

Structure and Properties of High Melt Strength Polypropylene Prepared by Combined Method of Blending and Crosslinking

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ABSTRACT: High melt strength polypropylene (HMSPP) was prepared by *in situ* heat induction reaction, in which pure polypropylene (PP) powders without any additives was used as basic resin, and low density polyethylene (LDPE) and trimethylolpropane triacrylate (TMPTA) were added as blending resin and as crosslinking agent, respectively. Microstructure of the obtained HMSPP (PP/LDPE/TMPTA blends) was characterized by FTIR, Wide-angle X-ray diffraction (WAXD), and testing of gel content. The effect of LDPE content on melt strength and melt flow rate of HMSPP were investigated. When the content of LDPE was 40 wt %, the melt strength of the HMSPP was above 16 CN, which was

much higher than those of pure PP powder (2.6 CN) and PP/LDPE blends without TMPTA (6.1 CN). Moreover, thermal behavior and mechanical properties of the HMSPP were also investigated. The results showed that the thermal stability and impact strength of HMSPP were greatly improved. In addition, HMSPP possessed good processing performance and good foaming properties. The foams produced by HMSPP showed uniform, closed, and independent cells. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 1739–1746, 2010

Key words: blending; crosslinking; melt; polypropylene (PP); strength

INTRODUCTION

Polypropylene (PP) is one of the most important synthetic polymeric materials. PP exhibits many good properties, such as, high mechanical strength, inertness for various chemical reagents, nontoxicity, high transparency, high glossiness, etc. However, there are still some weaknesses in the performance of PP. An example is low melt strength, which is ascribed to the linear structure of PP chain. The melt strength of PP is a measure of the melt resistance to extensional deformation. Low melt strength of PP influences the thermoforming behavior of this polymer, especially the tendency of the PP sheet to sagging. Recently, high melt strength grades of PP (HMSPP) have been developed to improve the processing performance in thermoforming.^{1–3}

HMSPP can be used in the place of polystyrene for foam products and poly(ethylene terephthalate)

for high-pressure bottle, which widen the application of PP. Especially, HMSPP is environmentally intimate material with a prohibition of any toxic gas discharge during incineration. Moreover, HMSPP can be recycled due to its thermoplastic behavior.

In general, there are long branching chains on the backbones or chain entanglement in the structure of HMSPP.^{4–7} It was found that HMSPP could be prepared through several routes, for examples, propylene polymerization catalyzed by metallocene catalyst or Ziegler-Natta catalyst,⁸ crosslinking via adding organic peroxide or using electron beam irradiation,⁹ or blending modification.^{10–12} Blending of PP with polyethylene (PE) has been extensively used to improve melt strength. There were some reports about enhancing melt strength of polymer by blending of high density polyethylene (HDPE), low density polyethylene (LDPE), and linear PP.^{10–12} Borsig et al. envisaged the crosslinking of PP/PE blend in all concentration ranges of polymer, with a peroxide and pentaerythritol tetra allyl ether as polyfunctional monomer.¹³ Chodak et al. used, in the case of PP/LDPE blends, a peroxide with a quinone that slightly delayed the crosslinking of the PE and activated the crosslinking of PP.¹⁴ According to Borsig et al. and Chodak et al.,^{13,14} under normal circumstances, in order to induce radical reaction, a certain

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amount of initiator, such as, peroxide compounds, should be added in the system. Meanwhile, Kim and Kim not only studied the crosslinking of HDPE during reactive extrusion but also researched the crosslinking of PP by peroxide and multifunctional monomer.^{15,16} As we know, the radicals from the initiator attack easily tertiary carbon atoms of PP, which accelerate degradation reaction of PP. If the initiators were not dispersed uniformly in the PP matrix, this will certainly lead to excessive degradation in some domains of the matrix, or even excessive crosslinking of PP chain in the presence of crosslinking coagents, which will damage performance of the resultant HMSPP.

In this work, we reported a novel method to prepare HMSPP via combining blending of PP and LDPE and then further crosslinking. Here, *in situ* heat induction reaction was used to initiate crosslinking reaction. In this method, the radicals were induced only by heat during blending. The benefit of this method lies in uniform dispersion of the resulting radicals and effectively avoiding excessive degradation of PP. At the same time, trimethylolpropane triacrylate (TMPTA) was added to act as crosslinking coagent that can effectively capture free radicals, which lead to the formation of microcrosslinked structure and improve melt strength of PP. In addition, LDPE added as blending resin, which has long-chain structure and high melt strength, can further improve the melt strength of PP. More importantly, PE radicals could be also formed by the reaction of LDPE with other free radicals or even heat induction. In this case, branched structure and microcrosslinked structure of PP/LDPE blend were formed in the presence of crosslinking coagent, thus the melt strength was further improved.

