

Different Fluorine Content of Poly(aryleneetherketone) Coated on Polyimide Films to Fabricate Hydrophobic and Low Moisture Absorption Films

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Polyimides have attracted considerable attention owing to their excellent properties; however, their hydrophilicity makes them susceptible to hydrolytic degradation. In this study, polyaryletherketones (PAEKs) with different fluorine contents were coated onto the surface of the polyimide films to fabricate the hydrophobic surface. The wettability of the films changed, with a transition from hydrophilicity to hydrophobicity, upon coating with PAEKs. Thus, the coated films exhibited low moisture absorption. Due to the excellent thermal stability of fluorinated polyaryletherketones (F-PAEKs), the coated polyimide films retained their hydrophobicity even after extreme treatment. Furthermore, the dielectric properties of the coated polyimide film were superior to those of the unmodified film. The hydrophobic polyimide film can meet the requirements of extreme environments.

Keywords: polyimide film, hydrophobic modification, fluorinated polyaryletherketone, durability

Introduction

Polyimides (PIs) are high performance polymers that have been extensively employed in numerous industries owing to their excellent thermal and chemical stabilities, mechanical strength, and dielectricity;^{1,2} however, PIs are hydrophilic. Thus, they undergo hydrolytic degradation as a result of moisture absorption.³ In addition to the reduction in the strength and electrical resistivity of PIs, hydrolytic degradation causes fatigue crack initiation and embrittlement, which permanently damage the PIs.³ To resolve these problems, many studies have proposed strategies to modify PIs, such as through electrospinning,⁴ plasma treatment,⁵ laser radiation,⁶ ultraviolet (UV) photo-oxidation,⁷ template curing processes,⁸ and coating in an effort to increase their hydrophobicity.^{9,10} Coating is a particularly simple method of introducing a hydrophobic surface, while the other methods involve complex processes and require sophisticated and expensive equipment. For example, Li *et al.*¹¹ immersed PI aerogels in a fluorocarbon resin solution for 12 h to synthesize superhydrophobic PI aerogels, which exhibited a high contact angle of 156°. Xu *et al.*¹² obtained a superhydrophobic PI film by incorporating nano poly(divinylbenzene) onto a PI film

via a one-step solvothermal method. The modified PI film retained its superhydrophobicity even after heating, immersion in liquid nitrogen, or UV irradiation.

PIs are extensively used in spacecraft, solar cells, and microelectronic devices owing to their excellent thermal properties. Substrates in spacecraft applications require particularly high thermal stability and must retain a stable hydrophobic surface in a high-temperature environment. Hydrophobic surfaces can be prepared using numerous polymers, including polystyrene (PS),^{13,14} polymethyl methacrylate (PMMA),^{15,16} and polyurethane (PU).^{17,18} However, these polymers cannot withstand prolonged exposure to heat, which limits their application in high-temperature environments.

In this study, fluorinated polyaryletherketones (F-PAEKs) are used to fabricate hydrophobic surfaces on PI films, which are a class of functional polymer materials with high thermal stability. The introduction of the fluorine group to the backbone of PAEKs improves their solubility, processability, and hydrophobicity. Herein, F-PAEKs are synthesized by the nucleophilic substitution polycondensation of phenolphthalein, 4,4-difluorobenzophenone, and 2,2-bis-(4-hydroxyphenyl) hexafluoropropane. F-PAEKs were coated onto the PI films to alter their wettability, upon transition from hydrophilicity to hydrophobicity. Moreover, the adhesion of the F-PAEKs on PI films was investigated. The modified

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films were found to retain their hydrophobicity even under harsh conditions.

Experimental

Materials

Phenolphthalein, 4,4-difluorobenzophenone (DFBP), 2,2-bis-(4-hydroxyphenyl) hexafluoropropane (BPAF), potassium carbonate (K_2CO_3), *N,N*,2-methylmethanone amide (DMF), *N*-methylpyrrolidone (NMP), toluene, anhydrous ethanol, glycerin, and ethylene glycol were purchased in AR grade from Adamas (Shanghai, China). Diiodomethane was purchased in AR grade from Shanghai Merrill Chemical Technology Co., Ltd. (Shanghai, China). The PI films were commercially obtained from Huate electrical insulation Co. Ltd. (Wenzhou, China). They were divided into 3 cm × 2.5 cm pieces, then ultrasonically washed in ethanol and dried under nitrogen atmosphere.

