized according to the polymerization method outline in PTATPD(O). M₄ (0.299 g, 0.378 mmol) and M₂ (0.171 g, 0.378 mmol) were added to a round bottom flask and placed under an inert atmosphere of argon using standard Schlenk line techniques. Anhydrous THF (8 cm³) and a saturated NaHCO₃ (2 cm³), previously degassed, were added, and the mixture was degassed again. Pd(OAc)₂ (6.2 mg, 27.6 µmol) and tri(o-toly)phosphine (16.3 mg, 53.6 µmol) were added, and the reaction mixture was degassed and placed under an inert atmosphere of argon using standard Schlenk line techniques. Anhydrous THF (8 cm³) and a saturated NaHCO₃ (2 cm³), previously degassed were added, and the system was degassed again. Pd(OAc)₂ (6.1 mg, 27.2 µmol) and tri(o-toly)phosphine (16.4 mg, 53.9 µmol) was added, and the reaction was degassed again. The polymerization was left for 24 hr. Upon completion, the mixture was cooled to room temperature, and bromobenzene (0.15 cm³, 0.94 mmol) was added. The mixture was degassed and heated to 90°C for 1 hr. Upon completion, the reaction was cooled to room temperature and phenyl boronic acid (150 mg, 1.23 mmol) was added. The mixture was degassed and heated at 90°C for 1.5 hr. The mixture was cooled to room temperature, precipitated in methanol, and left to stir overnight. The polymer was filtered through a membrane and subject to Soxhlet extraction with solvent in the following order: methanol, acetone, hexane, and tolune. The tolune fraction was concentrated in vacuo and precipitated in methanol (500 cm³). The resulting precipitate was stirred overnight and the bright red solid collected by filtration (245 mg, 0.306 mmol, 81%). GPC: tolune fraction, M₉ = 27,300 g mol⁻¹, Mₘ = 74,600 g mol⁻¹, Polydispersity index, PDI = 2.73. 1H NMR (500 MHz, C₂D₂Cl₄): ∂ (ppm) 9.53 (s, 2H), 8.83 (d, J = 7.48 Hz, 2H), 8.44 (d, J = 7.63 Hz, 2H) 3.75 (br, 2H), 1.79 (br, 2H), 1.50–1.25 (m, 52H), 0.91 (s, 3H). Anal. Calculated for C₅₀H₆₁NO₂SSi₂: C, 75.04; H, 8.19; N, 1.75; S, 3.41; found: C, 75.08; H, 8.13; N, 1.66; S, 3.87.
Acknowledgements

We thank the University of Sheffield (studentship for L. C.) and EPSRC, Grant number EP/M506618/1, (studentship for N. J. R.) for support of this work.

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