

Waste Ground Rubber Tire Powder/Thermoplastic Vulcanizate Blends: Preparation, Characterization, and Compatibility

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ABSTRACT: In this article, waste ground rubber tire (WGRT) powder was introduced into thermoplastic vulcanizate (TPV) to prepare the blends of WGRT powder/TPV. The mechanical, rheological, thermal aging, and dynamic properties of the blends were investigated with respect to the particle size and dosage of WGRT powder. The results showed that tensile strength, tear strength, elongation at break, and tensile permanent deformation of the blends increased with the decrease in WGRT particle size and decreased with the dosage of WGRT. The effects of different types and dosages of compatibilizers on mechanical and rheological properties of the blends were studied. The results showed that the compatibilizer PP-g-MAH could effectively improve the interfacial compatibility between WGRT and the TPV matrix to enhance the comprehensive properties of blends. The TPV/WGRT/PP-g-MAH blends obtained the best overall properties when prepared at the weight ratio 100/30/5. Rheological studies demonstrated that the WGRT/TPV blends represented lower apparent viscosity after PP-g-MAH were added, which means that processing performance of the blends was improved by PP-g-MAH. Scanning electron microscopy was used to study the morphologies of the blends. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2014**, *131*, 39868.

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INTRODUCTION

Waste rubber is a kind of solid industrial waste and ranks the second place in the waste polymers domain. With the rapid development of automobile industry in the world, every year more than 12,000,000 tons of waste tires are produced in the world,¹ so how to utilize the large amount of waste tires effectively relates to the conservation of resources and prevention of environmental pollution problems.

The importance of recycling crosslinked rubber used in automotive tires, sealing, and hoses has been wellknown.^{2–6} An important method to utilize waste tires is to turn them into waste ground rubber tire (WGRT) powder, which could be incorporated into a polymer matrix to obtain the blends filled with WGRT. As WGRT could be used as fillers in rubber compounding,⁷ some researches have been conducted on the use of WGRT as fillers in various thermoplastics to prepare thermoplastic elastomers, such as WGRT/PP,⁸ WGRT/PE,⁹ and WGRT/other thermoplastic.^{10–15} However, the mechanical properties of the blends were not desirable compared with thermoplastic elastomers due to the lack of reactive sites on the WGRT surface and the poor interfacial adhesion of WGRT with the thermoplastic matrix.

Thermoplastic vulcanizate (TPV) is a subclassification of thermoplastic elastomers, which is made from virgin rubbers and thermoplastics via dynamic vulcanization using curing agents like sulfur. The crosslinked rubber phase that forms during mixing has a length scale of micrometers and is firmly embedded in the thermoplastic phase, leading to superior mechanical properties of TPVs.¹⁶ Polypropylene/ethylene-propylene-diene monomer (PP/EPDM) is one of the most important TPVs, which has exhibited excellent comprehensive properties in comparison with those of blends composed of common thermoplastics and rubbers through a long period of usage. Nevertheless, high costs, poor oil resistance, large tensile permanent deformation, and other shortcomings limited the application and development of TPV (PP/EPDM) greatly.

In this work, we mixed WGRT into TPV (PP/EPDM) to prepare WGRT/TPV blends. As the greatest advantage of TPV is good processability compared to traditional vulcanizates, the research to rheological behaviors of the blends is of importance.^{17–19} Furthermore, we explored the effects of particle size and dosage of WGRT on the physical properties including mechanical properties, rheological properties, thermal properties, and dynamic mechanical properties. With regard to the weak interfacial compatibility between WGRT and TPV, consideration about using

compatibilizers can be made to improve compatibility among the blends.^{20,21} So we researched the interface compatibility and overall properties of the WGRT/TPV blends compatibilized by compatibilizers that could enhance the interfacial adhesion between WGRT and the TPV matrix. It is hoped that the blends compatibilized by compatibilizers could overcome the shortcomings of TPV (PP/EPDM) and decrease the cost of productions on the basis of retaining good performance of the original materials.

EXPERIMENTAL

Materials

WGRT was purchased from Zengcheng Faxing Rubber Co. (Guangzhou, China). The particles were divided into various size by sieving classification: 80 meshes (200–150 μm), 100 meshes (150–110 μm), 140 meshes (110–75 μm), and 200 meshes (75–58 μm). TPV (santoprene 201-80, PP/EPDM) was supplied by AES Co. Styrene–butadiene–styrene (SBS1401) was obtained from Baling Petrochemical Co. (China), and maleic anhydride-grafted polypropylene [PP-*g*-MAH (CA100)], ethylene–acrylate–glycidyl methacrylate (A8900), methyl methacrylate, butadiene and styrene copolymer (MBS) and ethylene and vinyl acetate copolymer (EVA2825) all were gained from Atofina Chemical Co. (France).

Blends Preparation

TPV was added in a laboratory two-roll mill whose rollers temperature was about 110°C, then mixed with WGRT (dried at 80°C in an oven) and compatibilizers gradually. The mixture was milled for 5 min and sheeted out at 2-mm nip gap. Compression molding of the sheeted-out stocks was carried out under the following conditions: preheating at 170°C for 15 min at low pressure, compressing for 5 min at 15 MPa at the same temperature, then cooling at ambient temperature in the mold at 10 MPa.

Measurement and Characterization

Rheological Properties. Torque rheological properties. The experiment was performed in an torque rheometer (Haake Poly-lab OS, Germany) at 180°C and rotor speed of 60 rpm. The blends (45 g) were added into the mixer and mixed for 10 min. The curves of torque-time were recorded automatically in the system.

Capillary rheological properties. Capillary rheological properties of the blends were tested at 180°C in a capillary rheometer (SHIMADZU CFT-500D, Japan). Barrel temperature was risen to 180°C, then the blends (2 g) were added to the barrel of capillary rheometer. Tests began after preheating for 90 min, then the melt was squeezed out from the die of capillary tube when the rod moved down.

