Polym. Bull. (2010) 65:551–563
DOI 10.1007/s00289-009-0226-9

ORIGINAL PAPER

Synthesis and characterization of novel optically active and photoactive aromatic polyesters containing 1,8-naphthalimidyl pendant group by step-growth polymerization

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Received: 5 October 2009 / Revised: 5 November 2009 / Accepted: 13 December 2009 /

Abstract Novel optically active polyesters (**PE**)s by step-growth polymerization of a chiral diacid containing naphthalimidyl and flexible chiral groups with different diols via direct polyesterification reaction with tosyl chloride/pyridine/dimethylformamide system as condensing agent were prepared. The resulting **PE**s were characterized using FT-IR, ^{1}H NMR, UV–vis spectroscopy, fluorimetry, and elemental analysis. Fluorescence properties of the **PE3h** as a representative one were examined in several polar aprotic solvents which reveal that this polymer has photoactive properties. Furthermore, thermal properties of these polymers were investigated using thermogravimetric and differential thermogravimetric analyses. The glass-transition temperatures of **PE3b** and **PE3f** were recorded between 264 and 220 °C by differential scanning calorimetry, and the 10% weight loss temperatures were ranging from 350 °C under nitrogen. The obtained macromolecules are readily soluble in many organic solvents.

Keywords Optically active polymers · Polycondensation · Polyesters · Photoactive polymers

Introduction

The development of biodegradable macromolecules has been performed to be an excellent alternative to decrease the volume of the plastic waste disposed into the environment every year [1, 2]. Polyesters (**PE**)s could be biodegradable polymers that have always been an attraction from the early days of Carothers [3, 4]. Different varieties of **PE**s have been synthesized over the past decades from various types of

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diacids and diols [5–7]. Aromatic **PE**s such as polymers from biphenol A have found a wide range of applications due to their notable properties such as good heat and chemical resistance [8–11]. However, their high melting temperature and insolubility in most organic solvents arises limitations in processing technology. For improving solubility of aromatic **PE**s, some approaches have been taken: introduction of kinks of flexible units in the main chain or replacement of the conventional monomers with ones containing bulky pendant groups [12]. Large side groups decrease the molecular mobility, so that the general observable effect is an increase of the glass transition temperature and enhance of solubility simultaneously. It is well known that a vast number of polymers containing heterocyclic ring in the side chain are resistant to high temperature conditions [13, 14].

The most general methods for synthesis of **PE**s comprise bulk polycondensation under elevated temperature and solution step-growth polymerization in high-boiling aprotic organic solvents [15]. Solution polyesterification using tosyl chloride (TsCl)/dimethylformamide (DMF)/pyridine (Py) as a condensing agent produces rather high-molecular-weight **PE**s from aromatic dicarboxylic acids and bisphenols [5–7, 16, 17].

Chiral polymers including those bearing main as well as side chain amino acid units are used extensively in the pharmaceutical industry for enantio-selective separations of drugs [18]. Frequently, a chiral host is prepared by using a natural chiral compound as precursor or modifier. Amino acids and peptides have often been employed as chiral sources in the synthesis of chiral receptors because of their accessibility and biological application [19]. Among the synthetic **PE**s, only those containing the naturally occurring (*L*)-α-amino acids, being structurally close to the natural polypeptides, possess potentially degradable linkages that make them appropriate as biomaterials [20–22].

Synthesis of functional optical materials with photoactive properties has become one of the most talented topics in materials science [23–27]. Fluorescent polymers have been broadly used as ionizing radiation recording materials, luminescent solar concentrators, materials for lasers, paints and varnish industries, luminescent photolayers, and luminescent probes in fibre-optic sensors [28].

Because of mentioned particular characteristics, we wish to report in this present article a facile and simple method for the synthesis of novel chiral and thermally stable **PE**s containing 1,8-naphthalimidyl pendant group with fluorescent properties.

Experimental

Materials

Reagents were purchased from Fluka Chemical (Buchs, Switzerland), Aldrich Chemical (Milwaukee, WI) and Riedel–deHaen AG (Seelze, Germany). *N*,*N*-Dimethylacetamide (DMAc) was dried over BaO and then was distilled under reduced pressure. 1,8-Naphthalenedicarboxylic anhydride was recrystallized from hot acetic anhydride. Diols obtained were used without further purification.

