

Polypropylene–Clay Blends Compatibilized with MAH-g-POE

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ABSTRACT: A series of new Polypropylene (PP)–clay blends, containing 5 wt % clay, were prepared by melt compounding with maleic anhydride grafted poly(ethylene-co-octene) (MAH-g-POE) as the compatibilizer by varying its content from 0 to 20 wt %. The effect of MAH-g-POE on the PP–clay miscibility was examined by X-ray diffraction (XRD), scanning electronic microscope (SEM) observation, differential scanning calorimeter (DSC) analysis, dynamic mechanical thermal analysis (DMTA), and rheological testing in sequence. The results showed that the addition of MAH-g-POE could improve the dispersion of clay layers in PP matrix and promoted the interaction between PP mole-

cules and clay layers. At 10 wt % MAH-g-POE, the PP–clay blend exhibited a highest value of $T_{c,onset}$ and T_g as well as a biggest melt storage modulus (G'), indicating the greatest PP–clay interaction. On the other hand, improved toughness and stiffness coexisted in blends with 5–10 wt % loading of MAH-g-POE. In view of SEM and DMTA observations, MAH-g-POE was well miscible with the PP matrix, even with the concentration up to 20 wt %. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 99: 2558–2564, 2006

Key words: polypropylene; clay; blends; miscibility; maleic anhydride grafted poly(ethylene-co-octene)

INTRODUCTION

Recently, polymer–clay nanocomposites have attracted considerable attention in thermoplastic industry because of their outstanding properties such as high stiffness and barrier resistance.¹ Furthermore, these improvements have been achieved at very low loading of about 5 wt % clay, which will be of great significance to produce lighter parts for industrial applications.^{2,3}

It is well known that the properties of polymer–clay composites are strongly relative, with the distribution of nanoclay in matrix. The more homogeneously the clay layers disperse in PP matrix, the better performances does the blend have.^{4,5} However, since PP does not have any polar group in its backbone, the homogeneous dispersion of clay platelets in PP matrix is not easy to be done via melting intercalation, even with organophilically modified clay.⁶ So, it is necessary to blend in a third component to improve the dispersion of clay layers in PP matrix. Until now, maleic anhydride grafted polypropylene (MAH-g-PP) has been widely used as a compatibilizer in PP–clay blends. It wets the modified clay surface more readily with the aid of the hydrogen bonding between MAH groups of PP chains and hydroxyl groups on clay

platelets while being miscible with the PP matrix. Nevertheless, the improvements of stiffness and modulus of these PP–clay blends compatibilized with MAH-g-PP are accompanied by a marked decrease in toughness, which greatly obstructs their applications. Consequently, a new compatibilizer is anticipated to compensate for the deficiency of PP–clay composites.

Poly(ethylene-co-octene) (POE), which was developed using a metallocene catalyst by Dow and Exxon, is a polyolefin elastomer with a narrow molecular weight distribution and homogenous octane distribution and often blended with other polymers by means of functional groups used for compatibilization, such as maleic anhydride (MAH) and epoxy.^{7–11} To our knowledge, MAH-grafted-POE (MAH-g-POE) has succeeded in improving the compatibility and mechanical properties of incompatible blends. For example, Zeng et al.¹² investigated the PP/PA6 blends and found that MAH-g-POE could improve the miscibility between PP and PA6 and achieved high performance PP/PA6 blends. Bai et al.¹³ studied the PP/PA6/POE ternary blends and concluded that the desired properties of the blends could be obtained through variations in PA6 and MAH-g-POE percentages in the blends. Li et al.¹⁴ studied the ternary PP/POE/BaSO₄ blends and found that the in situ formed MAH-functionalized POE could make POE compatible with BaSO₄, and good toughness of the blend was therefore obtained. But, so far, there is no report of MAH-g-POE in compatibilization of clay layers with PP matrix.

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TABLE I
Composition of the Blends Studied

Blend code	PP (wt %)	MAH-g-POE (wt %)	Clay (wt %)
PP	100	0	0
PPMC0	95	0	5
PPMC1	90	5	5
PPMC2	85	10	5
PPMC3	80	15	5
PPMC4	75	20	5
PPM	85	15	0

PPMC0–4: the number 0–4 represents the weight ratio of MAH-g-POE to clay.