Mechanical Properties

Dumbbell-shaped tensile specimens and pieces for tear test were punched out from the molded sheets. Tensile strength, elongation at break and tensile permanent deformation were measured on these tensile specimens according to ASTM D412 specification in a Zwick/Roell Z010 universal testing machine (Germany) at a strain rate of 50 mm/min at room temperature. Tear

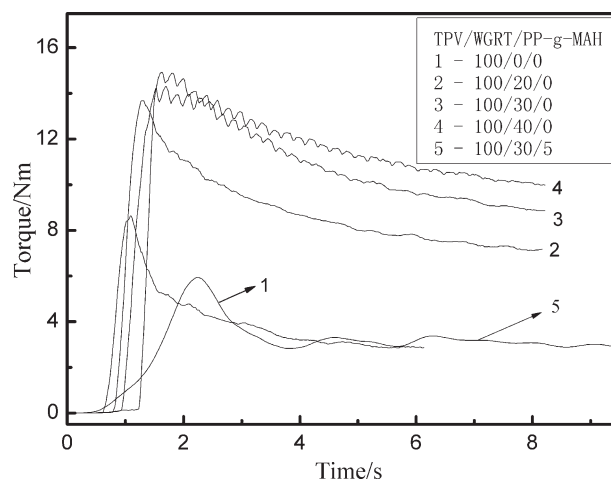


Figure 1. Effect of WGRT dosage and compatibilizer on rheological properties of the blends.

strength was tested in a same machine according to ASTM D624.

Thermal Aging Properties

Test samples were heated at 120°C in an air-circulating oven (GT-7017-M, Gaotie Detector Co., Ltd, China) for 72 h. After cooled down and conditioned at room temperature, tensile and tear test of the samples were performed at room temperature according to ASTM D412 and ASTM D624 respectively under the condition that was the same as the measurement of mechanical properties.

Thermal Properties

Thermal stability experiment of the blends was carried out in a Netzsch TG-209F1 (Germany) thermogravimetry analyzer. The test temperature range was from 30 to 650°C, heating rate was 20°C/min and the test atmosphere was in nitrogen. Meanwhile, dynamical mechanical analysis (DMA) was performed using a Netzsch DMA-242C (Germany). The samples were heated from –110°C up to 130°C at a rate of 3°C/min.

Scanning Electron Microscopy

Scanning electron microscopy (SEM; Nova Nano SEM 430) was used to qualitatively examine the fracture surface of the samples broken in liquid nitrogen.

RESULTS AND DISCUSSION

Effect of WGRT Dosage on Rheological Properties of Blends

Haake Rheological Experiment. Curves 1–4 in Figure 1 show the effect of 200 meshes WGRT dosage on rheological properties of the blends. During the mixing of TPV and WGRT, the peak value and equilibrium torque in the curves increased with the dosage of WGRT. This is owing to the denser three-dimensional network structure and stronger interactions between chemical bonds of vulcanized WGRT compared with dynamic vulcanized structure of TPV. When WGRT particles were added to TPV matrix, the chains of TPV entangled with network structured WGRT, bringing about the lower liquidity of TPV, and the surface of particles became softening under the role of heat, which means the sticking particles have strong tendency to obstruct the rotation of a rotor. On the other hand,

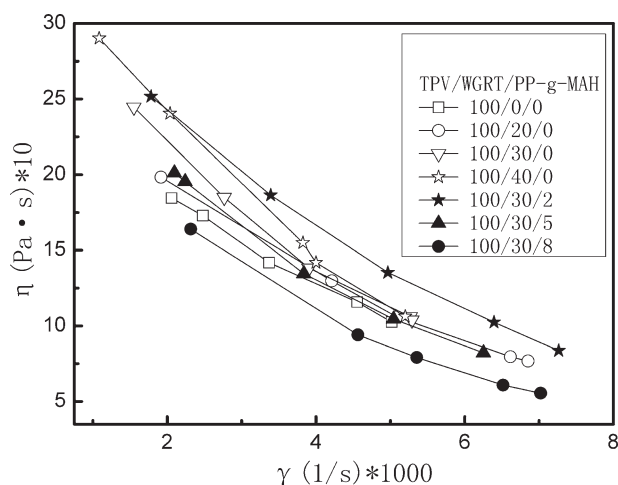


Figure 2. Relationship between shear rate and apparent viscosity of TPV and the blends.

WGRT that was added to TPV matrix without any additives could gather in large numbers in the matrix resulting in bad dispersion. The network structured and gathering WGRT brought about larger driving force that a rotor needed to keep constant speed, so the equilibrium torque of the blends became larger than that of neat TPV and increased with the dosage of WGRT.

Capillary Rheological Experiment. The relationship between apparent viscosity and shear rate of neat TPV and the blends without compatibilizers is shown in Figure 2. Apparent viscosity of all samples decreased with shear rate, indicating pseudoplastic flow behavior. Apparent viscosity of the blends increased accordingly with the dosage of 200 meshes WGRT, indicating that flowability of the blends became worse with the increase in WGRT dosage. This can be explained by Einstein equation: $\eta = \eta_s(1 + [\eta]\phi)$ where η is the apparent viscosity, η_s is the viscosity of pure solvent, $[\eta]$ is the intrinsic viscosity, ϕ is the volume fraction of WGRT. It can be considered that the cross-linked structure of WGRT tangled with more TPV chains and the dispersion of WGRT into TPV became worse with the

