Effects of different types of modifiers on the properties of EPDM/MVQ blended rubber

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Abstract: This study aims to enhance the compatibility of ethylene propylene diene monomer (EPDM) and silicone rubber (MVQ) based on the "principle of similar surface tension". Therefore, different types of modifiers are added to MVQ to increase its surface tension and narrow the gap with the surface tension of EPDM. Experiments show that the water contact angle of MVQ with different modifiers decreases significantly, and the interfacial thickness of the corresponding EPDM/MVQ blend increases, resulting in improved compatibility. Compared with the unmodified EPDM/MVQ blend, the mechanical properties before and after aging also increase to varying degrees.

Key words: blended rubber; contact angle; surface tension; phase interface; mechanical properties

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By blending EPDM, which exhibits excellent steam resistance, with MVQ, which boasts superior heat resistance, a product with superior comprehensive performance can be obtained. The heat resistance of this blended rubber is 10~30 ℃ higher than that of EPDM . Furthermore, it possesses higher strength and lower compression set, offering a reasonable cost-performance ratio. This meets the requirements for steam sealing products and is suitable for use as a flexible pipeline in thermal compression equipment such as steam cleaners, steam hammers, flat curing presses, and injection molding machines.

However, the blending of EPDM and MVQ faces the issue of poor process compatibility. When the two are directly blended, it is impossible for the molecules of the two rubbers to form strong intermolecular bonding forces. This is a thermodynamically incompatible system, where differences in molecular structure lead to various differences in polarity and surface tension . Generally, improving the compatibility of the blended system can be achieved from two aspects. Firstly, from the thermodynamic perspective of enhancing the interaction forces between polymer components, such as introducing intermolecular hydrogen bonds, dipole-dipole interactions, and chemical reactions between components ; secondly, from the process perspective of changing temperature, time, and adding a third-component compatibilizer. Therefore, this experiment uses acrylate rubber (ACM), ethylene-vinyl acetate rubber (EVM), nitrile rubber (NBR), and silane coupling agent KH-550 as modifiers for MVQ, allowing it to undergo certain crosslinking with MVQ. The crosslinked MVQ blend rubber improves its surface tension, achieving a surface tension similar to that of EPDM, strengthening the interaction forces between the two, and thus improving the compatibility of MVQ and EPDM. The results show that the EPDM/MVQ blend rubber using NBR as a modifier has the largest interphase thickness, the best compatibility, and the best aging resistance. In terms of macroscopic mechanical properties, due to the combined effects of compatibility and improved rubber-filler interaction, the blend rubber using KH-550 as a modifier exhibits the best mechanical properties before and after aging.

Biography: Dai Xin (2000-), female, is a graduate student pursuing her master's degree, primarily engaged in research on rubber blending and modification.

1 Experimental part

1.1 Experimental raw materials

EPDM 2650C, Arlanxeo; MVQ 9170 M, Elkem Silicones; ACM 121X, Jiujiang Dewei; EVM 700, Lanxess; NBR 1052, Zhenjiang Nadi; other additives are commonly used industrial products.

1.2 Experimental formula

Table 1 Experimental formula (in parts)

serial number	1#	2#	3#	4#	5#
ACM 121X	0	3	0	0	0
EVM 700	0	0	3	0	0
NBR 1052	0	0	0	3	0
KH-550	0	0	0	0	1.2

The experimental formula is shown in Table 1, with all other compounding ingredients being the same (in parts): EPDM 2650C 70, MVQ 9170 M 46.2, silica 10, ZnO 5, SA 1, DCP 2, TAIC 1.5, and antioxidant RD 1.5.

1.3 Sample preparation

Weigh various raw materials according to the recipe. Firstly, place EPDM on the open mill and add silica white in batches after the rollers are wrapped. Make 8 triangle packs and 5 rolls, then take it out for later use. Secondly, prepare the modified MVQ by placing MVQ in the open mill and wrapping the rollers. After that, add modifiers separately and mix them evenly before taking them out. Then, mix the EPDM masterbatch and modified silicone rubber, make 5 triangle packs and 3 rolls, add small materials for mixing, and finally add the curing agent. Mix them evenly before taking them out and let them stand for 16 hours. Then, cure them on the flat vulcanizing press under the conditions of 170 ℃ ×10 MPa×t_{90}.