Synthesis

Fluorinated polyaryletherketones (F-PAEKs) were prepared by nucleophile aromatic substitution, which was based on the methods described previously.¹⁹⁻²¹ The synthesis route is demonstrated in Scheme 1. 10 mmol DFBP were fixed, and the total molar amount of phenolphthalein and BPAF were 10 mmol. The mass ratios of phenolphthalein to BPAF (10:0, 7.5:2.5, 5:5, 2.5:7.5, 0:10), were named as F-PAEK-0, F-PAEK-1, F-PAEK-2, F-PAEK-3, F-PAEK-4, F-PAEK-5 and *x* represents the molar value of phenolphthalein in Scheme 1. Under a nitrogen atmosphere, phenolphthalein, BPAF, 10 mmol DFBP, 10 mmol K_2CO_3 , 30 mL DMF and 5 mL toluene were placed in a 100 mL flask equipped with a condenser and a dean-stark water segregator. The reaction was heated at 145 °C for 4 h to eliminate the generated water via azeotropic distillation with anhydrous toluene. Next, the

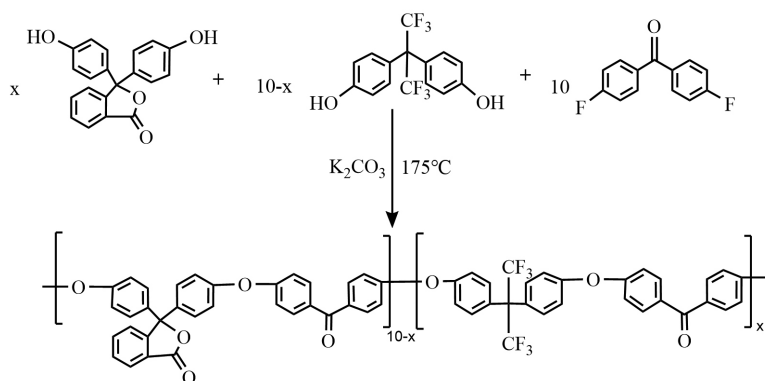
reaction was conducted at 175 °C for another 8 h. After the reaction, the obtained F-PAEKs were cooled to 50 °C and slowly poured into distilled water to precipitate the polymers. Finally, the polymers were washed 3 times with deionized water and ethanol to remove residual monomers and dried under vacuum at 60 °C for 12 h.

Preparation of F-PAEKs/PI films

F-PAEK (0.5 g) was dissolved in NMP (12 g) and filtered by suction followed by bubble elimination under vacuum. The polyimide film was lightly sanded, and the F-PAEK solution was coated on polyimide films using a 20 μm bar coater (denoted as F-PAEK/PI), dried at 80 °C for 2 h, and raised 20 °C *per* hour till 220 °C for 2 h.

Characterizations

Fourier-transform infrared (FTIR) spectra of F-PAEKs were performed on the infrared spectrometer (FT-IR370, Nicolet, USA), operating in the range of 2800-500 cm^{-1} by attenuated total reflectance (ATR) accessory. The structure of polymers was recorded by 1H nuclear magnetic resonance (NMR, 400 MHz, Bruker Corporation, Germany) and chloroform-*d* was used as the solvent. The molecular weight of the polymers was obtained by gel chromatography (GPC, Agilent 1200 s, Agilent, USA), and *N,N*,2-methylmethanone amide (DMF) as the eluent at a flow rate of 1 $mL\ min^{-1}$ and polyethyleneglycol (PEG) as the calibration standard. Differential scanning calorimeter (DSC 4000, PerkinElmer, USA) was employed to study the glass transition temperature (T_g). 3-6 mg specimens were first heated from 25 to 250 °C at a heating rate of 10 °C min^{-1} under N_2 atmosphere, and kept for 10 min to eliminate the heat history. Then, the samples were cooled to 25 °C at a rate of 10 °C min^{-1} , kept for another 10 min, and heated to 250 °C again at the rate of 10 °C min^{-1} . The thermal stability of F-PAEKs was obtained by thermo gravimetric analysis (TGA, TG209



Scheme 1. The schematic preparation process of F-PAEKs.

F3, Netzsch, Germany) over 50-800 °C with a heating rate of 10 °C min⁻¹ in N₂ atmosphere. The contact angle was measured by contact angle measurement (OCA20, PuSheng Instrument Company, China). The contact angle obtained by the average of five tests with 5 µL one droplet. The X-ray photoelectron spectroscopy instrument determined the chemical compositions of F-PAEKs (XPS, K-Alpha, Thermal Scientific, USA). Dielectric properties were measured at 25 °C and a frequency of 10³ Hz-10⁶ Hz using a dielectric analyzer (DMS500, Partulab, Germany). The standard scotch tape test (according to ASTM D3359)²² was used to evaluate the adhesion. The water uptake was weighed by the microbalance (model FA2104N, Shanghai Yiheng Technology Instrument Co., Ltd., China). PI and F-PAEK-2/PI films were dried at 60 °C for 12 h in vacuum. Dried films were dipped in double-distilled water, and the percentage of water absorption was calculated as equation 1:²³

$$\text{Water (\%)} = \frac{W_f - W_i}{W_i} \times 100 \quad (1)$$

where W_i and W_f are the initial and final weights of the films, respectively.

