

## Research Article

# Short Nylon Fibers Waste Modified with Glycidyl 3-Pentadecenyl Phenyl Ether to Reinforce Styrene Butadiene Rubber Tread Compounds

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Received 31 October 2018; Revised 16 January 2019; Accepted 29 January 2019; Published 14 February 2019

Academic Editor: Milan Marić

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The utilization of waste fibers represents an important environmental benefit and great economic savings for the community. In this study short nylon fibers waste was modified with Glycidyl 3-Pentadecenyl Phenyl Ether (GPPE) in the presence of Triethylamine/Ammonium persulfate by a simple two-step procedure. The reinforcing effects of modified fibers (MNSF-2) on the vulcanization characteristics, mechanical properties, dynamic mechanical properties, and the wear resistant property of Styrene Butadiene Rubber (SBR) tread compounds were investigated. The addition of the MNSF-2 resulted in slightly lower minimum torque ( $M_i$ ) and maximum torque ( $M_H$ ), as well as longer cure time ( $t_{90}$ ) and scorch time ( $t_{10}$ ) of tread compounds. The deterioration of tensile strength and elongation at break of the tread compound containing short nylon fibers waste (NSF) was apparent. Conversely, the modified fibers showed reinforcing effect on tread compounds. The tensile strength values of compounds increased with MNSF-2 content, passed through a maximum value, and then reduced slightly. The modulus and the tear strength of compounds increased significantly with fiber loadings. The highest tear strength value was observed in 8phr MNSF-2 reinforced SBR compounds, 31.9% higher than that of the gum compound. Meanwhile elongation at break of MNSF-2 compound maintained a relative high value than that of NSF/SBR compound. The addition of NSF exaggerated wear volume of compounds. However, the wear resistance of MNSF-2 compounds was superior to that of NSF compounds and comparable with that of the gum compound. The DMA results reveal that  $E'$  and  $\tan \delta$  values decreased at elevated temperature. Meanwhile enhanced storage modulus in MNSF-2/SBR tread compound can be observed. It is worth highlighting that MNSF-2/SBR compounds show higher  $\tan \delta$  at 0°C, indicating improved wet traction of tread compounds, while  $\tan \delta$  at 60°C maintains almost the same value as that of the gum sample. The results of this study are encouraging, demonstrating that the use of short nylon fibers waste in composites offers promising potential for the green tire application.

## 1. Introduction

Short fibers/rubber composites exhibit combined behaviors of the soft and elastic rubber matrix as well as stiff and strong fibrous reinforcement. These composites have been successfully used in production of V-belts, hoses, tire treads, and complex shaped mechanical goods [1]. Generally, the degree of reinforcement by short fibers depends on many factors such as the rubber matrix, the type of fiber, the concentration and orientation of the fibers, fiber aspect ratio, and the interaction adhesion between fiber and rubber [2, 3]. In more recent work, short nylon fibers were incorporated

into different rubber matrices such as Natural Rubber [4–7], Styrene Butadiene Rubber [8], Acrylonitrile Butadiene Rubber [9–12], and Chloroprene Rubber [13], and it was reported that modulus, tear strength, and heat build-up of rubber compound increase by the addition of fibers. However, tensile strength and elongation at break showed a drastic drop in all cases, which has been attributed to the relative higher modulus of short fibers and an indication of fiber-rubber adhesion [4, 7].

Fiber-matrix adhesion in short fiber-rubber composites has been a field of extensive researches [14]. Strong interface adhesion between fibers and matrices was achieved

by different techniques, which include a tricomponent dry bonding system based on hydrated silica–resorcinol–hexamethylenetetramine [4], the mercerization and acetylation chemical treatments of short fiber [15], the fiber surface treatment by resorcinol-formaldehyde latex (RFL) [2], the urethane based bonding agent [7], and NR-g-MAH compatibilizer [5]. The results showed that the strong fiber-matrix interface adhesion formed was feasible for the load being effectively transferred to the fiber to obtain a high-performance composite [4].

In engineered truck tread compounds, a low content of well dispersed short fibers is usually introduced to improve mechanical and dynamic properties [6]. The relative high modulus short fibers increased the rigidity of rubber tread compound effectively, resulting in lower tire deflection and superior rolling resistance of the tire in service [16]. Walker [6, 17] reported that tear strength and fatigue thermogenesis of the rubber tread compound were improved by the addition of modified cellulose short fibers. However the existence of strong interface further elevated the rigidity properties of rubber and consequently reduced the elasticity characteristic of the composites [1, 10]. A research [18] showed that the elongation at break, an indication of the elasticity, of resorcinol-formaldehyde treated short fiber/NR compounds dropped substantially to 70%, compared to that of their counterparts having no bonding agents. The reason was attributed to the strong adhesion and a rapid dissipation of energy at the filler-matrix interface due to the initiation of a catastrophic rupture [8].

Rubber elasticity is essential for many important tire properties such as wet traction performance [19, 20]. The elastic and more compliant rubber compound increases the contact area and that in turn increases friction coefficient, which is feasible of the improved wet traction of a tire [21]. It can be expected that adequate interphase adhesion between short fibers and rubber matrix is desired for obtaining the well-balanced properties of both wet traction and rolling resistance but also maintaining wear resistance of rubber tread compound in tire service.

