Comparative Rheological Studies on Jute-Fiber- and Glass-Fiber-Filled Polypropylene Composite Melts

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SYNOPSIS

An experimental study of the melt rheological properties of composites of isotactic polypropylene (IPP) filled with chopped jute-fibers (JF) and glass-fiber (GF) is reported. Fiber concentration is 5–20 wt %. Variation of shear stress with shear rate at constant temperature indicates increase in shear stress with shear rate and fiber loading. Shear stress values for IPP/GF melts are higher at the same shear rate and temperature. Variation of melt viscosity and melt elasticity with fiber concentration and shear stress or shear rate are also illustrated. Melt viscosity increases with fiber loading, while melt elasticity parameters such as die-swell and first normal stress difference decrease for both systems. For IPP/GF composite melts the viscosity values are higher, the die-swell and first normal stress difference values are lower compared to IPP/JF composites melts. Processing temperature of the filled IPP composites increase with respect to the nonfilled polymer, being a little higher in case of IPP/GF composites. © 1992 John Wiley & Sons, Inc.

INTRODUCTION

The mechanics and properties of composites have received considerable attention in the present literature.[1-4] Few studies of the rheological properties of fiber-filled polymer melts have appeared.[5-8] With the exception of Maiti and Hassan,[7] all these investigations deal solely with glass-fiber-filled composite melts.

There is a good potential for the use of natural fibers as reinforcing fillers in thermoplastics. The main advantage of these fibers is their low cost, low density, and resistance to breakage during processing.[9] There are many examples in the literature of the use of cellulosic fibers in their natural condition such as sisal, coir, jute, banana, sunhem, palm, flax, cotton, and paper for reinforcement of different thermosetting and thermoplastic resins such as phenol-formaldehyde, unsaturated polyester, epoxy, polyethylene, natural rubber, etc. Roe and Ansel[10] have made a comparison of the reinforcing potential of jute with respect to glass fiber. Although tensile strength and Young's modulus of jute are lower than that of glass, the specific modulus of jute fiber is superior to that of glass, and on a modulus per cost basis, jute is far more superior.[11-13] Also the specific strength per unit cost of jute approaches that of glass. Cost comparisons have been made by Kumar.[14] Therefore, where high strength is not a priority, jute may be used to fully or partially replace costly glass fiber.

IPP is a very useful commercial polymer containing little or no unsaturation. Its outstanding properties include: lowest density among commercial thermoplastics, exceptional flex life, good surface hardness and scratch resistance, very good abrasion resistance, steam sterilizability, and excellent electrical properties.[15]

In the present work we report the comparative study on the melt rheological properties of IPP/JF and IPP/GF composites. A piston-type capillary rheometer was used to obtain shear rate, shear stress data, melt viscosity, and melt elasticity parameters at various fiber loadings and at various temperatures.

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EXPERIMENTAL

Materials

IPP used was Koylene M 3030 (M.F.I. 3 and density 0.89 g/cm^3) supplied by Indian Petrochemicals Corporation Ltd., India. Glass fiber (Grade AA 408, $1/d$ = 330, l = 4.7 mm) was supplied by Owens Corning of U.S.A. Jute fiber (Grade W-2, white, 1 $\simeq$ 5 mm) was supplied by Jute Technological Research Laboratory, Calcutta, India.

Compounding

Glass fibers were dried at 100°C for 24 h in an air oven, and jute fibers were dried at 80°C for 48 h in a vacuum oven to expel moisture. Dried glass fibers were mixed with IPP granules and fed to a single-screw extruder (Windsor SX 30 type) to prepare IPP/GF composites containing 5, 10, and 20 wt % fibers. Temperatures maintained were 473, 483, and 493 K at feed, compression, and die zones. Screw rpm was maintained at 18 for both cases. IPP/JF composites containing 5, 10, and 20 wt % of jute fibers were prepared the same way. The extrudates were granulated. These granules were used for the rheological studies. Melt flow properties of IPP/GF and IPP/JF composites were measured on a high-pressure capillary rheometre, Rheograph-2001 of Messers, GOTTFERT, at temperatures of 483, 503, and 523 K.

RESULTS AND DISCUSSION

Shear Stress–Shear Rate Curves

Figures 1, 2, and 3 show representative plots of shear stress–shear rate data at fixed temperatures of 483, 503, and 523 K, respectively.

Shear stress at the wall, τ_w of the capillary viscometer was calculated using the following formula[16]:

$$\tau_w = \frac{F}{4A_p(L/D)} \tag{1}$$

where F is weight force, A_p is area of the plunger, L and D are the length and diameter of the capillary, respectively.

The apparent shear rate, γ_a was calculated using the following equation[16]:

