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Maleic Anhydride Modified Dicyclopentadiene Resin for Improving Wet Skid Resistance of Silica Filled SSBR/BR Composites

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Abstract: As commercial rubber in tires, silica-filled solution-polymerized styrene-butadiene rubber/butadiene rubber (SSBR/BR) compounds exhibited preferable wet skid resistance (WSR) properties, which could be further enhanced by the incorporation of some oligomeric resins. However, the untreated dicyclopentadiene (DCPD) resin shows a slight improvement in wet friction even if the good compatibility between DCPD and SBR owing to their common cyclic structures. For this problem to be addressed, we aimed to enhance its resin-silica interaction by reaction with maleic anhydride (MAH). In detail, the effect of MAH content on WSR, curing characteristics, physical-mechanical properties of the silica-filled SSBR/BR composites was investigated. When the MAH content is 4 wt% in the modified DCPD resin, the maximum enhancement of about 15% in $\tan \delta$ values at 0 °C, as well as that of 17% in British pendulum skidding tester (BPST) index is obtained, indicating a desirable improvement in WSR. In addition of these two commonly used methods, water contact angles of the vulcanizates increase gradually with increasing MAH content, further confirming the remarkable performance of modified DCPD resin in WSR.

Keywords: SSBR/BR compounds; wet skid resistance; dicyclopentadiene resin; maleic anhydride

1. Introduction

With the implementation of European Tire Labelling Regulation [1], high-performance tire tread is in high demand, i.e., both high wet skid resistance (WSR) and low rolling resistance, which correspond to the concept of “Green tires” as well. In recent years, the influences of various factors, such as filler types [2–4], rubber matrix [5,6], water film depth [7], tread and pavement patterns [8,9], etc., on tire performances during processing and operation have been studied, triggering extensive methods to enhance WSR. For one thing, the type and curing process of rubber blend influence performance, stimulating research directly on stimulation study of how rubber compound affects properties [10]. For another thing, frictions are consisted of energy dissipation in various forms. Likewise, deformation hysteresis and adhesion are recognized as two critical internal dissipation forms for wet friction. Moreover, according to Wang et al. [11,12], the wet friction coefficient between tire and slippery road is also closely associated with lubrication effect, i.e., elasto-hydrogen lubrication (EHL), boundary lubrication (BL) and microelasto-hydrogen lubrication (MEHL). In fact, the friction coefficient between elastomer and rigid surface, which depends mostly on whether lubricant is able to flow, increases evidently only if lubricant is thin enough to adhere tightly on the elastomer. In addition, as Giles [13] mentioned in “three area” conception, the key to enhance wet friction is to enlarge traction area in

contact area which divides into penetration area, transition area and traction area between tire and road, resulting from that water film thins under pressure during which elasto-hydrogen lubrication gradually transform to boundary lubrication. As a consequence, propelling the transition from EHL to BL by breaking the thick water film is exactly vital to improve wet friction. Many researchers acknowledge that the reason why silica-filled tire performs better than that with carbon black on wet road is that the silica-water interaction is much stronger than the silica-polymer interaction, making it capable of exposing silica to improve surface roughness and fulfil demand for boundary lubrication [3,11,12,14]. However, silica is easy to aggregate due to its polar surface, which even deteriorates the reinforcement of silica on account of causing the discrepancy of surface energy between silica and rubber chain. As a result, it is desirable to weaken the silica-filler network by adding some additives in order to improve the WSR of silica-filled SSBR/BR compounds.

There are two basic requirements for the agents: (1) sufficient compatibility with SSBR; (2) the ability to interact with silica. Many resins contain benzene or other ring structures, such as 2-phenyl-1-propene, phenolic resin, have been thus investigated [15]. However, most petroleum resins are restricted to being antiskid resins due to the primarily rare possibility to modify their nonpolar nature. Differently, dicyclopentadiene (DCPD) resin that is obtained by thermal polymerization and Diels-Alder reaction as shown in Figure 1, can exhibit strong polarity through copolymerizing with some polar monomers [16]. Furthermore, our previous work shows that modified rosin resins greatly improve the WSR of vulcanizates, mainly because of the introduced polar groups [17]. Therefore, we assumed a similarly expectable consequence by modifying DCPD resin and carried out relevant investigation.