EXPERIMENTAL

Materials

Isotactic PP powder with a melt flow rate (MFR) of 3.65 g/10 min was from Daqing Petrochemical Company. Low density polyethylene (LDPE) with a MFR of 1.5 g/10 min was also from Daqing Petrochemical Company. TMPTA was standard laboratory reagents used as received.

Preparation of HMSPP

The PP powder was tumble-premixed with LDPE and TMPTA. The blending and crosslinking processes were carried out in TSSL-25 corotating twin-screw extruder with a length/diameter ratio of 36/1 and a diameter of 25 mm. The screw configuration was designed to provide good dispersive and distributive mixing. The barrel temperatures from the

feed zone to the die zone were set as follows: 190, 210, 225, 230, 230, 230, 230, 215, and 195°C. The screw rate and feed rate were kept constant at 65 rpm and 21 rpm, respectively. The obtained ternary PP/LDPE/TMPTA blends are named as HMSPP. After the reaction, the resultant products were shaped into 1-mm thick compression-molded sheets as follows: the polymers were preheated at 210°C for 5 min and then pressed for 5 min under 15 MPa of pressure. After cooling, a sheet was cut into tensile bars. The specimens were stored under dried conditions before testing. As a control, binary PP/LDPE blends were also prepared under the same conditions.

Characterizations

FTIR spectra were recorded with a FTIR spectrometer (GX-2000) in the range of 600–4000 cm^{-1} with a resolution of 4 cm^{-1} . The products from the extruders were first purified using a dissolution-precipitation method in order to remove unreacted TMPTA. Samples for FTIR measurements were prepared by making films of the HMSPP in a hot press. Films (30 μm thick) were prepared by hot pressing at 210°C.

The impact strength was measured with a TY4020 Izod Impact Strength Testing. Wide-angle X-ray diffraction (WAXD) measurements were performed at room temperature by a D/max-2500/PC X-ray diffractometer (Rigaku, Japan) using $\text{CuK}\alpha$ radiation operated at 35 kV and 50 mA. The data were collected from 6° to 50° at a scanning rate of 3°/min. Before testing, all specimens were treated uniformly. Thermogravimetric analysis (TGA) was performed using PerkinElmer Pyris 1 under nitrogen atmosphere at a heating rate of 10°C/min.

The melting and crystallization behavior of the samples were investigated with a PerkinElmer DSC1 differential scanning calorimeter. The samples were first heated from 50°C to 200°C at a scanning rate of 10°C/min and then maintained at 200°C for 5 min before cooling to 50°C at the same rate. After that, the samples were reheated from 50°C to 200°C at a scanning rate of 10°C/min. The heats of fusion for LDPE (ΔH_m^0 , LDPE) and PP (ΔH_m^0 , PP) with 100% crystallinity were 287.3 J/g and 207 J/g, respectively.¹⁷

The MFR of the samples was determined using CEAST 6542 Modular Flow Index. The measurement condition was at 230°C under a load of 2.16 kg. MFR is generally expressed in terms of the weight of extrudate in grams per 10 min interval.

The sample (thickness is 0.5 mm) for testing the content of gel was made according to the GB/T 9352–88. 0.2–0.3 g pellets weighed by analytical balance (± 0.0001 g) were wrapped with the quantitative filter papers. The sample was packaged in a

copper net of 120 mesh and weighed by analytical balance, and then was put into boiling xylene for 16 h. After that, the sample was put in ventilates until tasteless. The sample was dried for 2 h in the drying oven at 105–110°C, and then was took out and put into the water extractor to cool 30 min and weighed by analytical balance. The gel percentage of the products was calculated as the percentage of the weight remaining with respect to the initial weight of sample.

Melt strength was measured by using Rheotens method with a capillary of die diameter of $D = 2$ mm and length/diameter (L/D) = 20/2 at 230°C (Fig. 1). Here, a melt strand was extruded through a capillary die and pulled down with increasing velocity (at constant acceleration 27 mm/s^2) by using a pair of wheels, and the force was measured till the rupture of the melt strand occurred. The average force is called as melt strength, and the drawdown velocity at break is a measure of melt extensibility.

Scanning electron microscopy (SEM) was used to examine the foaming morphology of samples by using a Japan JSM6360LA SEM.