$$\gamma_a = \frac{2}{15}\frac{V_{xh}d_p^2}{D^3} \tag{2}$$

where V_{xh} is crosshead speed and d_p is diameter of

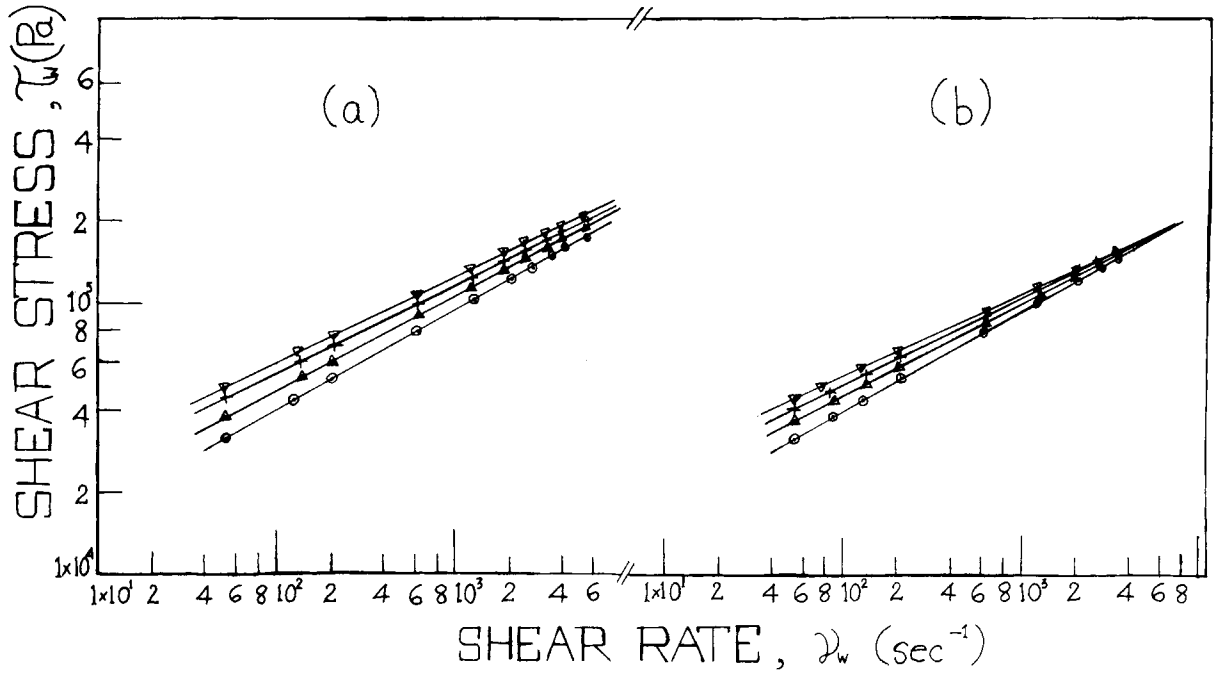

Figure 1 Shear stress (τ_w) vs. shear rate (γ_w) curves at 483 K. (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

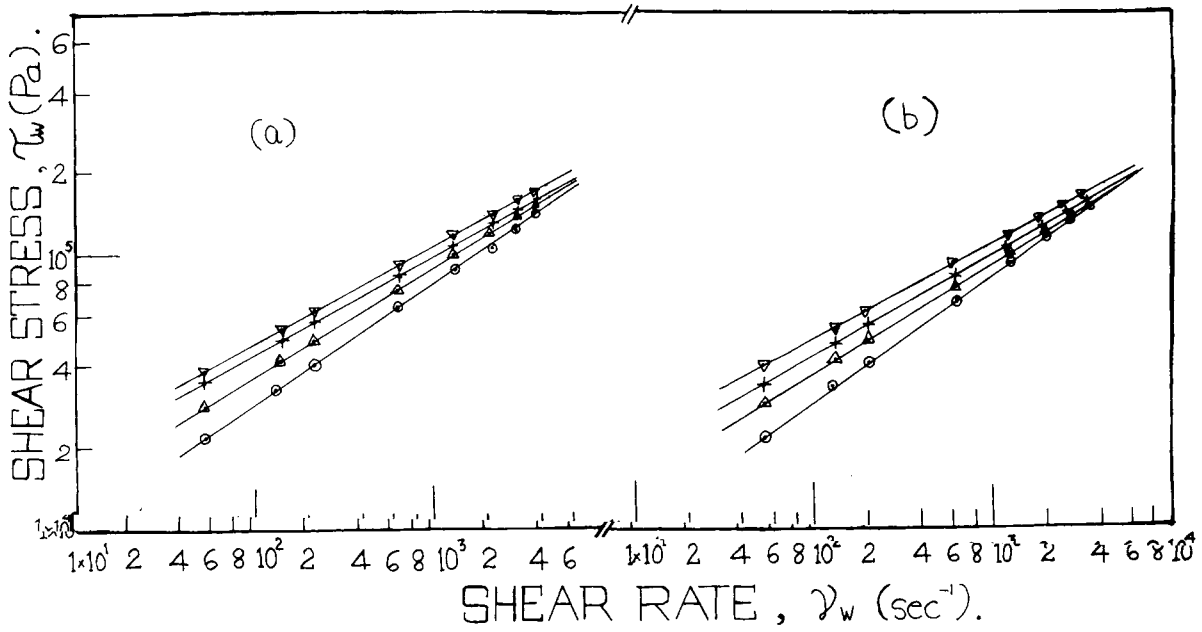

Figure 2 Shear stress (τ_w) vs. shear rate (γ_w) curves at 503 K. (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

the plunger, respectively. From the apparent shear rate values, the shear rate at the wall, γ_w, of the capillary was calculated after applying the Rabinowitsch correction.[17]

