

Synthesis, characterization and photovoltaics studies of 3-alkylthienyl thiophene based polymers prepared via direct arylation polymerization

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Abstract

The synthesis and characterization of two novel 3-thienyl based polythiophene derivatives PT and BT have been presented. These polymers have been synthesized by convenient and ecofriendly Pd catalyzed direct arylation polymerization method. In these polymers the alkyl side chain has been replaced by alkylthienyl side chain and polymer PT analogous to regioregular poly(3-hexylthiophene) (rr-P3HT) has been synthesized. These polymers have been characterized by ¹H NMR, GPC, TGA and UV vis absorption spectroscopy. The absorption maxima of these polymers are comparable to the rr-P3HT. The polymers have very good solubility in common organic solvents and therefore the solution processing of these materials has been easily done. Solar cells of these polymers have been fabricated by using PT and BT as donor materials and PC₆₁BM as acceptor material. It was observed that both the materials showed almost same photovoltaic characteristics. Solar cell performance of these materials has been found to be low and we are working on the optimization and improvement of power conversion efficiency of these materials. Copyright © 2017 VBRI Press.

Keywords: Direct arylation polymerization, P3HT, thienyl thiophene, solar cell.

Introduction

Over the past two decades, many classes of π -conjugated organic semiconducting polymers have been synthesized and characterized [1-3]. Till date regioregular (rr) poly(3-hexyl)thiophene (P3HT) have been proven to be one of the best material for various electronic devices like field effect transistor and organic solar cells etc [4-8]. There are different reports available in literature for the synthesis of rr-P3HT and are discussed here briefly [9-10]. Oxidative polymerization of thiophene using FeCl₃ is well known method but it does not give high degree of regioregular polymer [11]. McCullough and Lowe described the synthesis of rr-P3HT from 2-bromo-3-alkyl thiophene using Kumada coupling in presence of Ni catalyst [12-13]. Later on, Chen and Rieke synthesized rr-P3HT from 2, 5-dibromo-3-alkylthiophene using active zinc (Rieke Zn) and Ni catalyst [14]. McCullough also explained another method called Grignard metathesis reaction (GRIM) for the synthesis of rr-P3HT [15-17]. All these methods are very sensitive to moisture and oxygen. Therefore later on, with the process of direct arylation polymerization (DAP), the preparation of semiconducting polymers has become easy [18]. In rr-P3HT the alkyl side chain is completely

interdigitated in the polymer backbone which gives it a unique crystalline structure which is the key factor for its high stability, excellent charge mobility and high power conversion efficiency [19-24]. The device property of polythiophene (PT) can be easily tailored by altering the substitution pattern as this is considered one of the most powerful tools for modifying the properties of semiconducting molecules [25-27]. By incorporation of substituent at 3 and or 3, 4 positions many properties (band gap, solubility and solid state properties etc.) of PTs can be varied [28-30]. In our previous work we have synthesized and characterized a series of tetra and hexathiophenes with systematically varied alkyl thiophene side chain to study the effect of such substitution on conjugation and packing [31-33]. In the current work, we propose the synthesis and characterization of two novel polythiophene derivatives in which the substituent group has been modified. The alkyl side chain has been replaced by thienyl alkyl side chain and these monomers have been polymerized by DAP. The optical, thermal and photovoltaics study of these materials has been carried out.

Experimental

Material details

n-Butyllithium (n-BuLi) 1.6 M solution in hexane, anhydrous tetrahydrofuran (THF), palladium acetate (Pd(OAc)₂), cesium carbonate (Cs₂CO₃), dimethylformamide (DMF), dimethylacetamide (DMAc), pivalic acid, Ni(dppp)Cl₂ (dppp: 1,3-bis(diphenylphosphino)propane) were purchased from Alfa Aesar. All the solvents were purchased commercially, and used without further purification. All reactions were carried out in oven dried glassware under inert atmosphere.

