

Polymer Distribution During Bicomponent Melt Blowing of Poly(propylene)/Poly(ethylene terephthalate) and Its Improvement

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Received 2 October 2001; accepted 21 December 2001

ABSTRACT: Bicomponent melt blown (MB) microfiber nonwovens of poly(propylene) (PP) and poly(ethylene terephthalate) (PET) were produced in this study. It is interesting to analyze the polymer distribution uniformity across the web because it affects many end-use properties. By utilizing the technique of differential scanning calorimetry (DSC), a standard working line between heat of fusion and weight percentage was constructed for mixtures of PP and PET components. The fitted equations were used for determination of a component percentage in a certain position across the MB web. Measurements were conducted from DSC re-heating curves to achieve accurate results. The distribution of polymer varies with polymer mass ratio and processing conditions. The overall uniformity increased with the percentage of PP. When PP is the minor component in the polymer pair, it exhibits notably higher percentage in edge areas across the MB web. These results suggest the phase interface distortion of the polymer melt occurred at the entrance of the MB coat-hanger die tip. The polymer distribution uniformity is improved by adjusting temperature profile of the MB die. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 85: 2885–2889, 2002

Key words: poly(propylene); poly(ethylene terephthalate); differential scanning calorimetry (DSC); microfiber, melt blown

INTRODUCTION

Bicomponent (B/C) melt blown nonwoven fabrics are drawing significant attention from the nonwoven industry because of their great end-use potential. The side-by-side configuration of melt blown fiber is even more interesting because of the possibility of producing finer fibers and greater fiber crimp. With a low melting point component, such as poly(ethylene) (PE) or co-polyester (CoPET), this configuration provides the advantage of greater ease of thermal bonding to different types of substrate layers to develop var-

ious nonwoven composites. The 24-inch Reicofil® MB B/C pilot line (Figure 1), which was commissioned at the Textiles and Nonwovens Development Center (TANDEC) in March 1999, had produced high quality webs in both monocomponent (M/C) and B/C operating modes. The B/C microfibers possess side-by-side cross-sectional geometry.^{1–3}

In this work, PP and PET were utilized to produce bicomponent microfiber MB nonwovens. While producing bicomponent MB webs, it is important to know the uniformity of the web, especially in term of distribution of individual components. Furthermore, from the polymer distribution in the web, one may trace information back to the MB die, achieving data for optimizing operating conditions, polymer selection, and future im-

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Journal of Applied Polymer Science, Vol. 85, 2885–2889 (2002)
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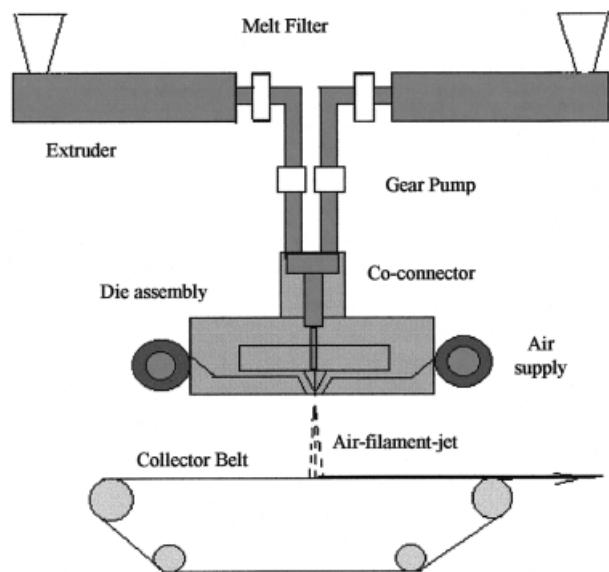


Figure 1 A schematic of the 24-in. Reicofil[®] MB B/C pilot line.⁷

provement in die design. The other aim of this study is to investigate the feasibility of better polymer distribution control by adjusting the MB die temperature profile.

Differential scanning calorimetry (DSC) is a convenient and accurate method for analyzing semicrystalline polymer blends.⁴ Because their melting temperatures are sufficiently different, PP and PET blends can be analyzed by DSC without difficulty.

EXPERIMENTAL

Materials and Equipment

The PP resin (Exxon Mobil Chemical Company), a white pellet with an average diameter size of 3–5

mm, is specially designed for the spunbond process. Its physical properties are as follows: nominal melt flow rate (MFR) = 35 g/10min at 230°C, melting temperature (T_m) = 165°C, glass transition temperature (T_g) = -17°C, and density (ρ) = 0.91 g/cm³. The PET resin (Wellman Inc.) is a semi-dull fiber grade, crystallized chip, with a nominal IV = 0.645 ± 0.017, T_m = 245–250°C, 0.3% TiO₂, and 2.10% (mol %) diethylene glycol. Resins were dried at 120°C for 4 h prior to extrusion.

