

Synthesis and characterization of conjugated polymers containing phenyl and bithiophene: controlling of optical properties with molarity

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Abstract Conjugated polymers containing phenyl and bithiophene units are synthesized by conventional polycondensation and crossing-coupling method of carboxylic acids with halides. The structure of both polymers formed by adding the ester group to the first polymer was confirmed by means of Fourier transform infrared (FTIR), nuclear magnetic resonance techniques, differential scanning calorimetry, and thermogravimetry and elemental analysis. The molecular weight distribution values of polymers were determined with a gel permeation chromatography (GPC) system. The optical properties of these different polymers with synthesis methods and structures were investigated for different molarities. The molar extinction coefficient values of the polymers decrease with increasing molarity. Transmittance (T) values of the polymers are increased with ester group addition and decreasing molarity. We obtained two types (direct and indirect allowed band gaps) of band gaps. The direct allowed optical band gap (E_{gd}) values of poly(2,2' bithiophene terephthalate) (polyBTTP) are lower than the indirect allowed optical band gap (E_{gid}) values of poly(5-phenyl-2,2' bithiophene) (polyPBT). The direct and indirect allowed band gap values of the polymers can be decreased with ester group addition and increasing molarity. The refractive index of the polymers can be decreased with decreasing molarity, increasing wavelength, and ester group addition.

Keywords Conjugated polymer · Bithiophene · Optical properties · Refractive index · Molarity

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Introduction

Conjugated polymers can be used for large-scale applications due to improved processability. At the same time, they draw attention with better electrical and physical properties. These polymers with a new structural design have brought innovation to some areas, for example, organic light-emitting diodes, organic transistors, organic solar cells, and electrochromic devices [1–6].

From the most notable of conjugated polymers, polythiophenes (PTs) have attracted much attention due to their good film-forming and optical properties [7–9]. Polythiophenes are located in many studies because of easy modification of the structure, excellent processabilities, controllability of the electrochemical behavior of doped and undoped states, high environmental resistance, and the rich chemistry, which thus provides the control of polymer properties [10]. Current important application areas of polythiophene and its derivatives include electrical conductors, optoelectronic devices, sensors, antistatic coatings, supercapacitors, transparent electrodes, electrochromic displays, light-emitting diodes, photovoltaic cells, batteries, and solar cells [11].

Optical constants include the valuable information for technological applications [12]. The determination of the optical constants of polymers such as refractive index and extinction coefficient is also important for optical applications. Furthermore, the refractive index and optical band gap are the fundamental parameters of an optical material, because these are closely related to the electronic properties of the material [13].

In this study, two different conjugate polymers were synthesized by esterification reaction of polycondensation and crossing-coupling method between 5, 5'-dibromo-2, 2'-bithiophene and terephthalic acid. Unlike the Suzuki

polymerization procedure, we used carboxylic acid instead of boronic acid and poly(5-phenyl-2,2' bithiophene) (polyPBT) with high molecular weight, and high yield was obtained. So, the polymerization of monomers was accomplished with bromine and carboxylic acid ended. Nuclear magnetic resonance (^1H -NMR, ^{13}C -NMR) and Fourier transform infrared (FTIR) spectroscopies, DSC, and TGA techniques were utilized for the characterization of the polymers. The analyses of both polymers were presented comparatively. The optical and thermal properties of the polymers formed by adding the ester group to the first polymer were compared. These different polymers with synthesis methods and structures showed significant differences in the thermal and optical properties. The optical properties of these polymers were also investigated for different molarities.

Experimental

Materials

5, 5'-Dibromo-2, 2'-bithiophene (Aldrich) and terephthalic acid (Aldrich) were used as received in the synthesis of polymers. Toluene (Merck), Na_2CO_3 (Merck) and dimethylformamide (Merck), chloroform (Merck), and methanol were utilized in polymerization. At the same time, tetrakis(triphenylphosphine)palladium(0) ($\text{Pd}(\text{PPh}_3)_4$) (Aldrich) was used without purification in the copolymerization as catalyst.

