Polycarbonate Composites Flame-Retarded by Polyphenylsilsesquioxane of Ladder Structure

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ABSTRACT: Polyphenylsilsesquioxane (PPSQ) of ladder structure synthesized in laboratory was incorporated into polycarbonate (PC) by means of a twin-screw extruder. Scanning electron microscopy (SEM) observations showed that spherical PPSQ particles of size $0.5-1.5 \mu$ m dispersed homogeneously in PC. The incorporation of PPSQ into PC improved the elongation at break and flexural strength. Thermal gravimetric analysis and Fourier transform infrared spectroscopy revealed that PPSQ made the initial thermal decomposition of PC happen fast. The early cleavages of C–C and C–O bonds in the PC chains induced crosslinking reaction in the condensed phase char. According to the cone calorimetry (CONE) tests, loading of 8 wt % PPSQ reduced the peak heat release rate of PC composites from 570 to 153 kW/m². The chars obtained after CONE testing of PC/PPSQ composites showed continuous, smooth, flaky, and cascading char and SiO₂ ceramic layer on the top. These indicated the good flame retardancy of PC/PPSQ, that is the attainment of a UL-94 rating of V-0 (1.6 mm), and limiting oxygen index of 42.0% when PPSQ was in 8 wt % in PC. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 4381–4388, 2012

Key words: polycarbonate; ladder polyphenylsilsesquioxane; thermal property; mechanical property; flame retardancy; char structure

INTRODUCTION

Polycarbonate (PC) usually refers to bisphenol A PC, the molecular structure of which contains flexible carbonate units and rigid benzene rings. It is an amorphous polymer with a series of advantages, including transparency, good electrical insulation, excellent dimensional stability, high impact strength, high heat distortion temperature (HDT), and high glass transition temperature (T_g). These features have made it the fastest developing engineering plastic and a subject of ongoing research interest in many industrial nations.^{1,2}

PC has a V-2 rating in the UL-94 test. However, more rigorous flame-retardant performance is often required for electrical and electronic, transportation, and building material applications, so the flame retardation of PC must be improved.³ Because of environmental concerns, halogenated flame retardants have been gradually prohibited. Accordingly, phosphorus-based flame retardants have been widely used in PC owing to their relative environmental friendliness.⁴⁻⁶

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However, phosphorus-containing flame retardants are prone to hydrolysis, and partially organic phosphonate flame retardants exert a plasticizing effect, which will significantly reduce the HDT and impact strength of PC.

Studies of the effectiveness of silicone flame retardants in PC date back to the 1980s and have mainly concerned polysilanes, polysiloxanes, polysilsesquioxanes and so on. Among these, poly(organic-silsesquioxane)s are a class of polysiloxanes with the formula $(RSiO_{1,5})_n$, where R is hydrogen or any alkyl, alkylene, aryl, or arylene group, or even an organicfunctional derivative of such groups. They combine the properties of inorganic compounds and organic polymers⁷ and can be blended with various organic polymers to form organic-inorganic hybrid reinforced polymers.^{8–10} In recent years, the use of polyhedral oligomeric silsesquioxane (POSS) as a flame retardant in PC has been the subject of much research interest. For instance, Li and Yang¹¹ studied the mechanical and combustion properties of PC/POSS and concluded that polyhedral oligomeric octaphenyl silsesquioxane (OPS) could improve the flame retardancy of PC composites by enhancing charring. Zhao and Schiraldi¹² observed that the mechanical properties of PC, including its tensile and dynamic mechanical moduli, were slightly enhanced by a loading of trisilanolphenyl-POSS (TPOSS). Song and Hu¹³ pointed out that the presence of TPOSS significantly affected the thermal degradation process of PC and

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Figure 1 Typical chemical structure of polyphenylsilsesquioxane (PPSQ).

lowered the peak heat release rate (PHRR) of the hybrids. In addition, a study by Wang and Xin¹⁴ on the PC/polysilsesquioxane system revealed that polymethylphenylsilsesquioxane (PMPSQ) microspheres incorporated into PC could induce crosslinking reactions and accelerate the thermal degradation of the polymer. At the same time, the PMPSQ microspheres caused a significant increase in impact strength and decreases in the flexural modulus and elongation at break of PC. Application of ladder polyphenylsilsesquioxane (PPSQ) in the flame retardation of PC has not hitherto been reported.