The diffusion measurement was according to the ASTM D-570,²⁴ and the appropriate solution of the diffusion coefficient given by Crank and Nicolson:²⁵

$$\frac{M_t}{M_\infty} = 4 \left(\frac{D_t}{l^2} \right)^{1/2} \left\{ \frac{1}{\sqrt{\pi}} + 2 \sum_{n=0}^{\infty} (-1)^n \text{ierfc} \left[\frac{nl}{2D_t^{1/2}} \right] \right\} \quad (2)$$

where M_t and M_∞ are the weights absorbed in time t and at equilibrium, respectively; D_t represents the diffusion coefficient at time t , l represents the coating thickness. This equation should be simplified:

$$\frac{M_t}{M_\infty} = \frac{4}{\sqrt{\pi}} \left(\frac{D_t}{l^2} \right)^{1/2} \quad (3)$$

The diffusion coefficient D_t based on the initial linear slope is calculated employing the equation 4 (m : the initial slope of the M_t/M_∞):

$$D = \frac{m^2 \pi l^2}{16} \quad (4)$$

Results and Discussion

Molecular structure of the F-PAEKs

To reveal the chemical structure of F-PAEKs, FTIR spectra of F-PAEKs are shown in Figure 1a. The characteristic peak of ester group of the phenolphthalein unit was at 1775 cm⁻¹. The absorptions at 1650 cm⁻¹ correspond to the stretching vibration of carbonyl groups in DFBP. The characteristic peak of the benzene ring occurs at 1589 cm⁻¹. The peak at 1020 cm⁻¹ is assigned to the asymmetric and symmetric stretching vibrations of C–O–C bonds. The peak at 1204 cm⁻¹ is attributed to –C–F stretching vibrations in BPAF, indicating the presence of the –F group in the F-PAEKs. And the characteristic peak of –C–F is not obtained in F-PAEK-0. The NMR characterization result of F-PAEKs is shown in Figure 1b, and it showed the positions of signal peaks that appeared as predicted, ranging from 7.05 to 8.00 ppm. This can be interpreted by the stronger effect of electron absorption of the –C–F group of BPAF and ester group of phenolphthalein, which appeared at a high chemical shift region of 7.71-7.90 ppm, respectively. Besides, –H next to the ether bonds showed relatively lower chemical shifts of 7.08-7.15 ppm, which confirmed this interpretation additionally. The ¹H NMR and FTIR spectra results confirmed that F-PAEKs were successfully synthesized.

The molecular weights and polydispersities (PDI) of F-PAEKs determined by GPC were summarized in Table 1. The molecular weights of F-PAEK were in the range of

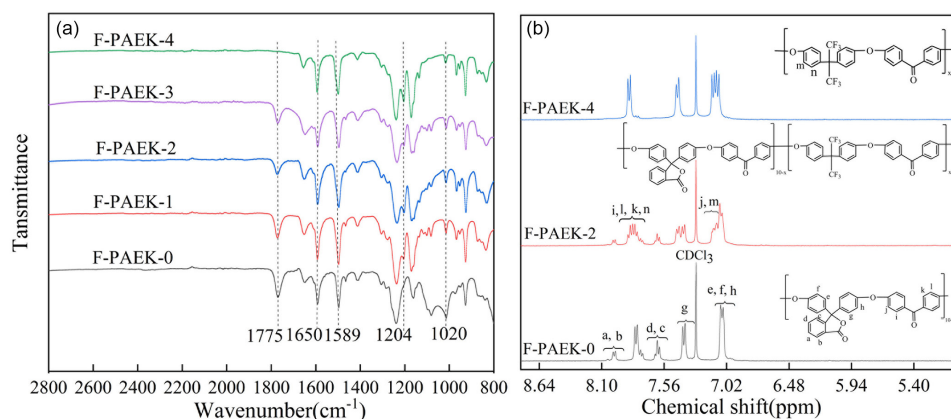


Figure 1. (a) FTIR-ATR and (b) ¹H NMR spectra of F-PAEKs.

