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Effect of melt viscosity of polypropylene on fibrillation of thermotropic liquid crystalline polymer in in situ composite film

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Abstract Various grades of polypropylene were melt blended with a thermotropic liquid crystalline polymer, a block copolymer of *p*-hydroxy benzoic acid and ethylene terephthalate (60/40 mole ratio). The blends were extruded as cast films at different values of draw ratio (slit width/film thickness). Fibrillation of TLCP dispersed phase with high fiber aspect ratio (length/width) was obtained with the matrix of low melt flow rate, i.e., high viscosity and with increasing film drawing. Melt viscosities of pure components and blends measured using capillary rheometer were found to decrease with increasing shear rate and temperature. Viscosity ratios (dispersed

phase to matrix phase) of the systems being investigated at 255 °C at the shear rate ranged from 10^2 to 10^4 s⁻¹, were found to lie between 0.04 and 0.15. The addition of a few percent of elastomeric compatibilizers; a tri-block copolymer SEBS, EPDM rubber and maleated-EPDM, was found to affect the melt viscosity of the blend and hence the morphology. Among these three compatibilizers, SEBS was found to provide the best fibrillation.

Key words Liquid crystalline polymers · In situ composite · Melt viscosity · Polypropylene · TLCP Fibrillation · Polymer blend

Introduction

An immiscible blend of a thermotropic liquid crystalline polymer (TLCP) with a thermoplastic resulted in a fiber-reinforced thermoplastic called an in situ composite, the term coined by Kiss (1987). Such composites arise due to the formation of a fibrillar TLCP phase during extensional melt flow. They are interesting because they have several outstanding features. In addition to the improvement on physical and dimensional stability of the matrix, the incorporation of TLCP also enhances the ease of processing, resulting from the reduced overall melt viscosity of the system. Processing conditions including temperature and the shear and elongational forces strongly affect the molecular orientation of the TLCP phase as well as the fiber aspect ratio which determines the final physical properties of the composite.

Research on this subject is reviewed by Dutta et al. (1990), Hanlos and Baird (1995), and Arcierno and Collyer (1996). There are several important factors affecting the morphology, i.e., the size, shape and distribution of TLCP domains in the finished product. These are, for example, blend composition, viscosities of the blend components, interfacial adhesion and processing conditions such as temperature, shear or elongational forces which depend on the mixing procedure and specimen preparation. The effect of the viscosity ratio (dispersed phase to matrix phase) on the morphology of in situ composites has been investigated by several researchers. Good fibrillation was found to be achieved when the viscosity of the TLCP phase is lower than that of the matrix. Heino et al. (1994, 1996) investigated polypropylene of various grades blended with two types of TLCP; a copolymer of *p*-hydroxy benzoic acid and

ethylene terephthalate (60/40 mole ratio HBA/PET), and Vectra A950 (a copolymer of 4-hydroxy benzoic acid and 6-hydroxy-2-naphthonic acid). They found that extruded strands with the most fibrous structure were achieved when the viscosity ratio ranged from about 0.5 to 1. The HBA/PET-PP blend system was also investigated by Rivera-Gastelum and Wagner (1996), who showed that the rheological properties depended, in a complex manner, on composition and applied shear. Both positive and negative deviations from the log additivity rule were observed at low shear rate, and the state of dispersion of TLCP influenced the viscosity reduction of the system. He et al. (1995) reported that a blend of 10% TLCP (a copolyester of 4-hydroxy benzoic acid and 2-chloro-4-hydroxy benzoic acid) and polyethersulfone or polysulfone with the viscosity ratio as low as 0.01 was sufficient for fiber formation. They concluded that in a blend system with low viscosity ratio and low TLCP content, the TLCP phase is easier to deform and has less chance to coalesce, and hence, good fibrillation could be achieved.

Addition of compatibilizer was found to improve the dispersion of the TLCP phase and in some cases enhance the final mechanical properties of the blend systems. Better dispersion of TLCP in the matrix through incorporation of a compatibilizer was expected to be due to the reduction of interfacial tension. Dutta and Baird (1995) and O'Donnell and Baird (1995) used a maleic anhydride grafted polypropylene as a compatibilizer for polypropylene-based composites and found an improvement in the modulus of up to about 30%. Seo (1997) found that maleic anhydride grafted ethylene propylene diene terpolymer (EPDM) could improve both the modulus and the toughness of in situ composites of Nylon 6 and polybutylene terephthalate. Using a tri-block copolymer of styrene (ethylene-butylene) styrene (SEBS), a thermoplastic elastomer as compatibilizer, Bualek-Limcharoen et al. (1999) observed a significant improvement in Young's modulus and in impact strength of in situ composites comprising TLCP (60/40 mol% HBA/PET) and polypropylene. The enhancement of impact strength was due to the action of SEBS as an impact modifier. Better dispersion of the TLCP phase was caused by the improved interfacial adhesion due to SEBS. In addition, SEBS was found to be a more effective compatibilizer than maleic anhydride grafted PP. This might be due to the influence of the elastomer on the rheological properties of the blend system.

In the present study, we investigated the effect of melt viscosity of various PP matrices on fibrillation of TLCP in the composite films, and the effect of elastomeric compatibilizers on melt viscosity and morphology of the blends. Melt flow-rate measurements obtained using a conventional method will be compared with the viscosity measurements performed using a capillary rheometer.