Nowadays, Management of End-of-Life Tires has become a critical problem worldwide. During the process of tire recycling, approximately 10% of waste short fibers which mainly consist of nylon, polyester, or cellulose are obtained [22]. Although the utilization of these large quantities waste fibers represents an important environmental benefit and great economic savings for the community, it has progressed slowly. Few studies in the literature focus on waste fibers on the properties of tire tread compounds [23, 24].

Our previous research results had confirmed that short nylon fibers waste maintained their length upon mixing and had reinforcing effects on Ethylene Propylene Diene Monomer rubber [25]. Moreover, in our lab we had successfully employed Glycidyl 3-Pentadecenyl Phenyl Ether (GPPE), a renewable derivative from cashew nut industry, as a compatibilizer to improve the interface adhesion between SBR and nanoparticle fillers [26, 27]. The presence of the epoxy propyl ether group and  $C_{15}$  unsaturated side chain in GPPE molecule, as shown in Figure 1, facilitates chemical reactions of GPPE with many chemical compounds [28].

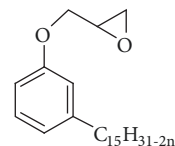


FIGURE 1: The molecule structure of GPPE.

The  $C_{15}$  unsaturated side chain of GPPE was available for creating strong and flexible interaction between filler and rubber matrix [26].

In this study we utilized GPPE to modified short nylon fibers waste to form adequate interfacial bonding between fibers and rubber matrix for obtaining high-performance tread compound. The reference compound used is a typical tire tread composition consisting of SBR matrix and the key ingredients. The modified method and the fibers contents on the properties of SBR tread compound were investigated.

## 2. Experimental

**2.1. Materials.** Short nylon fibers waste with 0.5~2 cm length and 15~45 $\mu$ m diameter was supplied by Dong-GuangHuangYu, Inc., China. Acrylic acid (AA), GPPE, Triethylamine (TEA), Ammonium persulfate (APS), and Polyoxyethylene(10) ether (OP-10) were purchased from ShangHai JingChun Sheng Hua Inc., China, and used as received. SBR (1502) was obtained from China Petrochemical Corporation. All other ingredients used were of commercial grade.

**2.2. Modified Fibers Preparation.** AA and APS were dissolved in purified water at certain ratio, stirring dispersion at 25°C for 1 hour to obtain fully mixed solution A. GPPE, TEA, and OP-10 were mixed in purified water, stirring at 25°C for 20min to prepare solution B.

A simple two-step method was used to modify fibers. In the first step, solution A was sprayed into short nylon fibers waste which was predispersing in a high-speed mixer at 1400r/min speed for 20min. Subsequently the resultant fibers were taken out and placed into a vacuum oven at 70°C for 2.5 hours to obtain the pretreated fibers (marked as MNSF-1). In the second step, MNSF-1 was mixed with solution B following the procedure similar to the first step to obtain the modified fiber (marked as MSNF-2). MSNF-2 was used in the rubber tread compound formula.

The modified fiber was dipped into ethyl alcohol for 12 hours then washed by deionized water several times to remove the unreacted monomer. After vacuum dried at 90°C for 24 hours, the purified fiber was characterized by FTIR and SEM.

**2.3. Preparation of Short Fibers/SBR Tread Compounds.** The formulation of the compounds was as follows (parts per hundred rubber, phr): 100 phr SBR, 5 phr zinc oxid, 1.5 phr stearic acid, 1.5 phr N-cyclohexyl-N-phenyl-p-phenylenediamine, 1.8 phr N-cyclohexyl-2-benzothiazole sulfonamide, 35 phr carbon black (N330), 35 phr silica, 25 phr aromatic oil, and 2

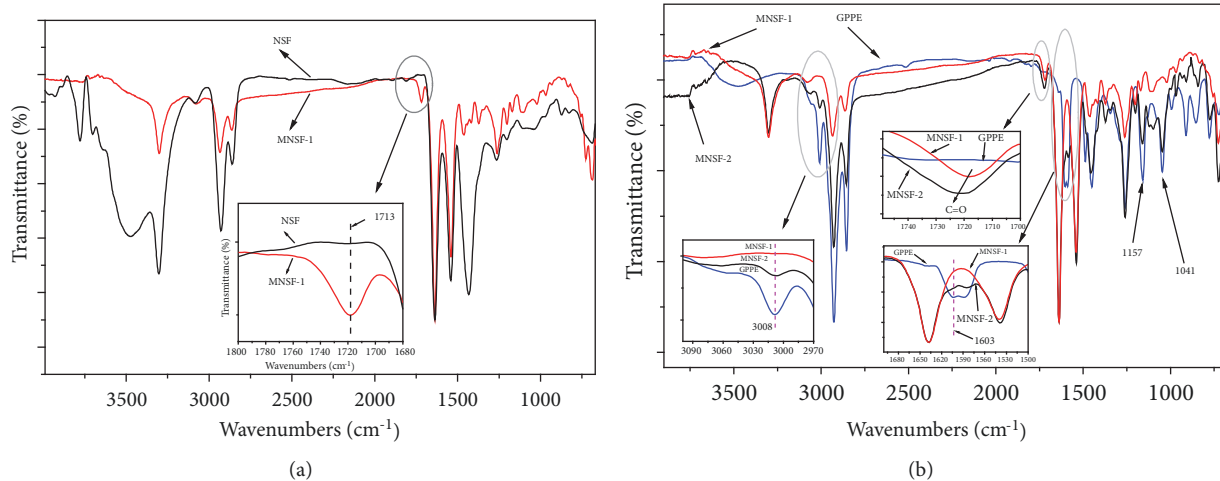


FIGURE 2: FT-IR spectra of GPPE and short fibers with and without modification.

phr sulphur. Various short fibers with different contents were compared in the formulation.

