

# Synthesis and properties of new fluorinated polyarylates derived from 1,1-bis(4-hydroxyphenyl)-1-phenyl-2,2,2-trifluoroethane and aromatic diacid chlorides

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## Abstract

A new series of fluorine-containing polyarylates were synthesized by interfacial or high-temperature solution polymerization of 1,1-bis(4-hydroxyphenyl)-1-phenyl-2,2,2-trifluoroethane with six aromatic diacyl chlorides. These polyarylates had inherent viscosities ranging from 0.47 to 1.37 dl/g that corresponded to weight-average and number-average molecular weights (by gel permeation chromatography) of 35,800–72,400 and 30,700–67,700, respectively. All polymers were highly soluble in a variety of solvents, and could afford tough, transparent, and colorless films via solution casting. The glass-transition temperatures of the polymers ranged from 209 to 271 °C. All of them did not show significant decomposition below 450 °C in both nitrogen and air atmospheres.

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## 1. Introduction

Polyarylates are aromatic polyesters consisting of aromatic diols and aromatic dicarboxylic acids [1]. Numerous efforts have been made to clarify the structure–property relationships in polyarylates [2,3], and some polyarylates are commercially available as high-performance engineering plastics [4]. The primary commercial polyarylates available today are the copolyesters based on 2,2-bis(4-hydroxyphenyl)propane (bisphenol A) and a mixture of isophthalic and terephthalic acids, that have a glass-transition temperature ( $T_g$ ) around 190 °C. There is still a need for the polyarylates having high  $T_g$ 's while retaining good solubility in organic solvents, thereby keeping the feasible processing. One of the approaches to obtain the polymer of this type is by the

introduction of a bulky pendent group along the polymer backbone [5,6]. In our previous publications we demonstrated that triphenyltrifluoroethane units-containing aromatic polyamides and polyimides exhibited moderately high  $T_g$ 's and enhanced thermal stability and solubility [7,8]. Therefore, the triphenyltrifluoroethane unit might be a promising segment for the preparation of polyarylates having high  $T_g$  and good solubility. This article deals with the preparation and basic characterization of a new class of fluorinated polyarylates from the polycondensation reactions of 1,1-bis(4-hydroxyphenyl)-1-phenyl-2,2,2-trifluoroethane with various aromatic diacid chlorides.

## 2. Experimental

### 2.1. Materials

$\alpha, \alpha, \alpha$ -Trifluoroacetophenone (Acros) and phenol (Wako) were used as received. Terephthaloyl chloride

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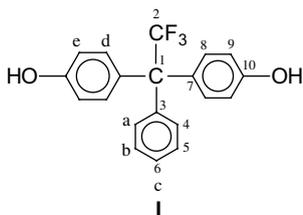
E-mail address: [shhsiao@ttu.edu.tw](mailto:shhsiao@ttu.edu.tw) (S.-H. Hsiao).

(**II<sub>a</sub>**) (from Wako) and isophthaloyl chloride (**II<sub>b</sub>**) (Fluka) were purified by vacuum sublimation. The other diacyl chloride monomers including 4,4'-biphenyldicarbonyl chloride (**II<sub>c</sub>**) (mp = 186 °C), 1,4-naphthalenedicarbonyl chloride (**II<sub>d</sub>**) (mp = 99 °C), 4,4'-oxydibenzoyl chloride (**II<sub>e</sub>**) (mp = 90 °C), and 4,4'-sulfonyldibenzoyl chloride (**II<sub>f</sub>**) (mp = 162 °C) were prepared by the chlorination of the corresponding commercially available dicarboxylic acids with thionyl chloride (SOCl<sub>2</sub>) in the presence of a trace amount of *N,N*-dimethylformamide. Benzyltriethylammonium chloride (BTEAC) (from TCI) used as the phase transfer catalyst in the interfacial polymerizations was used as received.