Physical measurements

Fourier transform infrared analyses of the polymers were performed with a Perkin Elmer Precisely Spectrum One spectrometer for the detection of functional groups. Samples were prepared as KBr pellets. A Bruker GmbH DPX-400-MHz FT spectrometer was used to obtain the NMR spectra of the polymers. Molecular weights of the polymers were determined with Agilent 1100 gel permeation chromatography. The eluting solvent was DMF and the flow rate was 0.7 mL min^{-1} . Calibration was achieved against polystyrene standard. Thermogravimetric analyses (TGA) of polymers were recorded under a nitrogen flow with a Perkin Elmer Pyris 1 at a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$. The melting point and glass transition temperature of the polymers synthesized were examined by means of a differential scanning calorimeter (DSC) named as Perkin Elmer Diamond. The differential scanning calorimetry experiments were carried out under N_2 atmosphere, between room temperature and $400 \text{ }^\circ\text{C}$, and the heating rate was $10 \text{ }^\circ\text{C min}^{-1}$. Optical measurements of polymers were carried out with a Shimadzu model UV-1800 spectrophotometer in the wavelength 1100–190 nm at room temperature.

Synthesis of the poly(5-phenyl-2,2' bithiophene)

The polymer was synthesized via formation cross-coupling reactions from palladium catalyzed by an carboxylic acid and an halide (I, Br, or Cl) in the presence of a base as an alternative to the traditional Suzuki conditions [14]. Monomers 5, 5'-dibromo-2, 2'-bithiophene (33.23 mg, 0.2 mmol) and terephthalic acid (64.82 mg, 0.2 mmol) were added to a 25-mL dry flask. The flask was purged with argon three times. To this flask, $\text{Pd}(\text{PPh}_3)_4$ (5.0 mg, 0.004 mmol) was added along with anhydrous/degassed toluene (12 mL mmol^{-1} of monomer). Reflux reaction took place for at least 36 h. Polymer solution is precipitated into methanol, redissolved in chloroform, and precipitated into methanol again. The polymer is dried under vacuum.

(Yield 70 %). Color: dark brown. Elem. anal. For $\text{C}_{14}\text{H}_8\text{S}_2$: C, 70; H, 3; S, 27 Found: C, 71; H, 4; S, 25. FTIR data (KBr, cm^{-1}): 3065–3023 (Ar–CH), 1625–1586 ν (Ar–C=C) 973, 793, 823 ν (C–S–C). ^1H NMR (400 MHz, DMSO-d_6): δ (ppm), 7.278 (4H, aromatic protons), 6987–6974/6875–6862 (4H, thiophene ring protons), ^{13}C NMR (400 MHz, DMSO-d_6): δ (ppm), 118.69, 119.50, 119.82, 120.14 (thiophene), 130.17, 123.88, 123.56, 123.24, 122.97, 122.65, 122.33 (C on aromatic ring), $M_n=170,690 \text{ g mol}^{-1}$ (polydispersity index, PDI, 1.14).

Synthesis of poly(2,2' bithiophene terephthalate)

Poly(2,2' bithiophene terephthalate) (polyBTTP) was synthesized via esterification reaction of 5, 5'-dibromo-2, 2'-bithiophene (33.23 mg, 0.2 mmol) and terephthalic acid (64.82 mg, 0.2 mmol) in the presence of triethylamine at $90 \text{ }^\circ\text{C}$. The reaction was carried out for 24 h with constant stirring. Then, methanol was added for precipitation and a brown solid was obtained by filtration. It was washed with methanol. The polymer is dried under vacuum.

(Yield 79 %). Color: brown. Elem. anal. For $\text{C}_{15}\text{H}_8\text{S}_2\text{O}_2$: C, 63; H, 3; S, 23 O, 11 Found: C, 62; H, 4; S, 24, O, 10. FTIR data (KBr, cm^{-1}): 3065–3023 (Ar–CH), 1625–1586 ν (Ar–C=C) 973, 793, 823 ν (C–S–C). ^1H NMR (400 MHz, DMSO-d_6): δ (ppm), 7.978–7.048 (4H, aromatic protons), 6495–6482/6306–6294 (4H, thiophene ring protons), ^{13}C -NMR (400 MHz, DMSO-d_6): δ (ppm), 140.68 (C=O), 123.95, 124.45, 124.77, 125.09 (C on thiophene ring), 130.56, 128.93, 128.50, 128.20, 127.92, 127.60, 127.28 (C on aromatic ring), $M_n=5.276 \text{ g mol}^{-1}$ (PDI 1.13).