1.4 Analysis and testing

1.4.1 Compatibility characterization

Based on the calculation of the surface tension of vulcanized rubber using the Owens two-liquid method (with water and α-bromonaphthalene as the test liquids), and the calculation of the interfacial thickness of blended vulcanized rubber according to literature to characterize its compatibility, a larger interfacial thickness of the blended rubber indicates better compatibility.

1.4.2 Mechanical properties

The tensile properties were tested using an electronic tensile testing machine of model I-7000S, manufactured by Taiwan High Speed Rail Corporation, in accordance with GB/T 528-2008. The tensile speed was 500 mm/min, and the test temperature was room temperature.

1.4.3 Aging resistance

The thermal-oxidative aging conditions are set at 120℃ for 72 hours.

1.4.4 Wear resistance

The Shao Poer abrasion test method (with a load of 10 N) was employed.

2 Results and Discussion

2.1 Vulcanization characteristic data

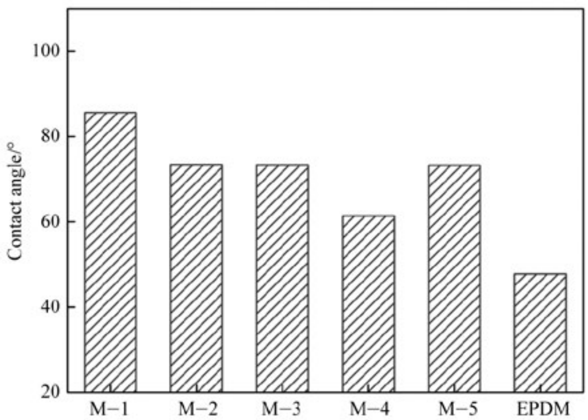
The vulcanization characteristic parameters of EPDM/MVQ blend rubber are shown in table 2. The scorch time t_{10} of the five blend rubbers is relatively short, indicating poor processing safety. The process optimum vulcanization time t_{90} of 4# blend rubber is the longest, and its vulcanization reaction rate is slower compared to the other four blend rubbers.

Table 2 Vulcanization characteristic parameters

Vulcanization characteristic data/number	1#	2#	3#	4#	5#
M_H /(dN·m)	22.14	22.27	22.83	21.33	22.88
M_L /(dN·m)	0.88	0.94	0.96	0.89	1.36
t_{10} /(min:s)	0:45	0:46	0:45	0:47	0:45
t_{90} /(min:s)	6:30	6:29	6:24	8:45	6:50

2.2 Compatibility characterization

The results of water contact angle tests for MVQ, modified MVQ, and EPDM are presented in Figure1, while the surface tension and interfacial thickness of the blends are illustrated in Figure2. Compared to the water contact angle of pure MVQ, the contact angle of MVQ with modifiers such as ACM, EVM, NBR, and KH-550 decreases, indicating improved wettability. The factors affecting the surface contact angle are also related to surface polarity . Specifically, the presence of polar groups (ester groups, cyano groups) in MVQ with modifiers increases its surface tension. For KH-550, one end hydrolyzes to form a hydroxyl group and the other end hydrolyzes to form an amino group under high temperature conditions , leading to a decrease in contact angle and an increase in surface tension. Among them, the addition of NBR significantly reduces the contact angle of MVQ. The addition of ACM, EVM, and KH-550 also reduces the contact angle of MVQ, but the reduction is not as significant as that achieved by adding NBR.



