Preparation and applications of the tertiary copolymer poly(ethylene glycol) methacrylate/methyl methacrylate/diethyl allylphosphonate

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ABSTRACT: A novel poly(acrylonitrile-*co*-butadiene-*co*-styrene) (ABS) antistatic and flame-retardant agent, poly(ethylene glycol) methacrylate/methyl methacrylate/diethyl allylphosphonate (PMMD), was synthesized from poly(ethylene glycol) methacrylate, methyl methacrylate, and diethyl allylphosphonate by free-radical precipitation polymerization in the aqueous phase to improve the antistatic and flame-retardant performance at the same time. Through adjustments of the molar ratios of the three monomers, various antistatic, flame-retardant copolymers (PMMD) were synthesized. The molecular structure and thermal stability of PMMD were analyzed with Fourier transform infrared spectroscopy and thermogravimetric analysis. The electrical resistivity and flame-retardant and mechanical properties of the ABS/PMMD composites were analyzed by a ZC90 megohmmeter, an oxygen index meter, a vertical burning tester, a memory impact testing machine, and a tensile testing machine. The morphology of PMMD in the ABS blends was characterized with scanning electron microscopy. The compatibilities of PMMD and ABS were characterized by the calculation of the thermodynamic work of adhesion via the measurement of the contact angle. The results show that the antistatic and flame-retardant performance of ABS were greatly improved by the PMMD copolymer and the mechanical properties of ABS showed little reduction. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 44126.

INTRODUCTION

A random copolymer, poly(ethylene glycol) methacrylate/methyl methacrylate/diethyl allylphosphonate (PMMD), was synthesized from poly(ethylene glycol) methacrylate (MPEGMA), methyl methacrylate (MMA), and diethyl allylphosphonate (DEAP) by free-radical precipitation polymerization in the aqueous phase. The random PMMD copolymer was used to improve the antistatic and flame-retardant performance of a poly(acrylonitrile-*co*-butadiene-*co*-styrene) (ABS) resin. ABS resin is an important engineering plastic; it has been widely used in various fields, including industrial electronics, mechanics, the automobile industry, and instruments, for its superperformance in chemical resistance, mechanical properties, and processability.[1–4] However, ABS has its own defects in its inflammability [its limited oxygen index (LOI) is only 17–18%[5] and high resistivity (its surface resistance is 10^{16} Ω, and its volume resistance is 10^{16} Ω cm). These defects of ABS, with its inflammability and high resistivity, have seriously hindered its applications. Through the addition of fire-retardant or antistatic agents to the ABS resin, its flame-retardant or antistatic capabilities can be improved.

There are two ways to overcome the problem of the high resistance of plastic. One method is coating with antistatic agents[6–9]; the second is its mixture with antistatic agents, such as carbon black,[10] metal and metal oxide powder,[11,12] or other hydrophilic substances that contain hydrophilic functional groups such as long-chain ether bonds.[13,14] Compared with carbon black, hydrophilic substances have advantages, including light color, good compatibility with the matrix, and little effect on the mechanical of polymer materials.

Traditionally, halogenated compounds have been widely used in ABS, polypropylene, and other plastics to improve their flame-retardant performances. However, with the daily increasing environmental consciousness, a lot of halogenated compounds are forbidden by Euro-American countries because poisonous and corrosive smoke can be produced when halogenated compounds are burned. So, it is necessary to develop new flame-

Additional Supporting Information may be found in the online version of this article.

retardant methods and agents. New methods, such as photo-crosslinking,[15,16] and new flame-retardant agents, such as nano-clay flame retardants,[17,18] red phosphorus,[5] and phosphorus- or nitrogen-based flame retardants,[19–21] have been discovered. these Phosphorus-based flame retardants have a good retardant effect; further burning of the matrix can be restrained by the formation of a protective layer when it is burning. Therefore, phosphorus-containing flame retardants are widely used for plastics flame resistance.

In the past decade, various investigations have been carried out to improve the antistatic and flame-retardant properties of ABS and other plastics. However, few researchers have reported the synthesis of polymers with both antistatic and flame-retardant properties in ABS resin. In this study, a novel ABS antistatic and flame-retardant agent PMMD was synthesized, characterized, and blended with ABS resin. The antistatic and flame-retardant performance of ABS was improved at the same time through the addition of only PMMD. The performance of the PMMD and ABS/PMMD composites are reported in this article.