Results and discussion

Synthesis and chemical characterization

The general synthetic routes toward polymers are outlined in Fig. 1. The first polymer was performed using dibromide and

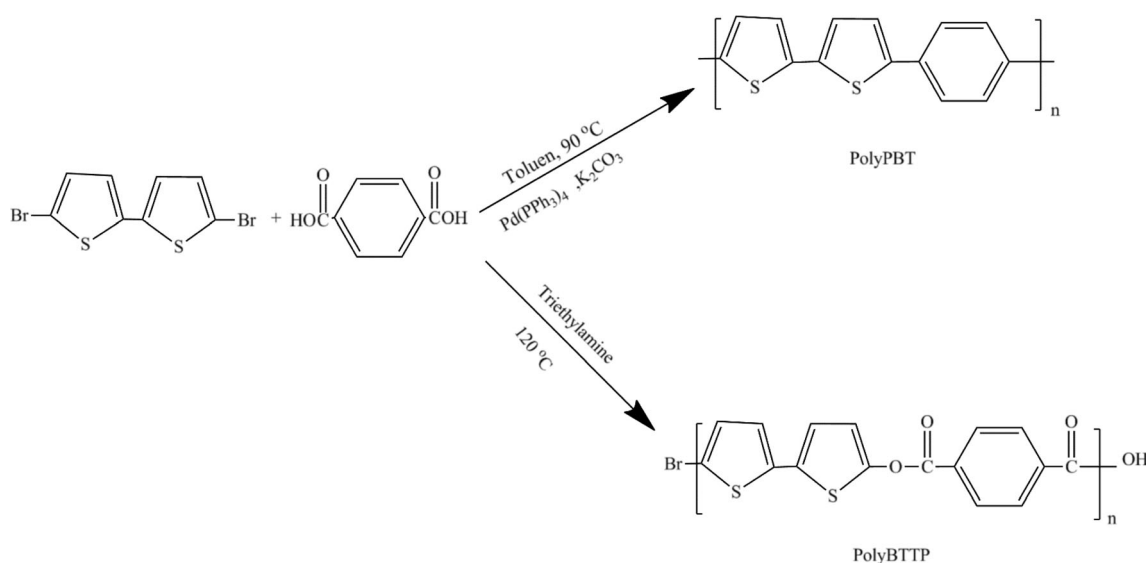


Fig. 1 The synthesis of polymers

dicarboxylic acid. Unlike the Suzuki polymerization procedure, we have accomplished polymerization of monomers with bromine and carboxylic acid ended. The second polymer (polyBTTP) was synthesized by the same reaction without catalyst by esterification reaction of polycondensation. Both polymers showed good solubility in common organic solvent. The number average molecular weights of polymers were determined by GPC using a polystyrene standard and DMF as an eluent. The number average molecular weights (M_n) of polyPBT and polyBTTP were determined to be 170.690 and 5.276 g mol⁻¹ with polydispersity indexes (M_w/M_n) of 1.14 and 1.13, respectively. The molecular weight of polyPBT was higher. All the major cross-coupling reactions have been used in polymerizations which generally gives the highest yields and molecular weights [15–17]. The polymers obtained by the general polycondensation method were accompanied by the formation of a low molecular weight by-product [18].

FTIR spectra of both synthesized polymers showed similar absorptions which were related to aromatic C–H, aromatic C=C, and C–S in thiophene ring stretchings (Fig. 2). The presence of a band at 1744 cm⁻¹ was a characteristic of stretching vibration of

the carbonyl group of esters of polyBTTP. The aromatic C=C stretching appears at about 1590 and 1490 cm⁻¹. At around 622 cm⁻¹, the C–S–C peak is overlapped by C–H and C–C out of plane vibrations of the aromatic ring. The medium absorption peak at 1244 cm⁻¹ is attributed to the O–C–O stretch of the ester group. The broad band observed at approximately 1648 cm⁻¹ proves the presence of polyconjugation [19]. The ¹H-NMR spectra of polymers have been carried out in DMSO-*d*₆ at room temperature. The ¹H-NMR spectrum of polymers showed the signals at 7–7.98 ppm corresponding to aromatic CH protons of the phenyl ring and at 6.98–6.86 ppm corresponding to CH protons of the thiophene ring.

Thermal stability of the polymers was investigated by TGA with a heating rate of 10 °C min⁻¹ and a nitrogen flow rate of 20 mL min⁻¹, and the TGA data of the polymers are listed in Table 1. As a result of TGA analysis (Fig. 3), polyPBT exhibited higher thermal stability compared with polyBTTP that may be due to the rigid molecular structure and the high molecular weight of polyPBT [18]. The thermal stabilities of both polymers are considerably different: the residues at 900 °C (%) of polyPBT and polyBTTP are about 56 and 13 %, respectively; their degradation temperatures of 5 % weight loss