In this paper, the new PP–clay blends (containing 5 wt % organoclay) were prepared by melt blending with different amounts of MAH-g-POE compatibilizer from 0 to 20 wt % by increments of 5 wt %. The influence of MAH-g-POE on the miscibility of PP and clay in blends was investigated in terms of X-ray Diffractometer measurement (XRD), scanning electronic microscopy (SEM), differential scanning calorimetry (DSC), dynamic mechanical thermal analysis (DMTA), and rheological testing. The mechanical properties of PP blends could also be examined in view of DMTA. In addition, the miscibility of MAH-g-POE with PP matrix was assumed by means of SEM and DMTA.

EXPERIMENTAL

Materials

The base material for the composites was polypropylene homopolymer F401 (MFI = 2.5 g/10 min, β = -0.91 g/cm³) produced by Sinopec Yangzi Petro Chemicals (Nanjing, China). The compatibilizer used was maleic anhydride- (MAH) grafted polyethylene octane elastomer (POE Engage 8999, the product of Dupont Dow Elastomer Co.) provided by Shanghai Sunny New Technology Development Co., LTD., having 0.8% MAH content. The organophilic clay was C18-clay DK1 (modified by octadecyl amine) supplied by Fenghong clay Co. China, as particles with average size of 25 μ m.

Preparation of PP-clay composites

The PP–clay composites including 5 wt % clay were melt blended in a thermoplastic mixer of Rheocord 900, Haake, by varying the content of MAH-g-POE from 0 to 20 wt % by increments of 5 wt % as a compatibilizer. Their compositions and abbreviations were listed in Table I. The melt blending process was performed at 190°C with a rotor speed of 100 rpm for 15 min, and the obtained composites were compression-molded into pieces of 100 \times 100 \times 1 mm³ for measurements.

Characterizations

The interlayer distance of the organoclay in the composites was studied at ambient temperature by using a X-ray Diffractometer measurement (D/MAXIII, Japan), with a Cu K α radiation (λ = 0.154 nm). All the samples were scanned by plates over a 2θ range of 1–11° at a rate of 2°/min. When the XRD pattern exhibits a peak, it indicates that there exists a significant fraction of material with a regular stacked structure of clay layers. The basal spaces between the clay layers of different samples can be determined from the {001} reflection peak in the XRD patterns by applying Bragg's diffraction law.

The phase structures of the composites were examined by Hitachi-S-2150 scanning electron microscope on the etched surfaces of cryogenically fractured specimens. All samples were etched in heptane (28°C) for 3 h to remove the compatibilizer MAH-g-POE from the PP matrix and thus improve contrast between the phases. These samples were sputter-coated with gold-palladium alloy before viewing.

The thermal property of PP composites was analyzed on a DSC Q10 differential scanning calorimeter thermal analyzer (TA Instruments). Test specimens (4–10 mg) were first heated from 20 to 210°C at a rate of 20°C/min and then maintained there for 5 min to eliminate the effects of preconditions. In succession, the samples were cooled down to 20°C at a rate of 10°C/min (crystallizing cycle), after that heated again from 20 to 210°C at a rate of 10°C/min (melting cycle). All the experiments were carried out under a nitrogen protection. From the thermograms, transition temperatures and enthalpies can be obtained and the degree of crystallinity also determined.

The dynamic mechanical behavior of the PP composites was studied on Rheometrics Dynamic Mechanical Analyzer (DMTA IV). Test samples were cut into bars (about 15 mm length, 4 mm width) from the compression-molded plaques. The experiments were performed in single point bending mode over a wide frequency range (0.1, 1, and 10 Hz), with a temperature range from -100 to 150°C and a heating rate of 3°C/min under a controlled sinusoidal strain. The dynamic mechanical properties, such as the storage modulus (E'), and the loss factor ($\tan \delta = E''/E'$, E'' represents the loss modulus) were recorded as a function of temperature and frequency. The activation energies of PP chain segments in different blend were also calculated.