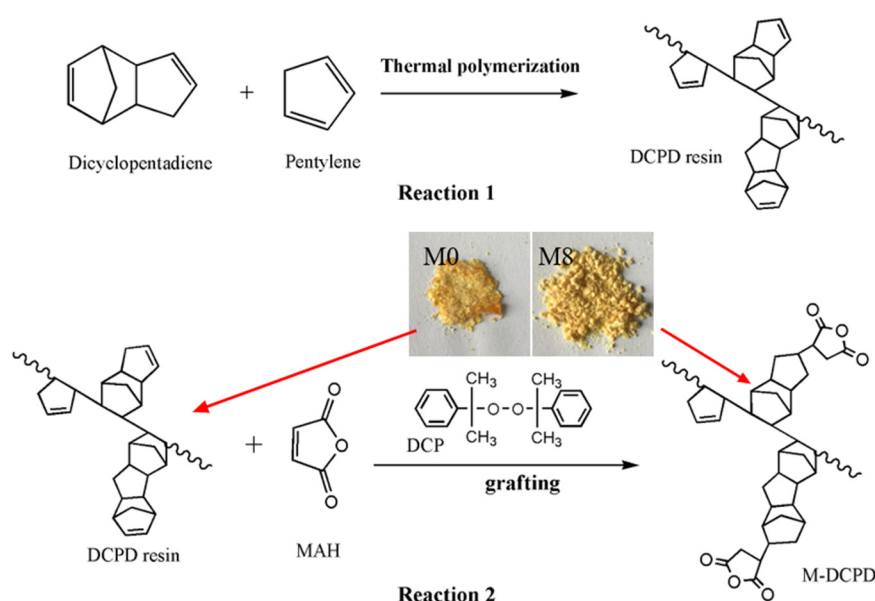


Figure 1. Reaction 1 refers to the diagram described preparation of dicyclopentadiene (DCPD) resin; reaction 2 refers to the preparation of modified DCPD resin.

In this study, the modified DCPD resin was prepared by grafting the reaction of DCPD resin with maleic anhydride. Fourier transform infrared spectrometer (FTIR) and differential scanning calorimetry (DSC) were used to confirm the reaction. Since the effect of MAH content on WSR is associated with both hysteresis and lubrication effect, we not only measured the dynamic properties at low temperature that are acknowledged by most researchers [18–21], but also innovatively introduced the water contact angle to analyze the interaction between rubber and water, in order to clearly understand how MAH content affects wet friction from the perspective of fluid lubrication. Furthermore, another portable British Pendulum Skid Tester (BPST) was also adopted to evaluate the maximum friction on different substrates, which stimulate real road conditions. Moreover, the effects of M-DCPD on the physical-mechanical properties and cure characteristics were also examined.

2. Experimental Section

2.1. Materials

Solution-polymerized styrene-butadiene rubber SSBR2535E (styrene content: 23%, vinyl content: 53%) with 37.5 wt% oil-extended was purchased from Butadiene rubber BR9000 (Cis 1,4-polybutadiene content: 96–99%), were purchased from Sinopec Yanshan Petrochemical Co. (Beijing, China). Highly dispersed silica 7000GR (surface area: 170 m²/g, grain size: 14 nm) was bought from Evonik Degussa Co. (Shanghai, China). Zinc oxide (ZnO), 1,3-diphenylguanidine (D), bis[3-(triethoxysilyl)propyl]tetrasulfide (TESPT), N-1,3-dimethylbutyl-N'-phenyl-p-phenylenediamine (6PPD), rubber waxes RW287, poly(1,2-dihydro-2,2,4-trimethyl quinoline) (RD), sulfur, N-tert-butylbenzothiazole-2-sulphenamide (NS), stearic acid, DCPD resin (molecular weight: 3250 g.mol⁻¹, softening point: 108.9 °C) and dicumyl peroxide (DCP) were obtained from Lianyungang Rebo Chemical Co. (Lianyungang, China). Maleic anhydride (MAH) was bought from Guangzhou Chemical Reagent Factory.