EXPERIMENTAL

Materials and Measurement Methods

MMA, toluene–p-sulfonic acid, hydroquinone, and $K_2S_2O_8$ were obtained from Ling Feng Chemical Reagent Co., Ltd. (Shanghai, China). Poly(ethylene glycol) monomethyl ether, triethyl phosphite, and allyl bromide were obtained from Shanghai Titan Technology Co., Ltd. ABS resin was offered by Taiwan's Chi Mei Industrial Co., Ltd. Antioxygen (168 and 1010) were purchased from BASF China Co., Ltd.

Instruments and Methods

Fourier transform infrared (FTIR) spectra of the samples were recorded between 4000 and 500 cm^{-1} on a Nicolet 6700 FTIR spectrometer (Thermo Nicolet Corp.). All of the FTIR specimens were dispersed in KBr and analyzed in the form of pressed discs. Thermogravimetric analysis (TG) was performed on a differential scanning calorimetry (DSC)–SP thermal analyzer (Netzsch STA 409, Germany). All of the samples were heated from 25 to 600 °C at a heating rate of 10 °C/min under a steady flow of an N_2 atmosphere.

The volume resistivity (ρ_v) and surface resistivity (ρ_s) of the ABS resin and its composites were detected with a ZC90 megohmmeter (Shanghai Precision Co., Ltd., China) according to GB/T 1410-2006. Each sample was kept at 23 °C and 50% relative humidity for 24 h before analysis. LOI were measured according to GB/T 2406.2-2009 with an HC-2 oxygen index meter (Jiangning Nanjing Analytical Instrument Co., Ltd.). The sample size in the experiment was $100 \times 10 \times 4$ mm^3. The UL-94 analyses were measured according to GB/T 2408-1996 with a CZF-3 vertical burning tester (Jiangning Nanjing Analytical Instrument Co., Ltd.). The sample size was $125 \times 6.5 \times 3$ mm^3. The contact angles were measured in a sessile drop mold with a JC2000A contact angle measuring instrument (Shang Zhong-chen Digital Apparatus Co., Ltd.). The notched impact strength was measured according to GB/T 13525-1992 with a JJ-20 memory impact testing machine (Changchun Intelligent

Equipment Co., China). The sample size was $100 \times 10 \times 4$ mm^3. The tensile strength was detected according to GB/T1040-1992 with a tensile testing machine (Changchun Intelligent Equipment Co., China). All of the mechanical properties were measured at a temperature of 23 °C and a relative humidity of 50%.

The morphologies of the ABS resin and its composites were observed with an S-4800 scanning electron microscope set with an accelerated voltage of 15 kV. The scanning electron microscopy (SEM) samples were etched with toluene for a given time and gold-sputtered before observation.

Synthesis of the Antistatic MPEGMA Monomer

An antistatic monomer of MPEGMA was synthesized with poly(ethylene glycol) monomethyl ether and MMA by an ester-exchange reaction. Poly(ethylene glycol) monomethyl ether (40 g, 0.1 mol), toluene–p-sulfonic acid (0.75 g), and hydroquinone (0.045 g) were added to a three-necked flask equipped with a stirring bar, a fractionating tower, and a thermometer. The reactant, polymerization initiator, and polymerization inhibitor were heated to 120 °C under a nitrogen atmosphere. Then, MMA (11 g, 0.11 mol) was added to the mixture and reacted for 3 h. Then, the fractionating tower was replaced with a distillation plant, and excess MMA was distilled out; this left behind the desired product as a viscous liquid. The synthesis route of the MPEGMA is shown in Scheme 1.

^{1}H-NMR (CDCl$_3$, ppm, δ): 1.8–2.0 (—CH$_3$, 3H), 3.3–3.4 (—O—CH$_3$, 3H), 3.5–3.8 (—O—CH$_2$—CH$_2$—O—, 36H), 4.2–4.4 (—COO—CH$_2$, 2H), 6.3–6.5 (=CH$_2$, 2H), 7.3 (CHCl$_3$).

Synthesis of the Flame-Retardant DEAP Monomer

The flame-retardant monomer DEAP was synthesized with triethyl phosphite and allyl bromide by a substitution reaction. Triethyl phosphite (20 g, 0.12 mol) and hydroquinone (0.11 g) were added to a three-necked flask equipped with a stirrer, a condensation reflux device, and a thermometer. The mixture were heated to 80 °C under a nitrogen atmosphere and stirred at 250 rpm. Then, allyl bromide (13.2 g, 0.11 mol) was added to the mixture and reacted at 80 °C for 4 h and then at 120 °C for 1 h. Finally, the excess triethyl phosphite was distilled out *in vacuo* to yield a colorless liquid. The synthesis route of the DEAP is shown in Scheme 1.

