Short Nylon Fibre Reinforced PP: Melt Rheology

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**Short fiber reinforced thermoplastics have generated much
interest these days since fibrous materials tend to increase both
mechanical and thermal properties, such as tensile strength, flexural
strength, flexural modulus, heat deflection temperature, creep
resistance, and some times impact strength of thermoplastics. If
the matrix and reinforcement are both based on polymers the com-
posite are recyclable. The rheological behavior of recyclable compo-
sites based on nylon fiber reinforced polypropylene (PP) is reported
in this paper. The rheological behavior was evaluated both using a
capillary rheometer and a torque rheometer. The study showed that
the composite became pseudoplastic with fiber content and hence
fiber addition did not affect processing adversely at higher shear
rates. The torque rheometer data resembled that obtained from
the capillary rheometer. The energy of mixing and activation energy
of mixing also did not show much variation from that of PP alone.**

1. INTRODUCTION

The incorporation of short fibers to thermoplastics and
elastomers to achieve cost reduction[1] and improvement in
mechanical properties has become increasingly important
in recent years. The rheological behavior of such compo-
sites is important for selecting the processing parameters
to fabricate products. A number of investigations on the
rheological behavior of short fiber reinforced thermoplastic
and elastomers have been reported[2–5]. Usually, the incor-
poration of short fiber in thermoplastics and elastomers
increases the melt viscosity and may result in unusual rheo-
logical effects. A decrease in melt viscosity as a result of
the incorporation of short fibers was also reported[6–9].
The influence of fiber length and fiber loading is more pro-
nounced at lower shear rate than at higher shear rate. The
melt viscosity of glass fiber-filled polyethylene (PE) and
polyproylene was studied by Becraft and Metzner[10–11]
and it was found that there is a significant increase in
viscosity with fiber loading at low shear rates and little
change in viscosity at higher shear rates.

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Short nylon fibers were used as recyclable reinforcing
material for polypropylene (PP). Because of the advantages
of repeated recyclability, the rheological behavior of such
composites is all the more important. In this article the
effect of temperature, fiber concentrations, and shear rate
on the melt flow behavior of composites is investigated.

2. EXPERIMENTAL

2.1. Materials

Composites were prepared using polypropylene (PP;
REPOL H200MA, Reliance Industries Limited), with a melt
flow index of 20 g/10 min (230°C/2.16 kgf) and nylon-6
fibers (0.03 mm) obtained from Apollo Tyres. Styrene
maleic anhydride (SMA)-*grafted*-PP was prepared as per
US patent, 4,753,997. The continuous nylon fiber was cut
to staples, having an average length of 8 ± 2 mm.

2.2. Composite Preparation

PP granules and nylon fibers were dried by keeping them
in an oven at a temperature of 100°C for 4 h. Nylon fiber
reinforced PP composites were prepared by adding nylon
fibers to PP melt in a Thermo Haake Rheocord 600 mixing
chamber with a volumetric capacity of 69 cm³ fitted with
roller type rotors. PP together with the nylon fibers was
added to the chamber. The temperature was kept at
170°C, which ensured proper melting of PP keeping the
nylon fibers intact. In the first series, 10, 20, and 30-wt.%
of nylon reinforced PP composites were prepared. In the
second series SMA-*grafted*-PP was used. A mixing time
of 8 min was given at a rotor speed of 40 rpm. In all cases
the torque stabilized to a constant value in this mixing time.

The hot mix from the mixing chamber was passed
through a two-roll mill and cut to thin strips. The test spe-
cimens were prepared by a semiautomatic plunger type
injection-molding machine, at a barrel temperature of
190°C, well below the melting point of nylon fibers.

For checking the recyclability[12] of the composites they
were mixed again in the Thermo Haake Rheocord 600.
This time temperature was kept at 215°C which is sufficient
to melt the nylon fibers at the shear involved.

2.3. Rheological Measurements Using Capillary

The melt rehological measurements were performed using Shimadzu capillary rheometer (50 kN) at different plunger speeds from 1–500 mm/min. The capillary used was made of tungsten carbide with a length to diameter (L/D) ratio of 40 at an angle of entry 90°. The sample for testing was loaded inside the barrel of the extrusion assembly and forced down the capillary using a plunger. After giving a residence time of 5 min the melt was extruded through the capillary at predetermined plunger speeds. The initial position of the plunger was kept constant in all experiments and shear viscosities at different shear rates were obtained from a single charge of the material. The measurements were performed at two different temperatures such as 180 and 220°C.

The shear stress at different plunger speeds were calculated using the equation

$$\tau_a = \frac{F}{4\,A_p(l_c/d_c)}$$

where F is the force on the plunger, A_p the cross-sectional area of the plunger and l_c and d_c are the length and diameter of the capillary, respectively. The shear rate at the wall is calculated using the equation

$$\gamma = \frac{R\Delta P}{2\,L}$$

The rheological measurements were also made using the Haake torque rheometer.