Equipments

Proton nuclear magnetic resonance (^{1}H NMR, 500 MHz) spectra were recorded in DMSO-d_6 solution using a Bruker (Germany) Avance 500 instrument. Proton resonances are designated as singlet (s), multiplet (m), and broad (br). FTIR spectra were recorded on Jasco-680 spectrophotometer (Japan). The spectra of solids were obtained using KBr pellets. The vibrational transition frequencies are reported in wavenumbers (cm^{-1}). Band intensities are assigned as weak (w), medium (m), strong (s), and broad (br). Inherent viscosities were measured by using a Cannon–Fenske Routine Viscometer (Germany) at concentration of 0.5 g/dL at 25 °C. Specific rotations were measured by a Jasco Polarimeter (Japan). Quantitative solubility was determined using 0.05 g of the polymer in 0.5 mL of solvent. Elemental analyses were performed by Tarbit Moalem University, Tehran, Iran. Fluorescence and UV–vis spectra were recorded on a spectrofluorometer with 5 nm, JASCO, FP-750 and UV/Vis/NIR spectrophotometer, JASCO, V-570, respectively. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) data were recorded on a Setaram instrument (Caluire, France) at a heating rate of 10 °C/min under nitrogen atmosphere.

Monomer synthesis

5-[3-Methyl-2-(1,8-naphthalimidyl)butanoylamino]isophthalic acid (**1**) as a diacid monomer was prepared according to our published article [29].

Polymer synthesis

The **PE**s were prepared by the following procedure: For synthesis of **PE3a**, a Py (0.20 mL) solution of TsCl (0.20 g; 1.08×10^{-3} mol), after 30 min stirring at room temperature, was treated with DMF (0.09 mL; 1.22×10^{-3} mol) for 30 min and the resulting solution was added drop wise to a solution of diacid **1** (0.10 g; 2.17×10^{-4} mol) in Py (0.40 mL). The mixture was maintained at room temperature for 30 min and then to this mixture, a solution of bisphenol A (**2a**) (0.05 g; 2.17×10^{-4} mol) in Py (0.20 mL) was added dropwise and the whole solution was stirred at room temperature for 15 min and at 120 °C for 2 h. As the reaction proceeded, the solution became viscous. Then the viscous liquid was precipitated in 30 mL of methanol to give 0.13 g of **PE3a** (83% yield). The other **PE3b–PE3g** were prepared by a similar procedure.

PE3a: FT-IR (KBr): 3325 (br), 2964 (m), 1744 (s), 1704 (s), 1666 (s), 1588 (s), 1540 (s), 1436 (m), 1377 (m), 1339 (s), 1238 (s), 1183 (s), 1099 (m), 1024 (s), 780 (m), 746 (s), 616 (w) cm^{-1}.

PE3b: FT-IR (KBr): 3353 (br), 2964 (m), 1774 (s), 1704 (s), 1666 (s), 1588 (s), 1542 (s), 1436 (m), 1378 (m), 1339 (s), 1287 (s), 1195 (s), 1016 (m), 909 (m), 781 (m), 746 (s), 539 (w) cm^{-1}.

PE3c: FT-IR (KBr): 3352 (br), 2965 (m), 1737 (s), 1703 (s), 1664 (s), 1587 (s), 1542 (m), 1435 (w), 1378 (m), 1340 (s), 1238 (s), 1193 (s), 1012 (w), 908 (w), 780 (m), 749 (s), 536 (w) cm^{-1}.

PE3d: FT-IR (KBr): 3353 (br), 2965 (m), 1748 (s), 1703 (s), 1665 (s), 1588 (s), 1541 (m), 1436 (m), 1378 (m), 1339 (s), 1239 (s), 1190 (s), 1072 (w), 909 (w), 781 (m), 742 (s), 567 (w) cm^{-1}.

