

Dynamic Properties of Rubber Vibration Isolators and Antivibration Performance of Ethylene–Propylene–Diene Monomer/Nylon 6 Blend Systems

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ABSTRACT: This article examines thermoplastic elastomers (TPEs) and thermoplastic vulcanizates (TPVs) as two types of elastomers from melt-blended and dynamically vulcanized ethylene–propylene–diene monomer (EPDM) rubber materials and nylon 6 plastic materials. A series of investigations were conducted on the mechanical properties, morphology, dynamic mechanical properties, hysteresis behavior, and dynamic antivibration properties with different nylon 6 contents. The experimental results showed that the incompatibility between EPDM and nylon 6 led to the easy destruction of the TPV materials in two interfacial polymers upon the application of an external force. Thus, after a dynamic vulcanization process, the mechanical properties of the EPDM/nylon 6 blends were not as good as those of the TPE materials. In terms of

morphology, nylon 6 plastics were uniformly distributed in the EPDM/nylon 6 blends during the EPDM rubber phase before vulcanization was performed. After the dynamic vulcanization, phase inversion was produced in which rubber microparticles were formed and dispersed in the nylon 6 plastic phase. The results of dynamic mechanical analysis, compression vibration hysteresis behavior, and dynamic property antivibration experiments showed that the blends provided better vibration isolation and antivibration performance after the amount of nylon 6 was increased and EPDM and nylon 6 were blended through dynamic vulcanization. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 108: 4114–4121, 2008

Key words: blends; compounding; elastomers; extrusion

INTRODUCTION

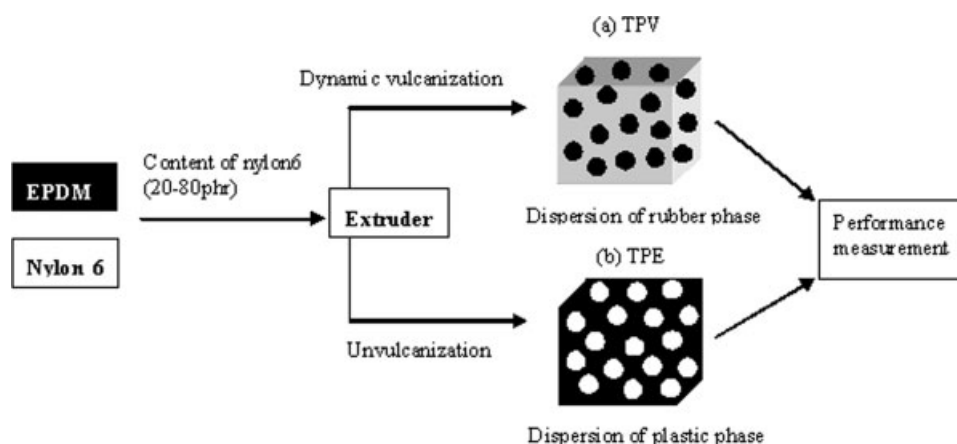
Polymers are widely applied as antivibration materials in machinery, transportation, and construction industries. Along with the development of high technology, demands for antivibration have been increasing rapidly, particularly in the area of aeronautics and precision instruments. Polymers exhibit a uniquely high damping behavior in their glass-transition zone because of the conversion of mechanical vibration into heat energy by means of friction between molecular chains and heat dissipation.¹ However, this high damping property cannot be maintained under large variations of temperature or frequency. Thus, many studies have proposed various methods to increase the damping of polymers within wide ranges of temperatures and frequencies,^{2–12} such as blending, copolymerization, and interpenetrating networks. However, the inherent sustaining strength of a polymer material cannot satisfy the antivibration structural design. Thus, in the earlier studies of Kerwin,¹³ Ugar and Beranek,¹⁴ Weibo and Feng-

chang,⁵ Oborn et al.,¹¹ and Yamada et al.,¹² a polymer/steel laminate sandwich structure was used to increase the stiffness of the polymer damping material. Some research has also used blended fillers^{5,15–17} and fibers^{18–22} in polymers to enhance their stiffness and antivibration performance. The energy loss mechanisms include friction between the polymer chains, friction between the polymer and fillers,¹ and interactions between the polymer and fibers.

