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High Optical Transparency and Low Dielectric Constant of Organosoluble Poly(aryl ether amides) Based on 1, 2-Bis (4-amino-2-trifluoromethylphenoxy) benzene and Aromatic Dicarboxylic acids

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High Optical Transparency and Low Dielectric Constant of Organosoluble Poly(aryl ether amides) Based on 1, 2-Bis (4-amino-2-trifluoromethylphenoxy) benzene and Aromatic Dicarboxylic acids

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A series of novel fluorinated poly(aryl ether amide)s(PEAs) were prepared from an aromatic diamine containing trifluoromethyl group ($-CF_3$), 1,2-bis(4-amino-2-trifluoromethylphenoxy)benzene(1),with various aromatic dicarboxylic acids using the phosphorylation polycondensation. The fluorinated PEAs were obtained in almost quantitative yields with inherent viscosities ranging from 0.55 to 0.78 dl/g. All the PEAs showed high solubility in many polar organic solvents such as N-methyl-2-pyrrolidinone (NMP), dimethyl sulfoxide (DMSO) and tetrahydrofuran (THF) and so on. Flexible and tough polymer films could be easily prepared by casting from DMAc solvent. The PEAs films exhibited high optical transparency with a cutoff wavelength of 336–357 nm and good mechanical properties with tensile strengths of 78.4–104.8 MPa and elongations at break of 5.4–10.3%. They showed high glass transition temperatures in the range of 227–244°C. They also exhibited good thermal stability, the 10% wt loss temperatures from 453 to 464°C in nitrogen atmosphere. Meanwhile, the obtained PEAs films possessed low dielectric constants of 2.42–2.80 at 1 MHz and low moisture absorption (1.27–2.36%). Due to their properties, the polyamides could be considered as engineering microelectronic and photoelectric materials.

Keywords: Trifluoromethyl group, poly(aryl ether amide)s, solubility, dielectric constant

1 Introduction

Wholly aromatic polyamides (PAs) have been well known for their high temperature stability, excellent mechanical strength and good chemical resistance, which qualify them as high-performance polymeric materials (1–3). Fibers obtained from anisotropic solutions of these high-performance materials have been used in applications where high thermal stability and mechanical strength are required. Aromatic PAs like other aromatic and heterocyclic polymers are quite intractable materials because they are infusible and insoluble in organic solvents. Purely aromatic polyisophthalamides, for instance, are soluble only in a few aprotic polar solvents containing dissolved inorganic salts which are mainly caused by the highly regular and rigid polymer backbones and the formation of intermolecular hydrogen bonding. Moreover, the high moisture absorptions of aromatic PAs, compared with other polymer materials, have resulted in obvious negative effect on their mechanical properties as well as electrical insulating and dielectric performance. Consequently, many efforts have been made to improve their solubility and dielectric properties without much impairing their thermal stability and mechanical properties. The successful approaches employed with those purpose include: introducing flexible bonds (4,5), cardo groups (6,7), unsymmetrical (8–10) and alicyclic units (11) into the macromolecular backbone. These structural modifications could disturb regularity of chain packing, thus providing better solubility.

Recently, considerable attention has been devoted to the fluorinated aromatic polymers, including fluorinated polyimides (12–14), PAs (15–21), poly(aryl ether)s (22–24), etc. It was found that the incorporation of flexible fluorinated groups into polymer backbones resulted in great benefits for promoting polymer solubility, as well as electrical and dielectric performance, which attributed to the small dipole

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and the low polarizability of the C–F bond as well as the increase in free volume (25,26).

As reported in previous publications (27), the fluorinated polyimides prepared based on 1,2-bis (4-amino-2-trifluoromethylphenoxy) benzene displayed excellent performance, such as solubility, thermal, optical and dielectric property. Under the above consideration, this paper mainly reported the syntheses a series of novel PEAs containing trifluoromethyl groups. The solubility, optical, dielectric, thermal, and physical properties of the fluorinated PEAs were also investigated and discussed.