Mixing was carried out in a laboratory two-roll mill (150×300mm) at a friction ratio of 1:1.2 according to ASTM standard D 3184-80. The roll temperature was kept at about 50°C during mixing. SBR was masticated on the mill for 2 min, followed by the addition of ingredients except fibers. Fibers were added at the end of the mixing process. We added the fibers evenly to the batch and passed the batch through the mill at a nip of 2 mm continuously for 2 min, then removed the batch from the mill. Subsequently the batch was passed through the mill five times at a nip of 1mm, folding it back on itself each time. We were paying attention to maintain approximately uniform orientation of fibers in the calender direction of mill and making sure most fibers were aligned in the same direction. Afterward the master batch was sheeted out to 2 mm thickness for the vulcanization.

The vulcanization was carried out in a hot press (QLB-400×400) for the optimum cure time determined by a disc rheometer. The test specimens were punched from the molded sheet along the fiber orientation. The specimen in which short fibers paralleled to the calender direction denotes as the longitudinal orientation direction (L direction), reversely as the transverse orientation direction (T direction).

**2.4. Characterization.** Fourier Transform Infrared spectroscopic analysis (FTIR) was carried out using the Nicolet 380 spectrophotometer (Thermo Scientific, Inc.). All samples were scanned as total reflection from 600  $\text{cm}^{-1}$  to 4000  $\text{cm}^{-1}$ .

The cure characteristics including minimum torque ( $M_L$ ), maximum torque ( $M_H$ ), optimum cure time ( $t_{90}$ ), and scorch time ( $t_{10}$ ) were determined with a Monsanto Oscillating Disc Rheometer R-100 at 160°C in accordance with ASTM method D-2084.

Mechanical properties of the vulcanized composites were measured by using a universal tensile testing machine (Shenzhen Sans Materials Testing Co., Ltd., China) under ambient conditions (at  $25 \pm 2^\circ\text{C}$ ). Tensile strength and tear strength

were tested according to ASTM: D412-06-2 and ASTM: D624-00 (2012) standard, respectively. The speed of jaw separation was 500 mm/min. The hardness of the rubber composites was measured by Shore A hardness tester as per ASTM: D2240-05(2010) standard.

Wear resistance, representing in terms of volume loss, was performed using Akron-type abrasion tester WML-76.

Dynamic Mechanical properties were investigated using Netzsch DMA tester 242; the temperature sweep test was performed; i.e., the temperature was scanned from -20°C to 80°C at 5°C/min under frequency and tensile strain of 1Hz and 1%, respectively.

The morphology of fibers and the tensile fractured surface of composites were measured by using Scanning Electron Microscopy (XL-30FEG, Philips, Inc.) at an accelerating voltage of 10kV. The surface was sputter coated with a thin layer of gold before SEM observation.

### 3. Results and Discussions

**3.1. FTIR Spectroscopy.** The FTIR spectra of GPPE and fibers before and after modification are shown in Figure 2. It can be seen from Figure 2(a) that the original fiber (NSF) presents the characteristic of nylon, with bands at 3300  $\text{cm}^{-1}$ , 3090  $\text{cm}^{-1}$ , 2933  $\text{cm}^{-1}$  and 2853  $\text{cm}^{-1}$  corresponding to the stretching vibration and the frequency doubling characteristic of NH and -CH<sub>2</sub>- groups, respectively. Meanwhile the peaks at 1550  $\text{cm}^{-1}$ , 1640  $\text{cm}^{-1}$  and 1265  $\text{cm}^{-1}$  can be attributed to N-H, C=O, and C-N vibration in amide group [29]. In case of the pretreated fiber (MNSF-1) similar nylon absorption characters can be observed, while the emergence of 1713  $\text{cm}^{-1}$  band confirms the introduction of carboxylic group onto the fiber surface [30].

Furthermore it can be seen from Figure 2(b) that 1041  $\text{cm}^{-1}$ , 1157  $\text{cm}^{-1}$ , 1603  $\text{cm}^{-1}$  and 3008  $\text{cm}^{-1}$  bands assigned to the structural characteristic of GPPE [31] appear in modified fiber (MNSF-2), indicating the occurrence of chemical reactions between GPPE and fiber surface [32]. The peak shift of C=O stretching vibration absorption observed in MNSF-2

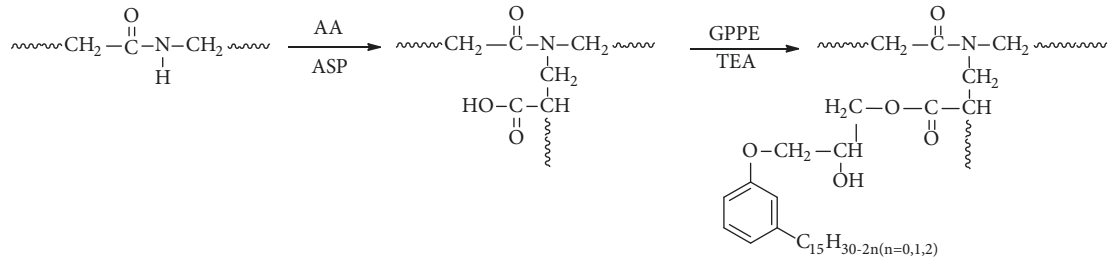


FIGURE 3: Grafting mechanism.

