Journal of Fluorine Chemistry 144 (2012) 176–181

Contents lists available at SciVerse ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

Soluble copoly(aryl ether ether ketone ketone)s containing xanthene and hexafluoroisopropylidene moieties

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ARTICLE INFO

Article history:
Received 27 June 2012
Received in revised form 9 September 2012
Accepted 11 September 2012
Available online 3 October 2012

Keywords:
Fluoropolymer
Poly(aryl ether ketone) copolymers
Xanthene unit
Trifluoromethyl group
Soluble

ABSTRACT

Several new copoly(aryl ether ether ketone ketone)s (PEEKKs) containing xanthene and hexafluoroisopropylidene moieties were prepared *via* nucleophilic substitution polycondensation of 1,4-bis(4-fluorobenzoyl)benzene with 9,9-bis(4-hydroxyphenyl)xanthene (BHPX) and 4,4′-hexafluoroisopropylidenediphenol (6F-BPA) by varying mole proportions of BHPX to 6F-BPA (5/95–75/25) in tetramethylene sulfone in the presence of anhydrous potassium carbonate. These polymers with weight-average molecular weights of 78,900–81,500 and number-average molecular weights of 38,500–40,800 were all amorphous and soluble in organic solvents such as *N*-methyl-2-pyrrolidone (NMP) and *N*,*N*-dimethylformamide (DMF), and even in THF and chloroform at room temperature. The copolymers showed glass transition temperatures (T_g's) between 180 and 216 °C, and the T_g values increased with increase in concentration of the BHPX units in the polymer. Besides, all the polymers were stable up to 470 °C with 5% weight loss temperatures ranging from 525 to 544 °C, and char yields of 58–64% at 800 °C in nitrogen. These copolymers could be cast into transparent, strong, and flexible films with tensile strengths of 52.4–75.1 MPa, elongations at break of 8–13%, and tensile moduli of 2.05–2.55 GPa. Their films had low dielectric constants of 2.58–2.84 (1 MHz) and low water absorptions in the range of 0.24–0.38%, and high transparency with an ultraviolet–visible absorption cut-off wavelengths in the 356–376 nm range.

1. Introduction

Aromatic poly(ether ketone)s (PEKs), known as high performance engineering plastics, have received considerable attention from industry and academy, due to their high mechanical properties, excellent thermooxidative stability and chemical resistance [1]. Among them, two of the most prominent examples, poly(ether ether ketone) (PEEK) and poly(ether ketone ketone) (PEKK) have achieved significant commercial importance as matrices for carbon fiber composites in aerospace, industrial and automotive applications and as dielectrics in the micro electronics field [1]. They are semicrystalline polymers with melting temperatures around 340–360 °C and glass transition temperatures (T_g's) around 140–160 °C. Moreover, they are generally insoluble in organic solvents at ambient temperature. As a result, these polymers cannot be processed by the casting method and require a high molding temperature resulted from their high melting temperatures. Additionally, the T_g's of these polymers are relative low, which leads to a low dimension stability [1]. Therefore, many efforts have been directed toward synthesizing soluble PEKs with enhanced processability, while keeping their desired properties. It has been proposed that introduction of pendant groups, heterocyclic moieties, asymmetry or noncoplanar structures into the polymeric chains of PEKs could improve the processability and/or glass-transition temperature [2–13]. Another successful approach to increasing solubility and processability of PEKs without an extreme loss of their outstanding properties is the introduction of a bulky cardo group into the polymer backbones. It has been demonstrated that the incorporation of cardo groups [14–19] into the backbone of PEKs results in polymers with enhanced solubility, processability, and good thermal stability. On the other hand, fluorine-containing polymers are currently of special interest because of their unique properties and high temperature performance. In general, the incorporation of fluorinated substituents into PEKs will decrease the dielectric constants, moisture absorption, and refractive index and increase the free volume, solubility, and thermal stability [15,20–24]. In particular, the polymers containing hexafluoroisopropyl moieties have been exploited for applications as optical wave-guide materials, microelectronic devices, and gas-separation membranes. Recently, our groups have prepared xanthene-based cardo poly(arylene ether ketone)s [25] with enhanced solubility, processability, and good mechanical strengths and thermal properties, as well as glass transition temperature above 210 °C.

