Rheological and Thermal Properties of Thermoplastic Natural Rubbers Based on Poly(methyl methacrylate)/ Epoxidized-Natural-Rubber Blends

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ABSTRACT: Epoxidized natural rubbers (ENRs) with epoxide levels of 10, 20, 30, 40, and 50 mol % were prepared. The ENRs were later blended with poly(methyl methacrylate) (PMMA) with various blend formulations. The mixing torque of the blends was observed. The torque increased as the PMMA contents and epoxide molar percentage increased in the ENR molecules. Furthermore, the shear stress and shear viscosity of the polymer blends in the molten state increased as the ENR content and epoxide molar percentage increased in the ENR molecules. Chemical interactions between polar groups in the ENR and PMMA molecules might be the reason for the increases in the torque, shear stress, and viscosity. All the ENR/PMMA blends exhibited shear-thinning behavior. This was observed as a decrease in the shear viscosity with an increase in the shear rate. The power-law index of the blends decreased as the ENR contents and epoxide molar percentage increased in the ENR molecules. However, the consistency index (or zero shear viscosity) increased as the ENR contents and epoxide molar percent-

INTRODUCTION

Thermoplastic elastomers (TPEs) are relatively new products in the rubber industry and most likely are the fastest growing sector in the polymer market. They exhibit properties typical of elastomeric materials but can be processed with thermoplastic processing equipment. Such materials can be produced either as block copolymers or as blends. Most polyolefin TPEs are based on ethylene–propylene–diene monomer (EPDM) or a modification of it. Some are commercially manufactured by companies such as Uniroyal, Inc., Goodrich, DuPont, Hercules, and Allied Chemical.¹ Interest in natural rubber (NR) and thermoplastic blends has also increased recently. These materials are also known as thermoplastic natural rubbers (TPNRs). Thermoplastics blended age increased. A two-phase morphology was observed with scanning electron microscopy. The small domains of the minor components were dispersed in the major phase. For the determination of blend compatibility, two distinct glass-transition-temperature (T_g) peaks from the tan δ /temperature curves were found. Shifts in T_g to a higher temperature for the elastomeric phase and to a lower temperature for the PMMA phase were observed. Therefore, the ENR/PMMA blends could be described as partly miscible blends. According to the thermogravimetry results, the decomposition temperatures of the blends increased as the levels of ENR and the epoxide molar percentage increased. The chemical interactions between the different phases of the blends could be the reason for the increase. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 92: 3561–3572, 2004

Key words: thermoplastics; rubber; rheology; thermal properties

with NR include polypropylene,^{2–6} linear low-density polyethylene,⁷ polystyrene,⁸ polyamide ^{6,9} poly(methyl methacrylate) (PMMA),¹⁰ and ethylene-vinyl acetate copolymer (EVA).¹¹ Apart from raw NR, modified forms of NR have also been used to prepare TPNRs. Epoxidized natural rubber (ENR) is one of the modified forms currently used to prepare TPNRs. This is done through the blending of ENR with thermoplastics such as poly-(vinyl chloride)^{12–15} and poly(ethylene-*co*-acrylic acid).¹⁶ Graft copolymers of NR with PMMA,^{16,17} polystyrene,⁸ and polyacrylonitrile¹⁷ have also been used to prepare TPNRs.

TPNRs of NR and PMMA blends combine the excellent processability characteristics of PMMA and the elastic properties of NR. In addition, they can be successfully used for the fabrication of automobile components. However, the NR and PMMA components are highly incompatible and hence exhibit poor mechanical properties. Therefore, the technological compatibilization of the immiscible pairs is necessary. The properties of the blends can be improved if the NR molecule is modified to be more polar. This will lead to more interchain interactions between the NR and

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TABLE I				
Blend	Formulation	for	ENR/PMMA	Blends

			Bl	lend co	de		
Ingredient	1	2	3	4	5	6	7
ENR ^a PMMA	0 100	20 80	40 60	50 50	60 40	80 20	100 0

^a Various types of ENR (i.e., ENR-10, ENR-20, ENR-30, ENR-40, and ENR-50).

PMMA molecules via the polar groups of each polymer.

In this study, an attempt was made to prepare TPNRs based on ENR and PMMA blends. ENRs with various levels of epoxide or epoxirane rings were used. Furthermore, the rheological, thermal, and morphological properties of the blends were investigated.

