

Novel Approach to Fibrillation of LCP in an LCP/PP Blend

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ABSTRACT: A novel concept of improving shear-induced fibrillation of liquid crystalline polymer (LCP) in LCP/thermoplastic blend systems was introduced. Silica fillers (SiO₂) were added to an LCP/polypropylene (PP) system to serve as a viscosity thickening agent and to improve the fibrillation of the LCP phase. The formation of LCP fibrils was found to enhance with the incorporation of 5–15 wt % of fillers. The presence of LCP fibrils improved the flow properties of the LCP/PP/SiO₂ composites. It was evident from the rheological and morphological studies that the addition

of silica led to an increase of the aspect ratio of the LCP fibrils, which, in turn, should improve their effectiveness as reinforcements and/or toughening agents. Substantial improvement in LCP aspect ratio was achieved by the introduction of hydrophobic SiO₂ fillers in the PP/LCP blends. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 86: 2070–2078, 2002

Key words: liquid-crystalline polymers; poly (propylene) (PP), fillers; reinforcement

INTRODUCTION

In recent years, there has been a great deal of emphasis placed on the development and production of high-strength lightweight materials. Blending of polymers has been widely established as a cost-effective method for producing new materials. Blends of thermoplastics and liquid crystalline polymers (LCPs) have been the focus of intense academic and industrial interest. Much of this interest stems from the unique fibrillar morphology that can be developed from the LCP dispersed phase in these blends upon processing operations.

Polypropylene (PP) is one of the widely used commodity polymers due to its good mechanical properties, ease of processing, and acceptance to various types of fillers.¹ Due to its low cost, PP has applications in houseware, packaging, and automotive industries. Various authors have demonstrated the possibility of blending PP^{2–8} or filled PP^{9–13} with LCPs, but not many^{6,9–10,13} investigated shear-induced fibrillation. There is also no methodical study reported on the quantification of LCP fibrillation as a function of the shear rate. In particular, the field of filled LCP/thermoplastic composites has been relatively unexplored. Although there have been articles which reported on LCP/thermoplastic blends containing fillers, the authors did not incorporate fillers intentionally to study the effects of fillers.

As early as 1983, Baird and Wilkes,¹⁴ who recognized the high cost of engaging pure LCP, invented a new method known as sandwich injection molding which produces composites of LCP forming the skin and wrapping around the filled PET core. The fibrillation of the LCP skin layer coupled with good adhesion between the skin and core structures rendered superior properties to the moldings.

It is common knowledge that LCP can ease the processability of thermoplastics when blended with the latter. Hence, He et al.¹⁵ introduced an LCP to improve the flow of glass fibre-filled PP. Under the influence of the LCP, the glass fibers were better oriented and the mechanical properties of the ternary composites were improved.

Although the fibrillation of LCPs can produce reinforcements that improve the mechanical properties of *in situ* composites, the blending of an LCP with PP, however, does not always result in the formation of fibrillar structures under a shear flow.¹⁴ Hence, the majority of the studies of LCP/PP *in situ* composites involve postextrusion drawing to facilitate the fibrillation process.^{16–19}

It has often been reported that the mechanical performance is directly proportional to the draw ratio since the enhanced orientation and reduced fibril diameter are manifested as better strength and modulus.^{16,17} However, Qin et al.¹⁸ found, from spun fibers of Vectra/PP blends, that a high draw ratio could only result in an initial increase in the mechanical properties. When the draw ratio was further increased, the fibers would generate a greater resistance toward

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drawing and impart adverse effects on the strength and modulus of the fibers.

The process of shear-induced fibrillation was governed by several factors that include the viscosities of each component polymer, miscibility between components, and processing conditions.^{20,21} It is commonly known that the viscosity ratio of an LCP to a thermoplastic matrix is an important factor that determines shear-induced fibrillation in *in situ* composites.^{6,20–22} Several authors^{23–25} have reported that fibrous microstructures were obtained only when the viscosity ratio of the dispersed phase to the matrix was below unity. Heino et al.⁶ investigated various types of LCP/PP blends and noticed that, while fibrillar LCP was formed at viscosity ratios ranging from 0.5 to 1, spherical or clusterlike morphology was exhibited for ratios above unity. Similar trends were also determined using other thermoplastic matrices.^{26–29} In fact, the above-mentioned parameters are related to each other through the capillary number (Ca),¹⁷ which is defined as the ratio of the shear stress acting on the LCP droplet by an external flow field to the interfacial tension which resists the deformation of the spherical droplet:

$$Ca = \eta_m [1 - (4\phi_d\phi_m)^{0.8}] \frac{d\gamma(16\lambda + 16)}{2\sigma(19\lambda + 16)} \quad (1)$$

where γ is the shear rate; d , the diameter of the dispersed phase droplet; σ , the interfacial tension; ϕ_d , the volume fraction of dispersed phase (LCP); ϕ_m , the volume fraction of the matrix phase; η_m , the viscosity of the matrix; and $\lambda = \eta_{LCP}/\eta_m$, the viscosity ratio.

The effective fibril transformation from an LCP droplet would take place when Ca exceeds the critical limit ($Ca_{critical}$) by many times.² In view of such a condition, a new approach of adding fillers as a viscosity thickening agent to increase Ca was put forward.