2 Experiment

2.1 Materials

The diamine, 1,2-bis (4-amino-2-trifluoromethylphenoxy) benzene (mp, 209–211°C) was synthesized according to the method reported in the preceding studies (27). The commercially available aromatic dicarboxylic acids such as terephthalic acid (**2a** or TPA; Aldrich), isophthalic acid (**2b** or IPA; Alfa), 4,4′-oxydibenzoic acid (**2c** or OBA; Alfa), and 2,2′-bis(4-carboxyphenyl)hexafluoropropane (**2d** or 6FBA; Chriskev) were used as received. Calcium chloride ($CaCl_2$) was dried under vacuum at 180°C for 4 h prior to use. NMP (Aladdin), pyridine (Py; Aladdin), triphenylphosphite (TPP; Aladdin) and other solvents were purified according to the standard methods.

2.2 Measurements

The ^{1}H-NMR spectra were measured on a Bruker AV400 instrument with dimethylsulfoxide-d_6 (DMSO-d_6) as a solvent and tetramethylsilane (TMS) as an internal standard. FT-IR spectra were recorded on a Nicolet Magna 470 spectrometer. Ultraviolet-visible (UV-Vis) spectra of the polymer films were recorded on a Lambda 35 (Perkin- Elmer) spectrophotometer at room temperature. Elemental analysis was carried out on a Carlo Erba1106 system. Differential scanning calorimetry (DSC) analysis was performed on a PE Diamond DSC instrument at a heating rate of 10°C/min in nitrogen atmosphere. Glass transition temperatures (T_g) were read at the middle of the transition in the heat capacity from the second heating scan. Thermogravimetric analysis (TGA) of the polymer samples was measured on a Netzsch TG 209F1 instrument at a heating rate of 20°C min^{-1} in nitrogen atmosphere, and $T_{10}$is reported as the temperatures where 10% weight losses was observed. Inherent viscosities ($\eta_{\text{inh}} = \ln\eta_r/c$) were measured at a concentration of 0.5 g/dL in DMAc at 25°C with an Ubbelohde viscometer. Mechanical properties of the films were measured with an AGS-500ND tensile tester at a crosshead speed of 5 mm/min. Measurements were performed at room temperature with film specimens (0.8 cm wide, 6 cm long, and about 0.05 mm thick), and an average of at least five individual specimens was used. Wide-angle X-ray diffraction (WAXD) patterns were obtained at room temperature on a Rigaku D/MAX 2500 powder diffractometer with a scanning speed of 4°/min, and the patterns were recorded in the 2θ range of 5–40°. Dielectric constants of the polymers were tested by the parallel-plate capacitor method using a HP4291B at a frequency region of 1 MHz–1.1 GHz. The equilibrium water uptake was determined by the weighing of the changes in vacuum-dried film specimens before and after immersion in deionized water at 25°C for 3 days.

2.3 Polymer Synthesis

A generally synthetic procedure for the poly(aryl ether amide)s is described as follows. A flask was charged with a mixture of 1.5 mmol diamine, 1.5 mmol aromatic dicarboxylic acids, 2.5 mL TPP, 1.0 mL pyridine, 5 mL NMP and 0.3 g calcium chloride. The reaction mixture was maintained at 110°C under nitrogen atmosphere for 4 h. As the polycondensation proceeded, the reaction mixture became gradually viscous. The resultant solution of the polymer was poured slowly into 300 ml of stirred methanol giving rise to a tough, fiber-like polymer precipitate. The product was collected by filtration, thoroughly washed by methanol and hot water, and dried.