EXPERIMENTAL

Materials

The PMMA used in this study was an injection-molding-grade, and it was manufactured by TPI Polyacrylate Co., Ltd. (Rayong, Thailand), with a melt-flow index of 14 g/10 min at 230°C. High-ammonia concentrated NR latex was used as a raw material for the preparation of ENRs (Pattani Industry Co., Ltd., Pattani, Thailand). The nonionic surfactant used to stabilize the latex during epoxidation was Teric N30 (alkylphenol ethoxylate), which was manufactured by Huntsman Corp. Australia Pty, Ltd. (Ascot Vale Vic, Australia). The formic acid used as a reactant for the preparation of ENRs was manufactured by Fluka Chemie (Buchs, Switzerland). The hydrogen peroxide used as a coreactant for the preparation of ENRs was manufactured by Riedel-de Haën (Seelze, Germany).

Preparation of ENRs

The ENR latex was prepared with high-ammonia concentrated NR latex with a dry rubber content of approximately 60%. Details for the preparation process of ENRs have been reported elsewhere.^{18,19} The reaction time was set according to the level of epoxide groups in the ENR products as described in our previous work.¹⁹ The level of the epoxide content in the ENR products was later confirmed by infrared spectroscopy via a calibration curve.

Mixing procedure

Blends of ENRs and PMMA were prepared via melt mixing with a Brabender PLE 331 plasticorder (Duisberg, Germany). Mixing was performed at 160°C. PMMA was first preheated for 6 min in the mixer without rotation of the rotors. The polymer was then melted for 2 min at a rotor speed of 40 rpm. ENR was added, and mixing was continued for 5 min. The torque versus the mixing time was plotted for each blend. The blend products were later cut into small pieces with a Bosco plastic grinder (Samutprakarn, Thailand). Various types of ENRs (i.e., ENR-10, ENR-20, ENR-30, ENR-40, and ENR-50) were used in this study. Furthermore, various rubber contents (i.e., 20, 40, 50, 60, 80, and 100 wt %) were blended with PMMA according to the blend formulations shown in Table I.

Rheological characterization

A Rosand RH7 single-bore capillary rheometer (Gloucestershire, UK) was used to characterize the shear-flow properties in terms of the shear stress and shear viscosity. The tests were carried out over a wide range of shear rates $(25-1600 \text{ s}^{-1})$ at a test temperature of 200°C. The capillary die was 2 mm in diameter and 32 mm long; it had a 180° entrance angle and an aspect ratio (length/diameter) of 16/1. The materials were first preheated in a barrel for 7 min under a pressure of approximately 2–4 MPa to obtain a compact mass. The excess molten material was automatically purged until no bubbles were observed. The test was then carried out at a set of shear rates in a program via a microprocessor. The pressure drop across the capillary channel and the melt temperature were captured via a data acquisition system during the test. The equations used to calculate the shear stress, shear viscosity, and shear rate are described in our previous articles.^{18,19,20,21}

Morphological studies

Morphological studies were carried out with a scanning electron microscope (Leo 1450 VP, Cambridge, UK). Molded samples of the blends were broken in liquid nitrogen to avoid any possibility of phase deformation. The minor phase of PMMA or ENR was preferentially extracted with acetone or chloroform, respectively. The samples were dried in an oven at



Figure 1 Epoxide-group levels in ENRs at various reaction times.



Figure 2 Torque-time curves for ENR-30/PMMA blends.



Figure 3 Effect of the shear rate on the melt viscosity of ENR-30/PMMA blends at various blend ratios.



Figure 4 Torque-time curves for 60/40 ENR/PMMA blends with various ENRs.

45°C for 48 h. The dried surfaces were gold-coated and then examined by scanning electron microscopy (SEM).

Thermal property characterization

Two techniques were used to follow the thermal behavior of the TPNRs: thermogravimetry and dynamic mechanical analysis (DMA). The thermogravimetric analysis was carried out under a nitrogen atmosphere at a heating rate of 20°C/min in a Shimadzu TGA-50 thermal analyzer (Kyoto, Japan). A constant sample weight was used in all cases. Thermograms were recorded from room temperature to 800°C. The temperature at which the rate of mass loss was at a maximum



Figure 5 Effect of the shear rate on the melt viscosity of various ENRs in 60/40 ENR/PMMA.



