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A Study of the Effect of Polystyrene Sulfonation on the Performance of Terephthaloyl Chloride-Dihydroxydiphenyl Sulfone Copolymer/Polystyrene System

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Thermal, morphological, and mechanical properties of composites of a liquid crystalline copolymer (LCP) poly(terephthaloyl chloride)-co-(p,p′-dihydroxydiphenyl sulfone) with polystyrene (PS) and sulfonated polystyrene (SPS) are presented and discussed. Sulfonation of polystyrene was expected to improve the interfacial adhesion by introducing hydrogen bonding in the LCP/PS system. The degree of sulfonation was 11%. The incompatibility (lack of proper interfacial adhesion) of the LCP/PS system resulted in sharp decrease in the composite tensile strength with LCP addition. The performance of the system did not change when processed at a higher temperature (270 °C instead of 225 °C). While a composite plate of 25% LCP/PS could not be fabricated, it was possible for LCP/SPS (processed at 215 °C), indicating some improvement in interfacial bonding by sulfonation. Sulfonation of PS resulted in fracture with some degree of plastic deformation for pure SPS matrix and also the LCP/SPS system with the lowest LCP content (1 wt%), whereas plastic deformation was not observed for PS used as received. The strength of the LCP/SPS system also decreased with increase in LCP content, indicating that 11% sulfonation is not sufficient to introduce significant compatibility, but it was not as dramatic as that for LCP/PS. The performance of the LCP/SPS system was not affected significantly by heat treatment at the process temperature.

Keywords composite, mechanical properties, polystyrene, sulfonation

1. Introduction

Interest in liquid crystalline polymers (LCPs) has grown in the past decade due to their inherently high stiffness and strength, high use temperature, excellent chemical resistance, and low coefficient of expansion (Ref 1-4). These materials represent an outstanding scientific development and have strong potential for technological applications ranging from structural to electronics and nonlinear optics.

Although many macroscopic properties of LCPs are extremely advantageous, the cost associated with production of these materials is high. The blending/reinforcing of a conventional resin with an LCP can produce improvements in cost effectiveness and can yield novel chemical and physical results (Ref 3-7). Recently, several researchers have studied blends/composites of LCPs and thermoplastics. Commercial or experimental LCPs (mainly aromatic polyesters, polyamides, or their copolymers) have been blended with various thermoplastics (Ref 1, 3-30). However, the drawback is that most of these LCP/thermoplastic systems suffer poor mechanical properties due to poor interfacial adhesion between the LCP and the thermoplastic (Ref 31). It was the objective of this study to investigate the possibility of improvement of the interfacial adhesion by introducing hydrogen bonding in an LCP/PS system.

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2. Experiment

The LCP was synthesized by condensation polymerization from its two monomers, terephthaloyl chloride (TC) and dihydroxydiphenyl sulfone (DHDPS), in a 1 to 1 mol ratio, expecting that this synthesis would result in a rigid rodlike structure, imparting liquid crystallinity (Ref 32).

LCP synthesis was done with toluene as the solvent (Ref 33). DHDPS (0.05 mol) was weighed out and dissolved in 100 mL of 0.1M NaOH. It was then diluted with 300 mL of water. The solution was charged in a blender. Then 3 g of sodium dodecyl sulfate in 30 mL of water was added to the blender while it was stirred slowly. It acted as a dispersing agent in the reaction. On the other hand, 0.05 mol of TC was dissolved in 150 mL toluene, and the solution was also charged in the blender. The reaction mixture was then blended at high speed for 5 min at room temperature. The reaction mixture was then poured into excess of acetone so as to coagulate the product. After leaving the system overnight, the slurry was filtered off and washed with excess of water to remove occluded salts and detergent. This step was followed by repeatedly washing with methanol and acetone to remove unreacted monomers and toluene.

The thermoplastics used in this study are polystyrene (PS) supplied by the Saudi Basic Industries Corporation (SABIC) and modified polystyrene by sulfonation. General purpose polystyrene beads were converted to PS powder for blending with LCP during the composite fabrication process. For that purpose, 120 g of polystyrene beads were dissolved in excess of methylene chloride. The system was maintained at 40 °C, while stirring continued until all the polystyrene was dissolved. The viscous solution was then added slowly to excess of methanol. The PS that precipitated out in thin strings was left overnight to soak in methanol. The methanol was then drained, and the PS was dried in vacuum at 80 °C for 4 h.