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http://dx.doi.org/10.1016/j.jfluchem.2012.09.007

Table 1
Synthetic data and some physical properties of the polymers.

Polymer	Yield (%)	η_{inh} (dL/g)[a]	M_w (g/mol)[b]	M_n (g/mol)[b]	M_w/M_n
Pa	97	0.75	78,900	38,500	2.05
Pb	98	0.80	80,800	39,900	2.03
Pc	97	0.77	79,700	40,600	1.96
Pd	97	0.82	81,500	40,800	2.00
Pe[c]	96	0.77	80,400	40,500	1.98

[a] Measured at a polymer concentration of 0.5 g/dL in NMP at 30 °C.
[b] Determined by GPC in tetrahydrofuran with polystyrene as standard.
[c] Derived from BFBB with BHPX in the literature [25].

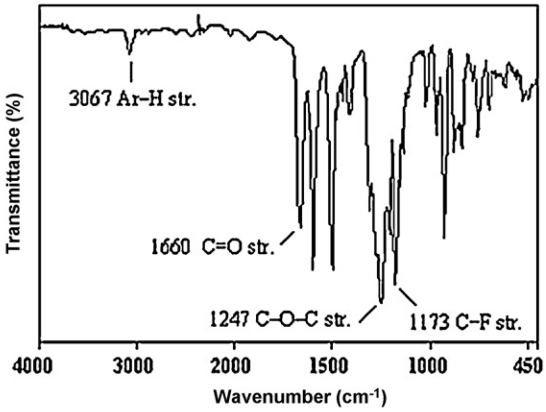


Fig. 1. FTIR spectrum of the PEEKK copolymer **Pc**.

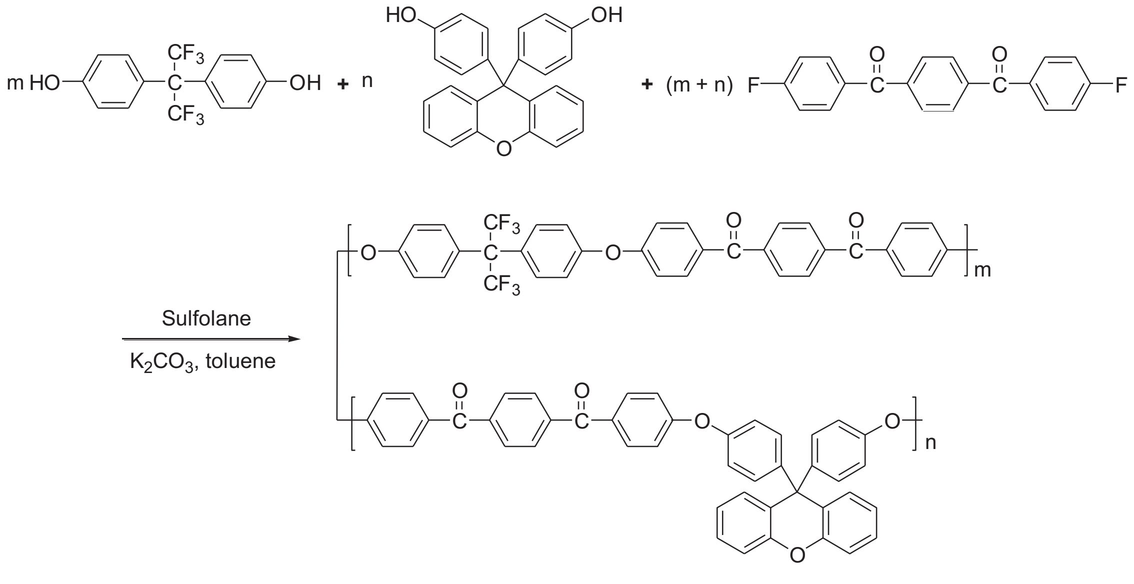
As part of an ongoing research program focused on the introduction of xanthene cardo group into the high-performance engineering thermoplastics, we here have developed several novel fluorinated PEEKK copolymers containing xanthene and hexafluoroisopropylidene moieties, which were prepared by an aromatic nucleophilic substitution reaction of 9,9-bis(4-hydroxyphenyl)xanthene (**BHPX**), 1,4-bis(4-fluorobenzoyl)benzene (**BFBB**) and 4,4′-hexafluoroisopropylidenediphenol (**6F-BPA**) in tetramethylene sulfone in the presence of potassium carbonate as an acid acceptor. In this paper, the solubility, tensile property, crystallinity, thermal property, water absorption, dielectric constant, as well as optical transparency of all the obtained polymers were investigated.