PE3e: FT-IR (KBr): 3235 (br), 2964 (m), 1754 (s), 1704 (s), 1666 (s), 1588 (s), 1540 (m), 1436 (m), 1377 (m), 1339 (s), 1238 (s), 1185 (s), 1099 (w), 1024 (s), 909 (m), 780 (m), 746 (s), 618 (w) cm^{-1}.

PE3f: FT-IR (KBr): 3357 (br), 2964 (m), 1754 (s), 1704 (s), 1666 (s), 1588 (s), 1540 (m), 1436 (w), 1377 (m), 1339 (s), 1250 (s), 1185 (s), 1099 (w), 909 (w), 780 (m), 746 (s), 618 (w) cm^{-1}.

PE3g: FT-IR (KBr): 3417 (br), 2964 (m), 1754 (s), 1703 (s), 1665 (s), 1588 (s), 1542 (m), 1436 (m), 1377 (m), 1339 (s), 1239 (s), 1194 (s), 1100 (w), 1031 (w), 909 (w), 781 (m), 746 (w), 640 (w) cm^{-1}.

PE3h: FT-IR (KBr): 3352 (br), 2964 (m), 1744 (s), 1705 (s), 1665 (s), 1588 (s), 1545 (m), 1436 (m), 1378 (m), 1339 (s), 1238 (s), 1169 (s), 1096 (w), 1028 (w), 907 (w), 779 (m), 743 (w), 661 (w) cm^{-1}.

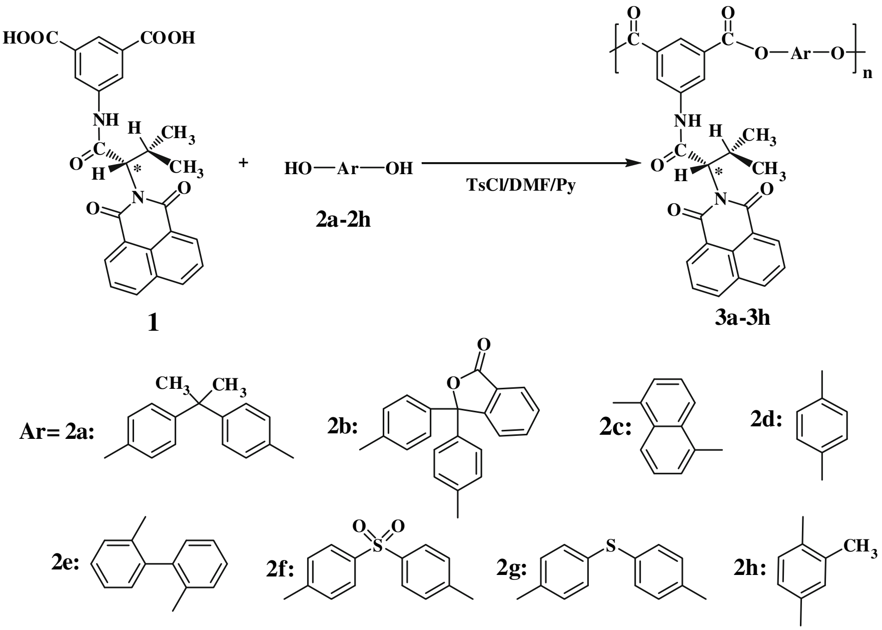
Results and discussion

PEs synthesis

Direct polycondensation of a dicarboxylic acid with a diols using TsCl/DMF/Py as condensing agent to form ester bonds is an efficient way to obtain **PE**s of moderate to high degree of polymerization on a laboratory scale. In this study, the Vilsmeier adduct was used for the polyesterification of aromatic diacid and aromatic diols in the following way (Scheme 1): TsCl was dissolved in Py to yield sulfonium salt and after a certain period of time (aging time) the solution was treated with DMF for 30 min to form Vilsmeier adduct as proposed by Higashi et al. [30–32]. The reaction mixture was added to a solution of diacid in Py to form activated diacid. After 30 min a solution of diol in Py was added and the whole solution was maintained at room temperature and then at an elevated temperature for a period of time. This method was successfully applied for the preparation of **PE3a**–**PE3g** from dicarboxylic acid (**1**) with various aromatic diols (**2a**–**2g**) (Scheme 1).