The PP/PET B/C MB webs with different polymer mass ratios were produced in the Reicofil[®] MB pilot line (Figure 1) at TANDEC, the University of Tennessee, Knoxville. The processing conditions and the descriptions of the webs are presented in Table I.

Instrument and Procedures

In this research, a Mettler thermal analysis system was utilized, which includes a TC11 controller, DSC25, and TMA40 module. The scans cover the temperature range 70–300°C in nitrogen under different heating/cooling rates.

Each scan consists of 4 segments: (1) heating from 70 to 300°C at a rate of 10°C/min; (2) staying at 300°C for 2 min, (3) cooling from 300 to 70°C at -15°C/min, and (4) re-heating from 70 to 300°C at a rate of 10°C/min. The data were used to construct a curve reflecting the relationship between heat of fusion (ΔH_f) and weight percentage of a polymer component. A set of DSC scans was performed for single polymer samples and their mixtures with different ratios, as shown in Figure 2.

The PP/PET B/C MB webs are ~23 in. in width. After being trimmed 1 in. on each side, samples were taken from the right side to left in

Table I Descriptions of MB Webs and Their Production Conditions

| ID | Component | Melt T , °C ^a | Throughput (kg/h) | | DCD, in. ^b | Air R, SCFM ^c | Air T, °C ^d | Belt, ft/min ^e |
|--------|----------------|----------------------------|-------------------|-------|-----------------------|--------------------------|------------------------|---------------------------|
| | | | PET | PP | | | | |
| Bico-1 | 28% PET/72% PP | 315 | 5.76 | 14.8 | 9.9 | 450 | 310 | 90 |
| Bico-2 | 48% PET/52% PP | 315 | 10.79 | 11.69 | 7.9 | 500 | 310 | 95 |
| Bico-3 | 74% PET/26% PP | 315 | 19.14 | 6.58 | 7.1 | 400 | 310 | 108 |

^a Melt temperature.

^b Die-to-collector-distance.

^c Airflow rate.

^d Air temperature.

^e Collector belt speed.

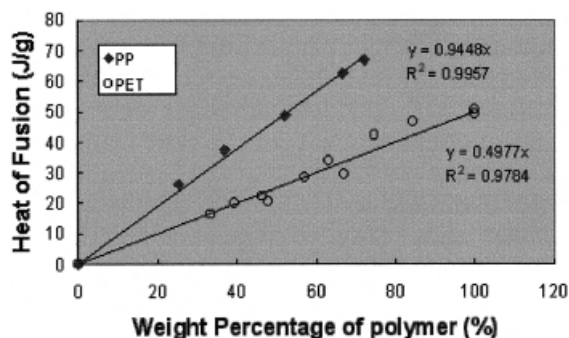


Figure 2 The relationship between heat of fusion and weight percentage of PET and PP measured by DSC.

the cross-machine direction (CD) at the positions of 2'', 4'', 6''... 20''. These samples were analyzed by DSC with the same conditions as already described.

RESULTS AND DISCUSSION

To quantitatively determine the distribution of individual component through the web, a standard curve was developed between the percentage of each component and their ΔH_f with the following assumptions: (a) the area of melting peak in a DSC curve is proportional to the crystallinity of the polymer, and (b) given the same conditions, a semicrystalline polymer develops to the same degree of crystallization.

The DSC data for the polymer mixtures of PP and PET, the linear relationship between ΔH_f and component weight percentage, and the fitted equations are illustrated in Figure 2. Typical DSC curves of PP/PET B/C MB web are presented in Figure 3. The PP endothermic peak can be readily identified around 165°C and PET is at ~253°C. Because the samples were taken from MB webs, the thermal histories were partially recorded in the supermolecular structures of the bico filaments. A heat treatment at a proper temperature above the melting point is necessary to minimize the thermal history effects. Therefore, the data of re-heating lines were taken to construct the standard working lines, which were then used to determine the polymer distribution of PP and PET in the PP/PET B/C MB webs.

Because of its flexible molecular chain, PP is able to rearrange the atoms after extrusion to crystallize with a relatively high rate even under lower temperature. In other words, PP develops its crystal structure to an extensive degree after

extrusion from the die. It is possible for PP to further develop crystals even after web formation. Therefore, the difference in ΔH_f between heating and re-heating is relatively small for PP. On the other hand, the molecular chain of PET is rigid compared with that of PP. This property leads to its low crystallization rate and the dependence on external drawing force for accelerated crystallization. However, in melt blown process, this positive drawing force is only significant in a very short distance from the die in the form of air drag. The microfibrils are rapidly quenched to slightly above the ambient temperature and reach the converter belt (or drum) to form the web.³ The PET parts in the filaments have insufficient time to crystallize before the temperature drops below the T_g ($T < T_g$). Therefore, the PET component is more heavily affected by the processing conditions and thermal history, as shown in Figure 3.