## 2.2. Synthesis of 1,1-bis(4-hydroxyphenyl)-1-phenyl-2,2,2-trifluoroethane (**I**)

In a three-neck, 300-ml round-bottom flask equipped with a stirring bar, gas inlet and drying tube, 25 g (0.14 mol) of  $\alpha, \alpha, \alpha$ -trifluoroacetophenone, 40 g (0.42 mol) of phenol, and 1 g of zinc chloride (ZnCl<sub>2</sub>) were added. The flask was immersed in a temperature controlled silicone oil bath at 100 °C. Via the gas inlet, dry hydrogen chloride (HCl) gas was bubbled into the reaction mixture. The reaction mixture gradually became viscous and formed a white solid at last. The crude product was washed repeatedly by boiling water and recrystallized from methanol/water (100 ml/100 ml) to afford 41.4 g (84%) of off-white, pure bisphenol **I** with a melting point of 234–235 °C (by DSC at a heating rate of 2 °C/min) (lit.[9] 231–232 °C).

IR (KBr): 3300–3600 (O–H), 1200–1260 (C–O), 1147 (C–F) cm<sup>-1</sup>. <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>,  $\delta$  ppm): 9.30 (s, 2H, –OH), 7.27 (d, 3H, H<sub>b</sub> + H<sub>c</sub>), 7.13 (d, 2H, H<sub>a</sub>), 6.90 (d, 4H, H<sub>d</sub>), 6.75 (d, 4H, H<sub>e</sub>). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>,  $\delta$  ppm): 158.60 (C<sup>10</sup>), 142.55 (C<sup>3</sup>), 132.43 (C<sup>8</sup>), 132.17 (C<sup>7</sup>), 131.20 (C<sup>4</sup>), 129.78 (q, <sup>1</sup>J<sub>C-F</sub> = 280 Hz, C<sup>2</sup>), 129.45 (C<sup>5</sup>), 128.95 (C<sup>6</sup>), 116.27 (C<sup>9</sup>), 64.37 (q, <sup>2</sup>J<sub>C-F</sub> = 23 Hz, C<sup>1</sup>). Elem. Anal. Calcd. for C<sub>20</sub>H<sub>15</sub>F<sub>3</sub>O<sub>2</sub> (344.33): C, 69.76%; H, 4.39%. Found: C, 69.54%; H, 4.21%.



## 2.3. Polymer synthesis

### 2.3.1. Interfacial polycondensation

A typical procedure for the synthesis of polyarylate **III<sub>e</sub>** is given as follows. A solution of 4,4'-oxydibenzoyl

chloride (**II<sub>e</sub>**) (0.5843 g; 2 mmol) in dichloromethane (6 ml) was added all at once to a solution of 3F-bisphenol **I** (0.6886 g; 2 mmol) and BTEAC (30 mg) in 1 M aqueous sodium hydroxide (5 ml). The reaction mixture was stirred vigorously at room temperature for 1 h. The polymer mixture was poured into boiling water (about 300 ml) to remove dichloromethane. The precipitated polymer was collected by filtration, washed with hot water thoroughly, and dried. The inherent viscosity of polymer **III<sub>e</sub>** was 1.37 dl/g, measured at a concentration of 0.5 g/dl in 1,1,2,2-tetrachloroethane (TCE)/phenol (40/60 by weight) at 30 °C.

Polyarylates **III<sub>a</sub>**, **III<sub>b</sub>**, **III<sub>d</sub>**, and **III<sub>f</sub>** were prepared from diol **I** with the corresponding diacid chlorides by a similar procedure described as above.

### 2.3.2. High-temperature solution polycondensation

Because diacid chloride **II<sub>c</sub>** was insoluble in dichloromethane, the polycondensation of **II<sub>c</sub>** and diol **I** was carried out in *o*-dichlorobenzene at an elevated temperature. A mixture of 0.5582 g (2 mmol) of **II<sub>c</sub>** and 0.6886 g (2 mmol) of 3F-bisphenol **I** in 10 ml of *o*-dichlorobenzene was heated to 200 °C and stirred at that temperature for 3 h. After cooling, the viscous solution was poured into 200 ml of methanol. The precipitated white fibrous polymer (**III<sub>c</sub>**) was washed thoroughly by methanol, collected by filtration, and dried. The inherent viscosity of polyarylate **III<sub>c</sub>** was 0.47 dl/g, measured at a concentration of 0.5 g/dl in TCE/phenol (40/60 by weight) at 30 °C.

