

HEAT STABLE AND ORGANOSOLUBLE BENZOXAZOLE OR BENZOTHAIAZOLE-CONTAINING POLY(IMIDE-ESTER)S AND POLY(ETHER-IMIDE-ESTER)S: SYNTHESIS AND CHARACTERIZATION

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Abstract Two series of poly(imide-ester)s (PIEs) and poly(ether-imide-ester)s (PEIEs), having benzoxazole or benzothiazole pendent groups, were conveniently prepared by the diphenylchlorophosphate-activated direct polyesterification of two bis(imide-carboxylic acid)s (**1**), such as 2-[3,5-bis(*N*-trimellitimidoyl)phenyl]benzoxazole (**1_O**) and 2-[3,5-bis(*N*-trimellitimidoyl)phenyl]benzothiazole (**1_S**) and two bis(imide-ether-carboxylic acid)s (**2**), such as 2-[3,5-bis(4-trimellitimidophenoxy)-phenyl]benzoxazole (**2_O**), and 2-[3,5-bis(4-trimellitimidophenoxy)-phenyl]benzothiazole (**2_S**) with various aromatic dihydroxy compounds in the presence of pyridine and lithium chloride. The structures, solubilities and thermal properties of obtained polymers were investigated in detail. All of the resulting polymers were characterized by FTIR and ¹H-NMR spectroscopy and elemental analysis. All of the resulting polymers exhibited excellent solubility in common organic solvents, such as pyridine, tetrahydrofuran and *m*-cresol, as well as in polar organic solvents, such as *N*-methyl-2-pyrrolidone, *N,N*-dimethylacetamide, *N,N*-dimethylformamide and dimethyl sulfoxide. The modified polymers were obtained in quantitative yields with inherent viscosities between 0.47 and 0.67 dl·g⁻¹. Experimental results indicated that all the polymers had glass transition temperature between 198 °C and 262 °C, the decomposition temperature at 10% weight loss between 398 °C and 531 °C under nitrogen.

Keywords: Poly(imide-ester)s; Poly(ether-imide-ester)s; Thermal properties; Solubility; Pendent groups; Structure-property relation.

INTRODUCTION

Due to the increasing demands for high-performance polymers as a replacement for ceramics or metals in the microelectronic, aerospace and automotive industries, thermally stable polymers have received much interest over the past decade. Aromatic polyimides are certainly one of the most useful classes of high-performance polymers, which have found many applications in industries because of their outstanding properties, such as excellent thermal and thermo-oxidative stability, solvent resistance, favorable electrical insulating properties as well as superior mechanical characteristics^[1–4]. Despite their widespread use, polyimides are generally intractable and lack the properties essential for successful fabrication into useful forms because of their high melting or glass-transition temperatures (*T_g*'s) and their limited solubility in organic solvents. Therefore, incorporating new functionalities to make polyimides more tractable without deteriorating their own excellent properties has become one important target of the investigators^[5–7]. One of the important modification for making processable polyimides is the preparation of copolyimides^[8–10] instead of homopolyimides, as well as the introduction of bulky, packing-disruptive groups which hinder the chain packing but do not markedly affect the *T_g*^[11–15], and flexible segments into the polymer chain which provide kinks between the rigid phenyl rings in the

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backbone^[16–24], replacing symmetrical aromatic rings by unsymmetrical ones^[25–30], and incorporation of the nonlinear moieties such as a bulky noncoplanar group in the polymer backbone^[31–33]. Combining these structural modifications minimizes the tradeoff between the processability and thermal stability of aromatic polyimides.

In previous work, we have reported the synthesis and characterization of aromatic copolyimides, such as poly(amide-imide)s^[34, 35], poly(ether-imide)s^[36], poly(ether-imide-amide)s^[37], and poly(imide-urea)s^[38] from a number of new imide-ring-containing dicarboxylic acids. From our previous studies, it was found that the incorporation of heterocyclic rings such as benzoxazole or benzothiazole as a bulky pendent group into rigid polymer backbone could substantially increase their solubility in various organic solvents as well as the thermal stability of polymers. It is important to note that polybenzoxazoles and polybenzothiazoles are recognized as a class of heterocyclic polymers with superior mechanical and thermal properties. Therefore, it can be expected that the resulting polymers with benzoxazole or benzothiazole pendent groups can provide a good balance of useful properties. Consequently, the aim of the present work was the preparation and evaluation of properties of two series of modified PIEs and PEIEs from imide-ring-containing dicarboxylic acids having benzoxazole or benzothiazole pendent groups with various aromatic dihydroxy compounds by diphenylchlorophosphate (DPCP)-activated direct polyesterification and compare their properties such as solubility and thermal stability with each other.

