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Preparation and mechanical properties of the magnetorheological elastomer based on natural rubber/rosin glycerin hybrid matrix

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Abstract

To improve the mechanical properties of the natural rubber based magnetorheological elastomers (MREs), rosin glycerin ester was added into the carrier matrix to enhance wettability and dispersibility of CI particles. Dynamic performance, including shear modulus, loss factor and viscosity of non-vulcanized matrix was measured by rheometer. In comparison to the natural rubber based MREs, the MR effect of these hybrid matrix MREs were higher and they can reach to 112% when the mass fraction of CI particles is only 60%. The contact angle was tested by drop shape analysis system (DSA) and it was found that the compatibility between the iron particles and matrix was improved. In combination of the microstructure and mechanical property analysis, a possible mechanism was proposed. Finally, the loss factor and tensile strength were studied.

(Some figures may appear in colour only in the online journal)

1. Introduction

Magnetorheological elastomers (MREs) are a specific class of smart materials named magnetorheological (MR) materials, whose rheological or mechanical properties can be controlled by external magnetic field [1–7]. Due to their wide applications in shock absorption, noise reduction and other areas, MREs have attracted great attention and have been used in various devices such as the adaptive tuned vibration absorbers, mass dampers, sensors, actuators, base isolation, etc [8–10]. Generally, MREs are fabricated with soft magnetorheological magnetic particles and elastomers, thus most works on improving the mechanical properties of MREs focus on these two parameters.

The MR effect, which is usually calculated by the shear storage modulus, has been applied to evaluate the MR performance of MREs. Li and Zhang [11] used two categories of iron products with different particle sizes of 50 and 5 μm to fabricate a series of bimodal MRE samples.

With the optimal volume fraction of small particles in the bimodal particle mixture (23.3 wt%), the MR elastomer will have the best MR effect. Evan and Pedro [12] elucidated the effects of particle shape on the mechanical properties of the magnetorheological composite and found that the maximum magnetostrictive strain was reached at saturation for a particle contraction of about 0.61 and an oblate particle shape with aspect ratio of about 0.67, and that the effect was enhanced by larger concentrations of particles and by both strongly oblate and prolate shapes. Ivaneyko *et al* [13] showed that the magneto-mechanical behavior of magneto-sensitive elastomers is very sensitive to the spatial distribution of the magnetic particles. The results indicated that Young's modulus of MRE with simple and body centered cubic lattice decreases with an increase of magnetic field. In contrast to the simple cubic lattice, the body centered cubic lattice model allowed for an expansion of the magneto-sensitive elastomers along the direction of a magnetic field.

Besides the magnetic particles, the elastomer matrix also exhibited high influence on the MR effects of the MREs, because it determined the movement of the magnetic particles. Over recent decades, different polymers such as natural rubber [14], poly (styrene-*b*-ethylene-ethylene-propylene-*b*-styrene) (SEEPS) [15], PDMS [16], silicone rubber [17] and hybrid matrix [18] have been selected as matrix for the MREs, since the matrix impact greatly on the properties of the MRE. For example, the MR effect of SEEPS based MRE can reach as high as 700% while the MR effect of silicone rubber/PU hybrid matrix based MRE was only 35% [15, 18]. To further improve the mechanical properties of these polymer based MREs, many other additives were also introduced. Lokander and Stenberg [19] found that the relative MR effect can be increased by adding the plasticizers into the matrix, due to the decrement of the zero-field modulus. Qiao [15] *et al* mixed titanate coupling agent modified CI with poly (styrene-*b*-ethylene-ethylene-propylene-*b*-styrene) (SEEPS) to develop high performance MREs. The titanate coupling agent remarkably softens the CI/SEEPS, thus a significant enhancement of the MR effect was found. The mechanical properties of the MREs can be selectively controlled by varying the characteristics of the polymer matrix, since the interface between the iron particles and the polymer exhibited high influence on the deformation polymer chains. Therefore, introduction of additives to improve both the movability of the polymer chains and the compatibility between the particles and polymer matrix is critical for the development of high performance MREs.

