Melt Shear Viscosity of PP/Al(OH)₃/Mg(OH)₂ Flame Retardant Composites at High Extrusion Rates

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Received 13 February 2010; accepted 31 May 2010 DOI 10.1002/app.32893 Published online 23 August 2010 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: The melt apparent shear viscosity (η_a) of polypropylene (PP) composites filled with aluminum hydroxide [Al(OH)₃] and magnesium hydroxide [Mg(OH)₂] was measured by means of a capillary rheometer under experimental conditions of temperature ranging from 180 to 200°C and apparent shear rate varying from 10 to 2 × 10³ s⁻¹, to identify the effects of the filler particle content and size on the melt viscosity. The results showed that the melt shear flow of the composites obeyed the power law and presented pseudoplastic behavior. The dependence of η_a on temperature was consistent with the Arrhenius equation. The sensitivity of η_a for the composite melts to temperature

INTRODUCTION

Polypropylene (PP) is a thermoplastic which has favorable performance in processing and practical applications as well as low price, hence it is used widely in many fields, such as in construction, transportation, electronic applications, in general house-hold materials, and so on.^{1–3} However, the poor flame resistance and impact toughness as well as obvious shrinkage rate limits its application scope. Therefore, one of the research hot-points in recent years has been how to modify PP resin to expand its applications.

Aluminum hydroxide [Al(OH)₃] and magnesium hydroxide [Mg(OH)₂], two important inorganic hydrated flame retardants for PP, are the environmentally most favored, readily available, nontoxic, and low-cost flame retardants. In the last few decades, work has been extensively reported on many aspects of inorganic flame retardant PP composites, including its flame retardancy,^{4–6} mechanical proper-

Contract grant sponsor: Research Committee of the Hong Kong Polytechnic University (Project code: G-U680).

was greater than that of the unfilled PP, and weakened with increasing apparent shear rate. The η_a increased linearly with an increase of the weigh fraction of the flame retardant, especially in the low apparent shear rate region. The η_a of the composites decreased slightly with an increase of particle size of flame retardant. Moreover, the variation for the η_a with particle size of flame retardant was much less than with apparent shear rate under these test conditions. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 1835–1841, 2011

Key words: polypropylene; flame retardant composites; melt apparent shear viscosity

ties,^{7–10} processing,^{11,12} thermal oxidative degradation,^{13,14} and so on.

Melt rheology is a branch of learning for studying the rheological behavior and mechanisms as well as the affecting factor during polymer processing. Melt apparent shear viscosity is one of the important characteristics for flow properties of polymeric materials. Studying the apparent shear viscosity and its affecting factors is helpful to reveal the flow mechanisms and also provide useful guidelines for optimum processing conditions for filled polymer systems. It has, therefore, received extensive attention in the past two decades.^{15–18} In a study on rheological properties of calcium silicate-filled isotactic PP, Maiti et al.¹⁹ found that the apparent melt viscosity of the composites decreased until a critical filler volume content of 8.5% was reached. However, with further increase in calcium silicate concentration, apparent melt viscosity increased. Muksing et al.²⁰ carried out melt rheology of organobentonite-filled PP composites by means of a twin-bore capillary rheometer, and found that the activation energy of the composites decreased with increasing shear rate. The addition of organobentonite enhanced the shear thinning behavior. With increasing organobentonite concentration, the shear stress/viscosity increased, while the power law index decreased, and this effect was more obvious at higher filler concentrations. Ghosh and Maiti²¹ found that the silver-

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Journal of Applied Polymer Science, Vol. 119, 1835–1841 (2011) © 2010 Wiley Periodicals, Inc.

powder-filled PP composites followed a power law in viscous behavior. The melt viscosity decreased with filler content up to 4.1% and increased with a further increase in filler content, but the viscosity of the composites was lower than that of unfilled PP. In the previous work, Liang²² investigated the influence of glass bead content and extrusion conditions on the melt flow properties of PP/ethylene-propylene-diene monomer copolymer (EPDM)/glass bead ternary composites, and found that the filler effect was insignificant. It may be attributable to the relatively weak polymer-particle adhesion or the smooth spherical surface of glass bead, so the flow resistance does not increase obviously like other inorganic rigid fillers, especially at high shear rates, leading to an improved sliding between neighboring melt layers. The temperature sensitivity somewhat increased with the improvement of the compatibility between PP and EPDM or with an addition of glass bead concentration.

