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### RHEOLOGICAL PROPERTIES OF ISOTACTIC POLYPROPYLENE/ALUMINA COMPOSITES

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## RHEOLOGICAL PROPERTIES OF ISOTACTIC POLYPROPYLENE/ALUMINA COMPOSITES

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*Melt rheological properties of alumina-filled isotactic polypropylene composites have been estimated at 0–22.2 vol% alumina at 493 K. The composite melts obeyed power law in shear stress-shear rate relations and were shear thinning. Apparent melt viscosity decreased initially up to 5.8% by volume alumina and increased with further increase in filler concentration. Melt elasticity decreased marginally upto  $\phi_F=5.8\%$  whereas the data increased upon further increase in alumina content. Surface modification of alumina particles by a titanate coupling agent LICA 38 brought about interesting modification of the rheological properties through a plasticizing/lubricating function and/or blocking of the surface groups of  $Al_2O_3$  particles.*

**Keywords:** Power law model, shear-thinning, apparent melt viscosity, first normal stress difference

### INTRODUCTION

Property modification of polymers by incorporation of various additives is a well known practice to widen the scope of application of the polymer [1–7]. Fillers constitute the bulk of these additives and inorganic particulates comprise the major volume amongst particulates. Properties that are gainfully enhanced are modulus, hardness, high temperature creep resistance, mold shrinkage resistance, etc., which are often accompanied by lower material cost, faster part fabrication, and less rejects.

In order to achieve the best combination of properties, the filler surface needs to be modified by a suitable coupling agent [4, 6, 8].

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Surface modified fillers alter compounding and processing of the composites. The rheological characterization of the systems are necessary to solve polymer processing problems. The melt viscosity and elasticity may decrease or increase with increase in filler concentration, depending on its shape and interaction with the matrix [9, 12]. In isotactic polypropylene filled with wood flour and  $\text{CaCO}_3$ , the melt viscosity increased and melt elasticity decreased with increase in filler concentration [9, 13]. The systems followed power law in their flow behaviors.

This article communicates a study on the melt rheological properties of alumina filled isotactic polypropylene. Rheological parameters shear stress, shear rate, melt viscosity and melt elasticity were generated on a piston-type capillary rheometer at alumina concentrations 0–22.2 vol% and at 493 K. The effects of filler surface treatment with a titanate coupling agent LICA 38 on the rheological behavior have also been elucidated.

## EXPERIMENTAL

### Materials

Isotactic polypropylene (i-pp) Koylene M0030 (density  $0.9 \text{ cm}^{-3}$ , MFI 10) obtained from M/s Indian Petrochemicals Corporation Ltd., Gujrat, was used. Commercial grade alumina (density  $2.21 \text{ g cm}^{-3}$ , average particle diameter  $69 \mu\text{m}$ ) was obtained from M/s Qualigens Fine Chemicals Ltd., Bombay and used. The liquid titanate coupling agent used to surface modify alumina was LICA 38 obtained from M/s Kenrich Petrochemicals Ltd., Bayonne, New Jersey, USA. Chemically LICA 38 is neopentyl (diallyl) oxy,tri(dioctyl) pyrophosphato titante [8].

### Alumina Surface Modification

To 0.4 g LICA 38 dissolved in 100 mL of moisture-free toluene 100 g  $\text{Al}_2\text{O}_3$  was added in a high-speed laboratory mixer (Sumeet, Model-2L) for 30 min. The coated alumina was dried at 393 K for 6 h to expel toluene. The hydrophobicity acquired by alumina was confirmed by a floating test on water. When dropped on water, the coated particles spread and floated on the water surface while the uncoated powders lumped and sank immediately. This technique of filler surface modification was employed in other works, too [4, 14, 15].

### Compounding

Vacuum-dried alumina (both untreated and treated) was mixed with i-PP on a two-roll mill (Richardson and Cruddas Ltd., Model-2-18  $\times$  8)

at  $433 \pm 2$  K for 10 min to make primary thin sheets with 0–22.2 vol% (0–70 phr) alumina. Squares 1 cm in sides cut from these sheets were fed to the rheometer for data generation.

## Measurements

A Rheograph-2001 (Gottfert, Germany) of cylindrical die with diameter 1 mm and L/D ratio 30 was used to generate rheological data at 493 K. The initial melting time of polymer was 2 min and the comparison interval was 8 s. The measurements were performed in the shear rate range 25–5000 ( $s^{-1}$ ). Rheological properties, apparent melt viscosity and melt elasticity, were calculated using RABKOR program (Version 9.3) following the instrument manual [16, 17].

