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(Graph Attention Networks)



کارگاه آنلاین آموزش استفاده از
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کارگاه آنلاین مقاله روزمره انگلیسی



Step-growth Polymerization of 5-(3-Acetoxy-naphtho-yl-amino)isophthalic acid with Different Aromatic Diols

Shadpour Mallakpour* and Zahra Rafiee

Organic Polymer Chemistry Research Laboratory, Department of Chemistry,
Isfahan University of Technology, Isfahan 84156-83111, Iran

Received 27 September 2007; accepted 16 February 2008

ABSTRACT

Soluble and photoactive novel aromatic polyesters containing acetoxy-naphthal- amide unit in the side chain have been synthesized with moderate molecular weights by direct polycondensation of 5-(3-acetoxy-naphtho-yl-amino)isophthalic acid with different aromatic diols using tosyl chloride (TsCl), pyridine (Py) and *N,N*-dimethylformamide (DMF) system as a condensing agent. The reaction was successfully applied to the preparation of thermally stable polyesters with good yields and inherent viscosities in the range of 0.28-0.62 dL/g. The resulted polymers are readily soluble in aprotic polar solvents such as *N*-methyl-2-pyrrolidone, *N,N*-dimethyl-acetamide, DMF, Py and dimethyl sulphoxide. Thermogravimetric analysis data showed that these polyesters are thermally stable, with 10% weight loss being recorded above 360°C. These macromolecules exhibited maximum UV-vis absorption at 269 and 320 nm in DMF solution. Their photoluminescence in DMF solution demonstrated fluorescence emission maxima around 360 and 428 nm for all polyesters obtained.

Key Words:

polyester;
TsCl/Py/DMF condensing agent;
thermal properties;
solubility;
direct polycondensation.

INTRODUCTION

In recent years many researchers have been interested in the synthesis and characterization of fluorescent polymers [1]. The derivatives of naphthalene, anthracene, other condensed aromatic hydrocarbons, carbazole and pyrazoline are more significant in the preparation of photoluminescence polymers. In

general, there are two approaches for preparation of fluorescent macromolecules. The first approach involves the polymerization of a monomer containing a fluorescent chromophore [2,3]. The second approach involves the chemical modification of commercially available polymers containing

(*) To whom correspondence to be addressed.
E-mail: mallak@cc.iut.ac.ir

reactive groups by using fluorescent dyes or organic molecules [4]. They find utilization as ionizing radiation recording materials, luminescent solar concentrators, materials for lasers, materials for the paints and varnish industries, luminescent photolayers, and luminescent probes in fibre-optic sensors [5].

Recently many attempts been made by researchers on high-performance polymers, with outstanding thermal stability and solubility, and provided great impetus to the discovery of a variety of thermostable and processable polymers. Aromatic polyesters are materials exhibiting an excellent pattern of physical properties. They are significant category of high performance and engineering materials, which exhibit good thermal stability, solvent resistance and good mechanical properties and therefore, they are applied widely in the aviation, automobile, and electronic industries [6-8]. Aromatic polyesters are also well known as rigid-rod liquid-crystalline polymers [9-11]. Polyethylene terephthalates are useful as engineering thermoplastics because of their good chemical resistance, good thermal and dimensional stability, high strength and rigidity, and good surface hardness and gloss [12,13].

Aromatic polyesters are generally prepared by solution or an interfacial reaction between aromatic dicarboxylic acids and bisphenols under severe conditions [14-16]. Higashi et al. reported that the reactions promoted by toluenesulphonyl chloride and *N,N*-dimethylformamide (DMF) were effective for the synthesis of aromatic dicarboxylic acids and bisphenols [17]. The direct polycondensation of dicarboxylic acids with diols using diphenylammonium triflate as catalyst was also reported [18]. These polymers are generally difficult to process, because of their high glass transition or melting temperatures coupled with insolubility in most organic solvents by virtue of their rigid structures. One of the approaches for improving solubility of aromatic polyesters without extreme loss of their high thermal stability is the introduction of flexible units in the main chain [19-22]. Incorporation of bulky, packing-disruptive groups into the polymer backbone [23-28] is also of particular concern because this approach causes the separation of polymer chains and lowers the chain packing, creating free volumes. Bulky side groups also hinder molecular mobility and therefore the over-

all observable and simultaneous effect is an increase in glass transition temperature as well as solubility improvement.