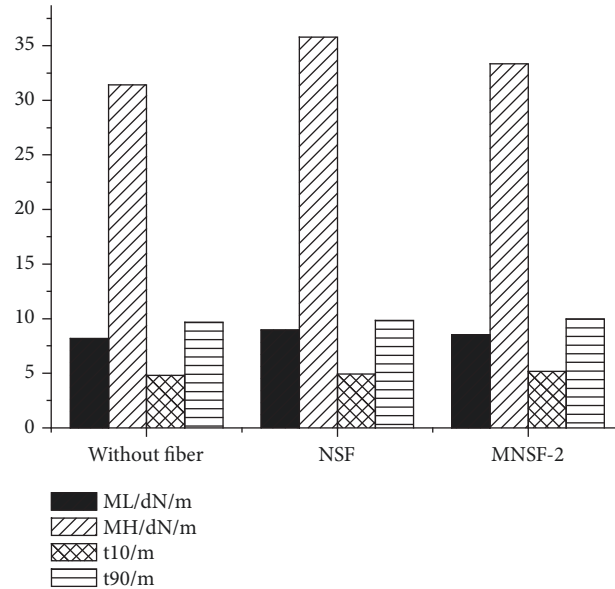


FIGURE 4: Effects of short fibers on cure characteristics of tread compounds.

suggests the ester formation during the fiber modified process [33].

Chahira Makhoulf [34] reported the grafting of acrylic acid monomer onto nylon6,6 fibers using benzoyl peroxide as an initiator by graft copolymerization. They confirmed that the reaction took place between the nitrogen atom of nylon molecule and carboxylic group of acrylic acid. So in much the same way it was reasonable that nylon molecular was motivated by APS initiator to become macromolecular radicals, which made feasible the free radical reaction with AA monomer [35], resulting in fibers grafted with carboxylic group (MNSF-1) at the first step. Subsequently the open-ring esterification reaction took place between carboxylic groups of MNSF-1 fibers and the epoxy group of GPPE in the presence of amine catalyst, to obtain the modified fiber (MNSF-2). The presumable reaction mechanism scheme is proposed in Figure 3. It is worth noted that  $C_{15}$  unsaturated carbon chain was introduced into MNSF-2 molecule structure [32].

**3.2. The Properties of Rubber Tread Compound.** The modified fiber (MNSF-2) was incorporated into SBR tread compounds formula. The effects of fiber contents and orientation on properties of tread compounds were investigated.

**3.2.1. The Cure Characteristics.** The cure characteristics of tread compounds with 2phr fibers and without fibers are shown in Figure 4. The addition of fibers resulted in slightly higher  $M_H$  and  $M_L$  due to the rigid characteristic of nylon fibers [11]. However, the  $M_H$  and  $M_L$  value of MNSF-2 compounds was lower than that of unmodified fiber (NSF). Meanwhile  $T_{10}$  and  $T_{90}$  values of MNSF-2 compounds were prolonged slightly. It can be explained that the interactions of the modified groups on the fiber surface with vulcanizing agents system probably occurred, which retarded vulcanization and thus reduced the crosslink density of the MNSF-2 compounds. Besides, some unreacted GPPE might play the role as a lubrication, leading to lower  $M_L$  and  $M_H$  of MNSF-2 compounds.

Figures 5 and 6 show the dependence of compounds vulcanization curves on MNSF-2 content.  $T_{90}$  value of MNSF-2 compounds increased with fiber contents, which might be attributed to the polar groups on modified fibers reacting with vulcanizing agents necessary for sulphur vulcanization [7]. In addition, vulcanization curves of tread compounds with or without fibers show flat plateaus, which means that high physical properties were liable to be maintained during vulcanization progress, which meets the processing requirements of tire rubber tread [36].

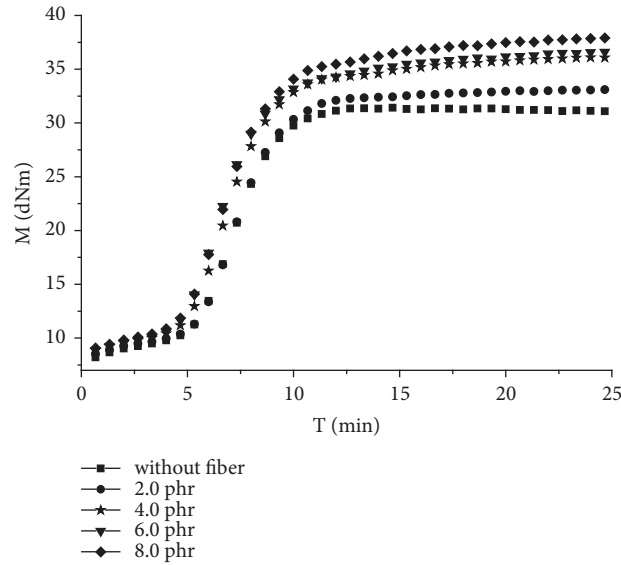


FIGURE 5: Effects of MNSF-2 content on cure characteristics of tread compounds.