2.2. Preparation of M-DCPD Resin

First, DCPD was completely melted in a 500 mL wild-mouth flask equipped with a mechanical stirrer and a thermometer at 200 °C. After MAH and initiator DCP were added simultaneously, the mixture was stirred at 200 °C for 2 h. The reaction mechanism was shown in Figure 1. After cooling down, the mixture was kept in xylene for 2 h to remove the unreacted MAH. The purified product was filtered and dried under vacuum. Table 1 lists the specific content of ingredients in each group and the MAH weight ratio measured by titration demonstrated in 2.4.1 The number in the names referred to MAH content. Moreover, M2, M4, M6 and M8 were collectively referred to the modified DCPD (M-DCPD) resin.

Table 1. The dosages of synthetic ingredients.

Sample	M0	M2	M4	M6	M8
DCPD(g)	100	100	100	100	100
DCP(g)	2	2	2	2	2
MAH(g)	0	2	4	6	8
AR *(%)	0	2.4	4.8	6.8	8.8

*: AR refers to the acid ratio of modified DCPD that described in Section 2.4.1.

2.3. Preparation of Vulcanizates

SSBR and BR were mixed uniformly in a two-roll mill at room temperature and blended for about 30 s at 120 °C as set temperature in a DEG C HAAKE mixer under 60 rpm. The mixture of silica and TESPT was added into the HAAKE mixer in three divided times and compounded for 90 s. After other additives were added and mixed for another 180 s, rubber compounds were discharged at 120 °C Sulfur and accelerator were mixed with blends on a two-roll mill at room temperature. Composites were vulcanized at 160 °C for optimum cure time (T90). The basic formulation was shown in Table 2. Vulcanizate containing M0 were set as the control group, while others with M-DCPD resin were testing groups.

Table 2. The compounds formula.

	Control Group (phr)	Test Group (phr)
SSBR (oil filled 37.5)	96.25	96.25
BR	30	30
TESPT	5.6	5.6
Silica	70	70
Stearic acid	2	2
ZnO	5	5

Table 2. Cont.

	Control Group (phr)	Test Group (phr)
6PPD	1.5	1.5
RW287	1.5	1.5
RD	1.5	1.5
NS	1.2	1.2
Sulfur	1.8	1.8
D	1.8	1.8
M0	10	0
M-DCPD resin	0	10

2.4. Characterization

2.4.1. Characterization of the M-DCPD Resin

Infrared transmission spectra were taken at room temperature on VERTEX70 FTIR (BRUKEROPTICS. Co. Ltd., Ettlingen, Germany) within 32 scans and resolution of 4 cm^{-1} over a range from 400 cm^{-1} to 4000 cm^{-1} . The glass transition of M-DCPD resin was observed through differential calorimetry scanning (NETZSCH DSC 204F1, Selb, Germany) under nitrogen atmosphere with a heating rate of $20\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$. The molecular mass of M-DCPD resin was measured through gel chromatography by using tetrahydrofuran as eluant at $35\text{ }^{\circ}\text{C}$ with the flow rate of $1\text{ mL}\cdot\text{min}^{-1}$.

The weight ratio of MAH in M-DCPD resin was studied by chemical titration with the following steps: 20 g samples were dissolved in 50 mL chloroform at room temperature. As soon as six beads of phenolphthalein indicator were added, potassium hydroxide/ethanol standard solution was dropwise added until it turned to tiny red and remained fadeless for 30 s. The weight ratio of anhydride (AR) in DCPD resin were calculated by the following equation,

$$\text{AR} = VMc/2m \times 100\% \quad (1)$$

where V and c are the volume and concentration of the standard potassium hydroxide/ethanol solution, M is the molecular weight of MAH, and m is the weight of M-DCPD resin.

2.4.2. Characterization of the Rubber Vulcanizates

The temperature sweep tests were conducted on a dynamic mechanical analyzer (DMA) (NETZSCH 242C, Selb, Germany) under tensile mode at 10 Hz with a heating rate of $3\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$ from $-80\text{ }^{\circ}\text{C}$ to $+100\text{ }^{\circ}\text{C}$. Mechanical properties were recorded using a tensile testing machine (UT-2080, U-CAN Technologies, Taiwan, China) following ISO 37:2005 under the speed of $500\text{ mm}\cdot\text{min}^{-1}$. Equilibrium swelling method was used to determine the cross-linking density. The BPST index was obtained from a Pendulum type friction coefficient measuring instrument according to other researchers [22]. The scanning electron microscope (SEM) images of fractured surfaces were taken by a scanning electron microscopy (ZEISS EVO 18 Special Edition, Jena, Germany) after etching the resin phases with methylbenzene. The static water contact angle was tested by a contact angle meter (DropMeter A-100).