The rheological properties of the compounds were investigated using Rheometric Scientific, ARES, with 25 mm diam parallel plates. The tests of nonlinear behavior of all the samples were performed in the range of deformation from 0.01 to 100% at 1 Hz and 190°C under ambient atmosphere, and the shear storage modulus (G') of the PP mixtures was measured

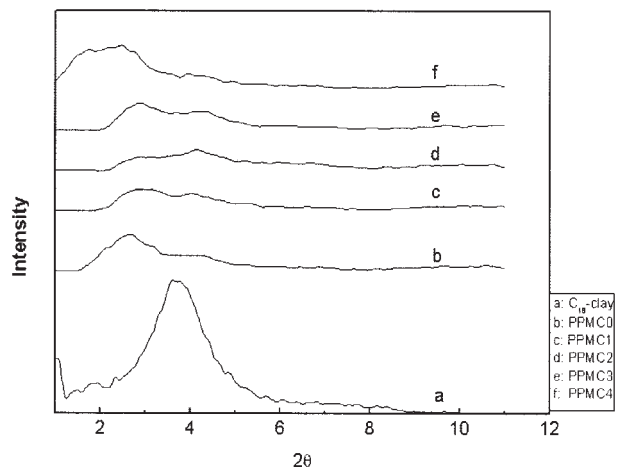


Figure 1 X-ray diffraction patterns of organoclay and its composites.

versus strain amplitude. Before test, samples were annealed between the parallel plates for 5 min.

RESULTS AND DISCUSSION

Clay distribution

Figure 1 shows the XRD patterns for the pristine C18-clay and its composites with different contents of the MAH-g-POE. C18-clay exhibits a single peak at 3.7° (2.4 nm basal space) before compounding. As shown in Figure 1, PPMC0 exhibited two diffraction peaks at about 2.7° and 4.2° , corresponding to a basal space of 3.2 and 2.1 nm, respectively. The increase of the basal space in PPMC0 indicates that without any compatibilizer, some PP molecules could intercalate into the interlayer of clay during melt blending. For PP-clay blends compatibilized with MAH-g-POE, the peak area widened and weakened with increasing MAH-g-POE loading from 5 to 10 wt %. It could be supposed that the strengthened interaction between the clay and PP matrix induced more clay layers exfoliated from the stacked structure of clay. However, when the concentration of MAH-g-POE increased from 15 to 20 wt %, the diffraction peak of blends appeared to be stronger and broader and shifted to a smaller angle. It could be assumed that the exfoliated action of clay layers was weakened with above 10 wt % concentration of MAH-g-POE, while the interlayer spacing of clay was expanded by the intercalation of MAH-g-POE.

As reported,^{15,16} the concentration of the intercalated clay layers with a given spacing is proportional to the area under the corresponding XRD peak, while the exfoliated structure of clay does not display any peaks in its XRD pattern. Comparing the XRD peaks presented here, the greatest exfoliation degree of clay layers in PPMC2 is quite likely to indicate the strongest surface interaction between the clay and PP matrix.

Phase structure

SEM micrographs of the etched cryogenic fracture surfaces of PP and PP-clay blends with different amounts of MAH-g-POE were shown in Figure 2. Some agglomerate structure of clay was observed in PPMC0, but for PP-clay blends compatibilized with MAH-g-POE, the agglomerate structure of clay disappeared gradually as the concentration of MAH-g-POE increased from 5 to 20 wt %. From these results, it is clear that the addition of MAH-g-POE could considerably improve the miscibility of clay layers with the PP matrix. On the other hand, the dark holes of MAH-g-POE were not visible in PP-clay blends with less than 10 wt % MAH-g-POE. When the MAH-g-POE increased up to 20 wt %, several small holes of MAH-g-POE gradually appeared without clear boundary from the PP matrix, as can be observed from Figures 2(c)–2(f). On the basis of these micrographs, it could be concluded that the MAH-g-POE molecules were principally bonded within the clay platelets below 10 wt % loading, and therefore not easy to be dissolved away by the heptane, from the whole blend. However, in PP-clay blends with 20 wt %, the removed holes of