In this work, a rosin glycerin ester/natural rubber mixed matrix was used to synthesize MREs with high MR effect and low loss factor. Due to the presence of the rosin glycerin ester with good adherence property, the viscosity of the rubber compound decreases, and thus the iron particles can move easily in the carrier matrix. In addition, the wetting ability of the unvulcanized rubber compound was improved and the MR effect of the MREs was increased. In this work, the influence of magnetic field strength, shear strain amplitude, frequency and CI content on the dynamic properties of the rosin glycerin ester/natural rubber based MREs are investigated. The wetting ability of the rubber compounds and the microstructures of the MREs with different content of rosin glycerin ester are observed and the possible mechanism for the high MR performance is discussed. To our knowledge, this is the first report on changing the wetting ability of rubber compound, and thus changing the dynamic properties, in particular the MR effect and loss factor of MREs.

2. Experimental details

2.1. MRE fabrication

Magnetic particles and elastic matrix are the main constituents of MREs. CI particles with average diameter of 7 μm were purchased from BASF in Germany (type CM). Natural rubber (NR) was used as the matrix, manufactured by the Hainan Rubber Group in China (type SCR WF). Rosin glycerin ester was selected as another part of the matrix, provided by

Table 1. Composition of sample matrix prepared (CI particles were fixed at 60% weight fraction).

Sample	Rosin glycerin ester	Natural rubber	Plasticizers and other additives
1	0	45	55
2	4.5	40.5	55
3	9	36	55
5	18	27	55

West Tech Chemical Co. Ltd in China. Plasticizer and other additives were provided by Hefei Wanyou Rubber Company of China. The fabrication of MREs consists of three major steps: mixing, pre-forming configuration and curing [20]. In this experiment, 100 phr rubber and rosin glycerin ester were mixed homogeneously using a two-roll mill (Taihu Rubber Machinery Inc. China, Model XK-160). Four samples with different content of rosin glycerin ester of 0, 10, 20, 40 phr were fabricated. The details of composition are shown in table 1. Then CI particles with a weight fraction of 60%, 100 phr plasticizer and 15 phr other additives were put into the mixture to form a rubber compound. The rubber compound was put into a mold under a pressure of 20 MPa in the presence of an external magnetic field (1300 mT) and the temperature was fixed at 80 °C for 15 min. After shut-down of the magnetic field, the temperature was further increased to 153 °C and maintained for 20 min. Finally, the sample was prepared. For the samples with different mass content of CI particles (50, 60, 70, 80 wt%), the ratio of rosin glycerin ester was kept at 9 wt%.

2.2. The viscosity of the non-vulcanized matrix

The viscosity of non-vulcanized matrix with different mass ratio of rosin glycerin ester was measured by rheometer (Physica MCR 301, Anton Paar). The non-vulcanized natural rubber was prepared as a disk with a diameter of 20 mm and thickness of 1 mm. The samples were tested at 90 °C with the shear rate from 10^{-1} to 10 s^{-1} , and the gap from the rotating disk was fixed at 1 mm.

2.3. Measurement of dynamic properties

The dynamic properties of the samples were measured by rheometer. A sample (disk of diameter 20 mm and thickness 1 mm) was put between the rotating disk and the base. A magnetic field strength controlled by the current applied to the electromagnetic coil was generated [21]. The magnetic field strength was paralleled with the thickness of the sample. A pre-pressure was exerted on the sample to prevent the sample from sliding on the rotating disk. The test was in an oscillating mode and the temperature was fixed at 25 °C, controlled with circulated water.

The samples were tested under different flux densities from 0 to 1000 mT—both strain amplitude sweep and frequency sweep modes were conducted. For the strain amplitude sweep test, the frequency was 5 Hz and the strain amplitude swept from 0.1% to 0.9%. For the frequency sweep