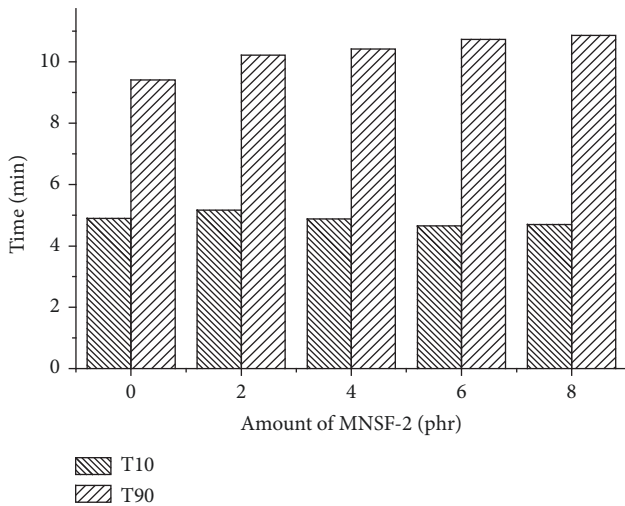


FIGURE 6: Effects of MNSF-2 content on  $t_{10}$  and  $t_{90}$  of tread compounds.

3.2.2. *The Mechanical Properties.* The superior interface adhesion of the modified fiber compounds to the unmodified fiber (NSF) has been verified by mechanical properties measurements as shown in Figure 7. The deterioration of tensile strength and elongation at break of tread compound with the addition of NSF was apparent. This might be attributed to the poor interface adhesion between original fibers and rubber matrix.

The modified fibers showed reinforcing effect on rubber compounds. With the increase of MNSF-2, the tensile strength increased to a maximum value and then decreased slightly. Both modulus and tear strength of fiber compounds were significantly higher than that without fibers. The highest tear strength value was observed in 8phr MNSF-2 reinforced SBR compounds, with 31.9% increase compared to

the gum rubber. Meanwhile, elongation at break value of MNSF-2/SBR compounds was higher than that of NSF/SBR compounds. It is known that the rigid short fibers are apt to restrict rubber matrix from deformation which contribute to elevated modulus and tear strength of composites [10]. MNSF-2 compounds showed lower hardness values and the modulus at 100% in T direction (a true measure of the rubber matrix property) compared to NSF compounds, indicating the effects of modified fibers on curing reactions of the rubber compounds, thus lowering the crosslink density as mentioned before. The stress-strain curve of the compound with 8phr modified fiber exhibited obvious yield plateau as shown in Figure 8, confirming the restricted effects with high fiber content.

The introduction of  $C_{15}$  carbon chain group enhanced the hydrophobicity of MNSF-2, promoting the uniform dispersion of this fiber within rubber matrix. Meanwhile, the cocrosslinking reaction might occur between the MNSF-2 fiber and SBR during vulcanization process, resulting in improved interface bonding and comparative mechanical properties of the modified fibers/SBR tread compounds. Furthermore, the superior elongation at break of MNSF-2/SBR compounds confirmed the formation of flexible interfacial bond between fibers and rubber matrix [26]. The developed flexible interface bond of tread compound is feasible to the well balance properties for a truck tire service.

In terms of the anisotropy characteristic, fibers aligned in L direction showed higher mechanical properties than in T direction, which should be due to the high L/D ratio and orientation of short fibers.

3.2.3. *The Wear Resistant Property.* The effect of short fibers content on the wear volume of tread compounds is shown in Figure 9. The higher fibers content was, the larger wear volume was obtained. The addition of NSF reduced the wear resistant property of tread compounds. However, the



