Rheological studies on mineral wool fibre reinforced PP/HDPE blend

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Mineral wool (MW) fibre reinforced polypropylene (PP)/high density polyethylene (PP/HDPE) blends have been developed by varying mineral wool fibre loading ranging from 5 to 15 wt%. Rheological data on reinforced blends are obtained by using a capillary rheometer at 220°C in shear rate ranging from 1 to \(10^3\) s\(^{-1}\). It has been found that incorporation of mineral wool fibre in to PP/HDPE blends increases the melt viscosity and reduces the recoverable shear strain \(\varepsilon_r\). Also, the shear rigidity \(G\) increases with the increase of mineral wool fibre loading.

Blends of isotactic polypropylene and high-density polyethylene are industrially useful materials. These blends are prepared for improving the impact strength and processability of PP and improving the environmental stress cracking resistance and processability of HDPE\(^1\)\(^2\). Large number of studies have so far been done on the rheological properties of PP/HDPE blends\(^1\)\(^6\)\(^9\)\(^13\)\(^16\)\(^17\), which include flow curves\(^1\)\(^6\)\(^9\)\(^13\)\(^16\)\(^17\), die swell\(^7\)\(^11\)\(^15\)\(^17\), melt fracture\(^5\)\(^6\)\(^16\), recoverable shear strain\(^8\)\(^14\), normal stress\(^13\)\(^14\), dynamic viscoelasticity\(^12\), relaxation spectrum\(^7\), and morphology.

Kuznetsov et al.\(^19\) prepared a composite material consisting of low density polyethylene (40-94%), chalk (4.85-48.50%), mineral fibre (0.99-9.93%) and silane coupling agent having improved melt flow index with increased strength. Polycarbonates were reinforced with mineral fibres to achieve good mechanical properties\(^20\).

Mineral wool fibre is made mainly from blast furnace slag and basalt rocks. It has calcium silicate along with \(Al_2O_3\), \(MgO\), \(Fe_2O_3\), \(Na_2O\), \(K_2O\) and oxides of other light-weight metals\(^18\). Not much data is available on rheology of mineral wool fibre reinforced thermoplastics and blends.

Chand et al.\(^18\)\(^21\)\(^23\) have recently reported the flow behaviour of PP and PP/LDPE blend reinforced with mineral wool fibre having different fibre loading ranging from 5-15 wt% of the blend.

**Note**

This paper describes the mineral wool fibre reinforced isotactic polypropylene/high density polyethylene blends developed at the laboratory. Rheological properties of these composites have been determined at 220°C.

**Materials and Methods**

**Materials**

The commercial grade isotactic polypropylene (PP) was supplied by M/s Shri Ramdev Plastics, India, and commercial grade semi-crystalline HDPE was supplied by M/s Monika Plastics, India. Chemical constitution of mineral wool fibres was reported in our earlier communication\(^18\). The density of PP, HDPE and mineral wool fibre was: 0.9, 0.93 and 2.7g/cc respectively.

**Preparation of filled blends**

To dry the materials, HDPE, PP and mineral wool fibre samples were kept in air circulating oven at 70°C for 24 h. These materials were mixed on a single screw extruder. Barrel diameter of the extruder was 1" and \(L/D\) ratio of the screw was 27.5. The capacity of the extruder was 16 lb/h. Temperature of the feed, compression and metering zones was 210, 220 and 235°C respectively and the temperature of die was 200°C. Rotation speed of the screw was 20 rpm.

Composition of some typical samples of the mineral wool (MW) reinforced blends was:

- PP/HDPE/MW (Sample A-160:40:00; Sample B-160:40:10; Sample C-160:40:20; Sample D-160:40:30).

**SEM analysis of minwool fibre**

Surface of minwool fibre was observed after gold coating on scanning electron microscope (model JEOL 35 CF).

**Melt rheology**

Melt rheological measurements were carried out by using MCR capillary rheometer attached to Instron 1185 model. \(L/D\) ratio of capillary was 33 and its diameter was 1.57 mm. Measurements were done at a constant temperature (220°C) in shear rate ranging
Apparent shear rate ($\dot{\gamma}_a$) was calculated by using Eq. (1):

$$\dot{\gamma}_a = \frac{4V}{\pi R^3}$$

where $V$ is volumetric flow rate and $R$ is radius of capillary die.

Rabinowitch correction was applied for determining true shear rate ($\dot{\gamma}_w$) by Eq.(2):

$$\dot{\gamma}_w = \frac{(3n+1)}{4n} \dot{\gamma}_a$$

where $n$ is the pseudoplasticity constant.