## RESULTS AND DISCUSSION

### Shear Stress-Shear Rate Curves

The variations of shear stress-shear rate were measured at 493 K and at varying pressures. The apparent shear rates  $\gamma_{app}$  were calculated following Equation (1) [18]:

$$\gamma_{app} = \frac{4Q}{\pi R^3} (s^{-1}) \quad (1)$$

where R is the radius of the capillary die (cm) and Q the volumetric flow rate ( $cm^3/s$ ), which in turn is given as:

$$Q = \frac{AS}{t} = AV (cm^3/s) \quad (2)$$

Here, V is the piston speed ( $cm/s$ ), A the piston free area ( $cm^2$ ), t the time (s), and S the piston path (cm).

From the  $\gamma_{app}$  values the wall shear rate values  $\gamma_w$ , were computed by applying Rabinowitsch-Weissenberg correction [19, 20]:

$$\gamma_w = \left( \frac{3n' + 1}{4n'} \right) \gamma_{app} (s^{-1}) \quad (3)$$

where  $n'$  is the flow behavior index calculated as the slopes of  $\log \tau_w$  vs.  $\log \gamma_{app}$  plots (not shown).

The apparent shear stress  $\tau_{w(app)}$  was estimated from equation (4) [16]:

$$\tau_{w(app)} = \frac{PD}{4L} \times 30 \text{ (Pa)} \quad (4)$$

where  $P$  is the pressure applied on the piston,  $L$  the capillary length (cm), and  $D$  the capillary diameter (cm). The wall shear stress  $\tau_w$  was determined by applying Ryder-Bagley correction [18, 19]:

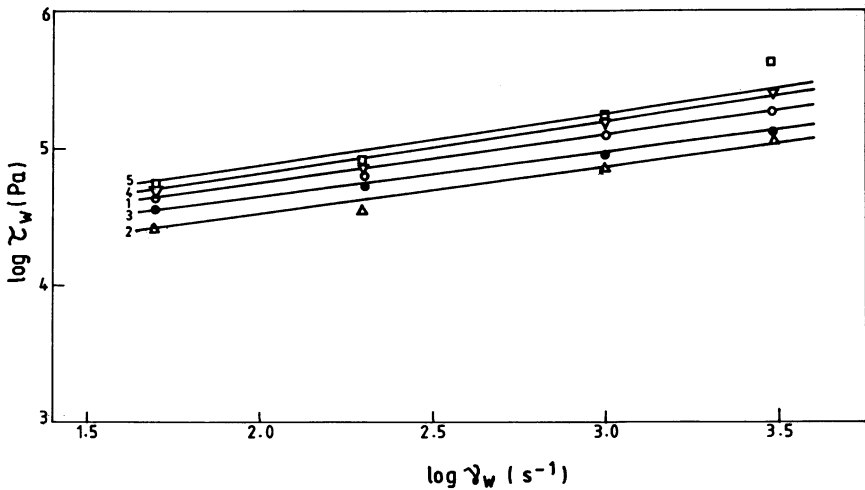
$$\tau_w = \frac{P}{2\left(\frac{L}{R} + e\right)} \quad (\text{Pa}) \quad (5)$$

where  $e$  is the negative of the slope of the  $P$ - $(L/R)$  plot in a range of shear rates.

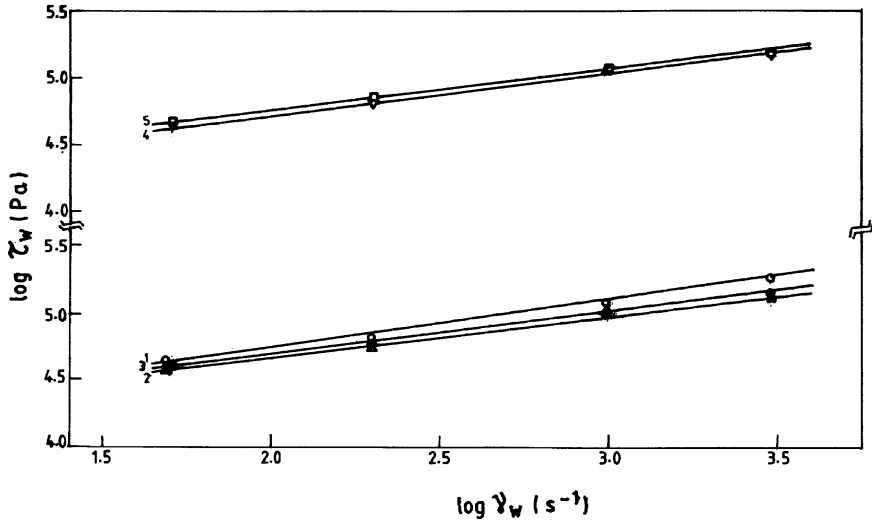
The apparent melt viscosity  $\eta_{\text{app}}$  were determined from the shear stress and shear rate data [19, 20]:

$$\eta_{\text{app}} = \frac{\tau_w}{\dot{\gamma}_w} \quad (\text{Pa.s}) \quad (6)$$

Variations of  $\log \tau_w$  vs.  $\log \dot{\gamma}_w$  in the i-pp/ $\text{Al}_2\text{O}_3$  and i-pp/ $\text{Al}_2\text{O}_3$ /LICA 38 systems at 493 K were linear in the shear rates studied, Figures 1 and 2, indicating that the melts obeyed power law model in their flow curves as were also reported in other works [9, 13]. In the i-pp/ $\text{Al}_2\text{O}_3$  composites up to alumina volume % ( $\phi_F$ ) 5.8, the  $\log \tau_w - \log \dot{\gamma}_w$  curve moves downward compared to i-PP. Beyond  $\phi_F = 5.8\%$  the curve moves upward and at  $\phi_F > 16.9\%$  the curve lies upper than that of i-PP. It implies that at low volumes of  $\text{Al}_2\text{O}_3$  up to 5.8% at a constant  $\dot{\gamma}_w$  the



**FIGURE 1** Variations of  $\log$  shear stress ( $\tau_w$ ) vs.  $\log$  shear rate ( $\dot{\gamma}_w$ ) of i-PP/ $\text{Al}_2\text{O}_3$  composites at 493 K: (○) i-PP; (Δ) i-PP/ $\text{Al}_2\text{O}_3$  5.8%; (●) i-PP/ $\text{Al}_2\text{O}_3$  12.5%; (▽) i-PP/ $\text{Al}_2\text{O}_3$  16.9%; (□) i-PP/ $\text{Al}_2\text{O}_3$  22.2%.



**FIGURE 2** Log  $\tau_w$ -log  $\gamma_w$  plots in i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 composites at 493 K. Descriptions of the curves are as in Figure 1.

composite exhibits lower shear stress and upon further increase in  $\phi_F$  the system shows higher shear stress. Higher shear stresses with increase in filler concentration at constant shear rate were observed in previous works [9, 13]. In i-PP/Al<sub>2</sub>O<sub>3</sub> composites at low  $\phi_F$  values a flow favoring orientation of acicular Al<sub>2</sub>O<sub>3</sub> particles may give rise to this lowering of shear stress (4). Similar results were observed in i-PP/Kaolin composites as well (11). In the i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 systems the flow curves also exhibit similar characteristics as their counterparts; at a fixed shear rate the extent of decrease and subsequent increase of shear stress is, however, less due to the modifying effect of LICA 38 through bonding of i-PP with Al<sub>2</sub>O<sub>3</sub>.

Table 1 presents values of power law indices  $n$  of the power law model [Equation (7)] obtained as the

