**Effect of Surface Treatment with Potassium Permanganate on Ultra-High Molecular
Weight Polyethylene Fiber Reinforced Natural Rubber Composites**

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Abstract: The effects of surface treatment using potassium permanganate on ultra-high molecular weight polyethylene (UHMWPE) fibers reinforced natural rubber (NR) composites were investigated. The results showed the surface roughness and the oxygen-containing groups on the surface of the modified fibers were effectively increased. The NR matrix composites were prepared with as-received and modified UHMWPE fibers added 0-6 wt%. The treated fibers increased the modulus and tensile stress at a given elongation. The tear strength increased with increasing fiber mass fraction, attained maximum values at 4 wt%. The hardness of composites exhibited continuous increase with increasing the fiber content. The dynamic mechanical tests showed that the storage modulus and the tangent of the loss angle were decreased in the modified UHMWPE fibers/NR composites. Several micro-fibrillations between the treated fiber and NR matrix were observed, which meant the interfacial adhesion strength was improved.

Keywords: UHMWPE fiber; potassium permanganate; natural rubber; composites; mechanical properties.

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1. Introduction

The rubber matrix composite is one of the most important materials and applied in many fields [1]. Power, belts, fibers and so on are usually used as reinforcements. Among all fiber reinforcing materials, ultra-high molecular weight polyethylene (UHMWPE) fiber is a promising material because of its extraordinary properties, such as high strength, high impact resistance, chemical stability, low moisture absorption, lightweight, low coefficient of friction and self-lubricating properties, etc [2-5]. However, UHMWPE fibers merely possesses insufficient adhesion to polymer matrix due to lack of polar functional groups and a low surface free energy [6,7], resulting in limiting the use of UHMWPE fiber reinforced composites.

To satisfy the various technological demands, a sufficient interfacial adhesion between fibers and composite matrix is of crucial importance [8,9]. In order to improve the interfacial adhesion of UHMWPE fibers and polymer matrix, many methods for the surface modification of UHMWPE fibers have been previously reported, such as acid etching [10, 11], plasma deposition [12, 13] and UV initiated graft copolymerization [14]. Through increasing the roughness and functional groups on the fiber surface, the researchers have proved the mechanical properties of UHMWPE fibers reinforced polymer composites are improved.

At present, the modification of the fiber is a key area of research [15]. The aim of the present work is to discuss the effects of potassium permanganates treatment on UHMWPE fibers reinforced polymer matrix composites. We used potassium permanganate solution, which was a strong oxidizing agent and low cost, to treat UHMWPE fibers in order to improve the bonding strength between the fiber and the natural rubber (NR) matrix. The NR composites including as-received and potassium permanganates modified UHMWPE fibers were prepared and the effect

of potassium permanganates treatment on the mechanical properties of the composites were discussed.

2. Experimental

2.1. Materials

The UHMWPE fibers in this study were supplied by Dacheng Advanced Material Co.,Ltd., China and the length was 0.5 cm. The density and the diameter of fiber were 0.97 g cm^{-3} and about 40 μm respectively. Natural rubber and other chemical reagents (analytical grade) were purchased from Ningbo Rubber Co., Ltd. and Sinopharm Chemical Reagent Co., Ltd, respectively.

2.2. Surface treatment of fibers

The UHMWPE fibers were treated by KMnO_4 solution and the solution was a compound of KMnO_4 and HNO_3 (weight ratio is 1:30). UHMWPE fibers were immersed in the KMnO_4 solution for about 1 min at room temperature and then rinsed with distilled water several times until a constant pH was reached. The treated fibers were finally dried in a vacuum oven under around 0 Pa at room temperature for 12 h.

2.3. Preparation of UHMWPE fibers reinforced NR composites

To obtain a vulcanizing mixture, 1.0 phr sulfur (1.0 parts per hundred resin, phr), 5.0 phr zinc oxide, 2.0 phr stearic acid, two kinds of vulcanizing promoters of 1.0phrMBT (2-mercaptobenzothiazole) and 0.5 phr TMTD (tetramethylthiuram disulfide), and two kinds of antioxidants of 1.0 phr antioxidant 4010 (N-cyclohexyl-N'-phenyl-p-phenylenediamine) and 1.5 phr antioxidant RD (poly(1,2-dihydro-2,2,4-trimethyl-quinoline)) were weighed for a hundred resin of NR. Small bits of NR (ca. 2 mm square) were put on a mixing roller and then the above mixture powder was added to a softened and drawn sheet of NR on the roller. In addition, as-received

and treated UHMWPE fibers, which were 1 wt%, 2 wt%, 3 wt%, 4 wt%, 5 wt%, 6 wt%, were added to it to make a pre-vulcanized rubber. Subsequently, the pre-vulcanized rubber sheet was cut into small pieces and a given amount of them was put in the metal mold and vulcanized.