Figure 6 Relationship between the shear stress and shear rate of ENR-30/PMMA blends at 200°C.



Figure 7 Relationship between the shear stress and shear rate of ENR/PMMA blends with various ENRs at 200°C.



Figure 8 Relationship between the shear viscosity and the epoxide-group level in NR molecules at various blend ratios and at a shear rate of 100 s^{-1} .

was evaluated from the differential thermogravimetry (DTG) curve and called the decomposition temperature (T_d) . The DMA measurements were carried out on a PerkinElmer DMA7 instrument (Boston, MA). The experiment was conducted in the tension mode. A temperature-time scan was carried out from -80 to +150°C at a frequency of 1 Hz. The heating rate was 2°C/min. Liquid nitrogen was used to achieve a subambient temperature.

RESULTS AND DISCUSSION

Preparation of ENRs

Figure 1 shows the relationship between the level of epoxide groups in ENR molecules and the reaction time. The epoxide groups increased in number with increasing reaction time. Therefore, ENRs with epoxide contents of 10, 20, 30, 40, and 50 mol % were prepared at reaction times of 2, 6, 12, 16, and 22 h, respectively.

Mixing torque

The torque-time curves of ENR-30/PMMA blended with various rubber contents are shown in Figure 2.

TABLE II

PMMA was first charged into the mixing chamber and preheated for 6 min without rotation of the rotor. This was to warm and dry the material. The rotors were later started, and mixing was continued for 2 min. The torque rose sharply when the rotors were started because of the resistance exerted on the rotors by unmelted PMMA. A reduction of the torque was later observed because PMMA was melted on account of mechanical shearing and heat transfer from the heating oil. Furthermore, the levels of the mixing torque increased with increasing contents of PMMA in the blend composition. This was the influence of the quantities of PMMA in the mixing chamber. A higher free volume in the mixing chamber was observed for a lower quantity of PMMA. At a mixing time of 8 min, ENR-30 was incorporated. The torque rose again because of unmelted ENR and the dispersion of ENR in the PMMA matrix or PMMA in the ENR matrix (in that case, ENR was a rich phase). As the ENR was blended well with PMMA, the torque started to decrease and level off at an approximate mixing time of 12 min. A comparison of the levels of mixing torque showed that the torque increased with an increase in the ENR concentration. This may be attributed to a higher shear viscosity for pure ENR-30 than for pure

n and K Values for ENR-30/PMMA Blends at 200°C n Blend code п Κ 0.40 1 23,687 2 0.37 34.868 3 0.3064,112 4 0.29 73,141 5 0.28 81,900 6 0.24 113,925 7 0.21 190,558

TABLE III
and K Values for ENR/PMMA (60/40) Blends at 200°C
with Various Types of FNRs

with valious types of Livins			
п	K		
0.31	43745		
0.29	74510		
0.28	81900		
0.27	91753		
0.26	108933		
	n 0.31 0.29 0.28 0.27 0.26		



Figure 9 *n* for ENR-30/PMMA blends with various blend ratios.

PMMA, as shown in Figure 3. Furthermore, the shear viscosities of ENR-30/PMMA blends were ranked as a combination of the shear viscosities of ENR-30 and PMMA. That is, the mixing torque of the ENR-30/PMMA blends was ordered as follows: 80/20 > 60/40 > 40/60 > 20/80.

Figure 4 shows the torque–time curves for various types of ENRs with a fixed blend ratio of ENR/PMMA = 60/40. A similar trend for the mixing torque (as shown in Fig. 2) was found. In a typical case, the mixing torque increased with an increase in the level of epoxide groups in the ENR molecules. This can be

ascribed to an increasing trend of the shear viscosity with an increasing level of epoxide groups, as shown in Figure 5. Furthermore, the higher chemical interactions between the polar groups of ENR and PMMA might be a reason for the increasing trend of the relationship between the mixing torque and shear viscosity.

Shear-flow properties of ENR/PMMA blends

Plots of the shear stress versus the shear rate of ENR-30 blended with various contents of PMMA are



Figure 10 K for ENR-30/PMMA blends with various blend ratios.



