

Extruded Blends of a Thermotropic Liquid Crystalline Polymer with Polyethylene Terephthalate, Polypropylene, and Polyphenylene Sulfide

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SYNOPSIS

Structure–property relationships were investigated for blends of a polyester-type thermotropic liquid crystalline polymer (LCP) with polyethylene terephthalate (PET), polypropylene (PP), and polyphenylene sulfide (PPS). The polymers were melt blended in a twin-screw extruder and the blends were extruded to strands of different draw ratios. Tensile properties of the blends were determined as a function of LCP content and draw ratio and compared with the results of morphological and rheological analyses. In general, the strength and stiffness of the matrix polymers were improved with increasing LCP content and draw ratio. At a draw ratio of 11, the blends of PET/30 wt % LCP exhibited a tensile strength about three times and an elastic modulus nearly four times that of pure PET. All blends exhibited a skin/core morphology with thin fibrils in the skin region. The formation and the sizes of the fibril-like LCP domains in the matrices were found to depend on LCP content and the viscosity ratio of the blend components.

INTRODUCTION

The main aim in blending thermotropic main-chain liquid crystalline polymers (LCPs) with thermoplastics is to achieve a composite-like microstructure and a mechanical reinforcement of the matrix polymer. A small amount of LCP can also act as a processing aid for thermoplastics that are difficult to process.

Blends of LCPs and thermoplastics consist of two phases since the two polymers are generally immiscible. The dispersed LCP phase exists as small spheres or fibers in the matrix and a skin/core morphology is created. The LCP phases are more oriented in the skin region and less oriented or in spherical form in the core.^{1–3} The formation of fibrillar LCP domains in the matrix depends on the ability of the rigid rod-like LCP molecules to orient in the direction of the melt flow. This orientation is retained when the material is solidified and a highly ordered fiber structure is obtained. The size, shape,

and distribution of the LCP domains in the matrix depend on the LCP content, processing conditions, viscosity ratio of the blend components, and the rheological characteristics of the matrix.^{4,5} In particular, sufficient shear forces are needed to obtain good mixing and extensional flow to deform the dispersed LCP domains into fibrillar shape. Increased orientation of the LCP microfibrils by additional drawing can greatly improve the mechanical properties of the blend.^{4,6–8}

In this article, we report on blends of a polyester-type thermotropic LCP with three different thermoplastics blended in a twin-screw extruder and extruded to strands of different draw ratio. As a polyester, polyethylene terephthalate (PET) is an interesting matrix polymer for a polyester-type LCP. Polypropylene (PP) is a common commodity plastic, and polyphenylene sulfide (PPS) is a high-performance engineering thermoplastic. In our earlier studies,⁹ we reported on the properties of similar blend systems processed by injection molding. In the present study, the possibility of improving the physical properties of extruded blends by enhancing the orientation of the LCP domains through elongational drawing was investigated. Strands of dif-

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ferent diameters were extruded with different take-up speeds. The effects of LCP content and draw ratio on the mechanical properties and morphology of the blends were studied and the results related to rheological measurements.

EXPERIMENTAL

Materials

The liquid crystalline polymer used in this work was Vectra A 950[®] produced by Hoechst Celanese. It is a totally aromatic polyester-type thermotropic main-chain LCP believed to consist of *p*-hydroxybenzoic acid (HBA) and 2,6-hydroxynaphthoic acid (HNA). The following properties of the LCP are given by the manufacturer: density, 1.40 g/cm³; melting point, 280°C; tensile strength, 165 MPa; elastic modulus, 9,700 MPa; and elongation at break, 3.0%.¹⁰

The matrix thermoplastics used in the experiments were polyethylene terephthalate (PET Arnite DO4 300 from Akzo), polypropylene (PP VB 65-11B from Neste), and polyphenylene sulfide (PPS Fortron 0214P1 from Hoechst Celanese). All polymers were supplied as extruded pellets.

Blending

Dry pellets of LCP and the matrix thermoplastics were manually mixed in a barrel. The amounts of LCP used in the blends were 5, 10, 20, and 30% by weight. The mixed polymer pellets were then carefully dried before melt blending in a Conair Micro-D dehumidifying dryer. The drying conditions for the blends, shown in Table I, were chosen separately for the different matrix polymers.

The melt blending of the materials was done with a Berstorff ZE 25 × 33D corotating twin-screw extruder. The cylinder temperatures of the extruder ranged from 300 to 310°C for PPS/LCP blends and from 285 to 290°C for the other blends. The screw speed was 150 rpm. The extrudate was immediately

Table I Drying Conditions for the Blends

Material	<i>T</i> (°C)	<i>t</i> (h)
LCP	155	5
PET/LCP	120 and 160	3 4
PP/LCP	70	2
PPS/LCP	140	3

Table II Cylinder Temperatures (°C) of the Extruder

Material	<i>T</i> ₁	<i>T</i> ₂	<i>T</i> ₃	<i>T</i> _{die}
LCP	280	280	285	285
PET	280	275	275	275
PET/LCP	280	275	275	275
PP	210	210	220	220
PP/LCP	280	280	285	285
PPS	285	285	290	290
PPS/LCP	285	285	290	290

quenched in a water bath and pelletized. After extrusion, excessive moisture was removed from the blends by drying them again in the dehumidifying dryer for about 1 h.

Processing

Before extrusion, the blends and the pure polymers were dried in a Conair Micro-D dehumidifying dryer. The drying conditions were the same as shown in Table I.