PPSQ is a ladder polymer with a stereoregular double Si—O chain structure (Fig. 1).^{15–17} We have successfully synthesized PPSQ by a new method.¹⁸ PPSQ has good thermal stability; its temperature at 5% weight loss in nitrogen is 521°C, and the residue char yield at 800°C is 77.18%. In the work described herein, flame-retardant composites of PC with PPSQ synthesized in the laboratory have been prepared through blending in the molten state. The effects of PPSQ on the mechanical properties, thermal stabilities, and flame retardancies of PC/PPSQ composites have been studied. The mechanism of the flame-retarding action of PPSQ on PC is also discussed.

EXPERIMENTAL

Materials

Phenyltrichlorosilane (>99%) was purchased from Dalian Yuanyong Organosilicon Plant, China. Tetramethylammonium hydroxide solution (N(CH₃)₄OH) was purchased from Xingfu Fine Chemicals Research Institute, China. Anhydrous aluminium trichloride (AlCl₃), benzene, methanol, ethanol, and hexane were purchased from Beijing Chemical Reagent Company and were used without further purification. PC (Makrolon 2805) was purchased from Bayer. Prior to processing, the polymer pellets were dried for at least 10 h under vacuum at 120°C.

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Synthesis of PPSQ

PPSQ was synthesized in our laboratory by the hydrolysis and condensation of phenyltrichlorosilane. Phenyltrichlorosilane (21.2 g) was hydrolyzed in benzene (100 mL) by the dropwise addition of water (200 mL) at 0°C. AlCl₃ (2.23 g) was dissolved in water (10 mL) to prepare an aqueous solution. The hydrolysate in the organic phase was mixed with the aqueous solution of AlCl₃ by stirring, the mixture was heated to 80°C, and then a methanolic solution of N(CH₃)₄OH (3 g) of a certain concentration was added. The condensation reaction in the solution was allowed to proceed for 24 h under vigorous stirring. When the reaction was complete, the filtrate was collected. A white precipitate was obtained upon addition of ethanol to the filtrate. This precipitate was collected by filtration, washed with hexane, and dried in a vacuum oven at 100°C. The final yield of PPSQ was 7.64 g.

The diameters of the spherical PPSQ particles were 0.5-1.5 µm. Gel permeation chromatography (GPC): $M_n = 5183$, polydispersity = 1.10. GPC was measured by means of Waters gel chromatography (Water 1515 HPLC pump and Waters 2414 RI detector) using polystyrene as a standard sample and tetrahydrofuran as mobile phase at 1 mL/min. Fourier transform infrared spectra (FTIR): v = 3050, 1594,1434 cm⁻¹ (phenyl), v = 1100, 1021 cm⁻¹ (Si–O–Si), $v = 580-780 \text{ cm}^{-1}$ (C–C, C–Si). Raman: v = 3053, 1000 cm⁻¹ (Ar-H), v = 1594 cm⁻¹ (phenyl). ¹H-NMR (C₆D₆, δ, ppm, TMS): 7.74, 7.15, 6.98 (H in phenyl). $^{29}\text{Si-NMR}$ (C₆D₆, δ , ppm): -82 to -76. ¹H-NMR and ²⁹Si-NMR spectra were recorded on a Bruker AV600 instrument with a pulse width of 5 µs and a pulse delay of 5 s. XRD (20): 7.3°, 19.4°. These results show that the PPSQ synthesized had a high content of ladder polymer without hydroxyl groups.¹⁸ The ²⁹Si-NMR spectra of PPSQ were shown in Figure 2.



Figure 2 ²⁹Si-NMR spectra of PPSQ.