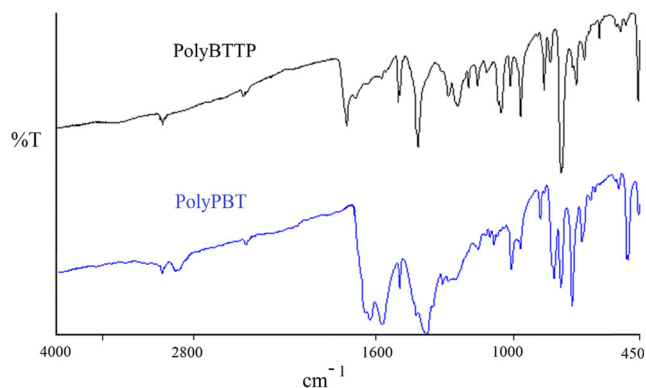


Fig. 2 The FTIR spectra of polyPBT and polyBTTP

Table 1 The TGA data of polyPBT and polyBTTP

	T_i^a	T_{50}^b	% weight loss at 500 °C	% weight loss at 600 °C	% weight loss at 700 °C	% weight loss at 800 °C	% remaining at 900 °C
polyPBT	118	853	9	24	37	45	56
polyBTTP	116	280	63	69	73	81	13

^a Initial decomposition temperature

^b Decomposition temperature at 50 % weight loss

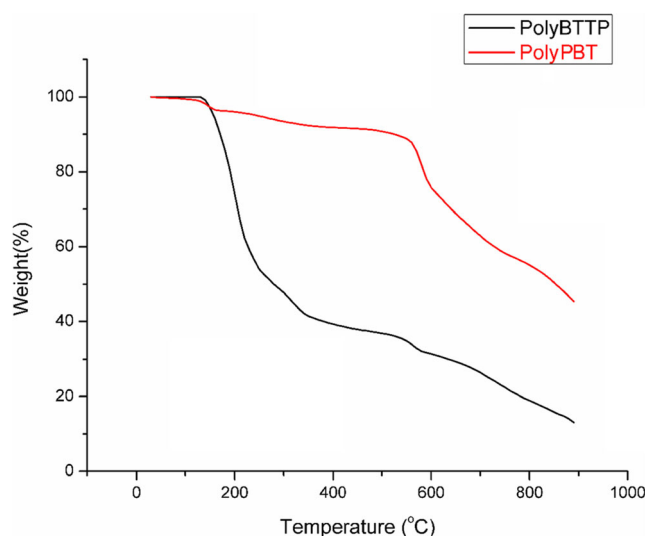


Fig. 3 The TGA curves of polyPBT and polyBTTP

are 247 and 158 °C, respectively; and the 50 % weight loss temperatures are 853 and 280 °C subsequently again.

DSC curves of polymers were recorded on Perkin Elmer Diamond (Fig. 4). The heating rate was maintained at 20 °C min⁻¹. The polyPBT showed only a melting temperature at 137 °C, but polyBTTP showed a glass transition temperature and two melting temperatures (T_c). The T_c values for polyBTTP were recorded as 136 and 130 °C. Its glass transition temperature is 108 °C, too. It is seen that the polymer gained flexibility by addition to the main chain of the ester functional group. While polyPBT has a more rigid structure, it was showing only the melting temperature due to polyBTTP having a C–O bond with high elasticity which showed the glass transition temperature [14, 20–23].

The optical properties

Absorbance (A) of a chemical substance can be obtained as follows [24–26]:

$$A = \log_{10} \left| \frac{I_0}{I} \right| = -\log_{10} T = \varepsilon CL \quad (1)$$

where I_0 and I are the intensity of incident and leaving monochromatic light on the substance of interest, respectively. Also, L is the optical path length of light through the cuvette, C is an accurate molar concentration of the absorbing molecule, and ε is a molar extinction coefficient or wavelength-dependent molar absorptivity of the absorbing molecule [25–27]. The ε values of the polyPBT and polyBTTP were obtained from Eq. (1) and given in Table 2. As seen in Fig. 5a and Table 2, the highest ε values (1.066×10^{13} and 0.704×10^{13} L mol⁻¹ cm⁻¹, respectively) of the polyPBT are obtained at 328 nm and for 0.28 and 0.56 pM, but these highest ε values of the polyPBT are not observed for 1.13, 2.25, and 4.5 pM due to high molarity. On the other hand,