For each B/C web, samples were taken at designated positions across the web for measuring ΔH_f of each component, as described in the *Experimental* section. The ΔH_f of PP and PET components was measured from corresponding melting peaks in re-heating curves. By utilizing the fitting equation of the standard line for PP (Figure 2), the weight percentage of PP in the B/C MB web at each position can be obtained. These calculated weight percentages were plotted against their positions in the web from the right to left in the CD direction (Figure 4). For all three cases, the edge areas present higher weight percentage of PP than the center area. However, PP component in web of 28% PET/72% PP distributed better than the other two cases. In contrast to the case of PP distribution, PET presented higher percentage in

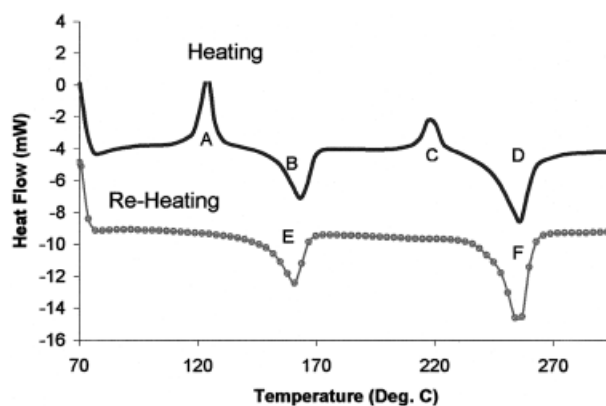


Figure 3 Typical DSC heating and re-heating curves of PP/PET B/C melt blown nonwoven (52% PET/48% PP at the position of 20 in.).

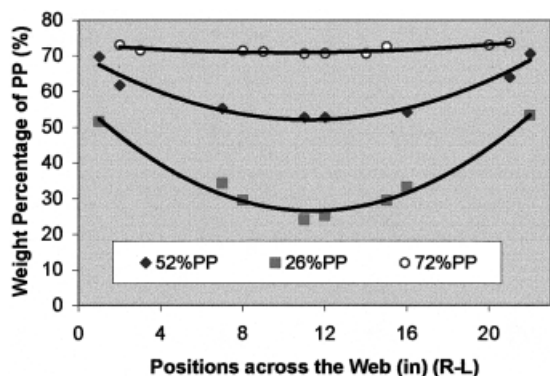


Figure 4 Distribution of PP in the PP/PET B/C webs.

the middle of the web and distributed most uniform in the web of 28% PET/72% PP (not shown in figure).

The coat-hanger die geometry of the 24-in. Reicofil® MB pilot line, different rheological properties of components under the processing conditions, the compatibility of the components and their ratios are among the factors affecting the polymer distribution in the web. The viscosity of the polymer melt is one of the most influential factors. Because PP and PET are incompatible, one may assume their melts in the coat-hanger die slot are a stratified two-phase system. According to Yu and Han,⁶ the pressure gradients in both phases are the same. The velocity profiles of the two phases are very much dominated by their viscosities. As shown in Figure 5, under processing conditions, the viscosity of PP is less viscous than that of PET. The data of polymer distribution and shear viscosity also suggest that the cross-sectional phase interface of the polymer

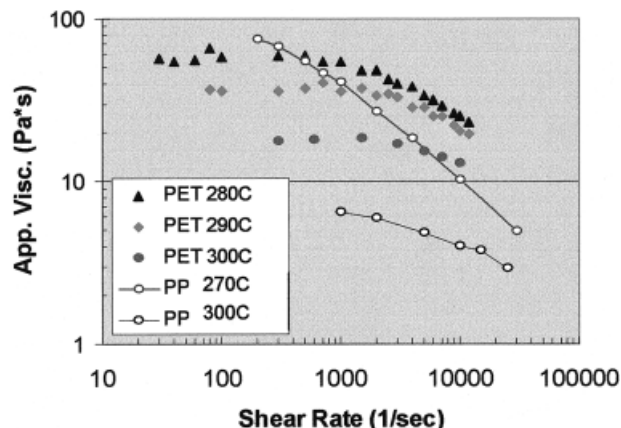


Figure 5 Rheological properties of PET and PP.

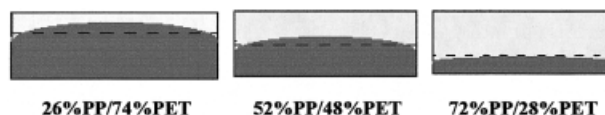


Figure 6 Cross-sectional phase interface profiles of the polymer melts before entering the MB die tip.

melt before entering the die tip changes with the in-input polymer ratio, as shown in Figure 6.