Sample	Tensile strength (MPa)	Elongation at break (%)	Flexural strength (MPa)	Flexural modulus (Mpa)			
PC control	60.8 ± 0.6	101 ± 3.0	91.4 ± 0.7	2589 ± 22			
2 wt % PPSQ	60.2 ± 0.6	101 ± 4.8	98.8 ± 0.5	2886 ± 23			
4 wt % PPSQ	58.6 ± 0.8	105 ± 5.0	97.9 ± 1.0	2896 ± 23			
6 wt % PPSQ	57.9 ± 0.5	109 ± 4.3	96.8 ± 1.0	2883 ± 10			
8 wt % PPSQ	56.1 ± 0.5	111 ± 3.3	90.2 ± 1.4	2631 ± 22			

TABLE I Mechanical Properties of PC and PC/PPSQ Composites

Preparation of PC/PPSQ composites

PC was blended with different levels of PPSQ, 0.3% polytetrafluoroethylene (PTFE) as a melt-dripping inhibitor, and a small amount of antioxidant, using an SJ-20 twin-screw extruder (Nanjing Giant Machinery). The blending was carried out at 255°C and a screw speed of 100 rpm, and the composites were finally cut into pellets. Samples for testing were molded by means of an injection-molding machine (HTF90X1, Haitian Plastics Machinery) at 260°C.

Measurements

DSC curves of the PC/PPSQ composites were measured using a Netzsch 204 F1 differential scanning calorimeter. Samples (5–10 mg) were tested at a heating rate of 10° C/min and results from the second heating in the range 25 to 180° C are reported.

HDT was measured by means of a ZWK1302-A testing machine (MTS Systems, China) according to GB/T 1634.2-04.

The mechanical properties were measured by means of universal testing machines (DXLL-5000, Shanghai D&G machinery equipment and CMT-4104, MTS Systems, China) according to ASTM test methods, such as D-638 for the tensile testing and D-790 for the flexural testing.

Scanning electron microscopy (SEM) experiments were performed with a Hitachi S650 scanning electron microscope. Samples for SEM were prepared by low-temperature fracturing and sputtering the surface with gold. Main accessories: X-ray energy spectrometer (EDAX9100).

Limiting oxygen indices (LOI) were obtained according to the standard GB/T 2406–93. An oxygen index instrument (Rheometric Scientific) was used. Dimension of the sample is $100 \times 6.5 \times 3 \text{ mm}^3$.

Vertical burning tests were performed according to UL-94 standard with samples of dimensions 125 \times 12.5 \times 3.2 mm³ and 125 \times 12.5 \times 1.6 mm³.

Combustion experiments were performed with a cone calorimeter (CONE, Fire Testing Technology) according to the standard GB/T 16172-2007. Samples of dimensions $100 \times 100 \times 3 \text{ mm}^3$ were exposed to a radiant cone (50 kW/m²). Three time measurements for every composite were achieved to obtain three combustion curves. A combustion curve at

middle level was chosen to make comparison among the composites.

Thermal gravimetric analysis (TGA) was performed with a Netzsch 209 F1 thermal analyzer at a heating rate of 10°C/min under a nitrogen atmosphere over the range from room temperature to 800°C.

TG-FTIR coupling: the sample (5 mg) was placed in the oven of Netzsch 209 F1 thermal analyzer. The temperature of the oven was raised at a heating rate of 10°C/min under a nitrogen atmosphere over the range from room temperature to 800°C. The dynamic FTIR spectra for gaseous products were obtained *in situ* during the thermal degradation processes. The real-time FTIR spectra were recorded using a Thermofisher 6700 spectrometer at a resolution of 4 cm⁻¹ with a total of 32 scans. The baseline correction was also based on 32-background scanning.

RESULTS AND DISCUSSION

Mechanical properties and morphology

The mechanical properties of the PC/PPSQ composites are shown in Table I. The tensile strength of PC is slightly decreased by the addition of PPSQ. It is worth emphasizing that PPSQ loading slightly increases the elongation at break of PC. This is a very valuable result because, in general, flame-retarded PC usually has a lowered elongation at break.^{11,14,19} In addition, with the exception of PC/8 wt% PPSQ, the flexural strengths and moduli of the PC/PPSQ composites are higher than those of the PC control. Evidently, the incorporation of PPSQ into PC enhances the toughness of the polymer.