3. Results and Discussion

3.1. Characterization of Copolymers

M-DCPD resin were obtained through reaction of MAH and DCPD resin, which were named as M0, M2, M4, M6, and M8 on the basis of MAH addition. The FTIR spectra of MAH and M-DCPD resin were shown in Figure 2. The sharp peaks around 1780 cm^{-1} and 1860 cm^{-1} observed in spectrum of M-DCPD resin are assigned to the typical stretching vibration of anhydride groups. Considering that the unreacted MAH has been removed by purification after graft polymerization of MAH and DCPD

resin as is shown in Figure 2, it could conclude that anhydride groups have been successfully grafted to the polymer chain. Moreover, an increasing tendency is observed in spectrum with the increasing dosage of MAH, as well as the increasing weight ratio of anhydride from titration method shown in Table 1, indicating that anhydride content in M-DCPD resin increases within more MAH addition.

Furthermore, the discrepancy in anhydride content probably enhance the resin polarity in varying degrees. DSC was conducted accordingly to characterize the resins. Figure 3a shows that the more MAH was added, the higher the glass transition temperature that was observed from M0–M8. Polymers before and after modification show similar elution time in gel permeation chromatography (GPC) as is shown in Figure 3b. Therefore, general increasing glass transition temperature demonstrated increasing polarity of resins. Since M-DCPD resin can totally dissolve in tetrahydrofuran, it is reasonable that most MAH added only reacted with the side-chain groups without cross-linking. The slight increasing of molecular weight from M0 to M8 was due to the increasing usage of MAH.

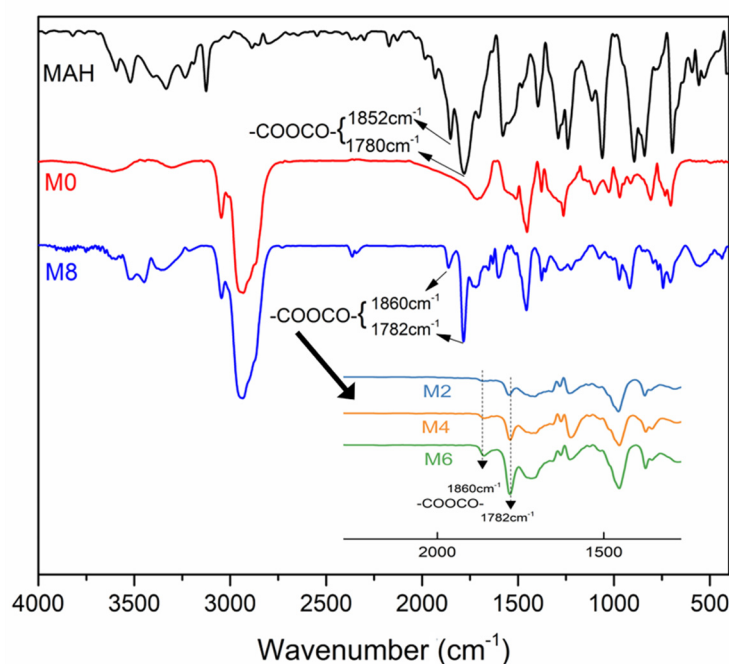


Figure 2. FTIR spectra of maleic anhydride (MAH), DCPD resin and M-DCPD resin.