^{1}H-NMR (CDCl$_3$, ppm, δ): 1.1–1.4 (—CH$_3$, 6H), 2.6–2.7 (—CH$_2$—P, 2H), 3.9–4.3 (P—O—CH$_2$—, 4H), 5.1–5.3 (=CH$_2$—), 5.7–5.9 (=CH—, 1H), 7.3 (CHCl$_3$).

Synthesis of the PMMD Copolymers

PMMD copolymers were synthesized with MPEGMA, MMA, and DEAP by free-radical precipitation polymerization in the aqueous phase. In the nitrogen atmosphere, deionized water and various monomers (MPEGMA, MMA, and DEAP) were added to a three-necked flask equipped with a stirrer, a condensation reflux device, and a thermometer and stirred at 250 rpm for 30 min. The molar ratio of the various monomers (MPEGMA, MMA, and DEAP) were 1:6:2, 1:6:3, 1:10:3, and 1:10:5, respectively. After stirring, a certain amount of initiator ($K_2S_2O_8$) was added to the mixture, which was heated to 70 °C and reacted for 12 h. Finally, the excess water was extracted by vacuum filtration and dried for 12 h *in vacuo* at 60 °C to yield a
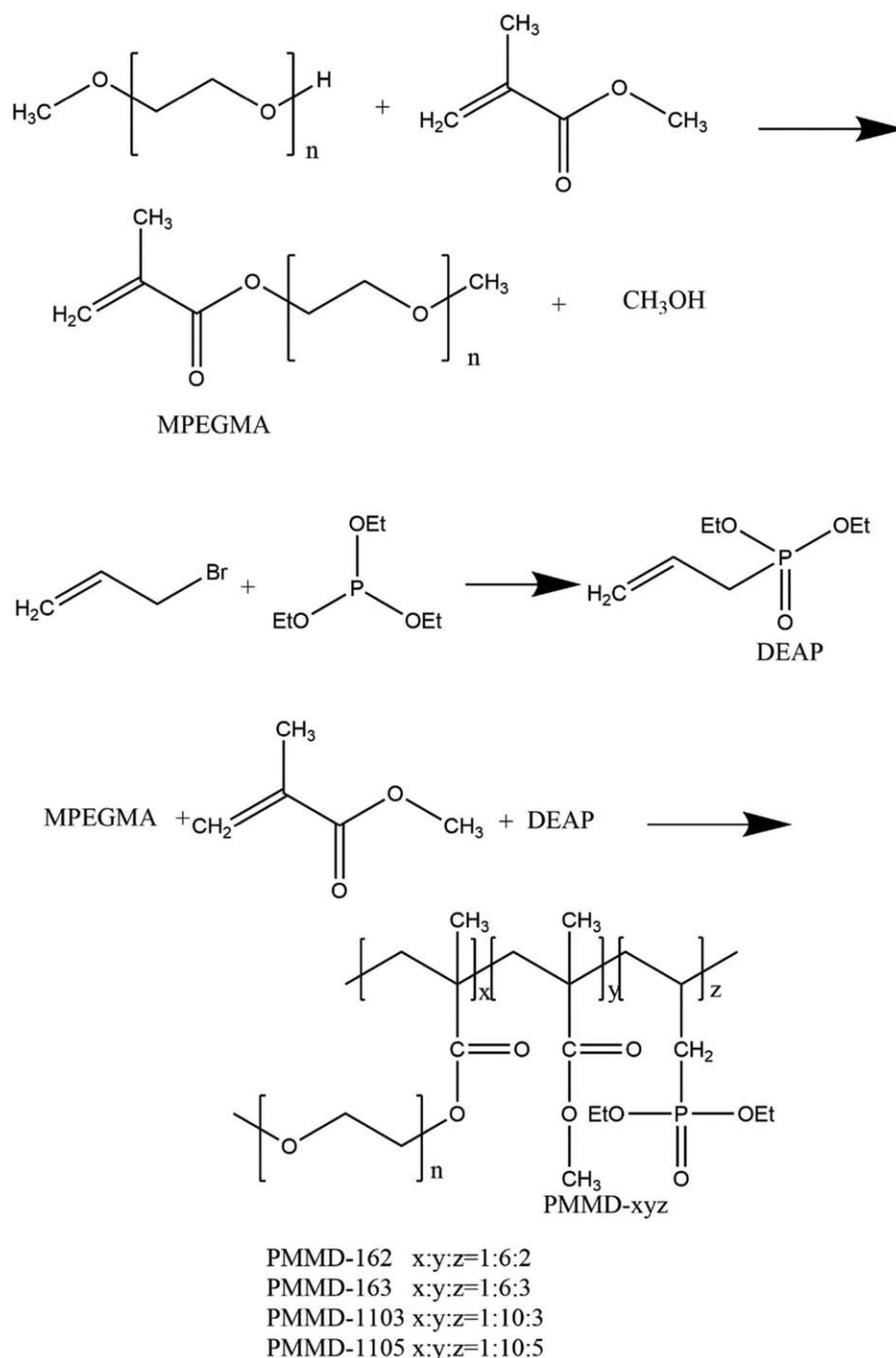
Scheme 1. Synthesis of the monomers and PMMD copolymer.