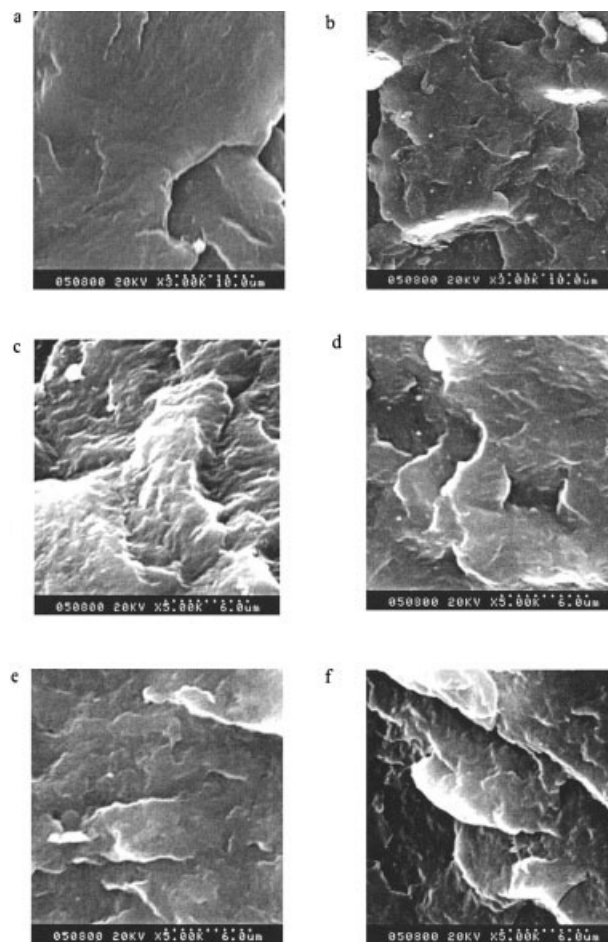


Figure 2 Cryogenic fractured and etched surfaces of PP and its composites: (a) PP, (b) PPMC0, (c) PPMC1, (d) PPMC2, (e) PPMC3, and (f) PPMC4.

TABLE II
Thermal Properties of PP and its Blends

Sample	$T_{c,onset}$ (°C)	T_c (°C)	$T_{m,onset}$ (°C)	T_m (°C)	ΔH (J/g)	ΔH_m (J/g wt % PP)	Crystallinity (%)
PP	117.7	112.3	157.1	167.8	90.2	90.2	43.2
PPMC0	120.8	117.2	158.7	164.8	85.8	90.3	43.2
PPMC1	121.6	116.0	158.4	166.7	69.5	77.2	36.9
PPMC2	123.3	118.8	158.8	165.4	69.7	82.0	39.2
PPMC3	119.7	113.8	157.0	165.5	68.7	87.1	41.7
PPMC4	121.9	117.3	157.8	165.0	61.6	82.1	39.3
PPM	119.9	115.6	158.1	165.3	68.4	80.5	38.5

$T_{c,onset}$ onset crystallization temperature; T_c , peak crystallization temperature; $T_{m,onset}$ onset fusion temperature; T_m , peak melting temperature; ΔH , heat of fusion of the blend; ΔH_m , heat of fusion of the PP in the blends.

MAH-g-POE were less than 150 nm, which hints the good miscibility of MAH-g-POE with the PP matrix, even at very high loading of 20 wt %.

Crystallization and fusion

Table II summarizes the DSC results for PP and its composites. As seen from the data, it is clear that adding either 5 wt % clay or 15 wt % MAH-g-POE to PP led to a slight increase in the values of $T_{c,onset}$ and T_c for PP crystallization. However, the effect on $T_{c,onset}$ caused by C18-clay was a bit higher than that by MAH-g-POE, which implies that C18-clay played preferable nucleation role in PP crystallization. For PP-clay blends with different content of MAH-g-POE, the values of $T_{c,onset}$ and T_c first increased with the increase of MAH-g-POE content from 5 to 10 wt %, and then decreased as the MAH-g-POE content further increased. It could be inferred that the nucleation effect of clay layers on PP crystallization in blends was promoted to the highest degree at 10 wt % loading of MAH-g-POE. According to the previous analysis of the clay distribution, the variation of $T_{c,onset}$ and T_c in PP-clay blends compatibilized with different amounts of MAH-g-POE was closely associated with the exfoliation degree of clay layers. A highest increase of $T_{c,onset}$ and T_c for PP crystallization corresponded to the greatest degree of clay delaminated structure in PPMC2. With the content of MAH-g-POE from 15 to 20 wt %, the slight decrease of $T_{c,onset}$ indicates the weakened nucleation effect of clay on PP crystallization in blends, which are corresponded to the more intercalated structure of clay distribution in these blends. This could be explained by the weakened interaction between clay layers and PP molecules attributable to the excessive incorporation of MAH-g-POE.