2.4. Instrumental analysis

Fourier transform infrared (FTIR) spectroscopy was used for analyzing the changes of chemical structure. UHMWPE fibers were mixed with KBr and pressed into pellets. The mixture was analyzed with a Thermo Fisher Scientific Inc. Nicolet 6700 FTIR spectrometer.

The morphological observations were executed using a scanning electron microscope (SEM) (TM3000, Hitachi High-Technologies Co., Ltd.). In order to be observed more clearly and have better conductivity, the surfaces of the specimens were sputter-coated with gold prior to their observation.

Dynamic mechanical analysis was performed with a DMA+1000 dynamic measurement system (Metravib, France) using a constant frequency of 10 Hz. the specimens were heated at a rate of 5 °C min⁻¹ from -80 to 0°C.

Tensile testing was performed on a WDW-20 universal testing machine (Zwick Roell, Germany) at a cross head speed of 500 mm min⁻¹ at room temperature. The dumbbell-shape specimen (Fig.1) with width 5 mm, thickness 2 mm and gauge length 20 mm was tested by following ISO 37:2005. Five specimens were tested and averaged for each sample.

Tear testing was also performed on the WDW-20 universal testing machine (Zwick Roell, Germany). The test specimen was unnicked angle shape (without a nick of specified depth) with 2 mm in thickness, according to ISO 34-1: 2004. The rate of separation of the grips was 500 mm min⁻¹ and five test pieces per sample were tested.

The test for Shore A hardness was carried on LX-A rubber Shore A hardness tester (Handpi Chemical Machinery Inc.), according to ISO 7619-1: 2004. For each test and type of composites, 10 specimens were tested and the average values were reported.

3. Results and discussion

3.1. Surface treatment of UHMWPE fibers

In this work, FTIR was used to explore the fine surface information of the fibers. It is noticed that for both as-received and treated UHMWPE fibers the peaks at about 2840~2940, 1630, 1470 and 720 cm^{-1} are observed (Fig.2). They are attributed to $-\text{CH}_2$ stretching vibrations, $-\text{C}=\text{O}$ stretching vibrations, $-\text{CH}_2$ deformation vibrations and $-(\text{CH}_2)_n$ - rocking vibrations [16-18]. The relative intensity of $-\text{OH}$ stretching vibrations at about 3340 cm^{-1} and $-\text{C}=\text{O}$ bands increases after the treatment, indicating the quantities of $-\text{OH}$ and $-\text{C}=\text{O}$ groups on the fiber surface increase. The functional groups can enhance the interface bonding between fibers and rubber matrix, and thus improve the performance of composites.

XPS was used to analyze the chemical composition on the UHMWPE fiber surface. Fig. 3 shows the XPS survey spectra of as-received and potassium permanganate treated fibers. The amount of oxygen increases and carbon inversely for the treated UHMWPE fibers. Table 1 shows the O 1s/C 1s ratio obtained from the XPS survey spectra. It is seen that the ratio of as-received fibers is 0.031. M. S. Silverstein et al. [19] discussed the origin of oxygen on as-received fibers. They thought the oxygen on the surface was more likely to originate in oxidized low-molecular-weight fragments excluded during the crystallization process. If the oxygen was simply spinning oils that remained on the surface then rinsing the fiber should remove the oil from the fiber surface. The ethanol rinse, however, did not change the surface oxygen content. The O 1s /C 1s ratio rises

rapidly to 0.563 after modification. This rise in surface oxygen is due to the increase of O-containing groups.

Potassium permanganate treatment produces a dramatic change, as shown in Fig. 4. It can be seen that as-received UHMWPE fibers show relatively smooth surface. After the surface treatment, the highly developed cracks have now replaced the longitudinal striations. The cracks are caused by the degradation of the molecule chains on the fiber surface as a result of potassium permanganate etching [20]. These cracks result in a great increase of the surface area and, thus, provide more favorable conditions for various physical and chemical interactions between fiber and NR. Of treated UHMWPE fibers, the remarkable crack, into which rubber can penetrate to produce a mechanical interlocking between fiber and rubber matrix, is one of the most important factors in improving the fiber/NR adhesion.