$$\tau_w = K\gamma_w^n \quad (7)$$

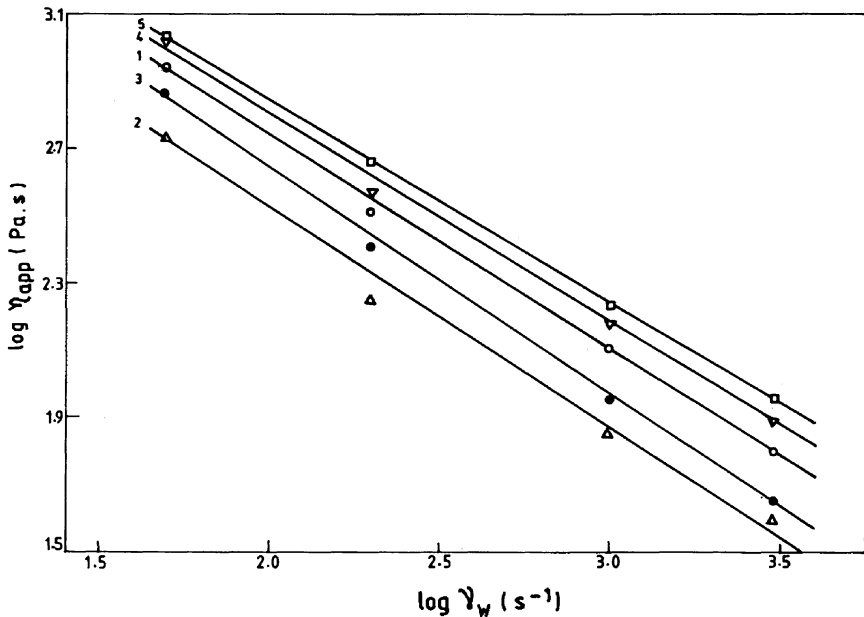
slopes of log  $\tau_w$  v.s log  $\gamma_w$  plots in Figure 1 and 2. The  $n$  values were less than unity, being  $0.348 \pm 0.018$  for i-PP/Al<sub>2</sub>O<sub>3</sub> and  $0.326 \pm 0.025$  for i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 systems. This implies that the composite melts were pseudoplastic common to thermoplastic systems [11, 13, 21].

At low shear rates, viz.  $10^{-2}$ – $10^0$  (s<sup>-1</sup>), some particle-filled composites exhibited yield stress [7, 22, 23]. These systems contained

**TABLE 1** Values of Power Law Index  $n$  for i-PP/Al<sub>2</sub>O<sub>3</sub> and i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 composites

$\phi_F$ (%)	Values of $n$	
	i-PP/Al <sub>2</sub> O <sub>3</sub>	i-PP/Al <sub>2</sub> O <sub>3</sub> /LICA 38
0	0.352	0.352
5.8	0.347	0.301
12.5	0.330	0.318
16.9	0.374	0.318
22.2	0.367	0.314

particles, less than 0.5  $\mu\text{m}$  in diameter, which showed strong particle-particle affinity. In the present work no such yield stress was noted, which may be due to larger size of alumina particles (average diameter  $\sim 69 \mu\text{m}$ ) in addition to higher values of  $\dot{\gamma}_w$  (25 to 5000  $\text{s}^{-1}$ ) used for the study. Other systems also reported absence of yield stress in the rheological behavior [5, 9, 13, 24].

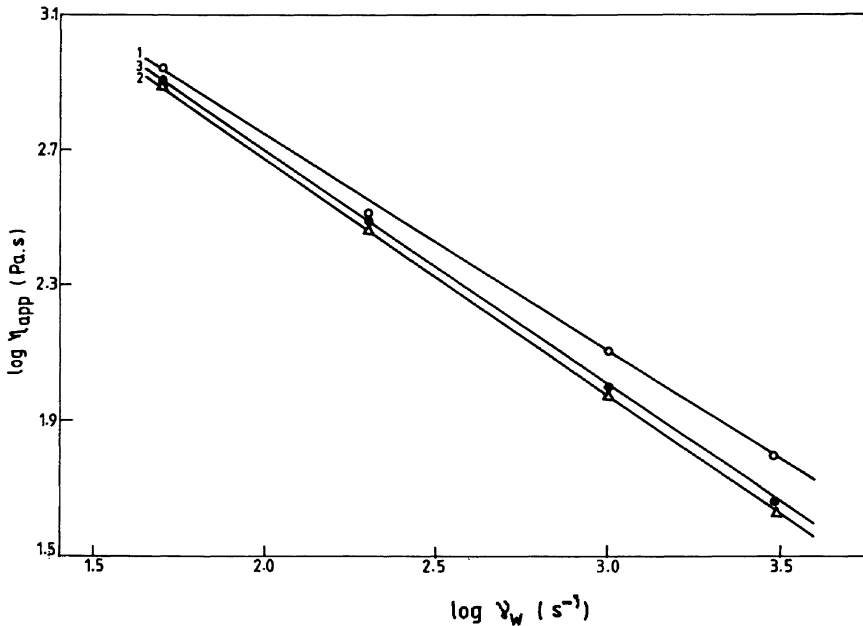
**FIGURE 3** Variations of log (apparent melt viscosity,  $\eta_{app}$ ) against log  $\dot{\gamma}_w$  in i-PP/Al<sub>2</sub>O<sub>3</sub> composites at 493 K. Descriptions of the curves are as in Figure 1.