As can be seen from the data in Table II, $T_{m,onset}$ of the PP blends shifted to a higher temperature and T_m to a lower temperature, compared with the plain PP. From these results, it could be assumed that the formative PP spherulites in blends were more regular and smaller than the pure PP crystals. Additionally, the

effect of the MAH-g-POE concentration on $T_{m,onset}$ is similar to the influence on $T_{c,onset}$; a maximum value observed at 10 wt % MAH-g-POE.

The relative crystallinity of the blends was calculated from the ratio of the enthalpy of fusion for the blend to the enthalpy of fusion for a theoretically 100% crystalline PP, taken as 209 J/g,^{17,18} and adjusted for the nominal mass fraction of PP in the blends. The results showed that PPMC0 blend had the same relative crystallinity as PP, while PP-clay blends with different amounts of MAH-g-POE showed a slight decrease in the relative crystallinity with respect to the pure PP.

In view of the DSC analysis, it can be proposed that 10 wt % MAH-g-POE was an optimum concentration for the affinity of PP molecules and clay layers in its blend, which is consistent with the dispersion analysis of clay layers in PPMC2.

Dynamic mechanical properties

DMTA is an important means, which could not only measure the dynamic mechanical properties of a material but could also detect changes in the solid structure of a polymer after compounding with other materials. Figures 3 and 4 depict the variation in loss factor ($\tan \delta$) and storage modulus (E') as a function of temperature at 10 Hz for PP and its composites.

As shown in Figure 3(a), one clearly resolved peak was evident for PP itself in the temperature range examined, centered at about 9.5°C and derived from the glass transition of the PP amorphous phase. The $\tan \delta$ curve for MAH-g-POE alone shows a sharply intense peak at -22.1°C, associated with the glass transition process of MAH-g-POE. For PPMC0, one $\tan \delta$ peak, attributed to the glass transition of PP, shifted to a higher temperature of 11.1°C, indicating that organoclay had some restriction on the mobility of PP chain segments. For PP-clay blends with different amounts of MAH-g-POE with respect to PPMC0, the T_g value moved to a maximum temperature of 15°C when MAH-g-POE presented at 10 wt % loading, and then slowly decreased to 12.0°C when MAH-g-

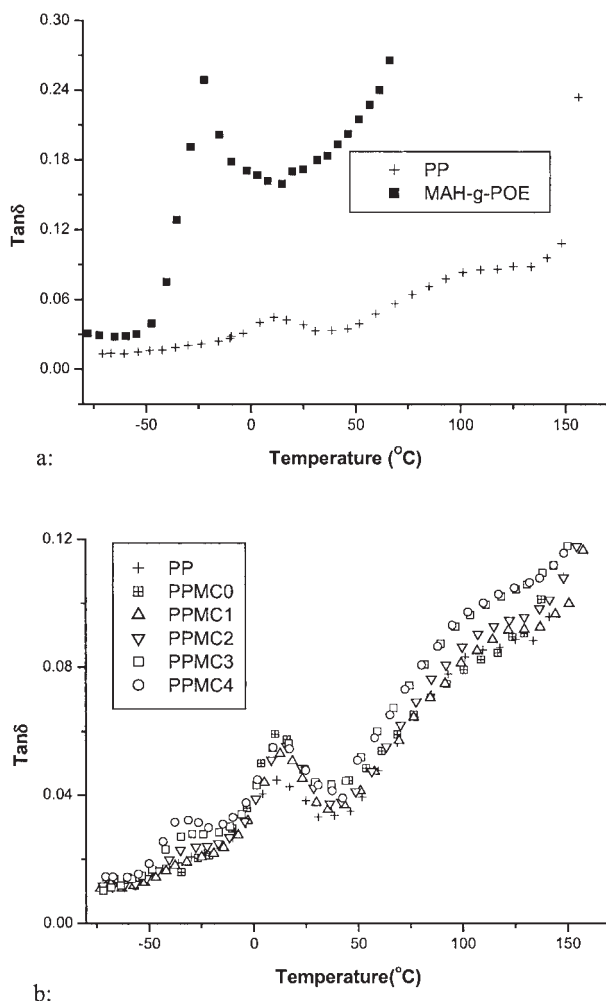


Figure 3 $\tan \delta$ of samples as a function of temperature: (a) PP and MAH-g-POE, (b) PP and its blends.