(Note: M-1 refers to MVQ, M-2 refers to MVQ/ACM, M-3 refers to MVQ/EVM, M-4 refers to MVQ/NBR, and M-5 refers to MVQ/KH-550)

Figure 1 Water contact angles of MVQ, modified MVQ, and EPDM

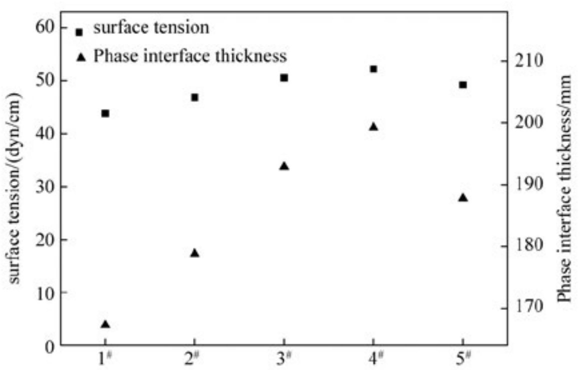


Figure 2 Surface tension and interfacial thickness of the blend

By modifying MVQ, its surface tension is increased, narrowing the gap with EPDM's surface tension, achieving similar surface tension, and enhancing the compatibility between MVQ and EPDM. As shown in Figure 2, the introduction of NBR can significantly increase the surface tension of the MVQ phase, enhancing its compatibility with the EPDM phase. This leads to an increase in the mutual attraction force between the two phases and an increase in the interfacial thickness. Among the five EPDM/MVQ blends, the 4# blend exhibits the best compatibility, with the closest surface tension between the two phases, the strongest mutual attraction force, the strongest interfacial adhesion, the thickest interphase, and the worst compatibility in the unmodified 1# blend, with the thinnest interphase.

2.3 Mechanical properties

As can be seen from Table 3, the addition of modifiers has increased the surface tension of the MVQ phase, making it closer to that of the EPDM phase. This has improved the compatibility between the two, enhanced the interfacial adhesion, and increased the tensile strength and tear resistance. The tensile strength of the EPDM/MVQ blend remains relatively unchanged and is at a similar level. The tear strength of the blends with EVM, NBR, and KH-550 as modifiers has been improved. Overall, the mechanical properties of blends 3#, 4#, and 5# are better than those of the unmodified blend. Furthermore, the increase in tensile strength of blend 5# is attributed to the enhanced interaction between rubber and filler due to KH-550. The significant decrease in the elongation at break is due to the condensation of hydroxyl groups generated by KH-550 with hydroxyl groups on the surface of silica, forming chemical bonds, and the formation of hydrogen bonds on the surface of amino silica. This increases the aggregation of fillers and the number of stress concentration points. In other words, the changes in the mechanical properties of blend 5# are caused by a combination of multiple factors.

Table 3 Mechanical properties of EPDM/MVQ blends

serial number	1#	2#	3#	4#	5#
Hardness / Shore A	60	59	61	60	59
Tensile strength / MPa	6.2	5.5	6.3	6.4	6.6
Elongation at break /%	457	428	465	422	387
50% modulus at fixed extension / MPa	1.1	1.1	0.9	1.0	1.1
100% modulus at fixed extension / MPa	1.4	1.5	1.3	1.4	1.6
200% modulus at fixed extension / MPa	2.1	2.1	2.0	2.2	2.8
300% modulus at fixed extension / MPa	3.2	3.2	3.0	3.4	4.4
Permanent set at break /%	17.5	15.8	14.2	13.3	11.7
Tear strength /(kN·m^{-1})	19.9	19.3	22.6	21.2	23.5

2.4 Wear resistance

Figure 3 compares the wear resistance of different EPDM/MVQ blends. Generally speaking, the greater the wear volume, the worse the wear resistance. As can be seen from the figure, blends 2# and 5# have smaller wear volumes and better wear resistance, while blends 1#, 3#, and 4# have larger wear volumes and poorer wear resistance. Among them, the wear resistance of blends modified with ACM and KH-550 is better than that of unmodified blends, while the wear resistance of blends modified with NBR is inferior to that of unmodified blends.