EXPERIMENTAL

Materials

Diphenyl chlorophosphate (DPCP) was purchased from Merck and used as received. Pyridine (Py, Merck) was purified by distillation under reduced pressure over calcium hydride and stored over 4-Å molecular sieves. 1,4-Dihydroxybenzene (a) (hydroquinone, Merck) and 4,4'-dihydroxybiphenyl (b, Fluka) were purified by recrystallization from ethanol. Bisphenol A (c, Merck) was purified by recrystallization from acetic acid. Commercially obtained anhydrous lithium chloride was dried under vacuum at 150 °C for 12 h. All other solvents were obtained from various commercial sources and used without further purification.

Techniques

¹H-NMR and ¹³C-NMR spectra were recorded on a Bruker AV-500 FT-NMR spectrometer in DMSO-*d*₆ at 25 °C with frequencies of 500.13 and 125.77 MHz for the ¹H and ¹³C spectra, respectively. FTIR spectra were recorded on a Bruker Tensor-27 spectrometer for the measurement of infrared absorption spectra of monomers and polymers. The spectra of solids were obtained using KBr pellets. Thermogravimetric analyses (TGA) were conducted with a Du Pont 2000 thermal analysis instrument under nitrogen atmosphere (20 cm³/min) at a heating rate of 10 K/min. Differential scanning calorimetry (DSC) was recorded on a Perkin Elmer pyris 6 DSC under nitrogen atmosphere (20 cm³/min) at a heating rate of 10 K/min. Inherent viscosities (η_{inh}) of polymers were determined for solution of 0.5 g/dL in *m*-cresol at 30 °C using a Canon-Fenske viscometer.

Synthesis of the Monomer

Two bis(imide-carboxylic acid) monomers (**1**), such as 2-[3,5-bis(*N*-trimellitimidoyl)phenyl]benzoxazole (**1_O**) and 2-[3,5-bis(*N*-trimellitimidoyl)phenyl]benzothiazole (**1_S**) and two bis(imide-ether-carboxylic acid) monomers (**2**), such as 2-[3,5-bis(4-trimellitimidophenoxy)-phenyl]benzoxazole (**2_O**) and 2-[3,5-bis(4-trimellitimidophenoxy)-phenyl]benzothiazole (**2_S**) were synthesized by the condensation of 5-(2-benzoxazole)-1,3-phenylenediamine, 5-(2-benzothiazole)-1,3-phenylenediamine, 5-(2-benzoxazole)-1,3-bis(4-aminophenoxy)benzene, and 5-(2-benzothiazole)-1,3-bis(4-aminophenoxy)benzene with two mole equivalents of trimellitic anhydride in glacial acetic acid, respectively, according to the procedure reported previously^[34, 37].

Synthesis of the Model Compound (M2_O)

In a three-necked round-bottomed flask equipped with a reflux condenser and a nitrogen gas inlet tube, a suspension consisting of bis(imide-ether-carboxylic acid) **2_O** (0.946 g, 1.25 mmol) and phenol (0.235 g, 2.5 mmol) in pyridine (2.5 mL) was heated at 120 °C for 5 min under nitrogen with stirring. A solution of DPCP

(0.09 g), lithium chloride (0.10 g), and Py (5 mL) aged at room temperature for 45 min was added dropwise through a dropping funnel over a period of 15 min. The heating was continued at 120 °C for 3 h and the reaction solution was trickled into methanol (100 mL), giving rise to a pale yellow precipitate, which was washed thoroughly with methanol and hot water, filtered off, and dried under reduced pressure at 80 °C for 5 h to afford 1.056 g (93%) of model compound **M2_O** (M.P. > 300 °C). Elemental analysis: calculated for C₅₅H₃₁N₃O₁₁ (**M2_O**), C 72.60%, H 3.43%, N 4.62%; found, C 72.41%, H 3.47%, N 4.69%.