Material synthesis

Fig. 1 illustrates the schematic for the preparation of monomers and polymers. Thiophene was monoalkylated by lithiation using n-BuLi followed by addition of 1-bromododecane which gave 2-dodecylthiophene in quantitative yield [34]. 2-dodecylthiophene was then brominated at 5- position by using 1 equivalent of N-bromosuccinimide (NBS). The Ni catalyzed Kumada coupling of 3-bromothiophene with **1** gave 3-(5-dodecylthienyl)thiophene, **2** as a white solid in 70% yield [35]. Dibromination of **2** was carried out under varied conditions like NBS/CHCl₃, NBS/acetic acid and NBS/DMF [36]. But the pure desired product was not isolated. Failure to get pure dibromo derivative of **2** led to the difficulty in polymerization by conventional methods

because this method of polymerization is extensively useful for the monomers which are difficult to functionalize. The Kumada coupling of 2-thienylmagnesium bromide with **3** in presence of catalytic amount of Ni gave 5''-dodecyl-[2,2':3',2''] terthiophene, **4** in 60% yield [42]. Monobromination of **4** by 1 equivalent of NBS gave monomer 5-bromo-5''-dodecyl-[2,2':3',2''] terthiophene, **5** in 90% yield [43]. All the intermediates were purified by column chromatography and characterized by ¹H NMR.

Synthesis of polymer PT

2-bromo-3-(5'-dodecyl-2'-thienyl) thiophene **3** (0.83 mmol, 343 mg), Pd(OAc)₂ (4 mol%, 23 mg), pivalic acid (0.83 mmol, 85 mg) and Cs₂CO₃ (2.5 mmol, 840 mg) were placed in a Schlenk tube with a magnetic stir bar. The reaction mixture was purged with N₂ for 15 minutes. To the reaction mixture THF (2.5 mL) was added and it was heated at 110°C in a preheated oil bath. After 3 hours the reaction mixture was cooled and poured into methanol and the precipitates were filtered. Soxhlet extractions with methanol and hexane were done to remove catalytic impurities and also small molecular weight polymers. The polymer after Soxhlet extraction was reprecipitated from CHCl₃/MeOH as red powder in 91% yield. Mn = 11,200 Mw/Mn = 1.45. ¹H NMR (400 MHz, CDCl₃): δ 2.82