This study concerned the addition of fine silica particles as a viscosity thickening agent to a PP matrix to modify its viscosity and, consequently, the LCP fibrillation. It was found that there was an improvement of the LCP fibrillation in the filled PP. While there are reports on filled LCP/PP systems,^{9–13} there has been little systematic discussion to provide a detailed understanding regarding the effect of fillers on LCP fibrillation. Hence, the improvement of LCP fibrillation in the filled PP was examined.

Since the mechanical and physical properties of the blends depend on their morphological structures, it is the intention of this work to quantify the morphology of the LCP phase as well. With the quantification of the fibrillar structures, the roles of different interdependent parameters could be better understood in (PP/SiO₂)LCP systems containing various silica con-

TABLE I
Temperature Profiles for Processing (SiO₂/PP)/LCP Composites

Process	Material	Temperature profile (°C)
Extrusion	SiO ₂ /PP	130/200/200/200/200/200
Extrusion	(SiO ₂ /PP)/LCP	230/280/280/275/275/270

tents. Recently, He et al.³⁰ introduced the new idea of *in situ* hybrid composites. Hence, the information gained from this study would reveal the feasibility of the new concept on employing fillers to achieve *in situ* hybrid LCP reinforcements by shear-dominating processing methods such as injection molding.

EXPERIMENTAL

Materials

The blends investigated were prepared using Rodrun LC5000 from Unitika (Japan) as the dispersed phase and polypropylene (PP) as the matrix. LC5000 is a copolyester consisting of 20% poly(ethylene terephthalate) and 80% poly(*p*-hydroxybenzoic acid). The PP matrix is a commercial product of Mitsui Toatsu Chemicals Inc. (Japan) with a melt-flow index of 4. Hydrophobic silica fillers of 12 nm in diameter were supplied by JJ Degussa (Singapore). The surface of the silica particles contained siloxane and silanol groups. The fillers were treated with organosilanes to render them predominantly hydrophobic. The hydrophobic silica was used because of its good compatibility with PP and its poor compatibility with the LCP.

Processing

Prior to processing, the polymers and fillers were dried for 24 h under a vacuum at 105°C. Preblending was achieved in two steps, where 0, 5, 10, and 15 wt % of silica were first mixed with PP followed by 30 wt % of the LCP using a Leistritz Mic 18/G1-30D twin-screw extruder with a feed rate of 5 rpm and screw speed of 120 rpm. The temperature profiles for the extrusion are given in Table I.

Rheological measurements

The melt-flow properties of individual components and blends were measured using a capillary rheometer (Gottfert Rheograph 6000). The length and diameter of the capillary die were 10 mm and 1 mm (L/D ratio = 10), respectively. A run angle of 180° was selected. Measurements were taken at 280°C over a range of shear rates of 50, 200, 500, 1000, and 3000 S⁻¹. The extrudates at the shear rates of 50, 500, and 3000 S⁻¹ were collected for morphological analysis.

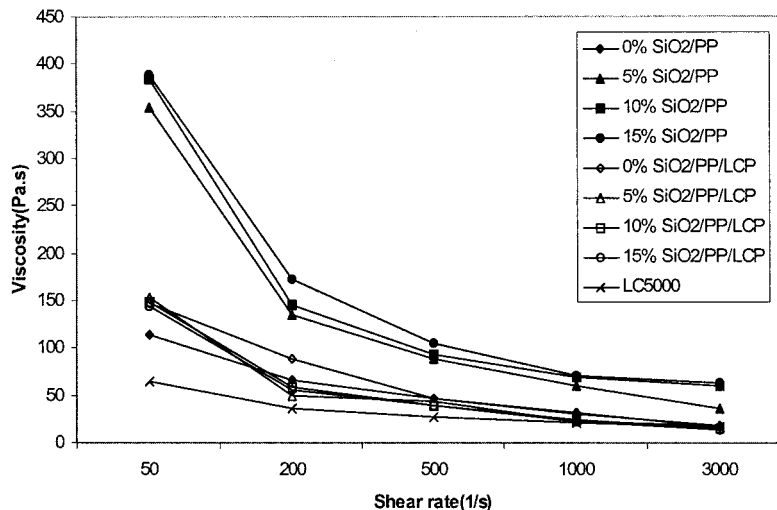


Figure 1 Viscosities of LC5000, PP/SiO₂ hybrid composites, and (PP/SiO₂)/LCP blends as a function of shear rate at 280°C.

Scanning electron microscopy

The morphology of specimens were examined using a JEOL JSM-5410 scanning electron microscope with an accelerating voltage of 10 kV. The extrudates were refluxed in toluene for 24 h to remove the PP matrix and the residue from both the skin and core was collected and dried for aspect ratio quantification.

Light microscopy

Polarizing optical microscopy was employed to observe the morphologies of the dispersed LCP phase. A thin film of the extrudate was applied onto a glass coverslip by melting the blend over a hot plate set at 200°C. Such a temperature would ensure that only the matrix material (PP) was melted, leaving the LCP phase relatively undisturbed. The LCP phase observed was taken from the entire extrudate which consisted of a mixture of LCP particles at both skin and core. An Optiphot2-Pol polarizing microscope was utilized to capture the microstructures.