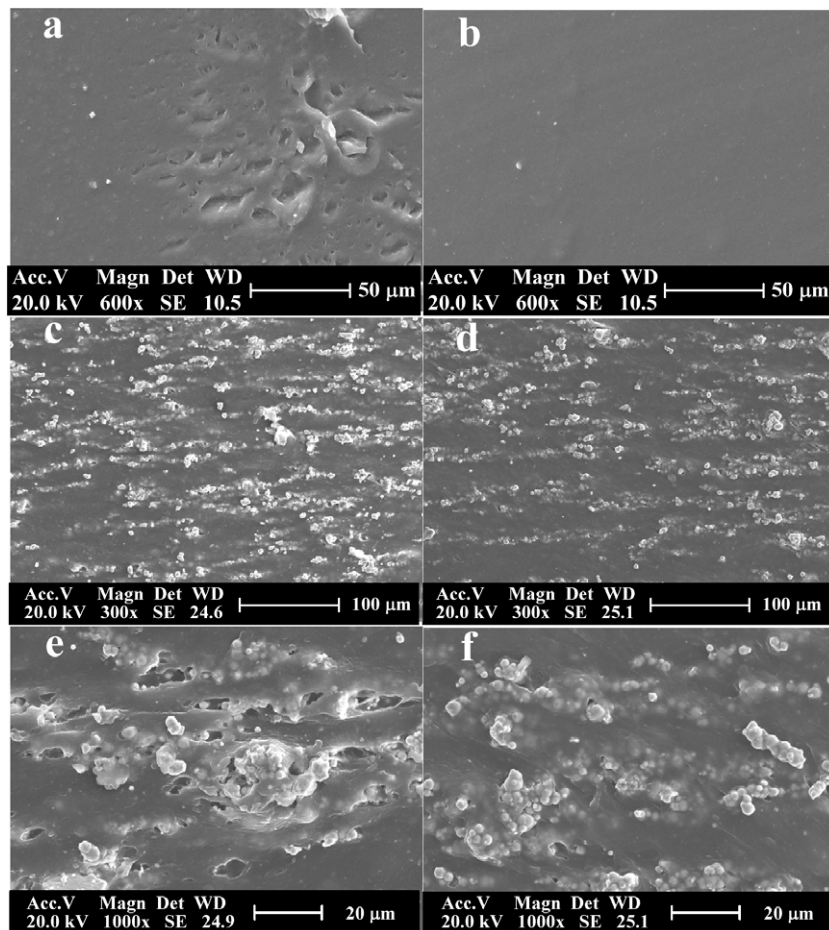


Figure 1. SEM images of the samples (a) contrast without rosin glycerin; (b) contrast with rosin glycerin ester; (c), (e) MRE without rosin glycerin ester; (d), (f) MRE with rosin glycerin ester.

test: the shear strain amplitude was 0.3% and the frequency was swept from 5 to 20 Hz.

2.4. Observation of microstructure

The microstructures of the samples were observed using an environmental scanning electron microscope (SEM, Philips of Holland, model XT30 ESEM-MP). The accelerating voltage was 15 kV. Before putting into the SEM, the samples were cut into pieces and coated with a thin layer of gold on the surface. The microstructure details of how rosin glycerin ester spread in NR, the dispersibility of CI particles, the structure formed by the CI particles in the matrix and the interface between rubber and CI particles were obtained.

2.5. Contact angle measurement

The contact angle was measured using a drop shape analysis system (DSA 30 Krüss of Germany). The CI particles were put into a mold and a force of 30 MPa applied in a tableting machine. Contrasts of rubber matrix without CI particles were fabricated in the same way as the MRE samples. Both the CI tablets and the contrasts were put on the test board of the DSA to measure the contact angle of pure water on the sample surface.

3. Results and discussion

3.1. Preparation and characterization of the MREs based on rosin glycerin ester/natural rubber mixed matrix

After mixing the precursor, magnetic pre-structure, and the final vulcanization, the rosin glycerin ester/natural rubber mixed matrix based MREs were obtained. The microstructure of the novel MREs was first investigated. Figure 1 shows the SEM image of the neat natural rubber and the rosin glycerin ester doped natural rubber. In comparison to the hybrid matrix, the cutting face of the neat natural rubber is rougher and some drapes are presented, which indicates the rosin glycerin ester not only can be well mixed with the natural rubber but can also improve the flexibility of the hybrid matrix. After introduction of the rosin glycerin ester, the compatibility between the iron particles and polymer matrix is highly improved, and the CI particles are homogeneously dispersed in the rubber matrix, while some big CI aggregations are found in the neat natural rubber based MRE. Due to the pre-structure process, the CI particles assemble to form a chain-like structure. From the high magnification SEM image of the MRE base on the hybrid matrix (figure 1(f)), it can be seen that the CI particles bond well with the matrix and the chains are straight and long. However, these CI particles prefer to form large aggregations

