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In situ compatibilized polypropylene/liquid crystalline polymer blends

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Ethylene–glycidyl methacrylate copolymer (EGMA) has been investigated as a reactive compatibilizer for the immiscible and incompatible blends of polypropylene (PP) and a liquid crystalline polymer (LCP). The epoxy functional groups of the EGMA copolymer can react with the carboxylic acid and/or hydroxyl end-groups of the liquid comptalline copolyester. The *in situ*-formed EGMA-*g*-LCP copolymer tends to reside along the interface of the PP–LCP and reduces the interfacial tension during melt processing. The compatibilized PP/LCP blends show finer disperse LCP domains and tend to shift the LCP fibrous structure near the skin region of the uncompatibilized blends into the droplet domains. The PP crystallinity in the compatibilized PP/LCP blends is lower than that of the corresponding uncompatibilized blends due to interference in the PP crystallization by the *in situ*-formed EGMA-*g*-LCP copolymers. The lower PP crystallinity of the compatibilized blends is reflected in a substantial reduction in stiffness (in terms of the tensile modulus). However, the improved adhesion of the compatibilized blends results in an improvement in the toughness (in terms of the tensile elongation and impact strength). Copyright © 1996 Elsevier Science Ltd.

(Keywords: polypropylene; liquid crystalline polymer; compatibilized blends)

INTRODUCTION

Since the pioneering work on polymer blends containing a liquid crystalline polymer (LCP) was reported during the early 1980s by a number of research groups[1–4], this subject has received considerable attention recently and has been reviewed in two recent publications[5,6]. Among the numerous LCP/thermoplastic (TP) blends, the system containing an LCP and polypropylene (PP) is one that has lately attracted particular interest[7–26]. Baird and coworkers[7] were probably credited for being the first to report this LCP/PP blend system in 1988. Yongcheng *et al.*[10] blended PP with a new class of LCPs containing flexible spacers and which possessed relatively lower melt temperatures and reported reductions in the shear viscosity and improved mechanical properties. Seppala and coworkers[11,12] reported that increases in the LCP content in the PP/LCP blends and in the draw ratio resulted in higher tensile strengths and moduli but lower values for the tensile elongation and impact strength. Baird and coworkers[13,22,23] applied a compatibilizer, a maleic-anhydride-grafted PP (MAGPP), in blends of PP with a copoly(ester–amide) LCP (Vectra B 950) and a copolyester LCP (Vectra A900), and observed significant improvements in the tensile strength and modulus but a reduced tensile elongation and toughness. Since the reaction between the maleic anhydride and the polyester hydroxyl or carboxylic end-groups has not yet been established, an interaction such as hydrogen bonding between the MAGPP and the LCP is probably the mechanism leading to the improved compatibilization of these MAGPP-compatibilized PP/LCP blends[23]. Miller *et al.*[24] used a polypropylene functionalized acrylic acid (PP–AA) as a compatibilizer for PP/LCP polyblend fibres and observed that the compatibilized fibres have improved interfacial adhesion, and higher fibre crystallinity, orientation, and mechanical properties. Based on the observed compatibilization effect, there appears to be an interaction between the polar acrylic acid groups and the LCP rather than a true covalent bond. Heino and coworkers[26] later used ethylene–ethyl acrylate–glycidyl methacrylate (E–EA–GMA) terpolymer as a reactive compatibilizer for PP/LCP blends, resulting in finer LCP domains and an improved unnotched impact strength. The reaction between an epoxy and the carboxylic acid or hydroxyl end-groups of polyester is well established and extensively reported, and has been recently summarized in a review from this laboratory[27]. Various types of compatibilizers, reactive or non-reactive, have been used to compatibilize many immisicble and incompatible thermoplastic polymer blends, with the results being summarized in a number of recent reviews[27–30]. However, such an approach has rarely been extended to TP/LCP blends. Until recently, only a limited number of articles in the area of compatibilized TP/LCP blends have been reported[13,17,22–24,26,31–35]. We have previously reported that the styrene–glycidyl methacrylate (SGMA) functions as an effective reactive compatibilizer for the blends of Noryl/LCP[31] and polystyrene (PS)/LCP[32]. The epoxy functional groups in the SGMA can react with the carboxylic acid or hydroxyl end-groups of the copolyester LCP to form various SGMA-*g*-LCP copolymers *in situ* at the interface during melt

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processing, with these copolymers functioning as compatibilizers.

Transesterification reactions can increase the compatibility between a liquid crystalline copolyester and an isotropic polyester[34]. The ester-exchanged products of polycarbonate (PC) and an LCP were employed to compatibilize PC/LCP blends[35].