2. Results and discussion

2.1. Polymer synthesis

As outlined in Scheme 1, several PEEKK copolymers with xanthene and hexafluoroisopropylidene units were prepared by aromatic nucleophilic polycondensation of **6F-BPA**, **BHPX** and **BFBB** by varying the mole ratio of **BHPX** to **6F-BPA** (5/95–75/25) with potassium carbonate as the base, tetramethylene sulfone as a solvent and toluene as azeotropic solvent for removal of water. The reaction system was heated to a reflux temperature to promote phenolate formation. Subsequently, the polymerization was conducted at 200 °C after completely removing toluene. In the experiment, the viscosity of the reaction systems was increased dramatically only about 0.5 h, and high molecular weight polymers were obtained readily in 2.5–3 h after the completion of bisphenolate formation. Furthermore, it was found that the existence of bulky pendant xanthene groups in the monomer did not have a significant effect on the polymerization reaction. The inherent viscosity values and some physical properties of the resulting PEEKKs based on different ratios of monomers were given in Table 1. These copolymers were obtained in almost quantitative yields, with inherent viscosities in the range of 0.75–0.82 dL/g in NMP. At the same time, their gel permeation chromatography (GPC) data showed that the number-average molecular weights (M_n's) were in the range of 38,500–40,800 g/mol, and the polydispersity values of these copolymers ranged from 1.96 to 2.05, using polystyrene as a standard, indicating that all the copolymers had sufficiently high molecular weights to permit the casting of tough and flexible films.

The FTIR spectra of all the polymers supported their structures. All polymers exhibited characteristic absorption bands around 1660 cm^{-1} corresponding to aryl carbonyl groups, around 1247 cm^{-1} due to aryl ether linkages, along with the characteristic absorption bands of C–F stretching near 1174 cm^{-1}. Fig. 1 displayed the typical FTIR spectrum of the PEEKK copolymer **Pc**.



Pa: m = 23.75 mmol, n = 1.250 mmol; **Pb**: m = 18.75 mmol, n = 6.250 mmol
Pc: m = 12.50 mmol, n = 12.50 mmol; **Pd**: m = 1.250 mmol, n = 23.75 mmol

Scheme 1. Preparation of fluorinated (aryl ether ether ketone ketone) copolymers.