Synthesis of Polymer

The general procedure to prepare the poly(imide-ester)s and poly(ether-imide-ester)s is as follows:

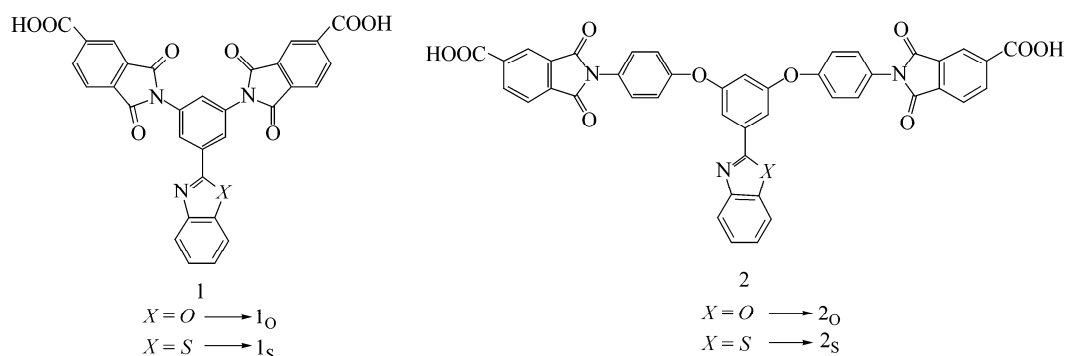
A solution of DPCP (0.17 g), lithium chloride (0.2 g), and Py (10 mL) was stirred under nitrogen atmosphere at room temperature for 45 min and then added dropwise through a dropping funnel over a period of 30 min to a solution containing dicarboxylic acid **1_O** (1.433 g, 2.5 mmol) and hydroquinone **a** (0.275 g, 2.5 mmol) in Py (5.0 mL) at the reflux temperature while stirring. The final solution was stirred and heated at 120 °C for 3 h under nitrogen. The polymer solution obtained was trickled into methanol (200 mL), giving rise to a stringy precipitate, which was washed thoroughly with methanol and hot water, collected by filtration, and dried at 80 °C under reduced pressure. The inherent viscosity of the polymer, **3_{Oa}**, was 0.47 dL/g. Elemental analysis: calculated for C₃₇H₁₇N₃O₉ (**3_{Oa}**), C 68.41%, H 2.95%, N 6.47%; found, C 68.28%, H 2.92%, N 6.19%.

Other poly(imide-ester)s, **3_Ob**, **3_Oc** and **3_{Sa}**–**3_{Sc}** and poly(ether-imide-ester)s **4_{Oa}**–**4_Oc** and **4_{Sa}**–**4_{Sc}** were synthesized in an analogous manner. Inherent viscosities of the polymers were measured in *m*-cresol at a concentration of 0.5 g/dL at 30 °C. Furthermore, the obtained FTIR and NMR spectroscopic data will be discussed in the results and discussion section.