FIG. 2. Variation of viscosity with shear rate at 180°C for nylon SMA-*g*-PP composites.

2.4. Die Swell Behavior

The extrudates were carefully collected as they emerged from the capillary die, taking care to avoid any deformation. The diameter of the extrudate was measured after 24 h of extrusion using a traveling microscope. The die swell was calculated as D_e/D where D_e is the diameter of the extrudate and D is the diameter of the capillary.
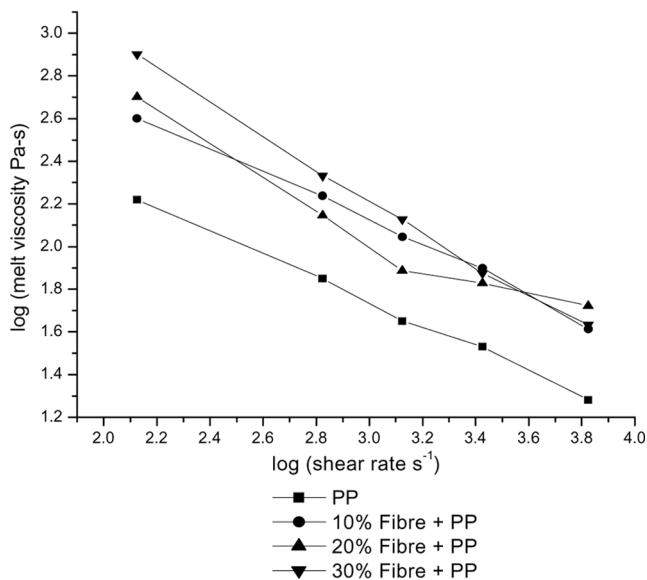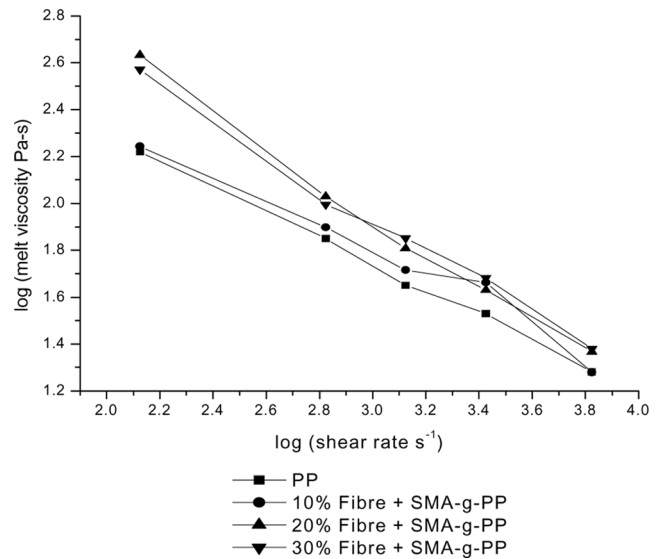

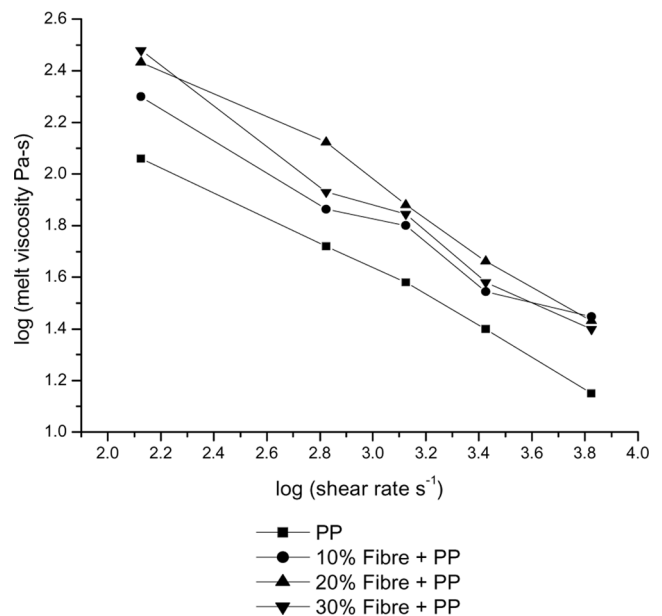FIG. 1. Variation of viscosity with shear rate at 180°C for nylon PP composites.