Polystyrene was sulfonated according to the procedure of Makowski et al. (Ref 34) by attaching $-SO_3H$ group at the paraposition of the benzene ring in the repeat unit of polystyrene. The sulfonating group was attached because it was expected to improve the interfacial adhesion by forming hydrogen bonding with the LCP chain in the composites of polystyrene and LCP. It has been reported that about 8% sulfonation is achieved through this procedure (Ref 34). A low level of sulfonation is preferable, because sulfonation of PS at high levels causes a substantial rise in viscosity resulting in difficulties in processing even at high temperatures.

The sulfonating agent used was acetyl sulfate, which was prepared by adding 198 mL of 1,2-dichloroethane to 38 mL of acetic anhydride. The mixture was cooled to 10 °C, and 14 mL of concentrated sulfuric acid was added. On the other hand, 104 g PS was dissolved in 490 mL of 1,2-dichloroethane and heated to 50 °C. It was then mixed with 35 mL of acetyl sulfate prepared earlier. The resulting solution was stirred for 60 min at 50 °C. Reaction was terminated by the addition of 25 mL methanol. The sulfonated polystyrene (SPS) was isolated by steam stripping. This steam stripping technique was somewhat different than the conventional method. Pure nitrogen gas was purged through the solution containing SPS while the whole system was maintained at 80 °C. The nitrogen was purged in order to enhance the removal of organic solvents from SPS. The polymer mass was pulverized with water in a blender, filtered, washed, and dried in vacuum at 90 °C. It was then powdered in a milling machine.

Composite Fabrication. The possibility of solution blending the LCP and the thermoplastics (PS and SPS) was checked first. Various solvents were tried, such as chloroform, acetone, dimethyl formamide (DMF), tetrahydrofuran (THF), phenol, acetic acid, concentrated sulfuric acid, dimethyl sulfoxide (DMS), and 1,2-dichloroethane. None of these trials was satisfactory. Composites of LCP with the thermoplastics were then mixed (in proportion) in a micromilling machine and fabricated by compression molding at a temperature above the glass transition but below the degradation temperature of the thermoplastics. Sheets about 0.7 mm thick were formed from PS, SPS, and composites of 1, 5, 10, and 25% LCP with these thermoplastics.

Characterization. Elemental analysis was performed on LCP and SPS by a Carlo Erba elemental analyzer. Thermal analysis of the materials was done by utilizing a differential scanning calorimeter (Perkin Elmer DSC-4) under an inert atmosphere of argon. The heating rate was 20 °C/min, and the temperature range covered from 50 to 320 °C. The mechanical properties were obtained using an Instron 1196 (Instron Corporation, Canton, MA) mechanical testing system. The tensile tests were conducted at a displacement rate of 0.1 mm/min. The samples for the tensile tests were cut from compression molded plates (and milled) as per specifications given in ASTM standard D 638M-89 (Type M II) (Ref 35). The arithmetic average

Table 1 Experimental and calculated values of percents of elements present in LCP and SPS

	Carbon, %		Hydrogen, %		Sulfur, %	
Polymer	Experimental	Calculated	Experimental	Calculated	Experimental	Calculated
LCP	60.8	63.1	3.4	3.2	8.8	8.5
SPS	55.37	52.17	4.52	4.34	1.9	17.4(a)

(a) Based on 100% sulfonation

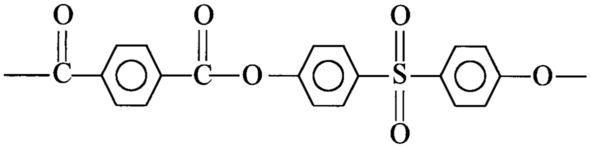

Fig. 1 Structural formula of a repeat unit of liquid crystalline copolymer

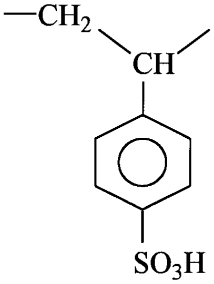

Fig. 2 Structural formula of a repeat unit of sulfonated polystyrene

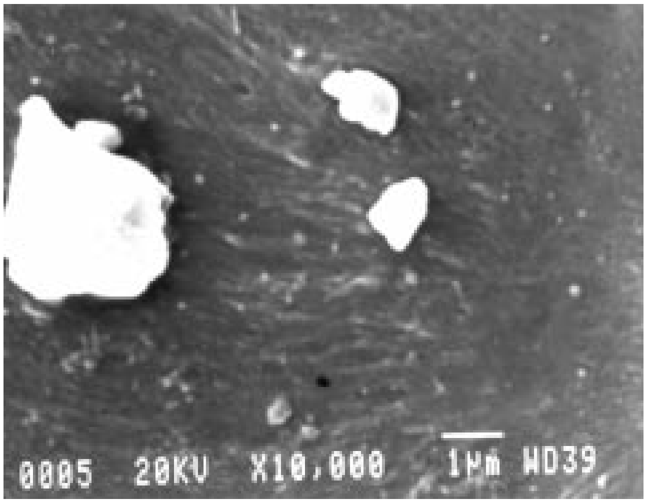

Fig. 3 SEM micrograph of a fracture surface of LCP/PS system showing the sharp interface boundary around an LCP particle. (Art has been reduced to 80% of its original size