Rubber/plastic blends have been commercialized as rubber-toughened plastics or as thermoplastic elastomers (TPEs).^{23,24} Thermoplastic vulcanizates (TPVs) form a special class of TPEs that can be processed under molten conditions as a thermoplastic polymer at elevated temperatures, at which the rubber phase is vulcanized with suitable curatives during mixing with a thermoplastic polymer. These materials exhibit the properties of conventional vulcanized rubber. Morphologically, TPVs are characterized by the presence of finely dispersed, micrometer-sized, crosslinked rubber particles distributed in the continuous thermoplastic matrix.^{25–31}

A number of reports on TPEs and TPVs based on ethylene–propylene–diene monomer (EPDM)/nylon blends have already been published. Ma et al.^{32,33} studied the structure and properties of an EPDM/nylon copolymer high-performance elastomer. How-

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Scheme 1 Morphology illustration of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends: (a) TPV and (b) TPE.

ever, the elastomer was prepared by static vulcanization. Huang and coworkers^{30,34,35} reported the effect of dynamic vulcanization on the crystallization behavior of an EPDM/nylon copolymer TPV and on the alteration of the crystallization behavior caused by a compatibilizer. Available atomic force microscopy measurements were used to evaluate the effect of a compatibilizer on the size of dispersed rubber particles and on changes in the crystallization behavior of a nylon copolymer matrix in TPVs. Oderkerk and coworkers^{36–38} investigated the influence of the rubber/thermoplastic viscosity ratio on the blend phase morphology, mechanical properties, and deformation–recovery behavior by changing the molecular weight of nylon 6. Wang et al.³⁹ investigated the toughening of nylon with epoxidized-EPDM rubber.

The main goal of this study was to evaluate EPDM/nylon blends (TPVs) prepared by the dynamic vulcanization process. The crosslinked rubber phase was sheared into small particles dispersed in the nylon matrix, and its phase morphology is shown in Scheme 1(a). Subsequently, its comparison with unvulcanized EPDM/nylon blends (TPEs) and its phase morphology were studied, as shown in Scheme 1(b). In our experiment, the variation of the nylon content in the TPV and TPE material systems

was investigated by the determination of the mechanical properties and hysteresis behavior and by dynamic property tests to evaluate the antivibration performance and vibration isolation.

EXPERIMENTAL

Materials

The polymers were Vistalon 7001 (ethylene content = 73 wt %, ethylidene norbornene content = 5 wt %), manufactured by ExxonMobil Chemical (Houston, TX), and nylon 6 (grade 2100), manufactured by Nan Ya Plastics Corp. (Taipei, Taiwan). Octylphenol–formaldehyde resin (SP-1045) was produced by Schenectady International, Inc. (Taipei, Taiwan). SnCl₂ was produced by Acros (Somerville, NJ).

Sample preparation

The materials used in this study consisted of EPDM, nylon 6, SP-1045, and SnCl₂ (Table I). They were prepared with a twin-screw extruder (model ZSK 26 Mega compounder, Werner and Pfleiderer, Stuttgart, Germany) with a rotor speed of 500 rpm. The elastomer was kept at 200–220°C during melt mixing with the thermoplastic.

TABLE I
Compositions of EPDM/Nylon 6 Blends (phr)

Material	TPV				TPE			
	ENV2	ENV4	ENV6	ENV8	EN2	EN4	EN6	EN8
EPDM	100	100	100	100	100	100	100	100
Nylon 6	20	40	60	80	20	40	60	80
SP-1045	7	7	7	7	—	—	—	—
SnCl ₂	0.4	0.4	0.4	0.4	—	—	—	—

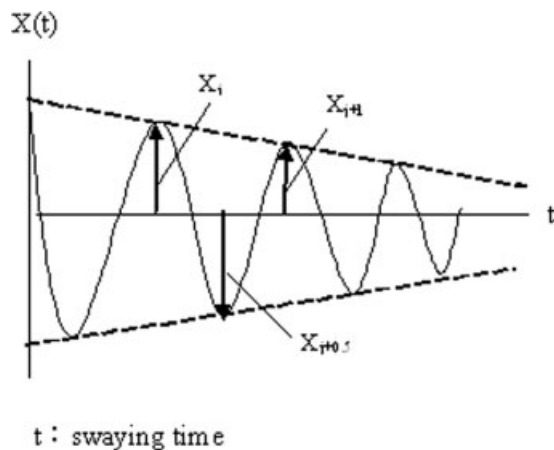


Figure 1 Typical hysteresis damping curve.

Mechanical property measurements

The tensile strength and elongation at break were measured with a universal tensile tester (QC506, Bennett Co., Ltd., Taiwan) with a tension velocity of 500 mm/min in compliance with the specifications of ASTM D 412C. The Shore hardness test was determined with a Shore A durometer (Elcometer Instruments Ltd., England) according to ASTM D 2240.