RESULTS AND DISCUSSION

Structural characterization of the HMSPP

In twin-screw extruder, a mixture of PP/LDPE/TMPTA was used to prepare HMSPP. To clear whether macromolecular free radicals of PP and LDPE could be formed under the process conditions, effects of process conditions on MFRs of PP and LDPE were investigated. When comparing with the original resins, after extrusion, the MFR of PP increased and that of LDPE decreased. This implies that macromolecular free radicals of PP and LDPE could be formed. Thus, the preparation process of PP/LDPE/TMPTA blends (HMSPP) involves three kinds of chemical reaction at least. Firstly, PP chains

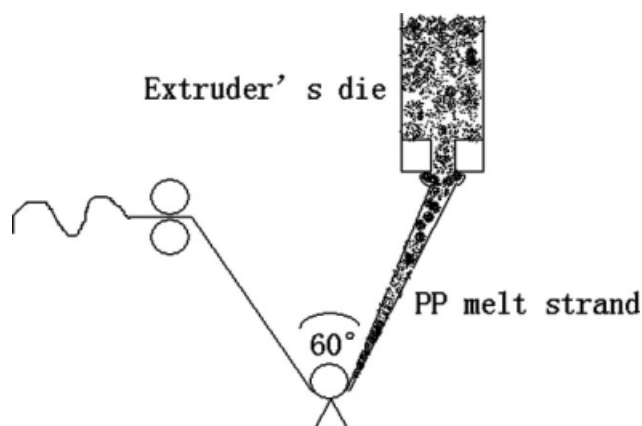


Figure 1 Schematic diagram of melt strength tester.

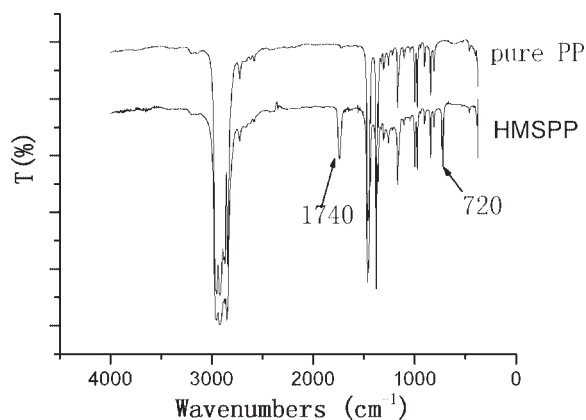


Figure 2 FTIR spectra of pure PP and HMSPP.

break to form free radicals under shear field at high temperature; meanwhile, a part of LDPE chains also break to form free radicals. Then the macromolecular free radicals react with TMPTA and molecular chains of PP and LDPE, the latter generates new macromolecular free radicals. At last, HMSPP is produced due to the formation of long branching chains or microcrosslinked structure through free-radical reaction among three components. Figure 2 shows FTIR spectra of the pure PP and purified HMSPP from the feed of PP/LDPE/TMPTA = 70/30/2 by weight. In the case of the HMSPP, two new absorption bands at 1740 cm^{-1} and 720 cm^{-1} are observed, which can be assigned to the absorption of C=O and LDPE, respectively. Meanwhile, there is no absorption peak of carbon-carbon double bond in the FTIR spectra of HMSPP ($1500\text{--}1700 \text{ cm}^{-1}$), confirming that the absorption band at 1740 cm^{-1} resulted from the crosslinking coagent that fully reacted with PP and LDPE and no residue of free TMPTA existed.

The content of gel is generally used to evaluate the crosslinking degree of polymers. Figure 3 shows

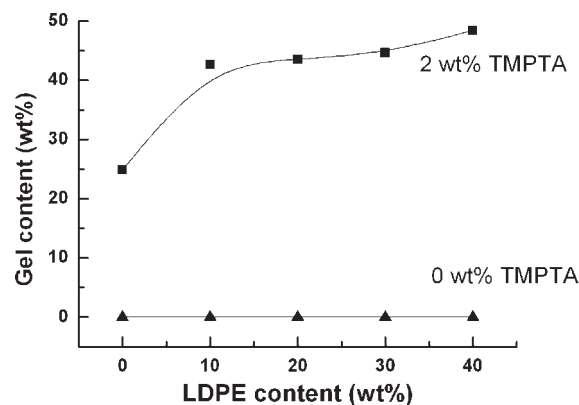


Figure 3 Effect of LDPE content on gel content of HMSPP.