The extrusion was carried out with a Brabender Plasti-Corder PLE 651 laboratory single-screw extruder connected to a belt capstan designed by Nokia Cables. The hot extrudate was immediately quenched in a water bath, and it was drawn at different speeds of the take-up machine to form strands of different diameter. The speed of the belt capstan ranged from 10 to 40 m/min. The draw ratio for each strand was determined as the ratio between the die and the strand cross-sections (S_0/S_s). The dimensions of the round hole capillary die were length (*L*) 30 mm, diameter (*D*) 5 mm, and thus *L/D* 6. The cylinder temperatures of the extruder are shown in Table II. In this stage, the blends of LCP and PPS were processed, without difficulty, at lower temperatures than recommended for PPS. The screw speed was maintained at 100 rpm for all blends.

Testing and Analysis

The mechanical properties of the extruded strands in the fiber direction were characterized by measurements of elastic modulus, tensile strength, and strain at break.

Before testing, the samples were maintained at the conditions of the testing room for at least 88 h.

An Instron 4204 tensile testing machine equipped with a computer was used in the testing. The test speed was 3 mm/min. The elastic modulus was de-

terminated as Young's modulus for the linear part of the curve between the initial region and the yield. Since the thickness of the samples varied, the diameter of each sample was measured before the test. The gauge length used for the calculation of the strain was 100 mm, which was also the distance between the grips before the test. The measurement was interrupted if the strain reached 35%. The results of the tensile tests are averages of at least five parallel samples.

The morphology of the fractured surfaces of the extruded strands was investigated with a JEOL JXA-840A scanning electron microscope (SEM). The samples were fractured at liquid nitrogen temperature, and the fractured surfaces were coated with a 15-nm layer of gold. Particular attention was paid to the effect of LCP content and draw ratio on the blend morphology and fiber formation.

The melt rheology of LCP and of the pure matrix polymers was investigated by measuring their melt viscosities in shear flow. This was done using a Göttfert Rheograph 2002 capillary viscosimeter. The measuring temperature was 290°C for PET and PP and 310°C for PPS. The L/D ratio for the die was 20 mm/0.5 mm, but a bigger die of 30 mm/1 mm had to be used for PET because it did not pass through the smaller die at the measuring temperature. The shear rate was varied from 5 to about 10,000 1/s.

RESULTS AND DISCUSSION

Mechanical Properties

Figure 1 shows the dependence of elastic modulus, tensile strength, and strain at break of pure LCP on the draw ratio in extrusion. When the draw ratio was increased from about 3 to 11, the tensile strength was increased by 68% to 274 MPa. The elastic modulus was increased even more significantly, by about 140%, up to 16,000 MPa. But while the strength and stiffness of LCP increased, the strain at break decreased to about 3%.

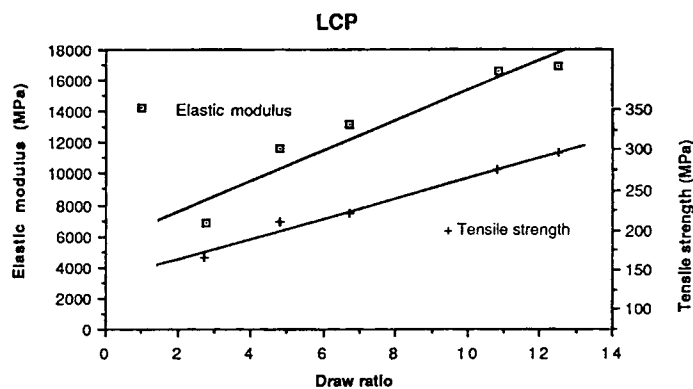
This clear dependence of the mechanical properties of LCP on draw ratio is explained by the ability of the LCP molecules to orient themselves in the direction of the melt flow during processing. In extrusion, the die and the belt capstan direct strong uniaxial elongational forces toward the molten material, leading to uniaxial orientation of the LCP molecules and further to an anisotropic fiber structure and properties characteristic of LCPs. This behavior of LCP makes it of interest to blend it with

thermoplastics for the purpose of obtaining a composite-like fiber structure and mechanical reinforcement of the matrix.

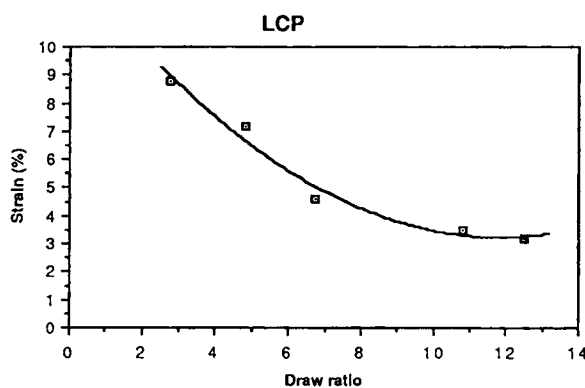
Tensile properties of the extruded blends of different compositions are presented as functions of draw ratio in Figures 2–4. The results of the tests showed that LCP acted as a mechanical reinforcement in all blends studied. Its addition to the thermoplastic matrices increased both the strength and stiffness of the matrix polymers while strain at break was generally decreased. The magnitude of the reinforcing effect depended significantly on LCP content but also on draw ratio. The effect of draw ratio on stiffness became more important at higher LCP contents as can be seen in Figures 2, 3, and 4. This effect was not found in pure PET, PP, or PPS, whose tensile properties remained constant at different draw ratios.

