Effect of thermo-oxidative aging on the fluidity and adhesion of compounded modified asphalt

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Abstract. This paper aims to investigate the aging behavior of high viscosity/SBS compound modified asphalt. To this end, asphalt specimens with different aging degrees were prepared separately, the fluidity of asphalt was evaluated by viscosity test, the adhesion between asphalt and aggregate before and after aging was investigated by contact angle test, and finally the correlation between the fluidity of asphalt and its adhesion was explored based on the gray correlation theory. The results showed that aging had a significant effect on the Newtonian fluid state of asphalt, but the trend and degree of the effect were related to the type of asphalt. Aging weakened the adhesion between asphalt and aggregate, but the compounding of high viscosity agents with SBS could slow down this aging effect. Asphalt fluidity can be used to predict its ability to adhere to aggregates, which in turn guides the design and construction of asphalt mixtures.

Keywords: Thermal-oxidative aging; High viscosity agent /SBS compound modified asphalt; Fluidity; Adhesion work; Open Graded Friction Course (OGFC).

1 Introduction

Open Graded Friction Course (OGFC) pavement is an open-graded asphalt pavement structure consisting of a combination of high-performance asphalt mastic, a high percentage of pore space, and an aggregate skeleton [1]. In order to achieve the effect of permeability and noise reduction, the internal voids of OGFC pavements are usually controlled to about 20%. As a result, the OGFC pavement surfaces reduce the noise of tires rubbing against the pavement as well as splash and rain spray during heavy rainfall extremes. The noise generated by OGFC pavements is typically reduced by about 3 dB compared to densely graded asphalt pavements [2]. In addition, the internal voids of OGFC are through and through, which can minimize the residue of surface water on the road surface under heavy rainfall, thus playing a role in preventing urban flooding, etc., and improving antiskid performance under high speed driving to ensure the operation safety of vehicles. Based on the above significant advantages, OGFC pavement

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has been widely used and promoted in the roads of many Asian countries such as Japan, China, and Singapore [3].

OGFC pavement surface can play its excellent durability and mechanical properties, the key lies in its use of high viscosity modified asphalt (HVMA). HVMA because of high power viscosity and softening point, can be more attached to the surface of the aggregate, therefore, OGFC does not easy to deformation in high temperatures and heavy loads, the internal voids are not destroyed, and the pavement's drainage function will not be affected [4]. At present, the more popular finished HVMA are TPS (TAFPACK-Super) of Japan, SINOTPS of Shenzhen Oceanpower of China. And scholars have also carried out a lot of research work on the development of HVMA and achieved more fruitful results. Hussei [5] et al. used nanoceramic powders with matrix asphalt to prepare high-viscosity modified asphalt and found that the interaction between Si-O-Si stretching vibrations and hydrocarbon molecules in the nanoceramic powders reassembles the asphaltene particles, thus increasing the viscosity of the asphalt. Zhang [6] et al. prepared an HVMA consisting of styrene-butadiene-styrene (SBS), plasticizer, and cross-linking agent. The mechanism of this is to improve the rutting resistance and viscosity of asphalt-by-asphalt plasticizer and to promote the swelling and dispersion of SBS in asphalt by promoting the formation of polymer network using crosslinking agent to obtain better processing ease, durability, and stability. Alam [7] et al. studied the effect of polyphosphoric acid and SBS on the modification of matrix asphalt with different dosing levels, and the results showed that with the increase in the content of polyphosphoric acid and SBS, the proportion of asphaltene and viscosity in the modified asphalt increased linearly. In conclusion, many research teams have made plenty of contributions to the preparation and performance characterization of HVMA, exploring the modification effects of SBS, resins, nanomaterials, rubber powders [8], recycled oils, waste building materials and other materials in different ratios, which favorably promotes the development and application of HVMA. However, there are few studies on the compounding of HVMAs with SBS modifiers. In fact, both HVMA and SBS modifier are finished product modifiers, and the study of the compounding effect between them is of practical significance in engineering.