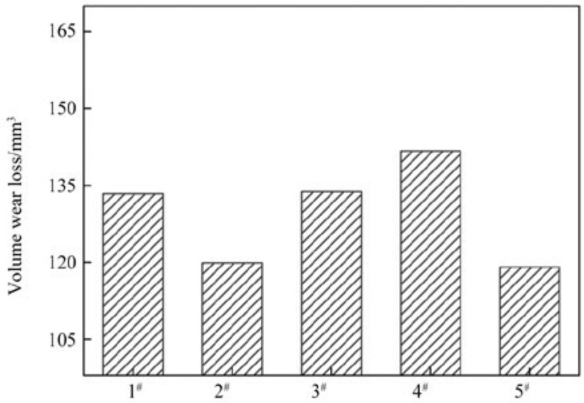


Figure 3 Wear resistance of EPDM/MVQ blends

2.5 Aging resistance

The samples of EPDM/MVQ blend were subjected to thermo-oxidative aging at 120 ℃ for 72 hours. During this process, the crosslinking density of the blend increased, and its hardness and modulus at a fixed extension increased compared to before aging. The mechanical property data of the aged blend is shown in Table 4. The decrease in tensile strength and elongation at break of blends 1#, 3#, and 4# is due to the excessive crosslinking density, which leads to an uneven crosslinking network. Under external forces, the number of stress concentration points increases, making them prone to fracture. The increase in tensile strength of blends 2# and 5# indicates that their vulcanization degree increased during the thermo-oxidative aging process, and the crosslinking network structure is relatively uniform. Overall, under the same aging conditions, the tensile strength and elongation at break of the modified blend are higher than those of the unmodified one.

Table 4 Mechanical properties of aged EPDM/MVQ blends

Serial number	$1^\#$	$2^\#$	$3^\#$	$4^\#$	$5^\#$
Hardness / Shore A	65	64	65	63	65
Tensile strength / MPa	5.6	5.7	5.7	5.8	6.8
Elongation at break /%	257	355	328	302	275
50% modulus at fixed extension / MPa	1.4	1.2	1.2	1.4	1.3
100% modulus at fixed extension / MPa	2.0	1.7	1.7	1.8	2.1
200% modulus at fixed extension / MPa	3.9	2.7	2.9	3.3	4.1
300% modulus at fixed extension / MPa	–	4.4	5.0	5.7	–
Permanent set at break /%	5.8	13.3	10.0	7.5	2.5
Tear strength /(kN·m^{-1})	23.8	22.4	21.3	17.6	23.0

The aging resistance of EPDM/MVQ blends was evaluated based on the changes in hardness and elongation at break before and after aging. As shown in Figure 4, the increase in hardness was the greatest for $5^\#$ and the smallest for $4^\#$. Furthermore, the elongation at break change rate of the modified EPDM/MVQ blends was lower than that of the unmodified blends. That is, considering both change rates, the aging resistance of $4^\#$ blend was the best, and compared to the unmodified $1^\#$ blend, the aging resistance of $2^\#$~$5^\#$ blends was slightly better.

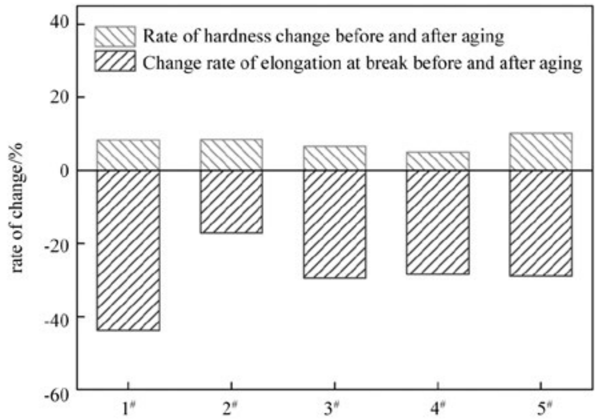


Figure 4 Change rate of hardness and elongation at break of the blend before and after aging