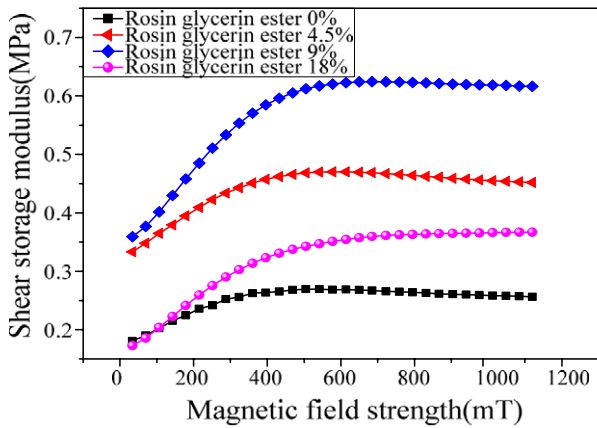


Figure 2. Shear storage of samples with different content of rosin glycerin ester under different magnetic field strength.

Table 2. The zero-field storage modulus (G_0), the magnetic induced modulus (ΔG) and the MR effect of the MREs with different CI particle content.

CI particle content (%)	G_0 (MPa)	ΔG (MPa)	MR effect (%)
50	0.12	0.04	43
60	0.33	0.12	51
70	0.59	0.31	55
80	0.81	0.74	87

in the absence of the rosin glycerin ester and some cracks are clearly observed between the CI particle chains and the neat natural rubber. As a result, the rosin glycerin ester can improve the compatibility between the CI particles and the matrix, which must be propitious to improve the MR effects of the MREs.

Usually, the MR effects of the MREs are evaluated by investigating their shear storage modulus (G). Define G_0 as zero-field modulus, G_{\max} as magnetic saturation modulus. Thus the magnetic induced modulus $\Delta G = G_{\max} - G_0$ and the relative MR effect is defined as $\Delta G/G_0$. In this work, keeping the CI weight percentage of 60% constant, all the samples' shear modulus show an increasing trend with magnetic field strength (figure 2), proving that all the MRE samples exhibit obvious MR effects. The influence of the rosin glycerin ester on the G is further studied (table 2). Without applying the magnetic field, G_0 of MRE samples with 9% rosin glycerin ester shows a higher value than the neat natural rubber based MREs, while with a larger weight fraction of rosin glycerin ester, the zero-field modulus decreased. Because rosin glycerin can decrease the viscosity and improved the flexibility of the precursor, the CI particles move more easily in the matrix when an external magnetic field is exerted. As shown in the SEM image (figure 1(f)), the chains formed by CI particles were perfect with the introduction of rosin glycerin ester. Similarly, the ΔG was larger than the samples without rosin glycerin ester due to the better chain structure (figure 2).

Keeping the weight ratio of the rosin glycerin ester constant (9 wt%), the influence of the CI content on the MR effects was further investigated (figure 3). Due to the strengthening effect, increasing the CI particles from 50

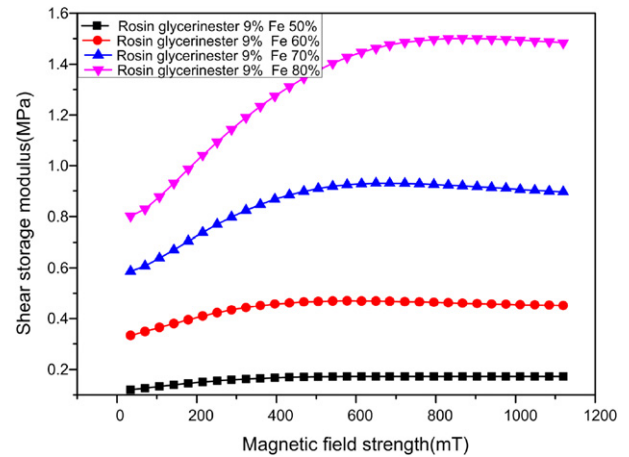


Figure 3. Shear storage modulus of MREs with different content of CI particles.