Since the reaction between an epoxy and a carboxyl group is well established we decided to choose a commercially available epoxy-containing copolymer, i.e. ethylene–glycidyl methacrylate (EGMA), as a reactive compatibilizer for the PP/LCP blends. The polyethylene segment of the EGMA copolymer is partially miscible with the PP, while the epoxy functional group can react with the LCP carboxyl end-group to form the EGMA-*g*-LCP copolymer at the interface. The *in situ*-formed EGMA-*g*-LCP copolymer is thus expected to act as a compatiblizer for the PP/LCP blends.

EXPERIMENTAL

The PP used in the study was PP-366, with a melt flow index of 3, supplied by the Taiwan Polypropylene Company. The LCP copolyester, Vectra A900, was obtained from Hoechst Celanese Corporation. EGMA, containing 12% glycidyl methacrylate (GMA), Igtabond E-grade, was purchased from Sumitomo Chemical Company of Japan. The catalyst, ethyl triphenylphosphonium bromide, was purchased from Merck.

To verify the reaction between EGMA and LCP on the basis of viscosity increase, a sample (40 g) was tested at 280°C and 30 r.p.m. in a Brabender Plastic-Corder machine. Melt blending was carried out using a 30 mm co-rotating twin-screw extruder by maintaining the barrel temperatures between 265 and 290°C. The extruded pellets were dried and injection moulded (280–290°C) into standard 1/8 in ASTM specimens by using an Arberg 3 oz injection moulding machine.

Capillary rheological measurements of the blends and matrices were performed on a capillary rheometer ($l/d = 40$, orifice radius = 0.02 in, and orifice length = 0.8 in) from the Layeness Company (Galaxy X model).

Differential scanning calorimetry (d.s.c.) was carried out on a Seiko DSC 220 calorimeter. Samples of ca. 10 mg were examined over the temperature range 25–300°C at a heating rate of 15°C min^{-1}. The crystallinity of the PP component in the blends was calculated by using the following equation:

$$X_c(\%) = (\Delta H_s/\Delta H_{PP}) \times (100/x) \qquad (1)$$

where X_c is the crystallinity of the blend, ΔH_s is the measured heat of fusion of the blend, $\Delta H_{PP}(= 209\ J\ g^{-1})$ is the heat of fusion of 100% crystalline isotactic polypropylene, and x is the mass fraction of polypropylene in the blend.

The LCP phase of the blends was observed on a hot-stage microscope (Model Eyesight, Zeiss) by heating the sample, pressed between two glass slides, to 200°C, thus forming a thin film. At this temperature, the PP phase is a transparent melt, while the unchanged LCP phase can be easily inspected with transmitted light. This technique appears to be advantageous for describing the changes in the size and shape of the dispersed LCP phase. The morphologies of the cryogenically fractured surfaces of the injection moulded specimens, the skin and core regions on planes parallel and perpendicular to the injection flow direction, were examined by a scanning electronmicroscope (Model S-570, Hitachi).

The procedures used for testing the mechanical properties, such as unnotched Izod impact and tensile tests, have been reported previously[31].

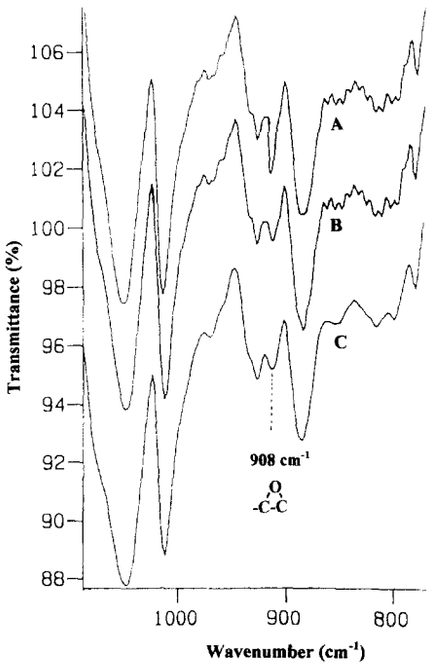


Figure 1 I.r. spectra of the uncompatibilized and compatibilized blends: (A) PP/LCP/EGMA (85/15/2.5) dry-blended; (B) PP/LCP/EGMA (85/15/2.5), melt-blended; (C) PP/LCP/EGMA/catalyst (85/15/2.5/0.02), melt-blended

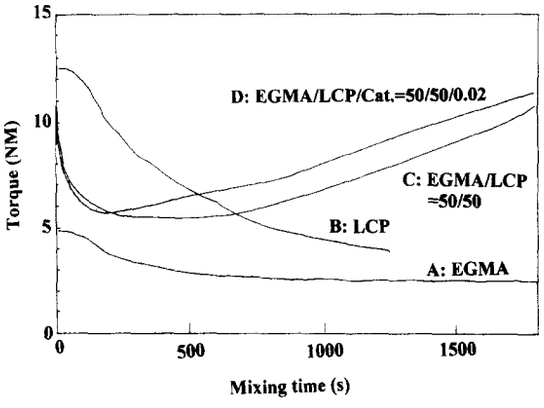


Figure 2 Plots of torque vs. time for the components and mixtures at 280°C

RESULTS AND DISCUSSION

The infra-red (i.r.) peak at 908 cm^{-1}, which is assigned to the epoxy ring has been used to monitor qualitatively the reactions between EGMA and the end-groups of the LCP. *Figure 1* shows the i.r., spectra of the compatibilized PP/LCP/EGMA (85/15/2.5) blends before and after melt blending. The sizes of the epoxy peaks of the melt-blended mixtures (curves B and C) are significantly