Figure 11 *n* for ENR/PMMA blends with various ENRs.

shown in Figure 6. The shear stress increased with an increase in the shear rate. Furthermore, at a given shear rate, pure PMMA exhibited the lowest shear stress, but pure ENR showed the highest value. The shear stress of ENR/PMMA blends was between those of the two pure parent polymers. Moreover, the shear stress increased with an increasing level of ENR-30 in the blend formulation. That is, a higher flow curve (on a plot of the shear stress vs the shear

rate) was found when an increased concentration of ENR-30 was added to the blends. The influence of the epoxide groups in the ENR molecules on a plot of the shear stress versus the shear rate is shown in Figure 7. Higher flow curves were observed for blends with more epoxide groups in the molecules. That is, the flow curves were in the following order: ENR-10 > ENR-20 > ENR-30 > ENR-40 and ENR-50. The higher degree of chemical interaction between the po-



Figure 12 K for ENR/PMMA blends with various ENRs.



Figure 13 Etched cryogenic fracture surfaces of ENR-30/PMMA blends with various blend ratios.

lar groups of ENR and PMMA might be responsible for the increasing trends of the shear stress.

The shear viscosity of pure ENR-30 blended with PMMA in various blend ratios is shown in Figure 3. As for the shear stress, the shear viscosity of pure ENR showed the highest values, whereas pure PMMA exhibited the lowest. Increasing the level of ENR-30 caused the shear viscosity to increase at a given shear rate. That is, a higher viscosity curve (on a plot of the shear viscosity vs the shear rate) was observed when the concentration of ENR-30 increased in the blends. The influence of the levels of the epoxide groups in the



Figure 14 Relationship between tan δ and the temperature for various ENRs.

ENR molecules on the viscosity curves is shown in Figure 5. The higher viscosity curves were found for blends of ENR with more epoxide groups in the molecules. This corresponds to the case of the shear stress. Furthermore, a shear-thinning behavior of the flow was observed for all cases. That is, the shear viscosity decreased with an increase in the shear rate. This indicated a pseudoplastic (shear-thinning) behavior of the blends. This may be attributed to the randomly oriented and entangled polymer chains becoming oriented and disentangled upon the application of high shear rates, leading to a lowered melt viscosity. A comparison of the shear viscosity for the blends with various types of ENRs and blend ratios (i.e., ENR/ PMMA = 40/60, 50/50, and 60/40) is shown in Figure 8. The shear viscosity at a given shear rate (100 s⁻¹) increased when the level of the epoxide groups increased in the ENR molecules and the level of ENR increased in the blend composition. This confirms our findings described in Figures 3 and 5.

TABLE IV T_g Values of NR (ADS) and Various Types of ENRs

		Material				
	ADS	ENR-10	ENR-20	ENR-30	ENR-40	ENR-50
T_g (°C)	-63	-52	-34	-26	-18	-10



Figure 15 Relationship between tan δ and the temperature for ENR-30 blended with PMMA in various blend ratios.

To clarify the shear-thinning behavior of the polymer melts, we applied the power-law equation to the relationship between the shear rate and shear stress:²²

$$\tau = K(\dot{\gamma})^n \tag{1}$$

where *n* is the power-law index or flow-behavior index, $\dot{\gamma}$ is the shear rate, τ is the shear stress, and *K* is the consistency or viscosity coefficient index.

From the linear relationship of a log-log scale, we can get the slope (n) and intercept (K), which are shown in Tables II and III. Plots of *n* and *K* values against the quantity of ENR-30 in the blend composition are given in Figures 9 and 10, respectively. The *n* values of the blends decreased with increasing levels of ENR-30 in the blend formulation. This indicates greater shear thinning in the flow with increasing ENR-30 content. The value of n also reflects the deviation of the flow profiles from uniform parabolic flow patterns (i.e., n = 1 for Newtonian flow) to pluglike flow profiles. The blends in this work had very low *n* values (i.e., n < 0.40). Therefore, the highly pseudoplastic fluid flowed through the capillary almost as a plug moving at a uniform speed as the melt was sliding down against the channel wall. The influence of the level of the epoxide groups on the n values is shown in Figure 11. The n values decreased with

TABLE V T_g Values of ENR-30 and PMMA Blends with Various Types of ENRs

Sample	T_{g1}	T_{g2}
Pure PMMA	_	119
20/80 ENR/PMMA	-18	112
40/60 ENR/PMMA	-22	110
50/50 ENR/PMMA	-24	116
60/40 ENR/PMMA	-21	113
80/20 ENR/PMMA	-20	110

 T_{g1} = glass-transition temperature of the rubber phase; T_{g2} = glass-transition temperature of the PMMA phase. increasing levels of epoxide groups in the ENR molecules. This also indicates greater shear-thinning behavior in the flow with an increasing level of epoxide groups.

