The influence of CM compounding amount on the mechanical properties of ACM/CM vulcanizate

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Abstract: This paper investigates the impact of CM blended amount on the vulcanization characteristics, physical and mechanical properties, and wear resistance of ACM/CM vulcanizates. The results indicate that as the CM blended amount increases, the Mooney viscosity (M_H) and Mooney viscosity-at-300% (M_H-M_L) of ACM/CM vulcanizates gradually increase, while the vulcanization rate significantly decreases. Before aging, the tensile strength, elongation at break, and tear strength of ACM/CM vulcanizates initially increase and then decrease with the increase in CM blended amount. When the CM blended amount is 40 parts, the mechanical properties of ACM/CM vulcanizates before and after aging are optimal, and the wear resistance is also the best.

Key words: CM blended amount; vulcanization characteristics; physical and mechanical properties; wear resistance

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Acrylate rubber (ACM) is an elastomer obtained through copolymerization using acrylate monomers as the main monomer. Its main chain is a saturated carbon chain, and its pendant groups are highly polar ester groups. Therefore, ACM exhibits excellent heat resistance, oil resistance, and resistance to thermal-oxidative aging, and is widely used in heat-resistant oil products. However, it also has disadvantages such as low mechanical strength, poor elasticity, poor low-temperature resistance, and poor processability, which severely limit its application fields and make it unable to meet the requirements for use in certain special environments. CM is a new type of specialty rubber relative to ACM. The main chain of CM molecules is saturated, similar to that of ACM, and has a certain degree of compatibility. In addition, CM has superior tensile properties, elasticity, low-temperature resistance, and processability compared to ACM, but its heat resistance and oil resistance are slightly inferior. The addition of a small amount of CM can improve the mechanical properties of ACM, while reducing costs without significantly affecting the main properties of the vulcanizate. Therefore, this paper investigates the changes in the mechanical properties of ACM/CM vulcanizate by adjusting the blending ratio of CM, aiming to find the optimal blending ratio of CM and improve the comprehensive performance of ACM. This is of great significance for expanding the application fields of ACM and enhancing its application value.

1 Experimental part

1.1 Raw materials

ACM-101X, Jiujiang Dewei Rubber Technology Co., Ltd.; CM135B, Weifang Yaxing Chemical Co., Ltd.; other additives are commonly sold raw materials in the rubber industry.

1.2 Experimental instruments and equipment

The instruments and equipment used in the experiment are listed in Table 1.

Biography: Liu Changshun (1999-), master's graduate student, mainly engaged in research on rubber blending and modification.

Table 1 Experimental instruments and equipment

Instrument Name	Model	Manufacturer
Open mill	X(S)K−160	Shanghai Shuangyi Rubber & Plastics Machinery Co., Ltd
Internal mixer	XSM−1/20−80	Shanghai Kechuang Rubber and Plastic Machinery Equipment Co., Ltd
Flat vulcanizing press	LCM−3C2−G03−LM	Shenzhen Jiaxin Electronic Equipment Technology Co., Ltd
Rotorless vulcanometer	GT−M2000−A	Taiwan High Speed Rail Corporation Limited
Electronic tensile machine	I−7000S	Taiwan High Speed Rail Corporation Limited
hardness tester	Shaw's LX−A type	Shanghai Xianfeng Film Machinery Factory
DIN abraser	GM−1	Yangzhou Tianfa Experimental Machinery Co., Ltd

1.3 Experimental formula

The formulations for comparing the influence of CM on the mechanical behavior of ACM/CM vulcanizates are shown in Table 2.

Table 2 ACM/CM blend ratio per formula/portion

Serial number	1#	2#	3#	4#	5#
ACM101x	100	90	80	70	60
CM135B	0	10	20	30	40

All other components of the formula are the same (unit: phr): 20 phr of carbon black N330, 15 phr of silica, 2 phr of TCY, 2 phr of BZ, 10 phr of CaCO3, and 5 phr of MgO.

Total (unit: phr): 184.

1.4 Sample preparation

Prepare ACM masterbatch and CM masterbatch in the internal mixer according to the experimental formula, with the mixer temperature at 65 ℃, rotor speed at 30 r/min, and rubber discharge after about 15 minutes; then prepare ACM/CM blend rubber with different raw rubber ratios on the open mill, add the curing system, make triangle packs 6 times, roll 6 times, take off the sheet, and make marks (1#~5#); let it stand for more than 8 hours, and cure the sample on the flat vulcanizing press with curing conditions of 165 ℃×t_{90}×10MPa.

1.5 Analysis and Testing

(1) Vulcanization characteristic test: The vulcanization characteristic is tested according to GB/T 16584-1996, with test conditions of 165 ℃×30 min, and the rotation angle is ±1°

(2) Tensile property test: The test is conducted according to the GB/T 528-2008 standard, with a tensile speed of 500mm/min and a test temperature of room temperature.

(3) Shore A hardness test: Conduct the test according to the GB/T531.1-2008 standard, using a hardness tester, and set the test temperature to room temperature.