Tensile properties of extruded blends of LCP and PET at different compositions are presented as a function of draw ratio in Figure 2. The tensile strength and elastic modulus of the blends changed in a regular and linear way as a function of LCP content and draw ratio, respectively. The tensile strength and elastic modulus for pure PET were about 45 and 2,085 MPa. When 10 wt % LCP was blended with PET, both properties reached values about 1.7 times as high. After an addition of 30 wt % LCP, the tensile strength increased to almost three times and the elastic modulus to nearly four times the value for pure PET. These increases were achieved with a draw ratio of only 11. LCP thus significantly improved the mechanical strength and stiffness of PET, and the degree of reinforcement was highly dependent on LCP content. The draw ratio became more important at higher LCP contents, though higher draw ratios than used in this study might lead to higher stiffnesses even at low LCP contents. The basic reason for the reinforcement became evident in the SEM studies (Figs. 5 and 6), which showed good fiber formation in PET/LCP blends. The strain at break was generally about 2–3% if the LCP content exceeded 10 wt %. Longer elongations were achieved at smaller LCP contents.

Zhuang et al.¹¹ found increase in tensile strength and elastic modulus and reduction in elongation of extruded and melt-spun PET/LCP blends with increasing LCP content. The relative improvements they report are slightly poorer than ours. Bhattacharya et al.¹² found similar effects with melt-spun blends. Ko et al.¹³ report that the modulus of blends of an HBA/HNA-based LCP and PET increased only at high LCP contents (80 wt %) with conventional extrusion, but the effect was also found at



a)



b)

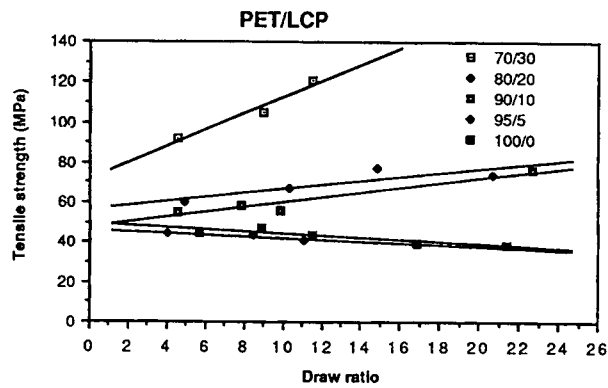
Figure 1 (a) Tensile strength and elastic modulus and (b) strain at break of the extruded LCP strands vs. draw ratio.

lower LCP contents with higher screw speeds. Amano and Nakagawa¹⁴ investigated the drawing behavior of blends of an HBA/PET-based LCP and PET using conventional and microwave heating. Higher moduli were obtained and drawn rod samples of the blends were produced under lower tensions and temperatures than for pure PET.

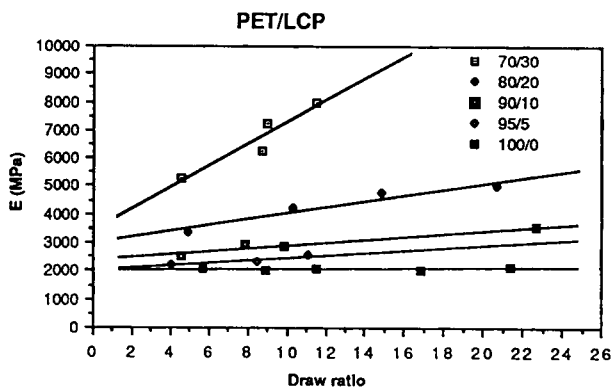
The blends of LCP and PP exhibited similar effects to those of LCP and PET, as can be seen in Figure 3. For the pure PP strand, the tensile strength and elastic modulus were about 24 and 1,284 MPa, respectively. In general, mechanical reinforcement was again achieved by increasing draw ratio and the content of LCP in the blend. At an LCP content of 30 wt %, the blend exhibited an elastic modulus of more than three times and tensile strength of about 2.5 times the value for pure PP. The addition of 20 wt % of LCP to PP doubled the modulus of PP and

increased the strength by about 50%. In these blends, the reinforcement took place at the cost of the strain at break, which decreased to about 2%. However, at smaller LCP contents some reinforcement was achieved without total loss of the ductility of PP. The blends with 5 wt % LCP elongated by more than 35%, and those with 10 wt % LCP exhibited a strain at break of about 6%.

Baird et al.¹⁵ blended three different LCPs with polypropylene. For Vectra/PP blends, they used a special mixing technique. The blend components were plasticized separately in two extruders, and the blending took place in a special mixing head before the die. The blends were extruded to flat films or blow molded to bottles. They reported a significant increase in torsional moduli of the drawn blends and formation of thin LCP fibers in PP matrix. Besides, the bottles exhibited some increase in tensile prop-



a)



b)

Figure 2 (a) Tensile strength and (b) elastic modulus of the extruded PET/LCP blends vs. draw ratio at different blend compositions.

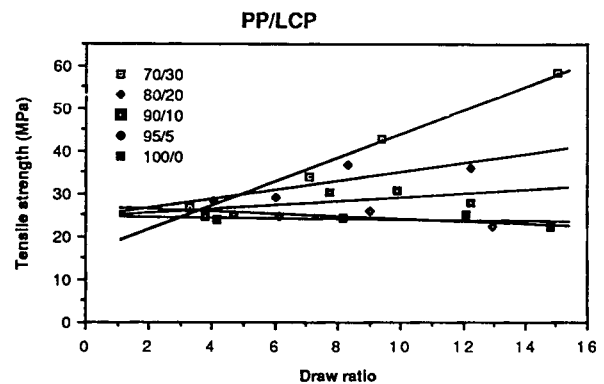
erties both in the axial and transverse directions. Our results are in qualitative agreement with those for the extruded flat films, although our blend components were mixed together directly in one extruder. The polypropylene grade was chosen so that the viscosity ratio for the blend is near unity at shear rates used in the extrusion.