The **PE**s were obtained in good yields (yields were above 83%) and had inherent viscosity values ranging between 0.27 and 0.61 dL g^{-1} (Table 1). The structures of **PE**s were confirmed by elemental analysis, FT-IR, ^{1}H NMR, and UV–vis spectroscopies. The incorporation of chiral unit into polymer chains was confirmed by measuring their specific rotation (Table 1). **PE**s based on different diols showed different optical rotation and these observations are the result of different polymer's structure. All of the **PE**s show optical rotation and therefore are optically active.

Elemental analysis data of **PE**s are listed in the Table 2. The chemical structures of these newly synthesized **PE**s were confirmed by the good agreement of the elemental analysis values with those of the calculated values.



Scheme 1 Polyesterification of monomer **1** with aromatic diols

Table 1 Synthesis and some physical properties of **PEs** (**3a–3g**)

Diol	Polymer					
	Polymer	Yield (%)	η_{inh} (dL/g)[a]	$[\alpha]^{25}_{\mathrm{Na},589}$ [a]	$[\alpha]^{25}_{\mathrm{Hg}}$ [a]	Color
2a	**PE3a**	86	0.45	+4.48	+8.38	White
2b	**PE3b**	83	0.50	−5.10	−8.48	Lavender
2c	**PE3c**	96	0.41	+7.80	+8.18	Pale-brown
2d	**PE3d**	85	0.27	+4.92	+6.84	White
2e	**PE3e**	86	0.42	+5.20	+6.40	Off white
2f	**PE3f**	91	0.61	+8.10	+8.40	White
2g	**PE3g**	88	0.37	+6.60	+6.80	White
2h	**PE3h**	87	0.27	+3.20	+4.12	Off white

[a] Measured at a concentration of 0.5 g/dL in DMF at 25 °C

Table 2 Elemental analysis of typical **PE**s

Polymer	Formula		Elemental analysis (%)		
			C	H	N
PE3a	$C_{40}H_{32}N_2O_7$	Calcd	73.61	4.94	4.29
	652.69 g/mol	Found	72.76	4.88	4.30
PE3f	$C_{37}H_{26}N_2O_9S$	Calcd	65.87	3.88	4.15
	674.14 g/mol	Found	64.94	3.73	4.25

Polymer characterization

FT-IR study

The structures of these polymers were confirmed as **PE**s by means of FT-IR spectroscopy. The FT-IR spectra of all polymers showed absorptions around 3300 cm^{-1} (υN–H), 1744 cm^{-1} (υC=O asymmetric, imide), 1704 cm^{-1} (υC=O symmetric, imide), and 1666 cm^{-1} (υC=O, amide). A strong band corresponding to C–O stretching for ester group can also be observed around 1190 cm^{-1}. All of these **PE**s exhibited absorption at 1375–1378 cm^{-1} (υC–N, imide) and 780 cm^{-1} that show the presence of the imide heterocycle in these polymers. The **PE3f** showed characteristic absorptions at 1250 and 1185 cm^{-1} due to the sulfone moiety (SO_2 stretching).

^{1}H NMR study

^{1}H NMR (500 MHz) data of **PE3a** as typical **PE** is summarized in Table 3 and the spectrum of **PE3f** is illustrated in Fig. 1. The ^{1}H NMR spectrum of this **PE**s showed a pattern similar to that of monomer [29] but the acidic protons of diacid are omitted and also showed all peaks for aliphatic and aromatic protons of diols which are consistent with the expected structure of corresponding **PE**s. In the ^{1}H NMR spectra of these two polymers (Table 3; Fig. 1), appearance of N–H proton of amide group around 10.00 ppm indicates the presence of amide groups in the polymer side chain. The resonance of aromatic protons appeared at a range of 7.10–8.70 ppm. The proton of the chiral center appeared at 5.32 ppm. The peak of C–H isopropyl group of *S*-valine appeared at 2.80 ppm as a broad peak. The resonance of the two diastereotopic CH_3 protons groups of *S*-valine appeared around 0.70 and 1.20 ppm, respectively.