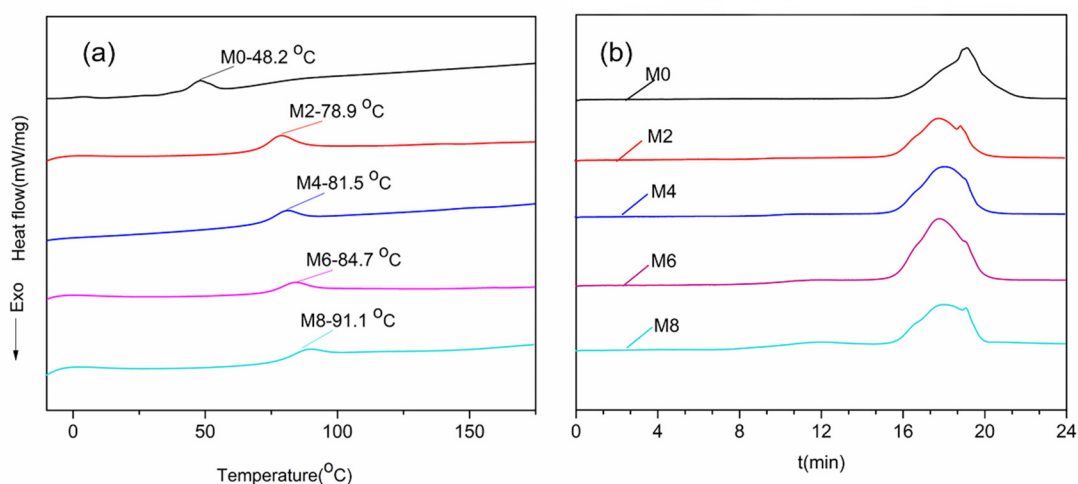


Figure 3. Curves obtained by DSC (a) and GPC (b) for M-DCPD resin.

3.2. WSR of the Vulcanizates

Vulcanizates with different content of M-DCPD resin were obtained after curing at 160 °C. They are named as R0, R2, R4, R6 and R8, representing composites with M0, M2, M4, M6 and M8 added, respectively. Using dynamic mechanical properties at low temperature to evaluate the WSR is accepted by most researchers in that deformation hysteresis is considered as one of major routes to dissipate energy under either the high frequency or low temperature [13,20]. The internal dissipation, i.e., $\tan\delta$ was then accordingly measured. As shown in Figure 4, the value of $\tan\delta$ at 0 °C increases with increasing MAH content and then drops. R4 presents the maximum value, indicating the best performance of R4 in WSR. However, adding M-DCPD resin slightly influences the rolling resistance of the vulcanizates according to the variation in $\tan\delta$ values at 60 °C.

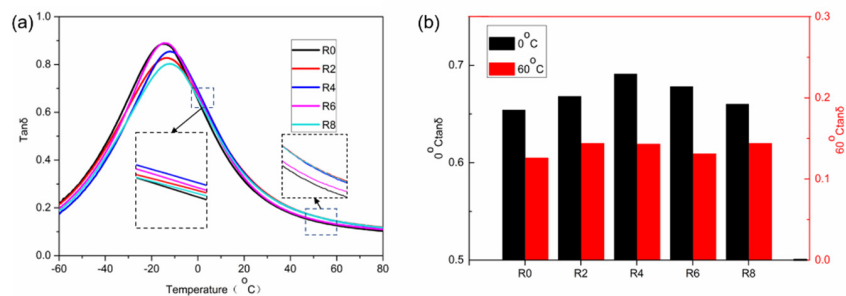


Figure 4. Stress strain curves (a), $\tan\delta$ values at 0 °C and 60 °C (b) of the vulcanizates.

The effect of resin on $\tan\delta$ is closely related to the compatibility between resin and rubber compounds, which results from the changes of glass transition temperature. The closer the peak value to 0 °C, the better improvement of the wet skid resistance of the vulcanizates [23]. Figure 4 shows that with increasing MAH content in the M-DCPD resins, the T_g of vulcanizates increases from −17.5 °C to −11.7 °C, values of $\tan\delta$ at 0 °C increases from 0.51 to 0.58 as well. The WSR performance of R8 was limited by inferior solvability of M8 in the rubber compounds due to its high glass transition temperature. For high hysteresis, two vital requirements are: (1) existence of filler aggregates; (2) destruction and reconstruction of filler agglomerates under dynamic stress [11]. Figure 5 visualized the supposed interaction mechanism of the modified resin with silica. The AR calculated above demonstrates that the polarity of resin increases with increasing amount of MAH, resulting in enhanced interaction between polar resin and silica, which can relatively weaken the filler-filler interaction. Furthermore, the interaction makes it more susceptible for filler aggregates to be damaged and reconstructed under periodic strain. For agglomerates containing trapped rubber, the destruction facilitates the release of restricted polymer chains, which exactly adds more energy consumption forms, leading to significant increase in loss modulus at low temperature (0 °C) and further in hysteresis. Although extended rubber volume fraction by released rubber chain enhanced viscosity, elastic modulus of vulcanizates increases as the polar interaction enhanced due to addition of M-DCPD resin, resulting in the decline of peak value of $\tan\delta$ of R8 at glass transition zone.