## 2.4. Preparation of the polyarylate films

A polymer solution of approximately 10 wt% was made by the dissolution of about 1.0 g of polyarylate in 10 ml of hot TCE. The clear solution was poured into a 11-cm-diameter glass culture dish, which was placed in a 90 °C oven overnight for slow release of the solvent. Then the obtained semidried polymer film was stripped from the glass substrate and further dried in vacuum at 160 °C for 6 h. The obtained films were about 0.08 mm thick and used for X-ray diffraction measurements, tensile tests, solubility tests, and thermal analyses.

## 2.5. Measurements

Elemental analysis was made on a PerkinElmer 2400 CHN analyzer. Infrared (IR) spectra were recorded on a Horiba FT-720 Fourier transform infrared (FTIR) spectrometer. <sup>1</sup>H and <sup>13</sup>C NMR spectra were measured on a JEOL EX 400 spectrometer. Wide-angle X-ray diffraction (WAXD) measurements were performed at room temperature ( $\approx$ 25 °C) on a Siemens Kristalloflex D5000 X-ray diffractometer (operating at 40 kV and 20 mA) with Ni-filtered CuK $\alpha$  radiation ( $\lambda$  = 1.5418 Å). The scanning rate was 2°/min over a range of

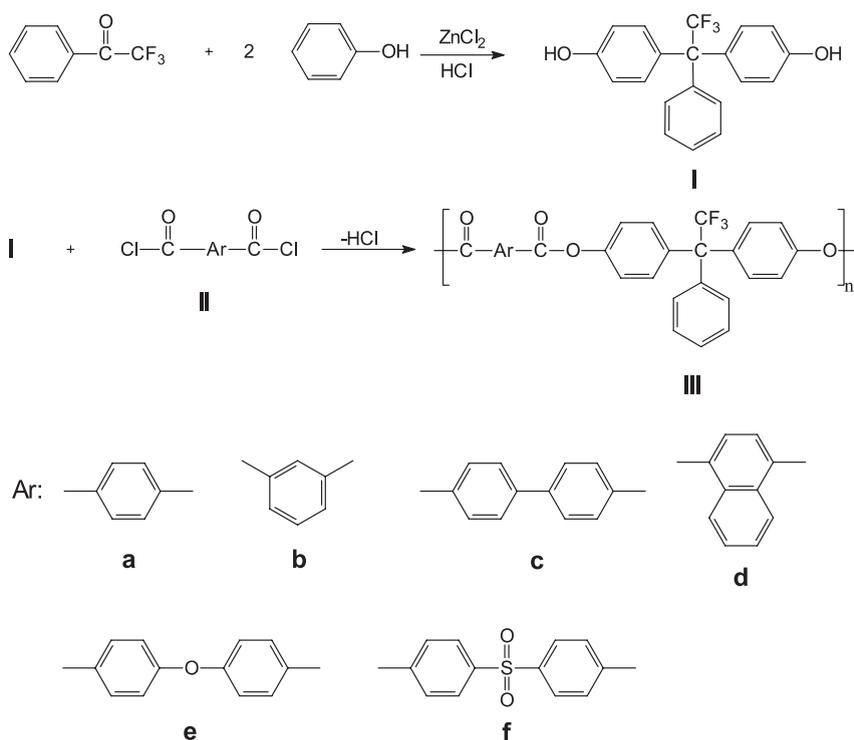
$2\theta = 5\text{--}45^\circ$ . The inherent viscosities of the polyarylates were determined with a Cannon-Fenske viscometer at  $30^\circ\text{C}$ . The molecular weight was determined by a Waters GPC system including a Waters 2410 refractive index detector using tetrahydrofuran as eluent and polystyrene standards. Differential scanning calorimetry (DSC) analyses were performed on a PerkinElmer Pyris 1 DSC at a heating rate of  $20^\circ\text{C}/\text{min}$  under nitrogen.  $T_g$ 's were read at the middle of the transition in the heat capacity and were taken from the second heating scan after quick cooling from  $400^\circ\text{C}$  at a cooling rate of  $200^\circ\text{C}/\text{min}$ . Thermomechanical analysis (TMA) was conducted with a Perkin Elmer TMA 7 instrument. The TMA experiments were conducted from  $40$  to  $300^\circ\text{C}$  at a scan rate of  $10^\circ\text{C}/\text{min}$  using a penetration probe  $1.0$  mm in diameter under an applied constant load of  $10$  mN. Softening temperatures ( $T_s$ 's) were taken as the onset temperature of probe displacement on the TMA traces. Thermogravimetric analysis (TGA) was conducted with a PerkinElmer Pyris 1 TGA. Experiments were carried out on approximately  $3\text{--}5$  mg of samples in flowing nitrogen or air (flow rate  $20\text{ cm}^3/\text{min}$ ) at a heating rate of  $20^\circ\text{C}/\text{min}$ . An Instron universal tester (model 4400R) with a load cell of  $5$  kg was used to study the stress-strain behavior of the polyarylate film samples. A gauge length of  $2$  cm and a crosshead speed of  $5$  mm/min were