Polymer properties

*Solubility of the **PE**s*

One of the main objectives of this study was producing modified **PE**s with improved solubility. Because of flexible bulky pendant groups, these polymers are expected to have good solubility. The solubility of **PE**s was tested at a concentration of 5 mg/mL and at ambient temperature in various solvents. All of the **PE**s are soluble

Table 3 ^{1}H NMR data of **PE3a**

Polymer code	δ (ppm)
PE3a	0.69 (s, br, 3H), 1.19 (s, br, 3H), 1.64 (s, br, 6H), 2.80 (s, br, 1H), 5.32 (s, br, 1H), 7.16 (s, br, 4H, Ar–H), 7.26 (s, br, 4H, Ar–H), 7.87 (s, br, 2H, Ar–H), 8.38 (s, br, 1H, Ar–H), 8.48 (m, br, 4H, Ar–H), 8.67 (s, br, 2H, Ar–H), 10.12 (s, NH).

[1] H NMR (500 MHz) were recorded in DMSO-d_6 at RT

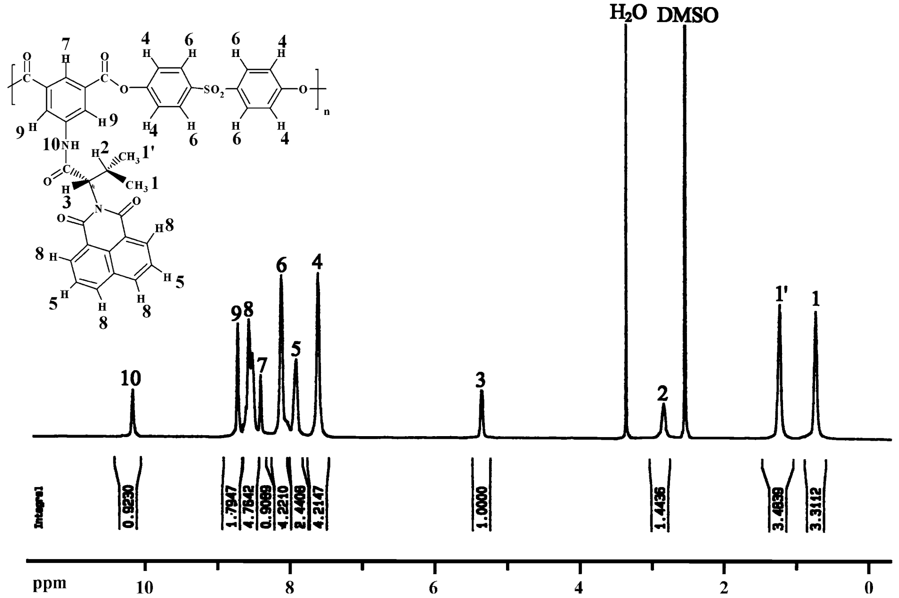


Fig. 1 ^{1}H NMR (500 MHz) spectrum of **PE3f** in DMSO-d6 at R.T

at this concentration in organic polar aprotic solvents such as DMF, DMAc, dimethyl sulfoxide (DMSO), and polar protic solvent such as H_2SO_4 at room temperature, and are insoluble in solvents such as chloroform, methylene chloride, methanol, ethanol, and water.

Thermal properties

The thermal stability of some **PE**s was investigated by TGA/DTG and DSC techniques under a nitrogen atmosphere. Figure 2 showed the TGA/DTG curves of **PE3b** and **PE3f**. Thermal stability of the polymers was studied based on 5 and 10% weight loss (T_5, T_{10}) of the polymers and residue at 800 °C (char yield) and the results are summarized in Table 4. The 10% weight loss temperatures of the aromatic **PE**s in nitrogen were recorded in 390 and 350 °C for **PE3b** and **PE3f**, respectively. Char yield can be used as criteria for evaluating limiting oxygen index (LOI) of the polymers in accordance with Van Krevelen and Hoftyzer equation [33]. LOI = 17.5 + 0.4 CR where CR = char yield. For **PE3b** and **PE3f** LOI values calculated based on their char yield at 800 °C was higher than 28. On the basis of LOI values, such macromolecules can be classified as self-extinguishing polymers. According to Table 4, it is clear that polymer based on phenolphthalein has better thermal stability and higher LOI as compared to other **PE**s. It could be pertained to aromatic and rigid structure of phenolphthalein compared to flexible structure of sulfone diol. The T_gs of the **PE**s were determined by DSC and the results are given in Table 4. The DSC analyses for **PE3b** and **PE3f** show T_g at around 264 and 220 °C, respectively.