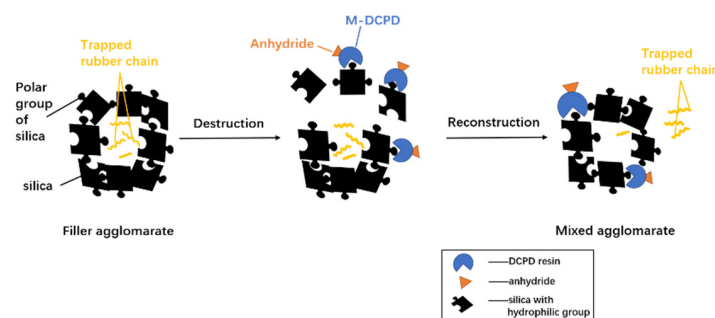


Figure 5. Schematic of the interaction between modified resin and silica.

However, the above two cases predict consecutively increased $\tan\delta$ values at low temperature as MAH content increasing, which does not completely correspond to the experimental results. Therefore, we suppose that the effect of M-DCPD resin on dynamic performance dose not only relate to the strength of the interaction with silica, but also relates to its dispersion in the rubber matrix. Resin would interact with only limited parts of filler aggregates if it cannot uniformly disperse in the rubber compound, leading to less effective improvement on hysteresis. That is likely to be the reason why the $\tan\delta$ value of R8 is significantly lower than those of R4 and R6. On the whole, M-DCPD resin remarkably enhances energy consumption by interacting with silica.

According to the mechanism of lubrication effect stated above, Figure 6 vividly described the trick to breakdown thick water film by enlarging surface roughness provided by silica asperity, which is formed as long as silica migrates to the surface of the vulcanizate through polar interaction between silica and water [12]. As a result, the ease with forming asperity could be deduced from the interfacial polar interaction of water with silica that is close to the interface.

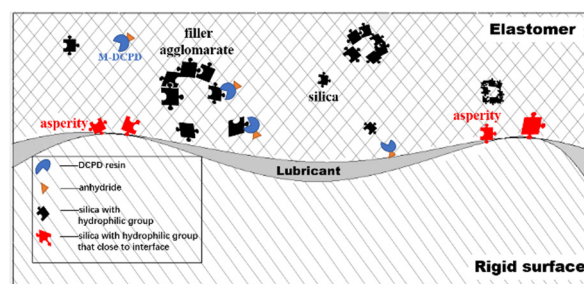


Figure 6. Interaction mechanism of vulcanizates and water effected by M-DCPD resin with anhydride and silica with hydroxy.

The surface polarity of vulcanizates could be reflected by water contact angle, as that shown in Figure 7, which had a negative relationship with the content of MAH (Figure 8), making it clear that content of hydrophilic groups on the surface of the vulcanizates increased with increasing MAH content. Obviously, the differences in hydrophilicity among different samples are caused by the quantity of silica on which a large number of hydroxyl groups exist, as well as that of M-DCPD resin that grafted with anhydride. Moreover, the quantity is closely associated with the dispersion of silica. The polar interaction between M-DCPD resin and silica contributes to reduction of surface energy difference between rubber and filler, leading to both uniform dispersion of silica and high surface polarity of the vulcanizates. Therefore, we have ground to speculate that the enhanced polar interaction of water with vulcanizates facilitates the break of water film.

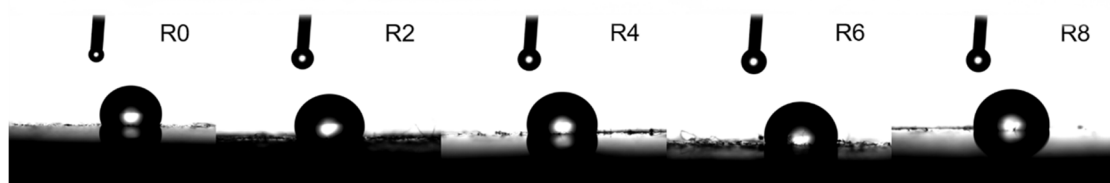


Figure 7. Water contact angle images of vulcanizates.