2.3.1. *FT-IR, ^{1}H-NMR, and elemental analysis data of PEA*

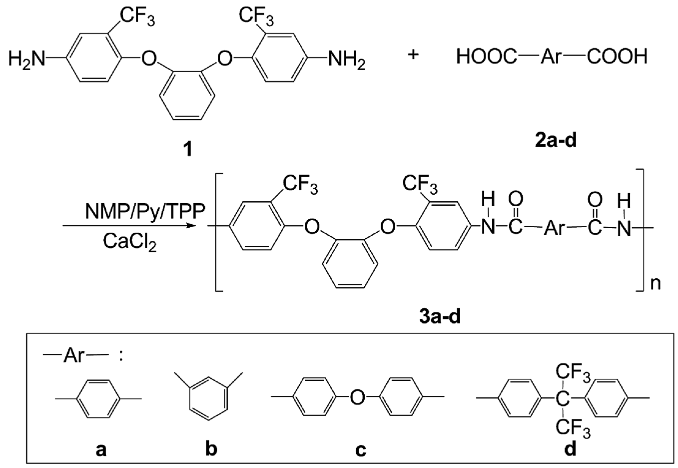
FT-IR (film): 3,295 (–NHCO–), 1,655 (C=O), 1,244 (C–O–C), 1,176, 1135 (–CF_3) cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d6, δ, ppm): 10.62 (s, 2H), 8.23 (s, 2H), 8.11 (m, 4H), 8.01 (d, $J = 9.1$, 2H), 7.32 (d, $J = 3.8$, 2H), 7.23 (d, J =4.7, 2H), 7. 04 (d, $J = 9.0$, 2H). Elemental Analysis: Calcd. For $(C_{30}H_{22}F_6N_2O_4)_n$ (588.5)n: C, 61.23; H, 3.77; N, 4.76. Found: C, 61.05; H, 3.64; N, 4.84.

2.3.2. *FT-IR, ^{1}H-NMR, and elemental analysis data of PEA*

FT-IR (film): 3,298 (–NHCO–), 1,658 (C=O), 1,246 (C–O–C), 1,179, 1135 (–CF_3) cm^{-1}. ^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 10.65 (s, 2H), 8.56 (s, 1H), 8.22 (d, $J = 2.0$, 2H), 8.16 (d, $J = 7.9$,2H), 8.03 (s, 2H), 8.00 (d, $J = 1.9$, 2H), 7.70 (t, $J = 2.8$, 1H), 7.30 (m, 2H), 7.21 (m, 2H), 7.07 (d, $J = 9.1$, 2H). Elemental Analysis: Calcd. For $(C_{30}H_{22}F_6N_2O_4)_n$ (588.5)n: C, 61.23; H, 3.77; N, 4.76. Found: C, 61.02; H, 3.61; N, 4.87.

2.3.3. *FT-IR, ^{1}H-NMR, and elemental analysis data of PEA*

FT-IR (film): 3,305 (–NHCO–), 1,656 (C=O), 1,243 (C–O–C), 1,175, 1136 (–CF_3) cm^{-1}.^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 10.46 (s, 2H), 8.21 (s, 2H), 8.05 (d, $J = 8.6$, 4H), 7.97 (d, $J = 8.9$, 2H), 7.31 (t, $J = 2.9$, 2H), 7.03 (d, $J = 9.1$, 2H). Elemental Analysis: Calcd.



Sch. 1. Synthesis of the fluorinated PEAs.

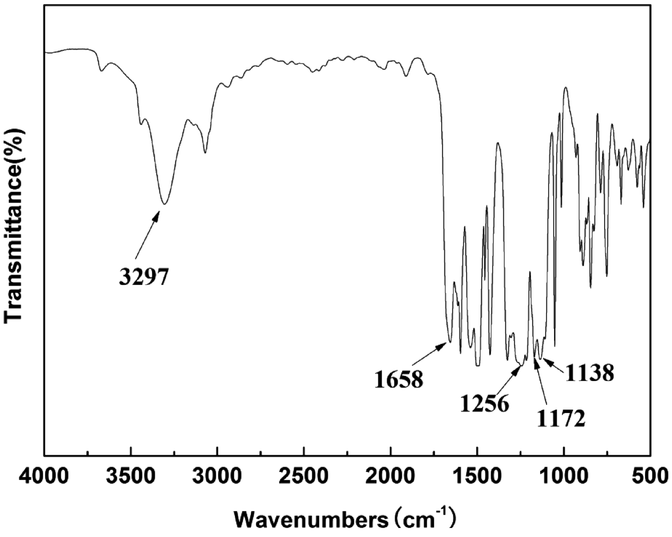


Fig. 1. FT-IR spectrum of PEA **3c**.

For $(C_{30}H_{22}F_6N_2O_6)_n$ (680.6)n: C, 58.07; H, 3.57; N, 4.51. Found: C, 57.91; H, 3.45; N, 4.62.