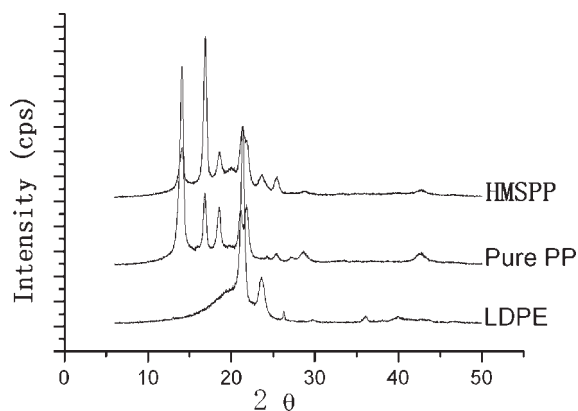


Figure 4 WAXD profiles of pure PP, LDPE, and HMSPP.

the effect of LDPE on the gel content of the HMSPP. It is noted that the gel content of the HMSPP increases gradually with increasing the content of LDPE in the presence of TMPTA. After adding 2 wt % TMPTA, the gel content of the sample is 24.9 wt % in the absence of LDPE. However, when the content of LDPE is 40 wt %, the gel content of the HMSPP is 48 wt %. As C—C double bond of TMPTA can be attacked by the free radicals originated from *in situ* heat induction reaction, the molecular chains of PP can form microcrosslinked structure besides long branching chains in PP/TMPTA mixture, which increases the gel content of HMSPP. Furthermore, in the presence of LDPE, TMPTA can effectively promote LDPE to combine with PP and form more microcrosslinked structure, thus the gel content of HMSPP increased further. In contrast, in the absence of TMPTA, the gel content of PP/LDPE blend is zero in spite of different content of LDPE. This implies that, in the absence of crosslinking coagent (such as TMPTA), the blend of PP and LDPE is just simple physical mixture, in which there is no crosslinking reaction under the same preparation conditions.

PP could crystallize in three polymorphic forms, α (monoclinic), β (pseudohexagonal), and γ (triclinic), depending on the composition of PP and crystallization conditions. Figure 4 shows the WAXD profiles of PP, LDPE, and HMSPP. The diffraction peaks of pure PP are located at 14.0° , 16.8° , 18.5° , 21.0° , and 21.8° in the range of 10 – 25° . The peaks at 14.0° , 16.8° , 18.5° , 21.0° , and 21.8° correspond to the α (110), α (040), α (130), α (111), and α (131) planes, respectively.^{18,19} When compared with pure PP, the intensities of diffraction peaks in some directions are changed in the HMSPP. There is a strong diffraction peak at $2\theta = 16.8^\circ$ for the HMSPP, which is much higher than that of pure PP. Furthermore, the intensity of diffraction peak at $2\theta = 14.0^\circ$ for the HMSPP is smaller than that of pure PP. These results indicate that the crystalliza-

tion state of PP in the HMSPP has changed after the blending and crosslinking.

Melting and crystallization behavior of HMSPP

Figure 5 presents the melting and crystallization curves of PP, LDPE, PP/LDPE blend and PP/LDPE/TMPTA blend (HMSPP). Pure LDPE and pure PP show single melting peak and single crystallization peak. Two well-separated melting peaks are found in PP/LDPE blend and PP/LDPE/TMPTA blend (the first for LDPE and the second for PP), reflecting two crystalline phases in all these materials. Adding 2 wt % TMPTA strongly affects the melting and crystallization behavior of pure PP and PP/LDPE = 70/30 (by weight) blend, especially the crystallization behavior of the latter. Both the crystallization peaks and the melting peaks of LDPE and PP in the PP/LDPE/TMPTA blend become broad.

From Table I, we can see that, comparing with pure PP, both melting temperature and crystallization temperature of PP in the PP/LDPE blend (in

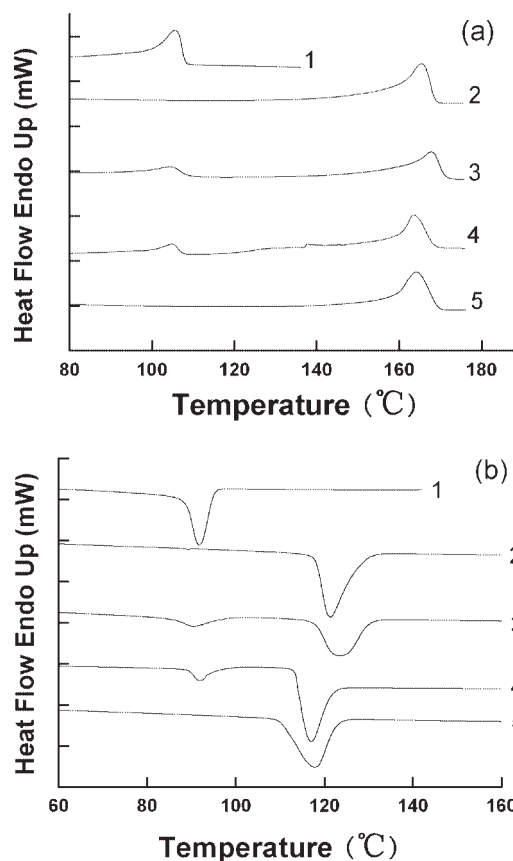


Figure 5 Comparison of melting curves (a) and crystallization curves (b) from DSC measurements. (1) Pure LDPE; (2) 2 wt % of TMPTA and 0 wt % of LDPE; (3) 2 wt % of TMPTA and 30 wt % of LDPE; (4) 0 wt % of TMPTA and 30 wt % of LDPE; (5) Pure PP.