We prepared the specimens in the form of thin films so as to minimize the skin-core morphology and maximize the homogeneity of the sample, i.e., no weld-lines are formed unlike the case of injection-molded specimens (Engberg et al. 1990). The fibrillation of the TLCP phase in the thin films can also be observed directly under a polarized optical microscope. A scanning electron microscope was used to investigate the composite fracture surface.

Materials and methods

Materials

Four grades of polypropylene obtained from HMC Polymer Co. were used in this study; PRO-FAX 6531, PW583, 6331 and 6231 with measured melt flow rates (MFR) of 5, 9, 14 and 28 respectively, and a melting point of 165 °C. For simplicity, these PPs are denoted as PP5, PP9, PP14 and PP28. The dispersed phase was a liquid crystalline polymer, Rodrun LC3000 (from Unitika Co.), which is a copolyester of *p*-hydroxy benzoic acid and ethylene terephthalate (60/40 mol% HBA/PET). The three compatibilizers used were the following elastomers: (1) a tri-block copolymer of styrene (ethylene-butylene) styrene (SEBS, KratonG 1652) kindly provided by Shell Chemical Co.; (2) ethylene propylene diene monomer (EPDM, Royalene 501); and (3) maleic anhydride grafted EPDM (MA-g-EPDM, Royaltuf 490, with 1% MA content) kindly provided by Uniroyal Co. All materials were dried in a vacuum oven at 70 °C for 12 h before use.

Blending and film extrusion

Blend compositions of 10 wt% TLCP and 3 wt% compatibilizer in the PP matrix were used throughout this study. All components were melt blended in a co-rotating twin screw extruder (PRISM TSC-16-TC) with a screw diameter of 16 mm, length-to-diameter ratio (L/D) of 25, a die diameter of 2 mm and a screw speed of 150 r.p.m. The temperature profile was 180/220/220/225/225 °C, representing the temperatures at the hopper zone, three barrel zones, and the die respectively. The extruded strand was immediately quenched in a water bath and subsequently pelletized. At the output rate of 1.2–2.1 kg h⁻¹, the apparent shear rate in the die was estimated to be 400–1000 s⁻¹. In the second step, the dried blend pellets were extruded using a mini-extruder with a screw diameter of 16 mm and a length to diameter ratio of 24, equipped with a cast film line (Rancastle-RCP-0625). The gap of the die lip and the die width were fixed at 0.65 mm and 152 mm respectively. The screw speed was fixed at 70 r.p.m. Two different temperature profiles were used, i.e., 190/220/220/240 °C and 220/240/255/255 °C, representing the temperatures at the hopper zone, two barrel zones, and the die respectively. After exiting the die outlet the film was immediately quenched on a water-chilled roll and uniaxially drawn at take-off speeds ranging from 1 to 6 m min⁻¹ (output rate approximately 0.6–1.2 kg h⁻¹). During this step the apparent shear rate in the die was estimated to be in the range 20 to 45 s⁻¹. The extruded films of draw ratio (slit-width to film thickness) ranged approximately 8–30, were obtained. The film thickness was in the range 70–20 μm.

Viscosity measurement

Melt flow rates of various grades of pure PP and all blends were measured on a melt flow index tester (Kayeness 7053) at 230 °C with an applied load of 2.16 kg (ASTM D1238).

Melt viscosities of all pure components and blends were measured using a Rosand RH710 capillary rheometer at 240 and 255 °C, equaling the die temperatures used for film preparation. A capillary of diameter 1 mm and length of 16 mm ($L/D = 16$) was used for the viscosity measurement. The shear rate was varied from 60 to 10^4 s^{-1} . The end effects (Bagley corrections) and the Rabinowitsch corrections were performed in all cases using two dies (a zero die and a 16 mm die) with flat ends, as specified for the Rosand system.

Morphology

TLCP fibrils dispersed in the thin film of the PP matrix could be directly observed under a polarized optical microscope (Olympus SC-35) at 200 \times magnification. The degree of fiber density per unit viewing area of different systems can then be compared. To see the actual size and shape of the TLCP fibers more clearly, PP was dissolved away by dropping xylene on a small piece of film placed on the heating stage at about 150 °C.

Morphology of the film fracture surface was investigated using a scanning electron microscope (Hitachi S2500) operated at 15 kV. The film fracture surface was prepared by freezing a strip of the blend film sandwiched between two polystyrene sheets in liquid nitrogen for about 30 min and snapping in half. A thin layer of palladium was coated (Hitachi E102 ion sputter) on the specimen to prevent charging on the surface.