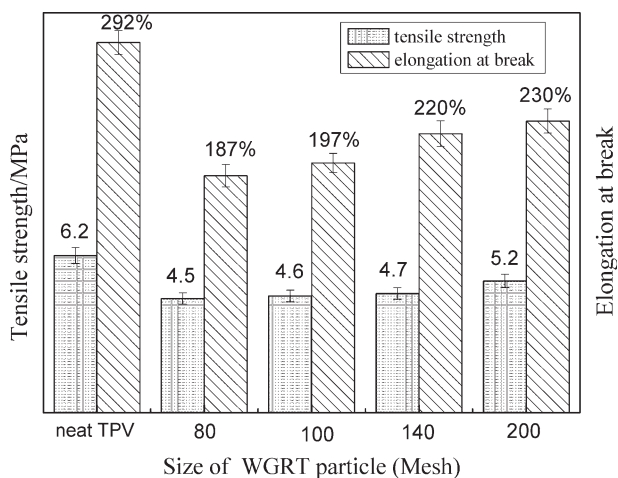


Figure 3. Effect of WGRT particle size on tensile strength and elongation at break of the blends.

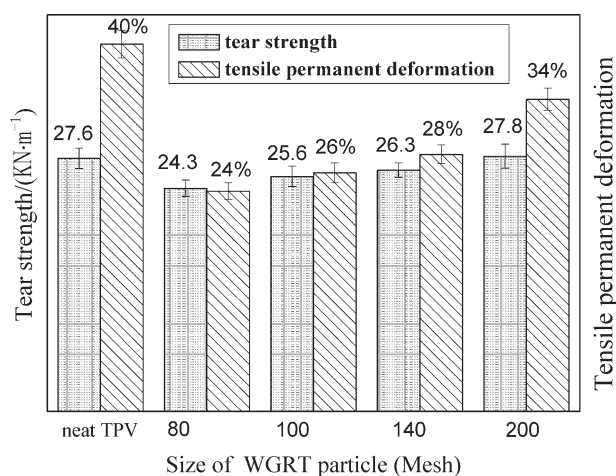


Figure 4. Effect of WGRT particle size on tear strength and tensile permanent deformation of the blends.

increase in WGRT dosage, thus the flowability of TPV was hindered by WGRT.

Effect of WGRT Particle Size on Mechanical Properties of Blends

Figures 3 and 4 show that all the mechanical properties of the blends decreased with the addition of WGRT. Mainly because WGRT particles that have been vulcanized could not disperse into the TPV matrix uniformly, poor mechanical properties arose from more stress concentrations produced by the bad dispersion. For another reason, PP and EPDM are the major components of TPV which are nonpolar substances, while WGRT used in the experiment is composed of styrene butadiene rubber, natural rubber and polybutadiene rubber which have certain polarity, so the difference in polarity leads to the weak compatibility between WGRT and TPV.

As also can be seen in Figures 3 and 4 that tensile strength, tear strength, elongation at break and tensile permanent deformation of the blends increased with the decrease in WGRT particle size. Generally smaller size particles show two aspects of effects on dispersion. On the one hand, smaller particles have stronger tendency to aggregate; on the other hand, specific surface area increases with the decrease in particle size, meaning the contact surface between WGRT and the TPV matrix will increase, which contributes to the interaction between WGRT and the TPV matrix. In this experiment, comprehensive effect of smaller size particles showed the improved dispersion of the blends. While tensile permanent deformation of the blends increased with the decrease in WGRT particle size, maybe because smaller WGRT powders are more likely to form continuous phase in the TPV matrix, resulting in the increased internal friction among the molecular chains. Consequently, the irreversible deformation became larger as the resilience of TPV molecular chains became worse, so the tensile permanent deformation of the blends increased with the decrease in WGRT particle size.

SEM photomicrographs of brittle fractured surfaces of neat TPV and WGRT/TPV blends are shown in Figure 5. Figure 5(a) shows little defect on the surface of neat TPV. But more microspores and cavities emerged on the surface of TPV/80 meshes