used for this study. Measurements were performed at room temperature with film specimens ( $0.5$  cm wide,  $6$  cm long, and about  $0.08$  mm thick), and an average of at least three replicas was used.

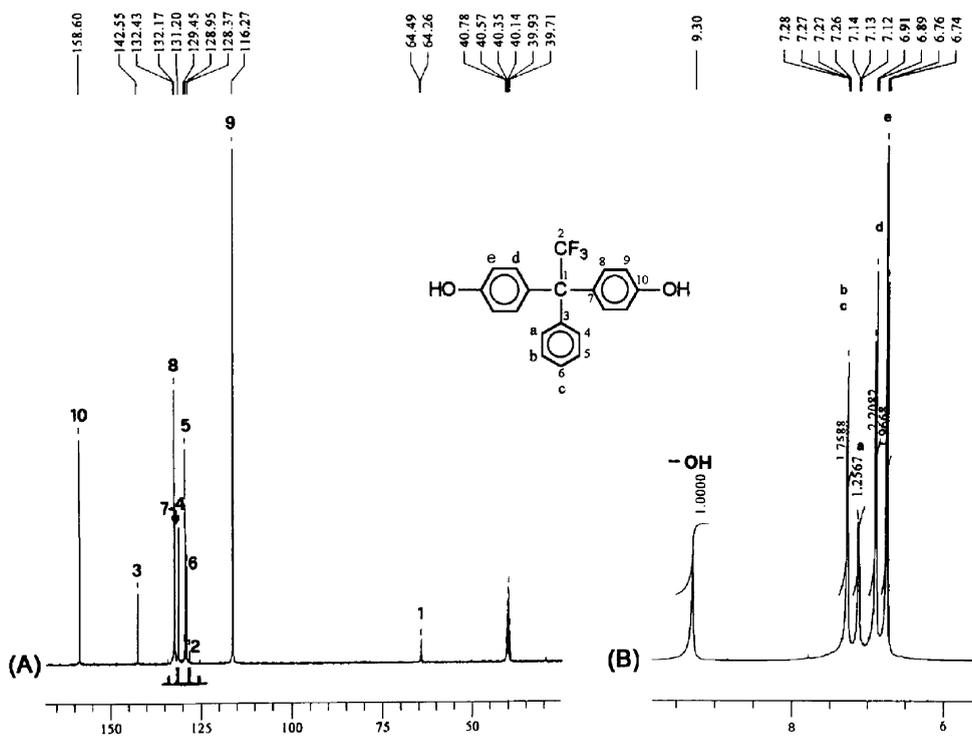
### 3. Results and discussion

#### 3.1. Polymer synthesis

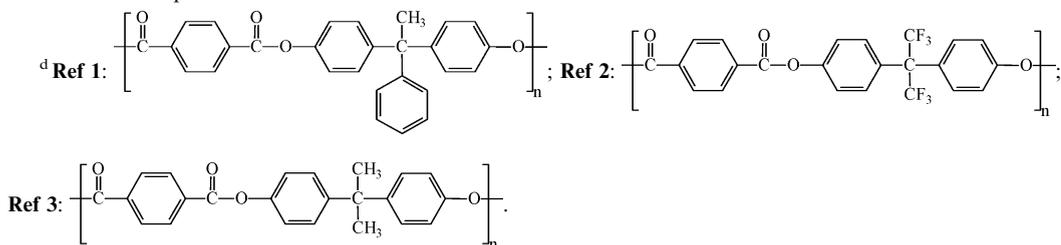
The fluorine-containing bisphenol **I** was obtained according to a well-established method [10] by the acid-catalyzed condensation of  $\alpha, \alpha, \alpha$ -trifluoroacetophenone and excess phenol (Scheme 1). The structure of fluorinated bisphenol **I** was confirmed by elemental analysis and IR and NMR spectroscopy. Characteristic IR absorption bands of hydroxyl O–H stretching and C–F stretching vibrations can be observed around  $3200$  and  $1150\text{ cm}^{-1}$ , respectively. Fig. 1 reproduces the  $^{13}\text{C}$  and  $^1\text{H}$  NMR spectra of **I**. The  $^{13}\text{C}$  NMR spectrum exhibited the expected absorption peaks for every carbon-13 atom of 3F-bisphenol **I**. The clear quartet centered at about  $129.8$  ppm was peculiar to the  $-\text{CF}_3$  carbons. The  $^1\text{H}$  NMR spectroscopic data of the aromatic protons of **I** are in the range of  $6.74\text{--}7.28$  ppm, and the resonance signal of hydroxyl protons is observed at  $9.30$  ppm.



Scheme 1.

Fig. 1. (A)  $^{13}\text{C}$  NMR and (B)  $^1\text{H}$  NMR spectra of bisphenol I in  $\text{DMSO}-d_6$ .Table 1  
Inherent viscosities, molecular weights, and tensile properties of polyarylates

| Polymer code       | $\eta_{\text{inh}}^{\text{a}}$ (dl/g) | Molecular weights <sup>b</sup> |        |       | Tensile properties of the polymer film <sup>c</sup> |                         |                       |
|--------------------|---------------------------------------|--------------------------------|--------|-------|---|-------------------------|-----------------------|