2.3.4. *FT-IR, ^{1}H-NMR, and elemental analysis data of PEA*

FT-IR (film): 3,297 (−NHCO−), 1,658 (C=O), 1,256 (C−O−C), 1,179, 1138 ($-CF_3$) cm^{-1}.^{1}H-NMR (400 MHz, DMSO-d_6, δ, ppm): 10.64 (s, 2H), 8.20 (d, $J = 1.5$, 2H), 8.03 (d, $J = 8.4$, 4H), 7.95 (d, $J = 8.9$, 2H), 7.51 (d, $J = 8.4$, 4H), 7.30(t, $J = 4.4$, 2H), 7.20 (m, 4H), 7.02 (d, $J = 9.1$, 4H). Elemental Analysis: Calcd. For $(C_{39}H_26F_{12}N_2O4)_n$ (814.6)n: C, 57.50; H, 3.22; N, 3.44. Found: C, 57.34; H, 3.13; N, 4.53.

3 Results and Discussion

3.1 Polymer Synthesis

According to the phosphorylation polycondensation first described by Yamazaki et al. (28, 29), a series of new fluorinated poly(aryl ether amide)s **3a–d** were synthesized from the aromatic diamine **1** with various aromatic dicarboxylic acids **2a–d** (Sch. 1). All the polymerizations of the diamine with various diacids in NMP proceeded and afforded homogeneous solutions, even at the end of polymerization, indicating the good solubility of obtained PEAs in the NMP. The polymers were obtained in almost quantitative yields and the inherent viscosities between 0.55 and 0.78 dL/g^{-1} in DMAc solution are listed in Table 1. All of the PEAs could be fabricated into transparent and flexible films, indicating medium- to high-molecular-weight polymer formation. The structure of PEAs was confirmed by elemental analysis, FT-IR and ^{1}HNMR spectroscopies, and corresponding data are given at the "***2.3.Polymer synthesis***" section.

The FT-IR spectra of the PEAs showed the characteristic absorptions around 3,297 and 1,658 cm^{-1} ,which were characteristic of N–H stretching and carbonyl stretching (C=O) respectively. The strong absorption band was observed in the region of 1100–1300 cm^{-1}due to the C−O−C and C−F multiple stretching. As a typical example, the FT-IR and ^{1}H-NMR spectra of PEA **3c** was illustrated in Figures 1 and 2, respectively. The assignments of each proton are given in Figure 2. The disappearance of amino groups

Table 1. Inherent viscosity and solubility behavior of these PEAs

		Solvents^c							
Polymers^a	η^b_{inh} *(dL/g)*	*NMP*	*DMAc*	*DMF*	*DMSO*	*THF*	*Cy*	*$CHCl_3$*	
3a	0.57	+++	++	++	–	++	+	+–	–
3b	0.55	+++	+++	+++	++	++	+	+–	–
3c	0.78	+++	+++	+++	++	++	+	(S)	–
3d	0.59	+++	+++	+++	++	++	+	(S)	–

[a] +++, 100 mg sample dissolved in 1 ml solvent (10%); ++, soluble at 5%; +, soluble at 1%; –, insoluble; S, swelling.

[b] Measured at a concentration of 0.5 g dL^{-1}in DMAc at 25°C.

[c] Abbreviations: NMP, N-methyl-2-pyrrolidinone; DMAc, N,N-dimethylacetamide; DMF, N,N-dimethylformamide; DMSO, dimethyl sulfoxide; Py, pyridine; THF,tetrahydrofuran; Cy, Cyclohexanone.