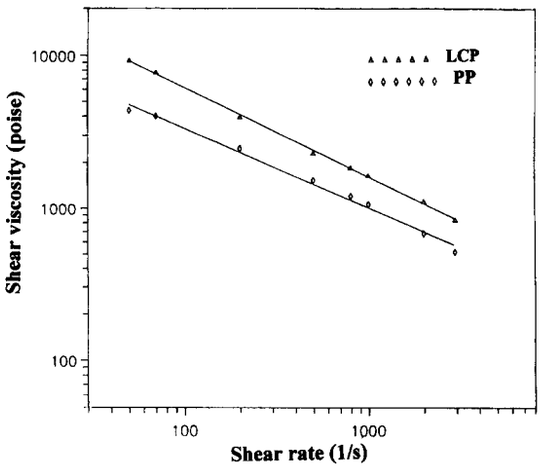


Figure 3 Plots of apparent viscosity vs. shear rate for PP and LCP at 280°C

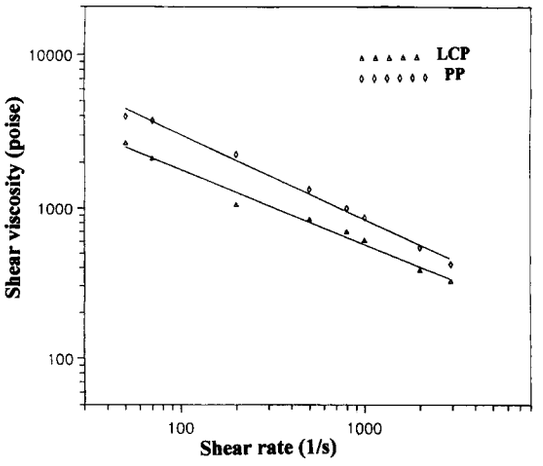


Figure 5 Plots of apparent viscosity vs. shear rate for PP and LCP at 290°C

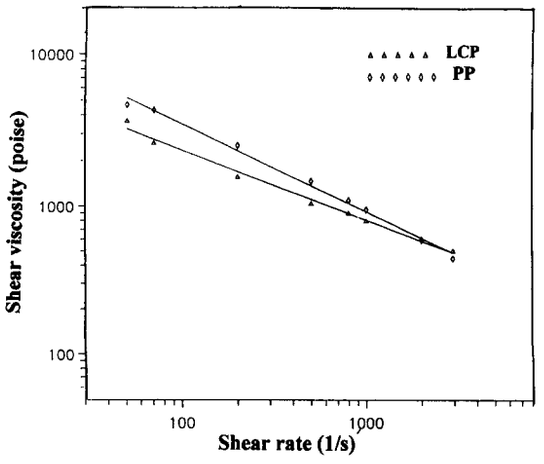


Figure 4 Plots of apparent viscosity vs. shear rate for PP and LCP at 285°C

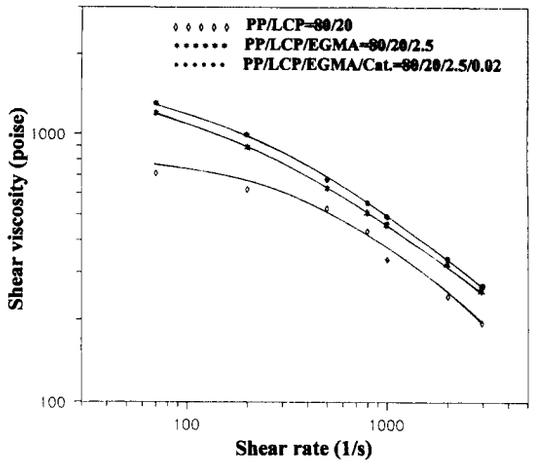


Figure 6 Plots of apparent viscosity vs. shear rate for the uncompatibilized and compatibilized PP/LCP (80/20) blends

smaller than that of the dry-blended mixture (curve A), which is an indication of the reaction of the epoxy groups. However, the i.r. spectra do not allow us to identify the products from the reactions between the epoxy and the –COOH and –OH end-groups. The epoxy group in EGMA prefers to react with the –COOH rather than with the aliphatic –OH group, due to the difference in acidity. Another competitive reaction, namely epoxy hydrolysis, is also expected to occur to a certain extent because epoxy-containing compounds or polymers are known to act as acid or water scavengers in many condensation-type polymers in the melt. Therefore, drying of the feeds is important for minimizing epoxy consumption through hydrolysis.