$$\gamma_w = \left(\frac{3n' + 1}{4n'}\right)\gamma_a \tag{3}$$

where n' is the flow behavior index obtained as the

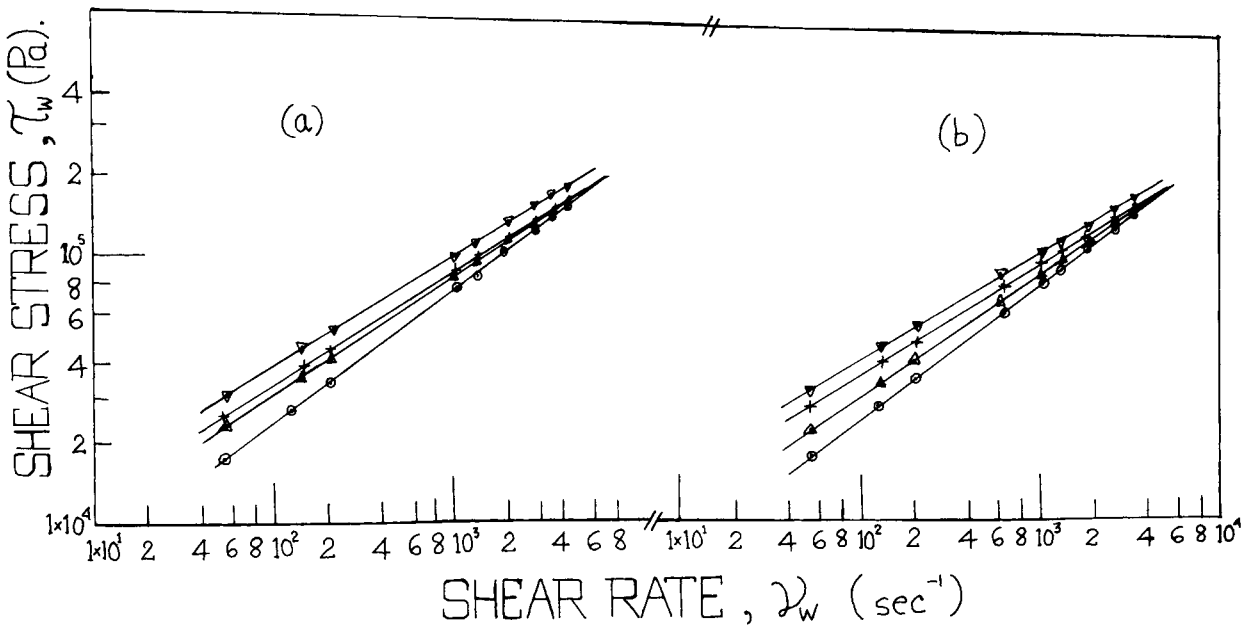

Figure 3 Shear stress (τ_w) vs. shear rate (γ_w) curves at 523 K. (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

Table I Values of Power Law Exponent for IPP/JF and IPP/GF Composites

Sample	Fiber (%)	N		
		483 K	503 K	523 K
IPP	0	0.38	0.44	0.49
IPP/JF	5	0.35	0.39	0.44
IPP/JF	10	0.33	0.37	0.42
IPP/JF	20	0.31	0.35	0.39
IPP/GF	5	0.34	0.38	0.43
IPP/GF	10	0.32	0.36	0.40
IPP/GF	20	0.30	0.33	0.38

slope of the linear plot of log τ_w versus log γ_a (curves not shown).

The melt viscosity was evaluated as

$$\eta = \frac{\tau_w}{\gamma_w} \tag{4}$$

The dependence of log τ_w versus log γ_w for both IPP/JF and IPP/GF composite melts are linear in the range of shear rates studied (10^1–10^4 s^{-1}). Same observation was noted by other authors for different fiber-filled composite systems.[7,9,18]

At the same shear rate and temperature, the shear stress was higher for IPP/GF composite melts compared to IPP/JF composite melts. This is in tune with the experimental observation (Figs. 7–9).

The nature of the curves indicate that power law behavior is followed by both IPP/JF and IPP/GF composite melts at all three temperatures of study. The values of power law flow indices n of the power law relation[7]

$$\tau_w = K\gamma_w^n \tag{5}$$

obtained from the slopes of the plots of log τ_w vs. log γ_w are shown in Table I.

For pure IPP melts, as well as for both IPP/JF and IPP/GF composite melts, the n values were less than unity showing pseudoplastic nature of the melts. For a particular filler loading the values of n increased with increase in temperature, whereas with increase in filler content n decreased.[7] The n values were lower for IPP/GF composite melts compared to IPP/JF composite melts indicating their higher pseudoplasticity.

It is now clear that the shear rates tested in the present study were not low enough to exhibit yield stress of the filled polymers (observed in earlier studies[21,22]). This yield stress is exhibited due to the formation of an interparticle network, which becomes quite strong at high filler loadings.[24–28] Yield stress is most pronounced at very low shear rates, e.g., 10^{-2} to 10^0 s^{-1}, and with very fine particle fillers. The finer the filler, the larger the ability of network formation and stronger the network exhibiting higher yield values. All systems exhibiting yield stress have filler diameters below 0.5 μm.[25] Apart from the high shear rates of study, the other cause for not observing any yield stress in the present work is probably due to the large size of both the jute and glass fibers. The absence of yield stress was also reported with large particles of wood flour,[7] glass-sphere,[29] and glass as well as for cellulosic fibers.[8,18]

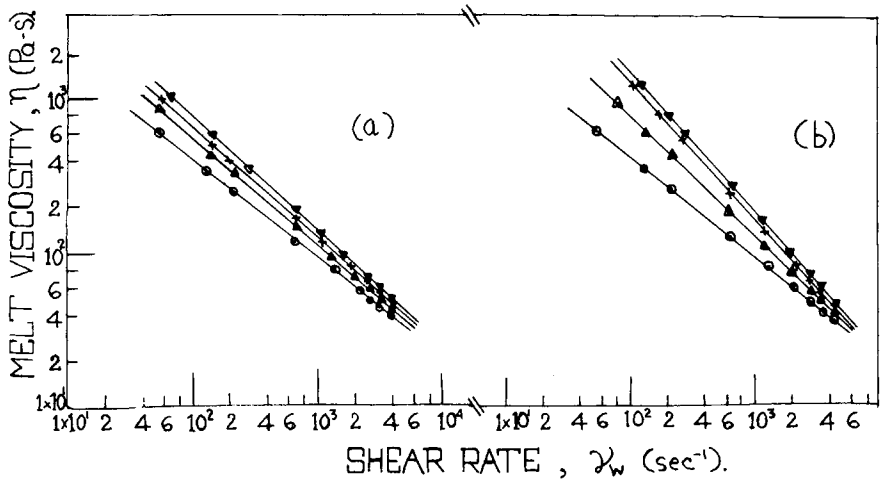

Figure 4 Variation of melt viscosity (η) with shear rate (γ_w) at 483 K for (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