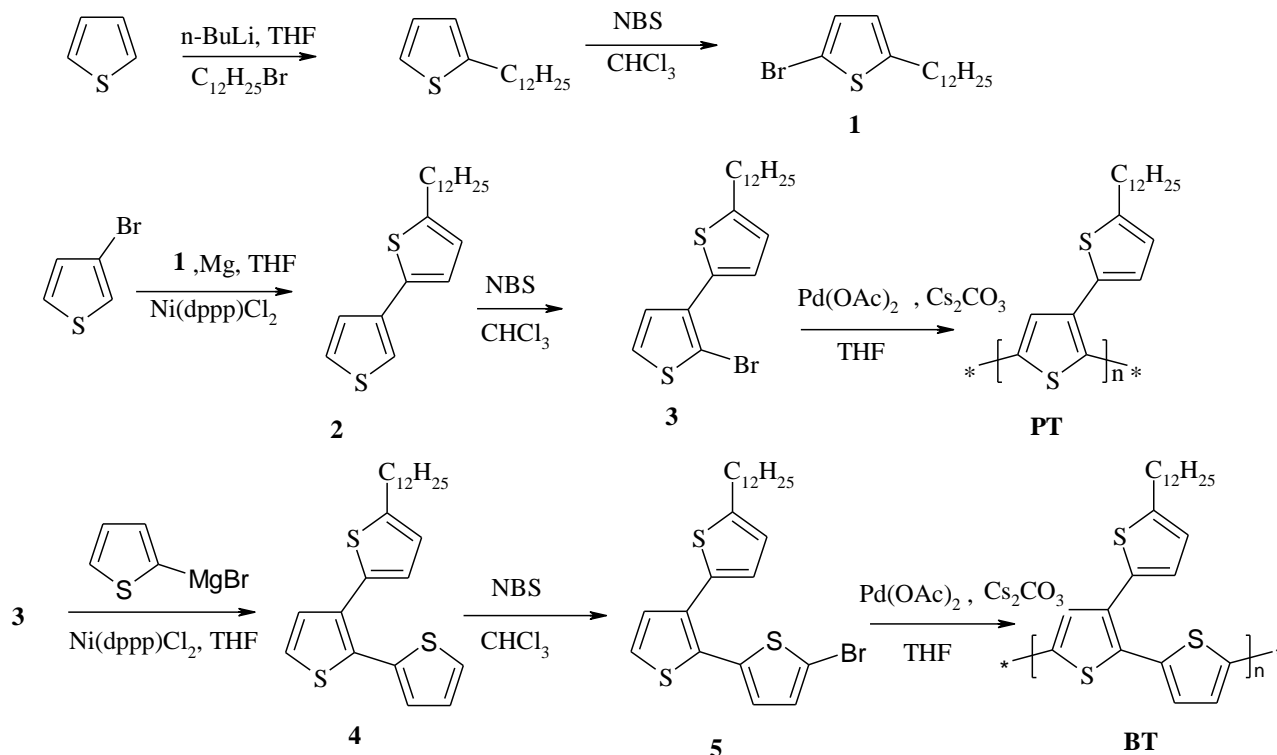


Fig. 1. Schematic for the preparation of monomer and polymer PT and BT.

like Suzuki or Stille polycondensation etc [37-38] because one of the mandatory requirement of these reactions is bifunctional monomer. **2** can be regio specifically brominated at C-2 position to give 2-bromo-3-(5'-dodecyl-2'-thienyl)thiophene, **3** which was then polymerized via DAP using Pd(OAc)₂ as catalyst [39-41]

(br, 2H), δ 1.7-0.8 (m, 23H), δ 6.7-7.2 (br, 3H).

Synthesis of polymer BT

5-bromo-5''-dodecyl-[2,2':3',2''] terthiophene **5** (0.698 mmol, 346 mg), Pd(OAc)₂ (4 mol%, 19 mg), pivalic acid (0.698 mmol, 70 mg) and Cs₂CO₃ (2.1 mmol, 700 mg)

were placed in a Schlenk tube with a magnetic stir bar. The reaction mixture was purged with N_2 for 15 minutes. To the reaction mixture THF (2.1 mL) was added and it was heated at $110^\circ C$ in a preheated oil bath. After 3 hours the reaction mixture was cooled and poured into methanol and the precipitates were filtered. Soxhlet extractions with methanol and hexane were done to remove catalytic impurities and also small molecular weight polymers. The polymer after Soxhlet extraction was reprecipitated from $CHCl_3/MeOH$ as red powder in 92 % yield. $M_n = 10,415$ $M_w/M_n = 3.3$. 1H NMR (400 MHz, $CDCl_3$): δ 2.82 (br, 2H), δ 1.7-0.8 (m, 23H), δ 6.7-7.2 (br, 5H).

Characterization and device fabrication

The 1H NMR spectra were recorded using JEOL 400 MHz NMR spectrometer using tetramethylsilane as internal standard and deuterated chloroform as solvent. The number average and weight average molecular weight of the polymer were determined by gel permeation chromatography (GPC) with polystyrene as the standard and THF as the solvent. Thermogravimetric analysis was carried out by Perkin Elmer Instrument. The UV Vis spectra were recorded on a T90+ UV/Vis Spectrometer (PG instruments Ltd). The bulkheterojunction (BHJ) solar cells using **PT** and **BT** polymers were fabricated on indium tin oxide (ITO) coated glass substrates with the following configuration ITO/PEDOT:PSS/active layer/Aluminum. Active layer of the fabricated devices was an heterojunction of **PT:PC₆₁BM** and **BT:PC₆₁BM** designated here as solar device SD1 (solar device) and SD2 respectively. ITO (10 Ω /sq) coated glass substrates were chemically etched in a predefined geometry using a standard wet chemical etching process. **Fig.2** shows ITO coated glass substrates (left) and substrate with the desired pattern (right) after wet chemical etching. The substrates were then cleaned with soap solution in ultrasonic bath followed by 15 min ultrasonication in deionized water. The substrates were then boiled in acetone (10 min), isopropanol (10 min) and trichloroethylene (10 min) followed by an oxygen plasma treatment (7 min).