Capillary rheometry is a usual instrument widely used in industry and research for the determination of the rheological properties of polymer melts or liquids and other non-Newtonian fluids such as blood and petroleum oil, within a wide range of shear rates. However, there are few reports on rheological properties of PP/Al(OH)₃/Mg(OH)₂ composite melts in open literatures systematically. Previously, Velasco et al.²³ investigated the melt rheology of PP/Mg(OH)₂ composites, and found that the melt flow index increased with a rise of weight fraction of Mg(OH)₂. More recently, the authors¹⁵ studied the melt flow properties of PP/Al(OH)₃/Mg(OH)₂ flame retardant composites using a melt flow indexer at low extrusion rates. The objectives of this article are to investigate the effects of extrusion conditions (e.g., temperature and apparent shear rate) and the fillers (e.g., content, size) on the melt apparent shear viscosity of PP/Al(OH)₃/Mg(OH)₂ composites at high extrusion flow rates by means a capillary rheometer.

EXPERIMENTAL

Raw materials

A polypropylene with trade mark of CJS-700G was used as a matrix resin in this work. This resin was supplied by Guangzhou Petrochemical Works in Guangdong province (PR China), and its density in the solid state and melt flow rate were 910 kg m⁻³ and 10 g/10 min, respectively. The weight-average molecular weight of the PP resin was 71,200.

Al(OH)₃ and Mg(OH)₂ with different particle sizes supplied by Foshan Jinge fire-fighting materials (P.R. China) were used as the fillers. The mean particle sizes (*d*) of both the Al(OH)₃ and Mg(OH)₂ particles were respectively, 1.25, 2.7, 5.0, and 9.0 $\mu m.$ The shape of both the two fillers was roughly spherical.

Fabrication

After the PP resin was simply mixed with the flame retardant, the mass ratio of Al(OH)₃ and Mg(OH)₂ was 1 : 2 in the flame retardant, they were blended in the PP molten state in a twin-screw extruder under conditions of temperature range from 170 to 180°C and screw speed of 100 rpm, then the extrudate of PP/Al(OH)₃/Mg(OH)₂ composites was granulated. The content of flame retardant (φ_f) were 10, 20, 30, 40, and 50 phr (per hundred parts of PP resin by weight), respectively. The diameter and length-diameter ratio of the screw were 35 and 40 mm, respectively. Finally, the PP/Al(OH)₃/Mg(OH)₂ composite particles were dried for 5 h at 80°C before rheological tests.

Instrument and methodology

The extrusion flow tests were performed on a capillary rheometer (Rheograph 25, Gottfertt, Germany) in a temperature range from 180 to 200°C, and apparent shear rate varied from 10 s⁻¹ to 2 × 10³ s⁻¹. The reservoir diameter was 15 mm, the diameter (*D*) and the length (*L*) of the capillary die were respectively, 1 and 30 mm, and die angle was 180°. The granular composites were placed in the barrel and forced through the capillary with a piston attached to the moving crosshead.

The apparent shear viscosity (η_a) can be determined by

$$\eta_a = \frac{\tau_w}{\gamma_a} \tag{1}$$

Where γ_a is the apparent shear rate, and τ_w is the wall shear stress which are expressed by:

$$\tau_w = \frac{(\Delta P - \Delta P_{\rm end})D}{4L} \tag{2}$$

and

$$\gamma_a = \frac{32Q}{\pi D^3} \tag{3}$$

where ΔP and ΔP_{end} are the total pressure drop and the end pressure drop in capillary extrusion of polymer melts, respectively, and Q is the volumetric flow rate. The end pressure drop consists of entry pressure drop and exit pressure drop, and the former is usually much more than the latter.