Morphology analysis

The morphology was measured with a JEOL JSM6360 scanning electron microscope (JEOL Ltd., Tokyo, Japan). A gold pattern was sputtered onto the sample fractured surface, and scanning electron microscopy (SEM) was used to observe the sample.

Determination of the compression stiffness

An MTS-810 material testing system (MTS-810, GOTECH Co., Ltd., Taiwan) was used to test the compression stiffness of the experimental piece with dimensions of $40 \pm 0.5 \text{ mm} \times 40 \pm 0.5 \text{ mm} \times 12 \pm 0.5 \text{ mm}$ within a deformation range of 2 mm. The calculation of the compression stiffness is as follows:

$$K_s = F/X \quad (1)$$

where K_s is the compression stiffness, F is the compression force, and X is the compression displacement.

Dynamic mechanical property analysis

Composites 6 mm wide were trimmed to specifications of $30 \text{ mm} \times 6 \text{ mm} \times 2 \text{ mm}$. A model Q800 dynamic mechanical analysis (DMA) instrument (TA Instruments) was used with a temperature rising rate of $5^\circ\text{C}/\text{min}$ within the range of -80 to 40°C under a frequency of 1 Hz for temperature scanning.

Determination of compression hysteresis

The measurement was carried out with an MTS-810 material testing system at a frequency of 1 Hz and amplitude of 2 mm. The loss of energy in each cycle (ΔW) was calculated from the hysteresis loop, and the damping constant (β) was calculated from ΔW :⁴⁰

$$\Delta W = \pi K \beta x^2 \quad (2)$$

where K is the stiffness of the material and x is the displacement (mL). The calculated β value was then converted into the hysteresis damping curve. X is the amplitude of the part in the j th cycle (similar to Fig. 1).⁴⁰

$$\frac{X_j}{X_{i+0.5}} = \sqrt{\frac{2 + \pi\beta}{2 - \pi\beta}} \quad (3)$$

$$\frac{X_{j+0.5}}{X_{i+1}} = \sqrt{\frac{2 + \pi\beta}{2 - \pi\beta}} \quad (4)$$

Testing methods for the dynamic properties of vibration isolation

The SRIS 3503-1990 nonresonance testing method⁴¹ was employed: the specimens were placed in the material testing system (MTS-810) and subjected to vibration at a frequency of 1 Hz and amplitude of 2 mm. Figure 2 presents the loading–amplitude graph under sine wave loading. The horizontal axis represents the amplitude, whereas the vertical axis represents the loading. The energy loss of the nanocomposites (ΔW) is the area surrounded by the loading–amplitude curve. P is the load, X is the amplitude, $|k^*|$ is the absolute resilience modulus, δ is the phase angle, k_1 is the storage modulus, k_2 is the loss modulus, c is the damping coefficient, ω is the angular frequency, $\tan \delta$ is the k_2/k_1 ratio, k_s is the compression stiffness, and R is the dynamic

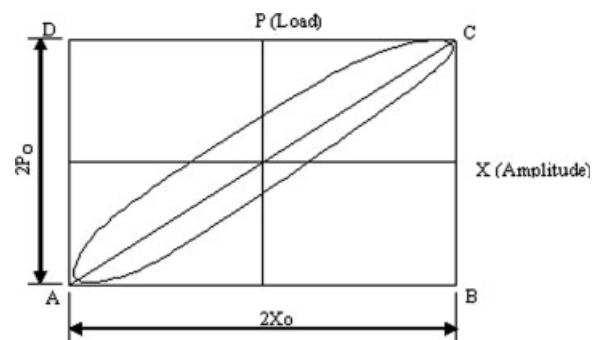


Figure 2 State of the load–displacement curve.

TABLE II
Mechanical Properties of EPDM/Nylon 6 Blends

Property	TPV				TPE			
	ENV2	ENV4	ENV6	ENV8	EN2	EN4	EN6	EN8
Hardness (shore A)	60	75	86	93	55	72	85	90
Tensile stress at break (kg/cm ²)	43.63	49.03	70.45	117.83	91.12	98.34	107.21	140.66
Elongation at break (%)	144.62	103.73	90.18	82.99	198.1	174.2	109.1	100.22
Compression stiffness (kg/mm)	136.2	295.3	338.1	378.6	122.2	255.5	328.0	344.5