TABLE I
Crystallization Properties of the Parental Resins and the Resultant HMSPP

Samples	T_m (°C)		T_c (°C)		Crystallinity (%)	
	LDPE	PP	LDPE	PP	LDPE	PP
PP	/	164.2	/	117.8	/	45.0
LDPE	105.7	/	91.7	/	20.7	/
c-PP ^a	/	165.4	/	121.3	/	46.2
PL-1 ^b	105.0	163.5	91.9	116.8	19.0	44.9
PL-2 ^c	101.3	166.6	90.1	127.7	16.9	45.8
PL-3 ^d	101.6	166.9	91.1	128.2	19.0	44.8
PL-4 ^e	104.8	167.9	90.4	124.1	17.6	44.2
PL-5 ^f	105.5	168.5	91.1	125.5	17.7	46.8

^a Containing 2 wt % TMPTA.

^b PP/LDPE = 70/30 by weight.

^c PP/LDPE/TMPTA = 90/10/2 by weight.

^d PP/LDPE/TMPTA = 80/20/2 by weight.

^e PP/LDPE/TMPTA = 70/30/2 by weight.

^f PP/LDPE/TMPTA = 60/40/2 by weight.

the absence of TMPTA) decrease slightly. However, owing to the addition of 2 wt % TMPTA which can react with LDPE and PP, both melting temperature and crystallization temperature of PP in PP/LDPE/TMPTA blends or PP/TMPTA blend (PL-2 to PL-5 or c-PP in Table I) increase. Moreover, when comparing with the PP/LDPE = 70/30 (by weight) blend (PL-1), the crystallization temperature and the melting temperature of PP in the PP/LDPE/TMPTA = 70/30/2 (by weight) blend (PL-4) are increased due to the crosslinking. Because the addition of TMPTA will lead to form some crosslinking sites in PP phase. The crosslinking sites, acting as nucleating agent, can promote the crystallization of PP. Therefore, in the case without LDPE, the crystallization temperature of PP in PP/TMPTA blends increases. At the same time, according to the results of Kim and Kim,¹⁵ crosslinking structures can restrict chain motion. So the melting temperature of PP in PP/TMPTA and PP/LDPE/TMPTA blends should also increase.

Melt properties of the HMSPP

Figure 6 shows the effect of LDPE content on the MFR of HMSPP. As a control, the MFR of PP/LDPE blends without TMPTA was also measured. All the samples were extruded in the twin-screw extruder under the same conditions before testing MFR. The first point in the first curve (0 wt % TMPTA) represents the MFR of the extrudate of pure PP powder without any stabilizer. It is clear that the MFR of pure PP powder without stabilizer increases due to thermal degradation of PP after extrusion. In comparison with pure PP powder without any stabilizer after extrusion, the addition of LDPE leads to the

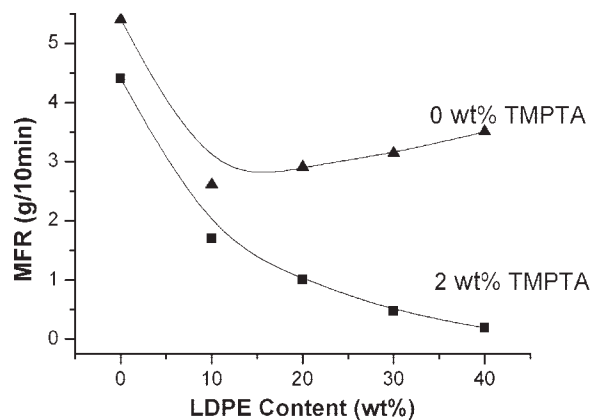


Figure 6 Effect of LDPE content on MFR of the HMSPP.

decrease of the MFR in the absence of TMPTA, and the MFR of PP/LDPE blends increases slightly again with the content of LDPE. However, after adding 2 wt % TMPTA, the MFR of HMSPP decreases with the increase of LDPE content. This is attributed to the fact that some crosslinking occurs, which makes the molecular chains extended and reduces mobility of the chains. When the content of LDPE was high in the HMSPP, more crosslinking structures were formed due to the participation of LDPE.