|                    |                                       | Mw                             | Mn     | Mw/Mn | Tensile strength (MPa)                              | Elongation to break (%) | Initial modulus (GPa) |
| IIIa               | 1.37                                  | 35,800                         | 22,900 | 1.56  | 68  | 14                      | 1.61                  |
| IIIb               | 1.20                                  | 64,300                         | 37,800 | 1.70  | 71  | 10                      | 1.52                  |
| IIIc               | 0.47                                  | 41,100                         | 26,500 | 1.57  | 67  | 18                      | 1.44                  |
| IIId               | 1.19                                  | 72,400                         | 50,500 | 1.43  | 66  | 8                       | 1.66                  |
| IIIe               | 1.33                                  | 51,600                         | 29,300 | 1.76  | 75  | 14                      | 1.82                  |
| IIIf               | 1.10                                  | 54,100                         | 33,700 | 1.60  | 66  | 11                      | 1.61                  |
| Ref 1 <sup>d</sup> | 1.16                                  | 51,600                         | 30,500 | 1.69  | 71  | 20                      | 1.68                  |
| Ref 2 <sup>d</sup> | 0.35                                  | —                              | —      | —     | —   | —                       | —                     |
| Ref 3 <sup>d</sup> | 0.26                                  | —                              | —      | —     | —   | —                       | —                     |

<sup>a</sup> Measured at a concentration of 0.5 g/dl in 1,1,2,2-tetrachloroethane/phenol (40:60 by weight) at 30 °C.<sup>b</sup> Relative to polystyrene standards, using THF as the eluent.<sup>c</sup> Films were cast from slow evaporation of polymer solutions in 1,1,2,2-tetrachloroethane. The cast films were dried under vacuum at 150 °C for 6 h prior to the tensile test.