ratio. They can be calculated with the following equations:⁴¹

$$|k^*| = P_0/X_0 = BC/AB \quad (5)$$

$$\sin \delta = (2/\pi) \times (\Delta W/W) \quad (6)$$

$$k_1 = |k^*| \cos \delta \quad (7)$$

$$k_2 = |k^*| \sin \delta \quad (8)$$

$$c = k_2/\omega \quad (9)$$

$$\tan \delta = k_2/k_1 \quad (10)$$

$$R = |k^*|/k_s \quad (11)$$

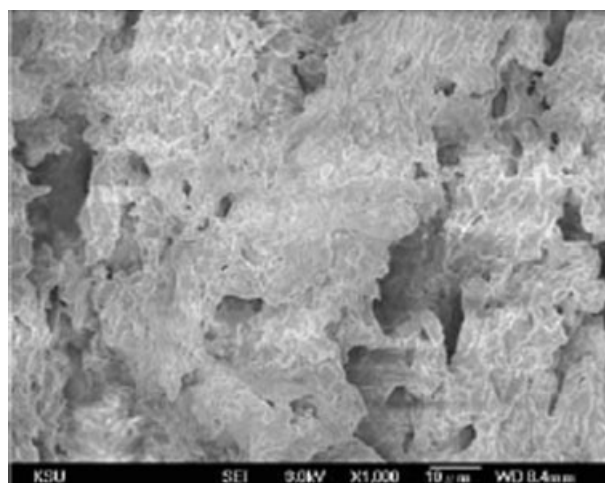
RESULTS AND DISCUSSION

Mechanical properties of EPDM/nylon 6 blends

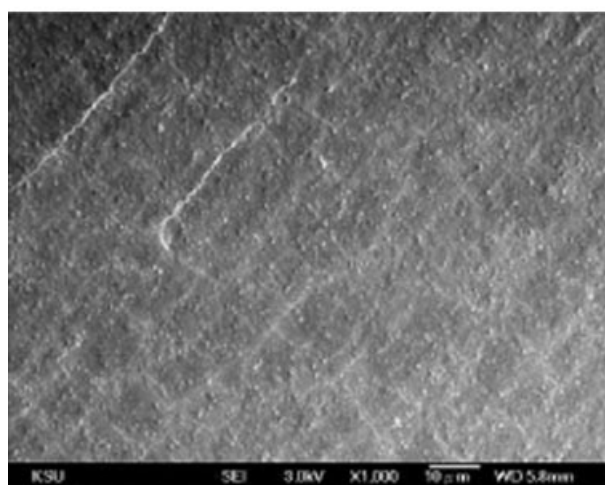
The mechanical properties of vulcanized and unvulcanized EPDM/nylon 6 blends are listed in Table II. As shown in the table, an increasing trend in the tensile strength of the blends occurs when the nylon 6 ratio is increased. The elongation experiences a decreasing trend. Another dimension of the analysis shows that the tensile strength and elongation of TPE materials are higher than those of TPV materials. In other words, because the rubber composition of TPV materials is fully crosslinked, the intensity, heat resistance, and chemical resistance of the materials are clearly improved. However, this is not the case in our research. Because of the considerably high interfacial energy between EPDM and nylon 6 (i.e., incompatibility) leading to the easy destruction of TPV materials in the frailest parts of the two polymers' interface when an external force is applied, the mechanical properties of the EPDM/nylon 6 blends after the dynamic vulcanization process are good and meet our expectations. The compression stiffness shows that TPV materials provide better functions. Because the defects and submicroscopic cracks of the materials are reduced during compression after vulcanization, the molecular network structure does not easily yield displacement deformities upon the application of an external force. The compressed material now produces a greater reaction force.

Morphology of EPDM/nylon 6 blends

The fractured surfaces of vulcanized and unvulcanized EPDM/nylon 6 blends after stretching are shown in Figure 3. This illustration reveals that when EPDM/nylon 6 blends undergo a dynamic vulcanization process during their melting state,



(a) TPV



(b) TPE

Figure 3 SEM photomicrographs of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends: (a) TPV and (b) TPE.