K is a Newtonian viscosity if *n* is 1. By definition, the *K* value is related to the zero shear viscosity (i.e., the shear viscosity at a shear rate of zero) of flowing polymer blends at a test temperature (200°C). *K* increased with the level of ENR in the blend composition (Fig. 10) and with the level of epoxide groups in the ENR molecules (Fig. 12).

Morphological studies

The etched cryogenic fracture surfaces of the ENR/ PMMA blends are shown in Figure 13. The minor phases of PMMA (i.e., at ENR/PMMA = 80/20 and 60/40) or ENR (i.e., at ENR/PMMA = 40/60 and 20/80) were preferentially extracted with acetone or chloroform, respectively. As a result, PMMA or ENR was dissolved at an elevated temperature for each type of blend and left some holes on the surfaces. The two-phase morphology is obvious. However, for the acetone-etched surface, the holes from the dissolved



Figure 16 Relationship between tan δ and the temperature for ENR-30 blended with PMMA in various blend ratios (tan δ was separated by the multiplication of a constant value).



Figure 17 Relationship between tan δ and the temperature for various ENRs blended with PMMA in a 60/40 ENR/PMMA ratio.

PMMA domains are not clear because of the swelling behavior of ENR-30 in the solvent. Therefore, a continuous phase (ENR-30) collapsed and covered the holes. The chloroform-etched fracture surface shows a clearer two-phase morphology in which the small particles of ENR domains dispersed in the PMMA matrix.

DMA

The variation of tan δ with the temperature for various types of pure ENRs and air-dried-sheet (ADS) NR is shown in Figure 14. The temperature at which damping (tan δ) peaks were observed is quoted as the glass-transition temperature (T_{q}) . Therefore, each type of ENR showed distinct peaks corresponding to the T_{o} values, as reported in Table IV. T_g of pure NR (ADS) was lower than those of ENRs. Furthermore, T_g of ENRs increased with an increase in the content of epoxide rings in the ENR molecules. This indicates a restriction of chain mobility due to the intermolecular forces exerted on the ENR molecules. The level of intermolecular forces depended on the concentration of polar groups in the ENR molecules. That is, higher epoxide contents caused lower chain mobility and hence higher T_g 's. This finding corresponds to the work of Gelling,²³ Baker et al.,²⁴ and Nakason et al.¹⁹ The variation of tan δ with the temperature for pure ENR-30, pure PMMA, and their blends at various blend ratios is shown in Figure 15 and Table V. Two distinct peaks corresponding to T_g values at low temperatures for ENRs and at high temperatures for PMMA were observed. To clarify the influence of the temperature on the glass-transition temperature of the blends, a given set of tan δ values for each pure polymer and blend was multiplied by a factor. Therefore, the tan δ /temperature curves of various blends and pure materials were separated and are clearly shown in Figure 16. A shift in the T_g 's was observed. That is, T_g of the elastomeric phase was shifted to a higher temperature, whereas T_g of the PMMA phase

was shifted to a lower temperature. The T_g values of both phases were slightly shifted for the 50/50 ENR/ PMMA blends. However, in the blends in which ENR or PMMA behaved as the minor phase, the shift of T_g was more pronounced. Therefore, a partially miscible blend system was observed for all blend types.

The tan δ /temperature curves for various types of ENRs blends with PMMA at ENR/PMMA = 60/40 are shown in Figure 17 and Table VI. We observed a shift in T_g for the rubber phase to a higher temperature and a shift in T_g for the PMMA phase to a lower temperature. The decreasing and increasing trends of T_g strongly depended on the level of epoxide groups in the ENR molecules. This was clearly evidence of a partly miscible blend between the ENR and PMMA.