RESULTS AND DISCUSSION

Rheology

Viscosities of the LCP (PP/SiO₂) and LCP/filled PP blends at 280°C are illustrated as a function of the shear rate in Figure 1. LC5000 exhibited the lowest viscosity among all melts, which is a characteristic of LCPs. The viscosity of PP was slightly higher than that of LCP. Upon the addition of silica, the viscosity of PP increased. It was observed, as expected, that a higher filler content generally means higher viscosity of the filled PP at all shear rates studied because interparticle interactions become stronger with larger amounts of

fillers and free movement of the polymeric chains would be restricted.

Pseudoplasticity was demonstrated for all compositions and drastic shear thinning was noticed for the filled systems. Since the silica particulates were merely 12 nm in diameter, they have a high specific surface area which led to significant aggregation. At low shear rates, the particle aggregation was strong, contributing to high viscosity. With mounting shear rates, the deformation force was large enough to break up the aggregates. Hence, the viscosity decreases abruptly with the breakage of the particle–particle bonds.

The rheological behavior of the blends responded differently with the introduction of the LCP. The flow curves of the LCP/(PP/SiO₂) blends fall between those of the pure components for the entire shear rate range studied, as noticed from Figures 2–6, except for the unfilled systems. The viscosity of the filled PP decreased drastically upon the addition of the LCP, indicating an improvement in the processability. However, for the pure PP, it was observed that the LCP did not lower the viscosity of PP at low shear rates. At shear rates such as 50 and 200 s⁻¹, the viscosity of the unfilled LCP/PP blend was higher than that of the pure PP. LCPs molecules form domains where the orientation is random prior to shearing.³¹ With a considerable amount of shearing, the domains would orient in the shear direction.³² When the shear rate applied is insufficient to induce alignment, the domains could only tumble and rotate. Hence, the viscosity of the LCP/PP blend was observed to be higher than that of pure PP at low shear rates, as shown in Figures 2 and 3.

With increasing shear rates, the viscosities of the blends also decreased. The unfilled LCP/PP blends approached the viscosity of PP while the viscosity of

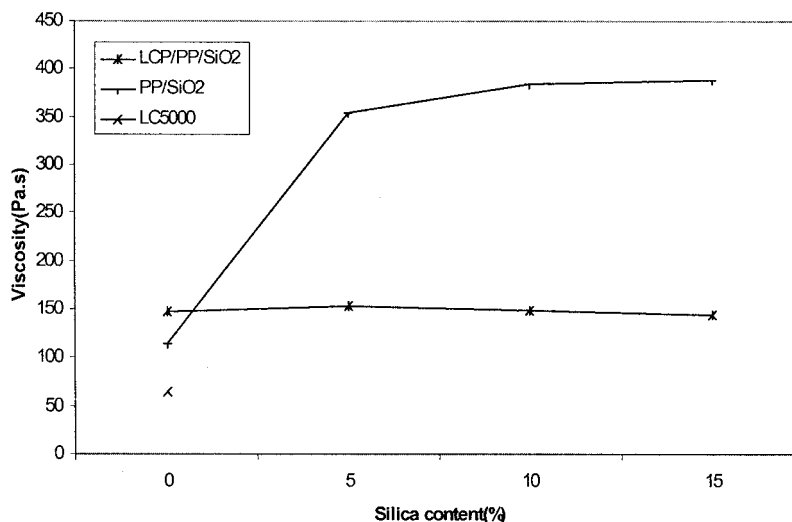


Figure 2 Viscosities of LC5000, PP/SiO₂ hybrid composites, and (PP/SiO₂)/LCP blends as a function of silica content at a shear rate of 50 s⁻¹ at 280°C.

filled blends decreased consistently with an increasing silica content. Inasmuch, the viscosity of the (PP/SiO₂)/LCP blends sheared at 500, 1000, and 3000 s⁻¹ were reduced to values even lower than those of the LCP/PP blend, as can be seen in Figures 4–6. This could possibly be ascribed to the formation of fibrillar LCP, since under high shearing, the domains would orient in a common direction and slide past each other, thereby facilitating flow.

It has been generally believed that among the factors that govern fibrillation of LCP the viscosity ratio plays a vital role. In fact, earlier work^{4–8} believed that a viscosity ratio less than unity would favor fibrillation. Thus, a graph of viscosity ratios obtained at various shear rates was plotted.

From the trends discerned in Figure 7, the viscosity ratio of the unfilled blend was highest over the whole range of shear rates measured. Subsequently, when progressively more fillers were added to the PP matrix, the more viscous melt was obtained, giving a lower viscosity ratio at each respective shear rate. However, the viscosity ratios of the filled composites did not decrease significantly by further addition of silica beyond 5 wt %.