(4) Permanent set at break: According to GB/T 528-2009, the test sample is left to rest for 3 minutes after being pulled apart, and the distance between two marked points is measured and recorded as L1. The permanent set at break is calculated using (1);

$$H=(L_1-L_0)/L_0\times100\% \quad (1)$$

Where L_0 is the initial gauge length of the sample (mm)

(5) Tear strength test: Conduct the test according to GB529 standard, with the test temperature being room temperature.

(6) Abrasion resistance test: Conduct the test according to GB/T 9867-2008, with a load of 10 N;

(7) Aging test: For hot air aging, follow GB/T 3512-2001, with aging conditions of 100 ℃×72 h; for hot oil aging, place the rubber sample in a 46# hydraulic oil tank according to GB/T 1690-92, and then place the oil tank in an aging oven according to GB/T 3512-2001, with aging conditions of 100 ℃×72h;

(8) Crosslinking density determination: Using methyl methacrylate as the solvent, the vulcanized rubber Vr (volume fraction of gel in rubber) was measured based on the equilibrium swelling method described in the literature. Vr was used to characterize the crosslinking density of rubber, and there is a positive correlation between Vr and the crosslinking density of rubber.

2 Results and Discussion

2.1 Vulcanization characteristics

The influence of different CM blend ratios on the vulcanization characteristics of ACM/CM blends is presented in Table 3.

Table 3 Vulcanization characteristics of ACM/CM rubber compound with different CM blending ratios

Serial number	1#	2#	3#	4#	5#
M_H/(dN·m)	16.30	20.20	20.61	21.42	22.03
M_L/(dN·m)	3.31	3.45	3.75	3.83	3.97
M_H-M_L/(dN·m)	12.99	16.75	16.86	17.59	18.06
t_{10}/min	0.23	0.31	0.35	0.38	0.45
t_{90}/min	4.08	19.00	21.23	22.28	23.24

According to the data in Table 3, the incorporation of CM significantly reduces the vulcanization rate of ACM/CM, leading to a notable increase in the t_{90} and t_{10} of the blend, thereby improving the processing safety of the blend. Under the condition of a fixed amount of vulcanizing agent, as the amount of CM incorporated increases, the M_H, M_L, and M_H-ML of the blend also continuously increase.

2.2 Apparent crosslinking density

The apparent crosslinking density V_r of ACM/CM vulcanizates before and after aging, obtained under different CM blend amounts through the equilibrium swelling method, is presented in Table 4.

Table 4 Vr of ACM/HNBR vulcanizate before and after aging under different CM blending amounts

Serial number	1#	2#	3#	4#	5#
Vr/% before aging	20.84	23.66	25.74	27.96	29.37
Vr/% after aging	22.07	24.12	26.07	27.82	29.61

The experiment utilizes Vr to characterize the crosslinking density of ACM/CM vulcanizates, where a higher Vr indicates a greater degree of crosslinking in the vulcanizate. As evident from Table 4, the Vr of the vulcanizates after aging is increased compared to that before aging. This is due to further crosslinking of the vulcanizates during the aging process, leading to an increase in the crosslinking network density of the ACM/CM vulcanizates.

2.3 Physical and mechanical properties

2.3.1 Effect of CM compounding amount on the physical and mechanical properties of ACM/CM vulcanizate before and after hot air aging

The experimental results of the influence of different CM blending amounts on the physical and mechanical properties of ACM/CM vulcanizate before and after aging are shown in Table 5.

Table 5 Physical and mechanical properties of ACM/CM vulcanizates before and after hot air aging under different CM blending amounts

Formula Number		1#	2#	3#	4#	5#
Hardness/Shore A	before aging	86	87	90	90	92
	After aging	90	92	92	93	92
Tensile strength / MPa	before aging	14.5	13.1	13.8	14.8	17.2
	After aging	14.3	15.4	16.3	15.7	17.2
Elongation at break, %	before aging	235	187	188	199	215
	After aging	106	163	165	115	142
50% modulus at fixed extension/MPa	before aging	5.0	4.2	4.1	5.1	5.0
	After aging	7.5	5.8	5.9	7.4	6.9
100% modulus/MPa	before aging	8.5	7.8	7.7	9.6	8.9
	After aging	13.8	10.8	11.5	13.9	13.2
200% modulus/MPa	before aging	13.4	12.8	–	–	16.4
	After aging	–	–	–	–	
Permanent deformation upon fracture /%	before aging	15.0	10.0	7.5	10.0	10.0
	After aging	5.0	7.5	7.5	5.0	6.0
Tear strength / (kN·m^{-1})	before aging	27.0	26.8	25.3	27.3	29.1
	After aging	22.7	25.1	25.6	23.1	27.4

As can be seen from Table 5, with the increase in the amount of CM co-used, the tensile strength, elongation at break, 50% modulus, tear strength, and permanent set of the ACM/CM vulcanizate before aging first decrease and then increase. When the amount of CM co-used is 40 phr, the mechanical properties of the ACM/CM vulcanizate are the best. This is because the addition of a small amount of CM cannot form a uniform cross-linked network with the ACM phase, but instead disrupts the uniformity of the ACM phase cross-linked network, introducing stress concentration points, which leads to a decrease in the mechanical properties of the blended rubber. However, as the amount of CM co-used increases, the CM phase cross-linked network in the blended rubber gradually improves, and the matching degree with the ACM phase cross-linked network gradually increases. When the blended rubber is stressed, the two phase cross-linked networks can respond simultaneously, thereby improving the physical and mechanical properties of the blended rubber.