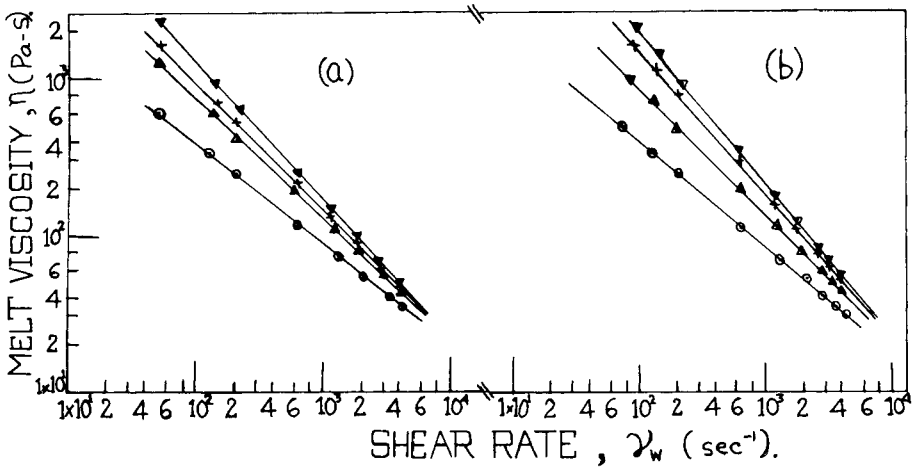

Figure 5 Variation of melt viscosity (η) with shear rate (γ_w) at 503 K for (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

Composite Melt Viscosity

Viscosity shear rate curves for IPP/JF and IPP/GF composite melts with varying levels of fiber loading and at the three different temperatures of study are shown in Figures 4–6. It is observed that in all cases the viscosity is increased by the presence of fibers and decreased with increasing shear rate. The glass fiber systems studied are the same as those of Crowson et al.,[19] and our results are in agreement with the above. The same trend was reported in other fiber-filled polymer systems as well.[9]

At low shear rates the presence of fibers causes an appreciable increase in viscosity, but the viscosities converge to a similar value at a shear rate in the range of 10^4 s^{-1}. This tendency of η vs. γ_w plot is exhibited by both the systems at all temperatures and have also been observed for other fiber-filled polymers.[6-8,20]

The plausible cause for an increase in viscosity of the fiber-filled polymer melts is the fact that incorporation of short fibers introduces discontinuity in the IPP matrix, which increases with increase in fiber content. The presence of these discontinuities provide obstruction to flow of the polymer melt, thus causing a rise in viscosity. This view is supported by other authors.[7] Another cause for an increase in viscosity may be stated as follows: In a melt (fluid) when a filler or additive (insoluble) is incorporated, the viscosity increase due to increased friction to-

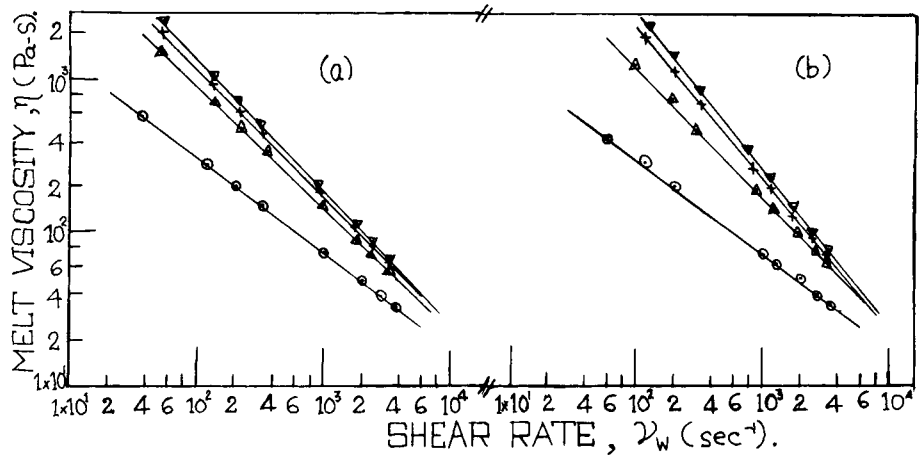

Figure 6 Variation of melt viscosity (η) with shear rate (γ_w) at 523 K for (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

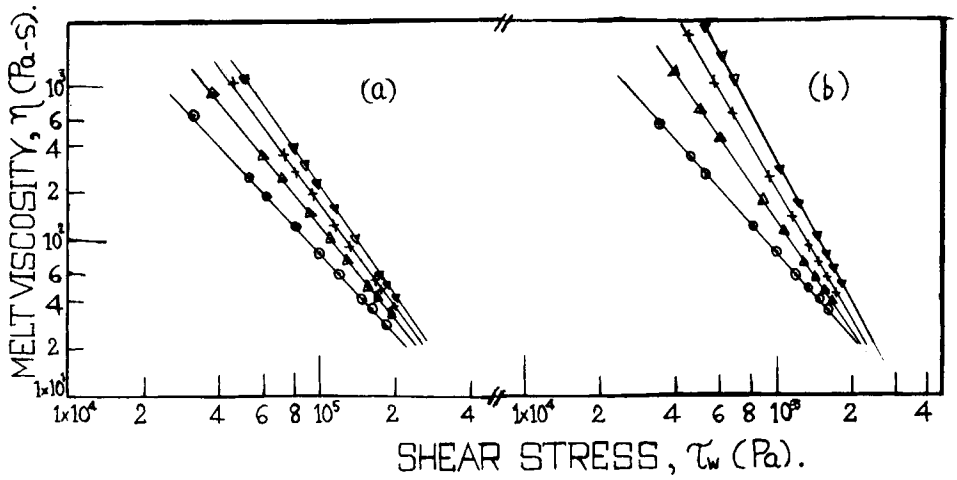

Figure 7 Variation of melt viscosity (η) with shear stress (τ_w) at 483 K: (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

ward the flow of the melt takes place. The greater is the filler–polymer interaction, the greater the resistance to flow. As adhesion of glass fiber to IPP matrix is better compared to that of jute fiber to IPP matrix, IPP/GF's rise in viscosity is higher at the same shear rate and temperature.

Earlier studies[9,19] have shown that filled polymers exhibit a constant η value in the flow curves when tested at very low shear rates (10^{-1}–10^{-2} s^{-1}). This has not been observed in our case because of the very high shear rates of study (10^{-1}–10^{-4} s^{-1}).