### Apparent Melt Viscosity ( $\eta_{app}$ )

Variations of  $\eta_{app}$  vs.  $\dot{\gamma}_w$  of the i-PP/Al<sub>2</sub>O<sub>3</sub> and i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 composites are presented in Figures 3 and 4, respectively. The apparent viscosity values in these composites were of similar order with i-PP filled composites containing wood fibers and inorganic fillers [11–13]; the values were, however, less than other i-PP systems by  $\sim 1$  order [9, 25]. This may be due to different grades of polymer used for the works.

The  $\eta_{app}$  values decrease quite sharply with increase in  $\dot{\gamma}_w$  in both i-PP/Al<sub>2</sub>O<sub>3</sub> and i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 composites, which implies that as with nonfilled i-PP, alumina-filled i-PP systems are shear-thinning as well. At a constant- $\dot{\gamma}_w$ , the  $\eta_{app}$  at first-decreases up to alumina volume %  $\phi_F = 5.8$  whereas beyond this  $\phi_F$  the data increased with  $\phi_F$ . Similar results were observed in i-PP composites with kaolin and CaSiO<sub>3</sub>[11, 12].

Variations of  $\eta_{app}$  vs.  $\phi_F$  (%) at a fixed shear rate 1000 (s<sup>-1</sup>) and at 493 K are shown in Figure 5. In the i-PP/Al<sub>2</sub>O<sub>3</sub> systems  $\eta_{app}$  at first decreased up to  $\phi_F = 5.8\%$ , exhibiting a minimum here, the decrease



**FIGURE 4** Plots of  $\log \eta_{app}$  vs.  $\log \dot{\gamma}_w$  in i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 composites at 493 K. Descriptions of the curves are as in Figure 1.



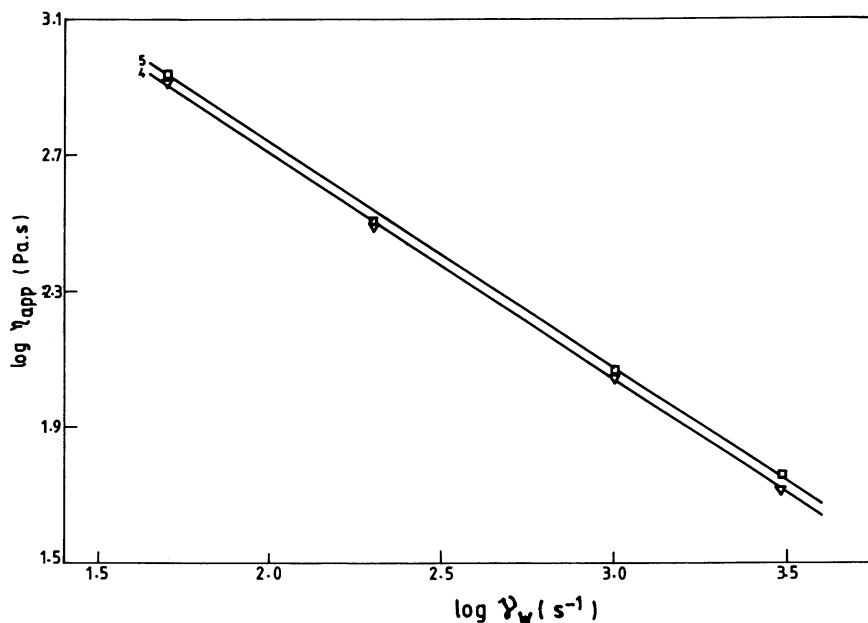
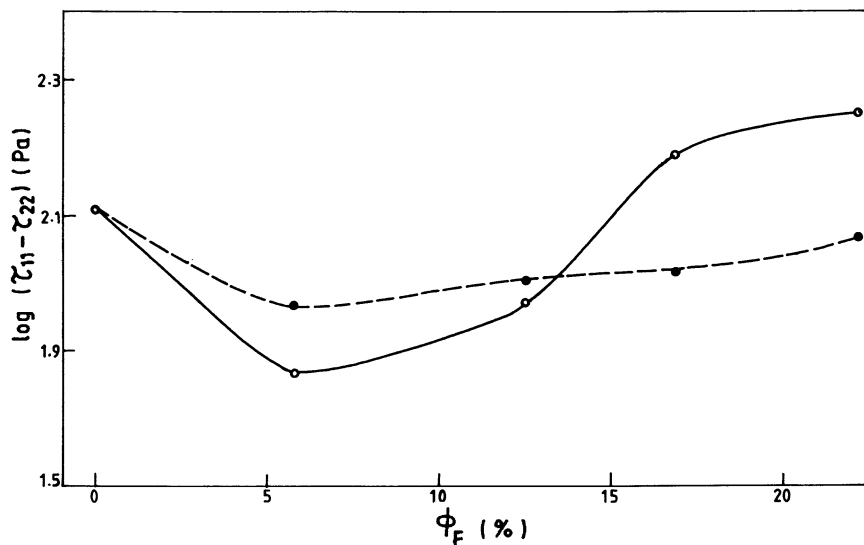