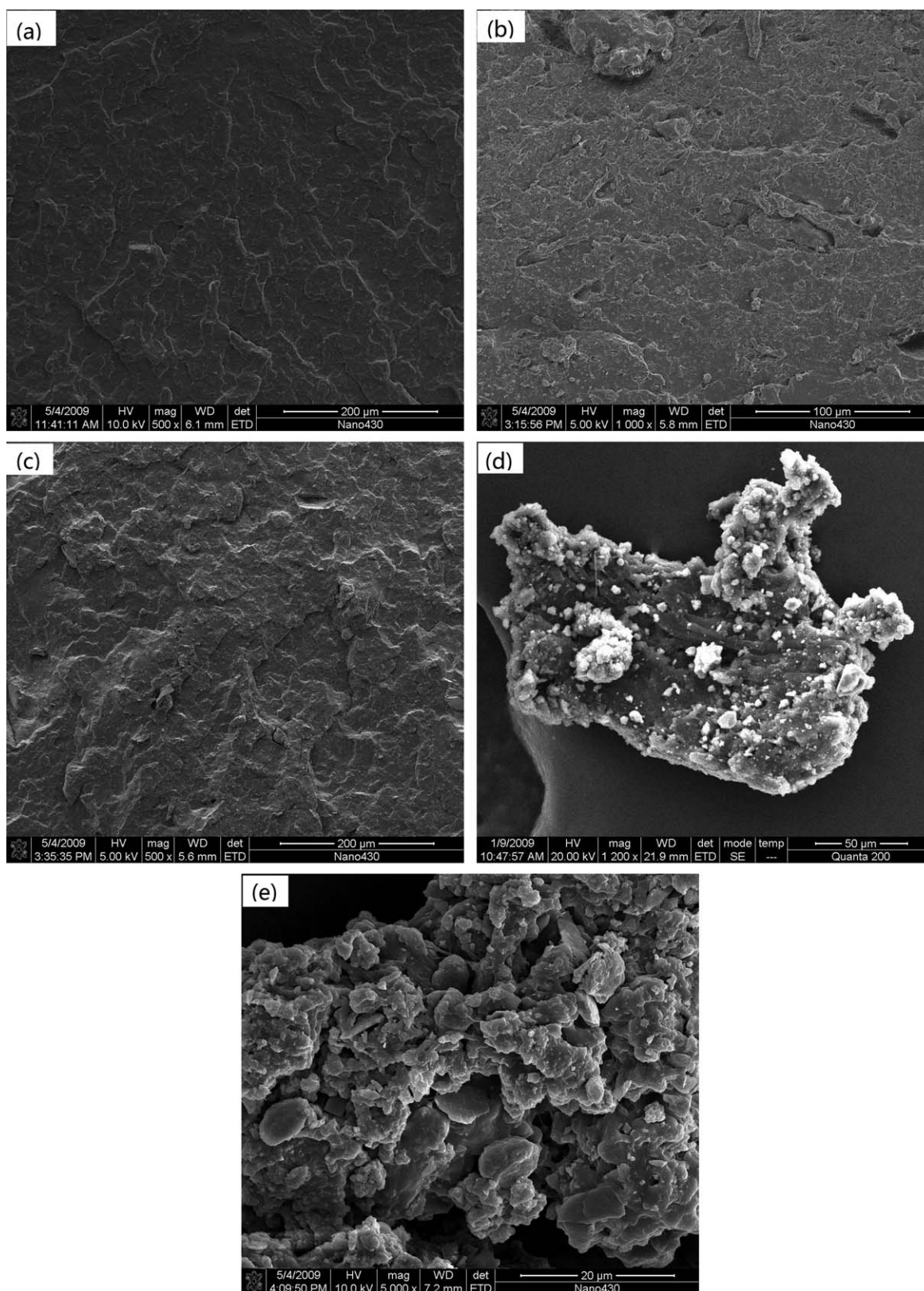


Figure 5. SEM photomicrographs of neat TPV, WGRT, and blends: (a) neat TPV; (b) TPV/80 meshes WGRT blends; (c) TPV/200 meshes WGRT blends; (d) 80 meshes WGRT; (e) 200 meshes WGRT.

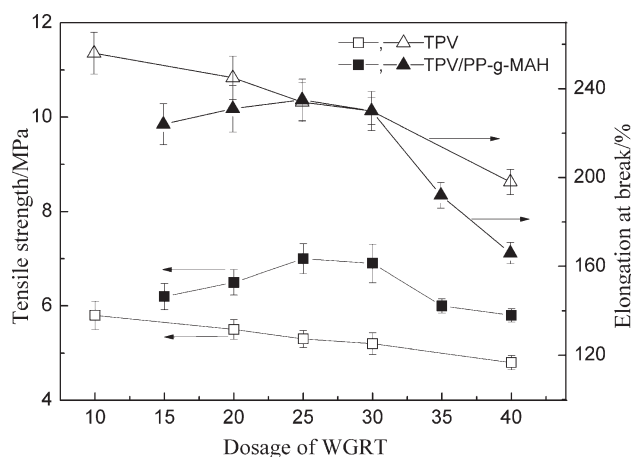


Figure 6. Effect of WGRT dosage on tensile strength and elongation at break of the blends (with and without PP-g-MAH).

WGRT blends than that of TPV/200 meshes WGRT blends. The reason is that it is more difficult for large particles to disperse into the matrix and stress concentrations in the matrix are more likely to grow up, which will damage the material performances. Nevertheless, smaller powders are rougher and have larger specific surface area that helps to improve the interfacial contact and reduce the possibility to produce stress concentration in the TPV matrix, so TPV/200 meshes WGRT blends have better mechanical properties.

From the above experiment, results can be concluded that it is more suitable to select 200 meshes WGRT to prepare TPV/WGRT blends. In the following, 200 meshes WGRT were selected to prepare TPV/WGRT blends except for special instructions in the experiment.

Effect of WGRT Dosage on Mechanical Properties of Blends

The effect of WGRT dosage on mechanical properties of the blends is shown in Figures 6 and 7. With the increased dosage of WGRT, the dispersion of WGRT particles into the TPV matrix and compatibility between WGRT and TPV both became worse, so tensile strength and elongation at break of the blends inevitably declined with the increase in WGRT dosage. While

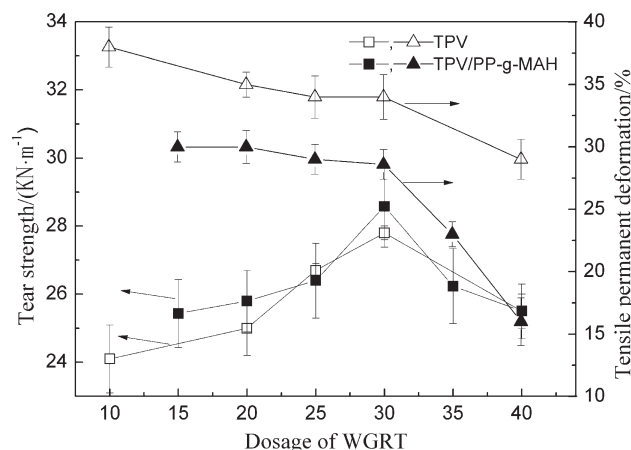


Figure 7. Effect of WGRT dosage on tear strength and tensile permanent deformation of the blends (with and without PP-g-MAH).