Figure 4 shows that the blends of LCP with PPS do not behave so regularly as those with PET and PP. At some draw ratios, the 80/20 PPS/LCP blend was mechanically stronger than the 70/30 blend, which exhibited lower values of strength and modulus than predicted. Nevertheless, the tensile strength of PPS was doubled in the 70/30 blend and the elastic modulus was increased more than 2.6-fold. Thus, we can conclude that 20–30 wt % LCP in PPS acts as a considerable reinforcement for the matrix. At a small draw ratio ($S_0/S_s = 4$), the pure PPS strand elongated by more than 35%,

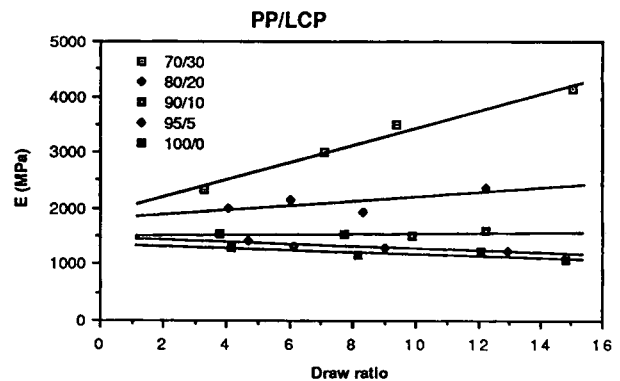
but at higher draw ratios, for PPS and for all PPS/LCP blend compositions, the strain at break was about 2–3%.

Morphology

The morphology of the extruded blends was characterized by SEM studies on the surface of the fractured strand. The studies revealed a close relation between the blend structure and its mechanical properties. As noted above, good reinforcement is achieved when LCP phases are deformed into fibers that orient in the flow direction. The degree of fiber formation depends on the amount of LCP in the blend and also on the elongational forces affecting the molten material. At small LCP contents, the LCP phases can form only a few small fibers and no significant reinforcement is achieved, while at larger LCP contents there are enough oriented LCP fibers in the matrix to carry a significant amount of the



a)



b)

Figure 3 (a) Tensile strength and (b) elastic modulus of the extruded PP/LCP blends vs. draw ratio at different blend compositions.

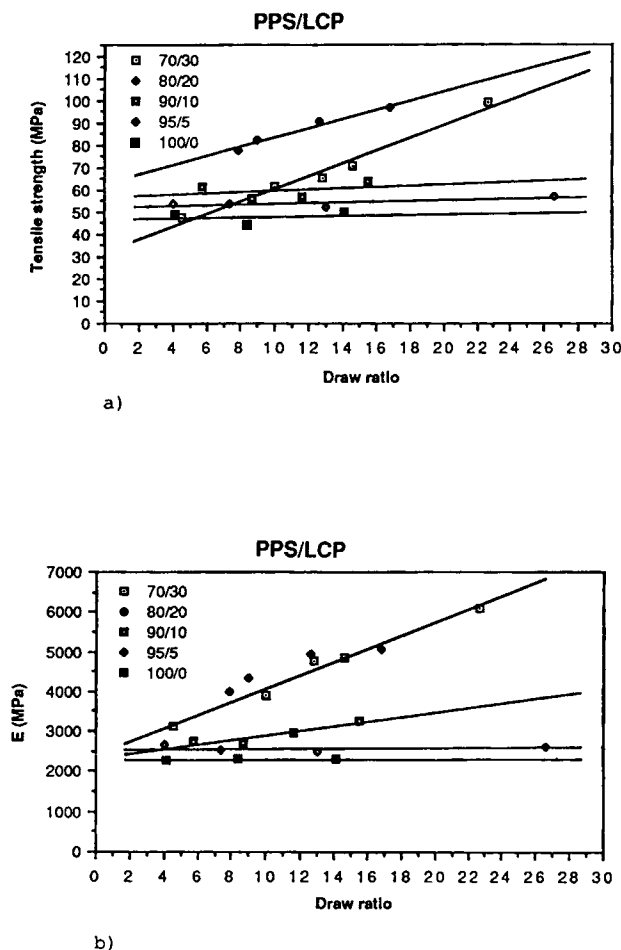


Figure 4 (a) Tensile strength and (b) elastic modulus of the extruded PPS/LCP blends vs. draw ratio at different blend compositions.

load and reinforce the matrix. In addition, the mutual compatibility and interfacial adhesion between the two polymers are of great importance in the reinforcing effect. In particular, the ratio of the melt viscosities is important for good dispersion and fiber formation.

The blends of LCP and thermoplastics are two-phase systems and they exhibit the skin/core morphology characteristic of pure LCPs. In the skin region, most of the fibrous LCP is oriented, while in the core the LCP domains are less oriented or in spherical form.

A clear skin/core structure was seen in all blends studied. In general, most of the LCP domains existed as fibers near or in the skin layer. At smaller LCP contents, there were fewer fibers, at greater contents more of them, but in every case fibers were found in the skin layer. In our earlier studies on injection-molded blends,⁹ no fibers were found at 5 wt % LCP.