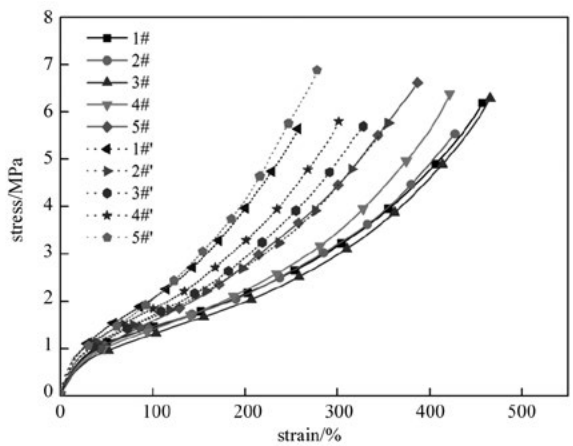


Figure 5 Comparison of tensile behavior of blends before and after aging

Note: $1^\#$~$5^\#$ represent the mechanical behavior curves of EPDM/MVQ blends at room temperature, while $1^{\#'}$~$5^{\#'}$ represent the mechanical behavior curves of EPDM/MVQ blends after aging at 120 ℃ for 72 hours.

In addition, by comparing the tensile behavior curves of EPDM/MVQ blends before and after aging, as shown in Figure 5, it can be observed that the tensile behavior of the unmodified EPDM/MVQ blend changes significantly before and after aging, once again verifying that the addition of modifiers improves the aging mechanical behavior of the blend. Meanwhile, to further investigate whether this type of blend can be used for long-term service under more severe high-temperature environments, the EPDM/MVQ samples were subjected to a thermo-oxidative aging test at 170℃ for 72 hours, as shown in Figure 6.

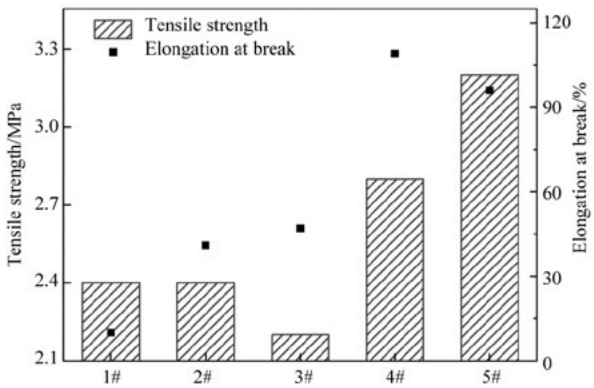


Figure 6 Tensile strength and elongation at break of the blended rubber after aging at 170 ℃

After undergoing a prolonged high-temperature exposure, the tensile strength and elongation at break of the blend samples have significantly decreased compared to those subjected to thermo-oxidative aging at room temperature and 120°C. Upon comparing five types of EPDM/MVQ blends, it was found that blends $4^{\#}$ with NBR and $5^{\#}$ with KH-550 exhibited higher strength and elongation, significantly surpassing the other three blends. This indicates that blends $4^{\#}$ and $5^{\#}$ are more resistant to high temperatures and have a longer service life, making them suitable for use in harsh high-temperature working environments.

3 Conclusion

(1) Using ACM, EVM, NBR, and KH-550 as modifiers for MVQ with high surface tension can significantly reduce its water contact angle, narrow the difference in surface tension with EPDM, and enhance the compatibility between the two. Among them, the addition of NBR has the most pronounced effect, resulting in the thickest interphase and the best compatibility of the blended rubber.

(2) From the perspective of macroscopic mechanical properties, the performance of $5^{\#}$ blend rubber is not only influenced by compatibility, but also by the interaction between fillers and rubber, as well as the aggregation degree of fillers. Overall, its mechanical properties are the best, followed by $4^{\#}$.

(3) After thermal-oxidative aging treatment at 120℃ and 170℃ respectively, the EPDM/MVQ blends $4^{\#}$ and $5^{\#}$ exhibited higher tensile strength and elongation at break, indicating excellent aging resistance.