Figure 2 illustrates the comparative torque vs. time curves for LCP, EGMA, and LC/EGMA (1/1) mixtures (with and without 200 ppm catalyst) at 280°C. The torque of the pure LCP shows a continuous decrease with time, indicating thermal degradation. The EGMA curve also decreases continuously with time, but the rate of torque decline is significantly lower than that of the LCP. The result indicates that the potential self-curing reaction of EGMA does not occur. The torques of the mixtures (curves C and D) increase progressively with time, which provides indirect evidence of an increase in the molar mass due to the anticipated graft reactions between EGMA and LCP. The presence of 200 ppm catalyst in the mixture further increases the rate of torque increase. This result indicates that certain epoxy-containing compounds (such as low-molecular-weight epoxy resin) can serve as coupling agents for restoring the LCP molar mass loss due to thermal degradation during the melt process. Similar results were also observed for a mixture of polystyrene and poly(phenylene oxide) and SGMA[31].

Figures 3–5 illustrate the shear viscosity vs. shear rate plots of LCP and PP at three different temperatures, i.e. 280, 285, and 290°C. Both matrices exhibit a non-Newtonian flow behaviour, where the viscosities shear thin continuously at approximately the same rate for the shear rates being investigated here. At 280°C, the viscosity of the LCP is higher than that of the PP, with this order being reversed for temperatures above 290°C. A viscosity ratio of dispersed phase vs. matrix (η_{LCP}/η_{PP}) of less than one is critical for forming the LCP fibrillar structure[19]. *Figure 6* gives the viscosity vs. shear rate plots of the uncompatibilized and compatibilized PP/LCP (80/20) blends. The compatibilized blend has a higher viscosity when compared to its corresponding

Table 1 Values obtained from d.s.c. for the melting temperatures, heats of melting, and crystallinities of the uncompatibilized and compatibilized PP/LCP blends

Composition[a]	T_m (°C)	ΔH (J g^{-1})	Crystallinity (%)
PP	166.4	86.6	41.1
PP/LCP (90/10)	166.0	99.7	53.0
PP/LCP/EGMA (90/10/2.5)	166.0	85.9	46.8
PP/LCP/EGMA/Cat (90/10/2.5/0.01)	166.0	81.3	44.3
PP/LCP/EGMA/Cat (90/10/2.5/0.02)	166.0	79.7	43.4
PP/LCP/EGMA/Cat (90/10/0.625)	166.1	96.0	51.4
PP/LCP/EGMA/Cat (90/10/0.625/0.02)	166.2	91.4	48.9
PP/LCP (95/5)	166.1	96.1	48.4
PP/LCP/EGMA (95/5/2.5)	166.0	90.1	46.5
PP/LCP/EGMA/Cat (95/5/2.5/0.02)	166.4	83.7	43.2
PP/LCP (97.5/2.5)	166.1	95.5	46.9
PP/LCP/EGMA (97.5/2.5/2.5)	166.2	91.8	46.2
PP/LCP/EGMA/Cat (97.5/2.5/2.5/0.02)	166.0	87.5	44.0

[a] Cat = catalyst, ethyl triphenyl phosphonium bromide

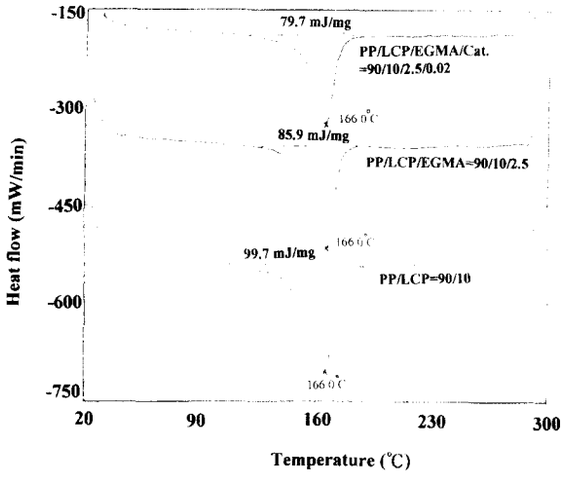
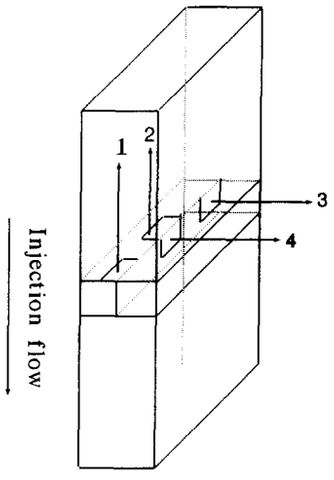


Figure 7 D.s.c. thermograms of the uncompatibilized and compatibilized PP/LCP(90/10) blends