According to a well-known technique [11,12], fluorine-containing polyarylates **III<sub>a-f</sub>** except **III<sub>c</sub>** were prepared from 3F-bisphenol **I** and various aromatic diacid chlorides **II<sub>a-f</sub>** by the two-phase polycondensation in a dichloromethane-aqueous sodium hydroxide solution system using BTEAC as the phase transfer catalyst (Scheme 1). Because 4,4'-biphenyldicarbonyl chloride (**II<sub>c</sub>**) is insoluble in dichloromethane, the polyarylate **III<sub>c</sub>** has been synthesized by a high-temperature solution polymerization reaction of bisphenol **I** and diacid chloride **II<sub>c</sub>** in refluxing *o*-dichlorobenzene. The inherent viscosities and average molecular weights of these polyarylates are reported in Table 1. These polymers exhibited inherent viscosities in the range of 0.47–1.37 dl/g. Their *M<sub>w</sub>*, *M<sub>n</sub>* and polydispersity index (*M<sub>w</sub>*/*M<sub>n</sub>*) determined by gel permeation chromatography were 35,800–72,400, 22,900–50,500, and 1.45–1.76, respectively. All polyarylates could be solution-cast into tough, transparent, and colorless films. The structural feature of the polyarylates was confirmed by IR spectroscopy. The characteristic absorptions of ester group appear around 1750 cm<sup>-1</sup> (C=O stretching) and in the region of 1200–1300 cm<sup>-1</sup> (C–O stretching). The strong absorption at about 1150 cm<sup>-1</sup> is due to the C–F stretching. For a comparative study, three referenced polyarylates derived from terephthaloyl chloride (**II<sub>a</sub>**) and three common bisphenols with different separator groups between benzene rings were also synthesized and characterized.

### 3.2. Polymer properties

The crystallinity of the prepared polyarylates was determined by WAXD analysis. The diffraction patterns of the representative polyarylates are presented in Fig. 2. Polyterephthalates **Ref 2** and **Ref 3** revealed some stronger diffraction signals, indicating a semicrystalline nature. However, polyarylates **III<sub>a</sub>** and **Ref 1** showed almost completely amorphous diffraction patterns. The other polyarylates of the **III** series also showed essentially amorphous patterns as those of **III<sub>a</sub>** and **Ref 1**. Apparently, the bulky, non-symmetric triphenyltrifluoroethane and triphenylethane cores interfere with the close chain packing, thus leading to amorphous polyarylates.

The organo-solubility of the polyarylates was determined qualitatively, and the results are listed in Table 2. These polyarylates were readily soluble at room temperature in strongly polar solvents, such as NMP, DMF, and DMSO, and several chlorinated solvents, but insoluble in acetone and methanol. Their high solubility could be attributed to the introduction of the bulky, packing-disrupting triphenyltrifluoroethane segments. The referenced polymers **Ref 2** and **Ref 3** revealed a relatively poor solubility because of their crystalline structures.

All the fluorinated polyarylates could afford flexible and creasable films. Their tensile properties are also re-

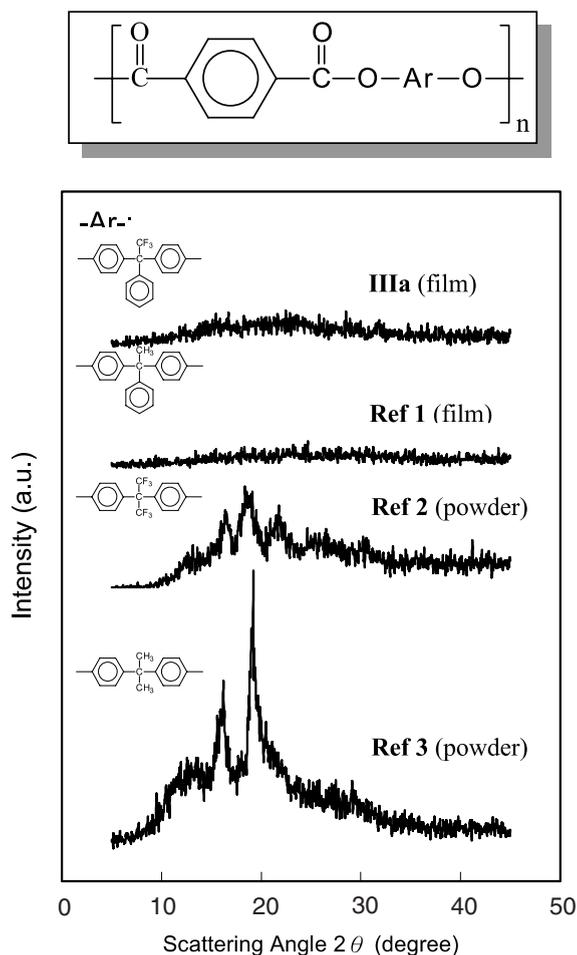


Fig. 2. WAXD patterns of **III<sub>a</sub>** and structurally related polyarylates.

ported in Table 1. These polyarylate films had tensile strengths of 66–75 MPa, elongations to break of 8–18%, and tensile moduli of 1.44–1.82 GPa. All the polymers behaved as ductile materials with good tensile strengths and moderate elongations to break.