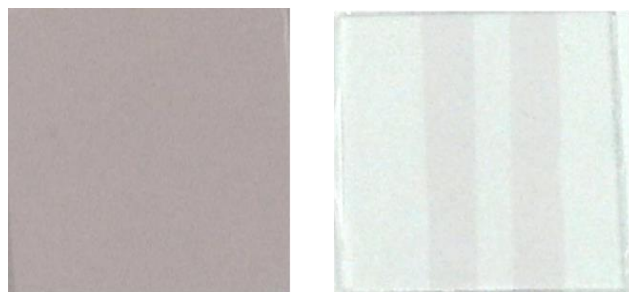


Fig. 2. ITO coated glass substrate (left) and substrate etched in desired geometry (right).

The photovoltaic devices were prepared by spin coating poly(3,4-ethylenedioxythiophene)-poly(styrene sulfonate) (PEDOT-PSS) as hole transport layer at a spin speed of 2000 rpm for one minute to get a thickness of ~ 50 nm. These were then annealed in vacuum oven at a temperature of $120^\circ C$ for 20 min. A solution of polymers (**PT** and **BT**) with **PC₆₁BM** having 1:1 blend ratio were prepared in o-

dichlorobenzene having a total concentration of 25 mg/mL followed by ultrasonication for 2h and 5h respectively. The active layers of **PT:PC₆₁BM** and **BT:PC₆₁BM** were spin coated at 800 rpm for one minute to maintain a film thickness of ~ 100 nm and ~ 150 nm respectively. These were then annealed in vacuum oven at a temperature of $100^\circ C$ for 30 min. The aluminum electrodes (~ 100 nm) were finally thermally evaporated in a vacuum of 10^{-6} torr. Solar simulator, SS50AAA, with 100 mW/cm^2 (AM1.5 condition) intensity and Keithley-2400 source meter were used for taking J-V measurements. The devices were fabricated and characterized in ambient conditions without encapsulation. **Fig. 3** shows the schematic diagram of the solar cell along with the digital photograph of the fabricated devices.

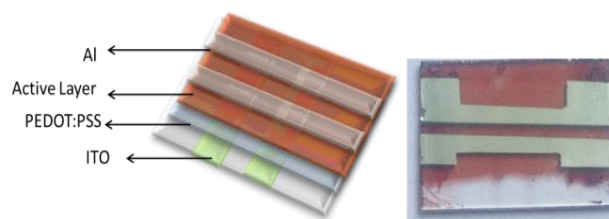


Fig. 3. Schematic diagram(left) and digital photograph of the fabricated devices (right).

Results and discussion

The polymers **PT** and **BT** were polymerized using Pd catalyzed DAP which is an economical and alternative route for conventional polymerization methods [44-45] As this method do not require any special conditions for polymerization and could be easily carried out on substrates which are difficult to functionalize. The reaction conditions were optimized in terms of catalytic system and solvent. The polymerization was carried out by using Hermann's catalyst {trans-bis-(acetato)bis[o-(di-tolylphosphino)benzyl]dipalladium(II)} and $Pd(OAc)_2$. It was found that polymerization reactions done with $Pd(OAc)_2$ as catalyst gave high molecular weight polymers while on the other hand with Hermann's catalyst only low molecular weight polymers were obtained [18]. The polymerization was done in various solvents like DMF, THF and mixture of THF and toluene. The best results in terms of solubility of the polymer were obtained by using THF as solvent. The polymers were characterized by UV-Vis absorption, 1H NMR, Gel permeation chromatography and thermo gravimetric analysis.