When the shear rate was increased the viscosity ratios of both the filled and unfilled blends increased as well. This relationship was attributable to the different shear-thinning effects of the dispersed phase and PP. Hence, the viscosity ratio depended not only on the quantity of the viscosity

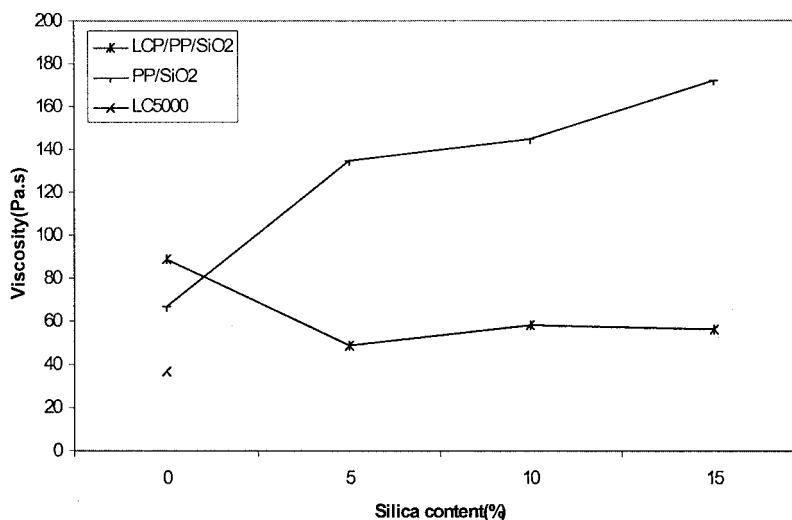


Figure 3 Viscosities of LC5000, PP/SiO₂ hybrid composites, and (PP/SiO₂)/LCP blends as a function of silica content at a shear rate of 200 s⁻¹ at 280°C.

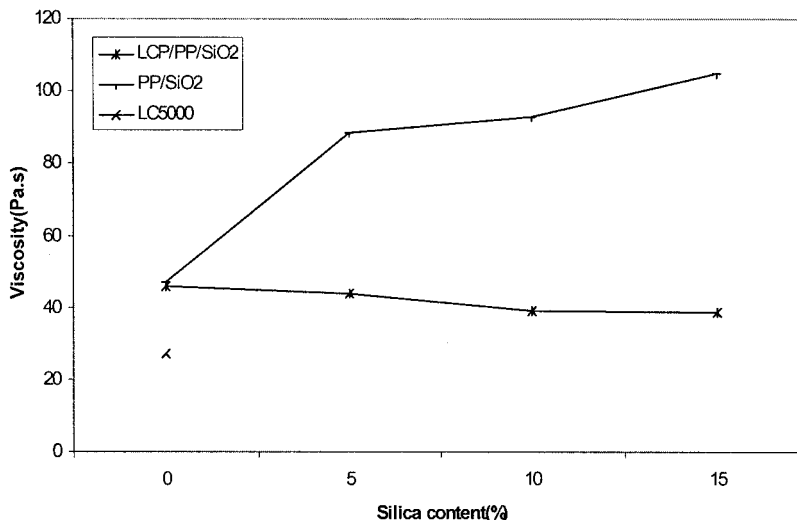


Figure 4 Viscosities of LC5000, PP/SiO₂ hybrid composites, and (PP/SiO₂)/LCP blends as a function of silica content at a shear rate of 500 s⁻¹ at 280°C.

thickening agent incorporated but also on the shear rate.

In this particular system selected, the viscosity ratios of the blends either with or without silica fillers were below unity despite the shear rate adopted. The PP/LCP blend possesses viscosity ratios ranging from 0.28 to 0.67, while that of the 15 wt % silica-filled ternary blend exhibited a range of viscosity ratios from 0.15 to 0.39. According to several authors,⁴⁻⁸ both filled and unfilled systems should have met the criteria for fibrillation if considering only the viscosity ratio. Thus, morphological studies were conducted to determine the fibrillation process.

Morphology

Figure 8 depicts the effect of the shear rate on the morphology of the blends. For each individual type of

composite, it was found that an increase in the shear rate from 50 to 3000 s⁻¹ improved fibrillation. From the series of micrographs, it can be seen that when no silica was added the majority of the LCP domains existed as spheroids even at high shear rates. With escalating shear rates, a few occasional fibrils were observed at a shear rate above 500 s⁻¹.

When fillers were introduced, more fibrillar structures at each corresponding shear rate were noticed. In particular, when 15 wt % of silica particles were added, even at a shear rate as low as 50 s⁻¹, fibrils began to appear. As the shear rate was further increased, more fibrous structures were generated.

The morphological studies provided strong support to the rheological data where blends with a high loading of fillers demonstrated lower viscosities due to the presence of fibrillar LCP (see Fig. 1). It was evident that the inclusion of silica fillers has facilitated the