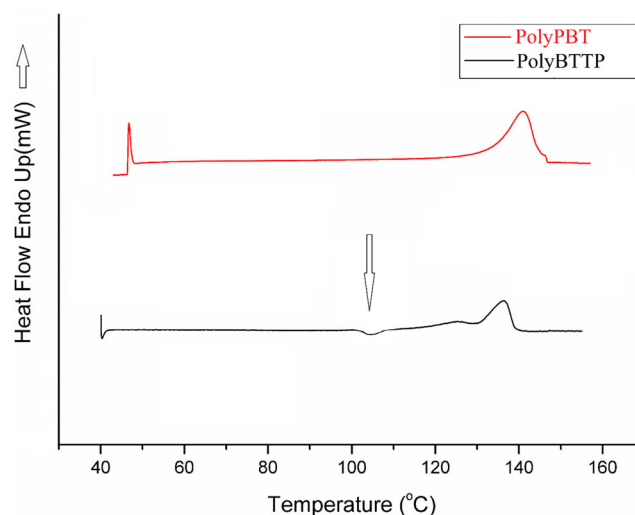


Fig. 4 The DSC curves of polymers

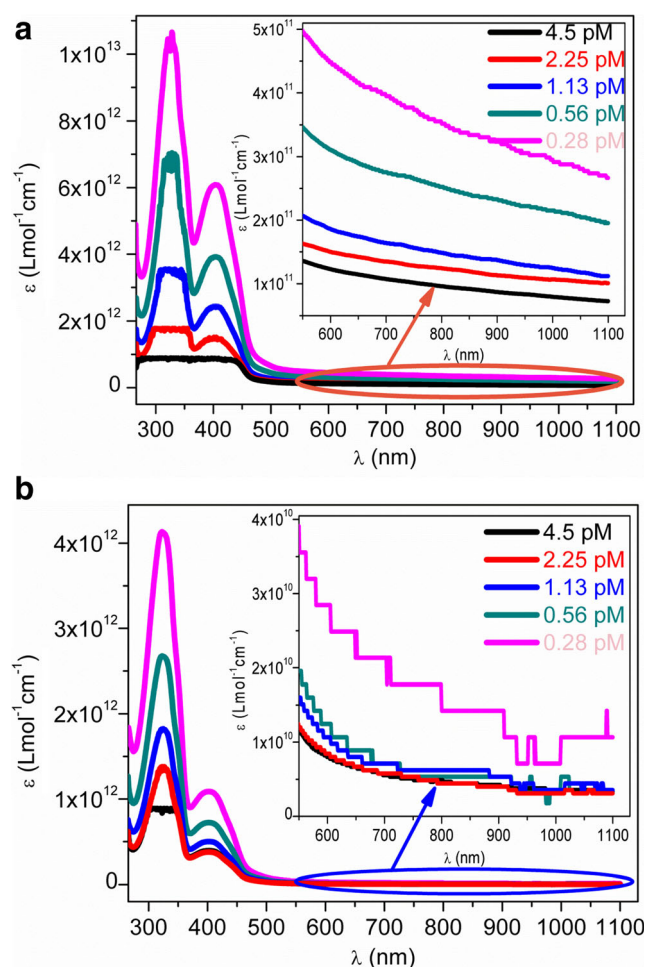
another peak of polyPBT is observed at 403 nm and for 0.28, 0.56, 1.13, and 2.25 pM, and these ε values of polyPBT for 0.28, 0.56, 1.13, and 2.25 pM are found to be 6.087×10^{12} , 3.920×10^{12} , 2.434×10^{12} , and 1.461×10^{12} L mol⁻¹ cm⁻¹, respectively. As seen in the inset of Fig. 5a, the ε of polyPBT at higher molarity decreases also with increasing molarity. As seen in Fig. 5b and Table 2, the highest ε values (4.135×10^{12} , 2.676×10^{12} , 1.807×10^{12} , and 1.3794×10^{12} L mol⁻¹ cm⁻¹, respectively) of polyBTTP are obtained at 323 nm and for 0.28, 0.56, 1.13, and 2.25 pM, but these highest ε values of polyBTTP are not observed for 4.5 pM due to high molarity. Similarly, another peak of polyBTTP is observed at 403 nm and for 0.28, 0.56, 1.13, 2.25, and 4.5 pM, and these ε values of polyBTTP for 0.28, 0.56, 1.13, 2.25, and 4.5 pM are found to be 10.880×10^{11} , 7.236×10^{11} , 5.013×10^{11} , 3.793×10^{11} , and 3.798×10^{11} L mol⁻¹ cm⁻¹, respectively. As seen in the inset of Fig. 5b, the ε values of polyBTTP at higher molarity are similar. The ε values of polyPBT and polyBTTP are higher than the ε values of the *N,N'*-dipentyl-3,4,9,10-perylenedicarboximide (PTCDI-C5) small molecule [24], poly[2-methoxy-5-(3',7'-dimethyloctyloxy)-1,4-phenylenevinylene] (MDMO-PPV) light-emitting polymer [28], 5,5'-di(4-biphenyl)-2,2'-bithiophene (PPTTP) nanofiber [29], and tris[4-(5-dicyanomethylidenemethyl-2-thienyl)phenyl]amine (TDCV-TPA) [27] in the literature. These results suggest that the ε and ε peak values of polyPBT and polyBTTP decrease with increasing molarity. As seen in Fig. 5a, b and Table 2, the ε and ε peak values of polyPBT are higher than those of polyBTTP. The obtained results show that the ε and ε peak values of polyPBT are decreased in polyBTTP with ester group addition and the highest ε peak wavelength (328 nm) of polyPBT can be decreased as 323 nm.