A possible explanation for the divergent results is the different processing method: LCP domains are more easily oriented in extrusion, where more effective elongational forces exist to deform them in the longitudinal direction. Even small particles are fibrillated. The injection-molded bars studied earlier were also much thicker than the present extruded strands, and it is known that in molded LCP specimens the width of the unoriented core relative to the oriented skin increases with the thickness of the specimen.¹⁶ At higher LCP contents, about 20–30 wt %, fibers exist throughout the extruded strand and the difference between the layers becomes blurred. Most fibers nevertheless continue to be found in the skin layer.

The skin/core morphology of PET/LCP blends can be seen in Figure 5. The thickness of the skin layer was generally about 100–200 μm . Although the lengths of the LCP fibrils could not be measured accurately at the transversally fractured surface, they ranged from about 5–10 μm at lower LCP contents (5 and 10 wt %) to at least tens of μm 's at higher LCP contents. The thickness of the LCP fibers and spheres was mostly 1–3 μm in the PET/LCP blends. The amount of fibrillar LCP domains increased with increasing LCP content and increasing draw ratio in parallel with the improvement in mechanical properties. The effect of the draw ratio is suggested in Figure 5, where the spherical particles in the core region at lower draw ratio are seen to be slightly deformed (upward from the paper level) at higher draw ratio. At 20 wt % LCP, fibers also existed in the core region, and at 30 wt % there were abundant fibers throughout the strand [Figs. 6(c) and (d)]; most of them continued to be well oriented in the skin region, however. At the highest LCP content, the skin layer was thicker (about 400 μm) than in other blend compositions.

Ko et al.¹³ have studied extruded cast films of blends of PET and a HBA/HNA-based LCP. In the blends containing 20–60 wt % LCP, the dispersed phase appeared as spheres or ellipsoids about 5–25 μm in diameter. Fibril-like LCP phases obtained with conventional extrusion only at an LCP content of 80 wt % were achieved at lower LCP contents by increasing the screw and gear pump speeds. This influenced the melt mixing and extensional flow behavior and resulted in better mechanical properties than with conventional extrusion. In our studies, good dispersion was obtained by melt blending in a twin-screw extruder, and fibril-like morphology was achieved even at low LCP contents.

The PP/LCP blends exhibited generally similar structure as the PET/LCP blends, but there was

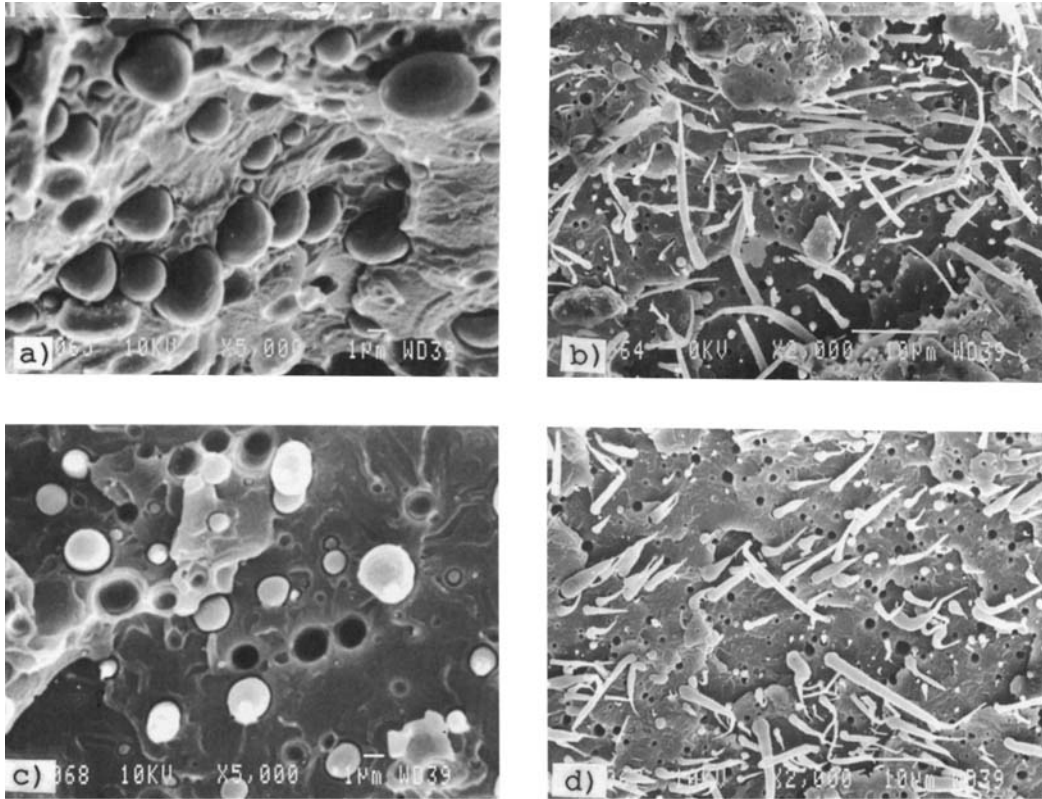


Figure 5 SEM studies of core (left, $\times 5,000$) and skin regions (right, $\times 2,000$) of the fractured surfaces of extruded PET/10 wt % LCP at draw ratios of 4 (above) and 20 (below).