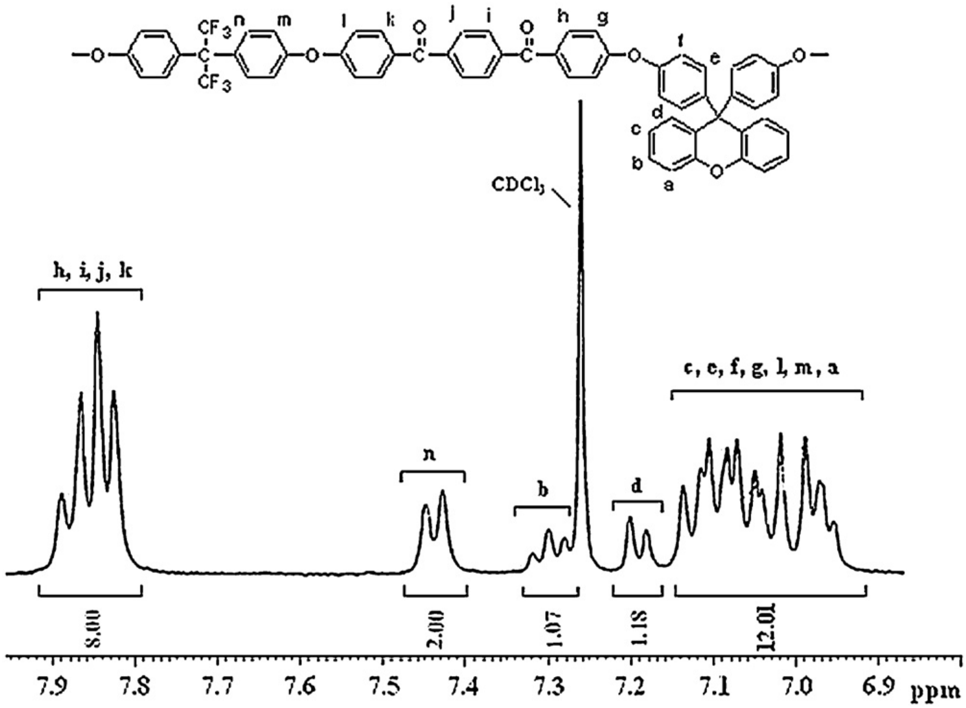


Fig. 2. ^{1}H NMR spectrum of the PEEKK copolymer **Pc**.

Besides, the chain structures of the polymers were also confirmed by ^{1}H NMR spectroscopy. In the ^{1}H NMR spectra of the polymers, the signals at 6.94–7.90 ppm were assigned to the aromatic protons in the polymer chains. A typical ^{1}H NMR spectrum of PEEKK copolymer **Pc** was shown in Fig. 2, and its spectrum agreed with the proposed polymer structure.

2.2. Thermal and crystal properties

The thermal properties of the new fluorinated PEEKKs were evaluated by DSC and TGA, and the results were summarized in Table 2. The T_g values of the polymers were 180–216 °C, and no melting endotherms were observed in DSC traces, which further confirmed the amorphous nature of these PEEKKs (Fig. 3). As depicted in Table 2, the T_g of the PEEKK copolymers increased with an increase of BHPX content in the polymers. This indicated that the gradual increase of the bulky pendant xanthene groups in the copolymer backbones hindered internal rotation of the polymer backbones, and increased the glass-transition temperatures of the corresponding copolymers, especially that of the copolymer **Pd**, for which higher T_g (216 °C) was observed.

Table 2
Thermal properties of the polymers.

Polymer	T_g (°C)[a]	T_{d0} (°C)[b]	T_{d5} (°C)[c]	T_{d10} (°C)[d]	R_w (%)[e]
Pa	180	478	528	543	61
Pb	189	471	525	541	61
Pc	203	483	527	543	64
Pd	216	487	544	563	58
Pe[f]	229	457	553	572	60

[a] Glass transition temperature base on the second heating trace of DSC measurements.

[b] Initial decomposition temperature measured by TGA under nitrogen atmosphere.

[c] Temperature for 5% weight loss measured by TGA under nitrogen atmosphere.

[d] Temperature for 10% weight loss measured by TGA under nitrogen atmosphere.

[e] Residual weight percentage heated to 800 °C under nitrogen atmosphere.

[f] Derived from BFBB with BHPX in the literature [25].

Fig. 4 presented TGA curves for all copolymers under nitrogen atmosphere. As disclosed in Table 2, it is interesting to note that **BHPX** contents in the polymer chains did not affect much on the thermal stability of the copolymers. The temperatures at 5 and 10% weight loss (T_5 and T_{10}) were in the range of 525–544 °C and 541–563 °C, respectively. Moreover, all polymers were stable up to 470 °C and gave a char yield of 58–64% at 800 °C. The high thermal stability is attributed to the rigidity of the aromatic chains and strong C–F bonds. Additionally, it should be noted that there was a large window between the T_g and the decomposition temperature for all PEEKK copolymers, which could be advantageous in the processing of these polymers. Compared with the structurally similar PEK containing-fluorine moieties [26,27,16], these newly prepared PEEKK copolymers seemed to exhibit better thermal stability due to the incorporation of the more rigid xanthene moieties to the main chain.