It is generally believed that the ratio of the entry pressure drop to the total pressure drop becomes

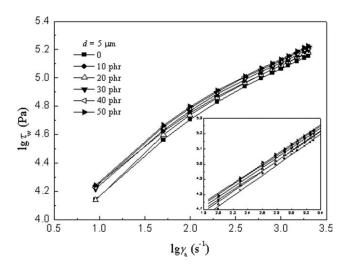


Figure 1 Flow curves of PP/Al(OH)₃/Mg(OH)₂ composite melts at 190°C.

smaller and smaller during die extrusion of polymer melts when the L/D is greater than 30. For a long die extrusion of polymer melts, therefore, the influence of inlet pressure losses on the melt flow may be neglected because of the small ratio of the inlet pressure drop to the total pressure drop. As a result, the entry correction might not be made owing the capillary used in this work is a long die (L/D = 30).

RESULTS AND DISCUSSION

Melt flow curves of PP/Al(OH)₃/Mg(OH)₂ composites

In general, the relationship between the shear stress at channel wall and the shear rate during flow of molten polymeric materials is referred to as the melt flow curve, which is an important method to characterize the processing properties of polymer melts.

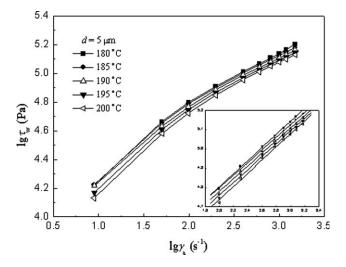


Figure 2 Flow curves of PP/Al(OH)₃/Mg(OH)₂ composite melts ($\varphi_f = 30$ phr).

TABLE I Values of k, n, and R₀ of PP/Al(OH)₃/Mg(OH)₂ Composite Melts at 190°C

(1)	1 (1 p -1)		
φ_f (phr)	k (kPa s ⁻¹)	п	R_0
0	11.0764	0.3393	0.9982
10	13.2941	0.3209	0.9988
20	12.1610	0.3343	0.9987
30	13.5584	0.3252	0.9988
40	14.3592	0.3218	0.9989
50	14.6120	0.3221	0.9990

 $\gamma_a \ge 99 \text{ s}^{-1}, d = 5 \mu \text{m}.$

Figure 1 shows the relationship between the $\lg \tau_w$ and $lg\gamma_a$ of the PP/Al(OH)₃/Mg(OH)₂ composite melts at different filler constants, temperature of 190°C and the particle diameter of 5 µm. As shown in Figure 1, with an increase in flame retardant concentration, the shear stress increases, but the difference among these composite systems are small and tend to close to each other at high apparent shear rate. Figure 2 displays the relationship between the $lg\tau_w$ and $lg\gamma_a$ of the PP/Al(OH)₃/Mg(OH)₂ composite melts at different temperature, φ_f of 30 phr and particle diameter of 5 µm. Similarly, the shear stress increases with an increase in flame retardant concentration, but the difference among these temperatures are small and tend to close to each other at high apparent shear rate. It can also be seen from Figures 1 and 2 that the plots of flow curves are roughly linear, it indicates that the relationship between τ_w and γ_a roughly accords with the power law in the case of higher shear rates, that is

$$\tau_w = k \gamma_a^n \tag{4}$$

where k is the consistency index, and n is the flow behavior index. The values of the k and n may be determined by means of a linear regression analysis method.

Although, the power-law relation does not predict the experimentally observed Newtonian plateaus in very low and very high shear rate scope, it is widely used for non-Newtonian fluids in many engineering applications.²⁴ Table I lists the values of k and n of the PP/Al(OH)₃/Mg(OH)₂ composite melts when the temperature is 190°C and γ_a is more than 10^2 s^{-1} (see the small picture in Fig. 1). The n of the composites is slightly smaller than that of the neat PP. That is, the $PP/Al(OH)_3/Mg(OH)_2$ composite melts show strong non-Newton behavior. In addition, the linear correlation coefficients (R_0) are more than 0.998.