One of the methods of improving the thermal properties of aromatic polyesters is to introduce some thermally stable groups, such as amide groups into their main chains. Improvements of the mechanical and thermal properties and crystallinity of polymers by the incorporation of a certain amount of amide groups into the main chains of aromatic polyesters have been reported [29,30].

Naphthalene-containing thermoplastic polyesters have attracted an increasing interest in recent years [31]. Poly(ethylene naphthalate) (PEN), poly(butylene naphthalate) (PBN) and poly(propylene naphthalate) (PPN) are the most well known members of this family of thermoplastics. These newly developed high performance polymers, containing a rigid naphthalene ring in the repeating unit and a flexible alkylene group, exhibit superior physical and mechanical properties than widely used corresponding phthalate-based polyesters. Naphthalene rings have been introduced into polymer structures by some research workers as it has a larger volume than benzene ring, which increases the proportion of the rigid group in the main chain and blocks the mobility of segments and therefore increases the glass transition temperature (T_g) of the polymer [32-37].

In this investigation, we wish to describe the successful synthesis and characterization of novel aromatic polyesters containing acetoxynaphthalamide moiety using tosyl chloride (TsCl)/DMF/pyridine (Py) as condensing agent. All the resulting polymers are new and they are reported for the first time. The incorporation of the bulky and rigid naphthalene pendant group into the polymer backbone not only can induce photoactivity, it may disrupt the molecular packing as well, reducing the intermolecular interactions and enhancing the solubility and thermal stability of the resulting new polymers.

EXPERIMENTAL

Materials

All chemicals were purchased from Fluka Chemical Co. (Buchs, Switzerland), Aldrich Chemical Co.

(Milwaukee, WI), Riedel-deHaen AG (Seelze, Germany) and Merck Chemical Co. Bisphenol A (**5a**) was purified by recrystallization from acetic acid-water. The other diols were used as obtained without further purification. DMF was dried over BaO and then it was distilled under reduced pressure.

Techniques

Proton nuclear magnetic resonance (^1H NMR, 500 MHz) spectra were recorded on a Bruker (Germany) Avance 500 instrument. FTIR Spectra were recorded on (Jasco-680, Japan) spectrophotometer. The spectra of solids were obtained using KBr pellets. The vibrational transition frequencies are reported in wave-numbers (cm^{-1}). Band intensities are assigned as weak (w), medium (m), strong (s) and broad (br). Inherent viscosities were measured by a standard procedure using a Cannon Fenske routine viscometer. Specific rotations were measured by a Jasco Polarimeter (Japan). Fluorescence and UV-vis spectra were recorded in DMF on a JASCO, FP-750 and UV/Vis/NIR, JASCO, V-570, spectrophotometer, respectively. Thermal gravimetric analysis (TGA) data for polymers were obtained by TGA-Perkin Elmer (Pyris 1) in nitrogen atmosphere at a heating rate of $10^\circ\text{C}/\text{min}$. Elemental analyses were performed in Iran Polymer and Petrochemical Research Institute, Tehran, Iran.

Monomer Synthesis

5-(3-Acetoxy-naphthoylamino)isophthalic acid (**4**) as a photoactive diacid was prepared according to our pervious published paper [38].

Polymer Synthesis

The aromatic polyesters were prepared by the following general procedure: as an example for the preparation of polymer **6a**,

A Py (0.15 mL, 1.9×10^{-3} mol) solution of TsCl (0.250 g, 1.25×10^{-3} mol) after 30 min stirring at room temperature, was treated with DMF (0.10 mL, 1.3×10^{-3} mol) for 30 min and was added dropwise to a solution of diacid **4** (0.100 g, 2.5×10^{-4} mol) in Py (0.15 mL). The mixture was maintained at room temperature for 30 min and then to this mixture, a solution of bisphenol A (**5a**) (0.058 g, 2.5×10^{-4} mol) in Py (0.15 mL) was added dropwise at room tempera-

ture and the whole solution was stirred at room temperature for 30 min and at 120°C for 2.5 h. As the reaction ensued, the solution became viscous. Then, the viscous liquid was precipitated in 30 mL of methanol to yield 0.153 g (97%) of polymer **6a**.