Table I. Effect of Different Compatibilizers on Mechanical Properties of the Blends (TPV/WGRT/Compatibilizer = 100/30/5)

Type of compatibilizers	Tensile strength (MPa)	Elongation at break (%)	Tear strength (KN m ⁻¹)	Tensile permanent deformation (%)
None	5.2	230	27.8	34
PP-g-MAH	6.9	230	28.6	29
MBS	4.0	77	13.5	6.4
A8900	4.2	164	14.7	20
EVA	4.6	247	23.5	34
SBS	4.7	248	26	35

tear strength increased with the dosage of WGRT before 30 parts per hundreds (phr of TPV). It is mainly owing to the three-dimensional network structure of WGRT, which also caused the lower tensile permanent deformation when WGRT was dispersed into the TPV matrix as elastomer particles.

Effect of Compatibilizers on Rheological Properties of Blends

Haake Rheological Experiment. Curve 5 in Figure 1 shows the effect of compatibilizers on rheological properties of the blends. As can be seen in the figure that the equilibrium torque of blends with compatibilizers decreased obviously compared with that of blends without compatibilizers, which is in balance with that of neat TPV on the whole. Because maleic anhydride in the compatibilizer PP-g-MAH interacted with WGRT and even reacted with the phenolic —OH group in WGRT when the crosslinks in WGRT structure were broken under mechanical mixing in the mill, which could help to disperse WGRT into the TPV matrix. The dispersion phase in the matrix phase was refined and space between particles in the matrix phase broadened and uniformed under the role of PP-g-MAH, so the WGRT inhibition to the rotation of a rotor declined. Therefore, the blends with compatibilizers kept the good processing liquidity of thermoplastic elastomers and even a little better than that of neat TPV.

Capillary Rheological Experiment. As can be seen in Figure 2, apparent viscosity of the blends decreased with the dosage of compatibilizers. It could be caused that the dispersion of WGRT into TPV matrix was improved after compatibilizers were added to the blends and the compatibilizer itself also has good flowability. As the dispersion of WGRT into the TPV matrix became better under the role of compatibilizers, the acting force among TPV segments became smaller and chains motion became easier, objectively contributing to the drop of apparent viscosity.

Effect of Compatibilizers on Mechanical Properties of Blends

The effect of different compatibilizers on mechanical properties of the blends is listed in Table I. When PP-g-MAH was used as compatibilizers, tensile strength of the blends improved obviously (from 5.2 to 6.9 MPa), which increased by about 32.7%. Other mechanical properties of the blends including tear strength, tensile permanent deformation, and elongation at break were also for the better. Elongation at break of the blends

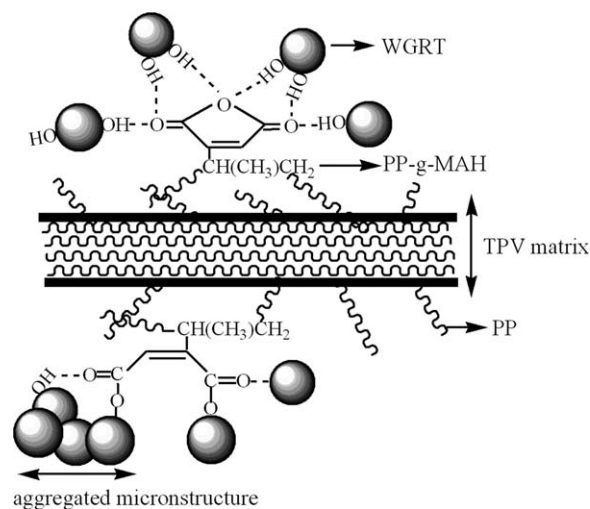


Figure 8. Schematic illustration of the compatibilization effect of the PP-g-MAH acting between WGRT and TPV.

with other two compatibilizers EVA and SBS is higher than that of the blends with PP-g-MAH, because EVA and SBS's own elongation at break are large. Nevertheless, tensile strength of the blends with compatibilizers EVA and SBS significantly decreased because of their poor compatibility with the blends. What's more, comprehensive performances of the blends with compatibilizer MBS and A8900 had considerable reduction. To sum up, the blends with PP-g-MAH had the best overall performance, because maleic anhydride in the compatibilizer PP-g-MAH could interact with WGRT and even react with the phenolic —OH group in WGRT, and nonpolar part of PP-g-MAH reacted with nonpolar TPV. This chemical interaction led to improved compatibility and adhesion between WGRT and TPV. Meanwhile WGRT particles also can disperse into the TPV matrix more uniformly because of the connection of PP-g-MAH. Additionally, the strength properties of PP-g-MAH are relatively better than those of other compatibilizers, which contributes to the mechanical properties of the blends. Figure 8 is the schematic illustration of the compatibilization effect of the PP-g-MAH acting between WGRT and TPV matrix.

SEM photograph was conducted to study the compatibility among the blends in the following. Figure 9 depicts SEM photomicrographs of brittle fractured surfaces of TPV/WGRT/PP-g-MAH and TPV/WGRT/SBS blends. The fracture surface in Figure 9(a,b) is more smooth and continuous than that in Figure 9(c). It can be argued that PP-g-MAH improved the interfacial compatibility and strengthened the interfacial bonding strength between WGRT and TPV molecule chains, so as to heighten the comprehensive mechanical performances of the blends.

In the context of the existence of compatibilizer PP-g-MAH (5 phr), as can be seen in Figures 6 and 7 that tensile strength and elongation at break of the blends increased with the dosage of WGRT first, then decreased after 25 phr, mainly because WGRT particles could uniformly disperse into the TPV on account of the effect of PP-g-MAH when WGRT dosage was small. Tensile strength of the blends increased with WGRT dosage due to the certain intensity of WGRT particles. However, with the increase

of WGRT dosage, it is easier for the blends to produce stress concentration around rubber phase, thus the rubber phase could not bear so much stress effectively that the mechanical performance of the blends degraded. At last, both elongation at break and tensile permanent deformation went down, and tear strength had a little reduction. From the above, the comprehensive performance of the blends was best when TPV/WGRT/PP-g-MAH was 100/30/5.