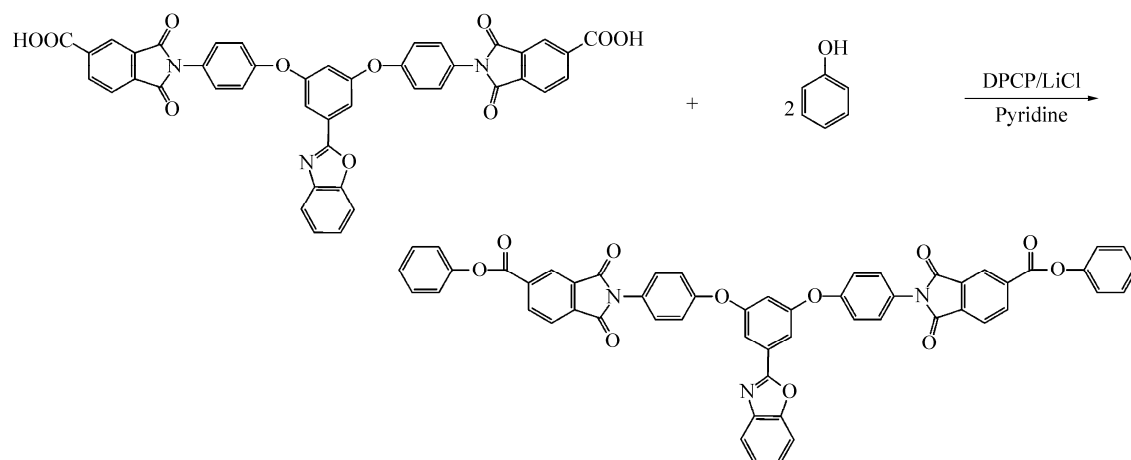
RESULTS AND DISCUSSION

Synthesis and Characterization

Four modified dicarboxylic acid monomers (Scheme 1) such as **1_O**, **1_S**, **2_O**, and **2_S** holding benzoxazole and benzothiazole moieties have been synthesized according to a modified procedure described in the literatures^[34, 37]. They were prepared by the condensation of the appropriate diamine with two mole equivalents of trimellitic anhydride in refluxing glacial acetic acid. The condensation reaction between the amines and anhydride groups, as well as the subsequent cyclodehydration reaction was carried out in the heterogeneous solution. In order to obtain information about the structures and the best preparation procedure for the final polymers, a model, **M2_O**, was synthesized by using a direct condensation method in the presence of diphenyl chlorophosphate (DPCP) with pyridine (Py) as condensing agent, as shown in Scheme 2.



Scheme 1 Structure of bis(imide-carboxylic acid) monomers **1_O** and **1_S** and bis(imide-ether-carboxylic acid) monomers **2_O** and **2_S**



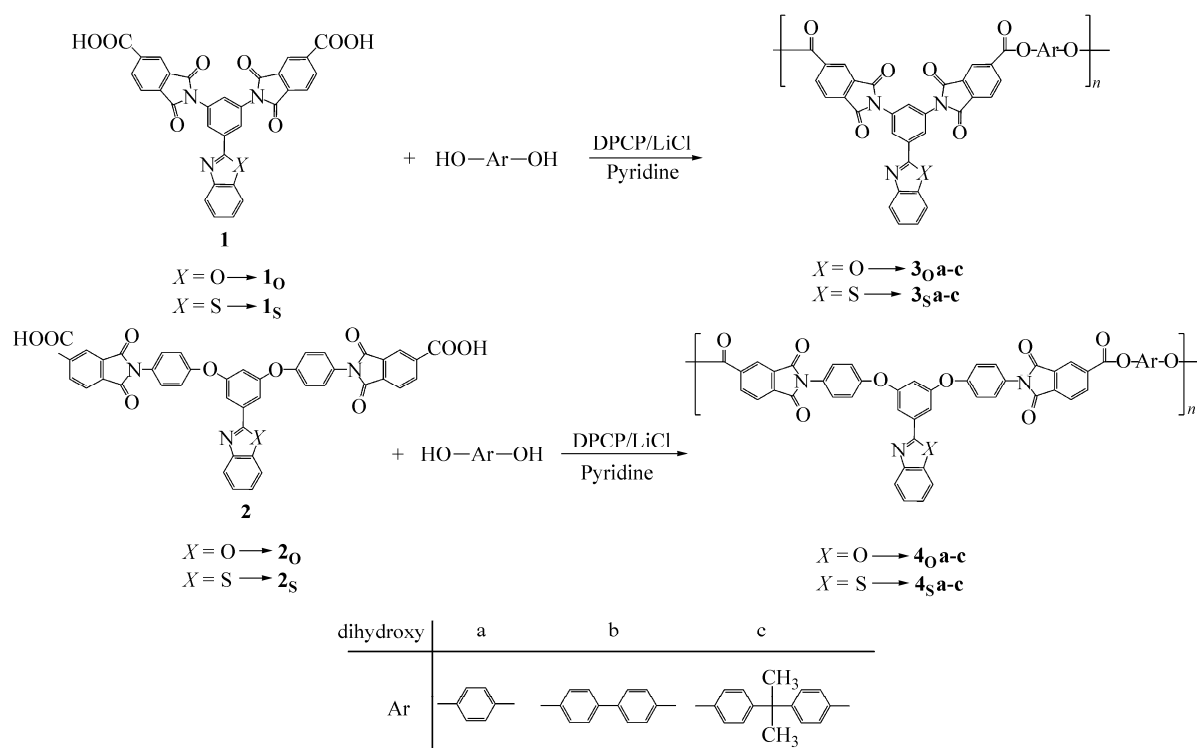
Scheme 2 Synthetic route used for the model compound **M2_O**