and the standard deviation of the tensile properties were calculated using four samples. All mechanical tests were carried out at room temperature. To investigate the interfacial adhesion between the LCP and the thermoplastics, fractured surfaces at cryogenic temperature (in liquid nitrogen) and tensile fractured surfaces were observed by scanning electron microscopy (SEM) on a JSM-840 scanning microscope.

3. Results

3.1 *Structures of LCP and SPS*

As the experimental details state, the liquid crystalline polymer was formed from terephthaloyl chloride and dihydroxydiphenyl sulfone in a 1 to 1 mol ratio. The structural formula of a repeat unit of LCP is shown in Fig. 1. This structure confirmed liquid crystallinity (as dissolved in sulfuric acid) by exhibiting birefringence through crossed polarizers. Sulfonated polystyrene was formed from acetyl sulfate (sulfonating agent) and concentrated sulfuric acid. Structural formula of a repeat unit of SPS is shown in Fig. 2.

Calculated (from the structure based on 100% synthesis) and experimentally determined weight percents of elements present in LCP were not significantly different (Table 1). However, for the case of SPS, about 11% sulfonation was achieved. This value is higher than the 8% level of sulfonation reported by Makowski et al. (Ref 34), probably due to the lower temperature used in this study for the removal of organic solvents from the product solution containing SPS. This study purged nitrogen gas through the SPS solution at 70 °C, rather than use the conventional steam stripping at 100 °C. Higher temperature might result in the breakage of SPS product back into its reactants.

3.2 *Thermal Analysis of the Materials*

Table 2 lists the approximate glass transition and/or degradation temperatures for the polymers used in this study. These two temperatures were important in determining the processing temperature range for plate fabrication. No glass transition temperature, T_g, was detected for pure LCP, and it did not melt either, but it did start to degrade at approximately 320 °C, making it impossible to fabricate a pure LCP plate.

The evidence of the immiscibility of the thermoplastics (PS and SPS) and the LCP (i.e., no dissolution of LCP in the thermoplastics) was seen in DSC thermal analysis. No shift in T_g was observed as the weight percent of LCP was increased. The T_g value for pure PS, SPS, LCP/PS, and LCP/SPS systems were all approximately 100 °C.

3.3 *Morphological and Mechanical Characterization*

LCP/PS system (processed at 225 °C) had two-phase morphology with roundish LCP particles dispersed in the PS matrix, as seen in Fig. 3. Size of the particles ranged from smaller than 1 μm to bigger than 5 μm in diameter. The striking features are the sharp and distinct phase boundaries, the loose LCP material lying on the surface, and the absence of polymer matrix on the LCP particles, all of which suggest poor interfacial adhesion. Processing at a higher temperature of about 270 °C did not appear to make any difference in the morphology of the system.

The study on morphology was complemented by characterizing the associated tensile modulus and strength. Representative tensile stress-strain curves for the LCP/PS system at different compositions are shown in Fig. 4. A composite with 25% LCP was also tried, but it could not be molded. Poor interfacial adhesion between the LCP and polystyrene with wetting and mixing problems at high compositions is believed to be the reason.

Composite modulus did not seem to change significantly with composition; however, tensile strength (and strain to break) dropped dramatically with increase in LCP content. Plots of modulus and strength versus LCP content are shown in Fig. 5 and 6, respectively. Also the plots show the prediction by the rule of mixtures for the case of no adhesion between the LCP and polystyrene. If the interfacial adhesion does not exist between the LCP and the thermoplastic, the volume occupied by the LCP can mechanically be regarded as void, and the simple rule of mixtures yields the following equations for the composite modulus (E_c) (Ref 36) and composite strength (σ_c) (Ref 11, 37):

$$E_c = (1 - V_f)E_m \quad \text{(Eq 1)}$$

$$\sigma_c = (1 - V_f)\sigma_m \quad \text{(Eq 2)}$$

Table 2 Glass transition/degradation temperatures of the polymers used

	PS	SPS	LCP
Glass transition temperature, °C	100	100	...
Degradation temperature, °C	280	240	320