Results and discussion

Rheology

Presented in Fig. 1 are the MFR values of pure PP, PP/TLCP blends, and blends containing a compatibilizer (SEBS, EPDM or MA-g-EPDM). Maximum errors were about ± 1 unit for the samples with high MFR

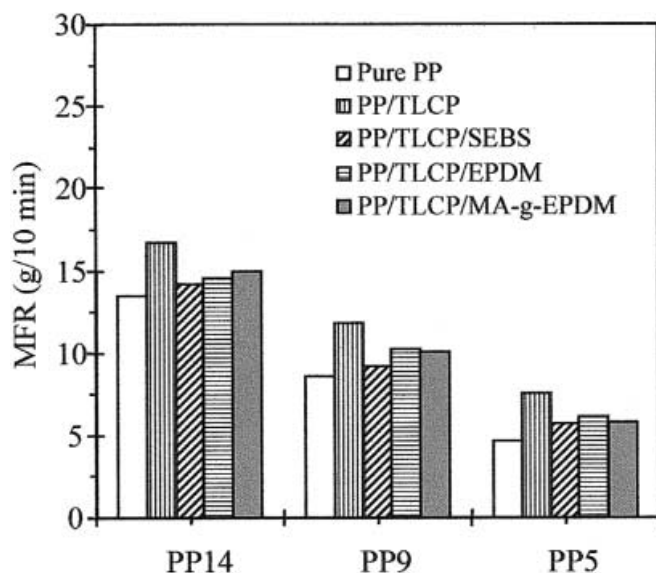


Fig. 1 Melt flow rate (MFR) of pure PPs (PP5, PP9 and PP14), PP/TLCP blends and blends containing compatibilizers (SEBS, EPDM and MA-g-EPDM)

and less for the samples with low MFR. When very low viscosity TLCP was added to PP, the blend MFR increased (and hence viscosity decreased). After addition of 3 wt% compatibilizer, the MFR of all blends decreased, implying that the apparent viscosity of the blend had increased. This leads us to expect that some of the elastomeric compatibilizer, in particular SEBS, is located at the interface between the fiber and the matrix having the two styrene end blocks embedded in the TLCP phase and the middle EB block in the PP matrix. This would enhance molecular entanglement at the interface so that at low shear rate the dispersed phase might have a certain level of resistance to flow.

Melt flow curves of TLCP and PP of different grades measured at 240 and 250 °C are shown in Fig. 2a and b

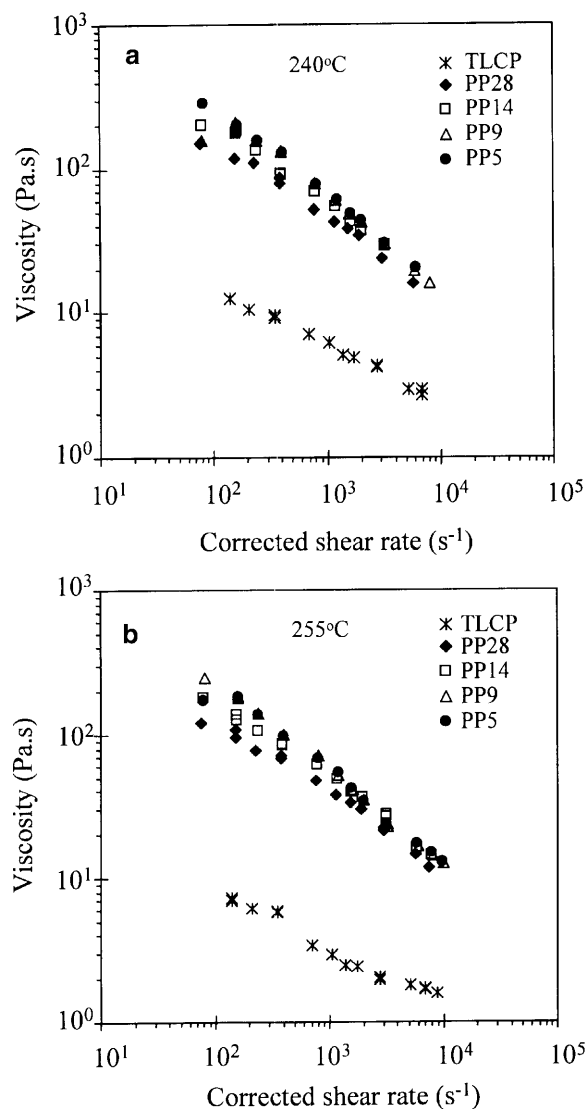


Fig. 2 Melt viscosity versus corrected shear rate for PPs and TLCP measured at (a) 240 °C and (b) 255 °C

respectively. It can be seen that the true viscosity of TLCP is very much lower than those of the pure PPs. As expected, the true viscosity of PP with low MFR is higher than that of PP with higher MFR. All pure components exhibit shear thinning behaviour due to the shear-induced chain orientation and hence the reduction of entanglement density at higher shear stress levels (Dealy and Wissbrun 1990). At all shear rates, the viscosity of TLCP at 255 °C is lower than that at 240 °C, demonstrating the improvement of flow behaviour of the TLCP melt with increasing temperature in this range. Our results are in good agreement with Tormes et al. (1998) who investigated the temperature dependence of the viscosity of LC3000 (the same TLCP used in our work) at a shear rate of 1 s^{-1} . Their results showed that the viscosity of TLCP decreased rapidly as the temperature increase in the range 200–260 °C, but was almost temperature independent in the range 260–300 °C. The lesser dependence of the viscosity on temperature in the high temperature range was probably caused by an opposing effect, that the increase in viscosity with temperature is due to the decrease in the degree of molecular ordering as the mesophase approaches the transition point to the isotropic melt. The shear thinning behaviour of TLCP is observed at both temperatures with a value of the exponent $n = 0.6$ in the power law equation, $\eta = k \dot{\gamma}^{(n-1)}$. On the other hand, the viscosity of each grade of PP does not significantly vary at these two temperatures. As a consequence, the viscosity ratio (defined as the ratio of the viscosity of TLCP to that of PP), $\rho = \eta_{\text{TLCP}}/\eta_{\text{PP}}$, at 240 °C is higher than that at 255 °C as shown in Fig. 3a and b respectively. The viscosity ratio at 240 °C lies in the range 0.07–0.23, whereas that at 255 °C is in the range 0.04–0.15. It can be seen that the viscosity ratio increases with increasing shear rate, due to the faster drop of the viscosity of PP than that of TLCP at high shear rate.