Chemical structures of the new model compound was confirmed by elemental analysis as well as FTIR, ^1H -NMR and ^{13}C -NMR spectroscopy. Elemental analysis values of C, H, and N of all the compounds are close to theoretical values as described in earlier section. The FTIR spectrum of the model compound **M2_O** exhibited characteristic absorption bands for the imide rings at 1775 and 1720 cm^{-1} , peculiar to the symmetrical and asymmetrical C—O stretching vibrations, respectively. Other absorption bands related to imide rings appeared at 1378 (C—N stretching vibration) 1099, and 724 cm^{-1} (imide ring deformation). Bands of ester functional groups appeared at about 1235 (C—O—C) and 1720 cm^{-1} (C=O), which was overlapped with the asymmetrical C=O stretching vibration of the imide rings. The ^1H -NMR spectrum of the model compound **M2_O** showed a variety of condensed sharp peaks in the region $\delta = 6.9$ –8.4 due to the aromatic protons with expected multiples and integration; the protons of the trimellitoyl groups revealed signals at downfield regions (*ca.* $\delta = 8$). The ^{13}C -NMR spectrum of model **M2_O**, with 27 different peaks, shows aromatic carbons at $\delta = 110$ –166 with expected multiples. Carbonyl carbons of ester and imide groups resonate in the downfield region above $\delta = 160$.

Four series of novel alternating aromatic poly(imide-ester)s, **3_{0a}–3_{0c}** and **3_{sa}–3_{sc}** and poly(ether-imide-ester)s **4_{0a}–4_{0c}** and **4_{sa}–4_{sc}**, containing benzoxazole and benzothiazole units were synthesized starting from dicarboxylic acid monomers **1₀**, **1_s**, **2₀**, and **2_s** and various aromatic dihydroxy compounds using the conditions applied to prepare model compound **M2_O** (Scheme 3).

The polycondensation proceeded gradually and gave the corresponding polymers in high yield up to 81%. Some characteristics of the resulting polymers including inherent viscosities measured in *m*-cresol at a concentration of 0.5 dL/g at 30 °C and yields of the resulting polymers are tabulated in Table 1.

The synthesized polymers were characterized by their FTIR and ^1H -NMR spectra. FTIR spectra of the polymers showed characteristic absorptions for the imide ring at around 1780 and 1720 cm^{-1} (imide-I), which are indicative of the asymmetrical and symmetrical C=O stretching vibration, and at 1370 (imide-II), 1090 (imide-III) and 725 cm^{-1} (imide-IV) whereas the imide-I, -III, -IV bands were assigned to axial, transverse, and out-of-plane vibrations of the cyclic imide structure, respectively. The absorption of ester carbonyl groups appeared at 1720 cm^{-1} , which was overlapped with the symmetrical C=O stretching vibration of the imide rings, and the C=N stretching band of benzoxazole and benzothiazole groups at 1612–1630 cm^{-1} and C—O stretching bands at 1230 cm^{-1} could also be observed. Figure 1 shows typical FTIR (A) and ^1H -NMR (B) spectra of **4_{sa}**. On the basis of the description above, we can conclude that the polymers have the expected structures.

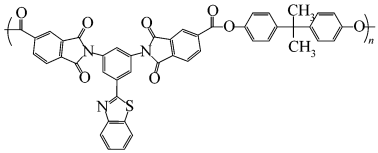
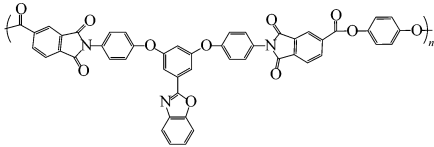
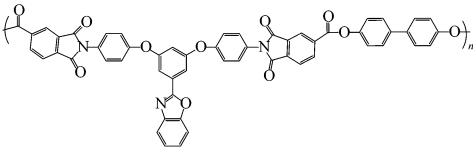
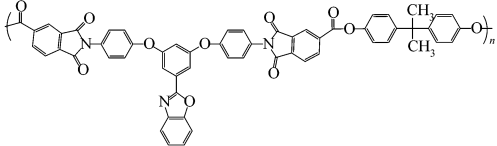
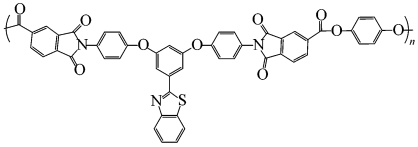
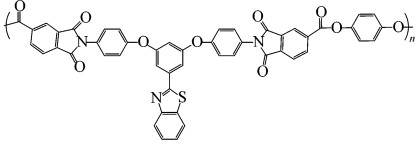
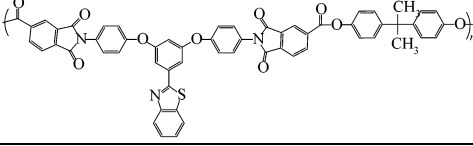