POE increased up to 20 wt %. As reported,¹⁹ the magnitude of T_g of a polymer depends on the mobility of the macromolecule chain segment in the polymer matrix, and if the molecular chain is restricted, motion or relaxation of the chain segment becomes difficult and so the glass transition temperature increases. According to the variation of PP's T_g in blends, it is clear that the geometric constraints of clay layers on the mobility of PP chain segments was intensified by MAH-g-POE. This is such that the constraint of clay attained a highest degree in PPMC2 accompanied with a highest T_g of PP, which reflected the strongest interaction of clay and PP in this blend. This is in agreement with the deduction from the variation of PP's $T_{c,onset}$ in PPMC2, closely relative to the high degree exfoliation structure of clay layers.

For PP-clay blends with MAH-g-POE from 5 to 20 wt %, the $\tan \delta$ curve showed a peak at very low temperature (-30 to -34°C), associated with the glass transition of the MAH-g-POE phase, and the peak became better resolved with increasing of the MAH-g-POE loading, which implies that good impact

strength and toughness were endowed by MAH-g-POE in these blends especially at low temperature, compared with the pure PP. In addition, the shifting of the peak maximum of the MAH-g-POE glass transition to a lower temperature and the significant reduction of the peak intensity could suggest that MAH-g-POE was miscible with the PP even at higher MAH-g-POE loading of 20 wt %, similar to the results reported by McNally.¹⁷ This is in good agreement with the observations in SEM, the dark holes of MAH-g-POE was not clearly visible even when MAH-g-POE was presented at 20 wt %.

The glass transition temperature (T_g) and the $\tan \delta$ values at T_g peak are also reported in Table III. A considerably higher $\tan \delta$ value can be observed at about -30 to -34°C in PP-clay blends compatibilized with more MAH-g-POE. It indicates that there exist some motion or relaxation of the chain segment in these PP-clay blends at such low temperature, which could suggest the improved toughness obtained in these blends at very low temperature.

The storage modulus (E') of MAH-g-POE is markedly lower than either PP itself or any PP-clay blend,

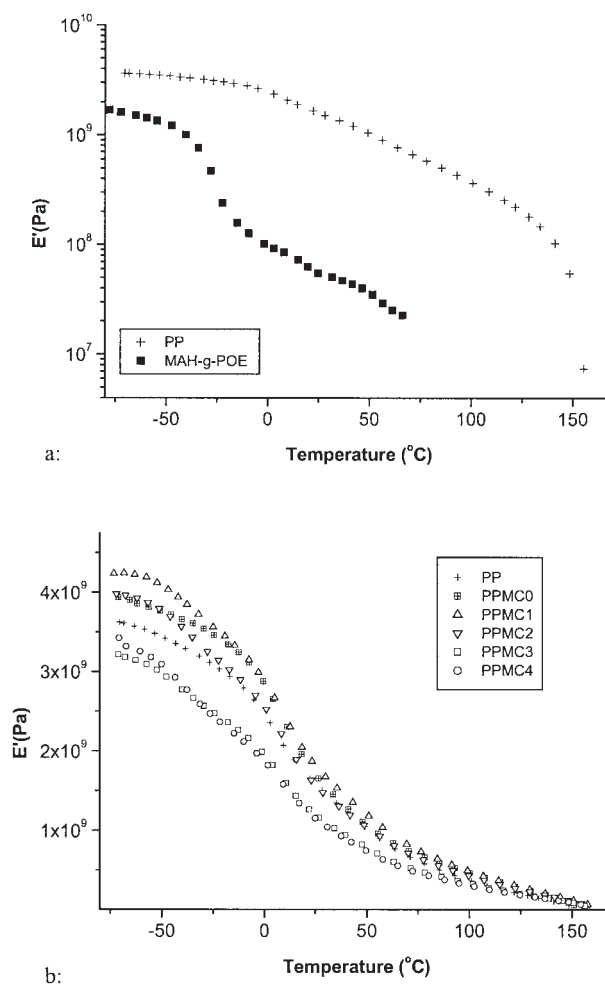


Figure 4 E' of samples as a function of temperature at 10 Hz: (a) PP and MAH-g-POE, (b) PP and its blends.