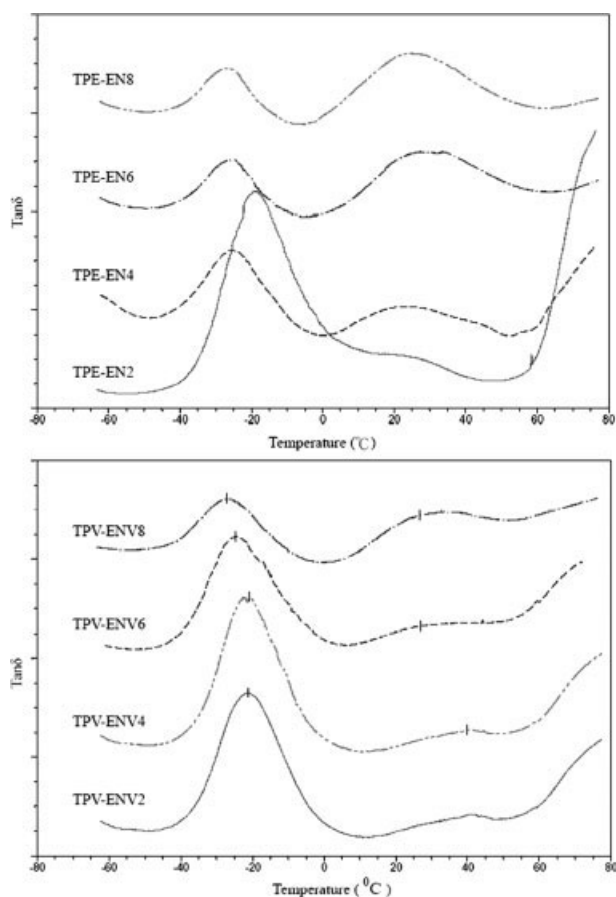


Figure 4 Effect of temperature on the damping ($\tan \delta$) of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends containing a variety of nylon 6 contents.

phase inversion takes place. The phase inversion makes the EPDM molecules produce a crosslinked reaction and causes a dispersion of rubber microparticles in the plastic phase. This illustration shows chunks of a white region in the fractured segments of TPV materials brought about by the extensive stress whitening of the plastic cover. When the material is stretched, the plastic shape changes and produces crystals. The darker regions are part of the fractured regions of rubber. Furthermore, we are able to observe some holes in the entire fractured sections. These are remnants of materials after stretching and a result of the interfacial incompatibility between EPDM and nylon 6. After the EPDM/nylon 6 blends undergo melt blending, the fracture segments become evenly dispersed with white spots. Meanwhile, Figure 3(b) shows that nylon 6 plastics are evenly distributed in the EPDM rubber phase.

DMA of EPDM/nylon 6 blends

A DMA diagram of vulcanized and unvulcanized EPDM/nylon 6 blends at a frequency of 1 Hz is presented in Figure 4. It shows two characteristic peaks:

one due to the molecular chain motion temperature of EPDM at a low temperature and another one due to the molecular chain motion temperature of nylon 6 at a high temperature. Before EPDM/nylon 6 blends are vulcanized, changing the nylon 6 contents will cause the EPDM molecular chain motion temperature to shift to a low temperature of 7°C, whereas there are no evident changes in the motion temperature of nylon 6 molecular chains. This may be attributed to the incompatibilities of the two polymers of the EPDM/nylon 6 blends, so the aggregate structures are looser. Thus, the molecular chain interactions produce a slippery transition. After the EPDM/nylon 6 blends are dynamically vulcanized, the EPDM and nylon 6 characteristic peaks move toward a high temperature at this time. This implies that an interaction exists between the two polymers. During the dynamic vulcanization in the EPDM rubber phase, thermal energy increases because the molecular chain movement experiences some sort of constraint as a result of crosslinking. In addition, the molecular chain of the nylon 6 plastic phase becomes stiff so that the TPV shifts to a high-temperature region when the nylon 6 ratio is increased. Figures 5 and 6 show the $\tan \delta$ values of TPEs and TPVs when the nylon 6 contents are changed, and the storage modulus varies. Increasing the amount of nylon 6 can help to increase the $\tan \delta$ and storage modulus properties. At this point, the $\tan \delta$ and storage modulus properties of TPVs are higher than those of TPEs. This occurrence may be attributed to the fact that when the EPDM rubber phase goes through dynamic vulcanization, the interactive strength of the nylon 6 macromolecule increases, and its molecular chain becomes stiffer. However, the movement temperature of the molecular chain is quite close to ambient temperatures. Therefore, EPDM/nylon 6

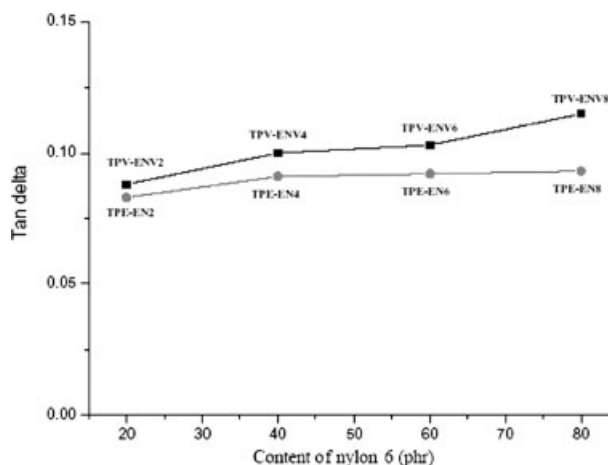


Figure 5 Effect of damping ($\tan \delta$) of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends containing a variety of nylon 6 contents (at 30°C).