The transmittance (T) spectra of polyPBT and polyBTTP were conducted to study their optical properties for 0.28, 0.56, 1.13, 2.25, and 4.5 pM. Figure 6 shows the plot of the T vs. λ . As seen in Fig. 6, there are two small peaks of polyPBT and polyBTTP in the near-ultraviolet (NUV) region. After the

Table 2 The optical parameters of polyPBT and polyBTTP for different molarities

Molarity (pM)	polyPBT			polyBTTP		
	ε (L mol ⁻¹ cm ⁻¹) $\times 10^{12}$ at 328 nm	ε (L mol ⁻¹ cm ⁻¹) $\times 10^{12}$ at 403 nm	E_{gd} (eV)	ε (L mol ⁻¹ cm ⁻¹) $\times 10^{12}$ at 323 nm	ε (L mol ⁻¹ cm ⁻¹) $\times 10^{12}$ at 403 nm	E_{gid} (eV)
0.28	10.66	6.087	2.678	4.135	1.088	2.305
0.56	7.044	3.920	2.651	2.676	0.724	2.245
1.13	—	2.434	2.623	1.807	0.501	2.173
2.25	—	1.461	2.584	1.379	0.377	2.155
4.5	—	—	2.519	—	0.394	2.107

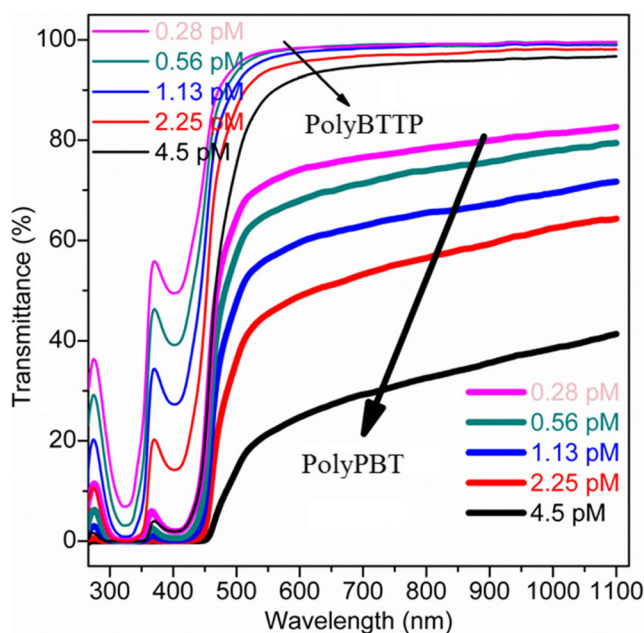
NUV region, the T values increase sharply with increasing wavelength until a certain wavelength; then, the T values are almost constant. As seen in Fig. 6, the T values of polyPBT and polyBTTP decrease with increasing molarity and the T values of polyPBT are lower than those of polyBTTP. These results suggest that the T values of polyBTTP increased with ester group addition and decreasing molarity. Also, it is observed that the T values of polyBTTP in the NUV region are close to the T values in the visible (V) region with decreasing molarity.