There is a close relationship between the microstructure and the macroscopic properties of composites. In polymer composites filled with particles, the size, shape, distribution of the dispersed particles, and phase interface play important roles in determining the mechanical properties.²⁰ Figure 3 shows that spherical PPSQ particles are uniformly dispersed in a PC matrix. The diameters of the dispersed particles are in the range $0.5-1.5 \mu$ m. However, it was noted that there is a lack of interfacial adhesion as the particles were dispersed, that is, the PPSQ particles were not bonded significantly to the PC phase. It is suggested that the submicron size and uniform dispersion of the spherical PPSQ



Figure 3 SEM micrographs of fracture surfaces of PPSQ/PC composites.

particles in PC may contribute to the good mechanical properties, as well fire retardancy of the composites. The good dispersion of PPSQ particles means good compatibility between PPSQ and PC; however, weak interfacial adhesion makes it be doubtful. It is thought that lack of the interfacial adhesion may be due to difference between thermal expansion coefficients of PPSQ particles and PC matrix. In addition,



Figure 4 DSC curves of PC and PC/PPSQ composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the mechanism through which PPSQ improves the toughness of PC is worthy of further research.

Thermal properties and thermal stability

The PPSQ chain shows rigidity because its ladder structure limits chain movement. DSC analysis showed that PPSQ samples synthesized in the laboratory do not exhibit a glass transition between room temperature and 350°C. A slight decrease in T_g of PC/PPSQ composites was observed compared with that of pure PC; see Figure 4. For PC/PPSQ with 8 wt % PPSQ, T_g decreased by just 1.3°C.

Table II lists the HDTs of PC and the PC/PPSQ samples. It can be seen that the HDTs of PC/PPSQ composites are slightly higher than that of PC itself. These are advantageous to applications of flame-re-tarded PC at high temperature.

TABLE II DSC and HDT Results for PC and PC/PPSQ Composites

Sample	T_g (°C; by DSC)	HDT (°C; 0.45 MPa)
PC control	145.1	133.0
2 wt% PPSQ	144.7	135.1
4 wt% PPSQ	144.2	135.0
6 wt% PPSQ	144.0	134.6
8 wt% PPSQ	143.8	134.0



Figure 5 TGA curves of PC and PC/PPSQ composites in N_2 .

The above results indicated that PPSQ does not have an obvious effect on the T_g and HDT of PC. The latter may be attributed to the stable ladder structure of PPSQ. On the one hand, the Si—O bond is not easy to break due to the high bond energy (422.5 kJ/mol)²¹; on the other hand, local chemical bond breakages within the ladder structure of the PPSQ chain do not have a significant impact on the structural integrity of the whole chain. Some improvement in thermal stability imparted to PC may correlate with the finely dispersed PPSQ rigid particles within the matrix.

Figure 5 shows the TGA curves of PC and the PC/PPSQ composites in nitrogen atmospheres. PPSQ seems to have little influence on the thermal decomposition of PC, and the residue from the PC composites at 800°C is increased a little by the addition of PPSQ.

In order to get a better understanding of the very interesting differences between PC and the PC/ PPSQ composites in their decompositions at high temperature, the combined TG-FTIR method was used to analyze how PPSQ affects the release of the volatile products from the thermal decomposition of PC. Figure 6 shows the 3D FTIR spectra of the gaseous products from PC and PC/PPSQ with time (temperature) in TG experiments.

PC has a high degree of thermal stability and undergoes little decomposition below 250°C. At higher temperatures, the main important volatile degradation products are carbon dioxide (CO2) and bisphenol A.²² In Figure 6 (3D-1), in the spectra for the PC, the strong peak at 2340 cm^{-1} is indicative of a substantial release of CO_2 ; the peak at 1783 cm⁻¹ may be attributed to the stretching vibration of the ester groups from the cracking of PC chains; and the peaks at 1187 and 1248 cm⁻¹ indicate the presence of isopropyl groups. Moreover, the absorbance bands at 1602 and 1511 cm⁻¹ can be attributed to aromatic rings. Similarly, thermal decomposition products of the PC/PPSQ composite had same FTIR characteristic absorbance bands in Figure 6 (3D-2). The most significant releases of gaseous products are seen to occur at the nearly same time for PC and PC/PPSQ.