Figures 7–9 presents melt viscosity–shear stress plots of IPP/JF and IPP/GF composite melts at the three temperatures of study. The melt viscosity shows a linear decrease with increasing shear stress at any given temperature for both systems. The same tendency was observed by other authors for filled IPP systems.[7,18] The effect of the presence of fibers on viscosity increases with increase in temperature for all cases, which is quite evident from the nature of curves 7 to 9. The power law relation for the pseudoplastic fluids obeying Eq. (5) is

$$\eta = K^{1/n}(\tau_w)^{(n-1)/n} \tag{6}$$

The slopes of the η vs. τ_w curves are in good agreement with Eq. (6), and the values of n are quite consistent with those shown in Table I.

The variation of melt viscosities of composites (η) with fiber content in weight percent (ϕ_F) at 483,

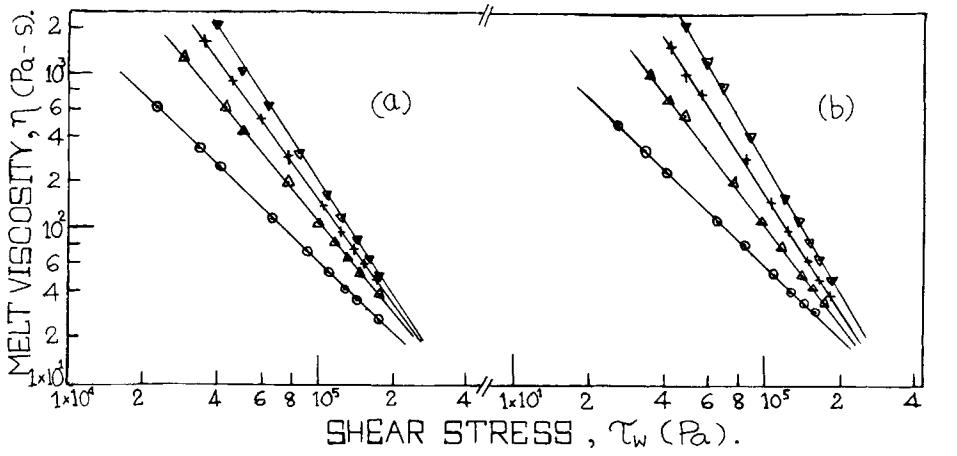

Figure 8 Variation of melt viscosity (η) with shear stress (τ_w) at 503 K: (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

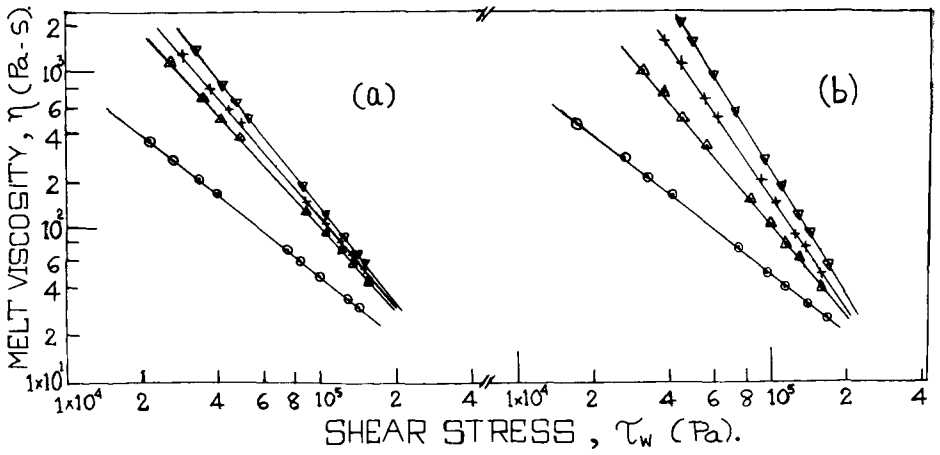

Figure 9 Variation of melt viscosity (η) with shear stress (τ_w) at 523 K: (a) (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20% and (b) (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

503, and 523 K and at a fixed shear stress of 30 kPa are shown in Figures 10 and 11. The IPP/GF composite melt viscosity increases 12- to 27-fold and that of IPP/JF increases by 5- to 8-fold compared to virgin IPP, depending on temperature.

Effect of Temperature on Viscous Property

In order to study the temperature dependence of viscosity, plots of log η vs. $1/T$ are plotted as shown in Figures 12 and 13 for IPP/JF and IPP/GF composite melts, respectively. The shear stress was fixed at 100 kPa. The slope of the curves for the filled systems are much lower than the slope for pure IPP melt and increased inappreciably with fiber content. This indicates that the melt viscosity of the composites is less temperature sensitive than that of the nonfilled polymer. This is true for both IPP/JF and IPP/GF systems. Such an observation is in good agreement with other reported results.[7,23,30]

Thus, addition of fibers to a polymer acts in its influence on viscosity like a reduction in temperature

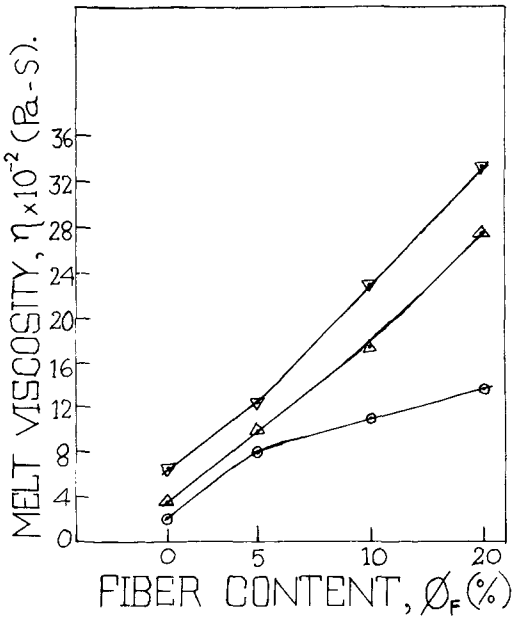

Figure 10 Plots of melt viscosity (η) with fiber content (ϕ_F) for IPP/JF composites at: (▽) 483 K; (△) 503 K; (○) 523 K at shear stress 30 kPa.