Shear stress ($\tau_w$) at capillary wall was determined by relation (3):

$$\tau_w = \frac{\Delta P R}{2L}$$

where $L$ is the length of capillary and $\Delta P$ is a total pressure drop between ends of the capillary. $\Delta P$ is calculated using Eq.(4):

$$\Delta P = \frac{\text{Load (N)}}{\text{Area (}\pi R^2)}$$

Melt viscosity ($\eta$), recoverable shear strain ($S_R$) and shear rigidity ($G$) were calculated by known procedures using relations (5)-(7):

$$\eta = \frac{\tau_w}{\dot{\gamma}_w}$$

$$S_R = \frac{(\tau_{11} - \tau_{22})}{2\tau_w}$$

$$G = \frac{\tau_w}{S_R}$$

where $(\tau_{11} - \tau_{22})$ is first normal stress difference.

Results and Discussion

Fig. 1 shows the microstructure of mineral wool fibre. Diameter and length of mineral wool fibres were respectively 2-5 microns and 80-300 microns (Fig. 1) and aspect ratio of fibres was 40-60.

Fig. 2 shows log shear rate vs log shear stress plot of samples A, B, C and D. It is shown that shear stress increases with increased shear rate.

Incorporation of mineral wool fibre has been found to increase the shear stress at all shear rates. These curves are linear over the entire range of data on double logarithmic scale. These linear curves are consistent with the following power law relationship:

$$\tau_w = K \dot{\gamma}^n$$

where $K$ and $n$ are fitted coefficients, and $n$ is known as pseudoplasticity index.

The values of $n$ calculated from the shear stress vs shear rate curves for samples A, B, C and D are: 0.55, 0.52, 0.50 and 0.46. The values of $n$ indicate that all these composites are pseudoplastic in nature in the entire range of shear rate from 1 to $10^1$ s$^{-1}$. These curves are similar in nature to flow curves of PP/LDPE blends as reported earlier. It seems that mineral wool fibres do not change the flow behaviour of the matrix. The decrease in $n$ from 0.55 to 0.46 with increasing MW reinforcing indicates the role of mineral wool fibre in increasing the pseudoplasticity of PP/HDPE blend melts.
Fig. 2 — Shear stress vs shear rate curves for samples A(●), B(●), C(■) and D(▲).

Fig. 3 — Melt viscosity vs shear rate curves for samples A(●), B(●), C(■) and D(▲).

Fig. 3 shows the variation of melt viscosity with shear rate, showing thereby that melt viscosity decreases with shear rate. But, increase in minwool loading increases the viscosity at all shear rates. This shows that minwool fibers have good adhesion with PP and HDPE. It has been reported in literature that the melt viscosity increases with increasing amount of perlite concentration with low-density polyethylene.\textsuperscript{22} Also, melt viscosity has been found to increase with increased amount of perlite for the other polyethylenes studied, which is similar to a mineral fibre reinforced PP/LDPE blends.\textsuperscript{18} Variation of melt viscosity with shear rate showed a linear change in the entire range of shear rate data. The overall increase of melt viscosity with increased MW fibre loading is due to the hindrance to the flow produced by the minwool fibre, a trend that has been reported frequently for rigid solid suspension.\textsuperscript{24,26}

The effect of addition of MJW fibres on recoverable shear strain of PP/HDPE blend matrix is represented in Fig. 4 at constant blending ratio and varying MW loading from 5 to 15 wt%. At high shear rates, the
scatter of data is higher as compared to the low shear rate. The scatter of data at higher shear rate may be due to the non-equilibrium state of elastic recovery. At low shear rate, the increase in $S_R$ was low but after that $S_R$ increased very sharply at higher shear rate region. $S_R$ reduced with the increase of MW fibre content but the trend of the curve was not changed with the change of loading on fibre content in the composites. Incorporation of MW fibre loading in the blend reduced the elastic recovery due to the hindrance effect. Similar observations were made by Chand and Hashmi\textsuperscript{23} in red mud filled PP/PE blend by varying the loading of red mud particles from 5 to 30 wt\% at fixed PP/PE ratio (3:1).

Fig. 5 shows the variation of shear rigidity with shear rate. It has been observed that shear rigidity improves with the incorporation of minwool fibre. This may be due to presence of rigid MW fibres suspended in to a molten blend which contribute their own modulus increasing the rigidity of the filled blend. Variation of shear rigidity with shear rate is nonlinear with considerable scatter of data points. The nonlinearity of melt elasticity with shear rate is attributed to nonequilibrium of elastic recovery in the
presence of mineral fibres. First normal stress difference also increases on increase of mineral wool fibre
loading in PP/HDPE blend matrix (Fig. 6).

Conclusions
 Addition of mineral wool fibre lowers down the pseudoplasticity index ‘n’. Melt viscosity shows an overall increase with increasing mineral fibre content in the PP/HDPE blend.

Melt elasticity of PP/HDPE/MW decreases with the increasing mineral fibre loading in the matrix blend. The nonlinearity of melt elasticity with shear rate is attributed to nonequilibrium of elastic recovery in the presence of mineral fibres. Recoverable shear strain decreased with the addition of mineral fibres and increased with the shear rate. Shear rigidity increased with the loading of mineral wool fibre in PP/HDPE blend.

References
21 Chand N & Vashishtha S R, Indian Pat No. 1111 (Filed to CSIR, New Delhi) 27 April 98.