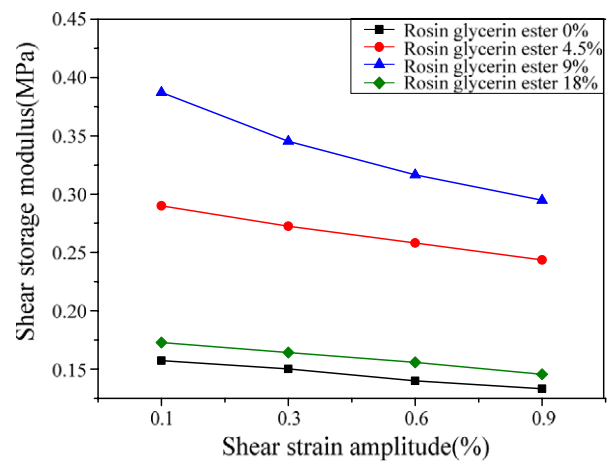


Figure 4. Shear storage modulus of four samples under different shear strain amplitude.

to 60, 70, and 80 wt%, the G_0 of the MREs increased from 0.12 to 0.33, 0.59, and 0.81 MPa, respectively. After applying the magnetic field, the inter-force between the CI particles significantly increased due to the interaction with the magnetic induced dipole. The increase of the CI content also leads to a reduction of the distance between the magnetic particles, which further increases the magnetic forces. Thus, the ΔG critically increased from 0.04 to 0.12, 0.31 and 0.74 MPa with the CI content increased from 50 to 60, 70 and 80 wt%. The value of the ΔG /CI weight ratio is not a constant, indicating that the magnetic induced forces between the CI particles are non-linearly dependent on the magnetic content.

The influence of shear strain amplitude and frequency on shear storage of the MREs is shown in figures 4 and 5. The storage modulus decreases with the strain amplitude, since with increasing shear strain the molecules of the rubber matrix began to slide. Due to the increase of the distance between the ends of two molecule chains, the chains more easily separated when the shear stress was applied. Therefore, the shear storage modulus decreases with the shear strain amplitude. When

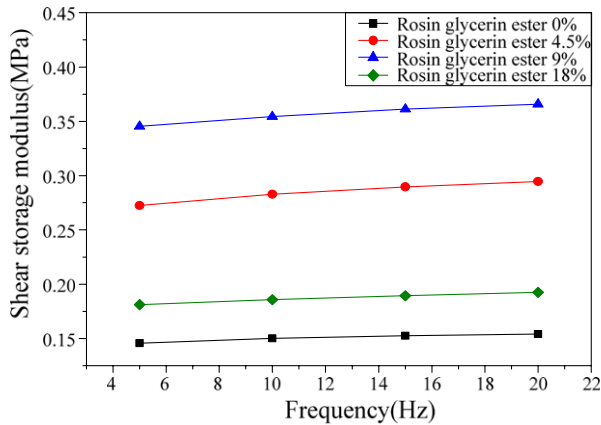


Figure 5. Shear storage modulus of four samples under different frequency.

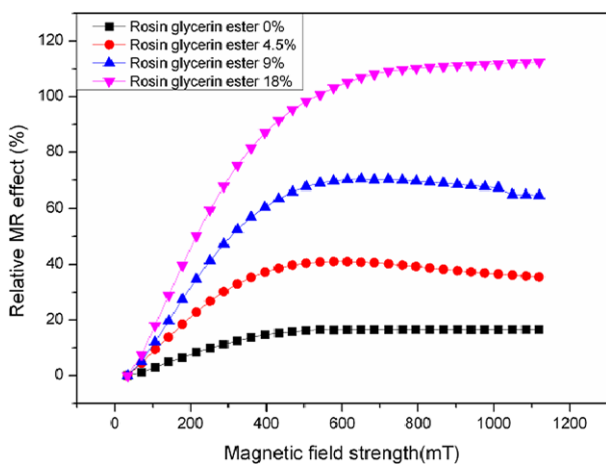


Figure 6. Relative MR effects of four samples under different magnetic field strength.

rosin glycerin ester reached 9 wt%, ΔG had the maximum value. In this mass ratio, the two phases were most tightly bound—more energy was needed to damage the cohesive boundary. In addition, the shear storage modulus gradually increased with the frequency (figure 5)—as the frequency rose, the crystallization in the natural rubber matrix increased while adding deformation. The crystallization can enhance the matrix, and thus the shear storage modulus increases. At a constant frequency, the performance of shear modulus identifies with the degree of cohesiveness of the two phases.