However, it should be noted that there are some differences in the amounts of the volatile degradation products between PC and PC/PPSQ. Relative to the main absorbance peak due to CO_2 , the absorbance peaks of ester groups, aromatic rings, and isopropyl groups at 1783, 1602, 1511, 1187, and 1248 cm⁻¹ are obviously stronger for PC/PPSQ than for PC. In addition, after the strongest absorbance peaks, the releases of CO_2 and aromatic species with time (temperature) for the PC/PPSQ are significantly lower than that for PC.

These results may indicate that PPSQ made the initial thermal decomposition of PC happen fast but the following slow. The early cleavages of C—C and C—O bonds in the PC chains contribute to the strong releases of CO₂, ester, and aromatic species, induce crosslinking reaction in the condensed phase char and, moreover, make the late thermal decomposition



Figure 6 FTIR spectra of the gaseous products from PC (3D-1) and from 8 wt % PPSQ /PC (3D-2) (N₂). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

 TABLE III

 Cone Calorimeter Data for PC and PC/PPSQ Composites

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wt % PPSQ	6 wt % PPSQ	8 wt % PPSQ
60	57	60
221.9	212.2	152.9
63.0	55.0	34.5
2170.4	1745.7	1472.4
0.13	0.11	0.11
2.41	2.39	1.74
	wt % PPSQ 60 221.9 63.0 2170.4 0.13 2.41	wt % PPSQ 6 wt % PPSQ 60 57 221.9 212.2 63.0 55.0 2170.4 1745.7 0.13 0.11 2.41 2.39

of the PC slow down. These features are very beneficial in flame-retarding PC.

Fire resistance performance

The combustion of PC and the PC/PPSQ composites has been studied by cone calorimetry (CONE). Several combustion parameters can be obtained from CONE, such as time to ignition (TTI), heat release rate (HRR), PHRR, total heat released, peak-specific extinction area (PK-SEA), effective heat of combustion, total suspended particles (TSP), and the CO and CO₂ emissions. CONE testing results for PC and the PC/PPSQ composites are shown in Table III and Figures 7 and 8. It is found that the TTI values of the PC/PPSQ composite are reduced compared with that of PC, and 8 wt % PPSQ significantly lowers the PHRR of PC from 570 to 153 kW/m². Significantly low PK-SEA, TSP, and the average CO and CO₂ emissions of PC/PPSQ composites are observed as well. These mean low smoke and harmful gas emissions in a fire.

Reduction of TTI as shown in Table III can correlate with the initial thermal decomposition of PC accelerated by PPSQ uncovered by TG-FTIR analysis in Figure 6. Furthermore, it is considered that fast initial decomposition reactions of PC support cross-



Figure 7 Heat release rate (HRR) of PC and PC/PPSQ composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

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linking reactions in the condensed phase, which help in the formation of the compact char and make PHRR decrease.

From Figures 7 and 8, it should be noted that the heat and smoke release of PC/PPSQ composites can be divided into two stages: the first is from TTI to 250 s, during which the heat and smoke release rate is very fast; the second is from about 250 to 500 s, during which the heat and smoke releases are relatively weak processes. During the first combustion stage, PPSQ may promote crosslinking and charring of the molten PC, thereby significantly reducing the exothermic combustion reactions of combustible gases and hindering the bubbling over of combustible gases from the decomposition of PC. However, the initial char may be broken down with increase of temperature and gas pressure inside the char layer. The breakage of the initial char layer results in the bubbling over and combustion of combustible gases, which contributes to the second relatively weak HRR and TSP processes as shown in Figures 7 and 8. When the content of PPSQ in PC is at 8 wt % PPSQ, then the collection of combustible gases in the dense char is very limited during the first stage, and thus the releases of heat and smoke in the second combustion stage are not so evident in CONE testing.



Figure 8 Total suspended particle (TSP) of PC and PC/ PPSQ composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



Figure 9 Optical photographs of the chars from PC and PC/PPSQ following cone calorimeter testing. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The charring of PC enhanced by PPSQ is evidenced by the optical photographs of the chars from the CONE tests shown in Figure 9. The char from the PC is collapsing and loose in small quantity. The chars from the PC/PPSQ composites are upstanding and intumescent in large quantity. In addition, on the top and surface of the chars formed by the PC/ PPSQ there exist white residue where X-ray energy spectral analysis showed that the fractions of Si, O, and C atoms are about 28.24, 71.76, and \sim 0%. They are attributed to SiO₂. In the inner of the char, the fractions of Si, O, and C atoms are 1.35, 13.11, and 85.54%, respectively, indicating the carbonaceous products. Such ceramic surface is very helpful to insulate transfer of heat and substances, which contributes to good flame retardancy of the PC/PPSQ.