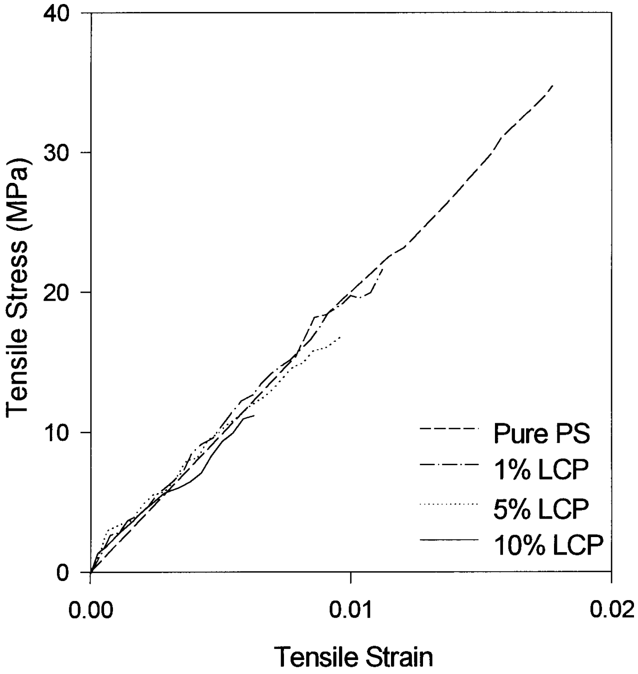

Fig. 4 Representative tensile stress-strain curves for LCP/PS system (compression molded at 225 °C) at various compositions

where V_f represents the volume fraction of the fiber (reinforcement) LCP, and σ_m and E_m are the tensile strength and modulus of the pure thermoplastic matrix, respectively. Volume fractions were calculated from the weight fractions using the specific gravities of PS and LCP of 1.05 and 1.5, respectively.

Composite modulus values were close to the predicted values for the case of no interfacial adhesion between the LCP and the polystyrene. However, the decrease in composite strength was much worse than the "no bond" prediction. This circumstance is understandable because the modulus is related to the stiffness of the material before fracture, and it could be predicted reasonably well by the rule of mixtures for the case of no interfacial bonding. On the other hand, composite tensile strength is a result of material fracture at the weakest point of the material, and it could drop well beyond the prediction by the rule of mixtures for the case of lack of adhesion between the components of the composite. When there is no (or poor) adhesion between the LCP and the thermoplastic matrix, existence of the LCP particles will cause stress concentrations at the interfaces of the two components (which will happen after the bond fails at some stress level for the case of some interfacial bonding). This concentration, especially around sharp edged LCP particles, could result in cracking and finally the failure of the material at a stress level quite below that predicted by utilizing the rule of mixtures in which the effect of discontinuities in the material is not taken into account.

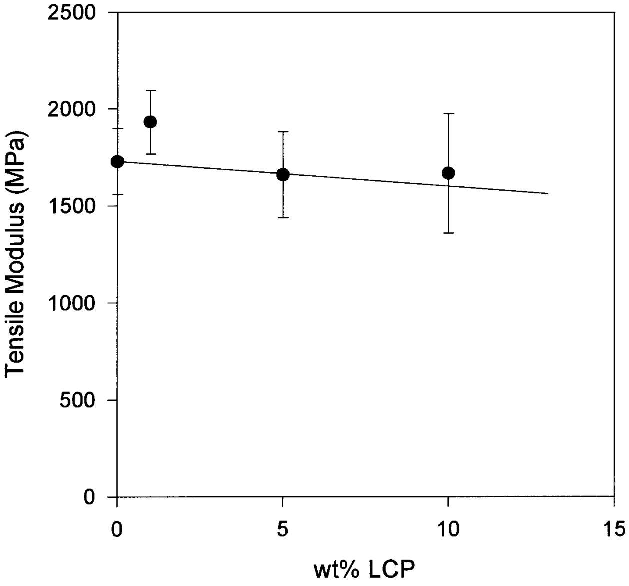

Fig. 5 Tensile modulus variation with LCP content for compression molded (at 225 °C) LCP/PS composite. The solid line corresponds to Eq 1.

Landon et al. (Ref 38) report that the equation:

$$\sigma_c = A\sigma_m - bV_f \qquad \text{(Eq 3)}$$

can be used to represent the experimental data, taking into account the stress concentration caused by the presence of spheres in the matrix, where *A* and *b* are constants. This equation represents the data well when *A* is 1 and *b* is about 6.75 σ_m, as shown in Fig. 6.

Tensile tests were also performed on the LCP/PS system processed at 270 °C (instead of 225 °C), the results were not different (Fig. 7). Higher processing temperature did not appear to help interfacial bonding of LCP/PS system. Composite modulus scattered about the prediction by the rule of mixtures for the no interfacial adhesion case (Fig. 8), and the composite tensile strength dropped again dramatically with increase in LCP content (Fig. 9).