The crystallinity of the PEEKK copolymers was examined by wide angle X-ray diffraction (WAXD). All the obtained polymers

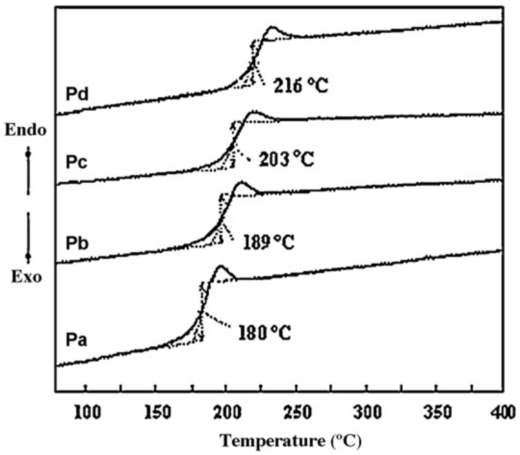


Fig. 3. DSC curves for 2nd heating of the PEEKK copolymers.

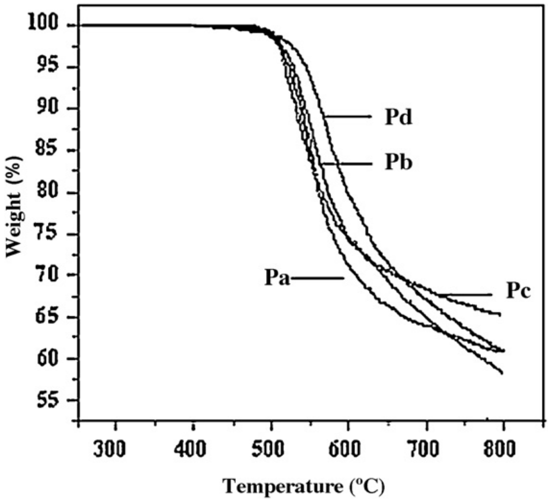


Fig. 4. TGA curves of the PEEKK copolymers.

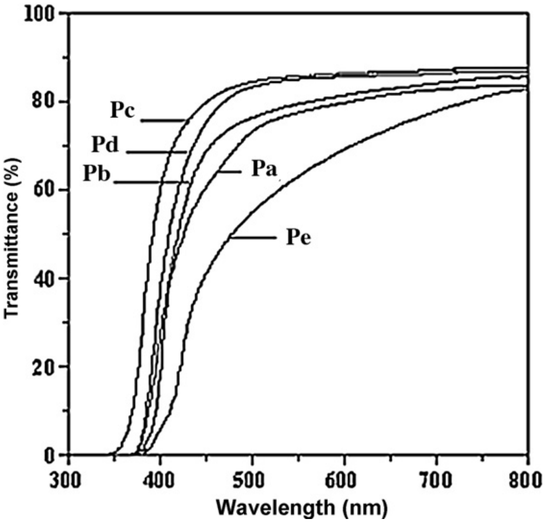


Fig. 5. UV–vis absorption spectra of the PEEKK copolymers.

showed amorphous patterns. The amorphous behavior of the polymers was mainly due to the presence of bulky xanthene pendant groups and trifluoromethyl substituents, which inhibited the close packing of the polymer chains, reducing intramolecular interactions and disrupting the regularity of molecular chains.