light yellow solid. The synthesis route of PMMD is shown in Scheme 1.

Preparation of the ABS/PMMD Composites

The ABS resin, PMMD, and antioxygen were dried *in vacuo* at 60 °C for 12 h before use. Certain amounts of the ABS resin, PMMD, and antioxygen were added to the torque rheometer and melted at 180 °C under a rotor speed of 60 rpm. Then, the ABS/PMMD blends were put into a press vulcanizer and molded at 180 °C to yield various kinds of samples. The torque values curves of the ABS and ABS/PMMD-163 (15%) are shown

in Figure S2 (see the Supporting Information). We observed that the maximum torque and final torque of the blends decreased with the addition of the PMMD-163 copolymer.

RESULTS AND DISCUSSION

Structural Characterization

The FTIR spectra of MPEGMA, DEAP, and PMMD-163 (MPEGMA/MMA/DEAP = 1:6:3) are shown in Figure 1. The FTIR spectra of PMMD-162, PMMD-1103, and PMMD-1105 were similar to that of PMMD-163. For the FTIR spectra of MPEGMA [Figure 1(a)], the absorption peak at $1635\,\mathrm{cm}^{-1}$
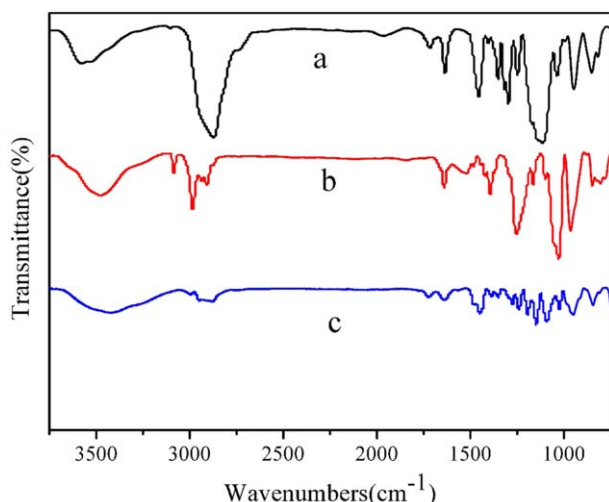
Figure 1. FTIR spectra of (a) MPEGMA, (b) DEAP, and (c) PMMD-163. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

corresponded to C=C stretching vibrations, and that at $1112\,cm^{-1}$ corresponded to antisymmetric stretching vibrations of the group of C—O—C; this indicated that the antistatic monomer was synthesized successfully and was composed of branched polyether and C=C groups. In the FTIR spectrum of DEAP [Figure 1(b)], the absorption band at $1639\,cm^{-1}$ corresponded to C=C stretching vibrations. The absorption peaks at 1253 and $1028\,cm^{-1}$ corresponded to P=O stretching vibrations and P—O—C stretching vibrations, which are characteristic of phosphate groups. The results indicate that the flame-retardant monomer was synthesized successfully and that it was composed of phosphate and C=C groups. In the FTIR spectrum of PMMD-163, the absorption at $1091\,cm^{-1}$ corresponded to the C—O—C stretching vibration absorption band in branched polyether. The peaks at 1241 and $1024\,cm^{-1}$ corresponded to P=O and P—O—C stretching vibrations in the phosphate group. The

Table I. TG Results for the PMMD-163, ABS/PMMD-163 Blends, and Pure ABS Resin

Material	Onset temperature (°C)	Weight loss (%)	Residual
PMMD-163	216	15.3	7.9
	287	13.2	5.4
	335	63.5	2.9
ABS/PMMD-163	396	94.6	
ABS	405	97.1	

PMMD-163 copolymer contained a branched polyether with antistatic properties and a phosphate group with a flame-retardant function.