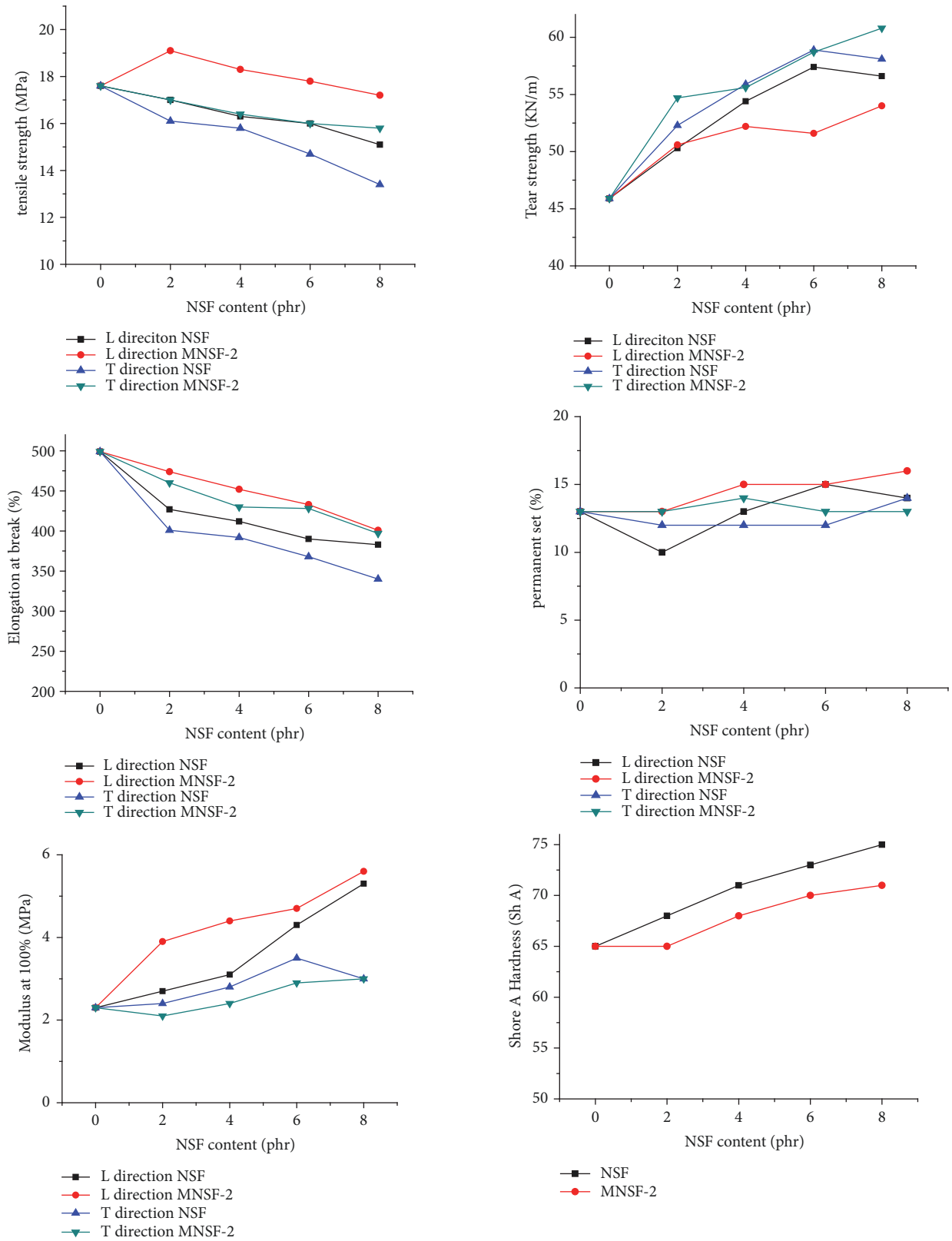


FIGURE 7: Effects of fibers content and the orientation on mechanical properties of compounds.

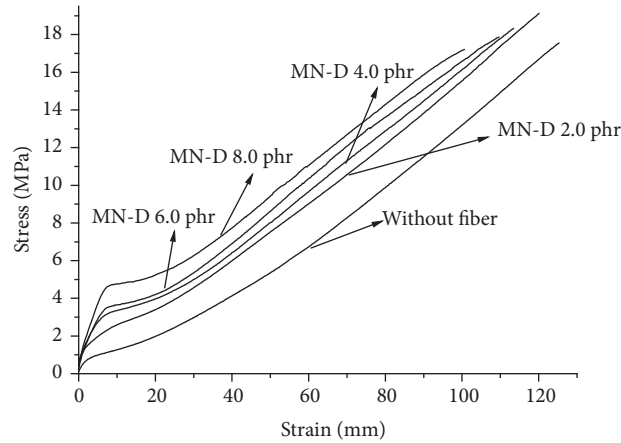


FIGURE 8: MNSF-2 contents on stress-strain curves.

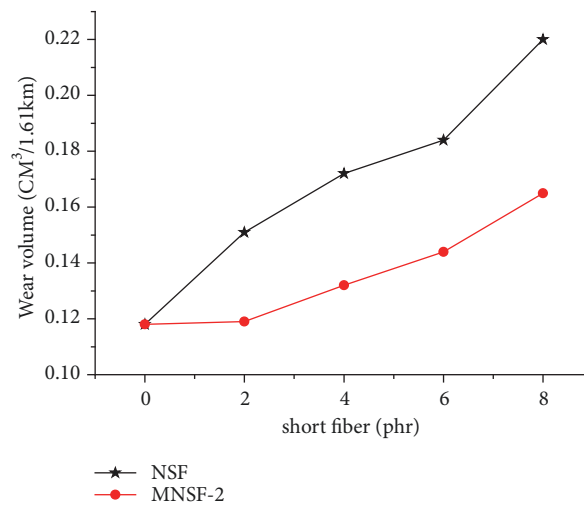


FIGURE 9: Short fiber contents on wear volume of tread compounds.

wear volume value of MNSF-2 compounds is lower than that of NSF compounds and is comparable with the wear volume of gum compound as MNSF-2 content being 2phr. MNSF-2 compounds showed better wear resistance than NSF compounds. The reason may be that wear resistant property of tire tread is related to the combination of strength and elasticity of rubber compounds [37]. The grafting of long carbon chains onto fiber surface was feasible to form strong and flexible interphase bond, which helps to maintain the elasticity of rubber [38] to some extent. Nevertheless, the decreased crosslink density due to the reaction of modified fibers with the vulcanizing agents had negative effects on the wear resistance.