2.3. Solubility and mechanical properties

The solubility behavior of the polymers in some solvents was listed in Table 3. It is well known that the conventional PEEK is insoluble in most known organic solvents. In contrast, the present fluorinated PEEKK copolymers were soluble at room temperature in aprotic polar solvents such as NMP, DMF and *N,N*-dimethylacetamide (DMAc) as well as in the less polar solvents like chloroform and tetrahydrofuran. Obviously, the good solubility of the copolymers could be attributed to the combined effects of the bulky xanthene pendant groups, trifluoromethyl substituents and flexible ether linkages in the polymer backbones, which prevented close chain-packing and allowed the solvent molecules to diffuse easily into the polymer chains [28]. The excellent solubility of these fluorinated PEEKK copolymers makes them easier for film preparation by spin-coating, and it also shows the potential for further device fabrication.

Table 3
Solubility of the polymers.[a]

Polymer	DMF	DMAc	NMP	THF	$CHCl_3$	Conc. H_2SO_4
Pa	+	+	+	+	+	+
Pb	+	+	+	+	+	+
Pc	+	+	+	+	+	+
Pd	+	+	+	+	+	+
PEEK	–	–	–	–	–	+

[a] The symbols represent the following: + = soluble at room temperature; – = insoluble even on heating. DMF: *N,N*-dimethylformamide; DMAc: *N,N*-dimethylacetamide; NMP: *N*-methyl-2-pyrrolidone.

Table 4
Mechanical properties of the polymers.

Polymer	Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (GPa)
Pa	52.4	8	2.05
Pb	58.7	10	2.37
Pc	64.3	11	2.16
Pd	75.1	13	2.55

The mechanical properties of the polymer thin film cast from DMAc were reported in Table 4. All the films were transparent, strong, and flexible. The polymer films had tensile strengths of 52.4–75.1 MPa, tensile moduli of 2.05–2.55 GPa, and elongations at break of 8–13%, indicating that these copolymers exhibited the expected ductile mechanical behavior characteristics of many materials containing aryl ether linkages [29,30]. These values were comparable to those of high-performance thermoplastic materials such as PEEK and Ultem PEI.

2.4. Optical transparency

The UV–vis absorption spectra of the polymer films with approximately 0.05 mm thickness were presented in Fig. 5, the cut-off wavelengths, optical transparency at 500 and 600 nm of all the polymers were also illustrated in Table 5. As seen from Table 5, except of PEEKK **Pe** only containing BHPX units, the cut-off wavelengths of these new fluorinated PEEKK copolymers ranging from 356 to 376 nm was lower than 380 nm, indicating that the polymers were colorless and had high transmittance. Furthermore, all the polymers showed higher than 80% optical transmittance at 600 nm. It is very interesting to note that polymer **Pc** with equal mole proportion of **BHPX** to **6F-BPA** exhibited the lowest cut-off wavelength (356 nm). The good optical transparency of the resulting polymers may be resulted from two reasons. The incorporation of xanthene cardo group into the polymer chains restricted the molecular alignment and therefore disturbed effective charge-transfer-complex formation. In addition, the extent of donor–acceptor complex formation could also be further reduced by lowering of the degree of charge separation along the polymer chains through the introduction of electron-withdrawing trifluoromethyl substituents, which separated the chromophoric groups and interrupted the intramolecular conjugation.

2.5. Water absorption and dielectric constants

Table 5 also summarized the water absorption and dielectric constants of these polymers. The fluorinated polymer films showed water absorptions in the range of 0.24–0.38%, which were lower than those of conventional PEEK [23] and PEEKK **Pe** derived from BHPX and BFBB [25], with water absorptions of 0.50 and 0.48%, respectively. The low water absorptions of the newly polymers are attributed to the hydrophobicity of the fluorine-containing groups. The dielectric constants of the polymers, as

Table 5
Optical transparency, water absorption and dielectric constants of the polymers.