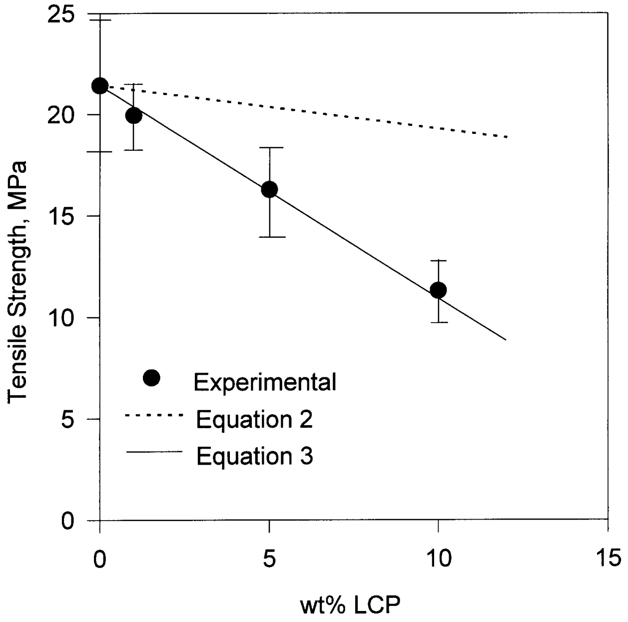

Fig. 6 Tensile strength variation with LCP content for compression molded (at 225 °C) LCP/PS composite

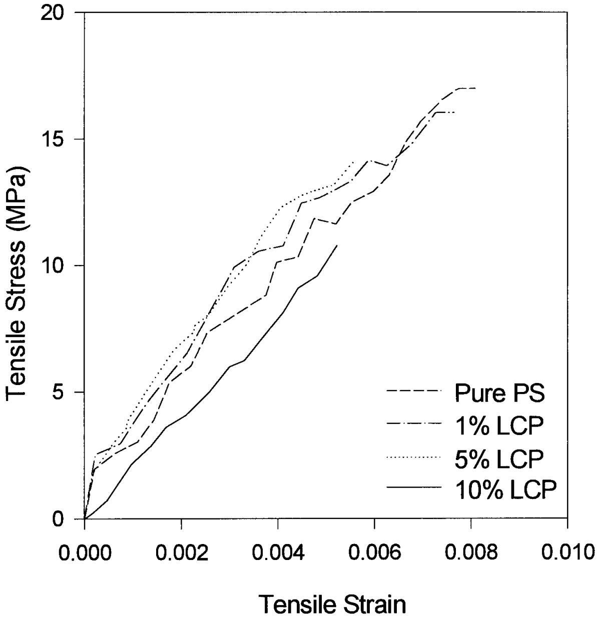

Fig. 7 Representative tensile stress-strain curves for LCP/PS system (compression molded at 270 °C) at various compositions

LCP/SPS System. Morphology of the LCP/SPS composite was not much different than the LCP/PS system. The interfaces between the LCP particles and the SPS matrix were also well defined (Fig. 10), indicating poor bonding between LCP and SPS. Low level sulfonation of PS (about 11%) did not seem to improve the compatibility appreciably. On the other hand, being able to form a composite plate with 25% LCP, while it was not possible (due to poor interfacial bonding) for the LCP/PS system, suggests that there should be some improvement in bonding between the LCP and SPS. However, this claim is not supported by the SEM micrographs.

Compression molding by keeping the material system at the process temperature (215 °C) for 1 and 2 h did not appear to change the overall morphology of the system; however, at least one LCP particle was found with not-so-well defined interface (Fig. 11), which might be an indication of some improvement in interfacial bonding.

Figure 12 shows the effect of LCP addition on the mechanical behavior of the LCP/SPS composite system. Pure SPS matrix and also the LCP/SPS system with the lowest LCP content resulted in a rugged fracture surface with some degree of plastic deformation (Fig. 13), whereas plastic deformation was at an insignificant level for the PS used as received. Further LCP addition resulted in embrittlement of the LCP/SPS system, decreasing the composite strength (Fig. 14) below that predicted by the rule of mixtures for the case of no interfacial