Scheme 3 Synthesis of poly(imide-ester)s and poly(ether-imide-ester)s

Table 1. Structures, yields and inherent viscosities of polymers

Polymer code	Polymer structure	Yield (%)	η_{inh}^a (dL/g)
3_{Oa}		88	0.47
3_{Ob}		92	0.51
3_{Oc}		81	0.57
3_{Sa}		91	0.61
3_{Sb}		90	0.48

Table 1. Continued

Polymer code	Polymer structure	Yield (%)	η_{inh}^a (dL/g)
3 _{sc}		84	0.45
4 _{0a}		89	0.52
4 _{0b}		90	0.58
4 _{0c}		87	0.65
4 _{sa}		94	0.67
4 _{sb}		91	0.54
4 _{sc}		91	0.53

^a Measured at a polymer concentration of 0.5 g/dL in *m*-cresol at 30 °C

Properties

The organosolubility behavior of all polymers was tested qualitatively in various organic solvents, with the results summarized in Table 2. One of the aims of the present investigation was the enhancement of the polymer solubility by introducing voluminous pendent groups along the polymer backbone. Almost all of the polymers were soluble in polar aprotic solvents such as DMF, NMP, DMAc and DMSO at room temperature or upon heating. They also showed good solubilities in *m*-cresol, pyridine, and tetrahydrofuran. Modified poly(imide-ester)s (PIEs) and poly(ether-imide-ester)s (PEIEs) displayed almost identical solubility in these solvents. In general, high solubility of these PIEs and PEIEs in weak polar solvents and high solubility of poly(amide-imide)s and poly(ether-imide-amide)s with the same structure, reported previously^[34, 37], in highly polar solvents can be explained that the strong solvation of the ester linkages by Py, THF, and *m*-cresol causes the better solubility of PIEs and PEIEs in less polar solvents, whereas the strong solvation of the amide and imide groups by polar aprotic solvents improved the solubility of poly(amide-imide)s and poly(ether-imide-amide)s in NMP, DMAc, DMF, and DMSO. Moreover, the good solubility of these polymers can be attributed to the presence of bulky benzoxazole and benzothiazole groups in the former, which increased the disorder in the chains and hindered dense chain packing, thus, reducing the interchain interactions to enhance the solubility. Furthermore,

the size of the kink in the dihydroxy unit would also (to some extent) play a role in affecting the solubility. For example, the relatively poor solubility of polymers **3ob** and **3sb** in some solvents is mainly the result of the presence of a rigid biphenyl, **b**, part where there is no kink to join the two aromatic rings.