Polymer	Cut-off wavelength[a] (nm)	Transparency[b] (%)	Transparency[c] (%)	Water uptake[d] (%)	Dielectric constant[e]
Pa	376	75	80	0.24	2.58
Pb	374	81	86	0.29	2.66
Pc	356	88	90	0.34	2.72
Pd	372	87	89	0.38	2.84
Pe[f]	384	75	80	0.48	3.22

[a] The wavelength range of UV measurement was from 200 to 800 nm.
[b] Transparency at 500 nm.
[c] Transparency at 600 nm.
[d] Calculated from the difference in the weights before and after immersion of the polymer films in water.
[e] The dry dielectric constants were obtained in the frequency of 1 MHz at room temperatures.
[f] Derived from BFBB with BHPX in the literature [25].

listed in Table 5, were measured on the thin films. Compared with conventional PEEK without pendant groups (3.27 at 1 MHz) [23], the resulting polymers had lower dielectric constants in the range of 2.58–2.84 (1 MHz). The dielectric constant also depended on the fluorine content, as the percentage of fluorine increased, a decrease in the dielectric constant was observed, as shown in Table 5. Also, the fluorine content in the polymer structure largely affected the water absorption value. There might be two related factors that could explain the observed results. First, the fluorine substitution would reduce moisture sorption and increase the free volume, leading to a lower dielectric constant. Second, the bulky xanthene pendant groups attached to the polymer main chains could inhibit close chain-packing, and this resulted in loosely packed polymers and a low dielectric constant.

3. Conclusions

In summary, four new poly(aryl ether ether ketone ketone) copolymers (PEEKKs) containing xanthene and hexafluoroisopropylidene moieties were synthesized with 1,4-bis(4-fluorobenzoyl)benzene with 9,9-bis(4-hydroxyphenyl)xanthene and 4,4′-hexafluoroisopropylidenediphenol by nucleophilic polycondensation. Compared with the conventional unsubstituted PEEK, all the copolymers exhibited enhanced glass transition temperatures and remarkably improved solubilities in organic solvents, as well as low dielectric constants, low water absorptions, and high optical transparency. These characteristics indicated that the present polymers might be considered as potentially promising candidates for processable high-temperature microelectronic devices, optical materials and gas-separation membranes.

4. Experimental

4.1. Materials

9,9-Bis(4-hydroxyphenyl)xanthene (**BHPX**) (mp = 239–240 °C) was synthesized according to our reported method [25]. 1,4-Bis(4-fluorobenzoyl)benzene (BFBB) (mp = 219–220 °C) (Yanji Chemical Plant, China) was recrystallized from toluene before use. 4,4′-Hexafluoroisopropylidenediphenol (**6F-BPA**) (mp = 161–162 °C) (Aldrich Chemical Corp.) was purified by sublimation and dried *in vacuo*. Anhydrous potassium carbonate (Beijing Chemical Reagent Co., China) was dried in vacuum at 100 °C before use. Tetramethylene sulfone (TMS) (Beijing Chemical Reagent Co., China) was dried with 4 Å molecular sieve and distilled over NaOH pellets under reduced pressure. The middle fractions were collected and stored over molecular sieves. The other reagents and solvents such as *N*-methyl-2-pyrrolidone (NMP), *N, N*′-dimethylformamide (DMF), *N,N*′-dimethylacetamide (DMAc), chloroform, tetrahydrofuran, toluene and methanol were obtained from commercial sources and used as received.