Dependence of melt apparent shear viscosity on temperature

Figure 3 illustrates the dependence of the melt apparent shear viscosity on temperature for the PP/

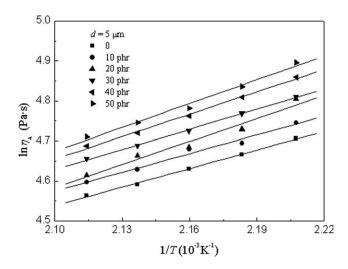


Figure 3 Dependence of apparent shear viscosity on temperature of PP/Al(OH)₃/Mg(OH)₂ composite melts ($\gamma_a = 1200 \text{ s}^{-1}$).

Al(OH)₃/Mg(OH)₂ composites at different filler content, under the testing conditions with γ_a of 1200 s⁻¹ and d of 5 μ m. Figure 4 shows the dependence of the melt apparent shear viscosity on temperature for the $PP/Al(OH)_3/Mg(OH)_2$ composites at different apparent shear rate, under the testing conditions with φ_f of 30 phr and d of 5 µm. As shown in Figures 3 and 4, the $ln\eta_a$ of the PP/Al(OH)₃/Mg(OH)₂ composite melts increases with an increase of the reciprocal of absolute temperature (1/T) at different filler contents or different apparent shear rates, and the relationship between them is roughly linear. This illustrates that the dependence of the melt shear viscosity of the PP/Al(OH)₃/Mg(OH)₂ composites on temperature obeys the Arrhenius equation under the experimental conditions. That is

$$\eta_a = A \exp(E/RT) \tag{5}$$

where *A* is the constant related to melt viscosity, *E* the activation energy of viscous flow, which is a characteristic for the sensitivity of melt apparent shear viscosity to temperature. *R* is the universal gas constant, *T* is the absolute temperature. *E* is also the slope of $\ln \eta_a - 1/T$ curves, and it can be determined by plotting.

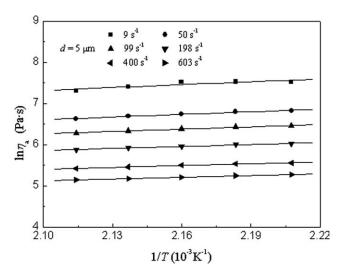


Figure 4 Dependence of apparent shear viscosity on temperature of PP/Al(OH)₃/Mg(OH)₂ composite melts (φ_f = 30 phr).

In general, the viscosity of a polymer melt decreases with increasing temperature due to greater free space and less the friction force available for molecular chain motion at higher temperature.²⁵ The values of constants A and E of the composite melts on different conditions obtained by means of linear regression analysis method from the curves of $ln\eta_a$ versus 1/T. Table II lists the values of E, A and R_0 of the PP/Al(OH)3/Mg(OH)2 composite melts at apparent shear rate of 1200 s⁻¹ and \hat{d} of 5 µm. It can be seen that the value of E increases with an increase of φ_f except individual data point. In addition, the E of the pure PP melt is close to the result reported in the previous literature.²⁰ Compared with the unfilled PP melt, the E of the $PP/Al(OH)_3/$ Mg(OH)₂ composite melts is relatively high, suggesting that the sensitivity of the apparent shear viscosity for the composite melts to temperature increases due to the addition of the fillers. Table III lists the values of E, A, and R_0 of the PP/Al(OH)₃/Mg(OH)₂ composite melts at φ_f of 30 phr and d of 5 µm. It may be observed that the activation energy for viscous flow decreases with increasing apparent shear rate. The reason may be that high apparent shear rate leads to a reduction of the entanglement points of the composites and the interaction force between chain segments.

 TABLE II

 Values of E, A, and R₀ of PP/Al(OH)₃/Mg(OH)₂ Composite Melts

φ_f (phr)	0	10	20	30	40	50
E/(kJ mol ⁻¹)	12.8475	12.9082	16.0016	13.9430	15.5034	16.4671
A	3.6443	3.7239	1.7184	3.0245	2.0939	1.6741
R ₀	0.9989	0.9900	0.9823	0.9992	0.9978	0.9934