Thermal Properties

The thermal stabilities of the PMMD-163 copolymer, ABS/PMMD-163 blends, and pure ABS resin were studied by TG and DSC. The TG curves of the PMMD-163 copolymer, ABS/PMMD-163 blends, and pure ABS resin are shown in Figure 2, and their onset temperature, weight loss, and residual are shown in Table I. We observed that the weight of PMMD-163 decreased gradually at the start because PMMD-163 was a hydrophilic polymer because it had a polyether branched chain, which absorbed water from the air easily. In addition, the PMMD-163 copolymer had three decompositions. The first onset temperature was 216 °C and was ascribed to the rupture of the P—O—C bond, the second onset temperature was 287 °C and involved the fracture of the C—O—C bond, and the last onset temperature was 335 °C and was attributed to the decomposition of the copolymer main chain. The weight of residual of the copolymer was 7.9%. The TG curve of the ABS/PMMD-163 blends was similar to that of the pure ABS resin. The onset decomposition temperature of the ABS/PMMD-163 blends was 396 °C, which was a little lower than the onset decomposition temperature of the pure ABS resin (405 °C). The weight of residual of the ABS/PMMD-163 blends was 5.4%, which was a little higher than the weight of the residual of the pure ABS
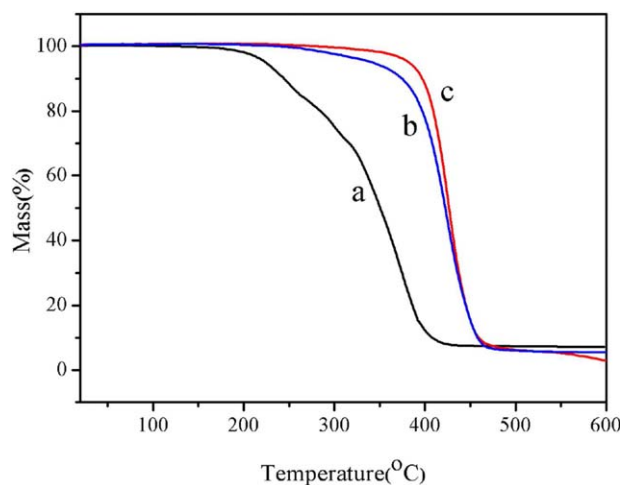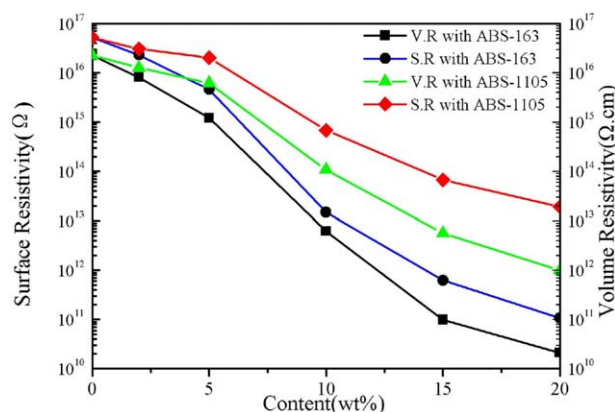

Figure 2. TG curves of the (a) PMMD-163, (b) ABS/PMMD blends, and (c) ABS. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 3. Influence of the copolymer content on the surface resistivity (S.R.) and volume resistivity (V.R.) of the PMMD/ABS blends. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table II. LOI and UL-94 Properties of the ABS/PMMD-163 and ABS/PMMD-162 Blends

		Additive (wt %)					
		0	5	10	15	20	100
LOI	ABS-163	17.8	18.9	19.5	20.4	20.8	23.2
	ABS-162	17.8	18.4	19.0	19.7	20.3	22.5
UL-94	ABS-163	Fail	Fail	Fail	HB	HB	V-2
	ABS-162	Fail	Fail	Fail	Fail	HB	V-2

resin. The results of TG indicate that the PMMD-163 copolymer was suitable for addition to the ABS resin because its onset decomposition temperature was higher than the processing temperature of the ABS resin (180 °C), and the ABS/PMMD-163 blends had moderate thermal stabilities.

The DSC curves of the PMMD-163 copolymer are shown in the Supporting Information (Figure S1). The DSC curves of other copolymers were similar to that of PMMD-163. From the DSC curve, only one glass-transition temperature near −28.9 °C was observed. So, we could roughly determine that the product we obtained was a copolymer instead of a mixture. There was no obvious melting peak until 200 °C; this indicated that the PMMD-163 copolymer was an amorphous copolymer.