The melt strength of HMSPP was measured by Rheometer-7D instruments. Here, the diameter of capillary die is 2 mm. Figure 7 shows the effect of LDPE content on expansion ratio and diameter of extrude bar of HMSPP. When the content of LDPE increases, the diameter and expansion ratio of the HMSPP and PP/LDPE blends increases gradually. As we know, in a melt extrusion process, the diameter expansion of polymer depends on relaxation time of molecular chain. The diameter expansion of polymer increases with relaxation time of molecular chain. For a polymer melt, the relaxation time at low shear rate is a function of the size of

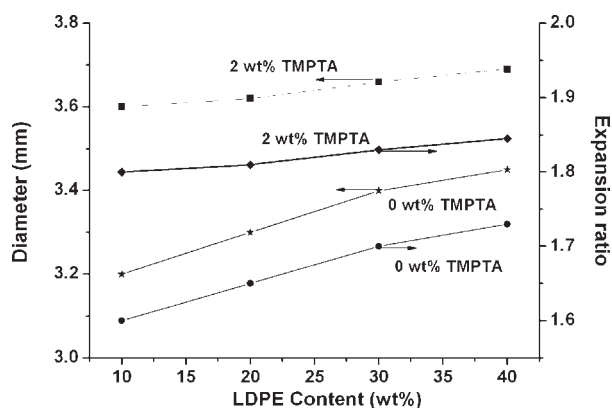


Figure 7 Effect of LDPE content on expansion ratio and diameter of extrude bar of HMSPP.

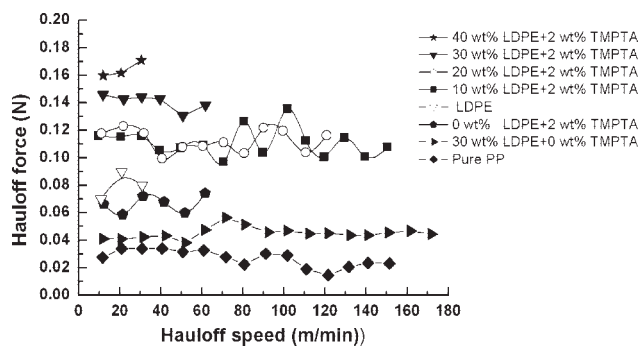


Figure 8 Effect of LDPE content on melt strength of HMSPP.

macromolecules. The formation of crosslinking structures increases the size of macromolecules, which leads to the increase of relaxation time at low shear rate. As a result, the crosslinking promotes the diameter expansion of polymer. At the same time, because the blending of LDPE and PP without TMPTA is simple physical mixture, in which there was no crosslinking reaction; therefore, the expansion ratio and diameter of PP/LDPE blends were smaller than those of HSMPP in spite of the composition.

Figure 8 shows effect of LDPE content on melt strength of HMSPP. The melt strength of the HMSPP increases with the content of LDPE. When the content of LDPE is 40 wt %, the melt strength of the HMSPP is above 16 CN, which is much higher than those of pure PP powder (2.6 CN) and PP/LDPE blends without TMPTA (6.1 CN). It indicates that, after crosslinking reaction, microcrosslinked structure was formed, which strongly increase the melt strength of HMSPP.

The temperature dependence of the melt strength can be further studied by using the Arrhenius type equation as shown in eq. (1) (see Figs. 9, 10). In eq. (1), C is a constant, E is the activation energy of

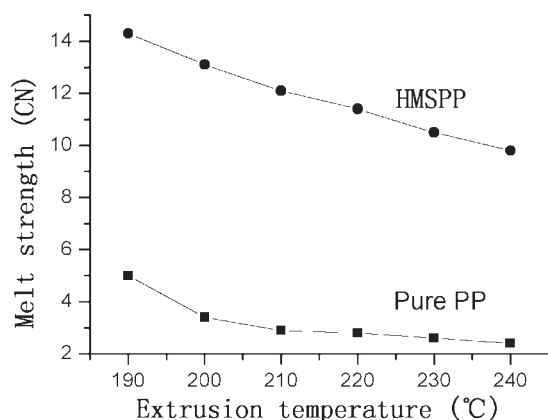


Figure 9 Melt strength of PP versus extrusion temperature.

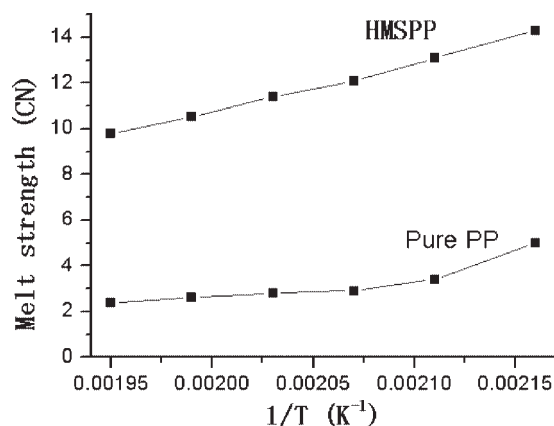


Figure 10 Arrhenius plot for melt strength of PP versus reciprocal of extrusion temperature.