In order to verify our theoretical inference about the effect of anhydride on filler dispersion, we therefore studied the BPST index. The BPST value directly reflects the degree of sliding friction between sample and testing surface by the following equation,

$$BPST\ index = 330\mu / (3 + \mu) \quad (2)$$

where μ refers to the friction coefficient between the vulcanizate and the test surface [12]. As it is shown in Figure 8, the BPST value raises by 16% as the MAH content increases to 4 wt% in M-DCPD resin, while it changes slightly with increasing loading. The dynamic properties from Figure 4 show that R4 and R6 can effectively promote energy dissipation because of high hysteresis. At the same time, it is inferred from water contact angle (Figure 8) that the increasing amount of silica near the internal surface is more likely to expose as surface protrusions in these two samples. The friction coefficient is accordingly higher with a rougher surface. Figure 8 also shows that though R8 exhibits better interaction with water than R4 and R6, the three samples show comparable ability to overcome friction. The BPST test, as well as water contact angle, prove that whether resins could evenly disperse in the compounds is vital to the performances of vulcanizates.

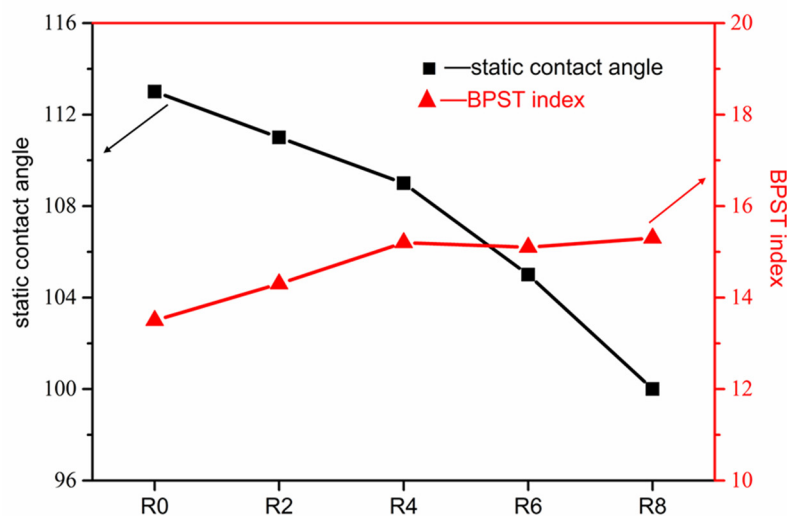


Figure 8. Water contact angles and British Pendulum Skid Tester (BPST) values of the vulcanizates.

3.3. Morphologies of the Vulcanizates

Figure 9 exhibits the SEM images of the vulcanizates after etching of the resins. It can be intuitively observed that the particle size of the resin decreases with increasing MAH content when its weight fraction is no more than 6% in M-DCPD resin, while the particle size of M8 oppositely increases. Apparently, the morphology of resin relates much to the quantity of silica they interact with. When the concentration of MAH varied from 0% to 6% in M-DCPD resin, the gradually diminishing particle sizes illustrate more even dispersion of resins, confirming the speculation above that M4, as well as M6, is able to weaken the filler network in a wider area than M8. According to the discussion about polarity of M-DCPD resin, M8 with high polarity probably phase separates with the nonpolar rubber and aggregates. As a consequence, the improvement of WSR is too weak with M-DCPD resin with low polarity, while the modified resin aggregates against dispersion and function if there is a large number of polar groups. Modified resin can effectively interact with silica and improve the WSR of the vulcanizates only when the content of polar anhydride is within an appropriate proportion.