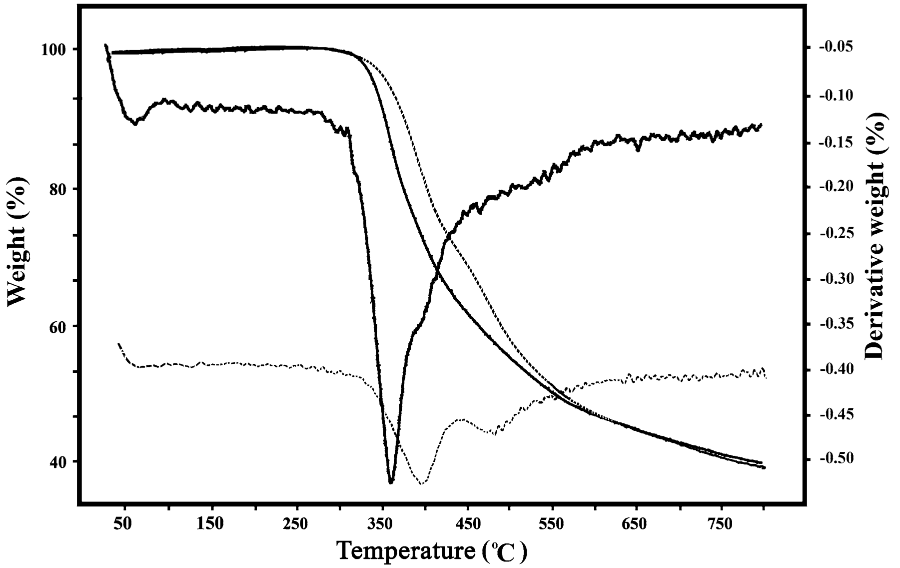


Fig. 2 TGA/DTG thermograms of **PE3b** - - - and **PE3f** — under N_2 atmosphere and a heating rate of 10 °C/min

Table 4 Thermal properties of **PE3b** and **PE3f**

Polymer	Decomposition temperature (°C)		Char yield[b] (%)	T_g[c] (°C)	DTG_{max} (°C)	LOI
	T_5^a	T_{10}^a				
PE3b	379	390	36.7	264	389	32
PE3f	340	350	27.1	220	360	28

[a] Temperature at which 5 and 10% weight loss was recorded by TGA at a heating rate of 10 °C/min in a nitrogen atmosphere

[b] Percentage weight of material left undecomposed after TGA analysis at maximum temperature 800 °C in a nitrogen atmosphere

[c] Glass-transition temperature recorded at a heating rate of 10 °C/min in a nitrogen atmosphere

Fluorimetric studies

Diacid exhibited maximum UV–vis absorption at 264 and 335 nm in DMF solution because of the $\pi \rightarrow \pi^*$ transitions of the aromatic chromophore (naphthalimide). **PE3h** as an example was selected for fluorimetric studies. UV–vis spectrum of **PE3h** was recorded in DMF, DMAc, and DMSO as a solvent, respectively. In the visible region of the spectrum generated well-resolved absorption spectra, showing absorbing bands separated by $\Delta\lambda = 59$ nm, which correspond to the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ bonds. The absorption wavelengths and molar extinction coefficient ($\varepsilon_{\pi\rightarrow\pi^*}$) data of **PE3h** in the above three solvents are listed in Table 5. $\varepsilon_{\pi\rightarrow\pi^*}$ of $\pi \rightarrow \pi^*$ bonds from the polymer system are different in diverse solvents, but there is no change in the shape or the position of the band maxima.