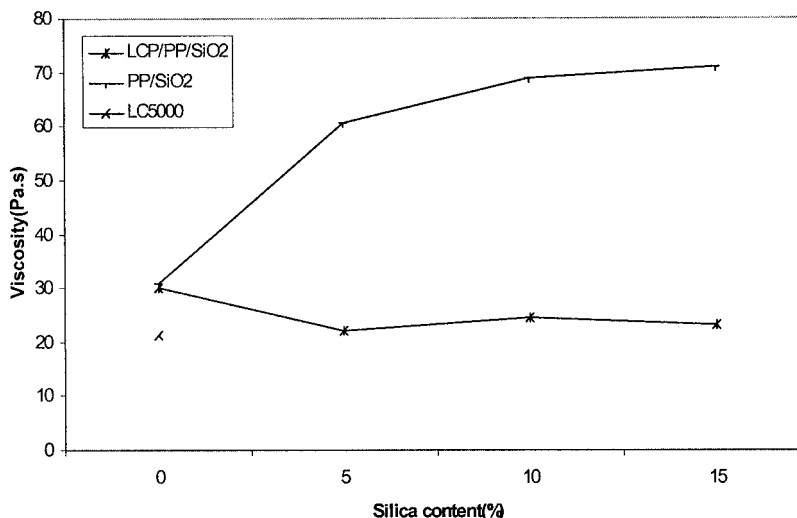


Figure 5 Viscosities of LC5000, PP/SiO₂ hybrid composites, and (PP/SiO₂)/LCP blends as a function of silica content at a shear rate of 1000 s⁻¹ at 280°C.

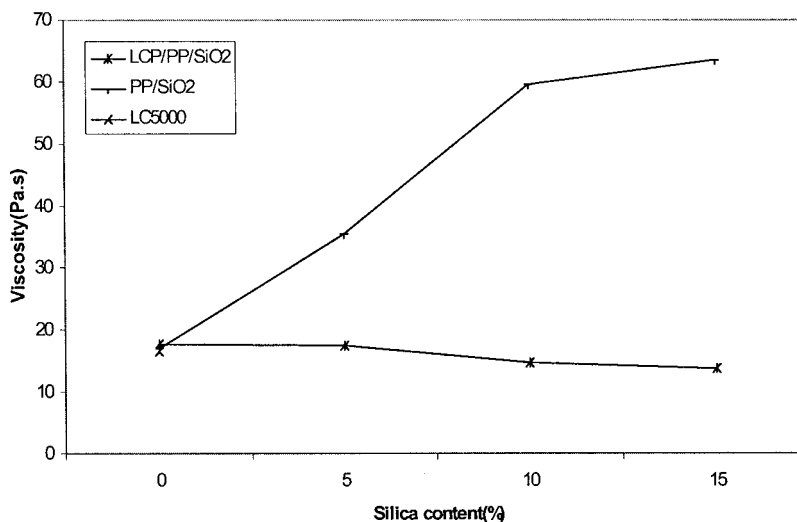


Figure 6 Viscosities of LC5000, PP/SiO₂ hybrid composites, and (PP/SiO₂)/LCP blends as a function of silica content at a shear rate of 3000 s⁻¹ at 280°C.

fibrillation of LCP. Hence, an attempt to quantify the morphology of the LCP domains using scanning electron microscopy (SEM) was adopted. Quantifying fibrils using an extracted LCP phase through SEM was believed to provide more realistic aspect ratios than did the polarizing optical micrographs, as the latter reflects fibrillation globally with bundles of fibrils entangling.

In line with light microscopy, the electron micrographs in Figure 9 revealed similar findings, where higher shear rates imparted fibrillar LCP and the dispersed phase became more fibrous with increasing filler content. The aspect ratio distribution of the LCP fibrils in (PP/SiO₂)/LCP polyblends are presented in Figure 10.

The histogram of the unfilled blends suggested that the majority of the LCP domains were of a low aspect ratio regardless of the shear rate. In fact, most of the LCP particles existed as spheroids, which had an as-

pect ratio of 1. As the shear rate became higher, there were fewer LCP spheres and LCP domains with a larger aspect ratio began to emerge. Without the addition of fillers, the maximum aspect ratio that can be reached was 15 at a shear rate of 3000 s⁻¹.

With as little as 5 wt % of silica fillers, there were fewer LCP particles with low aspect ratios of 1–5. As the shear rate increased, more fibrils with aspect ratios above 5 were obtained and the maximum aspect ratios attainable were in the range of 15–20. When the silica content reached 10 and 15 wt %, LCP fibrils having aspect ratios of 21–25 were noticed. Generally, as more fillers were added, the LCP domains were able to develop into longer and finer fibrils.

However, it should be noticed that the maximum aspect ratio obtained morphologically could only be an approximation. This is due to the existence of a skin-core phenomenon within the extrudate. As the preparation could not differentiate the skin and core

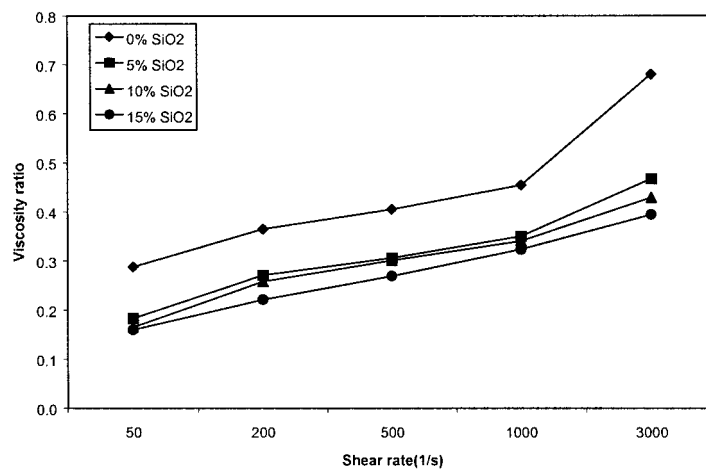


Figure 7 Viscosity ratios of (PP/SiO₂)/LCP blends with various filler contents as a function of shear rate at 280°C.