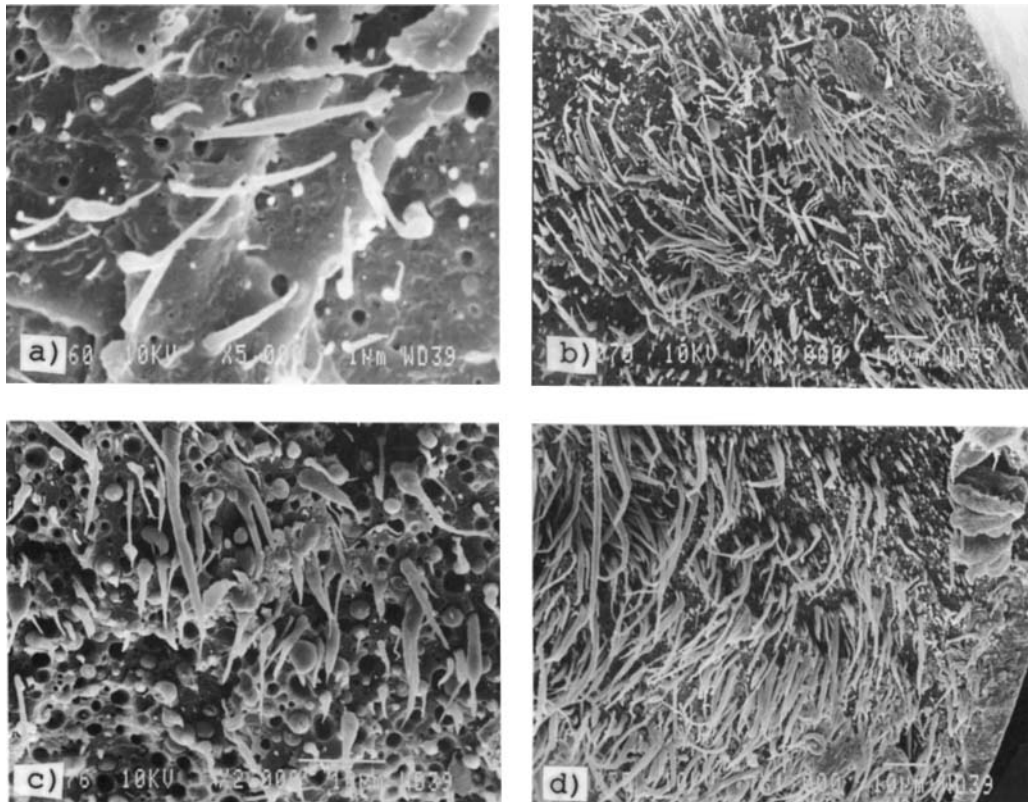


Figure 6 SEM studies of the skin region of the fractured surfaces of PET/LCP blends containing (a) 5 ($\times 5,000$), (b) 20 ($\times 1,000$), and (d) 30 ($\times 1,000$) wt % LCP, and (c) of the core region of the 70/30 blend ($\times 2,000$).

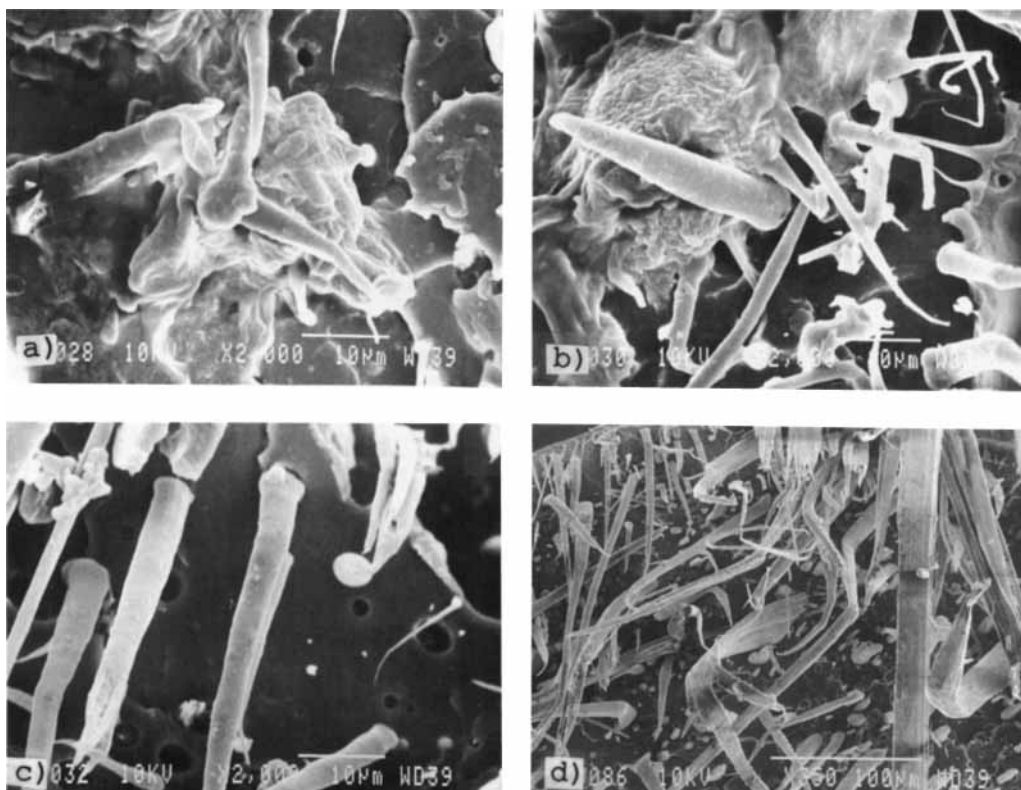


Figure 7 SEM studies of the skin region of the fractured surfaces of PP/LCP blends containing (a) 10, (b) 20, and (c) 30 wt % LCP ($\times 2,000$), and (d) the surface layer of the 70/30 blend ($\times 350$).

considerable difference in the size of the fibers, as can be seen in Figure 7. LCP formed longer fibrils in PP than in other matrices studied, ranging from tens of μm s at the lowest LCP contents to hundreds of μm 's at the highest. The longest LCP fibers were found in the surface layer [Fig. 7(d)]. In blends containing 20 and 30 wt % LCP, also thicker (up to 10–20 μm) and flat LCP domains existed. The formation of large LCP domains in PP is at least partly explained by the rheological findings discussed below.