TABLE III
Dynamic Mechanical Properties of the Studied Materials at 10 Hz

Sample	$E' (\times 10^9 \text{ MPa})$			$T (\text{°C})/\tan \delta (\times 10^{-2})$ at peak of MAH-g-POE	$T_g (\text{°C})/\tan \delta (\times 10^{-2})$ at peak of PP
	-68°C	45°C	124°C		
PP	3.6	11	0.2	—	9.5/5.0
PPMC0	3.9	1.2	0.26	—	11.1/6.0
PPMC1	4.2	1.3	0.32	-30.1/2.0	13.6/5.3
PPMC2	4.0	1.1	0.26	-32.2/2.4	15.0/5.4
PPMC3	3.2	0.86	0.20	-33.9/2.6	12.1/4.5
PPMC4	3.2	0.84	0.19	-34.0/3.2	12.0/5.4
MAH-g-POE	1.55	0.05	—	-22.1/24.9	—

as observed in Figure 4. When the MAH-g-POE was incorporated between 0 and 10 wt %, the E' of the PP-clay blends was higher than that of PP, which is directly attributed to the reinforced effect of clay, while the E' decreased in PP-clay blends with the concentration of MAH-g-POE above 10 wt %, which is due to the dominant plasticization of MAH-g-POE. To clearly see the variation of the storage modulus of the studied composites, the E' of different blends at -68, 45, and 124°C are listed in Table III. As seen in Table III, PP-clay blends with 5–10 wt % MAH-g-POE exhibited a higher stiffness with respect to the pure PP.

The relationship between the temperature at which a relaxation process is observed (T) and the frequency of excitation (f) can be given by the Arrhenius equation²⁰

$$f = f_0 \exp \frac{-\Delta E_a}{RT} \quad (1)$$

where f_0 is a constant, R is the gas constant (8.314 J/(K mol)) and ΔE_a is the activation energy for the relaxation process.

According to this equation, a plot of $\log f$ versus $1000/T$ (Fig. 5), should give a straight line with a slope that is proportional to the apparent activation energy

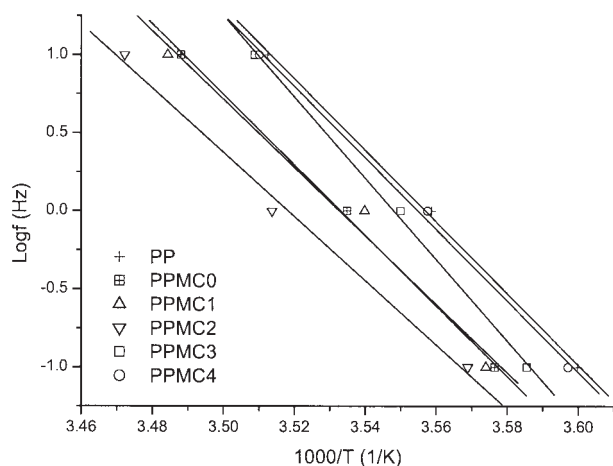


Figure 5 Calculation of the activation energies.

for relaxation process of PP and their composites. Calculated activation energies according to the eq. (1) are shown in Table IV. From the result, it seems that either clay or the incorporated MAH-g-POE had little effect on the ΔE_a of PP chain segments in blends.

On the basis of the dynamic mechanical analysis, the concentration of 10 wt % MAH-g-POE added to PP-clay blend was reasonably thought to be an optimum incorporation to improve the affinity of PP and clay layers and good mechanical properties came about as a consequence. It is in good agreement with the observations from DSC measurements.