Fig. 4 shows the absorption spectra of polymer in dilute $CHCl_3$ solution and in thin films. The polymer displays broad absorption spectra due to strong $\pi-\pi^*$ transition with absorption maxima is at 460 and 458 nm for **PT** and **BT** polymers respectively in $CHCl_3$ solution. The polymer exhibits absorption maxima at 526 and 485 nm for **PT** and **BT** polymers respectively in thin film and it is significantly red shifted than λ_{max} in solution. The bathochromic shift of 66 nm in **PT** is due to high degree of ordering of polymer in solid state [5, 16, 36] as high degree of crystallinity is observed from uniformly 3-substituted thiophenes [28]. These values closely resemble to that of the regioregular

P3HT [28, 46-48]. It also explains that by replacement of alkyl chain by thienyl alkyl side chains no increase in steric strain along the polymer backbone occur but instead it is providing increased conjugation in side chain along with main chain.

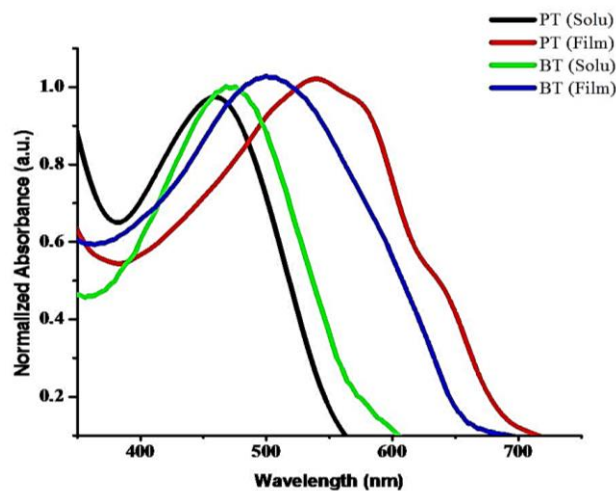


Fig. 4. Absorption spectra of polymer (PT and BT) in solution and film.

Table 1. Molecular Weight and Optical properties of polymer.

No	GPC			Absorption (nm)		T_d (°C)
	Mn (Dalton)	PDI	DP	Solution CHCl_3	Film	
PT	11,220	1.4	~37	460	526	420
BT	10,415	3.3	~31	458	485	400

Table 2. Device characteristics of SD1 and SD2.

Device Type	J_{sc} (mA/cm ²)	V_{oc} (V)	FF (%)	PCE (%)
SD1 (PT)	1.62	0.32	26.24	0.14
SD2 (BT)	1.26	0.37	25.26	0.10

The absorption maxima values of these materials have been summarized in Table 1. The blue shift of absorption maxima in BT than in PT may be due to cross couplings in the 3' and 4' position of the bithiophene monomer [49]. Thermo gravimetric analysis (TGA) was carried out to determine the degradation behavior of the polymer. The temperature (T_d) for 5% weight loss is 420 °C. ¹H NMR showed three signals in the aromatic region. Absence of any extra peak in the aromatic region suggests that there is no appreciable random coupling along the polymer backbone [13].

Bulk heterojunction (BHJ) organic solar cells of polymers PT and BT as donor materials and PC₆₁BM as acceptor material were fabricated in the ambient conditions. Fig. 5 and 6 shows the typical dark and illuminated current-density voltage (J-V) characteristics of ITO/PEDOT:PSS/PT:PC₆₁BM/Al (SD1) and ITO/PEDOT:PSS/PT:PC₆₁BM/Al (SD2) on a semi-log scale respectively. The curves show typical diode

characteristics in the dark. The photovoltaic parameters extracted from the illuminated J-V characteristics for SD1 were: short circuit current density (J_{sc}) of 1.62 mA/cm², open circuit voltage (V_{oc}) of 0.32 V, fill factor (FF) of 26.24 % and power conversion efficiency (PCE) of 0.14% and for SD2 were 1.26 mA/cm², 0.37 V, 25.26 % and 0.10%.