Antistatic Properties of the ABS/PMMD Blends
The effects of the copolymer (PMMD-163 and PMMD-1105) content and the proportion of MPEGMA monomer in the copolymer on the ρ_s and ρ_v values of the PMMD/ABS blends are shown in Figure 3. The ρ_s and ρ_v values of the pure ABS resin were 5.2×10^{16} Ω and 2.3×10^{16} Ω cm, respectively. On the one hand, the ρ_s and ρ_v values of the PMMD/ABS blends decreased gradually with increasing content of PMMD. On the other hand, the ρ_s and ρ_v values of the PMMD/ABS blends decreased with increasing proportion of the MPEGMA monomer in the copolymer. The ρ_s of ABS/PMMD-163 decreased to 1.2×10^{11} Ω when the content of PMMD-163 was 20 wt %; this was five orders of magnitude less than that of the pure ABS resin. Compared with PMMD-163, the ρ_s of ABS/PMMD-1105 decreased to 1.2×10^{13} Ω when the content of PMMD-1105 was 20 wt %; this was a little higher than that of PMMD-163. Similarly, when the copolymers content increased to 20 wt %, the ρ_v values of ABS/PMMD-163 and ABS/PMMD-1105 decreased to 2.1×10^{10} and 1.0×10^{12} Ω cm, respectively. In addition, as shown in Figure 3, the content of the copolymer was less than 5 wt %, with increasing content of the copolymer,

the ρ_s and ρ_v values of the blends increased slowly compared with those of the blends with copolymer contents high than 5 wt %. The variety tendency of the ρ_s and ρ_v values of the ABS/PMMD blends implied that there was a percolation threshold in the composites, and the percolation threshold was near 5 wt %. In addition, according to the military book DOD-HDBK-263,[22] the antistatic material had a ρ_s between 10^9 and 10^{14} Ω cm. Therefore, when the content of the copolymer was added to a certain amount, the blends reached the antistatic material requirement.

Flame-Retardant Properties of the ABS/PMMD Blends
The effects of the copolymer (PMMD-163 and PMMD-162) content and the proportion of DEAP monomer in the copolymer on the LOI and UL-94 of PMMD/ABS blends are shown in Table II. As the PMMD copolymer was added, the flame retardant of the ABS/PMMD blends was improved. When the contents of PMMD-163 and PMMD-162 were 20 wt %, the LOIs of composites improved to 20.8 and 20.3, respectively. The UL-94 of blends arrived to HB when the content of PMMD-163 was 15 wt % and that of PMMD-162 was 20 wt %. The flame-retardant properties of the ABS/PMMD-163 blends were a little better than those of ABS/PMMD-162 because the molar ratio of the flame retardant of PMMD in PMMD-163 was higher than that of PMMD-162. The LOI and UL-94 of composites improved with increasing PMMD. However, it had a limit because the LOI and UL-94 of PMMD were about 23 and V-2, respectively.

Thermodynamic Work of Adhesion of the ABS/PMMD Blends
The contact angles of the pure ABS, PMMD-163, and PMMD-1103 were measured in a sessile drop mold with a JC2000A contact angle measuring instrument and are listed in Table III. Then, the surface energies of ABS and PMMD were estimated from the contact angle data with the following two equations [eq. (1) for water and eq. (2) for glycol] according to Wu[23]:

$$(1+\cos\theta_{H_2O})=4\left(\frac{\gamma_{H_2O}^d\gamma^d}{\gamma_{H_2O}^d+\gamma^d}+\frac{\gamma_{H_2O}^p\gamma^p}{\gamma_{H_2O}^p+\gamma^p}\right) \quad (1)$$

$$(1+\cos\theta_{EG})=4\left(\frac{\gamma_{EG}^d\gamma^d}{\gamma_{EG}^d+\gamma^d}+\frac{\gamma_{EG}^p\gamma^p}{\gamma_{EG}^p+\gamma^p}\right) \quad (2)$$

where γ^d is the dispersion component of the surface energy, γ^p is the polar component of the surface energy, and θ is the contact angle of water (H_2O subscript) and glycol (EG subscript) with the materials. The numerical values used were as follows: $\gamma_{H_2O}^d=21\times(8 \text{ mJ/m}^2)$, $\gamma_{H_2O}^p=51\times(0 \text{ mJ/m}^2)$, $\gamma_{EG}^d=29\times(3 \text{ mJ/m}^2)$, and $\gamma_{EG}^d=19\times(0 \text{ mJ/m}^2)$. Then, the thermodynamic work of