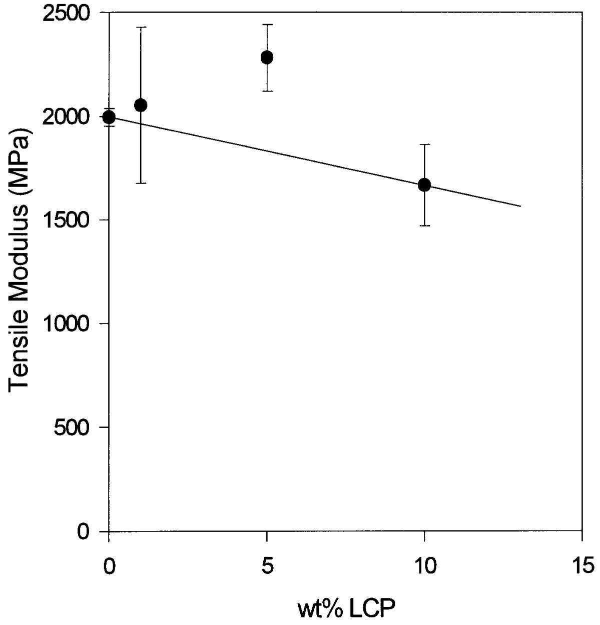

Fig. 8 Tensile modulus variation with LCP content for compression molded LCP/PS composite (at 270 °C). The solid line corresponds to Eq. 1.

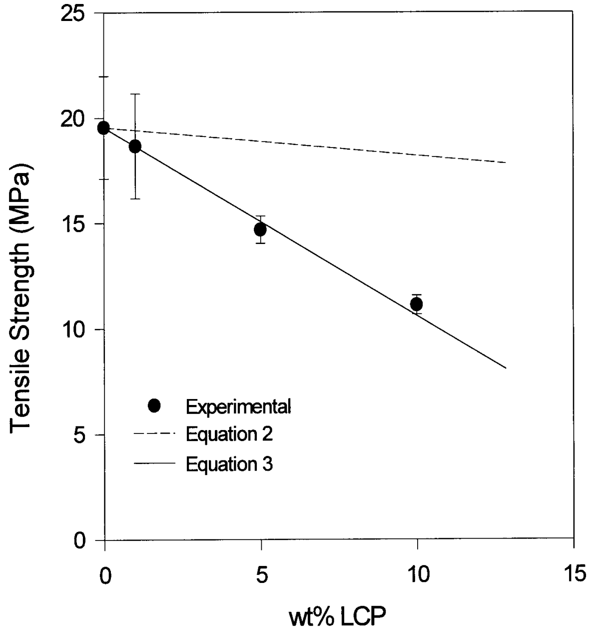

Fig. 9 Tensile strength variation with LCP content for compression molded LCP/PS composite (at 270 °C)

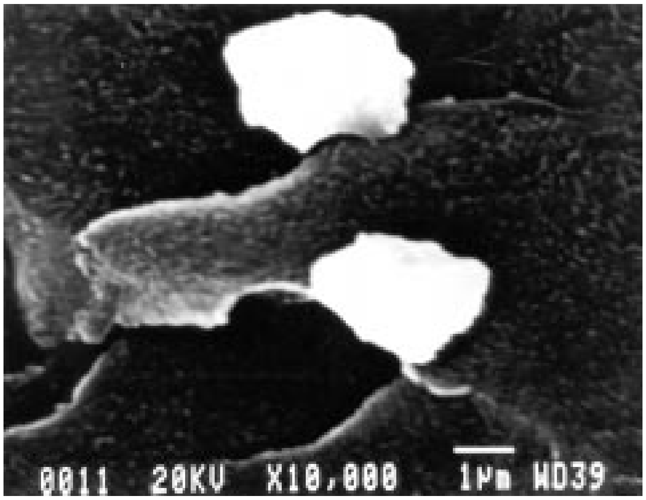

Fig. 10 SEM micrograph of a fracture surface of LCP/SPS system showing sharp interface boundaries around LCP particles. (Art has been reduced to 78% for printing.)

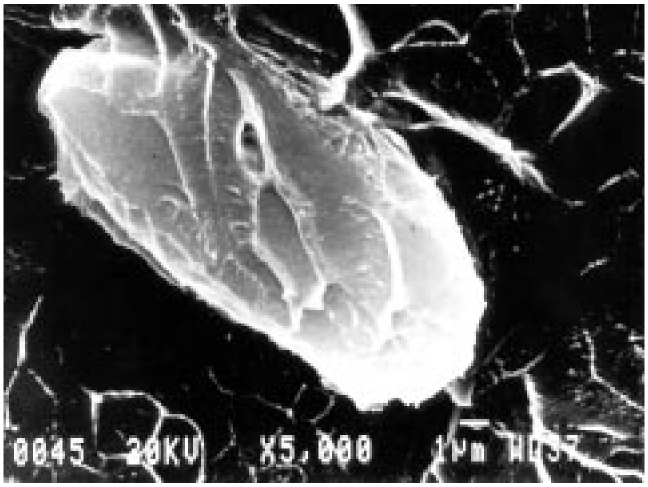

Fig. 11 SEM micrograph of a fracture surface of LCP/PS system (treated at the process temperature for 2 h) showing an LCP particle with not so sharp boundaries. (Art has been reduced to 78% for printing.)

adhesion due to the same possible reasons outlined earlier for the LCP/PS system. However, composite strength for any composition was higher for the LCP/SPS system than the LCP/PS system.