FTIR (KBr): 3417 (m), 3060 (w), 2966 (m), 1741 (s), 1684 (s), 1633 (s), 1599 (s), 1547 (m), 1504 (s), 1452 (s), 1413 (m), 1334 (s), 1199 (s), 1169 (s), 1079 (m), 1015 (m), 914 (m), 839 (m), 808 (m), 745 (s), 680 (m), 569 (m), 478 (m) cm^{-1} . λ_{max} (ϵ) = 268 (133157), 323 nm ($18762 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$).

The other polyesters (**6b-6j**) were prepared with similar procedures.

Polyester 6b

FTIR (KBr): 3411 (m), 3061 (m), 2956 (w), 1741 (s), 1700 (s), 1632 (m), 1599 (m), 1550 (s), 1486 (s), 1451 (s), 1400 (s), 1339 (s), 1278 (s), 1194 (s), 1162 (s), 1098 (s), 1033 (s), 1011 (s), 958 (m), 914 (m), 846 (m), 813 (m), 745 (m), 681 (s) cm^{-1} . ^1H NMR (500 MHz, DMSO- d_6): δ 2.24 (s, 3H, CH_3), 7.06-8.82 (m, 17H, Ar-H), 10.80 (s, 1H, N-H) ppm. λ_{max} (ϵ) = 267 (134137), 321 nm ($15592 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$).

Polyester 6c

FTIR (KBr): 3419 (m), 3076 (m), 2918 (w), 1745 (s), 1704 (s), 1632 (s), 1588 (s), 1489 (s), 1453 (s), 1406 (s), 1334 (s), 1294 (s), 1194 (s), 1152 (s), 1105 (s), 1033 (m), 1011 (m), 915 (w), 816 (w), 743 (w), 682 (w) cm^{-1} . λ_{max} (ϵ) = 269 (154283), 323 nm ($18392 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$).

Polyester 6d

FTIR (KBr): 3412 (m), 3062 (m), 2925 (m), 1744 (s), 1701 (s), 1675 (s), 1632 (s), 1600 (s), 1551 (s), 1453 (s), 1422 (s), 1338 (s), 1199 (s), 1159 (s), 1106 (s), 1056 (s), 1033 (s), 1009 (s), 959 (m), 906 (m), 815 (s), 746 (s), 681 (s) cm^{-1} . λ_{max} (ϵ) = 268 (149832), 321 nm ($17685 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$).

Polyester 6e

FTIR (KBr): 3411 (m), 3058 (m), 2967 (m), 1742 (s), 1682 (s), 1632 (s), 1599 (s), 1543 (s), 1504 (s), 1451 (s), 1422 (m), 1384 (s), 1337 (m), 1199 (s), 1169 (s), 1100 (s), 1079 (m), 1034 (m), 1015 (s), 958 (m), 914 (m), 846 (m), 815 (m), 746 (s), 710 (m) cm^{-1} . ^1H NMR (500 MHz, DMSO- d_6): δ 2.19 (s, 3H, CH_3),

7.08-8.71 (m, 13H, Ar-H), 10.79 (s, 1H, N-H) ppm.
 $\lambda_{\max} (\epsilon) = 271 (122769), 324 \text{ nm} (16986 \text{ L.mol}^{-1}.\text{cm}^{-1})$.

Polyester 6f

FTIR (KBr): 3308 (m), 3067 (m), 2965 (w), 1744 (s), 1661 (s), 1628 (s), 1598 (s), 1556 (s), 1505 (s), 1451 (s), 1412 (m), 1385 (m), 1338 (s), 1288 (s), 1197 (s), 1167 (s), 1117 (s), 1102 (s), 1084 (s), 1032 (m), 1016 (m), 970 (m), 928 (m), 870 (m), 848 (m), 815 (m), 746 (s) cm^{-1} . $\lambda_{\max} (\epsilon) = 268 (143296), 322 \text{ nm} (16877 \text{ L.mol}^{-1}.\text{cm}^{-1})$.