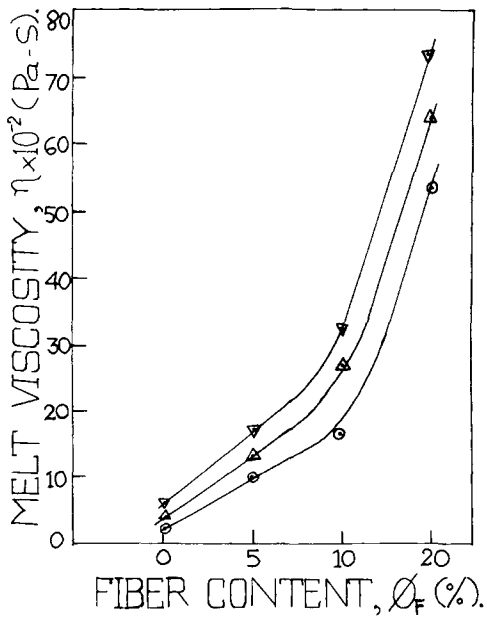

Figure 11 Plots of melt viscosity (η) with fiber content (ϕ_F) for IPP/GF composites at: (▽) 483 K; (△) 503 K; (○) 523 K at shear stress 30 kPa.

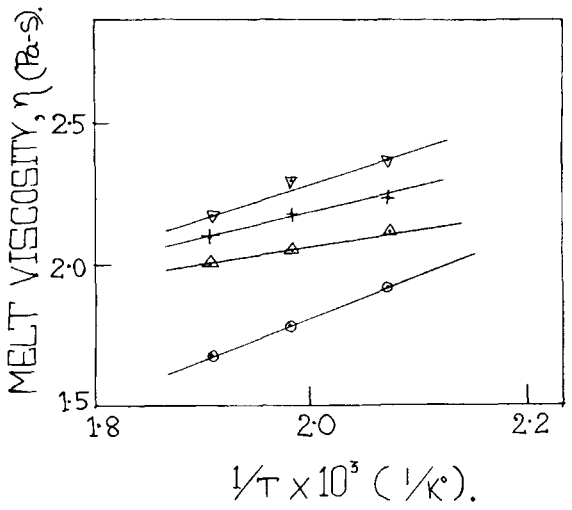

Figure 12 Plots of melt viscosity (η) vs. $1/T$ for IPP/JF composites at a constant shear stress 100 kPa: (○) IPP; (△) IPP/JF 5%; (+) IPP/JF 10%; (▽) IPP/JF 20%.

of the melt,[9,18] as both cause a rise in viscosity. This supports the idea that the enhanced viscosity is due to increased viscous energy dissipation in the matrix and that particle–particle interaction is not significant.[31-36]

Elasticity Parameters of the Melt

The present work evaluates the melt elasticity parameters such as die-swell ratio, first normal stress

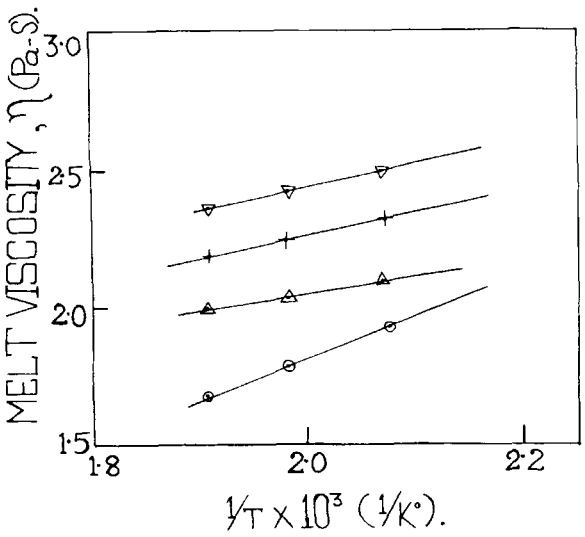

Figure 13 Plots of melt viscosity (η) vs. $1/T$ for IPP/GF composites at a constant shear stress 100 kPa: (○) IPP; (△) IPP/GF 5%; (+) IPP/GF 10%; (▽) IPP/GF 20%.

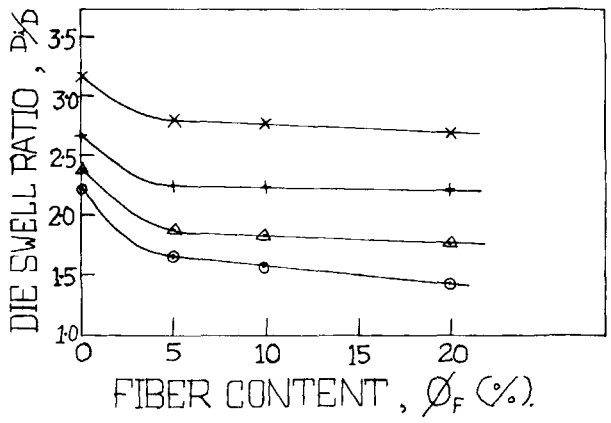

Figure 14 Variation of die-swell ratio (D_i/D) with JF content at 483 K and at constant shear stresses: (○) 80 kPa; (△) 100 kPa; (+) 130 kPa; (×) 160 kPa.

difference, and apparent shear modulus of IPP/JF and IPP/GF composite melts at a constant temperature of 483 K. The die-swell ratio is given by D_i/D where D_i and D are the diameters of the extrudate and the die, respectively. The plots of D_i/D versus fiber content (ϕ_F, wt %) at different shear stresses (160, 130, 100, and 80 kPa) are shown in Figures 14 and 15. It is observed that the die-swell ratio of virgin IPP is much higher than that of both the filled systems. This observation of melt elasticity decreases in filled polymers is in good agreement with other published results.[7,18,25,30] At any given shear stress, the decrease in elasticity with increase in fiber content was marginal in regions ϕ_F = 5%. The total drop in die-swell varies between 11 and 36% for IPP/JF system and between 32 and 47%