DSC, TMA, and TGA were used to evaluate thermal properties of all polymers. The thermal behavior data of the polyarylates are summarized in Table 3. The semicrystalline polyarylates **Ref 2** and **Ref 3** exhibited an obvious melting endotherm around 314 and 356 °C, respectively, in the first DSC heating traces. However, all the **III<sub>a-f</sub>** and **Ref 1** did not show melting endotherms up to the decomposition temperatures on the DSC thermograms. This result supports the amorphous nature of these polyarylates. The *T<sub>g</sub>* values of polyarylates **III<sub>a-f</sub>** were recorded in the range of 209–271 °C. The *T<sub>s</sub>* values of the polyarylate film samples were also determined with TMA by the penetration method. The *T<sub>s</sub>* values of **III<sub>a-f</sub>** stayed in the 221–282 °C range. Polyarylate **III<sub>a</sub>** seems to reveal a slightly higher *T<sub>g</sub>* and *T<sub>s</sub>* value as

Table 2  
Solubility behavior of polyarylates<sup>a</sup>

| Polymer      | Solvent <sup>b</sup> |      |     |            |     |              |               |      |         |
|--------------|----------------------|------|-----|------------|-----|--------------|---------------|------|---------|
|              | NMP                  | DMSO | THF | Chloroform | TCE | <i>o</i> -CP | <i>o</i> -DCB | MeOH | Acetone |
| <b>IIIa</b>  | +                    | +    | +   | +          | +   | +            | +             | –    | –       |
| <b>IIIb</b>  | +                    | –    | +   | +          | +   | +            | +             | –    | –       |
| <b>IIIc</b>  | +                    | +    | +   | +          | +   | +            | +             | –    | –       |
| <b>IIId</b>  | +                    | +    | +   | +          | +   | +            | +             | –    | –       |
| <b>IIIe</b>  | +                    | –    | +   | +          | +   | +            | +             | –    | –       |
| <b>IIIf</b>  | +                    | +    | +   | +          | +   | +            | +             | –    | –       |
| <b>Ref 1</b> | +                    | +    | +   | +          | +   | +            | +             | –    | –       |
| <b>Ref 2</b> | –                    | –    | –   | –          | –   | –            | –             | –    | –       |
| <b>Ref 3</b> | –                    | –    | –   | –          | +   | +            | +             | –    | –       |

<sup>a</sup> Qualitative solubility was tested with 10 mg sample in 1 ml solvent. Symbol: (+) soluble at room temperature; (+h) soluble on heating; (–) insoluble even on heating.

<sup>b</sup> DMSO: dimethyl sulfoxide; THF: tetrahydrofuran; *o*-CP: *o*-chlorophenol; NMP: *N*-methyl-2-pyrrolidone; *o*-DCB: *o*-dichlorobenzene; TCE: 1,1,2,2-tetrachloroethane.

Table 3  
Thermal behavior data of polyarylates

| Polymer      | $T_g^a$ (°C) | $T_s^b$ (°C) | $T_{5\%wt\text{loss}}^c$ (°C) <sup>c</sup> |        | $T_{10\%wt\text{loss}}^c$ (°C) <sup>c</sup> |        | Char yield (wt%) <sup>d</sup> |
|--------------|--------------|--------------|--|--------|---|--------|-------------------------------|
|              |              |              | In N <sub>2</sub>                          | In air | In N <sub>2</sub>                           | In air |                               |
| <b>IIIa</b>  | 246          | 254          | 526  | 495    | 546   | 513    | 53                            |
| <b>IIIb</b>  | 209          | 221          | 529  | 480    | 549   | 497    | 52                            |
| <b>IIIc</b>  | 269          | 277          | 530  | 492    | 551   | 515    | 56                            |
| <b>IIId</b>  | 221          | 231          | 517  | 493    | 538   | 510    | 54                            |
| <b>IIIe</b>  | 216          | 239          | 507  | 480    | 526   | 496    | 51                            |
| <b>IIIf</b>  | 271          | 282          | 507  | 488    | 526   | 507    | 47                            |
| <b>Ref 1</b> | 244          | 248          | 516  | 472    | 533   | 493    | 41                            |
| <b>Ref 2</b> | 240 (314)    | –            | 505  | 487    | 527   | 509    | 43                            |
| <b>Ref 3</b> | 202 (356)    | –            | 501  | 440    | 519   | 472    | 29                            |