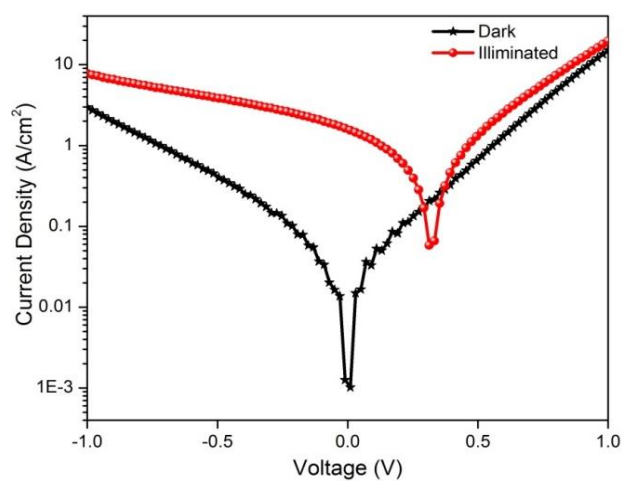


Fig. 5. Dark and illuminated current voltage (J-V) characteristics of prepared organic solar cell (SD1).

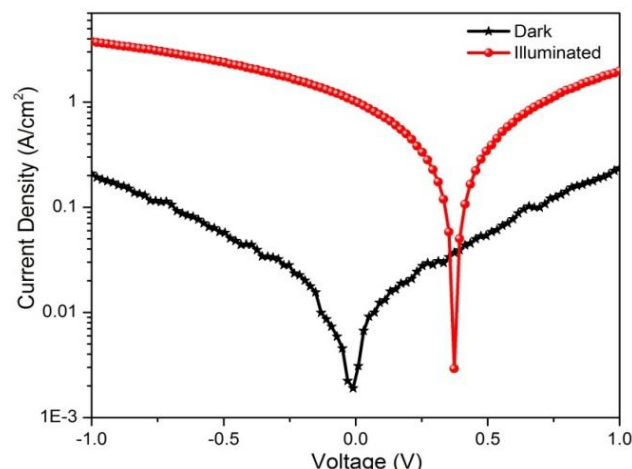


Fig. 6. Dark and illuminated current voltage (J-V) characteristics of prepared organic solar cell (SD2).

These photovoltaic parameters have been summarized in Table 2. It was observed that the power conversion efficiency of devices based on PT as donor material (SD1) is slightly greater than the devices based on BT as donor material (SD2). We are working on the further optimization for improvement in the efficiency of device.

Conclusion

A new class of solution processable 3-thienyl substituted polythiophene derivatives has been synthesized by direct arylation polymerization method. The materials have been characterized by various spectroscopic techniques. The substituent group at the 3-position of thiophene has been modified and it was observed that incorporation of the substituent is not providing steric hindrance to the polymer backbone as revealed by UV-Vis spectroscopy. The

absorption maximum of **PT** is comparable to P3HT. We have identified a new polymer whose optical properties are almost same as of rr P3HT. Solar cells were fabricated using these materials as donor with PC61BM as acceptor. The power conversion efficiency of 0.14 and 0.10 was achieved for **PT** and **BT** respectively, which is quite low as comparable to P3HT. The optimization of the solar cell performance of these materials is currently underway. Also, as the future perspective of this work, we will synthesize P3HT by DAP by keeping identical conditions for polymerization. This will provide us a good comparison of these materials with respect to P3HT as donor molecule in photovoltaics.

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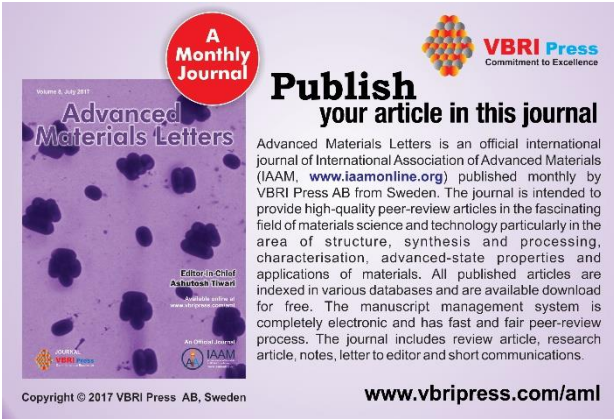
Author's contributions

The authors contributed equally to this work. All authors read and approved the final manuscript. Authors have no competing financial interests.

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