4.2. Measurements

^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra were recorded on Bruker AVANCE 400 MHz spectrometer in chloroform ($CDCl_3$). FTIR spectra of the monomer and polymers in KBr pellets were obtained on a Perkin-Elmer SP One FT-IR spectrophotometer. Differential scanning calorimetry (DSC) analyses were performed on a Perkin Elmer Pyris 1 DSC at a scanning rate of 20 °C/min in flowing nitrogen (20 mL/min). The glass-transition temperature (T_g) values were reported from the second scan after the first heating and quenching, and taken from the midpoint of the change in the slope of the baseline. Thermogravimetric analysis (TGA) was carried out on approximately 6–8 mg film samples with a Perkin Elmer Pyris 1 TGA under nitrogen atmosphere (120 mL/min) at a heating rate of 10 °C/min from 50 to 800 °C. Wide-angle X-ray diffraction patterns were recorded at room temperature (*ca.* 25 °C) on power with a Rigaku Geiger Flex D-Max III x-ray diffractometer, using Ni-filtered CuK_α radiation (operating at 40 kV and 15 mA); the scanning rate was 2°/min over a range of $2\theta = 5–40°$. The inherent viscosities were measured at 0.5 g dL^{-1} concentration in NMP with an Ubbelohde viscometer at 30 °C, in which the polymers were pretreated by drying in oven at 120 °C for 1 h to remove the adsorbed moisture. Molecular weights were determined by a gel permeation chromatography (GPC) with polystyrene calibration using a Waters 510 HPLC equipped with 5-μm phenogel columns (linear, 3× 500 Å) arranged in series and a UV detector at 254 nm using tetrahydrofuran as eluent. Polymer samples were dissolved in $CHCl_3$, filtered, precipitated in methanol and dried under vacuum at room temperature before performing GPC measurements. Mechanical properties of the thin films (about 0.2 mm, cast from DMAc solutions onto the glass plates, and followed by heating at 100–250 °C *in vacuo*) were measured at room temperature on an Instron 1121 instrument at a strain rate of 10 mm/min, and an average of at least four individual determinations was used. Ultraviolet–visible (UV–vis) spectra of the polymer films were recorded on a Shimadzu UV-1601 UV–vis spectrophotometer. Dielectric constants of the polymer films (diameter = 13 mm and thickness about 0.05 mm, prepared by compression molding, and then coated with silver by a vacuum evaporation method) were obtained using an HP 4192A LF impedance gain-phase analyzer with an HP-6047C capacitance meter at 1 MHz. The water uptakes of the polymer were measured by immersion of the polymer films (5 mm × 5 mm × 0.5 mm, prepared by compression molding) in deionized water at 100 °C for 2 h, followed by wiping with tissue paper for removal of any water on the surface. The water uptakes were calculated from the difference in the weights before and after this procedure.

4.3. Polymer synthesis

A representative polymerization procedure under nitrogen atmosphere for the synthesis of PEEKK copolymer (**Pc**) is as follows. To a 150 mL three-necked flask equipped with a mechanical stirrer, a nitrogen inlet with a thermometer, and a Dean–Stark trap with a condenser, was added **6F-BPA** (4.2029 g, 0.0125 mol), **BHPX** (4.5802 g, 0.0125 mol), **BFBB** (8.0578 g, 0.025 mol), anhydrous K_2CO_3 (4.485 g, 0.0325 mol), TMS (45 mL) and toluene (30 mL). The system was allowed to reflux for 2 h, and then the toluene was removed by distillation, and reaction mixture was heated to 200 °C and stirred at this temperature under nitrogen for 2.5 h. After cooling, the viscous solution was poured into methanol-water solution (500 mL), and the precipitated polymer was collected by filtration and then pulverized into a powder using a blender. The polymer powder was washed thoroughly several times with hot water, and then dissolved with chloroform, and precipitated in methanol twice. After filtration of the precipitates followed by heating the collected precipitates at 120 °C under vacuum overnight, the white colored polymer (**Pc**) was obtained in 97% yield. ^{1}H NMR ($CDCl_3$): δ = 7.89–7.83 (m, 8 H), 7.45 (d, J = 8.4 Hz, 2 H), 7.231 (t, J = 8.4 Hz, 1 H), 7.20 (d, J = 8.0 Hz, 1 H), 7.14–6.95 (m, 12 H); FT-IR (KBr): ν_{max} = 1660 (C=O), 1247 (Ar–O–Ar), 1173 (C–F) cm^{-1}.

Acknowledgments

We gratefully acknowledge financial support from the National Natural Science Foundation of China (Nos. 20664001, 21064003, and 51163008), the training program for science and technology leaders in major subjects of Jiangxi Province (No. 20113BCB22012), the Natural Science Foundation of Jiangxi Province (No. 20114BAB203014) and the Research Program of Jiangxi Province Department of Education (Nos. GJJ08166 and GJJ09138).

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