Melt viscosity plots of the blends of 10 wt% TLCP and PP with MFR 5, 9, and 14 are shown in Fig. 4a, b and c respectively. Viscosity plots of the blends containing 3 wt% compatibilizer (SEBS, EPDM or MA-g-EPDM) are also presented. It can be seen that for PP5 (Fig. 4a), the viscosity of all blends (with and without compatibilizer) are approximately the same. Compared with systems PP9 (Fig. 4b) and PP14 (Fig. 4c), the addition of compatibilizer, especially SEBS, reduces the viscosity of the blends and the effect is more pronounced in the shear rate region 60–500 s^{-1} . This result contradicts the observation obtained by measuring the melt flow rate. The discrepancy in the results obtained from these two methods might be due to the different ranges of shear rate involved, and the much different lengths of the two dies resulting in different entrance pressure drops. The results from the capillary rheometer showed, for instance, for PP5 at 255 °C and an apparent shear

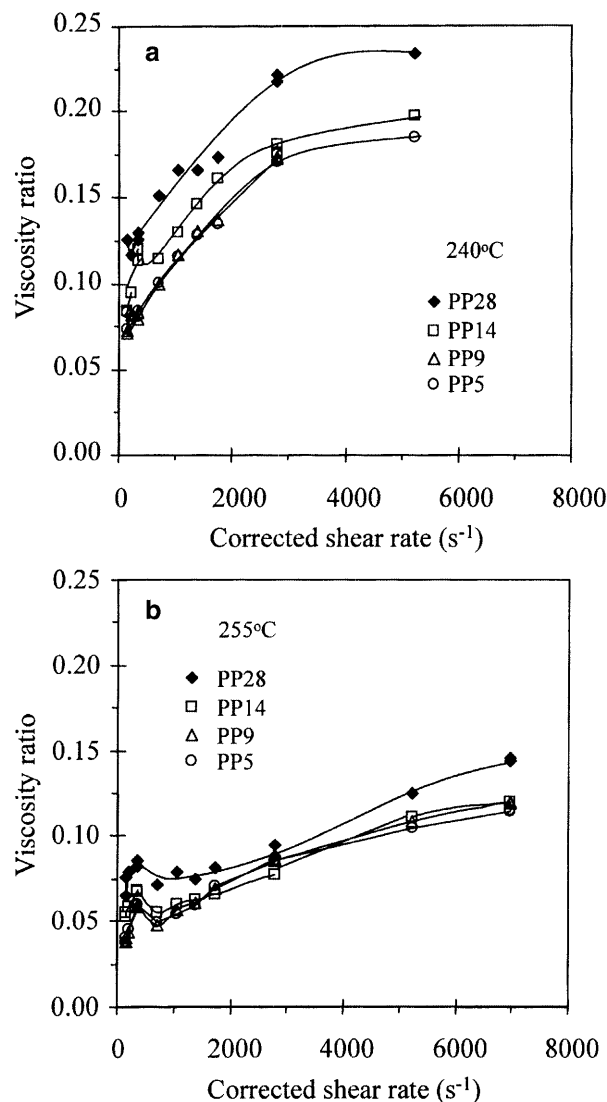


Fig. 3 Viscosity ratio versus corrected shear rate at (a) 240 °C and (b) 255 °C

rate of 120 s^{-1} , a pressure drop of 2.0 MPa in the long die, and a pressure drop of about 0.2 MPa in the short die. It should be noted that Bagley correction was not applied to the data obtained from the MFR tester as was the case for the capillary rheometer.

Gupta and Purwar (1984) investigated the melt viscosity of PP/SEBS blends using a capillary rheometer and observed a minimum viscosity in a blend containing 5% SEBS. They suggested this to be due to the high deformability of the small domains of the SEBS dispersed phase. Similarly, the reduction in the viscosity for PP/TLCP/SEBS blends obtained from the capillary rheometer at high shear rates might be due to the same effect.