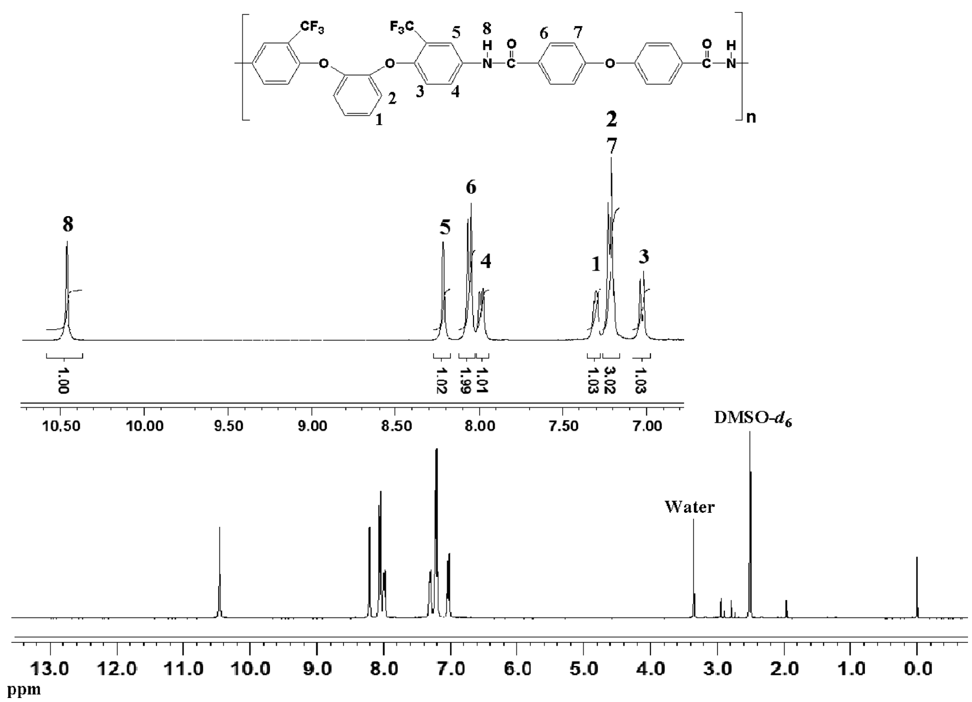


Fig. 2. The ^{1}H-NMR spectrum of **3c** in DMSO-d_6.

indicated a virtually complete conversion of the diamine **1** into PEA **3c**. The assignment of each proton was marked by Arabic numerals. The integrated area of each proton was consistence with their chemical composition. Elemental analyses are also generally in good agreement with the proposed structures, missing only the values found for the C-percentages that are always lower than the theoretical ones. That certainly can be attributed to the very aromatic nature of these polyamides, which commonly leave a small coal residue in the standard conditions of microanalysis.

3.2 Solubility of the Obtained PEAs

The solubility behavior of these fluorinated PEAs was tested qualitatively and the results are summarized in Table 1. It could be seen that all the newly synthesized PEAs showed excellent solubility in aprotic polar solvents, such as NMP, DMAc, DMF, THF, Pyridine and DMSO. The maximum dissolvability of the polymers in these solvents even exceeded 10.0%. The high solubility of these fluorinated PEAs is apparently due to the presence of the pendent trifluoromethylphenoxy groups which resulted in the decrease in the interaction of polymer chains by increasing the distance between polymer chains. Among these PEAs, **3c** and **4d** exhibited better solubility owing to the more flexible ether linkages in the macromolecular backbone or more $-CF_3$ pendant groups. For example, **3c** and **4d** could even be dissolved in common organic solvent like acetone and chloroform at room temperature.

3.3 Thermal Stability of the Obtained PEAs

The thermal behavior data of all the fluorinated PEAs are presented in Table 2. The glass transition temperature (T_g) values of the fluorinated PEAs are in the range of 227–244°C. As expected, **3d** exhibited the highest T_g value (244°C) because of the effect of the rigid macromolecular backbone, whereas **3b** obtained from 4,4′-oxydibenzoic acid showed the lowest T_g of 222°C. This is reasonable for its flexible ether linkage. The 10% weight loss temperature (T_{10}) and residual weight retention (R_w) at 800°C in nitrogen atmosphere were measured by TGA and corresponding TGA curves of the fluorinated exhibited in Figure 3. The T_{10} values of the fluorinated PEAs stayed in the range of 453–464°C in nitrogen and in the range of 433–451°C in air, respectively. They left more than 40% residual weight

Table 2. Thermal properties of various fluorinated PEAs

Polymer code	T_g^a *(°C)*	T_5^b *(°C)* *In* N_2	*In Air*	T_{10} *(°C)* *In* N_2	*In Air*	*Char Yield*^{c} *(%)*
3a	237	424	408	458	442	49
3b	227	423	415	456	441	52
3c	231	434	425	464	451	51
3d	244	412	415	453	433	42

[a] From DSC measurements conducted at a heating rate of 10°C min^{-1}.
[b] Temperature at 5% weight loss (T_5) and 10% wt loss (T_{10}) were determined by TGA in nitrogen atmosphere at a heating rate of 20°C min^{-1}.
[c] Residual weight retention (R_w) at 800°C in nitrogen atmosphere.