Effect of WGRT and Compatibilizers on Oil Resistivity of Blends

Oil resistivity of neat TPV is ordinary, such as Neoprene.¹ It can be seen in Table II that the oil resistivity of blends changed little when TPV was mixed with WGRT and PP-g-MAH separately, while the oil resistivity improved greatly when WGRT and PP-g-MAH were mixed into the TPV matrix simultaneously. It can be attributed to the plentiful polar groups in the structure of WGRT. In the mean time, the oil resistivity of PP-g-MAH is also very good as the polar group maleic anhydride abounds in the structure of PP-g-MAH, consequently the oil resistivity of TPV got advance with mixing PP-g-MAH. On the contrary, oil molecules could permeate into the blends without PP-g-MAH easily, leading the oil resistivity of the blends without PP-g-MAH to decrease. Table II also shows that the oil resistivity of the blends was best when WGRT dosage was 20 phr. It indicates that the compatibility of the blends reached its optimal state when the dosage of WGRT was 20 phr, thus it became difficult for oil molecules to enter into the blends. TPV/WGRT/PP-g-MAH = 100/30/5 should be the optimal choice considering the synthesise performance of the blends.

Thermal Aging Properties of Blends

Thermal aging is the common fault of polymer materials because of internal and external factors. Mechanical properties of thermally aged neat TPV and TPV/200 meshes WGRT/PP-g-MAH blends with different WGRT dosage are shown in Figures 10 and 11. Mechanical properties of the aged blends all decreased compared to those of unaged blends, especially elongation at break. Moreover the mechanical properties of aged blends increased with WGRT dosage up to 25 phr then decreased after 30 phr, which is the same trend as the change of unaged blends. It was attributed to the degradation of TPV, WGRT, and PP-g-MAH upon thermal aging at 120°C. However, the change of tensile strength was <10% when WGRT dosage was 25 and 30 phr. It indicated that the blends had a good aging property. This is owing to the reinforced interaction between WGRT and TPV compatibilized by PP-g-MAH and the formation of additional interaction between TPV and WGRT in virtue of the polymer chains motion upon thermal aging at 120°C.

Thermal Stability of Blends

The effect of WGRT particle size on the thermal stability of blends is shown in Figure 12. The variations of initial weight loss temperature and terminate temperature of blends with different WGRT particle size were not obvious. Weight-loss ratio of the blends slightly increased with particle size of WGRT. It means the thermal stability of blends became better with the decrease in WGRT particle size. It is believable that specific