Table 1. The molar ratio of reactants for F-PAEKs, molecular weight, and thermal properties of F-PAEKs

Sample	Mol(eq)		$M_n / (\text{g mol}^{-1})$	$M_w / (\text{g mol}^{-1})$	PDI	$T_g / ^\circ\text{C}$	$T_{5\%} / ^\circ\text{C}$	$T_{10\%} / ^\circ\text{C}$
	phenolphthalein/ BPAF/DFBP							
F-PAEK-0	10:0:10		29566	74215	1.680	205.69	436.28	472.13
F-PAEK-1	7.5:2.5:10		32174	77002	1.892	200.33	453.51	480.80
F-PAEK-2	5:5:10		38043	84587	1.736	180.06	446.48	497.77
F-PAEK-3	2.5:7.5:10		31132	79806	1.751	163.73	500.73	527.57
F-PAEK-4	0:10:10		36074	83889	1.854	135.53	453.02	511.87

BPAF: 2,2-bis-(4-hydroxyphenyl) hexafluoropropane; DFBP: 4,4-difluorobenzophenone; M_n : number-average molecular weight; M_w : weight-average molecular weight; PDI: polydispersity; T_g : glass transition temperature; $T_{5\%}$: temperature at 5% weight loss; $T_{10\%}$: temperature at 10% weight loss.

70000-85000, and the polymerization degree of F-PAEKs was shown in Table S2 (Supplementary Information (SI) section). From the results of FTIR, ^1H NMR, and GPC, it is confirmed that F-PAEKs were successfully synthesized.

Component analysis of F-PAEKs

XPS was utilized to expound the chemical compositions of F-PAEKs. It is evident that F-PAEKs contain the elements of F (688 eV, F1s), O (533 eV, O1s), and C (284.8 eV, C1s) as shown in Figure 2. It is shown that the C1s spectra of F-PAEK-0 contain C–C bond (284.8 eV), C–O–C bond (286 eV), and O–C=O bond (288.5 eV). The C1s spectra of F-PAEK-1-4 contain four peaks C–C (284.8 eV), C–O–C (286 eV), O–C=O (288.8 eV), and –C–F₃ (294 eV). This suggests that the fluorine group was successfully introduced into the polymer, and the XPS results were consistent with FTIR. As shown in Table 2, by increasing the addition of 2,2-bis-(4-hydroxyphenyl) hexafluoropropane, the content of fluorine increased. The high fluorine contents would reduce the surface free energy, resulting in the hydrophobicity of F-PAEKs.^{26,27}

Thermal properties of F-PAEKs

The thermal properties of the F-PAEKs were evaluated through TGA (Figure 3a) and DSC (Figure 3b); they are also listed in Table 1. The TGA curves of the F-PAEKs exhibited a single-step degradation profile (Figure 3a), and the weight loss was attributed to the degradation of the polymer backbone.²⁸⁻³⁰ The temperatures at 5% weight loss

($T_{5\%}$) of the F-PAEK samples were over 440 °C, while those at 10% weight loss ($T_{10\%}$) were in the range of 480-530 °C. Compared to previous literature (Table S4, SI section), F-PAEKs exhibited excellent thermal performance. These results demonstrate the high decomposition temperatures and thermal stability of F-PAEKs, which facilitate their application in high-temperature environments. The fluorine content of the F-PAEKs had a clear effect on T_g , which decreased with increasing fluorine content (Figure 3b). A reduction in the number of rigid phenolphthalein and incorporation of fluorine contributes to internal plasticization, resulting in reduced T_g values.

Hydrophobicity and adhesion properties of the F-PAEK/PI films

The hydrophobicity of the films is critical in preventing moisture absorption. The unmodified PI film is hydrophilic and exhibits a water contact angle of 75.89°. Coating the PI films with F-PAEKs imparts hydrophobicity to the PI film surface, which affects their wettability (Table 3). The adhesion between F-PAEKs and PI may be affected by the inertness of the PI surface; thus, the adhesion of the F-PAEK/PI films was analyzed using the tape test (Figure 4). The edges of the F-PAEK-2/PI film were completely smooth, with no flaking or detachment of the coating; thus, its adhesion was classified as 5B, the class with the highest adhesive strength as defined by the American Standard Test Method (ASTM). The adhesion of the F-PAEK-0/PI, F-PAEK-1/PI, and F-PAEK-3/PI films was classified as 2B, with coating removals of ca. 15%. The