Table 5 UV–vis data of **PE3h** in different solvents

Solvent	μ^{a}	$\lambda_{2(\pi\rightarrow\pi^{*})}$	$\lambda_{1(n\rightarrow\pi^{*}\text{ and }\pi\rightarrow\pi^{*})}$	$\varepsilon_{\pi\rightarrow\pi^{*}}$
DMAc	3.70	268	336	42,805
DMF	3.82	267	336	44,068
DMSO	3.96	269	336	45,137

[a] Dipole moment values at 25 °C [34]

Table 5 demonstrates the dipole moment amounts of the solvents that increase from DMAc to DMF and DMSO, respectively [34]. By increasing the polarity of solvents a bathochromic shift to lower wavelengths with an increase in ε was anticipating. The consequence of this phenomenon is conjugation of carbonyl group with the naphthalimide ring so by increasing the polarity of solvent the length of conjugation will be decreased and finally oxygen of the carbonyl group was solvated. In light of above, $\pi \rightarrow \pi^{*}$ transition must be difficult by according to the decreasing in length of conjugation, so the mentioned shift must be observed. But negligible difference between dipole moments of solvents only changed the displacement of $\varepsilon_{\pi\rightarrow\pi^{*}}$ and did not influence on $\lambda_{\pi\rightarrow\pi^{*}}$ [35]. A typical absorption spectra at constant polymer concentration in DMAc is shown in Fig. 3.

Investigation of the fluorescence behavior of naphthalimide pendant group was followed by exciting the **PE3h** at $\lambda_{\max}$ of naphthalimide moiety. Emission spectra of **PE3h** were recorded in DMF, DMAc, and DMSO, respectively, by fixing the

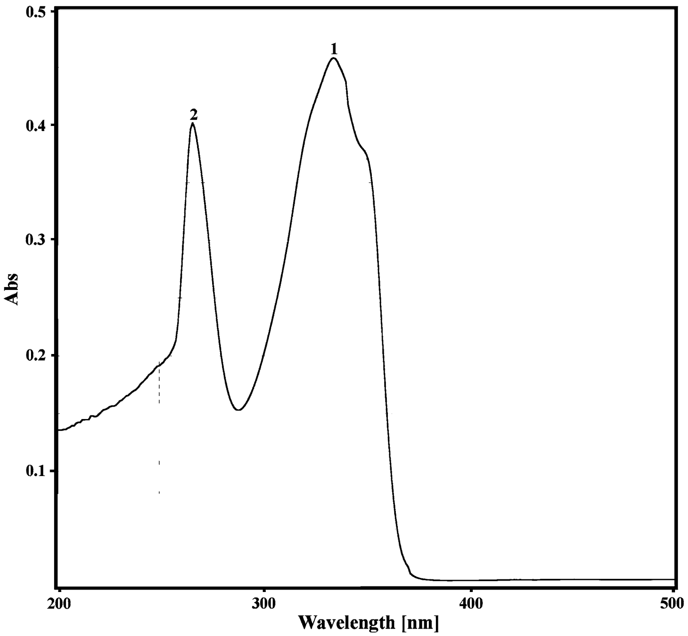


Fig. 3 Absorption spectrum of polymer **PE3h** in DMAc at 1.00 × 10^{-5} M concentration with 1.0 cm optical path

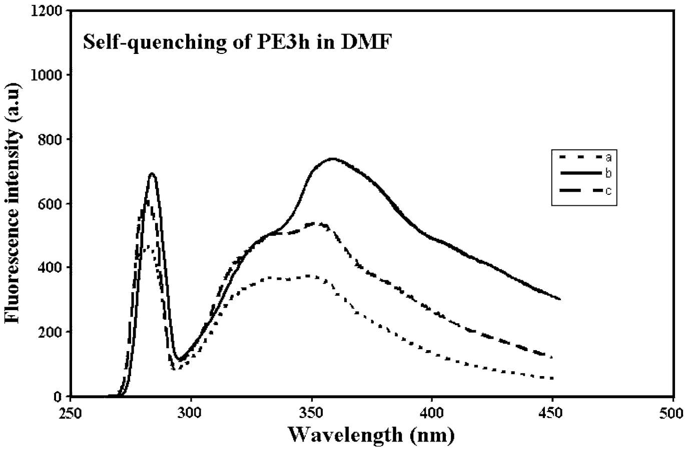


Fig. 4 Self-quenching of **PE3h** in DMF at (a) 1.00 × 10^{-5} M, (b) 1.00 × 10^{-6} M, and (c) 1.00 × 10^{-7} M (*au* arbitrary unit)