Composite modulus, on the other hand, did not change significantly with LCP addition, even with the addition of 25% LCP (Fig. 15). Being able to form LCP/SPS composite with as much as 25% LCP without losing the composite stiffness, while not possible for the LCP/PS system, indicates better bonding between the LCP reinforcement and the SPS matrix than the LCP/PS system. However, the lowering of the composite strength with LCP addition suggests that the interfacial bond might have been improved over the LCP/PS composite, but it is still weak.

Heat treating the LCP/SPS composite (containing 5% LCP) at the compression molding process temperature for 1 and 2 h did not seem to play an important role in the mechanical response of the material. As shown in Fig. 14 and 15, composite modulus and strength were lowered by heat treatment of 1 h, but they increased when heat treated for 2 h, over the system processed for about 5 min. The composite strengths for the two cases were still well below that predicted by the rule of mixtures for no interfacial adhesion case.

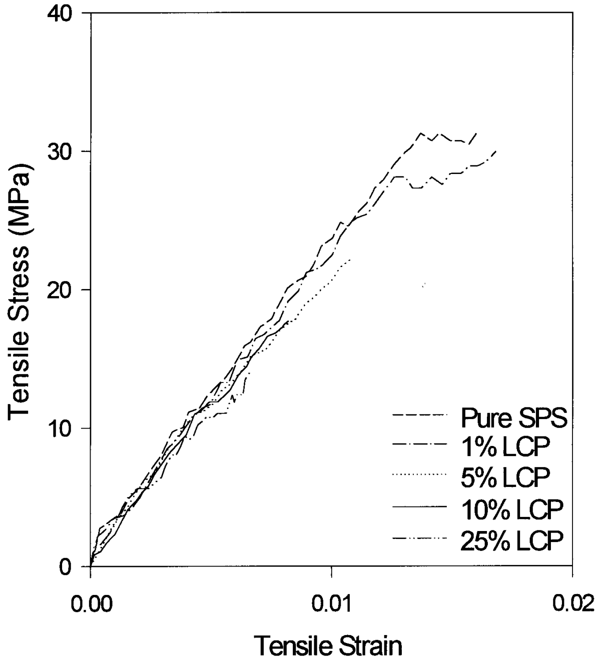

Fig. 12 Representative tensile stress-strain curves for LCP/SPS system (compression molded at 215 °C) at various compositions

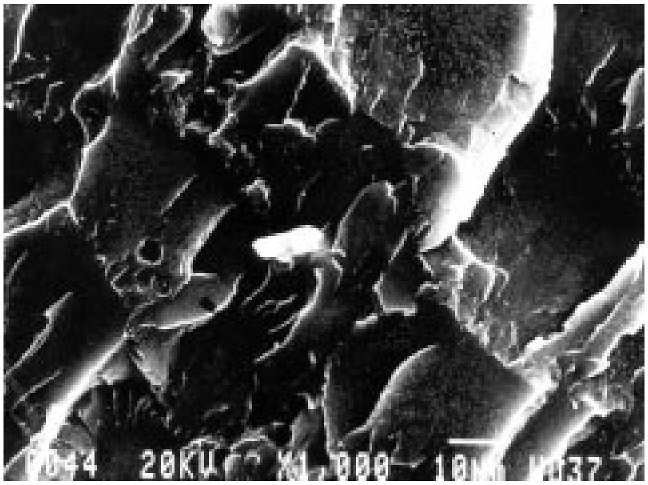

Fig. 13 SEM micrograph of a LCP/SPS system (with 1% LCP) showing a rugged fracture surface with some degree of plastic deformation. (Art has been reduced to 78% for printing.)