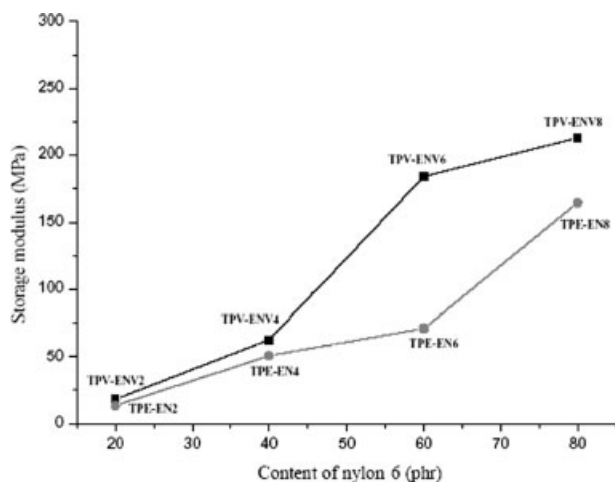


Figure 6 Effect of the storage modulus of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends containing a variety of nylon 6 contents (at 30°C).

blends rely on the increase in the nylon 6 content and acquire better damping and storage modulus characteristics at ambient temperatures.

Hysteresis of EPDM/nylon 6 blends

Under alternating stress, hysteresis occurs when the rate of deformation lags behind the rate of stress variation. In this case, because the energies absorbed and released are not balanced in each cycle, the stretching and recoil curves form a closed loop, which is known as the hysteresis loop. The area of the loop represents the energy loss. For elastomer materials, a larger hysteresis loop means higher damping, which can reduce vibration.⁴²

Figure 7 shows the hysteresis loop of EPDM/nylon 6 blends, vulcanized and unvulcanized, under compressive vibration at 30°C, 1 Hz, and a 2-mm amplitude. The energy loss in a cycle⁴⁰ can be obtained from the hysteresis loops. Figure 8 shows the antivibration performance of vulcanized and unvulcanized EPDM/nylon 6 blends. It indicates that EPDM/nylon 6 blends through the dynamic vulcanization process have better antivibration performance than unvulcanized blends. On the other hand, the antivibration property of TPE and TPV improves when the amount of nylon 6 is increased. We can therefore assume that after the rubber composition goes through vulcanization, the material becomes more conducive to transforming its kinetics to thermal dissipation upon the application of the external force. The addition of nylon 6 allows the material system to become stiff. In other words, the antivibration properties of the material system are determined on the basis of the plastic phase.

Polymer materials have viscous and elastic properties and hence exhibit hysteresis under the action of

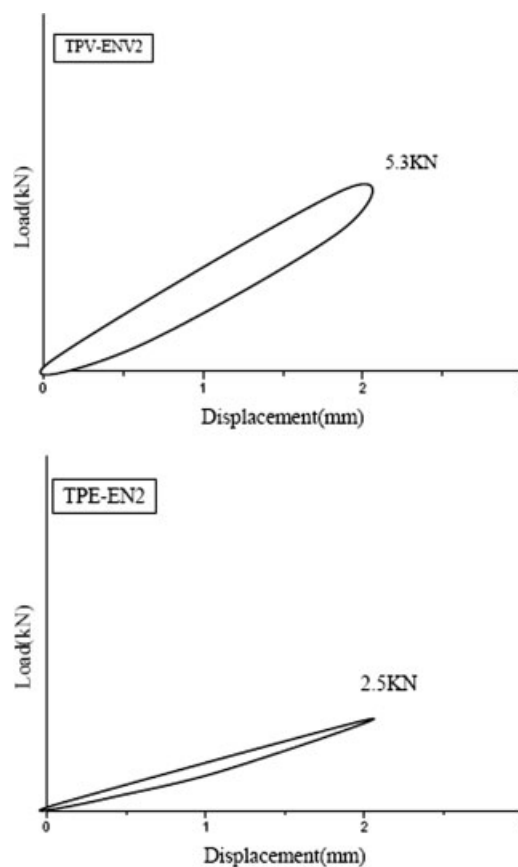


Figure 7 Hysteresis loops of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends (compression loading = 150 kg, frequency = 1 Hz, amplitude = 2 mm).