FIG. 3. Variation of viscosity with shear rate at 220°C for nylon PP composites.
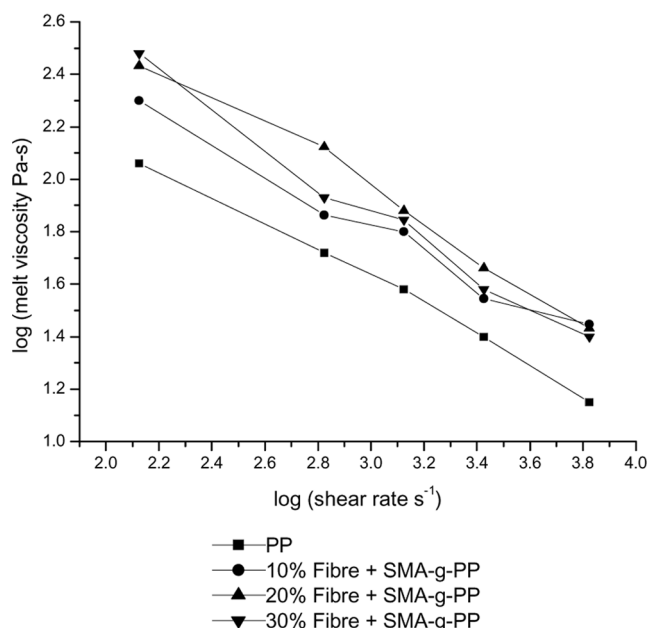
FIG. 4. Variation of viscosity with shear rate at 220°C for nylon SMA-g-PP composites.

2.5. Activation Energy

Variation of viscosity with reciprocal of absolute temperature for the composites at various fiber loading and a fixed rate of 1333 sec^{-1} is shown in Fig. 1. The slope of the lines is proportional to an apparent energy of activation for viscous flow and the activation energy at this shear rate was calculated.

2.6. Energy Required to Plasticize

The energy[13] required to plasticize a polymer for a period of time at a given temperature can be calculated from the
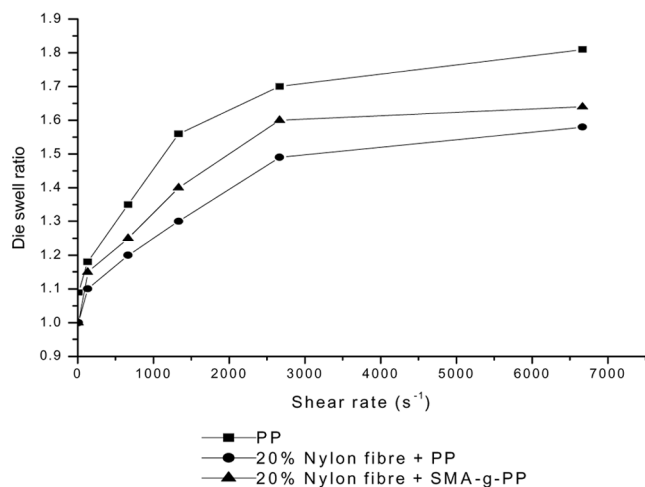

FIG. 5. Die-swell behavior of PP, PP/nylon composite and SMA-g-PP/nylon composite at different shear rate at const temperature 180°C.
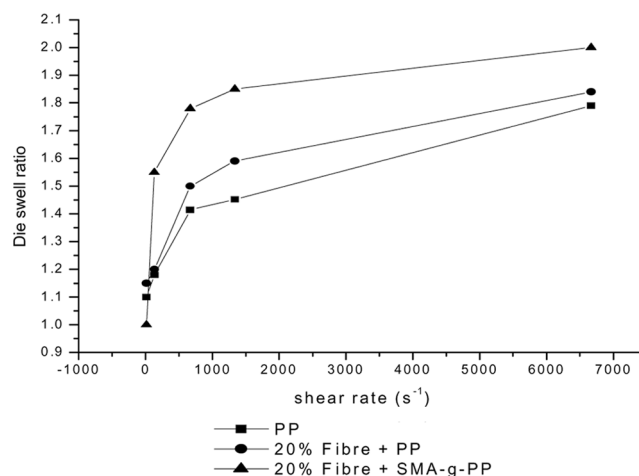
area under the torque-time curve at a preset temperature for the specified period of time. The energy W was calculated using the formula at a given temperature and shear rate.

$$W = 2^{\overline{\Pi}}n \int_{t_1}^{t_2} M.dt$$

where, n is the number of revolutions of the rotor per min, t_1 is the initial time, t_2 is the final time and M the torque in Nm.

3. RESULTS AND DISCUSSION

3.1. Effect of Fiber Loading and Shear Rate on Viscosity

Figs. 1–4 show the variation of viscosity of PP composites with shear rate and fiber loading at 180 and 220°C. These curves are typical of pseudoplastic materials, which show a decrease in viscosity with increasing shear rate.