Injection molded specimen

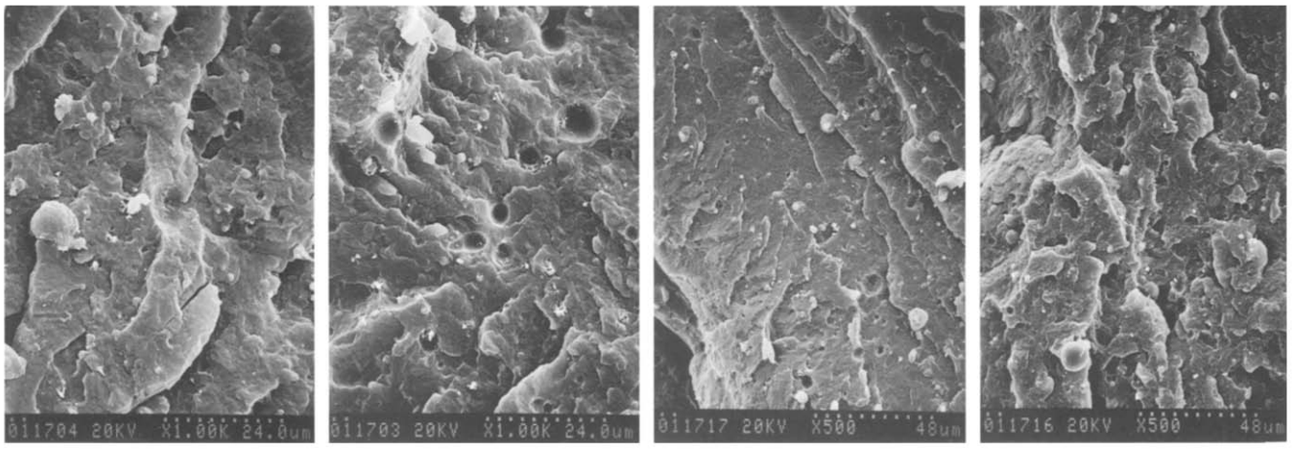
Figure 8 Schematic diagram showing the locations and flow direction of the injection moulded specimen used for scanning electron microscopy examination

uncompatibilized counterpart as a result of the grafting reaction. The compatibilized blend containing 200 ppm catalyst has an even higher viscosity, as would be expected from a higher grafted product.

Extruded pellets were employed in the d.s.c. analysis. *Table 1* summarizes the data from d.s.c. in terms of the melting temperature, heat of melting, and crystallinity of the pure PP and the PP component in the PP/LCP blends. *Figure 7* shows d.s.c. thermograms of the uncompatibilized and compatibilized PP/LCP (90/10) blends, where the melting points are essentially identical (at ca. 166°C), while the shapes and heats of fusion vary. The observed melting pionts (T_ms) for the PP component in the blends, compatibilized or uncompatibilized, are essentially identical to that of the pure PP. The PP crystallinities of the uncompatibilized PP/LCP blends are substantially higher than that of the pure PP, which is consistent with most of the previously reported results[23]. It is quite surprising to discover that the heats of melting and the crystallinities of the compatibilized blends are decreased significantly compared to the corresponding uncompatibilized blends. The presence of catalyst in the compatibilized blend further decreases its crystallinity. The formation and presence of the EGMA-*g*-LCP is expected to interfere with the PP crystallization, especially in the vicinity of the interface region. A better compatibilized blend results in smaller LCP domains, and the total interfacial area is also expected to be higher. An effective compatibilizer could increase the mutual solubilities of the various components, which could also cause a reduction in the PP component crystallinity in the compatibilized blend[27]. Similar results were also obtained from a corresponding blend between PP and poly(butylene terephthalate) (PBT), compatibilized by EGMA[35]. However, this observed result is in contrast to the PP–AA compatibilized PP/LCP blend system where the crystallinity of the PP in the compatibilized blend is higher than that in the corresponding uncompatibilized blend[24]. A lower PP crystallinity of the matrix in the blend is expected to reduce the stiffness of the blend, as will be discussed below.