Table 2. XPS data of the F-PAEKs

Sample	C1s		O1s		F1s	
	Position / eV	Atomic / %	Position / eV	Atomic / %	Position / eV	Atomic / %
F-PAEK-0	284.8	80.4	533.2	19.66		
F-PAEK-1	284.8	81.6	531.5	16.87	685.3	1.6
F-PAEK-2	284.8	77.4	532.1	14.24	688.0	8.4
F-PAEK-3	284.8	75.1	532.2	13.43	687.9	11.54
F-PAEK-4	284.8	73.13	532.3	10.56	688.1	16.30

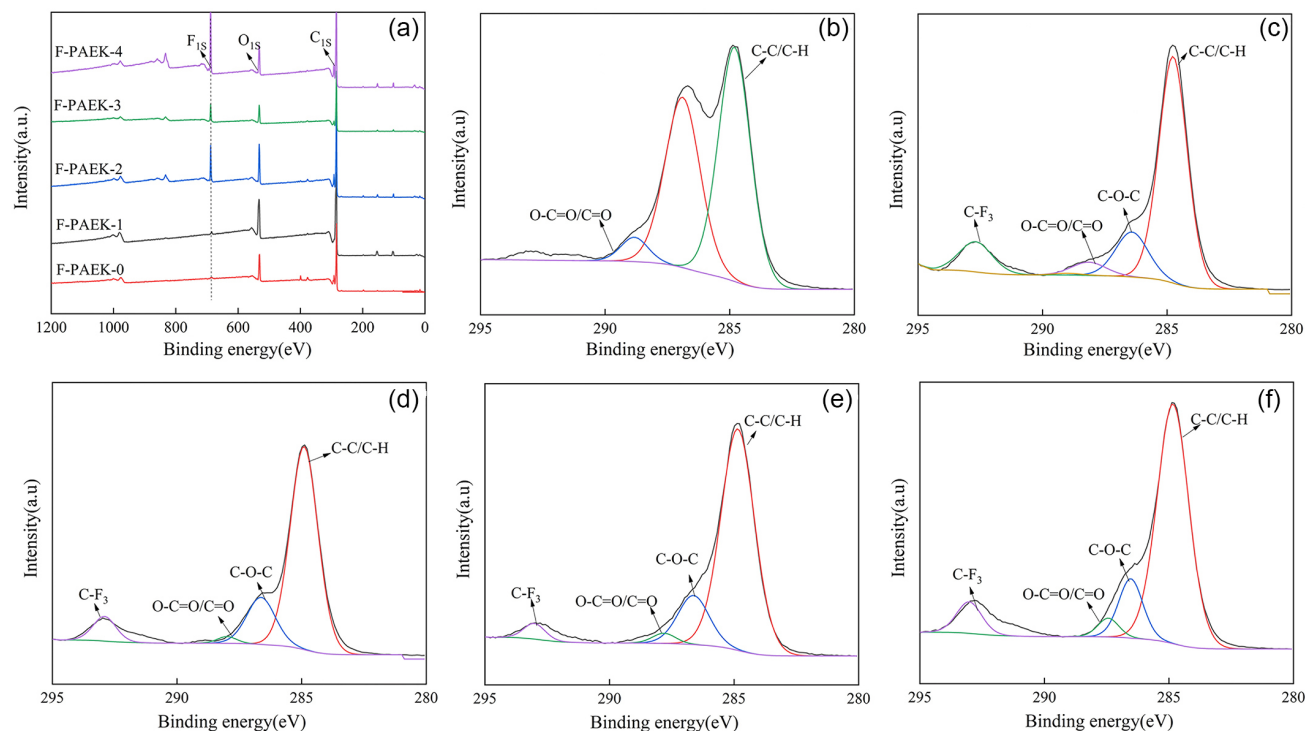


Figure 2. (a) XPS spectra of F-PAEKs, (b) C1s spectra of F-PAEK-0, (c) C1s spectra of F-PAEK-1, (d) C1s spectra of F-PAEK-2, (e) C1s spectra of F-PAEK-3, (f) C1s spectra of F-PAEK-4.