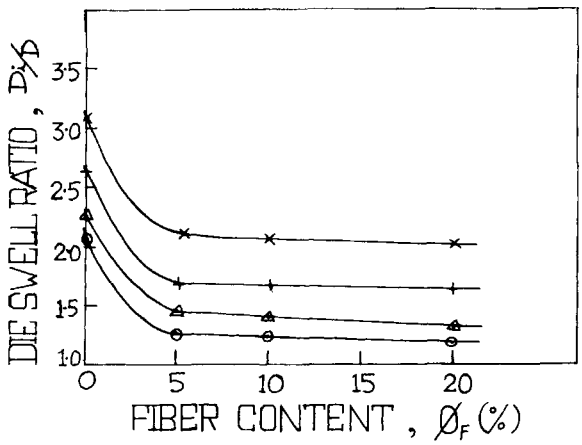

Figure 15 Variation of die-swell ratio (D_i/D) with GF content at 483 K and at constant shear stresses: (○) 80 kPa; (△) 100 kPa; (+) 130 kPa; (×) 160 kPa.

for IPP/GF system, based on nonfilled IPP depending on shear stress value. The higher die-swell values of pure IPP compared to both the filled systems was as a result of its low apparent shear modulus, i.e., higher recoverable shear strain (Figs. 18 and 19). These observations for D_i/D may also be justified by the presence of discontinuities in the polymer matrix mentioned before. Decrease in melt elasticity in these composites may be attributed to this phenomenon, which would cause discontinuity in stress transfer for elastic recovery or deformation.

The D_i/D values for IPP/JF composites are higher than that of IPP/GF composites. The plausible cause for this may be that some energy from elastic deformation or recovery stresses is being utilized in debonding of the glass fiber–IPP matrix, the bond strength of which compared to IPP–jute fiber is much higher.

Decrease in the melt elasticity of IPP in the presence of fibers aids in processing safety by increasing the critical shear stress for melt fracture. This will give smooth surface of the extrudates of the composites.

The first normal stress difference, $N_1 = (\tau_{11} - \tau_{22})$, was determined using Tanner's equation.[37]

$$N_1 = 2\tau_w[2(D_i/D)^6 - 2]^{1/2} \qquad (7)$$

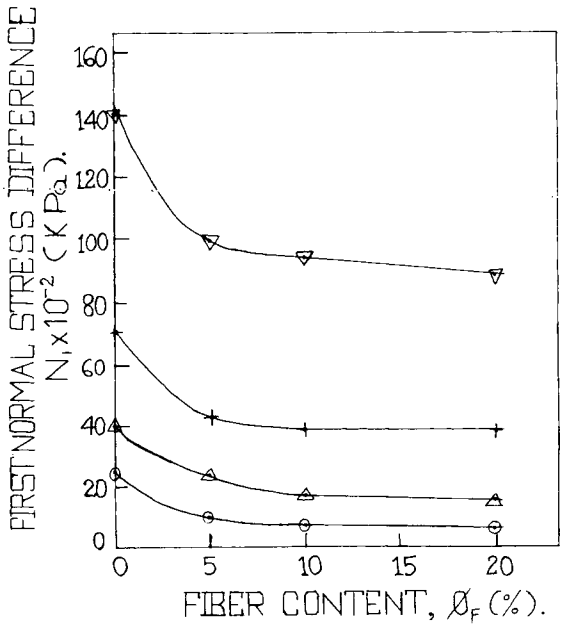

Figure 16 Variation of first normal stress difference (N_1) with JF content (ϕ_F, wt %) at 483 K and at constant shear stresses: (○) 80 kPa; (△) 100 kPa; (+) 130 kPa; (▽) 160 kPa.

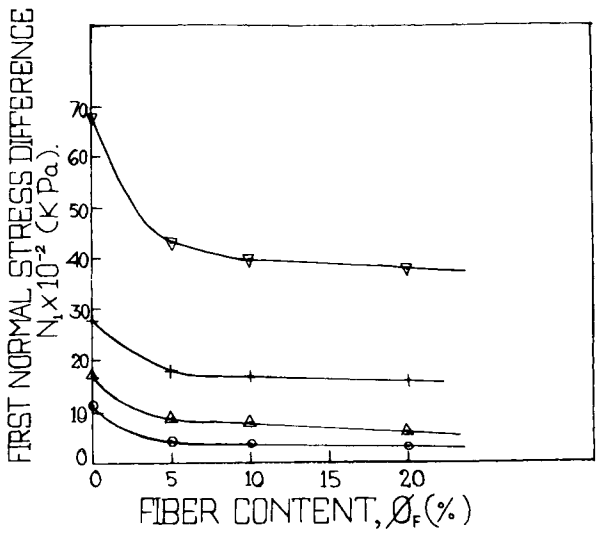

Figure 17 Variation of first normal stress differences (N_1) with GF content (ϕ_F, wt %) at 483 K and at constant shear stresses: (○) 80 kPa, (△) 100 kPa; (+) 130 kPa; (▽) 160 kPa.

Plots of N_1 vs. ϕ_F are shown in Figures 16 and 17 for IPP/JF and IPP/GF systems, respectively, at different shear stresses (160, 130, 100, and 80 kPa) and at a fixed temperature of 483 K. Drop in N_1 values for both systems with increasing fiber content is observed. This observation is supported by other authors.[7,18] The N_1 values of IPP/JF composite melts were found to be about twice that of IPP/GF composite melts.

The apparent shear modulus (G) and recoverable shear strain (γ_R) were calculated from[23,37]

$$\gamma_R = \frac{N_1}{2\tau_w} \qquad (8)$$

$$G = \frac{\tau_w}{\gamma_R} \qquad (9)$$

Plots of γ_R against γ_w and G against γ_w are shown in Figures 18–21 at a temperature of 483 K for IPP/JF and IPP/GF, respectively. It is seen that recoverable shear strain γ_R increases and apparent shear modulus G decreases with increasing shear rate, whereas for any fixed shear rate γ_R decreases and G increases with increase in fiber concentration. These observations are true for both the systems, as expected. The same observation was noted by other authors.[7] The γ_R values of IPP/GF melts are much lower compared to IPP/JF melts resulting in their higher shear modulus values, which is as expected.