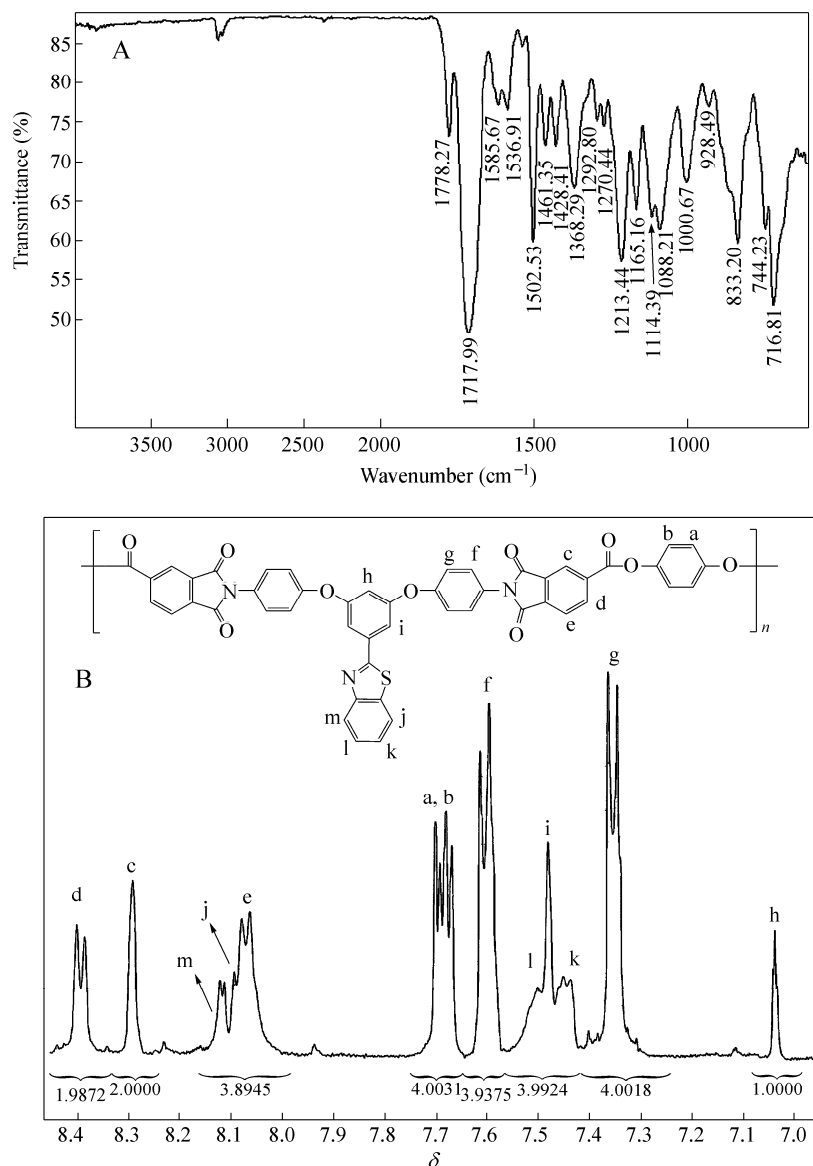


Fig. 1 FTIR (A) and ^1H -NMR (B) spectra of **4sa**

The thermal properties of both poly(imide-ester)s (PIEs) and poly(ether-imide-ester)s (PEIEs) were evaluated by means of differential scanning calorimetry (DSC) as well as thermogravimetric analysis (TGA/DTG) in a nitrogen atmosphere at a heating rate of 10 K/min. The glass-transition temperatures (T_g) and the temperature at 10 % weight loss (T_{10}) in nitrogen as well as the anaerobic char yield at 800 °C for all the polymers are listed in Table 3. Neither crystallization exotherms nor melting endotherms were observed by DSC, in the range 50–400 °C and only the inflection associated with the glass transition could be detected. The T_g s of the poly(imide-ester)s (PIEs) were 228–262 °C, which exhibited slightly higher T_g s than those of poly(ether-imide-ester)s (PEIEs), T_g s of which were 198–214 °C, respectively. This can be attributed to the presence of

flexible ether linkages in the main chains of PEIEs. Also the nature of the dihydroxy component had a significant influence on the T_g values. These went down in the series as follows **b** > **a** > **c**. In each series of polymers, the polymers derived from dihydroxy **b** exhibited the highest T_g due to the rigid biphenyl unit; and polymers obtained from dihydroxy **c** showed a lower T_g because of the presence of a flexible isopropylidene linkage as a kink unit in the polymer backbone. The T_g values of the synthesized polymers were also compared with those of the corresponding previously-prepared poly(amide-imide)s^[34] and poly(ether-imide-amide)s^[37]. It is revealed that the previous polymers showed a remarkable increase in the T_g values compared with the present polymers with the same structure. This could be attributed to the incorporation of flexible ester linkages along the polymers backbone, while the presence of amide groups with H-bond formation ability causes a restriction in free rotation of the macromolecular chains leading to enhanced T_g values.