3.2. Mechanical properties of UHMWPE fibers/NR composites

3.2.1. Tensile strength

The tensile mechanical behaviors of the NR and UHMWPE fibers/NR composites were characterized at room temperature. The stress-strain curves of pure NR and 2 wt% UHMWPE fibers/NR composites are shown in Fig. 5. Pure NR exhibited an elastic nonlinear behavior typical of amorphous polymers at a temperature above their glass transition temperature, where the stress continuously increased with the strain, and the stress of pure NR is lowest among the others. Addition of UHMWPE fibers increases the stress and beyond a certain strain, the stress starts to level-off approximately and then, rises up. To clarify the effect of surface treatment, we compare the two kinds of NR composites with as-received and treated fibers. In the case of as-received UHMWPE fibers/NR composites, the composites are apt to stretch at lower stress with

the increase of strain. This may be attributed to a lubricant-like behavior of untreated UHMWPE fibers which have only weak interaction to the rubber matrix. On the contrary, in the case of treated UHMWPE fibers/NR composites, it can be seen that the tensile stress becomes higher than that of NR and as-received UHMWPE fibers/NR composites. In our study, potassium permanganate solution was used to modify the inert fibers surface, and the mechanical properties of fibers/NR composites were improved. The main reason is that the better propagation of tensile stress to UHMWPE fibers was realized to some extent, because of the enhancement in the adhesion of fibers to NR matrix, as expected.

The Young modulus is determined from the first trace of the stress-strain curve at low deformation. From Fig. 5, we can see that in the system of treated UHMWPE fibers and NR, the modulus increases, compared with as-received UHMWPE fibers/NR composites. This implies that treated UHMWPE fibers can give rise to the remarkable interfacial interaction between the fibers and matrix rubber [21]. This result is in agreement with Sakurai et al. [22], who worked on UHMWPE fibers reinforced styrene-butadiene rubber composites.

The tensile stress at a given elongation is evaluated from Fig. 5, and the data is shown in Table 2. It is noticed that the tensile stress of as-received UHMWPE fibers/NR composites is increased by 133%, 87%, 34% and 14% at 50%, 100%, 200% and 300%, respectively, compared with the stress in the pure NR without UHMWPE fibers. The similar tendency is also observed in the treated UHMWPE fibers/NR composites. In other words, the treated fibers give rise to the increase in stress. The tensile stress of treated UHMWPE fibers/NR composites is increased by 224%, 144%, 70% and 42% at 50%, 100%, 200% and 300%, respectively, compared with the stress in the pure NR without UHMWPE fibers.

3.2.2. Tear strength

In the as-received and treated UHMWPE fibers/NR systems, the tear strength increases by 36% and 43% than that of NR (shown in Fig.6). A crack that happens in the matrix during drawing process cannot propagate across the fibers and has to stop there. Or the growing direction of the crack has to be changed to different directions. As a result, the interfaces between the fibers and natural rubber are peeled off to make larger crack at the expense of the energy. According to these behaviors, the strength is considered to be increased [23]. However, the strength increases no longer in the region of fiber content more than 4 wt%. It may be due to the occurrence of default in the interfacial region when the content of the fibers is high.

3.2.3. Hardness

Fig. 7 shows the hardness of the UHMWPE fibers/NR composites with different fiber loading. It is observed that the hardness increases with increasing fiber loading in both as-received and treated UHMWPE fibers/NR composites. Higher amounts of fibers are found to give higher hardness. Compared with pure NR, the hardness of as-received and treated UHMWPE fibers/NR composites is increased to 94% and 88%. However, the difference between as-received and treated UHMWPE fibers/NR composites added the same fiber content is not obvious.

3.2.4. Dynamic mechanical properties of UHMWPE fibers/NR composites

The dynamic mechanical properties of NR and UHMWPE fibers/NR composites were also performed. The variation of storage modulus (E') and tangent delta ($\tan \delta$) as a function of temperature are reported in Fig. 8 and 9. The dynamic properties reflect the amount of the energy in the composite stored as elastic energy and the amount of energy dissipated during the strain process. These properties are highly dependent on the existence of fillers: dispersion within the

matrix, volume fraction, geometrical characteristics, and load transfer from the filler to the matrix [24]. As illustrated in Fig. 8, at low temperatures, NR was in the glassy state and its storage modulus remained roughly constant at 1.5 GPa. This was due to the fact that in the glassy state, the molecular motions were largely restricted to vibration and short-range rotational motions. The glassy modulus are 1.9 and 0.5 GPa with the addition of as-received and treated UHMWPE fibers, respectively. It is remarkable that the as-received UHMWPE fibers/NR composites gives the highest energy dissipation in the temperature region over the glass transition temperature. This can be attributed to the very weak fiber/polymer bond in the composite [25]. As-received UHMWPE fibers/NR composites give an increase in E' in both the rubbery region and glassy region, treated UHMWPE fibers/NR composites only in the rubbery region. For instance, at -40 °C the storage modulus of the NR and the composites with as-received and treated UHMWPE fibers are 3.3, 20.7 and 16.1 MPa, respectively.