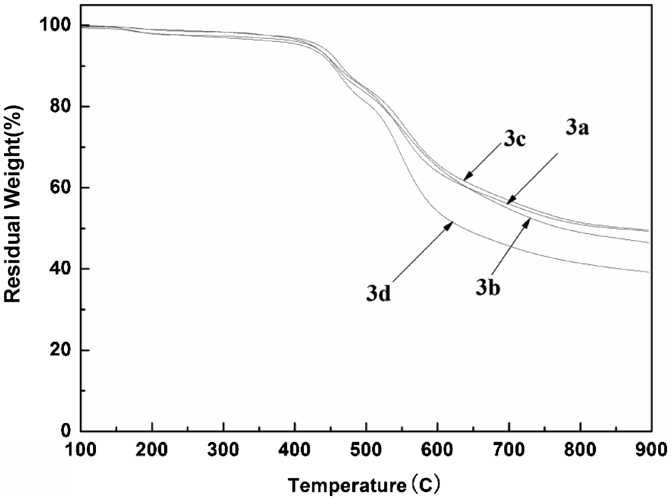


Fig. 3. TGA curves of PEAs **3a–d** at a heating rate of 20°C/min in N_2.

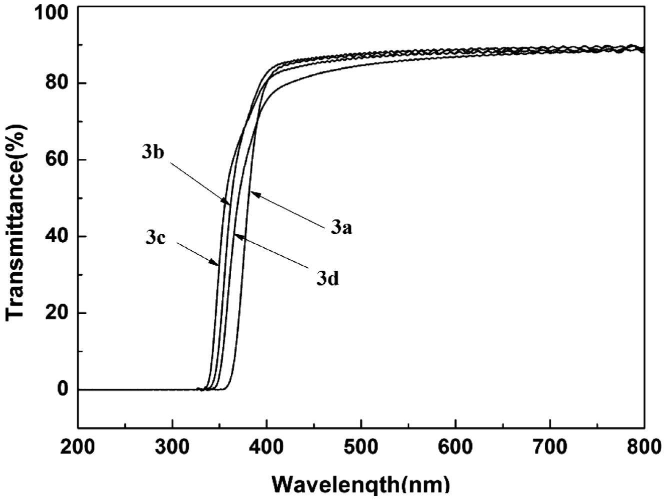


Fig. 4. UV-Vis spectra of various fluorinated PEA films.

at 800°C in nitrogen. The TGA data indicated that these PEAs had fairly high stability even with the introduction of bulky trifluoromethylphenoxy pendent groups.

3.4 Mechanical and Optical Properties

The molecular weights of all the PEAs were sufficiently high to permit the casting of flexible and tough films from solutions in DMAc, which were subsequently, converted into tough PEA films by extended heating at rising temperatures. These films were subjected to tensile tests and their mechanical properties were listed in Table 3. **3a–d** showed good tensile performance with tensile strengths, Young modulus and elongation at break in the range of 78.4–104.8 MPa, 1.65–2.21 GPa and 5.4–10.3%, respectively. These results indicated that the incorporation of the $-CF_3$ groups into the structure of PEAs not only improved solubility but also retained the good mechanical properties. The optical properties of the fluorinated PEA films are also given in Table 3, and the UV–Vis spectra of these films, about 20 μm thick, are shown in Figure 4. It could be seen from Table 3 that these fluorinated PEA films exhibited good transparency, the UV cutoff wavelength (λ_{cutoff}) was in the range of 336–357 nm, and the transparency at 450 nm (T_{450}) was higher than 83%. As expected, the introduction of bulky pendant groups effectively disrupt the dense packing of molecular chains and lead to low color and high optical transparent polymer films.