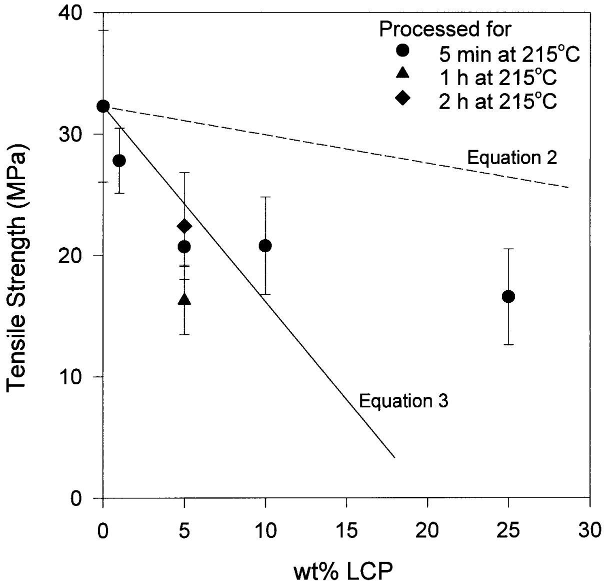

Fig. 14 Tensile strength variation with LCP content for compression molded LCP/SPS system

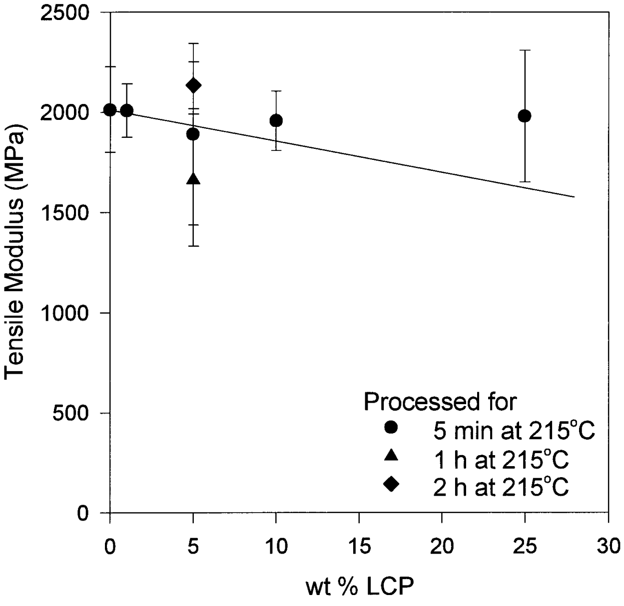

Fig. 15 Tensile modulus variation with LCP content for compression molded LCP/SPS system. The solid line corresponds to Eq 1.

4. Summary and Conclusions

The LCP was synthesized at room temperature by interfacial polycondensation reaction between terephthaloyl chloride and dihydroxydiphenyl sulfone. The copolymer synthesized was so stable and molecularly rigid that no glass transition temperature was recorded. Polystyrene was sulfonated to study the effect of the interacting group (–SO_3H) on the interfacial adhesion between LCP and polystyrene. Incorporation level of –SO_3H group to polystyrene was about 11%. The LCP was incorporated in PS and SPS in 1, 5, 10, and 25 wt%. However, the 25% LCP/PS plate could not be fabricated, possibly due to the lack of proper interfacial adhesion. The incompatibility (poor interfacial adhesion) of the LCP/PS system resulted in sharp decrease in the tensile strength of the LCP/PS composite with increase in LCP content. The morphology and the mechanical performance of the system did not change when it was processed at a higher temperature (270 °C instead of 225 °C).

In the case of LCP/SPS, again a poor bonding was observed between LCP and SPS, indicating that 11% of sulfonation of polystyrene was not enough to improve the compatibility significantly. However, a composite plate of 25% LCP/SPS was formed, unlike in the case of 25% LCP/PS, which could be an indication of some improvement in bonding between LCP and SPS. The strength of LCP/SPS system also decreased with increase in LCP content. However, composite strength for any composition was higher for the LCP/SPS system than the LCP/PS system. When 5% LCP/SPS system was processed for 1 and 2 h (instead of 6 min), overall morphology of the system did not seem to change much except in one SEM micrograph, in which the boundaries of LCP particle were not well defined indicating a chance of some improvement in interfacial bonding.

Investigation of the possibility of improving the interfacial adhesion between LCP and the thermoplastic by increasing the density of the functional groups in the LCP molecule, and/or incorporating strong interaction/ionic groups, such as OH or $SO_3^-Na^+$ in the thermoplastic, is pending.

The utilization of extrusion and/or injection molding processing techniques is also being considered. For these methods, a meltable LCP is essential, and larger quantities of the LCP and the thermoplastic are required. Due to high shear rates used in these techniques, the meltable LCP can be orientated in fibril form thus imparting high modulus and strength to the LCP/thermoplastic system in the direction of orientation (Ref 3-6, 26-31). The LCP is expected to be synthesized as meltable by introducing flexible spacers, side chains, or kinks in the main chain of the LCP molecule (Ref 39).

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