The more viscous PET phase pushed into the PP phase resulting in a concave interface for PP and a convex interface for PET. These results are in agreement with the experimental studies of Han and co-workers,⁶ who studied the co-extrusion system of low-density polyethylene (LDPE) and polystyrene (PS) through a slit die. It was found the interface moved from the less viscous LDPE side to the PS side.

In melt blowing process with a coat-hanger die, the interface distortion at the entrance of die tip controls the polymer distribution across the die; thus, it controls the component uniformity across the melt blown web. When PET is the predominant component in the polymer pair of PP/PET, the polymer distribution needs to be improved. In this study, the two polymer melts flow and distribute in the same coat-hanger, so their temperature can not be controlled separately. However, the die temperature profile along the die width can be adjusted. Three cases were studied as follows.

In Case 1, the die temperature was decreased from 315°C at the center to 299°C at the edge, with a polymer ratio of 15% PP/85% PET. In Case 2, the die temperature was decreased from 315°C at the center to 304°C at the edge, with a polymer ratio of 18% PP/82% PET. In Case 3, the die temperature was set constant at 315 °C across the die width, with a polymer ratio of 18% PP/82% PET. All the other processing conditions of these three cases were kept the same with a throughput of 40 kg/h. The polymer distribution profiles are presented in Figure 7.

Compared with Case 3, the polymer distribution is more uniform with a varied die temperature profile. It is obvious that the adjustment of die temperature profile has an effect on the polymer distribution. Therefore, it is practically possible to improve the polymer distribution across the web by adjusting the MB die temperature profile. When PET is the major component in the B/C MB fibers, the die temperature profile, such as in Case 1, is necessary for better polymer dis-

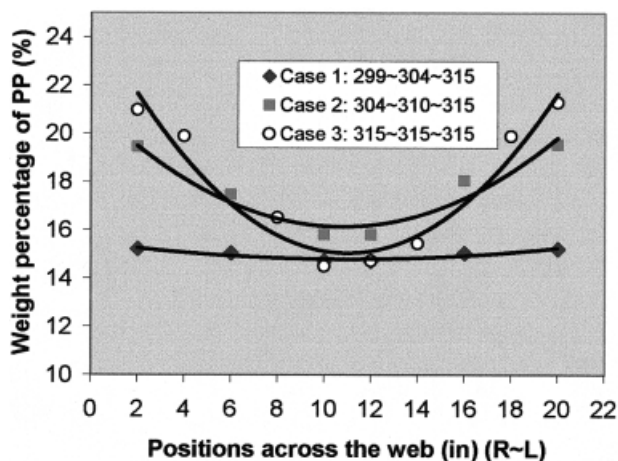


Figure 7 Improving polymer distribution across the MB web by adjusting the die temperature profile. (Case 1: 15% PP/85% PET; Cases 2 and 3: 18% PP/82% PET. The die temperature profiles are listed from edges to center with the unit of °C.)

tribution across the MB web. This observation may result from the differences in heat transfer effectiveness of the two components. Because PET phase takes the major volume of the coat-hanger, it is apparently slow in changing temperature, which results in minor viscosity variation. On the other hand, PP melt is a much thinner layer in the coat-hanger, it may respond quickly to the die temperature profile, resulting in increased viscosity while flowing towards the edges of the die. This effect probably led to better viscosity match of the two polymer melts and improved polymer distribution across the coat-hanger width. Because the polymer melt viscosity is a function of temperature and shear rate (among other factors), as polymer ratio and throughput varies, the viscosity match will change correspondingly. It is expected that a suitable die profile should be explored for a particular situation.

CONCLUSIONS

It is important to analyze the polymer distribution uniformity, which determines many other properties of the bicomponent MB webs. Using the technique of DSC, a standard working line between heat of fusion and weight percentage was constructed for different polymer ratios. The computer-fitted equations were used for determi-

nation of a component percentage in a certain position across the web. The processing (thermal) history must be eliminated by proper heat treatment to achieve accurate results. The measurements from DSC re-heating curves are recommended for the pair of PP and PET.

Under the experimental conditions in this work, PP is less viscous than PET. It exhibited higher percentage in edge areas across the MB web. These results suggested the phase interface distortion of the polymer melt at the entrance of the MB coat-hanger die tip varies with polymer ratio. When PP is the major component and the die temperature is constant across the die width, the polymer distributes more evenly across the web.

The polymer distribution uniformity needs to be better controlled when PP is not the major component in the polymer pair with PET. Adjusting the die temperature profile is found a practically feasible approach.

The authors thank Mr. Anthony Whaley and Mr. Vanston Brantley for their assistance in operating the melt blown line. They are grateful to Dr. Gajanan Bhat for his helpful advice on DSC analysis, and to Dr. David Garner, director of TANDEC, for his encouragement and support. Finally, the authors thank Reifenhauer Company, Germany, and The University of Tennessee for funding this work.

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