The relative MR effects of the four samples are shown in figure 6. When CI particles were fixed at 60 wt%, with the increase of rosin glycerin ester, the relative MR effect gradually increased significantly from 16.59% to 35.53%, 64.63% and 112.38%, almost 7 times the original value. The magnetic induced modulus increased at first and then came to a maximum value. When the weight ratio of the rosin glycerin ester was larger than 9 wt%, the ΔG was almost unchanged by the increase of rosin glycerin ester. However, due to the decrease of the G_0 , the relative MR effect also increased.

Figure 7 shows the loss factor of the samples under different magnetic fields. The damping property

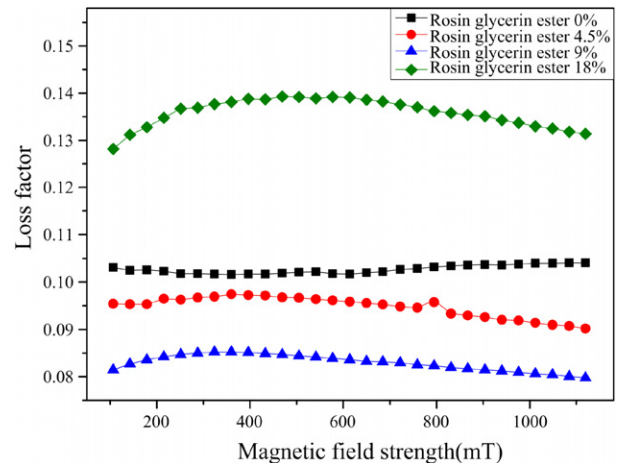


Figure 7. Loss factor of the four samples under different magnetic field strength.

influences the energy dissipation of the materials [22]. It was reported that the damping of MRE mainly consists of intrinsic damping, interface damping, and magneto-mechanical hysteresis damping [23]. For the MRE, the energy dissipation mainly comes from interfacial damping. Without adding rosin glycerin ester, the change of damping with external magnetic field produces almost a straight line (figure 7). Taking the sample with 18% rosin glycerin ester as an example, the loss factor shows a tendency to increase then decrease with increasing of the magnetic fields. For higher rosin glycerin ester content, the tendency seems to be clearer, which agrees well with the previous research [24]. The rosin glycerin ester helps the CI particles to move more easily in the matrix in the presence of an external magnetic field thus leading to a larger increase of loss factor on application of a magnetic field. The magnetic dependency of the loss factor first decreases and then increases with increasing of the rosin glycerin ester. The wetting ability of the non-vulcanized natural rubber was highly dependent on the rosin glycerin ester. Sample 3 has the maximum adhesion and the adhesion between rubber matrix and CI particles was the tightest, thus the relative slip between rubber matrix and CI particle was smaller.

The tensile strength of sample is shown in figure 8, indicating that the rosin glycerin ester led to a decrease of tensile strength. In the presence of the rosin glycerin ester, the vulcanization and crosslinking of natural rubber was inhibited with a large mass ratio, thus the additive spread homogeneously in the natural rubber. Because the cross-link network density of the system had decreased, the distance between the ends of two rubber molecules had increased, so the tensile strength decreased. Moreover, the crystallization of natural rubber molecules is very slow in room temperature, while they can crystallize immediately in tensile state. The crystalline molecules reinforced the composite matrix, thus the tensile strength increased. However, the rosin glycerin ester restrained the strain induced crystallization, thus tensile strength decreased due to the decrease of crystallization degree.

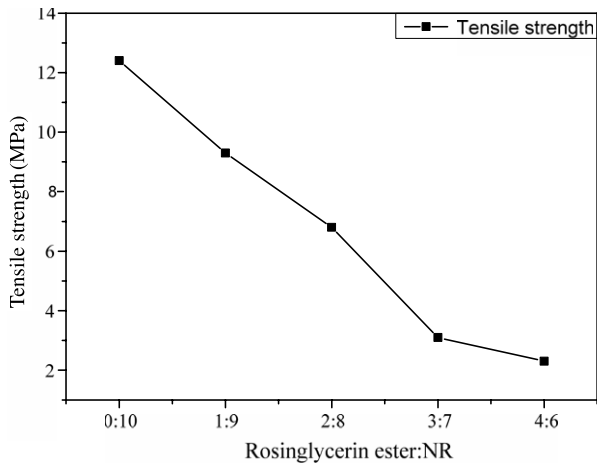


Figure 8. Tensile strength of MREs with different content of rosin glycerin ester.