melt strength (J/mol), R is the molar gas constant (8.314 J/mol K), and T is the absolute temperature (K).²⁰

$$\text{Log (melt strength)} = E/RT + \text{Log } C \quad (1)$$

The activation energy of the melt strength for pure PP and HMSPP (obtained from PP/LDPE/TMPTA = 70/30/2) can be calculated from the slope of the high extrusion temperature region (above 190°C) of Figure 10.²¹ Activation energies are shown in Table II. The difference between the activation energy for pure PP and HMSPP was small. This indicates that the temperature dependence of melt strength in the high extrusion temperature region is similar for both PP and HMSPP. According to Eyrings theory,²² activation energy of a polymer is the required energy for individual molecules to jump from one equilibrium state to another. It was found that below a certain level of molecular weight (i.e., 30 carbon units for paraffins), the activation energy increased with the molecular weight, but approached a constant value as the molecular weight was above this critical level of molecular weight. This suggests that for the microcrosslinked structure, only a short segment of the molecular chain can move at a time. Therefore, it is expected that a certain class of polymer with high molecular weight has a constant activation energy value.

TABLE II
Calculated Activation Energy for Melt Strength of HMSPP and Pure PP

Samples	Activation energy for melt strength (J/mol)
Pure PP	18.1×10^3
HMSPP	15.0×10^3

Mechanical properties of the HMSPP

Effect of LDPE content on impact strength is showed in Figure 11. In the absence of TMPTA, impact strength of PP/LDPE blends increases with the content of LDPE. This can be attributed to high impact strength of LDPE. Thus, the addition of more LDPE in the PP leads to the increase of impact strength. When compared with PP/LDPE blends without TMPTA, when 2 wt % of TMPTA is added into the PP/LDPE blends, impact strength is obviously improved. Owing to the crosslinking reaction, many linear chains of PP have been changed into three-dimensional networks, which lead to the increase in the impact strength. In this case, both crosslinking structure and the content of LDPE influence the impact strength of PP/LDPE/TMPTA blends (HMSPP). The relative increment of impact strength of PP/LDPE/TMPTA blends is smaller than that of PP/LDPE blends when the content of LDPE increases from 10 to 40 wt %. This means that the dependence of impact strength PP/LDPE/TMPTA blends on the content of LDPE is weak. From Figure 3, the gel content of PP/LDPE/TMPTA blends increases slowly with the content of LDPE, which confirm a little change of the microcrosslinked structure in the PP/LDPE/TMPTA blends. Therefore, it can be concluded that the impact strength of HMSPP strongly depends on the crosslinking reactions.

Thermal stability of the HMSPP

TGA results of the samples under nitrogen atmosphere at a heating rate of 10°C/min are shown in Figure 12. In comparison with pure PP, the addition of TMPTA obviously improves the thermal stability of PP due to the formation of crosslinking structure. It is noted that the thermal stability of the HMSPP (obtained from PP/LDPE/TMPTA = 70/30/2 by weight) is greatly improved comparing with two

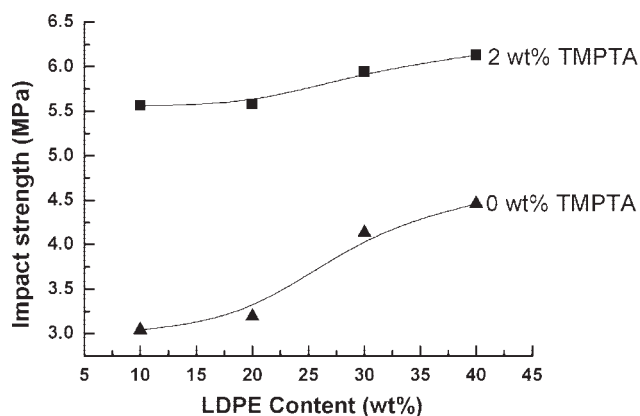


Figure 11 Effect of LDPE content on impact strength of HMSPP.