A: Location 1 B: Location 2 C: Location 3 D: Location 4

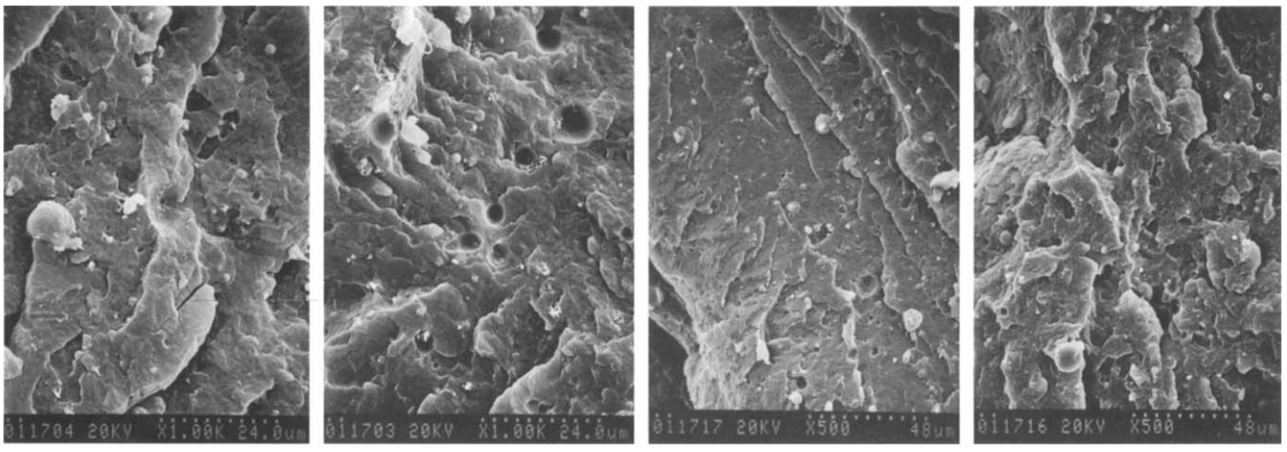
Figure 9 Scanning electron micrographs of the PP/LCP (85/15) blend: (A) location 1, perpendicular to the flow direction, near skin region; (B) location 2, perpendicular to the flow direction, at core region; (c) location 3, parallel to the flow direction, near skin region; (D) location 4, parallel to the flow direction, at core region



A: Location 1 B: Location 2 C: Location 3 D: Location 4

Figure 10 Scanning electron micrographs of the PP/LCP/EGMA (85/15/2.5) blend taken at the locations shown in *Figure 9*: (A) 1; (B) 2; (C) 3; (D) 4

The fractured surface morphologies of the injection moulded specimens were inspected on the planes perpendicular and parallel to the injection flow directions in both the core and near-skin regions. *Figure 8* is a schematic diagram which shows the four 'locations' of the specimen being examined. *Figure 9* shows scanning electron micrographs of the uncompatibilized PP/LCP(85/15) blend with reference to the above mentioned four locations. *Figure 9A* (×1000) was taken from the plane perpendicular to the injection flow direction, at the near-skin region (location 1), where the LCP fibrils are fairly long (high aspect ratio), with most of them being pulled out from the PP matrix, an indication of poor interfacial adhesion[24]. *Figure 9B* (×1000, location 2) shows the morphology at the core region for the same specimen as *Figure 9A*, where the LCP phase exists as large spherical particles. *Figure 9C* (×500) shows the micrograph obtained on the plane parallel to the flow direction near the skin region (location 3), and displays the presence of a large number of long LCP fibrils. *Figure 9D* (×500, location 4) shows the similar droplet LCP morphology to that seen in *Figure 9B*. Such a skin–core dispersed phase morphology has been observed in many polymer blends as a result of the shear difference and quenching rate difference in a typical injection moulding process. *Figure 10* shows the morphologies of the compatibilized PP/LCP/EGMA (85/15/2.5) blend, viewed at the same locations. *Figure 10A* (×1000, location 1) shows the presence of only LCP circular domains of different sizes. After closer examination, however, the larger LCP domains are actually found to be the fractured LCP fibrils. The improved interfacial adhesion of this compatibilized blend fractured the LCP fibrils, instead of pulling them out of the matrix, and additional evidence to support this claim will be shown in *Figure 10C*. *Figure 10B* (×1000, location 2) clearly shows that the size of the LCP dispersed droplets is considerably smaller than that of the uncompatibilized blend (*Figure 9B*), evidence for better compatibilization, as would be expected. *Figure 10C* (×500, location 3) shows that this compatibilized blend can still form LCP fibrils near the skin region but in this case the fibrils are relatively shorter and the number of fibrils is considerably less than that of the corresponding uncompatibilized blend. At the core region (*Figure 10D*, ×500, location 4), the size of the LCP droplets becomes rather small, with the phase contrast becoming blurred, indicat-



A: Location 1 B: Location 2 C: Location 3 D: Location 4

Figure 11 Scanning electron micrographs of the PP/LCP/EGMA/catalyst (85/15/2.5/0.2) blend taken at the locations shown in *Figure 9*: (A) 1; (B) 2; (C) 3; (D) 4

ing an improved compatibility between the PP and the LCP. *Figure 11* shows the morphologies of the compatibilized blend which contains an additional 200 ppm of catalyst, where no LCP fibril formation can be detected for any of the four locations. When the LCP content is reduced to 10% in the PP/LCP (90/10) blends, compatibilized or uncompatibilized, only droplet morphologies were observed at all four locations (micrographs not shown here). This means that a minimum LCP content of between 10 and 15% is necessary to form the LCP fibrils near the skin region under our experimental conditions.