**Fig. 5** The curves of the molar extinction coefficient (ε) vs. wavelength (λ) of **a** polyPBT and **b** polyBTTP for different molarities

The optical band gap (E_g) of polyPBT and polyBTTP can be obtained from the absorption spectrum using the Tauc relation [27, 30].

$$(\alpha h\nu) = A(h\nu - E_g)^n \quad (2)$$

where A is a constant and $h\nu$ and E_g are the photon energy and the optical band gap of the material, respectively. To calculate the E_g values of polyPBT for 0.28, 0.56, 1.13, 2.25, and 4.5 pM, the $(\alpha h\nu)^2$ plot vs. E of polyPBT is suitable and shown in Fig. 7a. The direct allowed band gap (E_{gd}) values of polyPBT for 0.28, 0.56, 1.13, 2.25, and 4.5 pM were calculated from the curves of $(\alpha h\nu)^2$ vs. E and are given in Table 2. As seen in Table 2, the E_{gd} value of polyPBT decreases with increasing molarity. The E_{gd} value (2.519 eV) of polyPBT for 4.5 pM is the lowest value, while the E_{gd} value (2.673 eV) of the polyPBT for 0.28 pM is the highest. The E_{gd} values of the polyPBT are higher than the E_{gd} values of the PTCDI-C5 small molecule [23], MDMO-PPV light-emitting polymer [28], PPTTPP nanofiber [29], and TDCV-TPA semiconductor [26] in the literature. To calculate

**Fig. 6** The plot of the transmittance vs. λ of polyPBT and polyBTTP for different molarities

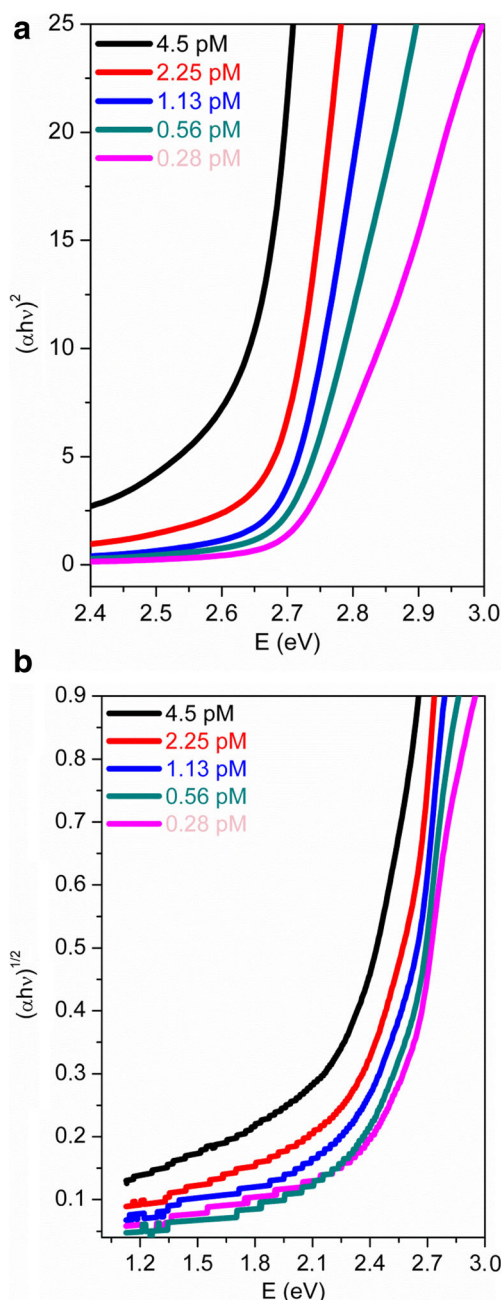


Fig. 7 The **a** $(\alpha h\nu)^2$ plot vs. the photon energy (E) of polyPBT and the **b** $(\alpha h\nu)^{1/2}$ plot vs. E of polyBTTP for different molarities

the E_g values of polyBTDMB for 0.28, 0.56, 1.13, 2.25, and 4.5 pM, the $(\alpha h\nu)^{1/2}$ plot vs. E of polyBTDMB is suitable and shown in Fig. 7b. The indirect allowed band gap (E_{gid}) values of polyBTDMB for 0.28, 0.56, 1.13, 2.25, and 4.5 pM were calculated from the curves of $(\alpha h\nu)^{1/2}$ vs. E and are given in Table 2. As seen in Table 2, the E_{gid} value of polyBTTP decreases with increasing molarity. The E_{gid} value (2.305 eV) of polyBTTP for 0.28 pM is the highest value, while the E_{gid} value (2.107 eV) of polyBTTP for 4.5 pM is the highest. The E_{gid} values of polyBTTP are close to the PTCDI-C5 small molecule [24] in the literature. We obtained two types (direct and indirect allowed

band gaps) of band gaps: The direct allowed band gap (E_{gd}) is suitable for polyPBT, while the indirect allowed band gap (E_{gid}) is suitable for polyBTTP. This situation results from the electronic band transition of polyBTTP. As seen in Table 2, the optical band gap (E_{gd}) values of polyBTTP are lower than the optical band gap (E_{gid}) values of polyPBT. It is observed that the direct and indirect allowed band gap values of polyBTTP can be decreased with ester group addition and increasing molarity.