Rheological behavior

A strain sweep dynamic test in which the sample is subjected to increasing oscillatory strain amplitude at controlled frequency can differentiate the linear and nonlinear regimes. The critical strain, corresponding to the transition from the linear to nonlinear regime characterized by a rapid decrease in storage modulus, represents the breakdown of the structure of melt polymer.^{21,22} Figure 6 showed the storage modulus (G') obtained from strain sweep tests for PP and its composites. As shown in Figure 6, the storage modulus of the pure PP was not strain dependent in a high strain region for strain <31%, and subsequently slowly decreased from 10,914 to 9877 Pa with increasing the strain deformation above the critical strain. In PPMC0, the linear strain regime slightly reduced to 24%, while the storage modulus exhibited a rapid

TABLE IV
Relaxation Activation Energies of the Investigated Samples

Sample	Activation energy (kJ/mol)
PP	190.5
PPMC0	187.7
PPMC1	182.0
PPMC2	170.8
PPMC3	214.2
PPMC4	190.4

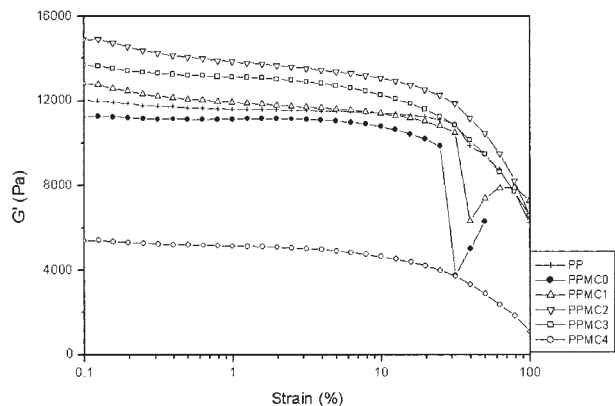


Figure 6 Storage modulus of PP and its composites in a strain sweep experiment at frequency of 1 Hz and $T = 190^{\circ}\text{C}$.

decrease from 9868 to 3769 Pa in the transition from the linear to nonlinear regime, which was similar to the molten PPMC1 by the fast decrease from 10,500 to 6331 Pa, compared with the pure PP melt. This phenomenon may be explained in terms of the breakdown process occurring in the clay agglomerates, as seen in SEM with distinct agglomerates of clay dispersed in PPMC0 and PPMC1. Nevertheless, the storage modulus of PP-clay blends compatibilized with MAH-g-POE from 10 to 20 wt % gradually decreased as the strain deformation increased from linear to nonlinear area, further indicating the improving PP-clay miscibility in blends. In the linear strain regime, the trend of G' in PP-clay blends with the loading of MAH-g-POE was almost the same as the observations of $T_{c,onset}$ and T_g of PP in different blends. The value of G' first increased with increasing MAH-g-POE because of the reinforced effect stemmed from the improving dispersion of clay layers. Secondly, the G' reached a maximum value at 10 wt % loading of MAH-g-POE, indicating the good synergic effect of clay and MAH-g-POE in PP matrix. Finally, the G' greatly decreased as MAH-g-POE increased up to 20 wt %, far below that of the plain PP owing to the excessive MAH-g-POE. From these results, it is obvious that the modulus of different PP-clay blends is sensitive to the concentration of MAH-g-POE and as a consequence the optimum effect of MAH-g-POE was easier to be examined than any other measurements before. Additionally, the critical strain of the compatibilized PP-clay blends reduced from 31 to 10% with increasing the concentration of MAH-g-POE.

CONCLUSIONS

In this study, PP-clay blends compatibilized with different amounts of MAH-g-POE was investigated in

detail, and the effect of the concentration of MAH-g-POE on the PP-clay interaction was discussed also. From XRD patterns and SEM photographs, the degree of compatibilization of clay with the PP matrix was raised by the incorporation of the MAH-g-POE compatibilizer. DSC and DMTA observations showed that when 10 wt % MAH-g-POE was added, the PP-clay interaction was promoted to the fullest extent and the blend displayed a highest $T_{c,onset}$ and T_g . By rheological testing, the PP-clay blend with 10 wt % MAH-g-POE exhibited a maximum G' . Furthermore, good toughness and stiffness were coexistent in PP-clay blends compatibilized with 5–10 wt % MAH-g-POE because of the synergic effect from clay layers and MAH-g-POE. In terms of SEM and DMA examinations, MAH-g-POE was well miscible with the PP matrix, even at a higher loading of 20 wt % MAH-g-POE.

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