Table 2. Solubility of the poly(imide-ester)s and poly(ether-imide-ester)s

Polymer code	Solvent ^a						
	DMAc	DMSO	DMF	NMP	<i>m</i> -Cresol	THF	Py
3_{0a}	+	—	+	—	+	+	++
3_{0b}	—	—	—	—	+	+	+
3_{0c}	++	+	++	+	++	++	++
3_sa	+	—	+	+	++	+	+
3_sb	—	—	+	—	+	+	+
3_sc	++	+	++	++	++	+	++
4_{0a}	+	+	++	++	++	+	++
4_{0b}	+	+	+	+	++	+	++
4_{0c}	++	+	++	++	++	+	++
4_sa	+	+	++	+	++	++	++
4_sb	+	+	+	+	++	+	+
4_sc	++	++	++	++	++	++	++

^aQualitative solubility was determined by dissolving 10 mg of poly(ether-imide)s in 1 mL of solvent at room temperature or upon heating;

++ : soluble at room temperature; + : soluble on heating; and — : insoluble even on heating; DMAc: *N,N*-dimethylacetamide; DMSO: dimethyl sulfoxide; DMF: *N,N*-dimethylformamide; NMP: *N*-methyl pyrrolidone; THF: tetrahydrofuran; Py: pyridine.

Table 3. Thermal properties of poly(imide-ester)s and poly(ether-imide-ester)s

Polymer code	T_g^a (°C)	T_{d10}^b (°C)	Char yield ^c (wt%)
3_{0a}	251	515	44
3_{0b}	262	531	48
3_{0c}	236	489	37
3_sa	239	496	38
3_sb	247	491	47
3_sc	228	463	31
4_{0a}	208	425	36
4_{0b}	214	452	33
4_{0c}	203	404	26
4_sa	202	416	32
4_sb	210	439	30
4_sc	198	398	28

^aFrom the second heating traces of DSC measurements with a heating rate of 10 K/min in nitrogen

^bTemperature at which 10% weight loss was recorded by TGA at a heating rate of 10 K/min in nitrogen

^cChar yield at 800 °C

The thermal stability was investigated by TGA with the T_{d10} values (temperatures which correspond to a 10% weight loss) as a criterion of thermal stability. All thermograms of the polymers obtained were almost similar with each other. In fact, all of them showed a two-stage thermal decomposition pattern in their thermograms (two maximas in the DTG curve). Assuming a different stability of the ester and imide units, the temperature maximum decomposition of the first stage is at around 465–520 °C, which might be attributed to the

early degradation of the ester linkages. Therefore, the 10% weight loss temperatures are mainly caused by the decomposition of ester units. The second-stage decomposition, started around 540–590 °C, is due to the decomposition of the imide linkages. The residual weights for all copolymers at 800 °C were above 26% under nitrogen atmosphere. Upon comparing the chemical structures of the pendent groups, it is seen that the polymers based on benzoxazole units showed somewhat more thermal stability than those of the polymers based on benzothiazole units with the same structure. This slightly higher thermal stability may be attributed to the greater ratio of thermally stable and polarity in benzoxazole ring-based macromolecular chains than those of benzothiazole ring-based polymers. For example, the TGA/DTG thermogram of **3Oc** is presented in Fig. 2.

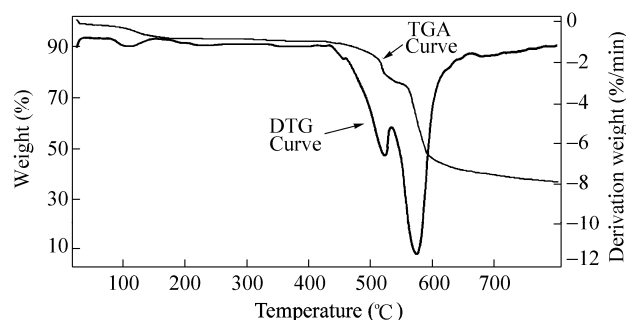


Fig. 2 TGA/DTG thermograms of **3Oc**

CONCLUSIONS

In conclusion, this work demonstrated that PIEs and PEIEs can be successfully prepared by DPCP-activated strategy with various aromatic dihydroxy compounds in the presence of pyridine and lithium chloride. The combination of flexible ether and ester linkages and also bulky heat-resistant benzoxazole or benzothiazole structure incorporated as pendent groups along with the polymer backbone remarkably endow the current polymers with some special characteristics, such as good solubility in common organic solvents and good thermal stability. Of course, thermal stabilities of PIEs exhibited slightly higher than those of PEIEs. In general, four modified dicarboxylic acid monomers such as **1o**, **1s**, **2o**, and **2s** can be considered as candidates for the synthesis of soluble and high-temperature-resistant poly(imide-ester)s and poly(ether-imide-ester)s.

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