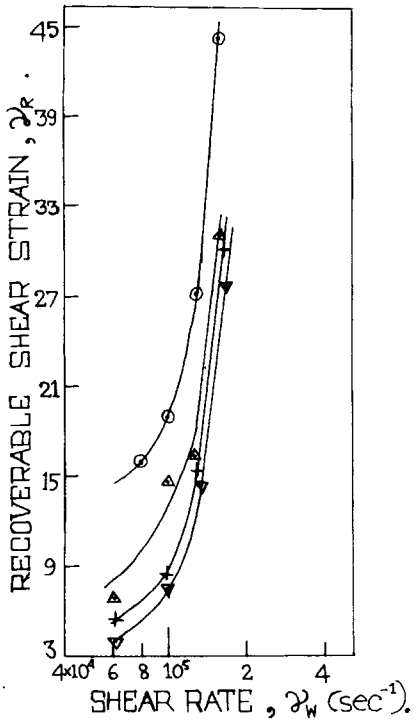

Figure 18 Recoverable shear strain (γ_R) vs. shear rate (γ_w) plots at constant JF concentrations: (○) 0%; (△) 5%; (+) 10%; (▽) 20%.

CONCLUSION

Both IPP/JF and IPP/GF melts are pseudoplastic in nature. They follow the power law relationship between shear stress and shear rate over the entire range of shear rates (10^1–10^4 s^{-1}) and at all fiber concentrations. IPP/GF composite melts is more pseudoplastic in nature compared to IPP/JF melts. The flow indices (n) values of IPP/GF melts varied from 0.30 to 0.43 and that for IPP/JF varied from 0.31 to 0.44. For pure IPP the range is 0.38–0.49

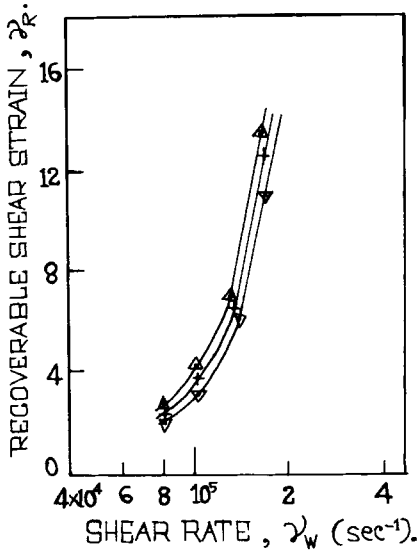

Figure 19 Recoverable shear strain (γ_R) vs. shear rate (γ_w) plots at constant GF concentrations: (△) 5%; (+) 10%; (▽) 20%.

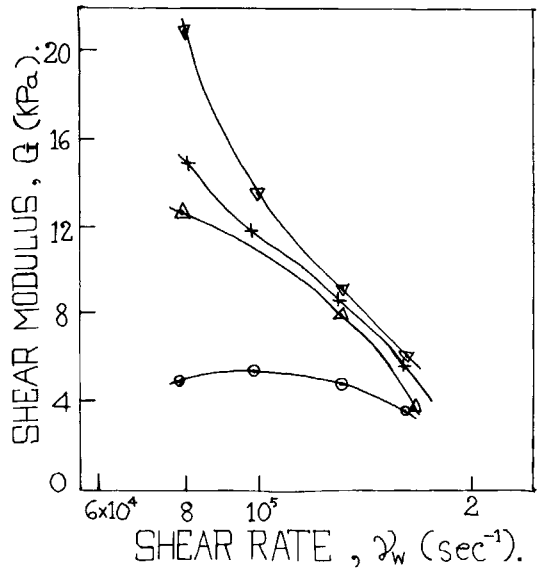

Figure 20 Plots of apparent shear modulus (G) vs. shear rate (γ_w) at constant JF concentrations: (○) 0%; (△) 5%; (+) 10%; (▽) 20%.

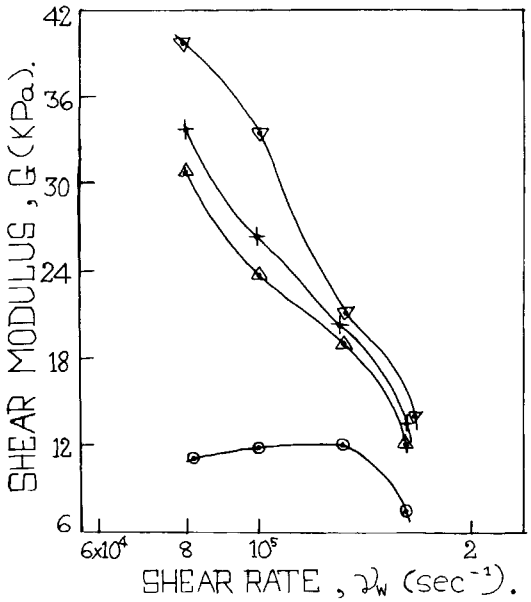

Figure 21 Plots of apparent shear modulus (G) vs. shear rate (γ_w) at constant GF concentrations: (○) 0%; (△) 5%; (+) 10%; (▽) 20%.

depending on temperature. At any given temperature pseudoplasticity decreases, with increasing fiber content and increases with temperature for both cases. At any given temperature melt viscosity of both filled systems increase with increasing fiber content and decrease with increasing shear rate, becoming almost equal at high shear rates. This similarity in viscosity values at high shear rates, for filled and unfilled thermoplastics, is an important factor in explaining the successful exploitation of these materials in injection molding technology, since very little additional power will be required to mold the filled materials.

Die-swell ratio, first normal stress difference, and shear modulus decrease while recoverable shear strain increases with increasing fiber concentration for both systems. The die-swell ratio, first normal stress difference, and recoverable shear strain of IPP/GF composite melts are lower compared to IPP/JF composite melts, whereas its shear modulus values are higher.

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