Thermal degradation

The thermal degradation of pure PMMA, pure ENR and blends of these two polymer was investigated with thermogravimetric analysis. Figure 18 shows thermograms for various types of ENR blended with PMMA at ENR/PMMA = 50/50. The degradation of ENR-10 started and completed at the lowest temperature, whereas ENR-50 exhibited the highest temperature. Therefore, the starting T_d 's of the various types of ENR/PMMA blends can be ranked as follows:

TABLE VI T_g Values for Various Types of ENRs Blended with
PMMA at ENR/PMMA = 60/40

Sample	T_{g1}	T_{g2}
ENR-10	-50	117
ENR-20	-32	116
ENR-30	-24	115
ENR-40	-18	113
ENR-50	-9	108

 T_{g1} = glass-transition temperature of the rubber phase; T_{g2} = glass-transition temperature of the PMMA phase.



Figure 18 Thermograms for 50/50 ENR/PMMA blends with various ENRs.

ENR-10 < ENR-20 < ENR-30 < ENR-40 < ENR-50. A comparison of the thermograms for ENR-30 blends with various contents of PMMA is shown in Figure 19. The blend formulation with a lower content of ENR-30 exhibited a lower starting T_d . T_d of the ENR/PMMA blends was evaluated from the main weight loss obtained from DTG curves (not shown here); the results are given in Table VII. We also found that pure PMMA exhibited the lowest T_d (i.e., at 334.2°C), whereas pure NR (ADS) showed a T_d at 358.1°C. Therefore, it is clear that T_d 's of the ENR/PMMA blends were higher than those of the parent polymer pairs. Furthermore, T_d increased as the level of epoxide groups increased in the ENR molecules and the content of ENR increased in the blends, as shown in Figure 20. In the case of the polymer blends, the thermal degradation depended on the morphology and extent of the interaction between the different phases in the blends. From DMA and morphology results, we concluded that the ENR/ PMMA blends had two-phase morphologies and were partly miscible. The extent of the interaction depended on the concentration of polar functional groups in the ENR and PMMA molecules. Therefore, the increasing trend of T_d corresponded to the increasing degree of chemical interaction between the two phases.

CONCLUSIONS

ENRs with various levels of epoxide groups (i.e., 10, 20, 30, 40, and 50 mol % epoxide) were successfully

prepared. They were later used to prepare ENR/ PMMA blends with various concentrations of PMMA in the blend formulation. The mixing torque increased with an increase in the PMMA contents in the blend formulation and an increase in the epoxide molar percentage in the ENR molecules. This was a result of increasing chemical interactions between the polar groups for each blend component. We also found that both the shear stress and shear viscosity increased as the ENR contents and epoxide molar percentage increased in the ENR molecules. Furthermore, all the blend types exhibited pseudoplastic or shear-thinning behavior. That is, the shear viscosity decreased with an increase in the shear rate. *n* of the blends decreased as the ENR contents and epoxide molar percentage increased in the ENR molecules. However, the reverse effect was observed for K (or zero shear viscosity). That is, the latter value increased with the levels of ENR and epoxide molar percentage in the blend formulation. From SEM, two-phase morphologies were observed with a small domain of the minor component dispersed in the matrix. Two distinct T_{q} peaks for the blends were observed from the tan δ /temperature curves. Furthermore, we observed a shift in T_{q} to a higher temperature for the elastomeric phase and to a lower temperature for PMMA phase. It can be concluded that the ENR/PMMA blends were partly miscible. The thermal degradation of the blends was also investigated. T_d of the blends increased with the level



Figure 19 Thermograms for ENR-30 blends with various blend ratios.

TABLE VII T_d Values of Pure PMMA and Various Types of ENRsBlended with PMMA at a 50/50 Ratio

	T_d (°C)				
Sample	40/60 ENR/ PMMA	50/50 ENR/ PMMA	60/40 ENR/ PMMA		
ENR-10	361.7	362.6	367.7		
ENR-20	364.7	366.4	370.1		
ENR-30	370.7	373.5	374.4		
ENR-40	371.5	375.1	376.7		
ENR-50	373.5	377.2	378.9		



Figure 20 T_d values for 40/60, 50/50, and 60/40 ENR/PMMA blends with various ENRs.

of ENR and the epoxide molar percentage. This again might be the result of the increasing trend of the chemical interaction between the different phases of the blends.

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