Table III. Contact Angles and Surface Energies of the ABS, PMMD-163, and PMMD-1103 and Thermodynamic Work of Adhesion of the ABS/PMMD Blends

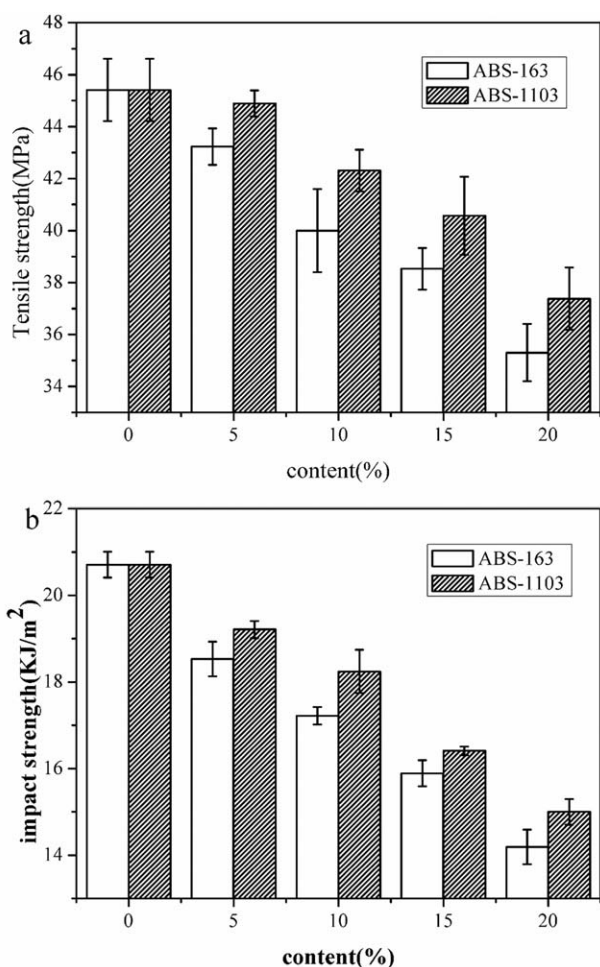|	Contact angle (°)		Surface energy (mJ/m²)			Work of adhesion (mJ/m²)	
Sample	Water	Glycol	Total	γ^d	γ^p	PMMD-163	PMMD-1103
ABS	94.0	53.4	41.1	37.9	3.2		
PMMD-163	44.1	62.4	68.6	3.2	65.4	51	61.6
PMMD-1103	54.6	47.5	46.2	10.7	35.5		

Figure 4. Influence of the copolymer content on the mechanical properties of the ABS/PMMD blends.

the ABS/PMMD-163 and ABS/PMMD-1103 blends were calculated with eq. (3):

$$\text{Work of adhesion} = 2(\gamma_A^d \gamma_B^d)^{1/2} + 2(\gamma_A^p \gamma_B^p)^{1/2} \quad (3)$$

where γ_A and γ_B are the surface energies of the two materials in contact. The thermodynamic work of adhesion values of ABS/PMMD-163 and ABS/PMMD-1103 were 51 and 61.6 mJ/m^2, respectively. The results of the thermodynamic work of adhesion of ABS/PMMD-163 and ABS/PMMD-1103 blends show that the compatibility of PMMD with the ABS resin copolymer improved with increasing molar ratio of MMA in the PMMD copolymer.

Mechanical Properties of the ABS/PMMD Blends

The effect of the copolymer (PMMD-163 and PMMD-1103) content and the proportion of MMA monomer in the copolymer on the tensile strength and notched impact strength of the PMMD/ABS blends are shown in Figure 4(a,b). The tensile strength and notched impact strength values of the pure ABS resin were 45.4 MPa and 20.7 KJ/m^2, respectively. When the content of PMMD increased to 20 wt %, the tensile strength and notched impact strength decreased to 35.3 MPa and 14.2 kJ/m^2 for the ABS/PMMD-163 blends and 37.4 MPa and 15 KJ/m^2 for the ABS/PMMD-1103 blends. We observed that the tensile strength and notched impact strength of the blends decreased with increasing PMMD content and decreasing MMA monomer in the PMMD copolymer. However, the change with the addition of PMMD to the ABS resin was small and would not impact its use.