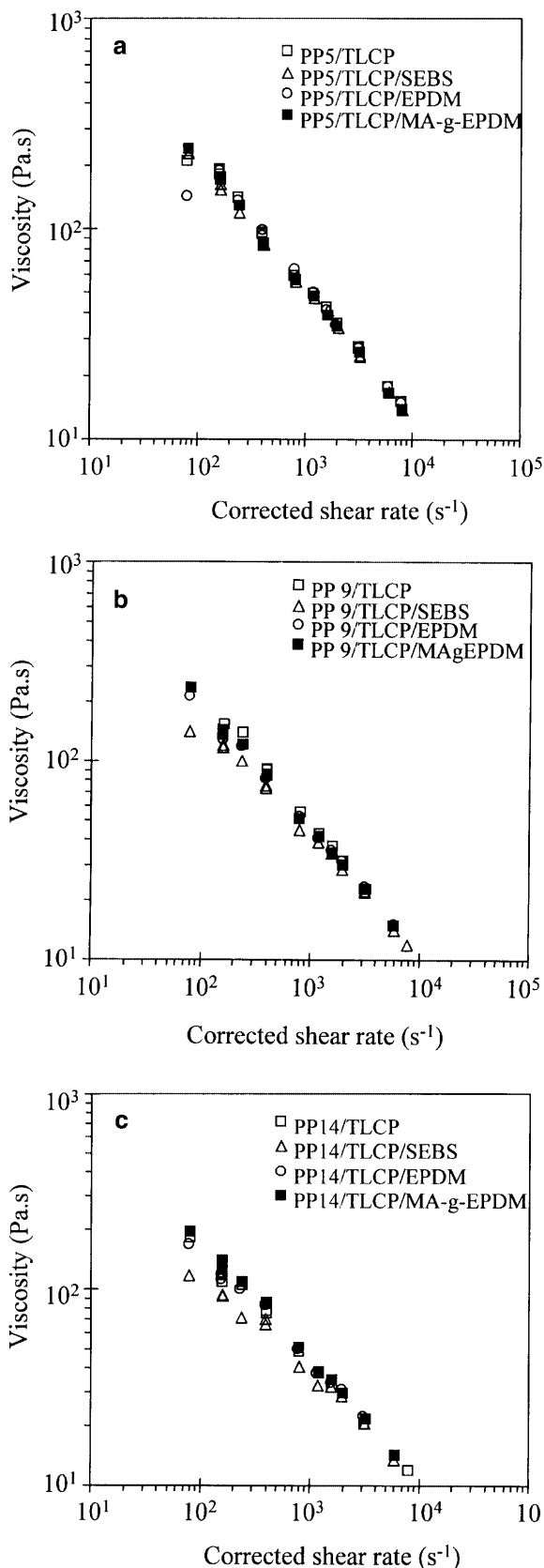


Fig. 4 Melt viscosity versus corrected shear rate for PP/TLCP blends without and with compatibilizer (SEBS, EPDM and MA-g-EPDM) measured at 255 °C, for different PP grades: (a) PP5, (b) PP9 and (c) PP14

Morphology

Effect of melt viscosity of PP matrix

The morphology of in situ composite films observed with an optical microscope are illustrated in Fig. 5. The films were prepared using a die temperature of 255 °C, a draw ratio of 30, and PP matrices of different MFR; (a) PP5, (b) PP9, (c) PP14 and (d) PP28. The effect of the melt viscosity of PP on fibrillation of TLCP is clearly demonstrated here. The PP5 matrix gives the thinnest TLCP fibers and also the highest number of fibers per unit viewing area, whereas PP28 gives the thickest and shortest fibers, and also the lowest number of fibers per unit area. As discussed above, molten PP5 is the most viscous fluid among all the PPs used in this study, and thus could exert the highest shear force to break up the TLCP phase into fine droplets and deform them into thin elongated fibrils. SEM micrographs of the fracture surface of the films (the same series as shown in Fig. 5) are presented in Fig. 6. The film specimens were fractured perpendicular to the machine direction. The pull-out feature of TLCP fibers are seen in every sample, suggesting poor interfacial adhesion between the fibers and the matrix. The effect of melt viscosity of the PP matrix on fibrillation of TLCP is evident from the size of the fibers. The approximate widths of the fiber (measured at the point adjacent to the matrix) are about 6, 5, 4 and 3 μm for the films prepared from PP matrices with MFR 28, 14, 9 and 5 respectively. That means the PP matrix with lower MFR (higher viscosity) generates thinner TLCP fibers. The highest number of fibers per unit cross-sectional area is also evident in the PP5/TLCP composite film. This suggests that the fiber dispersion is improved with increasing melt viscosity of the matrix.

Effect of film drawing

The effect of the additional drawing of the film on the length of TLCP fibers is shown in Fig. 7. These are optical micrographs of TLCP fibers extracted from the films produced at a draw ratio of 8 (column I) and a draw ratio of 30 (column II). The samples in both columns are prepared from the matrices; (a) PP5, (b) PP9, (c) PP14 and (d) PP28. Hot xylene was used to extract the fibers and spread them out in order to minimize overlapping. Comparison of the micrographs in the two columns reveals that TLCP fibers in films produced at the draw ratio 8 are shorter than those

Fig. 5 Optical micrographs of PP/TLCP films prepared from (a) PP5, (b) PP9, (c) PP14 and (d) PP28