Table 3. Mechanical and optical properties of fluorinated PEAs films

Polymer code	*Tensile strength (MPa)*	*Tensile modulus (GPa)*	*Elongation at break (%)*	*λ_{cutoff} (nm)*	*T_{450} (%)*
3a	94.7	1.85	9.7	357	82
3b	78.4	1.65	5.4	340	83
3c	104.8	2.21	10.3	336	84
3d	84.7	1.73	7.5	344	86

[a] Measured in THF relative to polystyrene standards.

3.5 X-Ray Diffraction of the PEAs

The wide angle X-ray diffraction (WAXD) of these PEAs was illustrated in Figure 5. The obtained PEAs displayed almost amorphous patterns in nature, this could be attributable to the presence of pendent which reduces attraction between molecules and hindrances macromolecular packing regularly. Generally, these polymers with similar structure containing $-CF_3$ groups are amorphous (30–34).

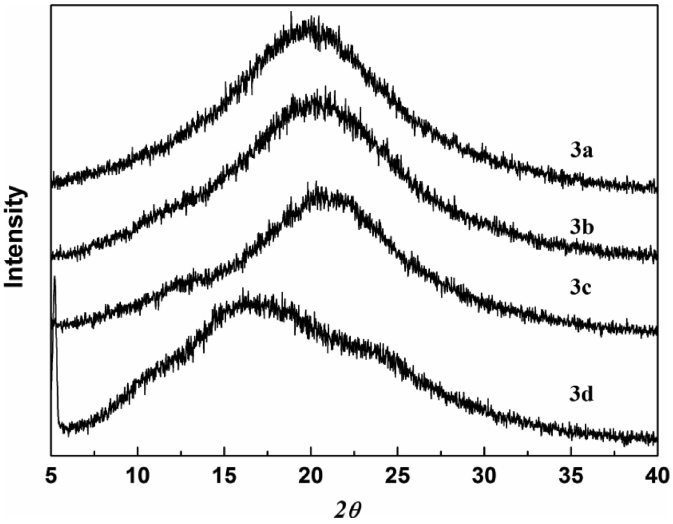


Fig. 5. X-ray diffraction patterns of various fluorinated PEAs.

Table 4. Dielectric constant and water absorption of various fluorinated PEA films

Polymer code	Film thickness (μm)	Dielectric constant				Water absorption (%)
		1 MHz	*10 MHz*	*100 MHz*	*1000 MHz*	
3a	73	2.93	2.54	2.50	2.47	2.02
3b	64	3.29	2.86	2.80	2.77	1.97
3c	60	2.99	2.58	2.54	2.51	1.72
3d	60	2.84	2.47	2.42	2.39	1.27

3.6 Dielectric Constants and Water Absorption

The dielectric constants and water absorption of all PEAs were summarized in Table 4. The PEAs **3a-d** showed the dielectric constants at 1 MHz and 1 GHz in the range of 2.84–3.29 and 2.39–2.77. The decreased dielectric constants could mainly be attributed to the trifluoromethyl groups in the polymer chains. The low polarizability of trifluoromethyl groups, combined with the bulky free volumes of laterally dimethyl groups, endowed the PEAs with low dielectric constants. PEA **3d** exhibited the lowest dielectric constant among this series of polymers because of the highest fluorine content in the repeat unit. These results suggested that the $-CF_3$ groups in the polymer chains play an important role in the electrical and dielectric performance. It presents utility for the microelectronics industry, in which a low dielectric constant is desired to prevent crosstalk between conducting paths. As expected, the PEAs also exhibited lower water absorptions (1.27–2.02%) due to the hydrophobic nature of the trifluoromethyl groups. The low water absorptions also ensure that these PEAs have stable dielectric performance.

4 Conclusions

A series of new organosoluble PEAs containing trifluoromethyl were successfully prepared by the phosphorylation polycondensation. The introduction of bulky pendant groups is an effective way to improve their solubility, optical and dielectric properties without substantial diminution of their physical properties. All the obtained PEAs showed a high solubility, good thermal stability, low dielectric constants and water absorption and could be easily processed to flexible and tough films with good mechanical properties. Furthermore, they displayed high optical transparency. Thus, these obtained PEAs demonstrated an eminent combination of several desired properties and could be considered as potential high-temperature resistant materials for optical or microelectronic applications.

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