**3.2.4. Dynamic Mechanical Properties.** The effects of modified fiber content on dynamic mechanical properties of the tread compound at various temperatures between  $-20^{\circ}\text{C}$  and  $80^{\circ}\text{C}$  are shown in Figure 10. The storage modulus ( $E'$ ) values decrease at elevated temperature. The probable reason is that the elasticity of rubber and the interphase adhesion become weak as temperature increases, leading to the considerable decrease of  $E'$  value. Nevertheless, the utilization of modified

fibers enhances storage modulus of tread rubber compound compared to the original fibers. The improved  $E'$  value of the compound with high MNSF-2 content is most probably due to the rigidity characteristic of fibers and the enhanced interface bonding, which effectively restrict the rubber from deformation [36]. The loss factor ( $\tan \delta$ ) curves showed similar drop trend at elevated temperature as  $E'$  curves.

The relationship between MNSF-2 content and  $\tan \delta$  is presented in Figure 11. It is worth highlighting that all MNSF-2/SBR compounds show higher value of  $\tan \delta$  at  $0^{\circ}\text{C}$ , while maintaining almost the same  $\tan \delta$  at  $60^{\circ}\text{C}$  as the gum sample. Normally it is an industry accepted practice in tire performance prediction to use loss factor at  $0^{\circ}\text{C}$  and at  $60^{\circ}\text{C}$  of the tread compound as predictors of tire wet traction and rolling resistance respectively [39]. The higher  $\tan \delta$  at  $0^{\circ}\text{C}$  of MNSF-2 compounds suggests the improved wet traction or wet-skid resistance of tread compounds. This may be related to the flexible interfacial adhesion between modified fiber and rubber matrix, as tread rubber compound which remains flexible at low temperatures can establish close contact with the underlying surface that increases the wet-skid resistance of the tire tread [40].

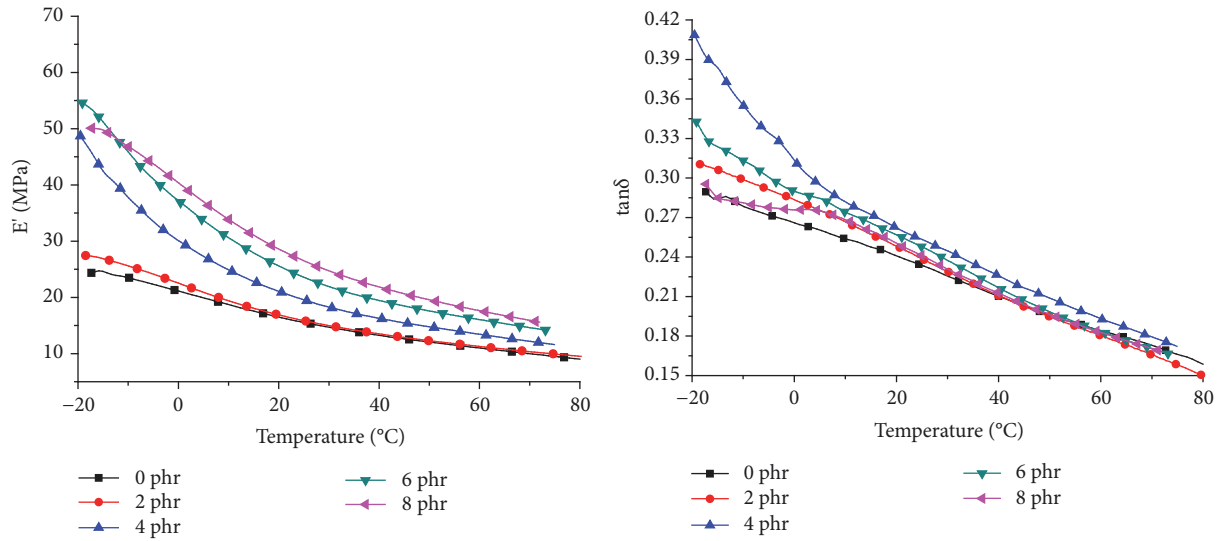


FIGURE 10: MNSF-2 content on  $E'$  and  $\tan \delta$  of compounds at various temperatures.

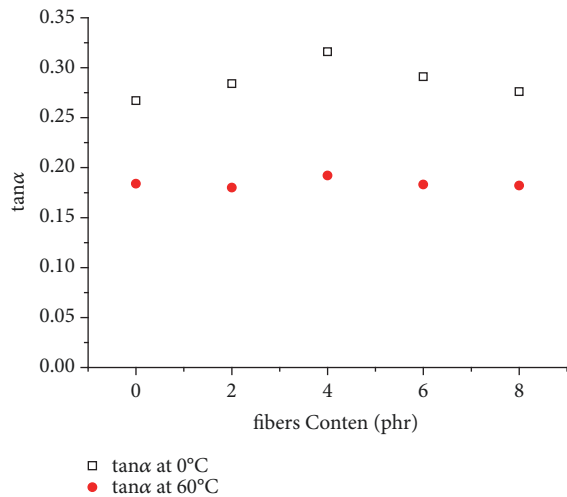


FIGURE 11: The relationship between MNSF-2 content and  $\tan \delta$ .

**3.3. Morphology.** The SEM micrographs of short fibers and fractured surface of compounds are shown in Figure 12. It can be observed that the modified fiber exhibits rougher surface compared to the NSF. Meanwhile broken and pull-out fibers from the distorted holes can be seen in MNSF-2 compound sample, suggesting better interfacial adhesion of modified fiber compound than that of original fiber. The SEM results agreed well with the improved mechanical properties of MNSF-2 tread compounds mentioned above.