 $\gamma_a = 1200 \text{ s}^{-1}, d = 5 \ \mu\text{m}.$

Journal of Applied Polymer Science DOI 10.1002/app

values of L, A, and K_0 of $\Gamma\Gamma/An(OII)_3/Mg(OII)_2$ Composite Metis						
$\gamma_a(s^{-1})$	9	50	99	198	400	603
$E/(kJ mol^{-1})$	19.2361	17.7211	15.8925	13.3131	12.2336	12.0616
Α	11.6742	8.4137	9.4532	12.1249	10.1049	7.9531
R_0	0.8963	0.9862	0.9859	0.9888	0.9941	0.9975

TABLE III Values of E, A, and R_0 of PP/Al(OH)₃/Mg(OH)₂ Composite Melts

 $\phi_f = 30 \text{ phr}, d = 5 \mu \text{m}.$

Dependence of melt apparent shear viscosity on apparent shear rate

Figure 5 illustrates the dependence of the apparent shear viscosity on apparent shear rate for the PP/Al(OH)₃/Mg(OH)₂ composite melts with different concentrations of flame retardant at temperature of 190°C. In a $lg\eta_a - lg\gamma_a$ bi-logarithm coordinate system, the apparent shear viscosity of the PP/Al(OH)₃/Mg(OH)₂ composite melts with different contents of flame retardant decreases roughly linearly, this indicates that the relationship between them obeys also the power law. That is, it is evident that the melt apparent shear viscosity reduces quickly with an increase of apparent shear rate. Namely, the PP/Al(OH)₃/Mg(OH)₂ composites have obvious shear thinning effect (i.e., pseudoplastic property).

Generally speaking, the melt macromolecules deviate from equilibrium conformation and orientate along the flow direction simultaneously with a rise of apparent shear rate. Moreover, interaction force between the PP macromolecules and the filler particles decreases, the rate of destruction of network junctions is greater than the rate of creation. Consequently, the macromolecular movements are relatively easy, and the apparent shear viscosity of the PP/Al(OH)₃/Mg(OH)₂ composite melts reduces accordingly.

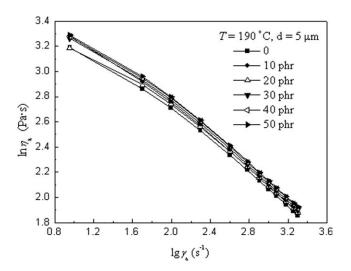


Figure 5 Dependence of apparent shear viscosity on temperature on apparent shear rate of $PP/Al(OH)_3/Mg(OH)_2$ composite melts.

Relationship between melt apparent shear viscosity and filler content

The effect of flame retardant content on the apparent shear viscosity of the PP/Al(OH)₃/Mg(OH)₂ composite melts at 195°C is shown in Figure 6. It can be seen that the melt apparent shear viscosity of the PP/Al(OH)₃/Mg(OH)₂ composites increases linearly with increasing the filler concentration. In other words, the flow resistance of the composite melts macromolecules increases owing to the addition of flame retardant. Meanwhile, many new network junctions form between the melt macromolecules and flame retardant, leading to significantly increasing the melt apparent shear viscosity with an increase of φ_{f} . The relationship between them is given by:

$$\eta_a = f_1 + f_2 \varphi_f \tag{6}$$

where f_1 and f_2 are the coefficients related to the melt apparent shear viscosity. Similarly, the values of the f_1 and f_2 of the composite systems may be determined by using a linear regression analysis method, as summarized in Table IV. It is known from eq. (6) that the f_2 reflects the sensitivity of the melt apparent shear viscosity to the flame retardant content. It can be concluded that the f_2 decreases

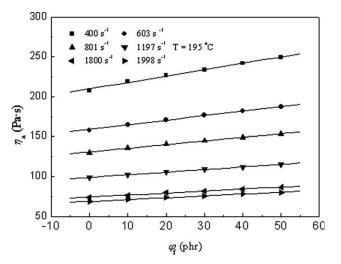


Figure 6 Relationship between filler weigh fraction and apparent shear viscosity of $PP/Al(OH)_3/Mg(OH)_2$ composite melts.