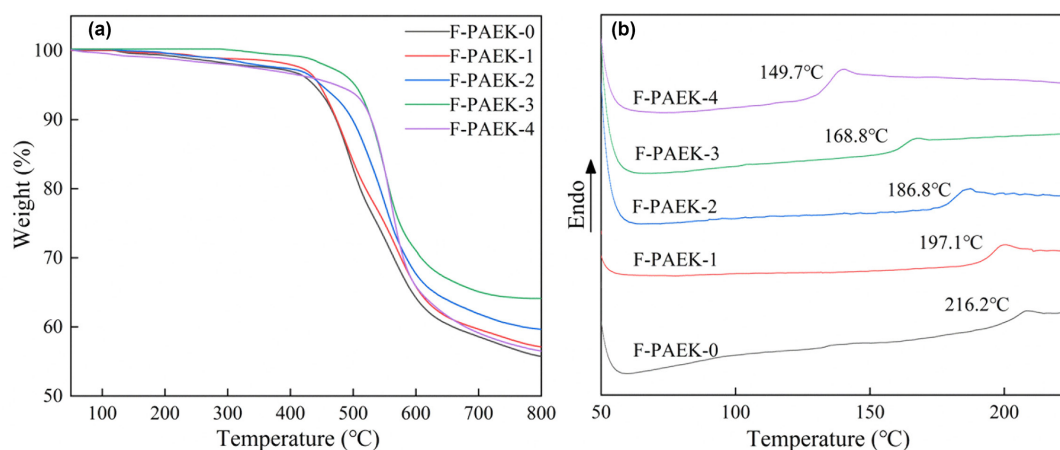


Figure 3. (a) TGA curves; (b) DSC curves of F-PAEKs.

F-PAEK-4/PI film exhibited the weakest adhesion, with a coating removal of ca. 30%.

Strong adhesion is attributed to noncovalent interactions and two surfaces with similar surface free energies. On the one hand, π - π stacking interactions between F-PAEK and PI would enhance the entanglement. Interactions between the π -rich 2D surfaces and polymers having aromatic groups have been already reported.^{31,32} During coating, the swelling behavior happened on the surface of PI films. F-PAEK entered the PI film interfaces, and the entanglement of F-PAEK and PI occurred via π - π stacking interactions. T_g increases with the number of aromatic

groups, thereby inhibiting the movement of the chain and weakening the entanglement effect. F-PAEK-2 exhibited the best adhesion on PI film, since it has balanced the π - π stacking interactions and T_g .

On the other hand, the similarity between the surface free energies of the adhered surfaces is required to ensure strong adhesion. Bao *et al.*³³ demonstrated that a significant difference in the surface free energy (γ_s) resulted in high interfacial tension, which adversely affected the adhesion between the coating film and substrates. γ_s can be estimated by the van Oss-Chaudhury-Good model:³⁴

$$\gamma_i = \gamma_i^{LW} + \gamma_i^{AB} = \gamma_i^{LW} + 2(\gamma_i^+ \gamma_i^-)^{0.5} \quad (5)$$

where γ_i^{LW} : the Lifshitz-van der Waals (LW) component of surface free energy; γ_i^{AB} : the polar acid-base component of surface free energy from the Lewis theory; γ_i^+ : the parameter of the surface free energy of a Lewis acid; γ_i^- : the parameter of the surface free energy of Lewis base; i = S: represents solid, i = L represents liquid.

The surface free energy of the model liquids can be calculated by measuring the contact angle. Water, ethylene glycol, and diiodomethane were used as model liquids, representing polar, semi-polar, and non-polar solvents, respectively, to measure the wetting angles. The surface energy parameters of these liquids were obtained from literature and their apparent contact angle on solid surfaces are listed in Tables 3^{33,34} and 4, respectively. Table 3 also tabulates the surface free energies of the PI film and F-PAEKs, which were calculated using equation 5. The surface free energy of the F-PAEK-2 film is similar to that of the unmodified PI film; thus, the strongest adhesion exists between F-PAEK-2 and the PI film.

Moisture absorption, durability and dielectricity

Hydrophobic modification prevents moisture absorption and PI aging. The effect of hydrophobic modification was investigated using F-PAEK-2/PI owing to its optimal adhesive properties. Figure 5 shows the moisture absorption isotherms and diffusion mechanisms of the PI and F-PAEK-2/PI films. The moisture absorption and diffusion rate of the F-PAEK-2/PI film were lower than those of the PI film, demonstrating that hydrophobic

Table 3. Contact angle (θ) for standard liquid and the surface free energy parameters of PI and F-PAEKs films

Sample	θ_w / degree	θ_E / degree	θ_D / degree	γ_s / (mJ m ⁻²)
PI	75.89	62.89	36.81	35.97
F-PAEK-0	104.81	84.17	41.09	37.11
F-PAEK-1	122.33	98.15	47.37	37.65
F-PAEK-2	128.50	96.26	53.49	35.42
F-PAEK-3	134.25	90.59	56.57	32.39
F-PAEK-4	140.35	81.75	66.15	18.12

W: water; E: ethylene glycol; D: diiodomethane; γ_s : surface free energy.