excitation wavelength at absorption wavelength, i.e., at 267 nm. When it was excited at this wavelength, emission fluorescence wavelengths were observed with maximum around 284, 334, and 359 nm, respectively, (monomer was excited at 264 nm too and emission fluorescence wavelengths were observed with maximum around 288 and 324 nm, respectively). Figure 4 shows the correlation between fluorescence intensity of **PE3h** and emission wavelengths at different polymer concentrations in DMF as a polar aprotic solvent. Dependency of fluorescence intensity on polymer concentration illustrates the self-quenching phenomenon [35]. Decreasing the polymer concentration to an ideal concentration (a concentration, which maximum fluorescence intensity has been observed), the fluorescence intensity enhances and then it will decrease upon falling polymer concentration. The spectra obtained from the polymer systems in different solvents, vary in the intensity and shape of the maxima bands.

The solvent character has an effect on the relative fluorescence intensity of the maxima emission fluorescence wavelength. Table 6 shows the maximum emission of **PE3h** in different solvents. In **PE3h**, the maximum fluorescence intensity was studied in DMF and all intensities in DMAc were less than DMF and also in DMSO were less than DMAc. Separation between macromolecules is increased by raising the dielectric constant of the solvent, and thus self-quenching of a polymer with another one will be decreased. Consequently, the relative fluorescence intensity

Table 6 Maximum fluorescence wavelengths and fluorescence intensity of **PE3h** in diverse solvents

Solvent	$\lambda_{\text{max emission}}$	Fluorescence intensity (au)
DMF	284	693.78
DMAc	284	310.48
DMSO	284	166.08

au arbitrary unit

must increase. In accordance with this theory, it is predictable that the comparative fluorescence intensities will pursue the trend based on dielectric constant, i.e., DMSO > DMAc > DMF [34], whereas, the observed inclination is on the contrary with above one. Accordingly, another parameter, which is the consequence of viscosity on intermolecular separation, must be measured. Viscosity values of DMAc, DMF, and DMSO as solvent at 50 °C are 0.927, 0.794 and 1.987, respectively. With increasing the viscosity of solvents, the quenching of polymer with molecule of solvents will raise but, the self-quenching is decreased. Hence, the results of these two reverse phenomena promote the relative fluorescence intensity [35].

Conclusions

Novel photoactive and optically active **PE3a–PE3h** have been synthesized by the reaction of diacid **1** with aromatic diols (**2a–2h**). The resulting polymers show excellent solubility due to bulky pendant groups, good thermal stability with glass-transition temperature around 200 °C, and fluorescence emission phenomena. Since the obtained polymers are optically active they may have potential to be used in the column chromatography technique for the separation of enantiomeric mixtures. The choice of 1,8-naphthalenedicarboxylic anhydride was due to the many derivatives of l,8-naphthalic anhydride exhibit strong fluorescence emission and serve for this reason as fluorescent dyes and fluorescent whitening agents. For the investigation of the fluorescence behavior of the **PE**s, one polymer was selected as representative and its emission fluorescence pattern was recorded in some aprotic polar solvents by fixing the excitation wavelength at maximum absorption wavelengths. The resulting polymers could have many applications such as photoactive materials which may be used in solar energy collectors as electro-optically sensitive materials and for laser activity.

Acknowledgments We wish to express our gratitude to the Research Affairs Division, Isfahan University of Technology (IUT), for partial financial support. Further financial support from National Elite Foundation (NEF) and Center of Excellency in Sensors and Green Research (IUT) is gratefully acknowledged. We also thanks in advanced Prof. B. Rezaie (Analytical Chemistry Division of IUT), Prof. M. Amirnasr (Inorganic Chemistry Division of IUT), Mr. M. Dinari, Mr. M. Taghavi, and Dr. Z. Rafiee for helpful discussions.

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