3.2. The mechanism of the enhanced MR effects for the MRE based on the rosin glycerin ester/natural rubber hybrid matrix

The mechanism of interaction between rubber matrix and CI particles is shown in figure 9. The dispersion and adhesion between CI particles and rubber matrix were characterized by the contact angle of the samples [25–29]. Free energy of immersion of the CI particles in the rubber matrix was used to quantify the influence. In this section ΔG is defined as the free energy of immersion of the interfacial energy between the CI particles and rubber matrix. γ_s , γ_l are the surface energy of the CI particles and the rubber matrix:

$$\Delta G = \gamma_{sl} - \gamma_s. \tag{1}$$

If ΔG has a positive value, the wetting of CI particles by the rubber matrix is thermodynamically unfavored. If ΔG has a negative value, the wetting will be preferred. Direct measurement of the interfacial energy γ_{sl} is impossible. Young’s equation is introduced as

$$\gamma_s - \gamma_l = \gamma_{sl} \cos \theta. \tag{2}$$

Combination of equations (1) and (2) yields

$$\Delta G = -\gamma_l \cos \theta. \tag{3}$$

Strongly connected with the free energy of immersion is the influence of adhesion between CI particles and rubber matrix W_a , which also determines the wetting process of the filler surface and the polymer [30]. The work of adhesion was first introduced by Hankins [31]. Between a solid and a liquid phase W_a is defined as

$$W_a = \gamma_s + \gamma_l - \gamma_{sl}. \tag{4}$$

By combination of equation (2) with Young’s equation, get the well-known Young–Dupré equation for the work of adhesion:

$$\Delta W_a = \gamma_l(1 + \cos \theta). \tag{5}$$

As γ_l is a constant, the work ΔW_a increases at first and then decreases. The correlation between wetting and adhesion is

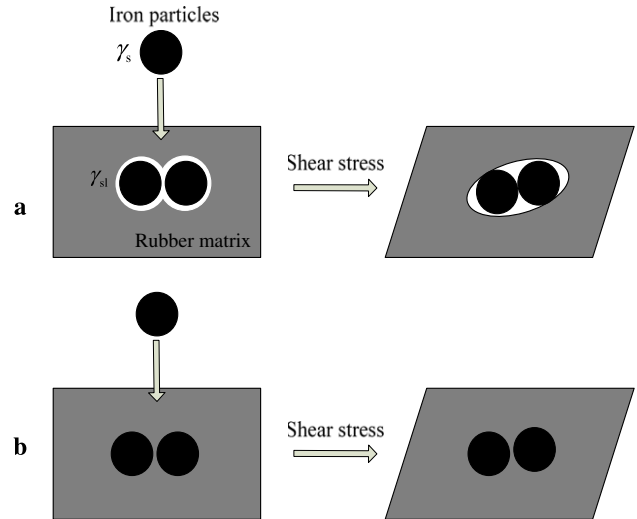


Figure 9. The mechanism of bonding degree and interaction between rubber matrix and CI particles (white space is the gaps between rubber matrix and CI particles). (a) Without rosin glycerin ester, loose bonding; (b) with rosin glycerin ester, close bonding.

Table 3. The contact angle of each sample.

Samples	Contact angle
NR: rosin glycerin ester = 10:0	120.8
NR: rosin glycerin ester = 9:1	112.2
NR: rosin glycerin ester = 8:2	100.2
NR: rosin glycerin ester = 6:4	111.6
Carbonyl iron tablet	143.7

that for complete wetting ($\theta = 0^\circ$), $\Delta W_a = 2\gamma_l$ the rubber and CI particles were cohesive as one single phase. On the other hand, for non-wetting ($\theta = 180^\circ$), $\Delta W_a = 0$, the work of adhesion equals zero, thus the rubber matrix and CI particles are separated into two completely non-connected phases. The experiment results in table 3 show that the adhesion between rubber matrix and CI particles is the strongest when rosin glycerin ester is 9 wt%.