an external force. The β values may be derived from the area surrounded by hysteresis loops. According to the theory of free vibration, the vibration isolation of materials can be evaluated from β and the hysteresis damping characteristics.⁴⁰

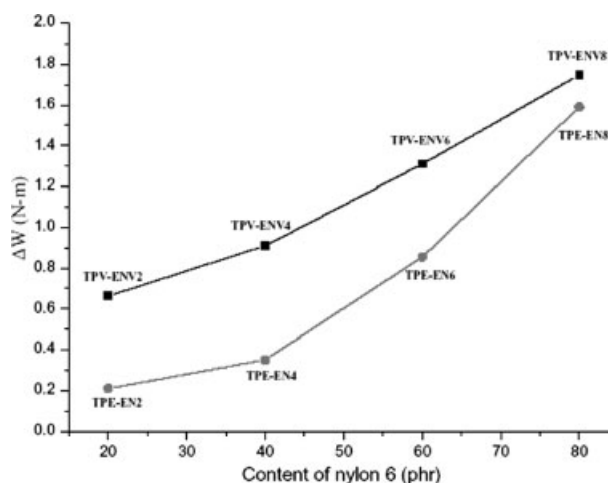


Figure 8 Energy loss in a cycle (ΔW) of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends containing a variety of nylon 6 contents.

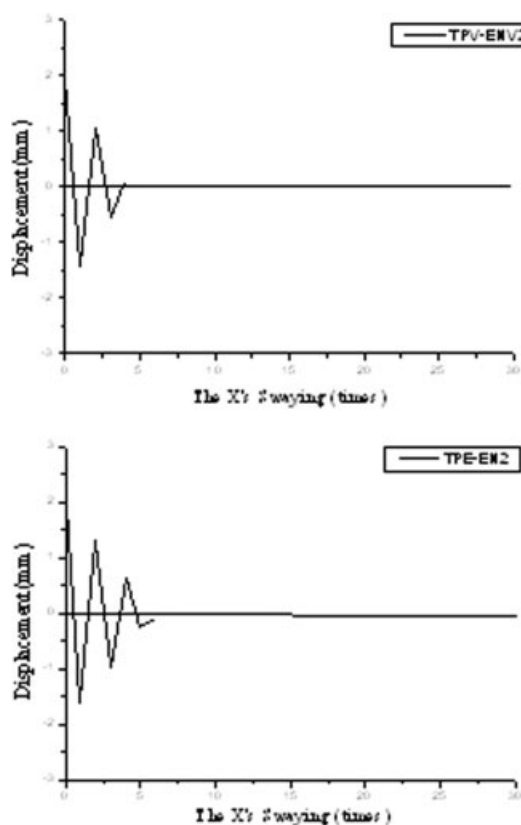


Figure 9 Hysteresis damping curves of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends.

Figure 9 shows the vibration–damping curves of vulcanized and unvulcanized EPDM/nylon 6 blends. The β values derived from the hysteresis loops are shown in Figure 10, which presents the β values of TPEs ($\beta = 0.133\text{--}0.304$) and TPVs ($\beta = 0.199\text{--}0.323$). With a higher β value and greater viscosity factor, TPV energy dissipates faster at certain amplitudes and reaches stability with less vibration. These experimental results indicate tendencies similar to those found in Figure 8. Therefore, it can be noted that the dynamic vulcanization and nylon 6 content play important roles in the design of materials in terms of vibration isolation and antivibration performance.

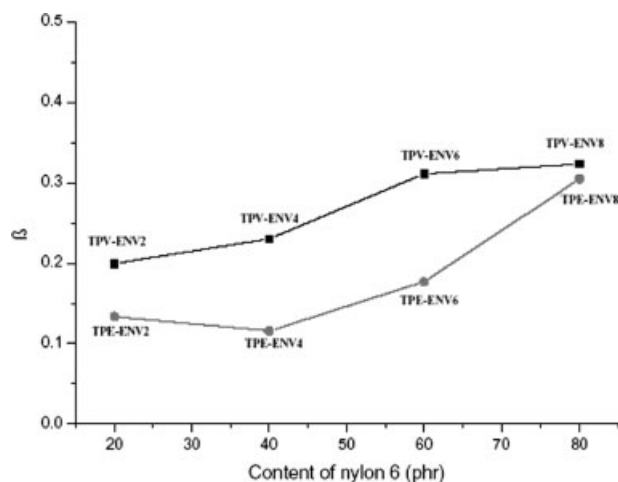


Figure 10 β of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends containing a variety of nylon 6 contents.