Table 6 Change rate of tensile properties of ACM/CM vulcanizate before and after hot air aging under different CM blending amounts

Performance	1#	2#	3#	4#	5#
Change rate of tensile strength, %	–1.4	17.6	18.1	6.1	0.0
Rate of change of elongation at break, %	–54.9	–12.8	–12.2	–42.2	–34.0

Compared with before aging, the hardness, tensile strength, and modulus at various extension rates of ACM/CM vulcanizate after aging have significantly increased, while the elongation at break and permanent set at break have decreased. Based on Table 5, this is attributed to the further crosslinking of the vulcanizate during the aging process, leading to an increase in crosslinking density and resulting in a harder and brittle vulcanizate. Referring to Table 6, when the amount of CM used is 20 phr, the heat-resistant air aging performance of ACM/CM vulcanizate is the best.

2.3.2 Effect of CM blending ratio on the tensile properties of ACM/CM vulcanizate after hot oil aging

To further investigate the effect of CM and its dosage on the heat oil aging resistance of ACM/CM vulcanizate, the tensile properties and heat oil volume change rate of the blended rubber after heat oil aging were measured. The experimental results are presented in Table 7.

Table 7 Change rate of properties of ACM/CM vulcanizate after hot oil aging under different CM blending amounts

performance	1#	2#	3#	4#	5#
Tensile strength/MPa	13.7	14.1	14.1	14.2	16.1
Elongation at break, %	169	137	172	139	191
Change rate of tensile strength, %	−5.5	7.1	2.2	−4.1	−6.4
Change rate of elongation at break, %	−28.1	−26.7	−8.5	−30.2	−11.2
Oil resistance volume change rate /%	0.52	1.82	2.13	1.67	0.35

Combining Table 5 and Table 7, it can be concluded that compared to before aging, the tensile strength of ACM/CM vulcanizate after hot oil aging increases, while the elongation at break decreases. This is due to further crosslinking of the blend caused by high temperature. Further analysis shows that when the amount of CM used is 20 phr, the change rate of tensile properties of the blend before and after hot oil aging is minimal. However, when the amount of CM used is 40 phr, the mechanical properties are optimal. This is because when the amount of CM used is 40 phr, the CM phase crosslinking network in the blend is highly perfected, and the ACM phase crosslinking network has a certain degree of matching. Oil is not easily absorbed, and small molecules in the blend are not easily precipitated, resulting in minimal changes in oil resistance volume change rate.

2.4 DIN abrasion

As can be seen from Figure 1, with the increase in the amount of CM used, the abrasion volume of ACM/CM vulcanizate first increases and then decreases. When the amount of CM is 40 phr, the abrasion resistance of the blended rubber is the best.

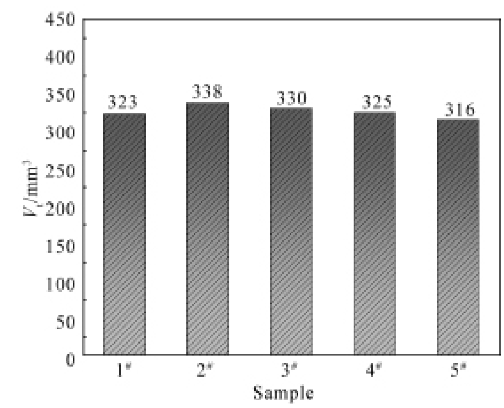

Figure 1 Wear volume of ACM/HNBR vulcanizate with different CM blending amounts

3 Conclusion

(1)With the increase in the amount of CM used, the MH, ML, and MH-ML of the blend gradually increase, and the vulcanization rate of the blend significantly decreases, significantly improving the processing safety of the blend.

(2)With the increase in the amount of CM used, the tensile strength, elongation at break, and tear strength of ACM/CM vulcanizate before aging first increase and then decrease. When the amount of CM used is 40 phr, the comprehensive performance of the blended rubber is the best. After hot air aging, the tensile strength of ACM/CM vulcanizate increases, while the elongation at break decreases. When the amount of CM used is 20 phr, the resistance to hot air aging is the best, but the tensile properties are optimal when the amount used is 40 phr. After hot oil aging, when the amount of CM used is 20 phr, the change rate of tensile properties before and after aging is the smallest, but the tensile properties are optimal when the amount used is 40 phr, and at this time, the oil resistance volume change rate of the blended rubber is the smallest.

(3)When the combined amount of CM is 40 phr, the wear resistance of ACM/CM vulcanizate is the best.