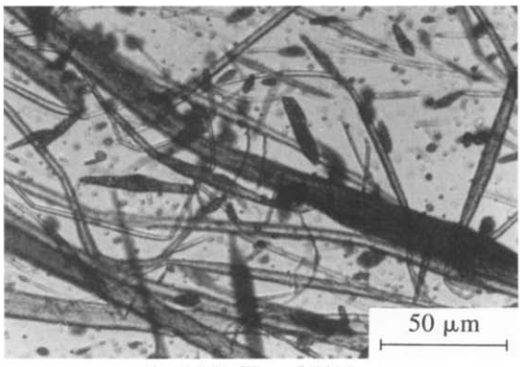
Figure 12 shows the hot-stage photomicrographs of the extruded strands from a few selected blends. For the uncompatibilized PP/LCP (85/18 blend) (*Figure 12A*), the LCP exists mainly as on fibrils with a small fraction being present as droplets. In contrast the LCP phase in the corresponding compatibilized blend (*Figure 12B*) exists mainly in a droplet form with only a few short fibrils being present. For the uncompatibilized blend with a lower LCP content, i.e. PP/LCP (90/10) (*Figure 12C*), the LCP exists essentially as droplets of different sizes. Qin *et al.*[14] employed essentially the same PP and LP in his study and observed clear LCP fibril formation when the LCP contents are 5 parts per hundred parts of rubber (phr) or above when using a single-screw extruder equipped with a single-hole spinneret in the die head. The higher uniaxial extensional flow achieved by the spinneret employed by Qin *et al.*[14] is able to form LCP fibrils at a relatively lower LCP content (5 phr) than that used in our study (15%).

The concentration of LCP within a PP matrix (or any other flexible thermoplastic) and the appropriate processing conditons have been shown to be critical factors in the creation of a fibrous morphology[14,23]. An increased dispersed phase size created by an increased rate of coalescence at high LCP concentrations is believed to be one factor responsible for the formation of the LCP fibres in the blend.

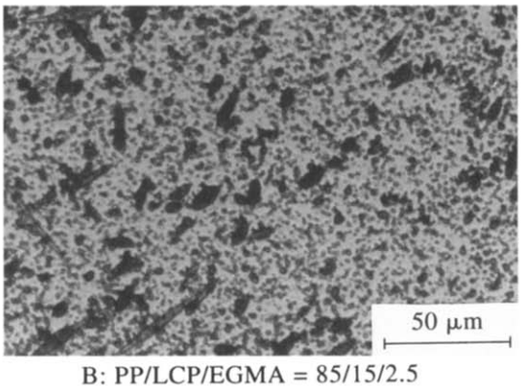
The addition of an effective phase compatibilizer to an incompatible flexible thermoplastic (TP) blend, will in general, result in a reduced interfacial tension in the melt, finer phase domains, increased interfacial adhesion and enhanced toughness. However, a similar approach, when applied to the TP/LCP blends, may not result in the expected toughness improvement. In order to create an ideal fibril-reinforcement of the TP/LCP blends, the formation of longer and finer LCP fibrils, with a higher aspect ratio, within the TP matrix and an increased interfacial adhesion are the two most important factors. Unfortunately, most of the compatibilized TP/LCP blends tend to hinder LCP fibril formation, due to a lower interfacial tension and finer dispersed phase domains, even though enhanced interfacial adhesion is obtained.

The observed mechanical properties are summarized in *Table 2*. *Figure 13* clearly demonstrates the trend of impact strength enhancement for the compatibilized blends over the uncompatibilized counterparts. The presence of 200 ppm of catalyst further increases the impact strength. Tensile elongation also shows the same trend as the impact strength (*Table 2*). The tensile strength results are not very consistent, with some values being higher and some lower, but the variation is not very substantial. *Figure 14* shows a significant decrease in the tensile modulus for the compatibilized blends. Similar results were also observed previously[26]. Three factors are believed to be responsible for the lower moduli obtained for the compatibilized blends in this PP/LCP blend system, namely the rubbery nature of the compatibilizer, droplet LCP morphology, and the reduced PP crystallinity. Earlier d.s.c. data showed that the PP crystallinity of the compatibilized blends is significantly lower than that of the uncompatibilized blends. A significantly reduced modulus was also obtained for reactive ethylene-based terpolymer compatibilized blends as a result of the decrease in the PP crystallinity[17]. The anticipated reactions between our reactive compatibilizer and the LCP produced the grafted copolymer which interferes with the PP crystallization.