<sup>a</sup> The samples were heated from 50 to 400 °C at a scan rate of 20 °C/min; this was followed by rapid cooling to 50 °C at 200 °C/min in N<sub>2</sub>. The midpoint temperature of the baseline shift on the subsequent DSC trace (from 50 to 400 °C at 20 °C/min) was defined as  $T_g$ .

<sup>b</sup> Taken as the onset temperature of the probe displacement on the TMA trace. The film samples were heated at 300 °C for 30 min prior to the TMA test.

<sup>c</sup> Decomposition temperature recorded via TGA at a heating rate of 20 °C/min and a gas flow rate of 40 cm<sup>3</sup>/min.

<sup>d</sup> Residual weight percentage at 800 °C in nitrogen.

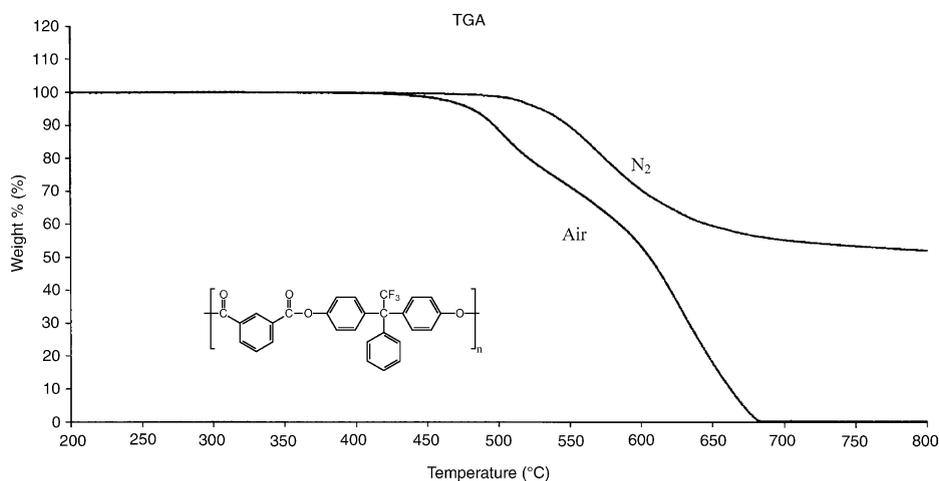


Fig. 3. TGA curves of polyarylate **IIIb**, at a heating rate of 20 °C/min.

compared to its non-fluorinated analog, **Ref 1**, probably due to the increase of structural hindrance caused by the bulkier  $-\text{CF}_3$  groups.

Thermal and thermo-oxidative stabilities of these polyarylates were evaluated by TGA under both nitrogen and air atmospheres using a 5 or 10 wt% loss temperature for comparison. Typical TGA curves in nitrogen and air atmospheres of the representative polymer **III<sub>b</sub>**, are shown in Fig. 3. The thermal and thermo-oxidative behavior data of the polyarylates are listed in Table 3. The decomposition temperatures ( $T_d$ ) of polyarylates at 10% weight loss were all above 500 °C. In nitrogen, the fluorinated polyarylate **III<sub>a</sub>** exhibited higher  $T_d$  values in comparison to its non-fluorinated counterpart (**Ref 1**), because of the higher C–F bond strength.

#### 4. Conclusions

A new class of fluorine-containing polyarylates (**III**) were synthesized from 1,1-bis(4-hydroxyphenyl)-1-phenyl-2,2,2-trifluoroethane and various aromatic diacid chlorides by the conventional techniques. All the polyarylates were readily soluble in polar solvents and could be cast into amorphous and tough films. These polyarylates also showed moderately high thermal stability. Thus, these polyarylates can be considered as candidates of high-performance polymeric materials.

#### Acknowledgements

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