Under the shear strain amplitude, the elastomer began to generate a deformation. With an external magnetic field applied, the CI particles tended to keep a linear structure in the vertical direction under the magnetic field strength while the rubber matrix moved in the direction of the shear force, and the rubber matrix and CI particles began to slip. If the bond between rubber matrix and CI particles was loose, the work of adhesion was also weak (figure 9(a)), thus gaps were present at the interface due to the debonding (figure 1(e)). The gap between CI particles and rubber matrix was huge when shear stress was applied, the sliding friction tends to be bigger, and the interaction between the two particles can also increase the friction, so the energy dissipation was more, and the loss factor larger. Figure 9(b) shows a strong work of adhesion, with the bond between rubber matrix and CI particle tight, as seen in the SEM image figure 1(f). The gap between CI particles was small, so the sliding friction tended to be smaller, the CI particles were separated by the rubber matrix, thus decreasing the interaction between the particles, and the energy dissipation was less, and the loss factor tended

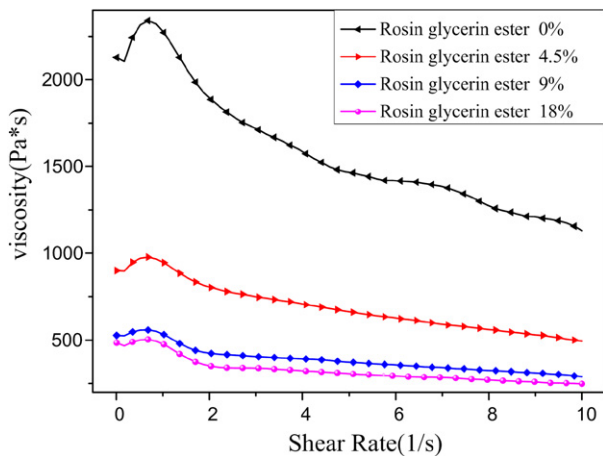


Figure 10. Viscosity of natural rubber with different rosin glycerin ester content.

to be smaller. Also, with the addition of rosin glycerin ester, the rubber matrix tended to be softer, the intrinsic damping caused by the rubber matrix became larger, so the loss factor of the sample came to a minimum value, then increased again.

The flexibility of the matrix was proved by the viscosity of the matrix. The dispersion and movement of the iron particles in the matrix is the first and most important step in the fabrication of MR elastomers. The viscosity of the matrix has a great effect on the dispersion of the filler particles [32]. Figure 10 shows the viscosity of non-vulcanized rubber matrix with different weight ratios of rosin glycerin ester under different shear rates. For neat NR, the plasticizer is hard to dissolve into the rubber while mixing. With the addition of rosin glycerin ester, the plasticizer was well dispersed in the hybrid matrix. The figure shows that the viscosity of the matrix is decreased with the increase of rosin glycerin ester, so that CI particles can move more easily in the matrix. This analysis agrees well with the SEM results, that the CI particles in samples with rosin glycerin ester form a thicker and longer linear structure.

In summary, the rosin glycerin ester can change the viscosity of the non-vulcanized rubber matrix, and thus change the wetting ability of the matrix on CI particles which affects the properties of the MREs. With an external magnetic field exerted, the CI particles tend to form a longer and stronger linear structure. Addition of rosin glycerin ester leads to a higher MR effect and a smaller loss factor in an appropriate weight fraction. The shear storage modulus of the MREs tends to be smaller for larger shear strain amplitude. With a higher frequency, the shear storage modulus tends to be larger. A high content of rosin glycerin ester leads the tensile stress to be smaller.

4. Conclusion

In this study, novel rosin glycerin ester/natural rubber mixed matrix based MREs with high MR effects were fabricated. Addition of rosin glycerin ester decreased the viscosity of rubber matrix, and the CI particles moved more easily in the matrix under the magnetic field strength to form

a better linear structure. MREs with additive have lower zero-field modulus—the MR effect was 7 times greater than the non-additive value. Also, additive can change the wetting ability of the matrix. With an appropriate content, the work of adhesion increases to a maximum value, the interfacial adhesion can affect the interaction of the two phases, and thus have a great effect on damping properties. MRE with higher MR effect and lower loss factor has been achieved, which can be applied to many fields such as adaptive tuned absorbers based on MRE. A wider load of force can be exerted on the absorber due to the high MR effect—the lower the loss factor, the higher the absorber effectiveness.

Acknowledgments

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