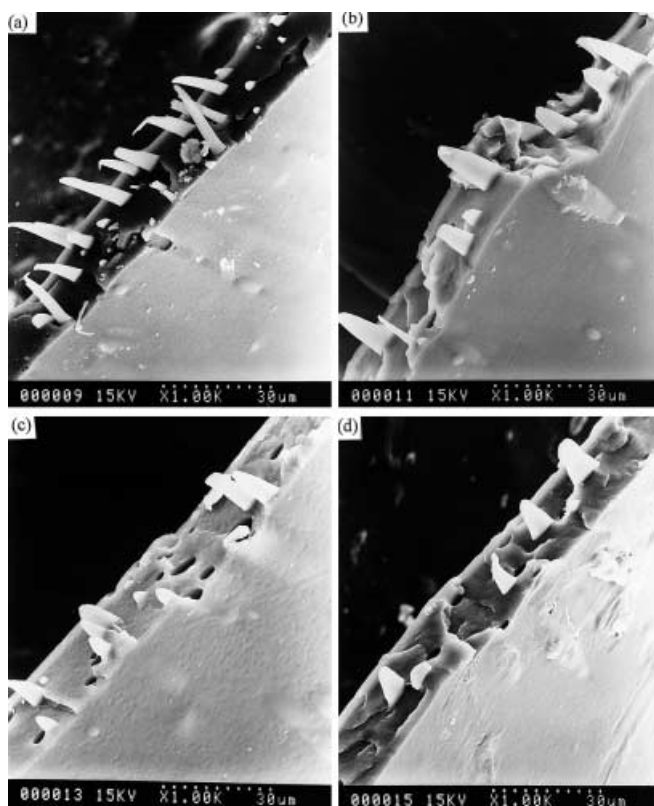
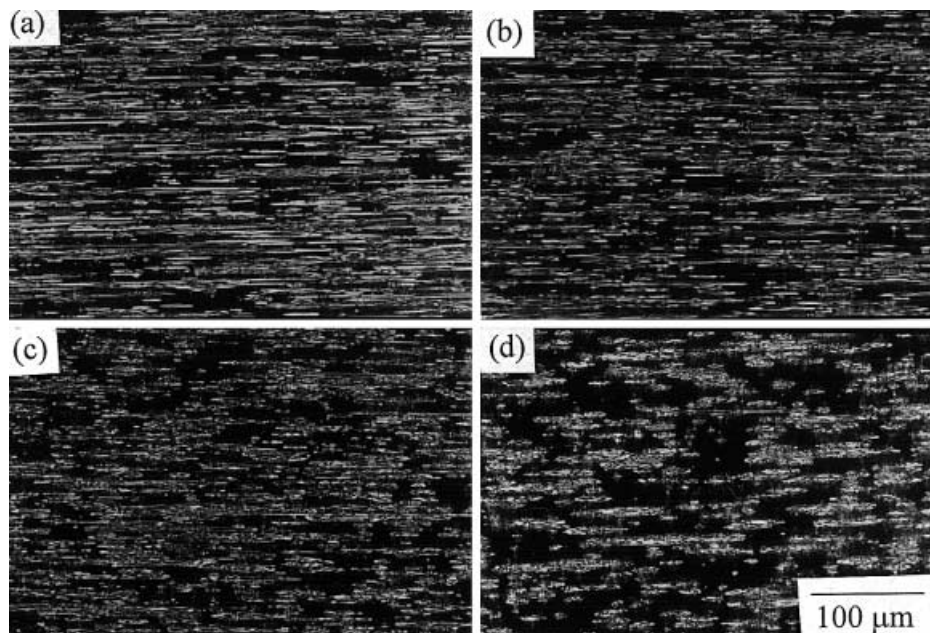


Fig. 6 SEM micrographs of fractured surfaces of PP/TLCP films prepared from (a) PP5, (b) PP9, (c) PP14 and (d) PP28