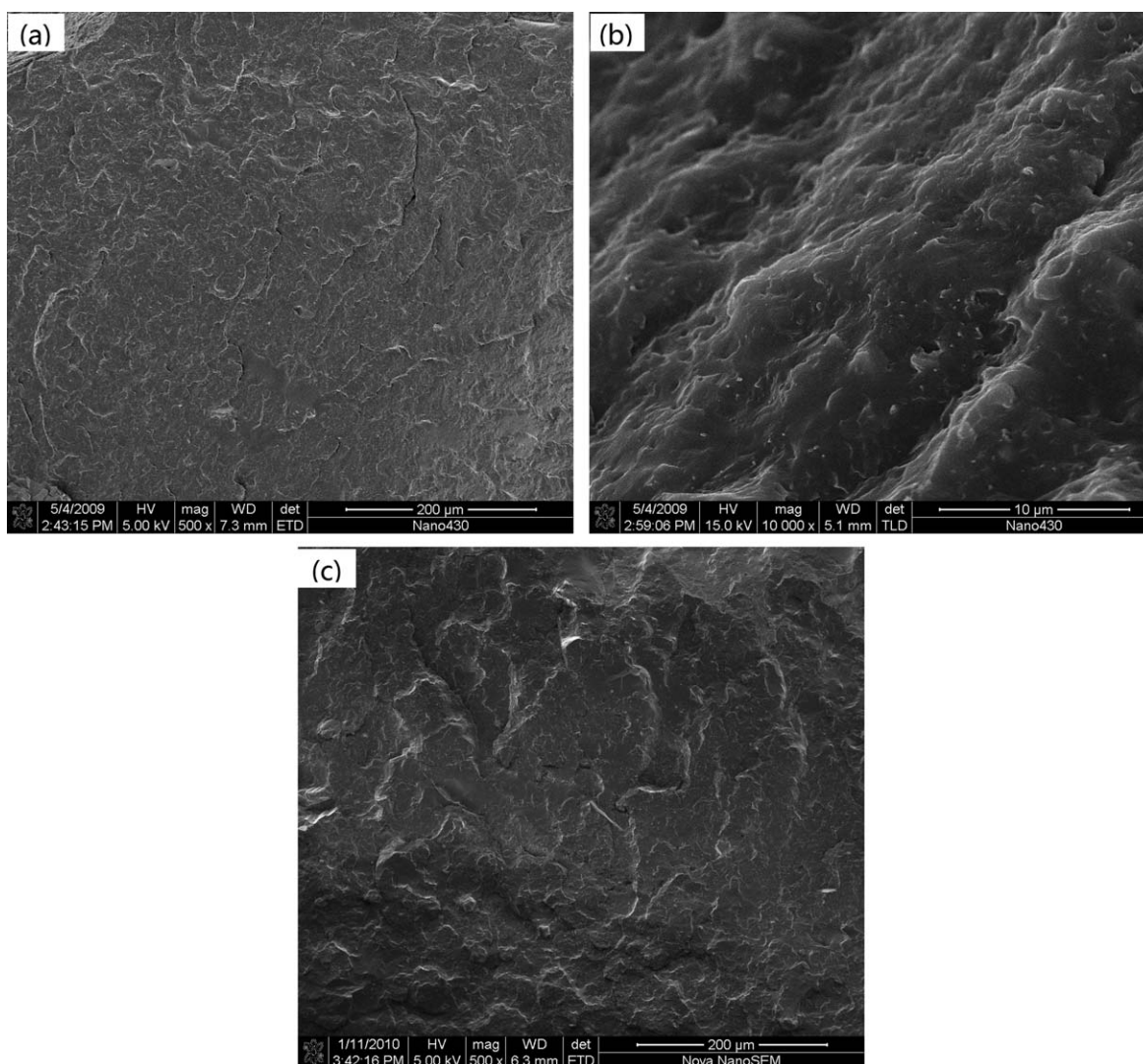


Figure 9. SEM photomicrographs of blends: (a,b) TPV/WGRT/PP-g-MAH blends; (c) TPV/WGRT/SBS blends.

surface area of WGRT particles increased with the decrease in particle size of WGRT, bringing about the increased interaction between WGRT and TPV. Thus the thermal stability of blends improved slightly because of the accretion of contact surface.

Figure 13 shows the effect of WGRT dosage on the thermal stability of blends. Compared with that of neat TPV, initial and terminate temperature changed little and only the weight loss retention increased. It is owing to the large amount of carbon

black in WGRT, so the weight loss retention increased with WGRT content. For this reason, the blends could retain good thermal stability.

Table II. Effect of the Dosage of WGRT and PP-g-MAH on Oil Resistivity of the Blends

TPV/WGRT/ PP-g-MAH	Mass change (%)	TPV/WGRT/ PP-g-MAH	Mass change (%)
100/0/0	22.6	100/10/5	18.5
100/10/0	24.8	100/20/5	15.2
100/30/0	24.0	100/30/5	17.0
100/0/5	20.2	100/40/5	19.1
100/0/10	17.7		

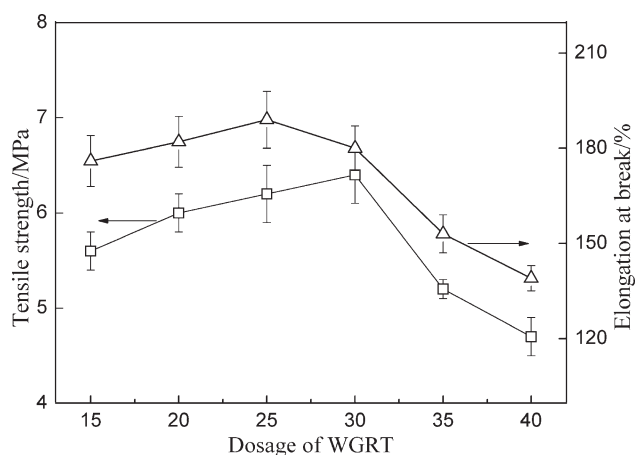


Figure 10. Effect of WGRT dosage on tensile strength and elongation at break of the aged blends (TPV/PP-g-MAH = 100/5).

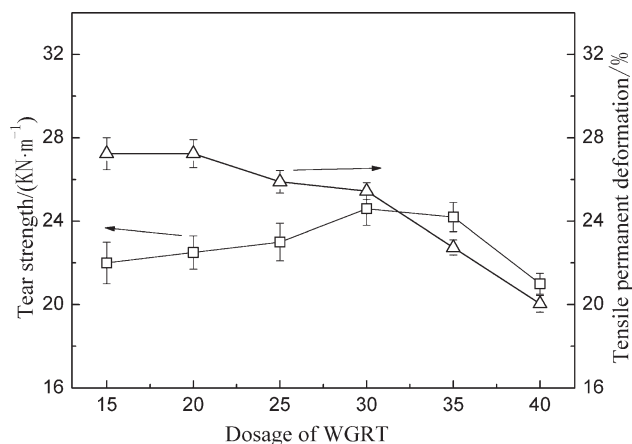


Figure 11. Effect of WGRT dosage on tear strength and tensile permanent deformation of the aged blends (TPV/PP-g-MAH = 100/5).

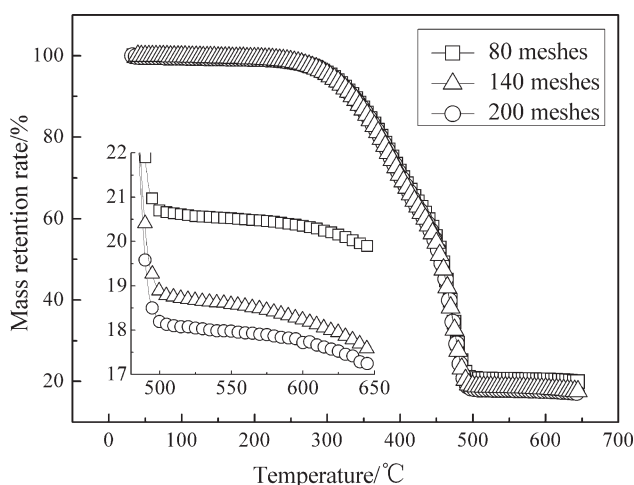


Figure 12. Effect of different particle size on TG curves of the blends.