FIGURE 4 (Continued).

being  $\sim 43\%$  compared to the value of i-PP. The viscosity increased with further increase in  $\phi_F$  and at  $\phi_F > 12.5\%$ ; the data increased by 20–37% from the value of i-PP. Alumina particles are acicular and may have interparticle interactions through surface groups, which could lead to thixotropic effect in polymers composite [26, 27]. At filler concentration up to 5.8% the shear forces appear to disrupt the particular networks, which draws the particles to a flow favoring orientation decreasing the melt viscosity. Upon increasing the  $\phi_F$  further the particle network reformation is facilitated, which obstructs the flow of the polymer increasing the melt viscosity. Similar observations were reported in i-PP/Kaolin and i-PP/CaSiO<sub>3</sub> composite also [11, 12].

In the i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 systems  $\eta_{app}$  also decreased up to  $\phi_F = 5.8\%$ , the data then increased marginally with increase in  $\phi_F$ , remaining always lower than that of i-PP. Although a minimum was shown at  $\phi_F = 5.8\%$ , the minimum was less pronounced compared to i-PP/Al<sub>2</sub>O<sub>3</sub> (untreated) systems. The decrease in  $\eta_{app}$  were 29–9% depending on  $\phi_F$ . This indicates a higher extent of i-PP-alumina interaction, as was also observed in the solid state properties of i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 composites (26), which prevents the alumina



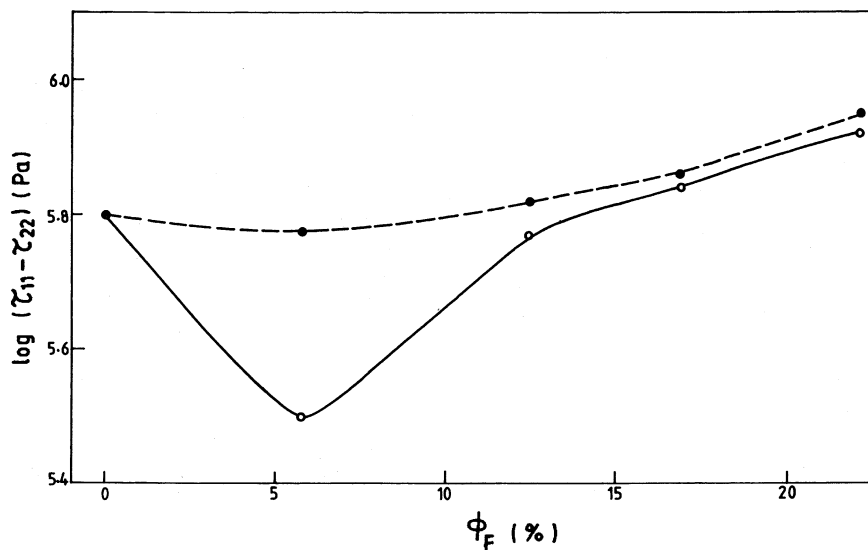
**FIGURE 5** Variations of  $\log \eta_{app}$  vs.  $\phi_F$  (%) at 493 K at a constant  $\dot{\gamma}_w$  value of 1000 ( $s^{-1}$ ) in (○) i-PP/Al<sub>2</sub>O<sub>3</sub> and (●) i-PP/Al<sub>2</sub>O<sub>3</sub>/LICA 38 composites.

particles to the flow favoring orientation to an extent so that  $\eta_{app}$  decrease is lower up to  $\phi_F = 5.8\%$ . Since some of the surface groups are bonded with the polymer particle network formation is impeded even at  $\phi_F > 12.5\%$  so that melt flow obstruction was lower contrary to the previous composites. This effect coupled with the plasticizing/lubricating effect of the coupling agent [4, 25] seems effective at these  $\phi_F$  values so that the  $\eta_{app}$ , although increased marginally than that at  $\phi_F = 5.8\%$ , remained lower than i-PP as well as i-PP/Al<sub>2</sub>O<sub>3</sub> composites.