Journal of Applied Polymer Science DOI 10.1002/app

with an increase of melt apparent shear rate. In other words, the viscosities for the composite melts tend to converge at high apparent shear rate; the variation of the apparent viscosity at low apparent shear rate was more significant than that at high apparent shear rate due to the strong pseudoplasticity of filled systems. This phenomenon also can be observed in other PP-based composites²⁶ as well as LDPE-based composites,²⁷ ABS-based composites.²⁸

In general, the filling of filler particles will block the movement of molecular chains of the matrix resin, leading to an increase of the melt viscosity. It can also be seen from Figure 6 that the melt apparent shear viscosity of the composites increases linearly with increasing the filler concentration, and the correlation coefficients are greater than 0.99. This indicates that the relationship between the melt apparent shear viscosity and the weight fraction of the fillers is consistent with the rule of mixture. In other words, the melt apparent shear viscosity dose not fluctuate with an addition of the $Al(OH)_3/Mg(OH)_2$ particles. It means that the dispersion of the filler particles in the PP matrix is roughly uniform under these experimental conditions.²⁹

Effect of flame retardant particle size on melt apparent shear viscosity

Figure 7 shows the effect of the flame retardant particle size on the melt apparent shear viscosity for the $PP/Al(OH)_3/Mg(OH)_2$ composite melts at φ_f of 20 phr. It may be observed that the melt apparent shear viscosity decreases linearly with increasing the particle diameter under different temperature. This is because the number of filler in the composite melts reduces with an increase of the particle size under the same flame retardant concentration. In this case, the distances between flame retardant particles increase, and the flow resistance of PP macromolecules decreases. On the other hand, the smaller the particle size, the larger the surface energy is, which makes the composite melts form more network junctions to produce larger flow resistance. Consequently, the composite melts present a better flow

TABLE IV Values of f_1 , f_2 , and R_0 of PP/Al(OH)₃/Mg(OH)₂ Composite Melts at 195°C

	-		
$\gamma_a(s^{-1})$	f_1	f_2	R_0
400	209.7619	0.8029	0.9970
603	158.9048	0.5771	0.9979
801	131.0476	0.4514	0.9962
1197	98.8857	0.3286	0.9985
1800	74.3333	0.2480	0.9986
1998	68.8381	0.2311	0.9988

 $d = 5 \ \mu m.$

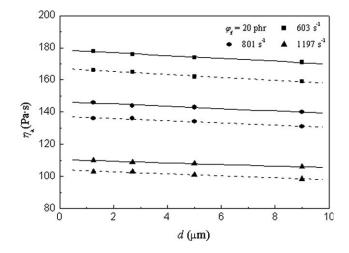


Figure 7 Effect of filler diameter on apparent shear viscosity of $PP/Al(OH)_3/Mg(OH)_2$ composite melts (-190°C, -200°C).

behavior during extrusion with increasing particle size. However, it is interesting to note in Figure 7 that a weak trend of decrease in melt apparent shear viscosity of the composites with increasing particle size of flame retardant can be perceived at different apparent shear rates and temperatures. In comparison, the variations with the particle size of the flame retardant are much less significant than with apparent shear rate for the apparent shear viscosity in this study.

CONCLUSIONS

Under the experimental conditions of temperature from 180 to 200°C and apparent shear rate varying from 10 to 2×10^3 s⁻¹, the effects of extrusion conditions as well as the filler content and size on the flow behavior of the PP/Al(OH)₃/Mg(OH)₂ composite melts were significant.

The melt shear flow of the composites obeyed roughly the power law. The dependence of the melt apparent shear viscosity on temperature was consistent with the Arrhenius equation. The sensitivity of the apparent shear viscosity for the composite melts to temperature was greater than that of the unfilled PP, and decreased as apparent shear rate rises.

The melt apparent shear viscosity of the composites decreased with an increase of apparent shear rate. The melt apparent shear viscosity increased linearly with an increase of the content of the flame retardant, but this increase rate decreased with a rise of apparent shear rate. The melt apparent shear viscosity of the composites decreased slightly with an increase of particle size of flame retardant. Furthermore, the changes with particle size of flame retardant for the apparent shear viscosity were much less significant than with apparent shear rate.

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