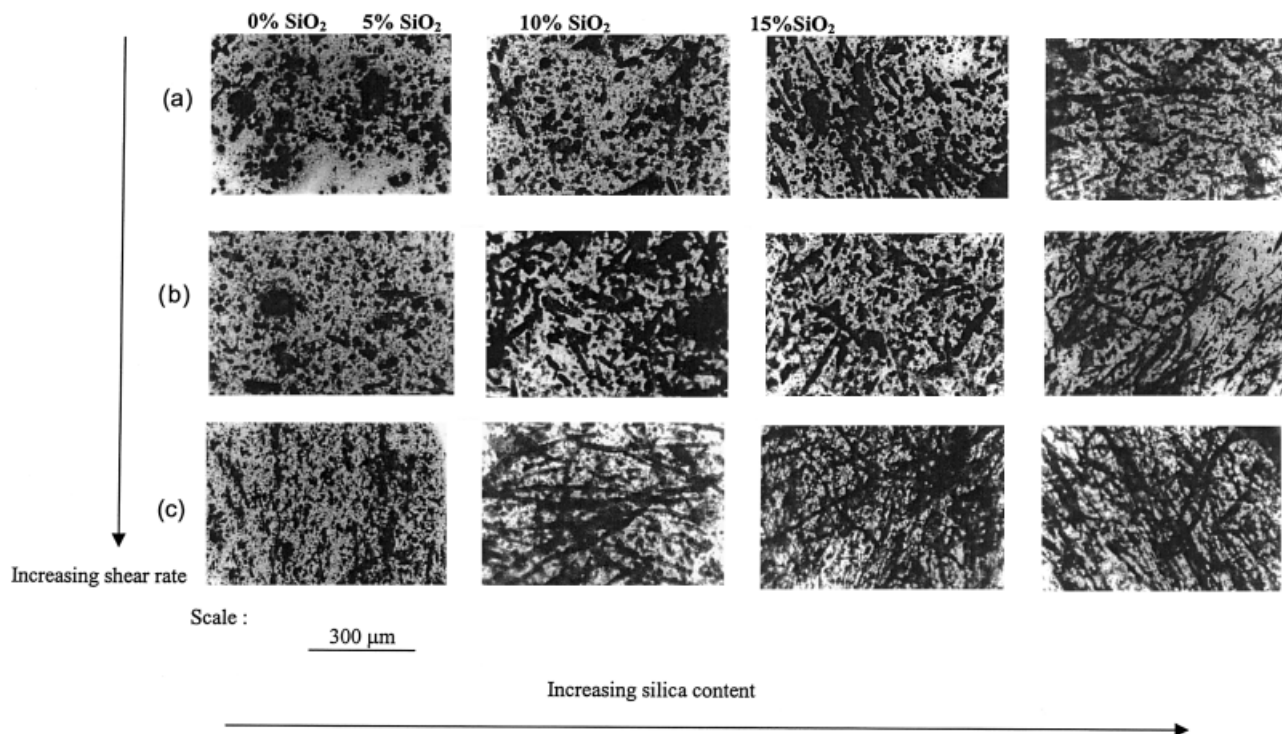


Figure 8 Polarizing optical micrographs of (PP/SiO₂)/LCP blends extruded at 280°C with shear rates of (a) 50 s⁻¹, (b) 500 s⁻¹, and (c) 3000 s⁻¹.