Polyester 6g

FTIR (KBr): 3308 (m), 3063 (m), 2929 (w), 1742 (s), 1661 (s), 1630 (s), 1599 (s), 1551 (s), 1506 (m), 1474 (m), 1450 (s), 1338 (s), 1186 (s), 1119 (s), 1065 (m), 1034 (w), 1009 (m), 955 (m), 915 (w), 874 (m), 837 (m), 815 (m), 746 (m) cm^{-1} . $\lambda_{\max} (\epsilon) = 269 (154789), 324 \text{ nm} (16087 \text{ L.mol}^{-1}.\text{cm}^{-1})$.

Polyester 6h

FTIR (KBr): 3411 (m), 3069 (m), 2928 (w), 1743 (s),

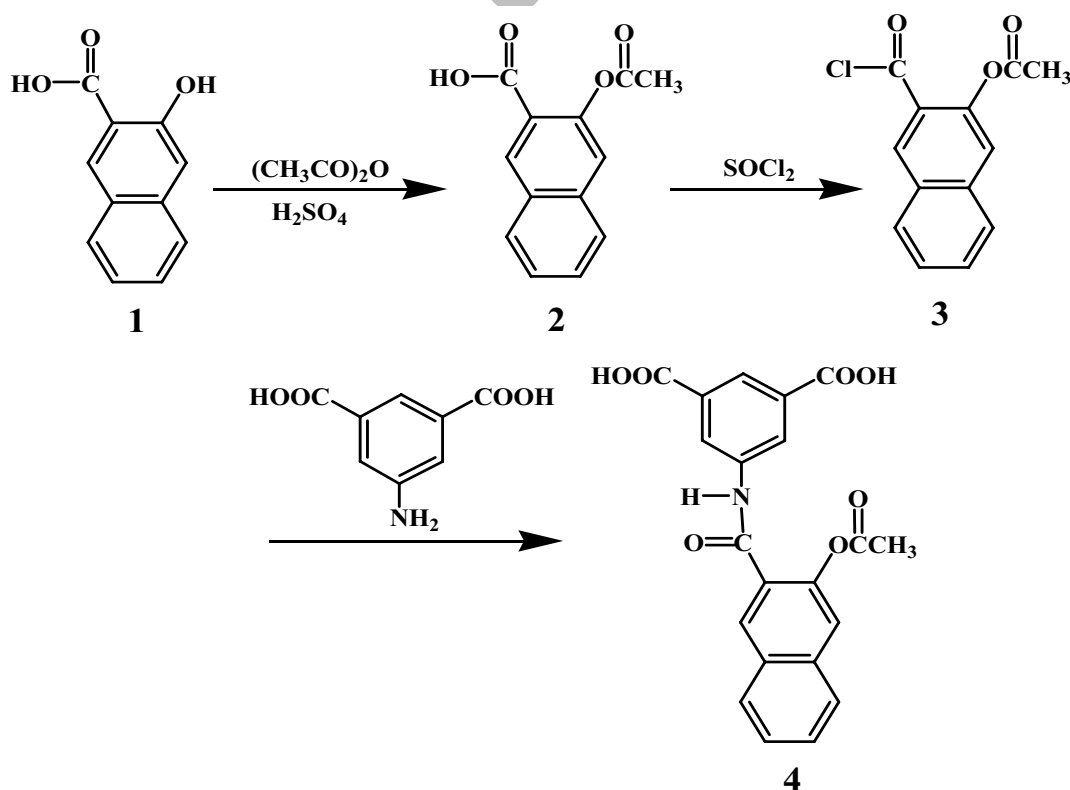
1662 (s), 1632 (s), 1605 (s), 1556 (s), 1497 (s), 1451 (s), 1485 (s), 1339 (s), 1201 (s), 1159 (s), 1124 (s), 1060 (m), 1033 (m), 1009 (m), 985 (m), 907 (m), 816 (w), 746 (w), 681 (m), 610 (m) cm^{-1} . $\lambda_{\max} (\epsilon) = 267 (134896), 321 \text{ nm} (17574 \text{ L.mol}^{-1}.\text{cm}^{-1})$.