Figure 10 shows SEM images of the inner chars from PC/PPSQ from cone calorimeter tests, which reveal a continuous, smooth, flaky, and cascading char structure. Comparing the char from the 4 wt % PPSQ/PC with that from the 8 wt % PPSQ/PC, we can seen that there are some holes in the former, whereas the latter is continuous and compact without holes. These char layers play an important role in preventing the transmission of heat, gas, and particulates and, thus, lower the heat and smoke release.

As a whole, PPSQ accelerates initial thermal decomposition of PC, induces crosslinking reactions in condensed phase, which results in intumescent and compact inner carbonaceous char and SiO_2 ceramic layer on the top of the char. Therefore, PPSQ loading made PC have good flame retardancy.

LOI and vertical burning classifications (UL-94) for PC and the PC/PPSQ composites are listed in Table IV. As can be seen, 4 wt % PPSQ modifies PC to the point of being nonflammable (LOI = 32.7%), and the LOI of PC with 8 wt % PPSQ increases by 16% (LOI = 42.0%). Correspondingly, PC composites of thickness 3.2 and 1.6 mm attain UL-94 ratings of V-0 by addition of at least 4 and 6 wt % PPSQ, respectively. LOI values and the UL-94 rating indicate that the flame retardancy of PC is effectively improved by PPSQ.

As a comparison, when using OPS with a cage structure as a flame-retardant for PC,¹¹ the LOI of the PC/OPS composite with 8 wt % OPS was found to be 36.0% and the PHRR was about 300 kW/m². This indicates that PPSQ with a ladder structure is



Figure 10 SEM images of the inner chars of PC/PPSQ following cone calorimeter testing.

TABLE IV
Flame Retardancy of PC and PC/PPSQ Composites

	LOI (%)	UL-94	
Sample		3.2 mm	1.6 mm
PC control	26.0	V-2	NR
2 wt % PPSQ	27.6	V-1	V-2
4 wt % PPSQ	32.7	V-0	V-1
6 wt % PPSQ	39.8	V-0	V-0
8 wt % PPSQ	42.0	V-0	V-0

more effective than OPS with a cage structure in flame-retarding PC.

CONCLUSIONS

PC/PPSQ composites have been prepared by means of a twin-screw extruder. It was observed by SEM that spherical PPSQ particles of size $0.5-1.5 \mu m$ disperse homogeneously in PC. For the mechanical properties, the incorporation of PPSQ into PC improved the elongation at break and flexural strength, but as a consequence, the tensile strength was marginally reduced.

The PPSQ did not have an evident influence on the thermal properties, including the T_g and HDT. TG-FTIR analysis showed that PPSQ made the initial thermal decomposition of PC happen fast. The early cleavages of C—C and C—O bonds in the PC chains induced crosslinking reaction in the condensed phase char and, moreover, made the late thermal decomposition of the PC slow down.

According to CONE tests, PPSQ loading reduced the TTI of the PC/PPSQ; the PHRR was reduced from 570 to 153 kW/m² by the addition of 8 wt % PPSQ; and the smoke release was greatly suppressed by PPSQ; significantly low PK-SEA, TSP, and the average CO and CO₂ emissions of PC/PPSQ composites were observed as well. According to SEM observations of the char layer from CONE tests, PPSQ made PC form a flaky and cascading char and SiO₂ ceramic layer on the top. It should be pointed out that for the PC/PPSQ, the fast initial thermal decomposition, crosslinking reactions in condensed phase, formation of the compact inner carbonaceous char, and SiO_2 ceramic layer contribute to the good flame retardacy of PC. In fact, the LOI of PC was raised from 26.0 to 42.0% by the addition of 8 wt % PPSQ; UL-94 tests indicated that at least 4 and 6 wt % loadings of PPSQ made PC composites of thicknesses 3.2 and 1.6 mm, respectively, attain ratings of V-0.

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