The refractive index (n) is an important parameter for optoelectronic technologies. It is significant to determine optical parameters of polyPBT and polyBTTP. The optical properties of a material can be defined by the refractive index. The n is obtained from the following equation [31–33]:

$$n = \left\{ \left[\frac{4R}{(R-1)^2} - k^2 \right]^{1/2} - \frac{R+1}{R-1} \right\} \quad (3)$$

The n values of polyPBT and polyBTTP for 0.28, 0.56, 1.13, 2.25, and 4.5 pM were obtained from Eq. (3). Figure 8a shows the plot of n vs. λ of polyPBT for 0.28,

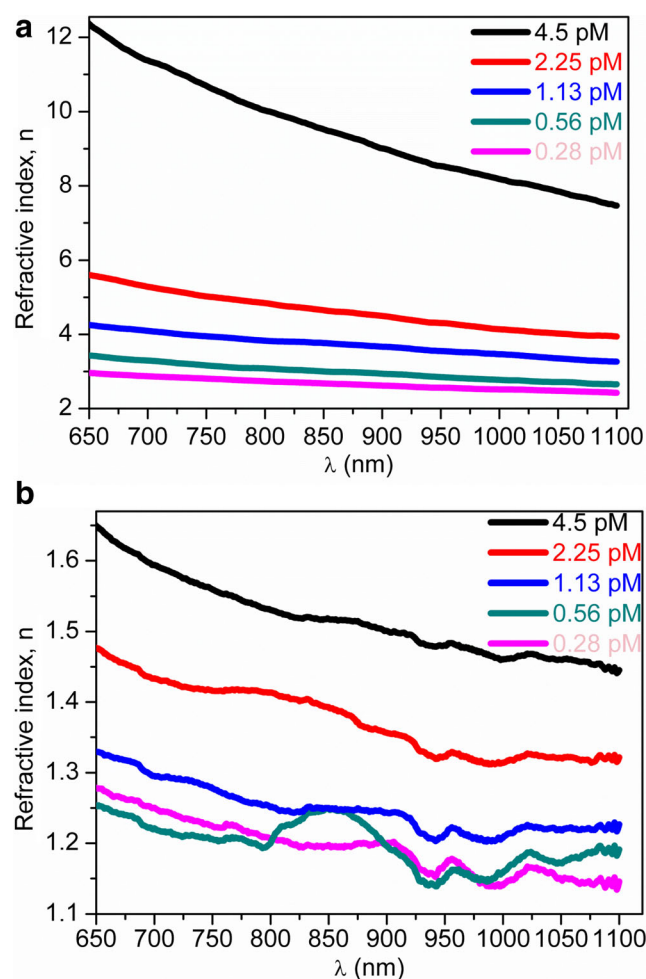


Fig. 8 The plot of n vs. λ of **a** polyPBT and **b** polyBTTP for different molarities

0.56, 1.13, 2.25, and 4.5 pM. As seen in Fig. 8a, the refractive index of polyPBT decreases with decreasing molarity and increasing wavelength. The n value of polyPBT varies from 12.398 to 2.456 with changing of the molarity and wavelength. As seen in Fig. 8b, the refractive index of polyBTTP for molarities higher than 1.13 pM decreases with decreasing molarity and increasing wavelength. The n value of polyBTTP varies from 1.653 to 1.134 with changing of the molarity and wavelength. As seen in Fig. 8a, b, the n values of polyBTTP are lower than the n values of polyPBT. These results suggest that the refractive index of polyBTTP can be decreased with decreasing molarity, increasing wavelength, and ester group addition.

Conclusions

The polymers were synthesized successfully using the coupling reaction and general polycondensation. PolyPBT, having a more rigid structure, exhibited higher thermal stability and higher molecular weights. PolyBTTP gained flexibility by addition to the main chain of the ester functional group. The polymers show good solubility in common organic solvents. The optical properties of the polymers were controlled with ester group addition and molarity. The molar extinction coefficient (ϵ) and ϵ peak values of polyPBT are higher than those of polyBTTP. The transmittance (T) values of polyPBT are lower than those of polyBTTP. The direct allowed band gap (E_{gd}) value (2.519 eV) of polyPBT for 4.5 pM is the lowest value, while the E_{gd} value (2.673 eV) of polyPBT for 0.28 pM is the highest. The refractive index value of polyPBT varies from 12.398 to 2.456 with changing of the molarity and wavelength.

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