Figure 14 shows that the variations of initial and terminate temperature were not obvious as well as weight loss retention. But the weight loss retention increased a little with PP-g-MAH dos-

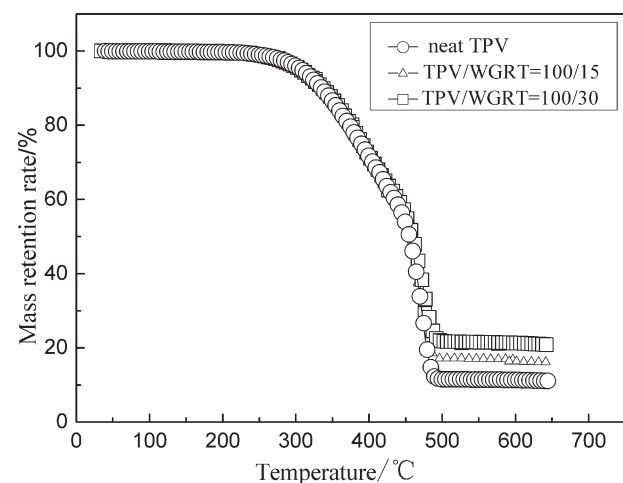


Figure 13. Effect of different WGRT dosage on TG curves of the blends.

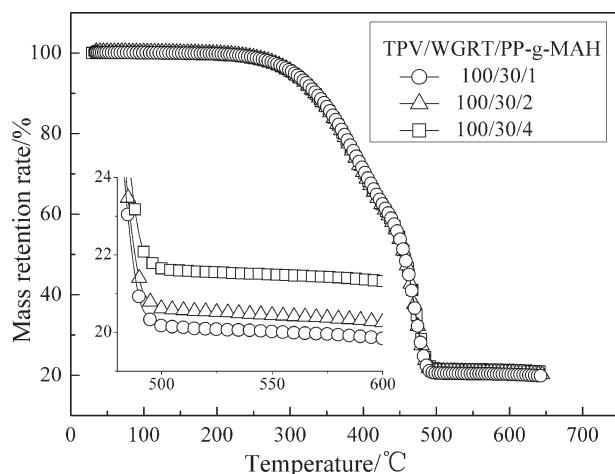


Figure 14. Effect of different compatibilizer dosage on TG curves of the blends.

age, as a result of the increased interface bonding between the WGRT powders and TPV that was compatibilized by more PP-g-MAH. So the addition of PP-g-MAH had a positive influence on the thermal stability of blends.

Dynamic Mechanical Analysis of Blends

The curves in Figure 15 reveal that the storage modulus of blends increased with WGRT dosage. WGRT is composed of crosslinked structure which has strong ability to store elastic deformation, so the storage modulus of blends could be improved significantly by mixing WGRT.

Figure 16 shows the two maximum values at -78°C and -7.7°C , which could be used to characterize the glass-transition temperature (T_g) of the blends.²² The low glass-transition temperature is the contribution of rubber phase and the high glass-transition temperature is the contribution of plastic phase.²³ Hereby, it can be considered that -78°C is the T_g of EPDM and -7.7°C is the T_g of PP. Moreover WGRT had a glass-transition temperature at -69.5°C , and the two glass-transition temperatures of the blends moved toward the lower temperature

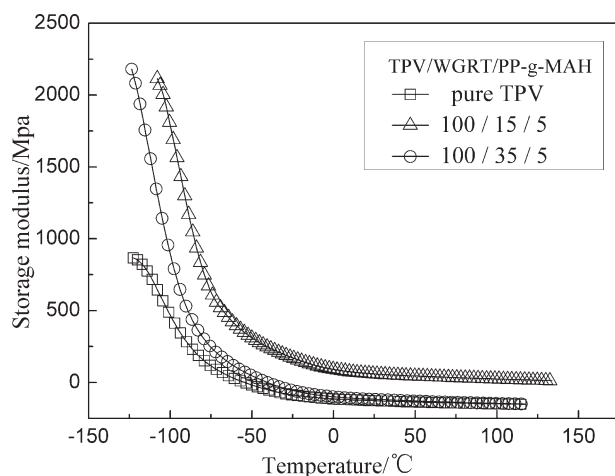


Figure 15. Effect of WGRT dosage on storage modulus of the blends.

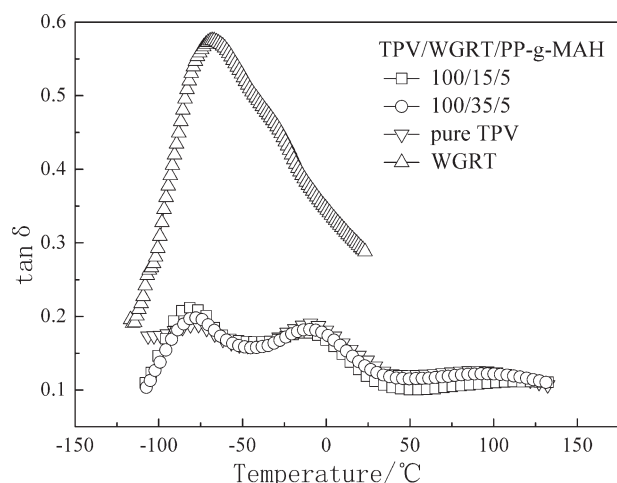


Figure 16. Effect of WGRT dosage on temperature-damping curves of the blends.

direction compared with that of neat TPV. It means the addition of WGRT improved the low-temperature property slightly.

CONCLUSIONS

Mechanical properties of the blends mixed with WGRT became worse than that of neat TPV. Tensile strength, tear strength, and elongation at break of the blends increased with the decrease in WGRT particle size and decreased with WGRT dosage. Meanwhile processing liquidity became worse by mixing WGRT.

The comprehensive performances of blends retained optimum when PP-g-MAH was used as compatibilizers between WGRT and TPV, and tensile strength and tensile permanent deformation were superior to neat TPV. The flowability of blends became better with the increased dosage of PP-g-MAH. SEM photomicrographs also showed that PP-g-MAH could effectively increase the compatibility to enhance the comprehensive properties of blends.

Thermal stability of the blends maintained well through thermal stability analysis. And it was evident that the low-temperature property of the blends was improved by WGRT through dynamic mechanical analysis.

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