## Melt Elasticity

Melt elasticity parameter first normal stress difference ( $\tau_{11} - \tau_{22}$ ) of the composites were evaluated at 493 K. These data have been directly calculated from the viscosity function measured on Rheographs 2001 (Version 9.6) and corrected by using the "RABKOR" program [16, 17].

Variations of  $\tau_{11} - \tau_{22}$  vs.  $\phi_F$  at a constant shear rate value 1000  $s^{-1}$  are presented in Figure 6. The value of  $\tau_{11} - \tau_{22}$  were of similar order with i-PP/WF composites (28) and about one order lower than the data of other i-PP based systems [11, 13, 29]. In the i-PP/Al<sub>2</sub>O<sub>3</sub> composites the  $\tau_{11} - \tau_{22}$  decreased initially upto  $\phi_F = 5.8\%$ , showing a minimum here, the total decrease was 7–50% depending on  $\phi_F$ , compared to nonfilled i-PP. It may be pointed out that  $\eta_{app}$  also decreased up to



**FIGURE 6**  $\log(\tau_{11} - \tau_{22})$  vs.  $\phi_F$  (%) plots at 493 K at a fixed  $\gamma_w$  value of 1000 ( $s^{-1}$ ) in (○) i-PP/ $Al_2O_3$  and (●) i-PP/ $Al_2O_3$ /LICA 38 composites.

$\phi_F = 5.8\%$ , which was postulated to be due to the rupture of particle networks and a flow-favoring orientation of the acicular  $Al_2O_3$  particles. It appears that this orientation is disturbed after the emergence of the melt from the die and the particles are quickly disposed in a flow restricting orientation which decreases the elastic recovery of the melt. At  $\phi_F > 5.8\%$  the elasticity increased and at  $\phi_F > 12.5\%$  the increase was by 10–32% over that of i-PP. At these  $\phi_F$  values particle networks may be reformed, at least partially, and the polymer is squeezed out to the surface, which partially establishes matrix continuity after the melt's emergence from the die so that elastic energy recovery is facilitated, increasing the melt elasticity. Similar behavior of i-PP based systems were observed earlier [12, 28].

In the i-PP/ $Al_2O_3$ /LICA 38 composites the elasticity decreased only marginally at  $\phi_F = 5.8\%$  the data then increases by 5–41% over the values of i-pp depending on  $\phi_F$ . The lubricating/plasticizing effect of the coupling agent seems quite apparent, which facilitates elastic recovery of the melt over that of i-PP/ $Al_2O_3$  composites [4, 25].

## CONCLUSIONS

Power law model was followed by i-PP/ $Al_2O_3$  composites in the shear stress-shear rate variations in the range of 25–5000 ( $s^{-1}$ ) of shear rate

at 493 K. The values of the power law flow indices varied in the range  $0.348 \pm 0.018$  which indicate a pseudoplastic nature of the composite melts. Apparent melt viscosity decreased initially up to  $\phi_F = 5.8\%$  due to a flow-favoring orientation of flaky alumina particles arising out of rupture of interparticle network in the presence of shearing forces. At  $\phi_F > 5.8\%$  the  $\eta_{app}$  increased due to obstruction to the melt flow by the particle networks. Melt elasticity also decreased up to  $5.8\% \phi_F$  due to orientation of acicular  $Al_2O_3$  particles in a flow disturbing disposition upon the melt's exit from the die. At  $\phi_F > 5.8\%$  the elasticity increased due to the squeezing out of the matrix polymer to the surface by the particle networks which partially restores matrix continuity facilitating elastic recovery.

In the i-PP/ $Al_2O_3$ /LICA 38 systems similar rheological behaviors were noted. The melts followed power law model in the flow curves, the values of  $n$  varied in the range  $0.326 \pm 0.025$ . The melts were shear thinning. The variations of  $\eta_{app}$  and  $\tau_{11} - \tau_{22}$  vs.  $\phi_F$  were also quite similar as in i-PP/ $Al_2O_3$  composites. The coupling agent LICA 38 modified these variations through lubricating/plasticizing functions and/or blocking some of the surface groups of alumina, however.

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