Polyester 6i

FTIR (KBr): 3308 (m), 3066 (m), 2965 (w), 1745 (s), 1700 (s), 1673 (s), 1633 (s), 1590 (s), 1550 (s), 1503 (m), 1486 (m), 1453 (s), 1408 (s), 1337 (s), 1187 (s), 1166 (s), 1120 (s), 1061 (s), 1031 (s), 1008 (s), 957 (m), 909 (m), 869 (m), 815 (m), 798 (m), 741 (m), 723 (m) cm^{-1} . $\lambda_{\max} (\epsilon) = 270 (149822), 321 \text{ nm} (16966 \text{ L.mol}^{-1}.\text{cm}^{-1})$.

Polyester 6j

FTIR (KBr): 3413 (m), 3065 (m), 2980 (w), 1743 (s), 1663 (s), 1631 (s), 1600 (s), 1552 (s), 1507 (m), 1452 (m), 1401 (s), 1339 (s), 1197 (s), 1160 (s), 1103 (s), 1062 (s), 1032 (m), 1009 (m), 957 (m), 906 (m), 814 (w), 781 (m), 746 (m) cm^{-1} . $\lambda_{\max} (\epsilon) = 269 (137519), 322 \text{ nm} (16712 \text{ L.mol}^{-1}.\text{cm}^{-1})$.



Scheme I. Synthesis of 5-(3-acetoxynaphthoylamino)isophthalic acid (**4**).