PPS/LCP blends exhibited a porous microstructure different from that of the other blends. SEM micrographs of the skin regions are shown in Figure 8. The porosity of the material and the consequent poor mechanical properties are evidently due to a lack of interfacial adhesion between the blend components. The more porous structure of the 70/30 blend than of the 80/20 blend explains its unexpectedly poor strength. There were also fewer oriented fibrous LCP domains in the 70/30 blend than in the 80/20 blend. In spite of the porous structure, fibers were found in the skin region at all blend compositions and some reinforcement was achieved.

In extruded films of LCP (Vectra)/PPS blends, Ramanathan et al.⁴ found a foam structure without any fiber formation of the LCP phases, and they suggested that additives in the Vectra may have caused a reaction between the blend components leading to the porous structure and poor properties. By contrast, HBA/PET-type LCPs blended with PPS exhibited a fibrillar morphology and physical properties were enhanced.

All blends studied were two-phase systems and in SEM studies no miscibility was found between the blend components. Fracturing of the extruded strands did not break the LCP domains, but caused a fiber pull-out from the matrix, indicative of relatively poor interfacial adhesion between the blend components. The interfacial adhesion seemed to be better in the blends with PET and PP than in those with PPS.

Rheology

The viscosity ratio of the blend components is important for the formation of a fibrous blend structure. According to many researchers,^{17–19} maximum

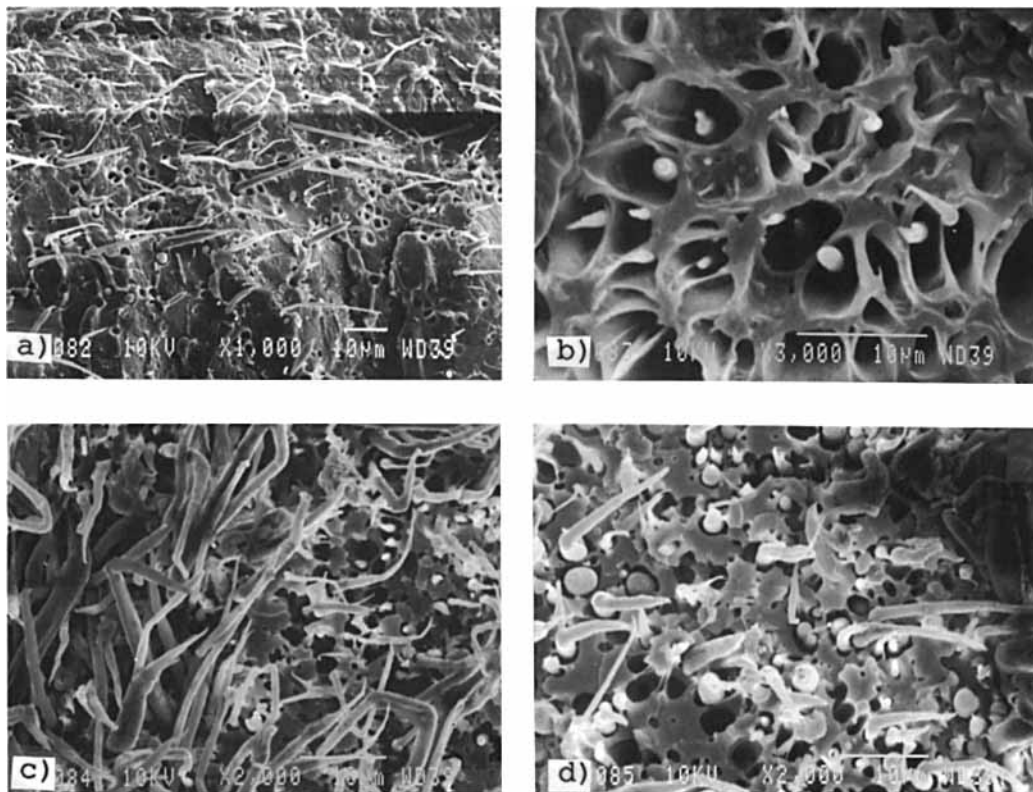


Figure 8 SEM studies of the skin region of the fractured surfaces of PPS/LCP blends containing (a) 5 ($\times 1,000$), (b) 10 ($\times 3,000$), (c) 20, and (d) 30 wt % LCP ($\times 2,000$).

fibrillation occurs when the viscosity ratio of the original components is close to unity. Formation of long continuous fibers is usually found in the blends of amorphous and semicrystalline polymers, when the viscosity ratio of the dispersed phase to the matrix is small. When the viscosity ratio is near unity, the dispersed phase bursts into smaller fibrils or droplets and the droplet size reaches its minimum.²⁰ With proper stretching, the small droplets can then be deformed into fine fibrils, provided that the coalescence of droplets is not prevented. According to hydrodynamic stability theory, without coalescence of the droplets in the matrix the surface energy of the smaller particles is too large for deformation to occur.²¹ Hence, the formation of fibrillar morphology also depends on the critical shear stress, which promotes coalescence of the dispersed phase.²²

The melt viscosities of LCP and the three matrices studied are presented as a function of shear rate in Figure 9. The measuring temperature was the same as the blending temperature, 310°C for PPS and 290°C for PP and PET. Thus, the viscosity of LCP can be compared with the viscosities of the matrices in conditions approximating those of processing. In extrusion, the shear rate varies in the

range 100–1,000 1/s and in injection molding from 1,000 to 10,000 1/s.^{23,24}

At small shear rates (100–200 1/s), at 290°C, the melt viscosities of LCP, PET, and PP were closely similar. At higher shear rates, LCP became clearly less viscous than PET and the ratio of their viscosities varied between 0.5 and 1. This led to good dispersion of LCP in PET and good fiber formation at all LCP contents.