## 4. Conclusions

In this work short nylon fibers waste modified with GPPE was obtained by a simple two-step procedure. The effects of short fibers on vulcanization characteristics, mechanical properties, dynamic mechanical properties, and wear resistance of SBR tread compounds were investigated.

The fiber modified method and fibers contents affected the curing properties of rubber compounds. The addition of the MNSF-2 resulted in slightly lower  $M_H$  and  $M_L$  of tread compounds. The  $t_{90}$  value increased with the modified fibers contents. Meanwhile all SBR tread compounds with fibers show the same flat plateau vulcanization curves as the gum compounds.

The deterioration of tensile strength and elongation at break of tread compound containing NSF was apparent. Conversely, the modified fibers showed reinforcing effect on tread compounds. The tensile strength values of compounds increased with MNSF-2 content, passed through a maximum value, and then reduced slightly. The modulus and the tear strength of compounds increased significantly with fiber loadings. The highest tear strength value was observed in 8phr MNSF-2 reinforced SBR compounds, with 31.9% higher than that of the gum compound, while elongation at break of the MNSF-2 compound maintains a relative high value than the NSF/SBR compounds.

The addition of NSF exaggerated abrasive volume of compounds at high fiber content. However, the wear resistance of MNSF-2 compounds is superior to that of NSF compounds and comparable with that of gum compound. The DMA results reveal that  $E'$  and  $\tan \delta$  values decrease at elevated temperature. The utilization of modified fibers enhanced storage modulus of tread compound. MNSF-2/SBR compounds show higher  $\tan \delta$  at  $0^\circ\text{C}$  indicating improved wet-skid resistance, while  $\tan \delta$  at  $60^\circ\text{C}$  maintains almost the same value as that of the gum sample.

## Data Availability

The data used to support the findings of this study are available from the corresponding author upon request. There are no linked research data sets for this submission. The following reason is given: data will be made available upon request.



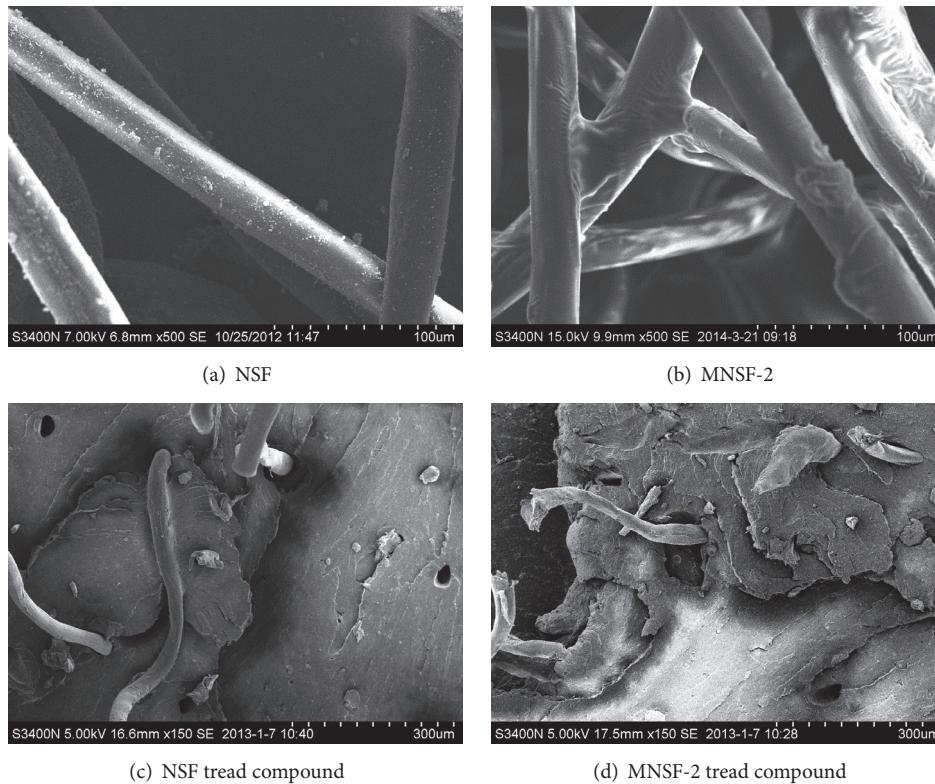


FIGURE 12: SEM of short fibers and tread compounds.

## Conflicts of Interest

The authors declare that they have no conflicts of interest.

## Acknowledgments

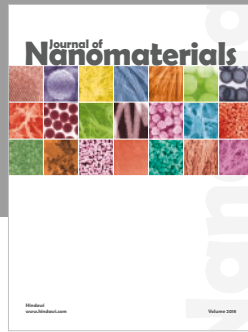
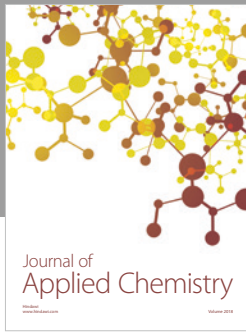
We would like to acknowledge the Science & Technology Key Program of Guangzhou, China (Grant No. 2013Y2-00116), South China Tire & Rubber Co., Ltd., for supporting this research.

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