RESULTS AND DISCUSSION

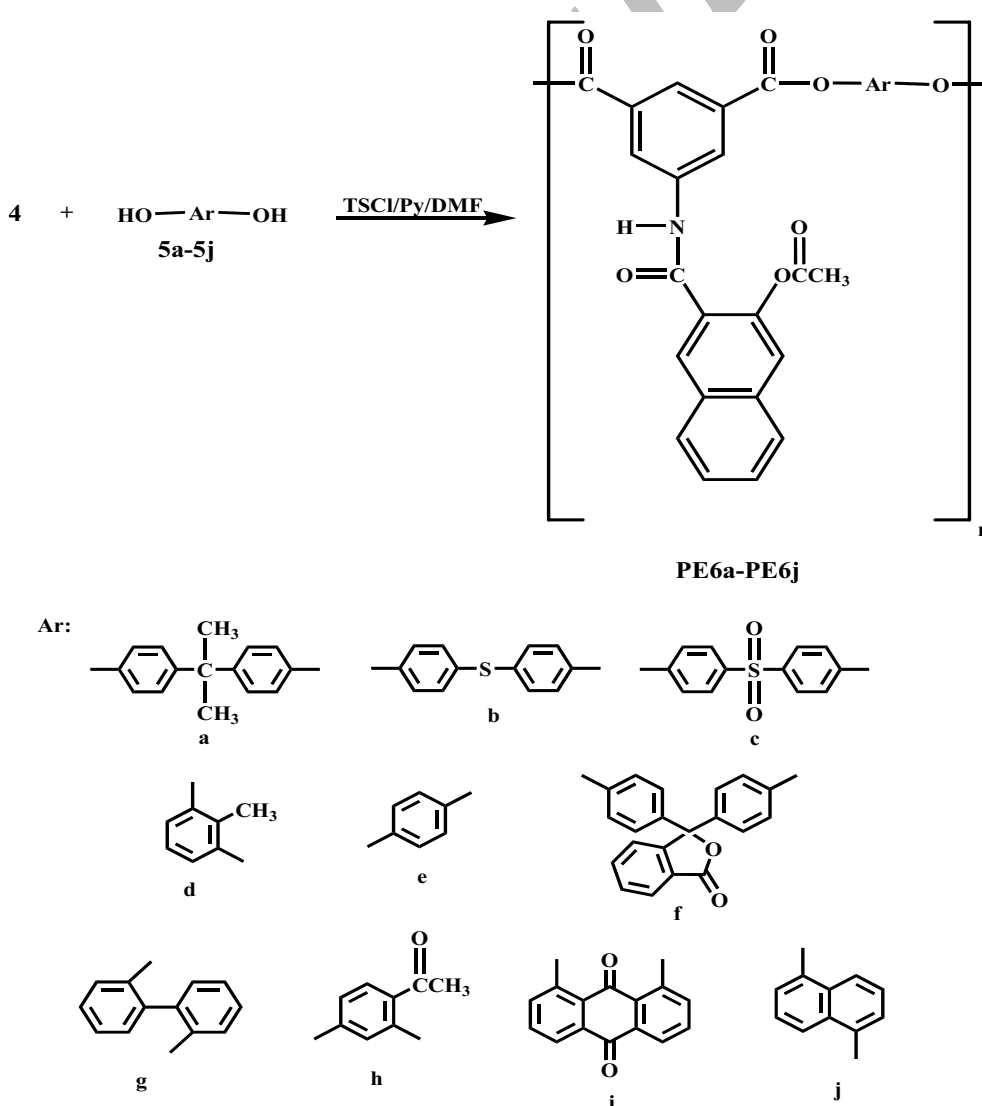
Monomer Synthesis

As shown in Scheme I, monomer diacid **4** was synthesized in three steps according to our previous work [38].

Polymer Synthesis

Polyesterification is an important polycondensation reaction from both the scientific and the technological point of view. New all-aromatic polyesters with pendant substituted naphthalamide group were synthesized with the aim of achieving better solubility and thermal stability. These new polymers **6a-6j** were

prepared by direct polycondensation of a novel diacid monomer **4** with several different aromatic diols (**5a-5j**) in a system of TsCl/Py/DMF (Scheme II). In this investigation for the polycondensation reaction of diacid **4** with aromatic diols, a Vilsmeier adduct was prepared by dissolving TsCl in a mixed solvent of Py and DMF. The polycondensation was carried out in the following way: TsCl was dissolved in Py and after a certain period of time (aging time) the solution was treated with DMF for 30 min. The reaction mixture was added to a solution of diacid in Py. After a period of time a solution of diol in Py was added and the whole solution was maintained at elevated temperature for several hours.



Scheme II. Polyesterification reactions of monomer **4** with aromatic diols.

Table 1. Synthesis and some physical properties of polyesters **6a-6j**.

Entry	Diol	Polymer			
		Polymer	Yield (%)	η_{inh}^a (dL/g)	Colour
1	5a	6a	97	0.62	Off-white
2	5b	6b	74	0.35	Off-white
3	5c	6c	89	0.38	Off-white
4	5d	6d	89	0.34	Light-brown
5	5e	6e	95	0.42	Off-white
6	5f	6f	94	0.41	Off-white
7	5g	6g	78	0.28	Off-white
8	5h	6h	79	0.39	Light-brown
9	5i	6i	77	0.29	Light-brown
10	5j	6j	96	0.31	Light-brown

(a) Measured at a concentration of 0.5 g/dL in DMF at 25°C.

All the reaction parameters such as aging times, reaction time, temperature and molar ratio of chemical additives to diacid which have considerable effect on the reaction progress were optimized in previous work [21] and used for direct polyesterification of diacid **4**. The reaction yields and some physical data of the polyesters obtained are listed in Table 1.

The inherent viscosities of the resulting polymers under optimized conditions were in the range of 0.28-0.62 dL/g and the yields were 74-97%.

Polymer Characterization

The formation of aromatic polyesters was confirmed by FTIR spectroscopy analysis. FTIR Spectra of all polymers show the characteristic carbonyl absorption peaks for the amide and ester at 1745, 1660, and 1632 cm^{-1} , due to the symmetrical and asymmetrical carbonyl stretching vibrations. Band of amide N-H group appeared around 3400 cm^{-1} . A strong band corresponding to C-O stretching can also be observed at 1196

cm^{-1} . ^1H NMR (500 MHz) spectra of polymers **6b** and **6e** were consistent with their structures. The results of elemental analysis were in agreement with the calculated values (Table 2).