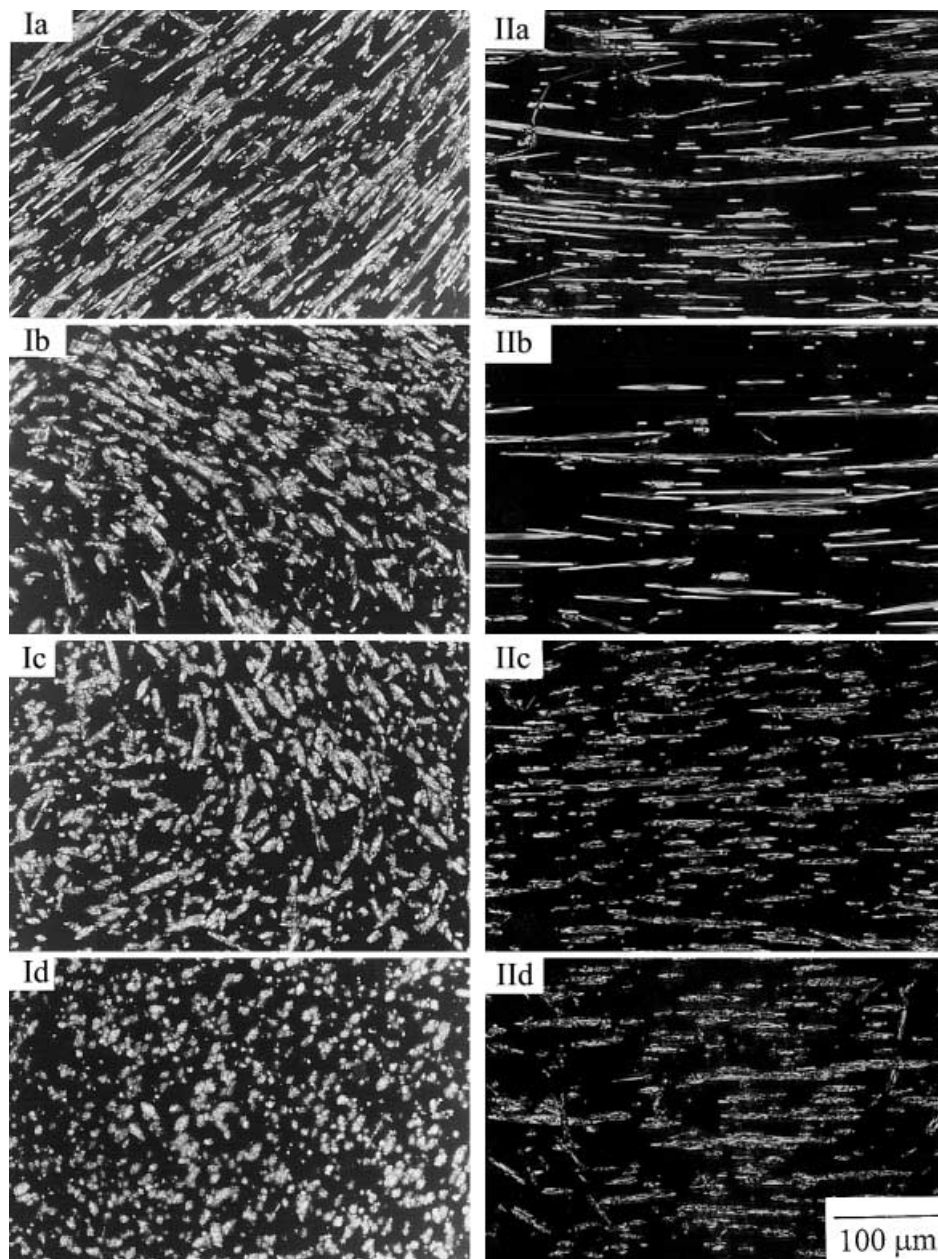
prepared at the draw ratio 30. For the film with the PP28 matrix, produced at draw ratio 8, the TLCP dispersed phase appear as droplets. The dispersed phase becomes

more elongated as the MFR of PP decreases. Films produced at the draw ratio of 30 exhibit elongated TLCP fibers. It is evident that the additional drawing of the films just after exiting the die greatly increase the fiber aspect ratio. Our observation agrees with that of Qin (1994) who studied polyblend fibers of PP/TLCP (Vectra A900 and B950) and concluded that the drawing at high temperature preserved the long length of TLCP fibrils due to the high degree of mobility of TLCP. Although the shear viscosity ratio obtained from the capillary rheometer of each blend system in this study is quite low (i.e. in the range 0.04 to 0.15), compared to that reported by Heino et al. (1994). We have demonstrated that it is possible to obtain good fibrillation in the last step of specimen preparation, i.e., film extrusion at high draw ratio. In this case, the extensional flow played an important role on elongation of the TLCP droplets and therefore the ratio of elongational viscosities should be considered. We have estimated the apparent elongational viscosities by using the relations described by Cogswell (1981). From the values of the apparent shear viscosities obtained at the apparent shear rates of 60 s^{-1} and 1200 s^{-1} , from the pressure drops at the zero die, and from the power law indices for PP and TLCP (0.4 and 0.6 respectively), the apparent elongational viscosity ratios of TLCP/PP5 to TLCP/PP28 systems were found to be in the range 0.06 to 0.23 at 60 s^{-1} shear rate and 0.12 to 1.22 at 1200 s^{-1} shear rate.

Effect of compatibilizers

The effect of compatibilizer on fibrillation of the TLCP dispersed phase is shown in Fig. 8. These are

Fig. 7 Optical micrographs of TLCP fibers extracted from PP/TLCP films prepared from (a) PP5, (b) PP9, (c) PP14 and (d) PP28: films were prepared at draw ratio 8 (column I) and 30 (column II)



optical micrographs of TLCP fibers extracted from various composite films prepared from PP5 with and without 3 wt% of a compatibilizer; (a) no compatibilizer, (b) SEBS, (c) EPDM and (d) MA-g-EPDM. SEM micrographs of the fracture surface of the same series of specimens are illustrated in Fig. 9. It is clearly seen that TLCP fibrils in the composites containing compatibilizer are thinner than in the uncompatibilized specimen. Finer and shorter fiber pull-out is observed in the film containing SEBS, indicating the improvement on the dispersion of the TLCP phase. SEBS thus seems to be a more effective compatibilizer than the other two. This is due to the

tri-block structure of the thermoplastic elastomer, SEBS, where the middle EB block is compatible with PP and the styrenic blocks at the two ends are compatible with the aromatic groups of TLCP. Hence, SEBS should be present at the interface, with each block diffused into corresponding phases of similar chemical structure. This will help reduce the interfacial tension between PP and TLCP phases and therefore improve the dispersion of TLCP. As reported in our previous work (Bualek-Limcharoen et al. 1999), significant improvement of tensile modulus and impact strength were observed in the SEBS compatibilized blend of PP (MFR = 12) with TLCP.