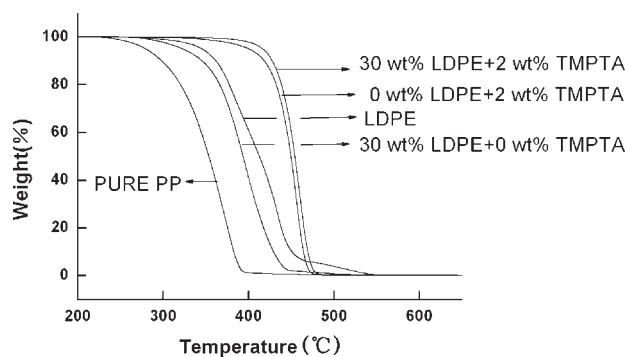


Figure 12 Comparison of TGA curves of the parental resins and the resultant HMSPP.

kinds of the parental resins and the corresponding PP/LDPE blend. The degradation temperature of the HMSPP is higher than those of other samples. For example, the temperatures for the 10% weight loss of PP and the HMSPP are 298°C and 424°C, respectively. The pure PP and PP/LDPE blend without TMPTA degrade completely at temperatures up to about 400°C and 450°C, whereas the temperature for the HMSPP is above 480°C.

Foaming performance of HMSPP

The resultant HMSPP (obtained from PP/LDPE/TMPTA = 70/30/2 by weight) was further tried to produce foaming materials. The foaming process was carried out in TSSL-25 corotating twin-screw extruder with a weight ratio of HMSPP/blowing agent = 100/2. Here, azodicarbonamide (AC) was used as a blowing agent. From Figure 13, it can be seen that the foam cells show opening pore and uneven distribution in the foam produced from pure PP powder as basic resin. This is because pure PP has low melt strength and low viscosity, which cannot have adequate capacity to capture gas produced by AC. In contrast, the foam cells in the foam of HMSPP as blowing material are uniformly distributed and closed. This can be attributed to the existence of microcrosslinked structure in the HMSPP, which results in high melt strength and high viscosity in the melt state of HMSPP. Thus, diffusion rate of foaming gas is relatively low, and HMSPP has stronger ability to enclose gas than that of pure PP. Thus, HMSPP shows better foaming properties than that of pure PP.

CONCLUSIONS

The HMSPP was successfully prepared by *in situ* heat induction reaction, in which pure PP powders without any additives was used as basic resin, and TMPTA as crosslinking coagent and LDPE as blending resin. The results indicated that the melt

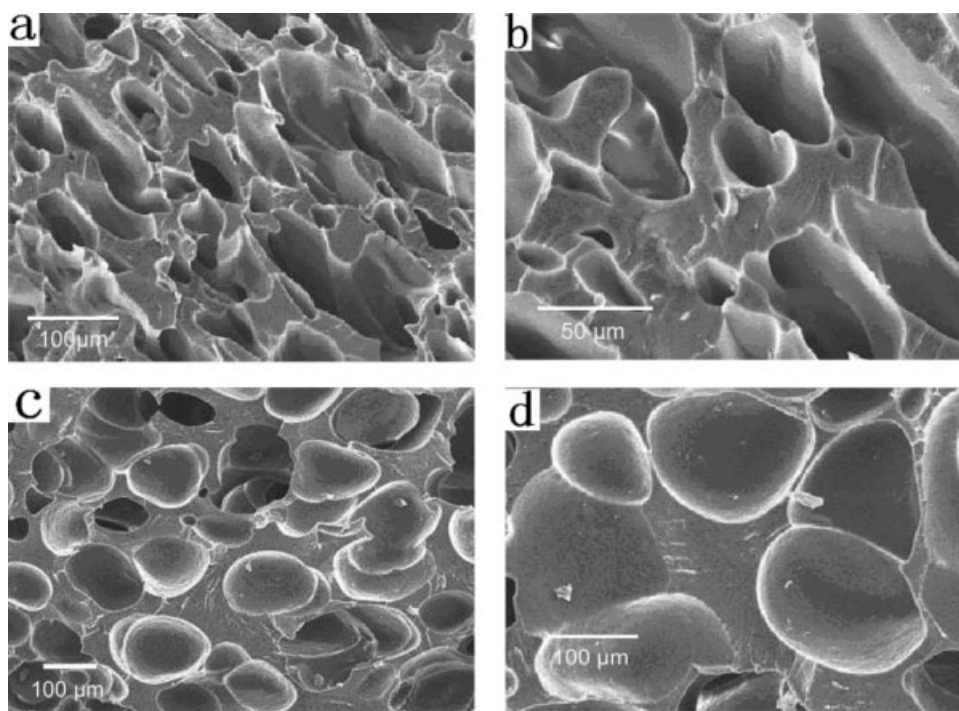


Figure 13 SEM images of foamed pure PP (a and b) and HMSPP (c and d).

strength of HMSPP was greatly improved due to the formation of microcrosslinked structure. The content of LDPE strongly influenced melt strength of HMSPP. The HMSPP had higher impact strength than that of pure PP. Furthermore, the TGA results showed that the thermal stability of the HMSPP was higher than that of the pure PP. Meanwhile, HMSPP showed good processing performance and good foaming properties. The foams produced by HMSPP showed uniform, closed, and independent cells.

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