UV-Vis Absorption and Fluorescence Characteristics

These polymers exhibited maximum UV-vis absorption at 269 and 320 nm in DMF solution because of the π - π^* transitions of the aromatic chromophore (naphthalene). The absorption spectrum of polyester **6a** is shown in Figure 1. In comparing the UV-vis absorption of the resulting polymers with a series of segmented poly(urethane-urea)s containing naphthalene it becomes evident that the latter polymers gave absorption at 300 and 360 nm in DMF solution [39]. The fluorescence spectra of the polyesters were also recorded in DMF. For example, in the case of the polyester **6a**, the polymer was excited at 260 nm, and emission fluorescence wavelengths were observed at 360 and 428 nm, respectively. The emission fluores-

Table 2. Elemental analysis of polyesters **6c** and **6f**.

Polyester entry	Entry	Measurements	Elemental analysis (%)		
			C	H	N
6c	$(\text{C}_{33}\text{H}_{21}\text{NO}_9\text{S}_2)_n$ (607.6) _n	calcd	65.23	3.48	2.31
		found	65.64	3.58	2.74
6f	$(\text{C}_{41}\text{H}_{25}\text{NO}_9)_n$ (675.1) _n	calcd	72.88	3.73	2.07
		found	72.19	3.64	2.19

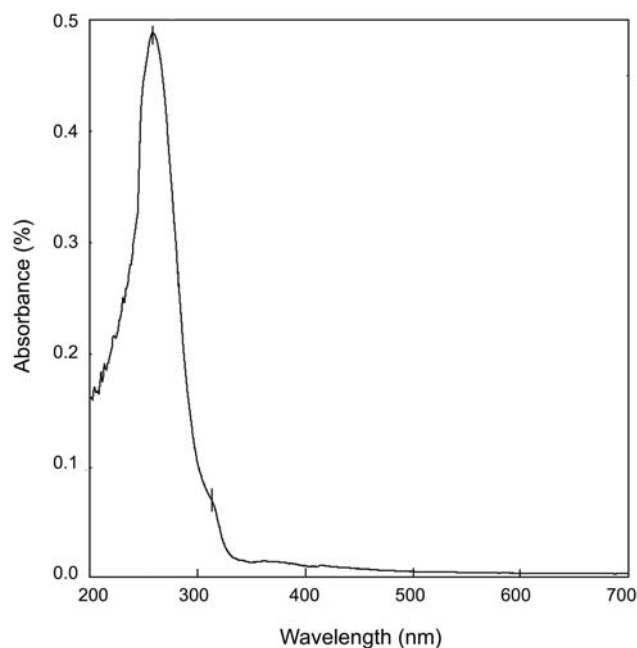


Figure 1. UV-Vis spectrum of polyester **6a**.

cence pattern of these polymers was also compared with the same segmented poly(urethane-urea)s which gave similar pattern, but the emission intensity is lower than those of poly(urethane-urea)s [35]. All these compounds show almost similar UV-vis and fluorescence spectra pattern.

Solubility of Aromatic Polyesters

The solubility of polyesters was tested quantitatively in various solvents as listed in Table 3. All the poly-

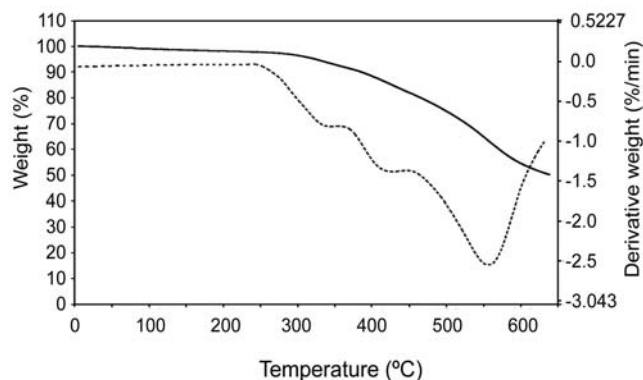


Figure 2. TGA/DTG thermogram of polyester **6a** with a heating rate of 10°C/min in nitrogen atmosphere.

esters are soluble in organic solvents such as DMF, DMAc, dimethyl sulphoxide, *N*-methyl-2-pyrrolidone, Py and in H₂SO₄ at room temperature, and are insoluble in solvents such as chloroform, methylene chloride, methanol, ethanol and water. The incorporation of the pendant acetoxynaphthalamide in the side chain polyesters enhanced their solubility in polar solvents.