The shear thinning behavior of PP changed more abruptly than that of LCP; thus, PP was the less viscous phase at higher shear rates. Perhaps this is the reason for the formation of larger LCP fibers and spheres in PP than in other matrices. As the more viscous component, LCP can not disperse into smaller domains and thin fibrils are not formed. The viscosity ratio of LCP to PP varied from about 1 to 3 in the range studied. Nevertheless, it was close to unity over a fairly broad range of shear rate.

The viscosity ratio of LCP to PPS at 310°C varied from about 0.2 at higher shear rates to 0.4 at lower shear rates. However, throughout the range LCP was the more easily flowing component, which enabled good dispersion of LCP in PPS. Good formation of short fibers was also seen in the SEM

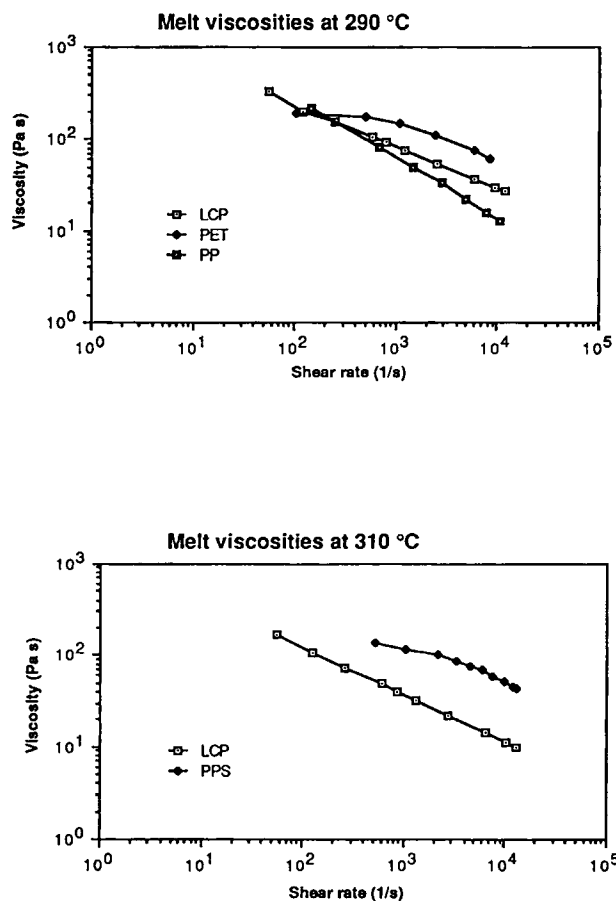


Figure 9 Melt viscosities as a function of shear rate for LCP and the pure matrix polymers at their blending temperatures.

studies (Fig. 8) in spite of the porous structure of the blends.

According to the rheological studies, Vectra A 950 seemed to be well suited for blending with the three thermoplastics of the study. Similar blends were prepared with polyamides (PA 66 and PA 6) that were less viscous than Vectra A 950. The dispersed LCP phase appeared as small spheres in all compositions, no fibrils were found, and the reinforcement was minimal. The poor result was assumed to be due to the high viscosity ratio of LCP to PA.

CONCLUSIONS

A polyester-type totally aromatic thermotropic main-chain LCP was melt blended with PET, PP, and PPS and the blends were extruded to strands of different draw ratio.

LCP was found to act as a significant reinforcement for all matrices studied. Tensile strength and elastic modulus of the blends increased with increasing LCP content and draw ratio, while strain at break decreased. The improvement in strength and stiffness was most significant at higher LCP contents (20–30 wt %), and at these compositions also the effect of draw ratio was greater. The relative improvements in tensile properties were greater than obtained earlier for injection-molded blends of the same polymers.⁹ At a draw ratio of only 11, the elastic modulus of PET/30 wt % LCP reached the level of 8,000 MPa, about four times the value of pure PET, and the corresponding tensile strength (120 MPa) was about three times that of PET.

The mechanical properties achieved were closely related to the blend morphology. All blends consisted of two discrete phases and exhibited a clear skin/core structure with the thickness of the skin layer ranging from about 100 to 400 μm . Fibrils were formed in the skin region in all blend types and compositions in contrast to our studies on injection-molded blends. The size of the LCP domains increased with increasing LCP content and the formation of LCP fibrils was enhanced with LCP content and the draw ratio. At 20–30 wt % LCP, fibril-like LCP phases also existed in the core of the strand. In PP, the size of the dispersed LCP phases was much greater than in the other matrices at all compositions.

The melt viscosities of LCP and the pure matrices were quite similar in the shear rate range used in processing, which enabled good dispersion of LCP in the matrices and formation of fibrils. However, at some shear rates PP was slightly less viscous than LCP, which could explain the formation of large LCP domains in PP.

SEM studies did not reveal any significant miscibility between LCP and the matrix polymers. The exceptionally poor interfacial adhesion in PPS/LCP blends was evidently the cause of the porous blend structure.

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