Dynamic properties of EPDM/nylon 6 blends: vibration isolation and antivibration

The relationship of the dynamic ratio and the loss factor may be used to evaluate the antivibration and vibration isolation effects of the elastomer material. Table III presents the dynamic testing results of EPDM/nylon 6 under the loading conditions of 150 kg at 30°C, 1 Hz, and a 2-mm amplitude. With a lower dynamic ratio, there is a better antivibration effect along with a higher damping or loss factor, which is favorable for shock absorption. The relationship of the dynamic ratio and loss factor is depicted in Figure 11.

While comparing the vibration isolation of vulcanized and unvulcanized EPDM/nylon 6 blends, we found that the loss factor assumes an increasing tendency with an increasing amount of nylon 6. The EPDM/nylon 6 blends after vulcanization show an equally favorable vibration isolation performance with the same nylon content ratio. At 80 phr nylon 6, TPE eventually achieves a vibration isolation performance that is close to that of TPV with only a 40 phr nylon 6 ratio. This means that the source of the vibration isolation performance in EPDM/nylon 6

TABLE III
Dynamic Properties of the Antivibration Performance of EPDM/Nylon 6 Blends

	TPV				TPE			
	ENV2	ENV4	ENV6	ENV8	EN2	EN4	EN6	EN8
$ k^* $	270.41	566.33	341.84	438.77	127.55	244.90	392.85	423.47
δ	7.878	7.626	12.538	13.039	5.181	4.583	7.082	12.498
k_1	268.332	562.266	335.229	429.604	127.128	244.264	390.427	415.335
k_2	33.376	67.678	66.889	89.241	10.367	17.615	43.613	82.602
c	5.312	10.771	10.646	14.023	1.651	2.803	6.941	13.146
Tan δ	0.123	0.120	0.196	0.203	0.081	0.072	0.111	0.195
R	1.98	1.91	1.19	1.15	1.04	0.96	1.19	1.22

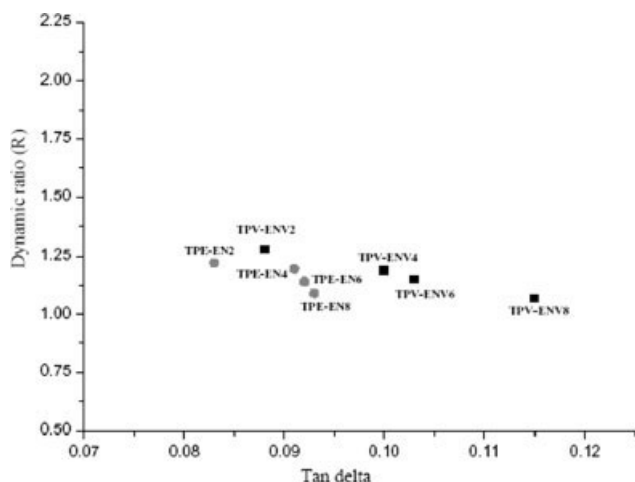


Figure 11 Loading dynamic ratio (R) and loss factor ($\tan \delta$) of dynamically vulcanized and unvulcanized EPDM/nylon 6 blends containing a variety of nylon 6 contents.

blends is the vulcanized rubber. The meaning of physical is considering that the actual viscosity of this material is known. Rubber also forms a three-dimensional network structure that effectively converts the external mechanical energy to kinetic energy. Nevertheless, the vibration isolation effect is at its best when nylon 6 is at 80 phr. It seems that the antivibration performance of both vulcanized and unvulcanized EPDM/nylon blends increases when the nylon content is increased. The difference between the two is not significant. To sum up the observations stated previously, the viscoelasticity of EPDM/nylon blends, as well as the vibration isolation and antivibration performance of these materials, can be adjusted by changes in the proportion of nylon.

CONCLUSIONS

EPDM rubber and nylon 6 plastic materials have been combined to form blends. The two methods of manufacturing TPE and TPV materials, melt blending and dynamic vulcanization, have been compared, and the effects of nylon 6 have been discussed. Because EPDM and nylon 6 are incompatible, TPV materials are not easily damaged in a polymer environment when an external force is applied. Thus, the mechanical properties of TPV are found to be inferior to those of TPE materials. With respect to the morphology, EPDM/nylon 6 blends produce a crosslinking reaction after the dynamic vulcanization process, and the formed microparticles are dispersed inside the plastic phase. The antivibration properties of TPV materials have been proven to be better than those of the TPE material system. Therefore, it can be noted that the dynamic vulcanization and nylon 6 content play important roles in the design of materials in terms of their vibration isolation and antivibration performance.

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