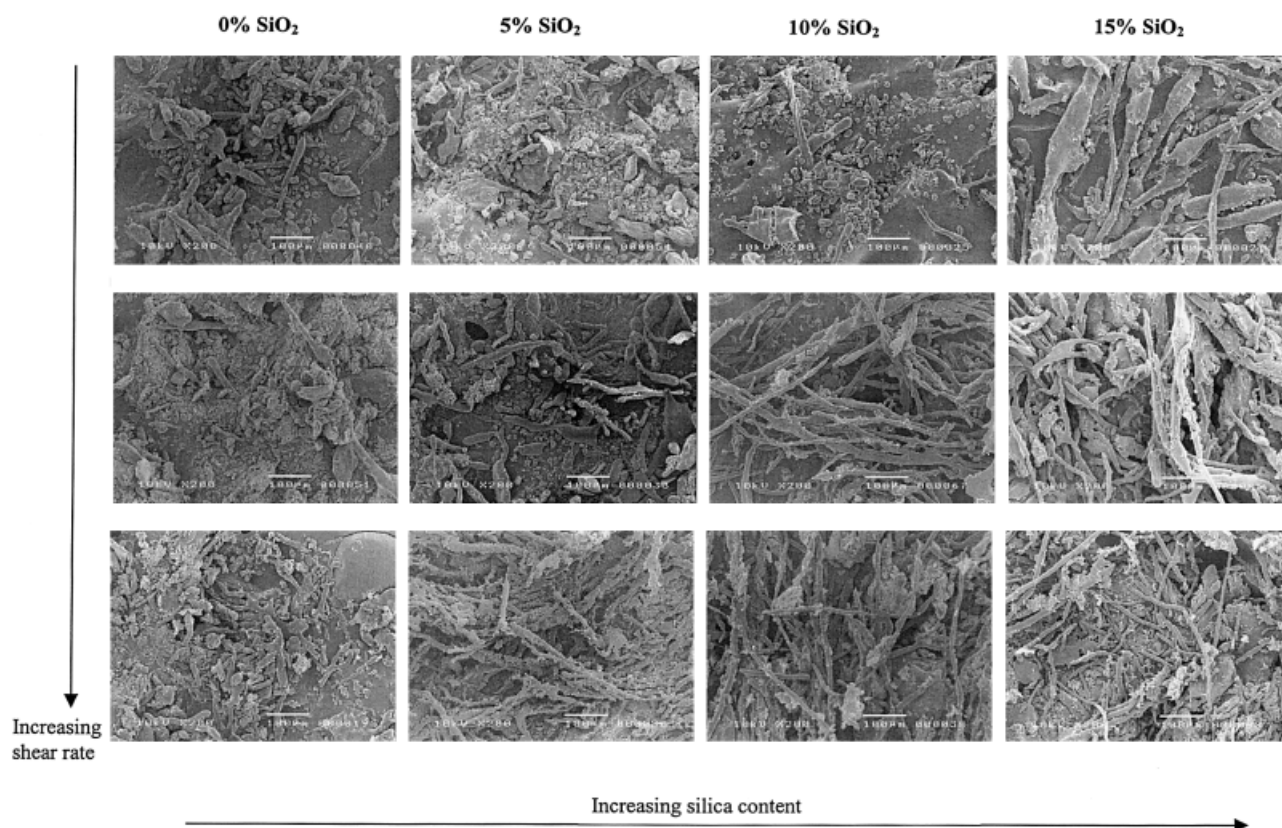


Figure 9 Electron micrographs of extracted LCP extruded at 280°C with shear rates of (top) 50 s⁻¹, (center) 500 s⁻¹, and (bottom) 3000 s⁻¹.

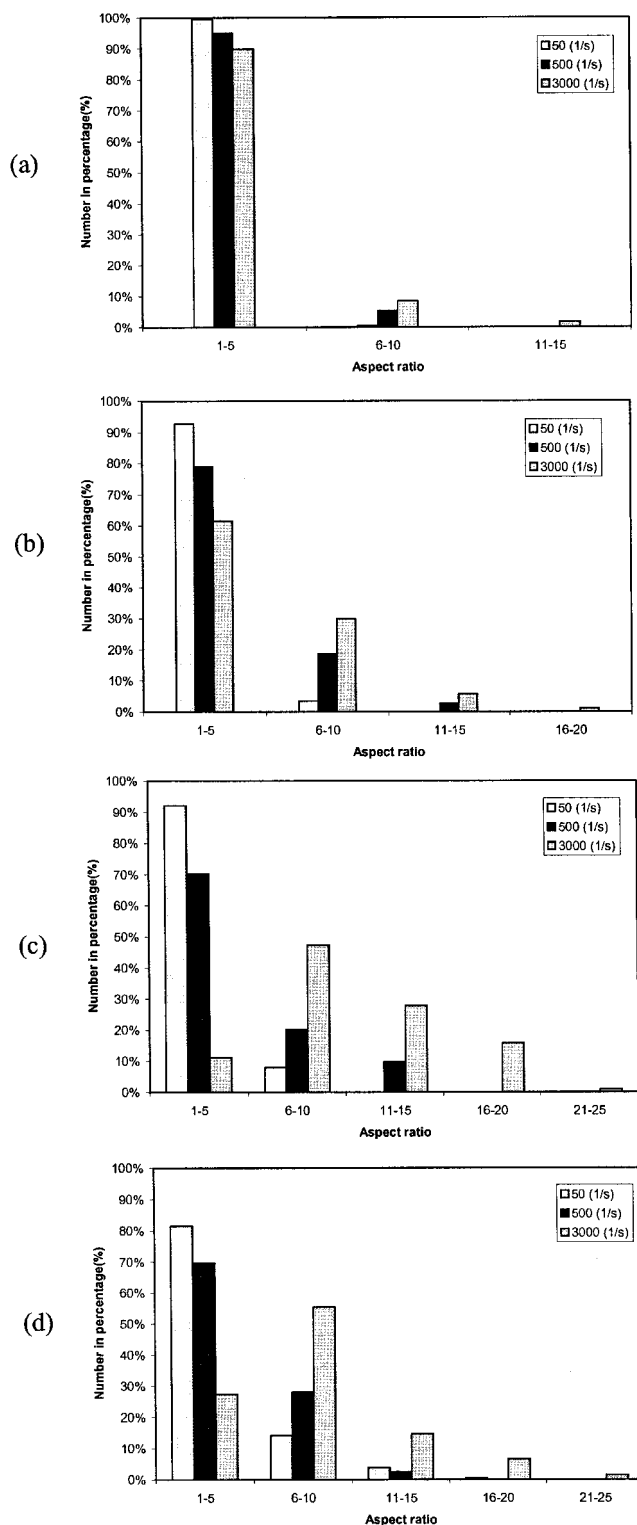


Figure 10 Aspect ratio distributions of LCP fibrils in (PP/SiO₂)/LCP composites with different silica contents and shear rates: (a) 0 wt % SiO₂; (b) 5 wt % SiO₂; (c) 10 wt % SiO₂; (d) 15 wt % SiO₂.