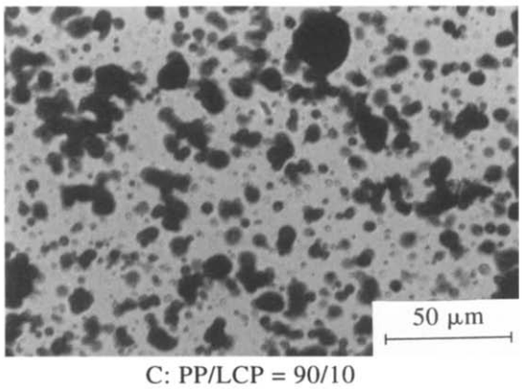
As mentioned earlier, in order for a compatibilizer to enhance the reinforcement of a thermoplastic by liquid crystalline polymers, the compatibilizer must not cause the loss of the fibrous morphology. This situation has rarely been achieved, except in a few cases[31,32]. Earlier morphological results clearly demonstrated that the use of a compatibilizer PP/LCP in the blends investigated in this present study tended to shift the fibrous LCP structure near the skin region into the droplet domains.



A: PP/LCP = 85/15



B: PP/LCP/EGMA = 85/15/2.5



C: PP/LCP = 90/10

Figure 12 Photomicrographs of various blends at 200°C: (A) PP/LCP (85/15); (B) PP/LCP/EGMA (85/15/2.5); (C) PP/LCP (90/10)

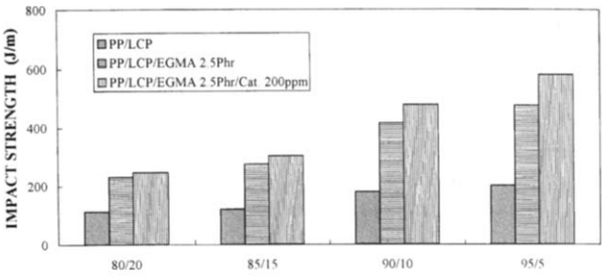


Figure 13 Effect of compatibilizer and catalyst on the unnotched impact strengths of the PP/LCP blends

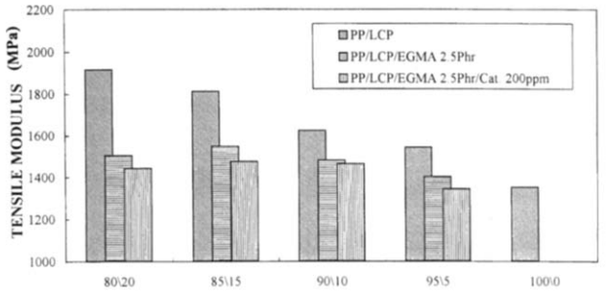


Figure 14 Effect of compatibilizer and catalyst on the tensile moduli of the PP/LCP blends

CONCLUSIONS

The EGMA copolymer has been shown to be an effective reactive compatibilizer for PP/LCP blends. The epoxy functional group of the EGMA is able to react with the –COOH and/or the –OH end-groups of the LCP copolyester to form a EGMA-*g*-LCP copolymer at the interface. The *in situ*-formed EGMA-*g*-LCP copolymer tends to reside along the PP–LCP interface, and reduces the interfacial tension during melt processing, thus resulting in finer dispersed LCP phase domains. The expected increase in adhesion of the compatibilized blends results in an improvement in the toughness (impact strength and tensile elongation). However, the increase in the compatibility of the PP/LCP blends tends

Table 2 Tensile properties and values obtained for the unnotched impact strength of the uncompatibilized and compatibilized PP/LCP blends

Composition[a]	Tensile strength (MPa)	Tensile modulus (MPa)	Tensile elongation[b] (%)	Unnotched impact strength (J m^{-1})
PP/LCP (95/5)	32.0	1543	n.b.	200
PP/LCP/EGMA (95/5/2.5)	24.8	1401	n.b.	475
PP/LCP/EGMA/Cat (95/5/2.5/0.02)	38.2	1342	n.b.	580
PP/LCB (90/10)	24.3	1623	23.6	179
PP/LCP/EGMA (90/10/2.5)	30.4	1480	n.b.	415
PP/LCP/EGMA/Cat (90/10/2.5/0/02)	28.9	1463	n.b.	477
PP/LCP (85/15)	26.4	1812	14.5	120
PP/LCP/EGMA (85/15/2.5)	25.1	1548	21.1	274
PP/LCP/EGMA/Cat (85/15/2.5/0.02)	26.7	1474	23.7	302
PP/LCP (80/20)	26.5	1915	9.3	112
PP/LCP/EGMA (80/20/2.5)	26.1	1504	15.5	230
PP/LCP/EGMA/Cat (80/20/2.5/0.02)	26.7	1443	15.8	246

[a] Cat = catalyst, ethyl triphenyl phosphonium bromide
[b] n.b. indicates no break observed in the tensile test after at least 60% elongation

to shift the fibrous LCP morphology of the uncompatibilized blends into the droplet domains in the compatibilized blends. The PP crystallinities of the compatibilized blends are lower than those of the corresponding uncompatibilized blends and this results in a significant decrease in the tensile modulus of the product. The presence of the ethyl triphenylphosphonium bromide catalyst promotes the grafting reaction and further improves the blend compatibilization.

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