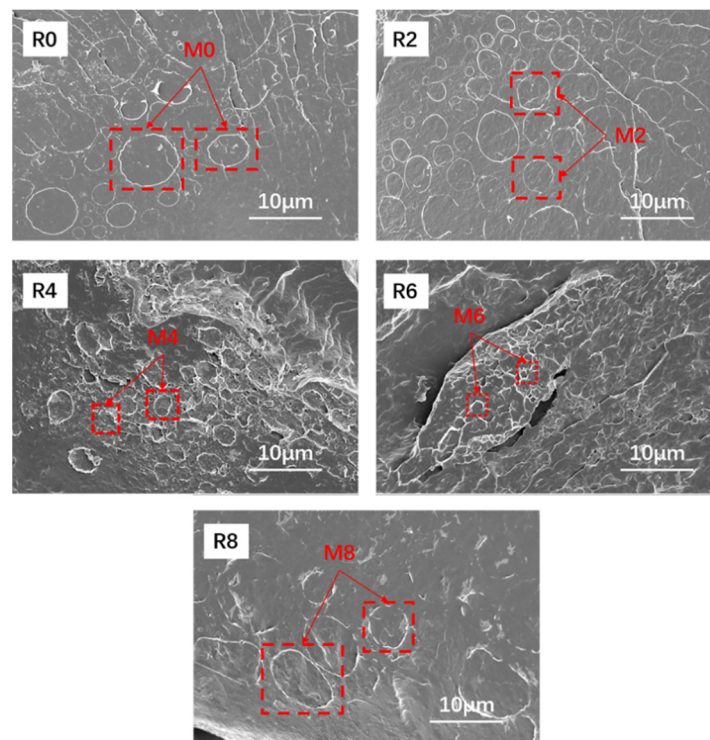


Figure 9. SEM images of the vulcanizates after etching of the resins.

3.4. Mechanical Properties of the Vulcanizates

Table 3 summarizes the physical-mechanical properties of the vulcanizates. Cross-linking density, the 100% modulus and 300% modulus increase, whereas the elongation at break decreases slightly from R0 to R6. R8 shows the opposite trend. Meanwhile, hardness, tensile strength and tear strength fluctuate in a narrow range, resilience as well. As is discussed in Sections 3.2 and 3.3, intermolecular force was enhanced by the addition of M-DCPD resins due to improved polarity of vulcanizates, resulting in increased 300% modulus. But the 300% modulus of R8 was lower than that of R6 due to the relevantly poor dispersion of M8. The enhanced 300% modulus also indicates improved dispersion of silica, since uniform dispersion is beneficial for more chemical bonding between silica and rubber chain and ultimately higher intermolecular force.

Table 3. Physical-mechanical properties of silica-filled solution-polymerized styrene-butadiene rubber /butadiene rubber (SSBR/BR) vulcanizates.

Sample	Hardness (shore A)	Cross-Linking Density (%)	100% Modulus (MPa)	300% Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)	Tear Strength (kN/m)	Resilience (%)
R0	54	20.8	1.6	10.6	17.6	414	33.1	40
R2	53	22.0	1.6	11.2	17.7	404	32.1	41
R4	54	21.2	1.7	12.6	16.7	352	29.9	39
R6	54	21.4	2.1	14.4	16.0	320	29.7	40
R8	55	21.4	1.9	13.3	16.4	339	30.6	39

4. Conclusions

WSR is significantly enhanced by M-DCPD resin with MAH content of around 4 wt% to 6 wt% from the two following aspects. On the one hand, more energy dissipation during disintegration and reconstruction of the filler aggregates under filler-resin interaction lead to a high hysteresis. On the other hand, anhydride plays a dominant role in balancing filler-filler interaction with filler-lubricant interaction, boosting surface polarity of vulcanizates within an adequate proportion. Consequently,

with the addition of DCPD resin modified with MAH in 4 wt%–6 wt%, the BPST index and the value of $\tan\delta$ at 0 °C increased by around 16% and 15%, respectively, as well as a decrease in contact angle by around 10%, indicating that both external and internal friction are obviously improved, the surface polarity of vulcanizates are also enhanced. As a result, the modification of DCPD resin with appropriate dosage of MAH could give a great improvement in WSR of the silica-filled SSBR/BR. Moreover, the enhancement of WSR is valid to improve driving safety and desirable duration of tire. Our work proposes a detailed strategy to use modified DCPD resin as antiskid resin by introducing polar groups, and we anticipate further efficient methods to explore more multifunctional resins.

Author Contributions: Z.C. conceived and designed the experiments; K.F. and R.H. performed the experiments and analyzed the data; Q.P. and R.H. wrote the paper. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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