The evolution of the tangent of the loss angle as a function of temperature for NR and UHMWPE fibers/NR composites is presented in Fig. 9. The dynamic glass transition temperature was for all the materials close to -55 °C. An insignificant decrease of the maximum $\tan \delta$ values was observed when adding the UHMWPE fibers. The $\tan \delta$ peak height is 2.47, 1.35 and 1.22 respectively for NR, as-received and treated UHMWPE fibers/NR composites. The decrease of $\tan \delta$ peak reflects that UHMWPE fibers have effectively immobilized NR chains near the polymer-fiber interface due to favorable interfacial interactions with the NR matrix [26]. Meanwhile, the maximum $\tan \delta$ value of the composites with treated UHMWPE fibers are lower than that of as-received UHMWPE fibers/NR composites. This result indicates that there is a stronger interaction between treated UHMWPE fibers and NR matrix [27].

3.2.5. SEM observation of the fractured surface in UHMWPE fibers/NR composites

Fig. 10 shows the SEM images of the fractured surfaces of as-received and treated UHMWPE fibers/NR composites, which reflect the structural integrity and the adhesive features of the compositions in the composites. It can be evidently observed that for as-received UHMWPE fibers/NR composites, many fibers are pulled out from natural rubber (Fig. 10(a)) and UHMWPE fiber is rather clean with almost no distinct interfacial bond (Fig. 10(b) and (c)), showing poor interfacial adhesion. This is because as-received UHMWPE fibers with surface inertness cannot well bond with NR. However, in Fig. 10(d), it can be seen that less fibers were pulled out from matrix and the failure of the treated UHMWPE fibers/NR composites was accompanied by several micro-fibrillation of the fiber (as the arrows showed in Fig. 10(e) and (f)), indicating UHMWPE fibers are better bonded with NR. Such better interface bond should be attributed to that the chemical activity of the hydroxyl group on the surface of treated UHMWPE fibers promotes the adhesion between UHMWPE fibers and NR. Due to the effect of increased amount of hydroxyl group on the surface of treated UHMWPE fibers, UHMWPE fibers are bonded with NR [28].

4. Conclusions

In the current work, the effects of potassium permanganate surface treatment on the UHMWPE fibers reinforced NR composites were investigated and the following conclusions were obtained.

UHMWPE fibers were treated using potassium permanganate solution, and the chemical and the morphological changes were characterized by FTIR, XPS and SEM. The analyses indicated that after surface treatment, the content of oxygen-containing groups were increased on the surface of UHMWPE fibers and the SEM observation showed the increasing surface roughness with potassium permanganate treatment.

The modulus and tensile stress at a given elongation of treated UHMWPE fibers/NR composites increased markedly, compared with as-received UHMWPE fibers/NR composites and pure NR. This was due to the improved interfacial adhesion between fiber and NR matrix. The tear strength and the hardness of the composites increased with adding as-received and treated UHMWPE fibers. The results of the dynamic mechanical tests showed that the storage modulus and the tangent of the loss angle are decreased in the potassium permanganate treated UHMWPE fibers/NR composites. From SEM images, it was observed that there were several micro-fibrillations between the treated fiber and NR matrix, which meant the interfacial adhesion strength in the composite was great.

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Figure captions

Fig. 1. Dumbbell-shape tensile testing specimen.

Fig. 2. FTIR spectra of UHMWPE fibers.

Fig. 3. XPS survey spectra for as-received and treated UHMWPE fibers.

Fig. 4. SEM micrographs of (a) as-received fiber, (b) treated fiber.

Fig. 5. Stress-strain curves of NR and 2 wt% UHMWPE fibers/NR composites.

Fig. 6. Tear strength of UHMWPE fibers/NR composites.

Fig. 7. Hardness of UHMWPE fibers/NR composites.

Fig. 8. The curves of storage modulus versus temperature for NR and UHMWPE fibers/NR composites.

Fig. 9. The curves of loss factor ($\tan \delta$) versus the temperature for NR and UHMWPE fibers/NR composites.

Fig. 10. SEM images of fractured surface: (a), (b) and (c) as-received UHMWPE fibers/NR composites, (d), (e) and (f) treated UHMWPE fibers/NR composites.

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Fig. 8.

Fig. 9.

Fig. 10.

Table 1 [O1s]/[C1s] ratio of as-received and treated UHMWPE fibers.

Sample	[O1s]/[C1s]
As-received fibers	0.031
Potassium permanganate treated fibers	0.563

Table 2 Tensile stress of NR and UHMWPE fibers/NR composites at a given elongation.

Samples	Tensile stress at 50%/MPa	Tensile stress at 100%/MPa	Tensile stress at 200%/MPa	Tensile stress at 300%/MPa
NR	0.447±0.013	0.639±0.019	0.990±0.030	1.471±0.044
As-received fibers/NR composites	1.041±0.031	1.195±0.036	1.330±0.040	1.671±0.050
Treated fibers/NR composites	1.448±0.043	1.561±0.047	1.684±0.051	2.083±0.062