Table 4. Surface free energy of standard liquids³³

Liquid	γ_s / (mJ m ⁻²)	γ_s^{LW} / (mJ m ⁻²)	γ_s^+ / (mJ m ⁻²)	γ_s^- / (mJ m ⁻²)
Water	72.8	21.8	25.5	25.5
Ethylene glycol	48.0	29.0	1.92	47
Diiodomethane	50.8	50.8	0	0

γ_s : surface free energy; γ_s^{LW} : Lifshitz-van der Waals (LW) component of surface free energy; γ_s^+ : parameter of the surface free energy of a Lewis acid; γ_s^- : parameter of the surface free energy of Lewis base; i = S: represents solid, i = L represents liquid.

modification of the PI films effectively impeded moisture absorption.

The durability of the F-PAEK-2/PI film was also investigated. The F-PAEK-2/PI film, which was stored at room temperature for three months, exhibited a water contact angle of 125.34° (Figure 6b). Thermal and subzero treatments were conducted to examine the durability of the F-PAEK-2/PI film. The F-PAEK-2/PI film retained its hydrophobicity after heating at 300 °C for 1 h, and exhibited a water contact angle of 110.20°. The F-PAEK film exhibited no mass loss below 400 °C owing to its

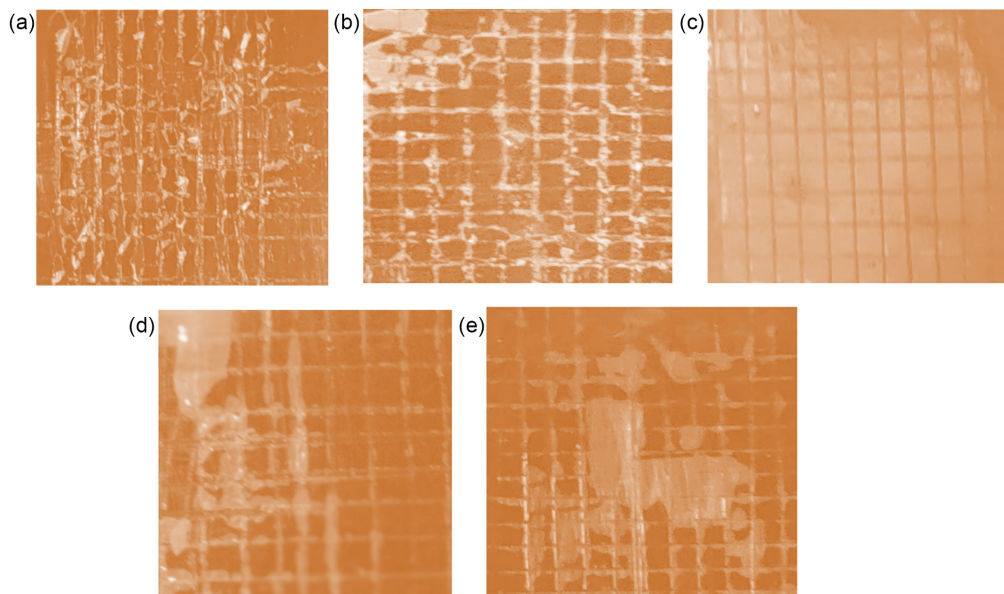


Figure 4. Images of scotch tape test of F-PAEKs on PI films: (a) F-PAEK-0, (b) F-PAEK-1, (c) F-PAEK-2, (d) F-PAEK-3, (e) F-PAEK-4.

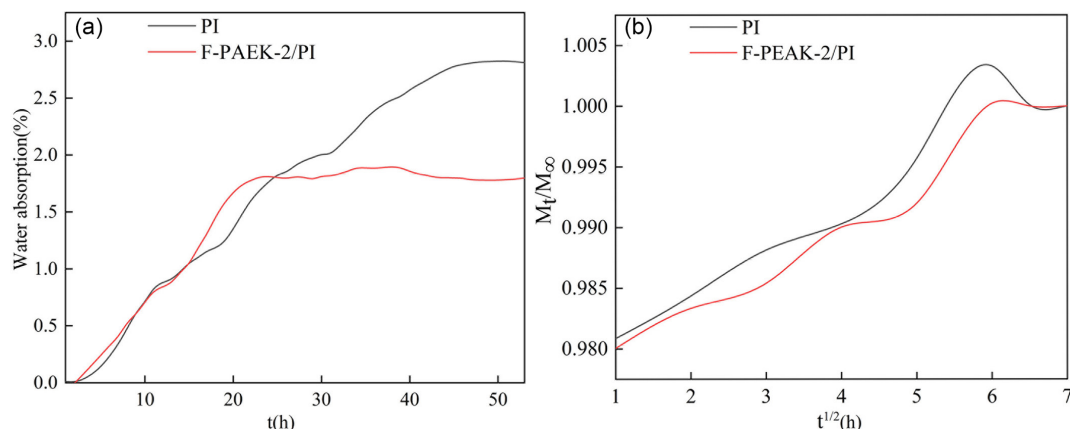


Figure 5. (a) Water absorption isotherms of the PI and F-PAEK-2/PI film; (b) diffusion mechanisms as a function of square root of time for the PI and F-PAEK-2/PI films.