Fig. 8 Optical micrographs of TLCP fibers extracted from PP5/TLCP films containing (a) no compatibilizer, (b) SEBS, (c) EPDM and (d) MA-g-EPDM

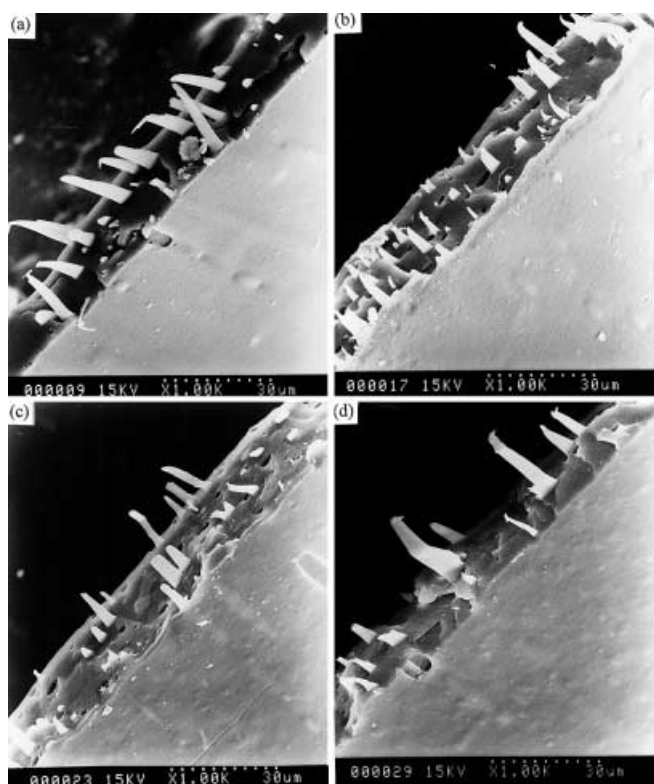
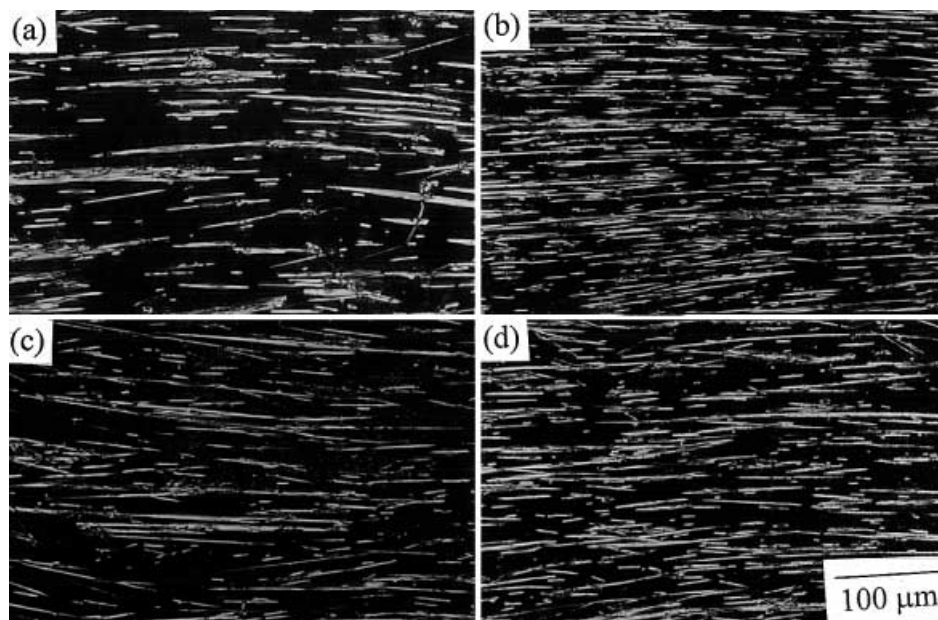


Fig. 9 SEM micrographs of the fractured surfaces of PP5/TLCP films containing (a) no compatibilizer, (b) SEBS, (c) EPDM and (d) MA-g-EPDM

Conclusion

TLCP (Rodrun LC3000) was melt blended with different grades of polypropylene (MFR 5, 9, 14 and 28). The specimens were prepared in the form of extruded films at various draw ratios. Melt viscosities of these blends were measured using a capillary rheometer. The effect of melt viscosity of the PP matrix, as well as the effect of elastomeric compatibilizers on fibrillation of TLCP were investigated. We have demonstrated that, although the shear viscosity ratio of PP/TLCP in this study is quite low (from 0.04 to 0.15 at 255 °C), long TLCP fibrils can be formed in the last step by drawing the extruded films at high draw ratio. Addition of an elastomeric compatibilizer caused a reduction in the blend melt viscosity as measured by a capillary rheometer. However, the MFR values showed an increase in viscosity with the addition of an elastomeric compatibilizer. The discrepancy might be due to the higher molecular entanglement at the interface caused by the added elastomer and this effect resists the shear flow especially at the low shear rate used in MFR measurement.

The morphology of the blends suggests that all compatibilizers help improve dispersion of the TLCP phase. SEBS is found to be a more effective compatibilizer than EPDM and MA-g-EPDM, as longer TLCP fibers are observed in the former.

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