In general, the incorporation of fibers in polymer systems increases the viscosity and increases further with
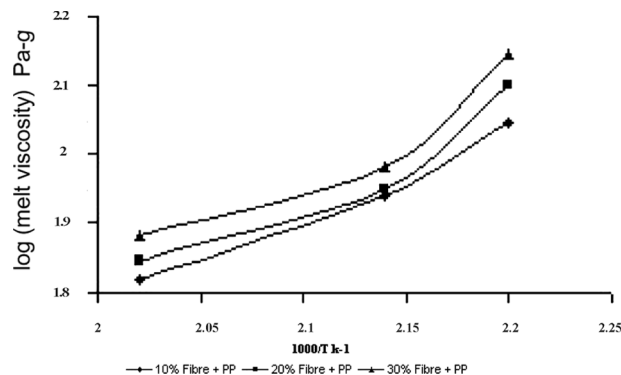

FIG. 7. Variation of melt viscosity with reciprocal absolute temperature of nylon PP composites at a fixed shear rate 1333 sec^{-1}.
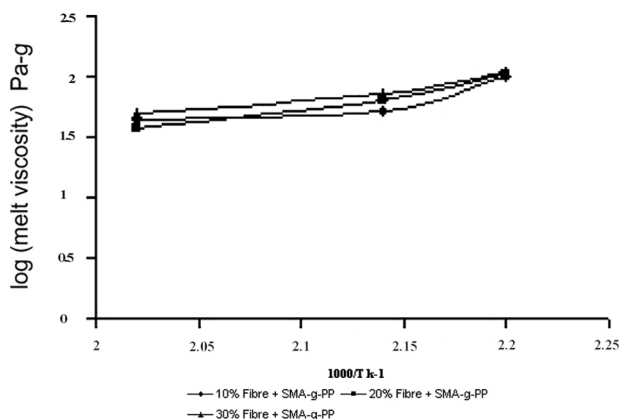
FIG. 8.   Variation of melt viscosity with reciprocal absolute temperature of nylon SMA-*g*-PP composites at a fixed shear rate $1333 \, \text{sec}^{-1}$.

fiber content. At low concentration levels, the viscosity is expected to increase rapidly with increasing concentrations of the fibers because of the rapidly increasing collisions between particles as they become packed more closely to each other. However, at a critical concentration level, random packing ceases to a more orderly anisotropic structure of the fibers in suspension, and these may now slide readily past one another. Hence, above the critical concentration level, further increase in the fiber concentration progressively decreases the viscosity of the system until very high concentration levels of the fibers are reached. The increase in viscosity is found to be more predominant at lower shear rates where fiber and polymer molecules are not completely oriented. The addition of fiber to a polymer system will perturb the normal flow of the polymer and will hinder the mobility of the chain segments in flow. As the fiber content increases the phenomenon becomes more predominant and hence the viscosity increases further.

3.2. Die Swell Behavior

Figs. 5 and 6 show the die swell behavior of the composite at two different temperatures. At lower temperature (180°C) the incorporation of fibers reduces the swelling of matrix indicates that the fiber is not getting melted at this

TABLE 1
Activation energy for nylon PP composites

Composition	Activation energy (kJ)
10% Fiber + PP	10
20% Fiber + PP	11
30% Fiber + PP	12
10% Fiber + SMA-*g*-PP	14
20% Fiber + SMA-*g*-PP	20
30% Fiber + SMA-*g*-PP	15

processing, but acts as a filler. On the other hand, at higher temperature (220°C), the incorporated fiber also gets melted and as a result the swelling increase than that of the matrix.

3.3. Activation Energy

Figs. 7 and 8 show the variation of melt viscosity with reciprocal absolute temperature of nylon PP composites at a fixed shear rate $1333 \, \text{sec}^{-1}$. The activation energy can be obtained by finding the slope of the above curve and is given in Table 1.

3.4. Energy Required to Plasticize

The energy required for making the composite at different rpm for a period of 8 min is given in Table 2. The energy needed is found to be increasing with the fiber loading and also with the increase in rpm.

4. CONCLUSION

The study shows that the viscosity of PP increases with fiber content. However, the composite becomes progressively pseudoplastic and hence at high shear rate the flow curves crowd together showing that at higher shear rates the fibers may not adversely affect processing. The elastic behavior of the matrix decreases with fiber content at 180°C because the fiber acts as filler at this temperature. However, the reverse behavior is observed at 220°C, where the fiber melts.

TABLE 2
Energy required for plasticization of nylon PP composites at different rpm

	Energy required for plasticization (J) at 30 rpm	Energy required for plasticization (J) at 40 rpm	Energy required for plasticization (J) at 80 rpm	Energy required for plasticization (J) at 100 rpm
10% Fiber + PP	4521	14871	32153	50240
20% Fiber + PP	6028	16076	36172	60288
30% Fiber + PP	7536	318086	40192	62800

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