Thermal Properties

The thermal stability of polymers was studied by TGA/DTG under a nitrogen atmosphere. Figures 2 and 3 present TGA thermograms of polyesters **6a** and **6e**, respectively. The 5 and 10% weight loss (T₅ and T₁₀) of the polymers and residue at 600°C (char yield) for these polymers are summarized in Table 4. The

Table 3. Solubility of polyesters **6a-6j**^a.

Solvent	6a	6b	6c	6d	6e	6f	6g	6h	6i	6j
DMAc	+	+	+	+	+	+	+	+	+	+
DMF	+	+	+	+	+	+	+	+	+	+
NMP	+	+	+	+	+	+	+	+	+	+
DMSO	+	+	+	+	+	+	+	+	+	+
Py	+	+	+	+	+	+	+	+	+	+
H ₂ SO ₄	+	+	+	+	+	+	+	+	+	+
MeOH	-	-	-	-	-	-	-	-	-	-
EtOH	-	-	-	-	-	-	-	-	-	-
CHCl ₃	-	-	-	-	-	-	-	-	-	-
CH ₂ Cl ₂	-	-	-	-	-	-	-	-	-	-
H ₂ O	-	-	-	-	-	-	-	-	-	-

(a) Concentration: 5 mg.mL⁻¹; (+) soluble at room temperature; (-) insoluble at room temperature.

Table 4. Thermal properties of polyesters **6a** and **6e**.

Polyester	Decomposition temperature (°C)		Char yield ^c (%)
	T ₅ ^a	T ₁₀ ^b	
6a	327	386	55.0
6e	315	360	45.5

(a) Temperature at which 5% weight loss was recorded by TGA at a heating rate of 10°C/min in a nitrogen atmosphere. (b) Temperature at which 10% weight loss was recorded by TGA at a heating rate of 10°C/min in a nitrogen atmosphere. (c) Percentage weight of material left undecomposed after TGA analysis at maximum temperature 600°C in a nitrogen atmosphere.

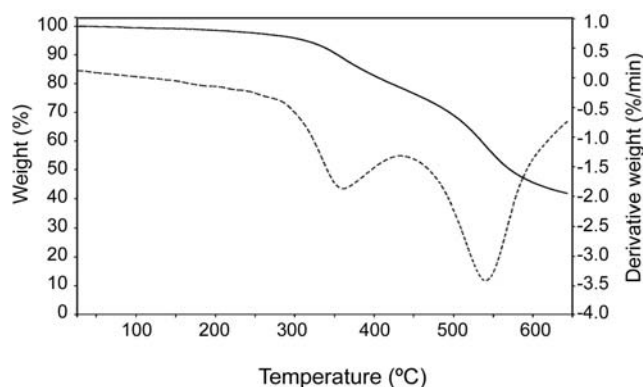


Figure 3. TGA/DTG thermogram of polyester **6e** with a heating rate of 10°C/min in nitrogen atmosphere.

polymers are thermally stable up to above 310°C as indicated by the weight loss in TGA thermograms.

CONCLUSION

In this research work, direct polycondensation was carried out by the reaction of diacid monomer **4** with several aromatic diols using TsCl/DMF/Py as a condensing agent to prepare novel aromatic polyesters. The polycondensation leads to the formation of polymers having inherent viscosity ranging from 0.28-0.62 dL/g. By introducing acetoxynaphthalamide pendant group to the aromatic polyesters side chain, polymers with substantially increased solubility and good thermal stability were obtained. Since the resulting polyesters have naphthalene functional group, they have the potentiality to be used as photolabing and photoresponsive materials and since polyesters are readily soluble in many organic solvents, they can be used for film forming and coating materials.

ACKNOWLEDGEMENTS

We wish to express our gratitude to the Research Affairs Division of Isfahan University of Technology (IUT), for financial support. Further financial support from the Center of Excellency in Sensors and Green Chemistry Research (IUT) is gratefully acknowledged.

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