particles, the aspect ratio revealed only an average of the particle dimension. Hence, the true aspect ratios of the fibrils could not be exactly reflected. Nonetheless,

the quantification of the fibrils could provide useful information that the addition of silica fillers could serve to improve the LCP fibrillation.

Through the rheological and morphological studies, it was determined that ascending shear rates were capable of inducing fibrillation within the (PP/SiO₂)/LCP ternary systems. This was attributed to the LCP experiencing a larger shear deformation that accompanied the higher shear rates. With escalating shear rates, the higher shear stress could deform the large LCP domains and/or make small LCP particles coalesce to generate LCP fibrils with a higher aspect ratio as revealed by Figures 9 and 10. The larger deformation force by higher shear rates would result in the coalescence of LCP domains into longer fibrils.

From the standpoint of Ca, the positive relationship between the shear rate ($\dot{\gamma}$) and Ca would denote that a higher shear rate would cause a larger Ca, thereby increasing the efficiency of the LCP droplet transforming into fibrils. In addition to the effect of the shear rate, the hydrophobic silica content was another factor that affects the fibrillation of the LCP phase. The silica content could also influence the value of Ca. This was achieved indirectly through variations in both the matrix viscosity (η_m) and the viscosity ratio (λ). With the hydrophobic silica in the PP phase, η_m would increase when more hydrophobic silica particles were added. Consequently, the value of Ca would be higher. As λ is given by η_{LCP}/η_m , any modifications to η_m would alter λ as well. Hence, the high silica concentration would reduce λ .

However, through the expression of Ca, it could be seen that the influence of the term λ on Ca is small compared to the effect of η_m . The expression of Ca with λ , that is, $(16\lambda + 16)/(19\lambda + 16)$, would approach unity as λ decreased to infinity. Hence, it is believed that λ should have an insignificant effect on Ca. With the assumption that $Ca_{critical}$ remained constant with an increasing silica content, the addition of more silica particulates would increase Ca and improve the fibrillating ability of the system.

Hence, from the observations, it was concluded that fibrillation of the LCP phase could be achieved through an increase in the shear rate and/or hydrophobic silica content. From the statistical results, it was concluded that the hydrophobically natured silica would serve as an effective viscosity thickening agent.

Furthermore, it was realized from the rheological and morphological analyses that the fibrillation was a complicated process. Although it was shown experimentally in Figure 7 that the viscosity ratios of all the LCP/PP composites were below unity for the entire range of shear rate studied, there were not many LCP domains being deformed into fibrils for this particular system. Thus, having a viscosity ratio smaller than unity might not necessarily imply fibrillation of LCP. In fact, there were articles^{9,11,13} which reported that

fibrillation of LCP in the PP matrix was still possible even though the viscosity ratio exceeded unity. Hence, this suggests that the fibrillation is a complicated process and the viscosity ratio may not be the only factor governing fibrillation.

From the mathematical representation of Ca , it is illustrated that λ , γ , and η_m are the three factors that affect fibrillation. However, changes in λ would not affect Ca very much. Moreover, both λ and η_m are shear-rate dependent. The effect of having a viscosity ratio less than unity on fibrillation of the dispersed phase is thus a complex issue, as it is a dynamic situation dependent on the shear rate applied. Indeed, it was felt instead that the shear rate that a system experiences would be a major determinant for fibrillation. It is evident from Figures 7–9 that more LCP fibrils were formed in all the compositions when the shear rates were high despite the increasing viscosity ratio. Therefore, the better LCP fibrillation noted in the hydrophobic (PP/SiO₂)/LCP system was due mainly to the increases in the shear rate and η_m . Through the optimum adjustments made by varying shear rate and content of silica particles, fibrillar LCP structures could be obtained.

CONCLUSIONS

A new method of enhancing the fibrillation of LCP in an LCP/thermoplastic blend system was successfully demonstrated employing the hydrodynamic theory. The approach was designed based on the equation of Ca . While the equation is commonly used to explain the fibrillation of LCP in binary LCP/thermoplastic systems, no attempt has yet been made to correlate fibrillation of LCP in a ternary system such as the LCP/filled PP blend. Our new concept is the addition of fillers as a viscosity thickening agent to modify the viscosity of the matrix. At the same time, the environment for LCP fibrillation has been modified. Through this work, a better understanding of LCP/filled thermoplastic ternary blends has been revealed both theoretically and experimentally. Specifically, silica fillers were incorporated in an LCP/PP composite and this third phase was noticed to improve the LCP fibril formation. The addition of more fillers could increase

the aspect ratio of the LCP fibril so that the development of an advanced *in situ* hybrid composite could be realized.

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