high thermal stability (Figure 3a). Subzero treatment was performed by immersing the F-PAEK-2/PI film in liquid nitrogen to evaluate the stability of its hydrophobicity under low-temperature conditions. The F-PAEK-2/PI film exhibited a water contact angle of 119.13° , after the subzero treatment, and retained its hydrophobicity. These results indicate that the hydrophobicity of the F-PAEK-2/PI film is not affected under extreme temperature conditions.

PI is widely applied in engineering fields, due to its low dielectric properties. Therefore, we investigated whether the dielectric constant of the PI film was affected by modification with F-PAEK. The dielectric properties of the PI and F-PAEK-2/PI films were evaluated in the frequency range of 10^3 - 10^6 Hz (Figure 7). The F-PAEK-2/PI films exhibited a lower dielectric constant ($1.425 @ 10$ GHz) and

dielectric loss ($0.016 @ 10$ GHz) than the unmodified PI films. However, modification with F-PAEK did not affect the dielectricity of the PI film in the investigated frequency range.

Conclusions

F-PAEKs have been successfully synthesized and coated onto the surface of PI films. The T_g values of the F-PAEKs decreased with increasing fluorine content, while the thermal stability and hydrophobicity increased. The coating of the PI films with F-PAEKs imparted hydrophobicity to the film surface. The adhesion between F-PAEK-2 and the PI film is optimal, due to the similarity of surface free energy. The water contact angle of F-PAEK-2/PI was 128° . The coated films maintained their hydrophobicity after both after low-

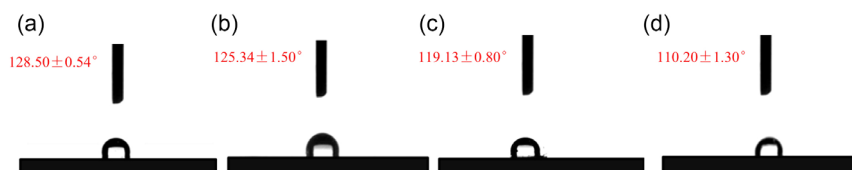


Figure 6. The contact angle of F-PAEK-2/PI (a) un-treated, (b) after 3 months, (c) after thermal treatment, (d) after subzero treatment.

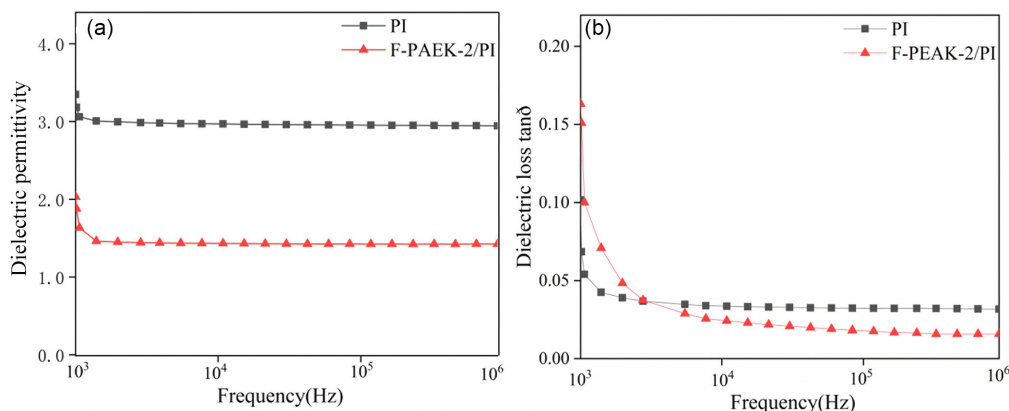


Figure 7. Electrical characterization of PI and F-PAEK-2/PI: (a) dielectric permittivity, (b) dielectric loss.

and high-temperature treatment, and the water contact angle stabilized at approximately 110°. The coated films exhibited lower moisture absorption, and a low dielectric constant. With these favorable properties, the PI films modified with hydrophobic F-PAEK possesses excellent potential in spacecraft devices, solar cells, and circuit boards.

Supplementary Information

Supplementary data are available free of charge at <http://jbcbs.s bq.org.br> as PDF file.

Acknowledgments

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