Morphology

The SEM micrographs of the pure ABS and ABS/PMMD-163 blends are shown in Figure 4(a–f), respectively. All of the SEM samples were etched by toluene for a given time and gold-
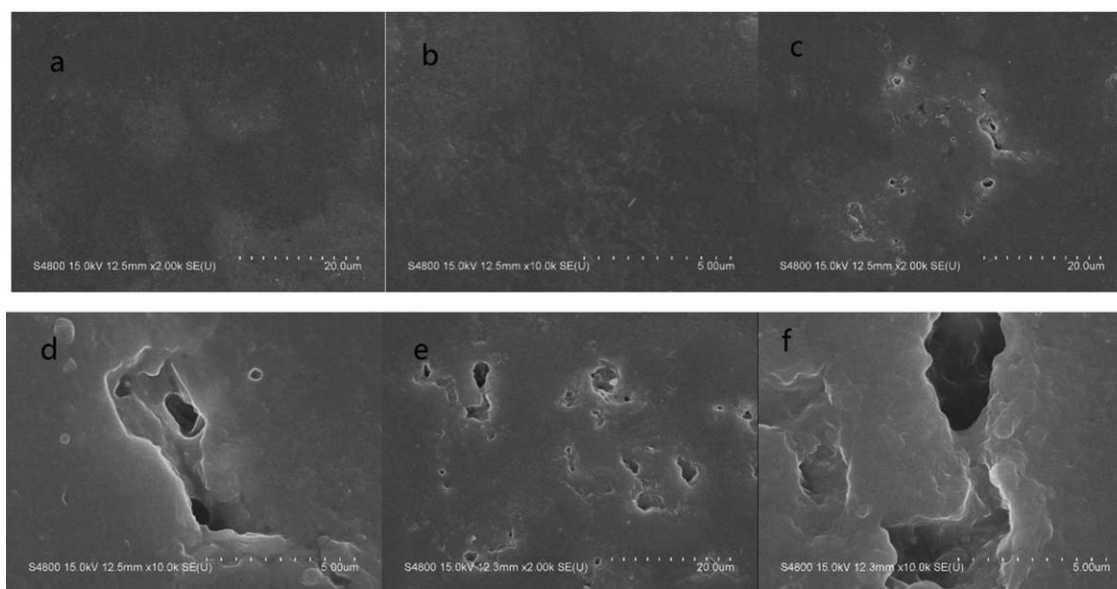

Figure 5. SEM micrographs at different magnifications of the pure ABS and ABS/PMMD-163 blends with different PMMD-163 contents: (a) 0 wt % and 2000×, (b) 0 wt % and 10,000×, (c) 5 wt % and 2000×, (d) 5 wt % and 10,000×, (e) 15 wt % and 2000×, and (f) 15 wt % and 10,000×.

sputtered before observation. We observed that there were seldom black microspheres, as shown in Figure 5(a,b), for the ABS resin insoluble in toluene. Some black microspheres randomly dispersed in etched ABS/PMMD-163 composites were detected when the PMMD-163 content reached 5 wt % in Figure 5(c,d). However, the number of those microspheres was restricted in a definite range, and the size of those microspheres was relatively small (ca. 2 μm). This means that when the content of antistatic agent was small, it was not enough to form continuous conducting tunnels or networks in ABS blends; this directly resulted in the relatively lower conductivity of the composites. When the content of PMMD-163 widened to 15 wt %, more and bigger black microspheres were observed, as shown in Figure 5(e,f). The size of those microspheres was increased to about 5 μm. This means that with increasing content of PMMD-163, agglomeration occurred; this could explain the phenomenon in which the mechanical properties decreased and the resistivity decreased with increasing content of the PMMD copolymer.

CONCLUSIONS

The antistatic flame-retardant PMMD copolymer was successfully synthesized with MPEGMA, MMA, and DEAP by free-radical precipitation polymerization in the aqueous phase. The structure of the PMMD copolymer was studied. TG reveals that PMMD had sufficient thermal stability and little effect on the stability of the ABS resin. The ρ_s and ρ_v values of the PMMD-163/ABS composites drastically decreased with increasing PMMD content from 10^{16} to 10^{11} Ω and from 10^{16} to 10^{10} Ω cm, respectively. PMMD had a relative good flame-retardant performance for ABS resin; this was verified by LOI and UL-94 testing. The results of the tensile strength and notched impact strength show that the PMMD copolymer had little effect on the mechanical properties of ABS. The calculation of the thermodynamic work of adhesion revealed that the compatibility of ABS with PMMD increased with increasing molar ratio of MMA in the PMMD copolymer. All of the tests showed that the PMMD copolymer was suitable for ABS to improve its antistatic and flame-retardant performance.

ACKNOWLEDGMENTS

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