At the same time, due to higher porosity, OGFC pavements face severe challenges of transitional exposure to oxygen, ultraviolet light, humidity, and temperature during long-term service, causing them to deteriorate at a higher rate than densely matched asphalt pavements. Severe aging reactions lead to increased asphalt stiffness and brittleness, decreased asphalt-aggregate adhesion, and a decline in stress relaxation properties and flexibility [9]. This is manifested in the pavement by an increase in macroscopic distresses such as spalling, potholes, rutting, etc., which accelerates the durability failure of the pavement [10]. Therefore, the service life of OGFC pavements is usually lower than that of dense-matched asphalt pavements. Much work has been put into indoor testing and outdoor testing to investigate the effects of different aging on asphalt and asphalt mixtures. Molenaar [11] et al. investigated the effects of short-term aging and long-term aging on the mechanical properties of HVMA and compared them with field-aged binders. The results showed that aging increases the tensile strength of HVMA but decreases the strain at fracture. Aging starts to increase the fatigue properties of the asphalt, but field aging leads to a significant decrease in fatigue resistance after 7 years. Hu [12] et al. on the other hand, compared in detail the rheological properties, chemical compositions, and micromorphology of HVMA, SBS, and base asphalt before and after thermo-oxidative aging. The results showed that short-term aging leads to the degradation of the polymer phase structure of HVMA, resulting in a decrease in the rheological properties of HVMA, while the oxidation of matrix asphalt is an important cause of the change in the rheological properties of HVMA after long-term aging. Li [13] et al. investigated the aging behavior of HVMA in a coupled heat-light-water environment, and examined the rheological properties and microstructures of the asphalt by DSR, BBR and ESEM tests, respectively. The results showed that acid rain solution and UV radiation had significant effects on the viscoelastic properties, high and low-temperature properties, and fatigue properties of HVMA. However, the above studies pay more attention to the analysis of the single performance of HVMA, and rarely discuss the relationship between the rheological properties of asphalt and its adhesion. In fact, it is easier to obtain the rheological properties of asphalt by testing, but due to the complexity of instruments and operations, asphalt adhesion performance parameters are rarely obtained quickly. If the rheological properties can be used to predict and evaluate the corresponding adhesion ability of asphalt, it will be helpful to the design and construction of HVMA.

In view of this, in this paper, a high viscosity/SBS compounded modified asphalt was prepared using commercially available finished high viscosity modified asphalt with SBS modified asphalt and compared with the finished high viscosity modified asphalt and SBS modified asphalt. The fluidity of the asphalt under different aging levels was evaluated by viscosity tests, and contact angle tests were conducted to quantitatively examine the adhesion between asphalt and aggregate. Finally, asphalt fluidity and asphalt adhesion were analyzed using gray correlation theory. This study proves the effectiveness of high viscosity agent and SBS in improving the aging resistance and adhesion of asphalt, which can provide useful data and theoretical support for the design and construction of HVMA.

2 Test materials and methods

2.1 Materials

In this paper, SBS modified asphalt and Finished high viscosity modified asphalt (FHVMA) are both provided by PetroChina Foshan Company Limited, and their basic properties are shown in Table 1.

Table 1. Basic performance of SBS and FHVMA asphalt.

Item	Unit	SBS	FHVMA	Test method
25℃ Penetration	0.1mm	46.9	47.8	T0604
Softening point	℃	75.5	88.4	T0606
135℃ Viscosity	Pa·s	1.96	2.75	T0625

The high viscosity agent was from Tianjin Zhongyou Gaoyuan New Material Co. Ltd. and its basic technical performance indexes are shown in Table 2. In addition, the aggregate used in the test was limestone.

Table 2. Basic performance of 70# asphalt and SBS modified asphalt.

Item	Unit	Result	Requirement	Standards
Appearances	/	Granular, uniform, full, no lumps	Granular, uniform, full, no lumps	/
Color	/	Light red	Light red	/
Single particle mass	g	0.0096	≤0.03	T0606
densities	g/cm3	0.958	≤1.0	GB/T 1033
Lit. melting index	g/10min	2.436	≥2.0	GB/T 3682
Ash content	%	0.76	≤1.0	T0614-2011

2.2 Compound high viscosity modified asphalt preparation

Compound high viscosity modified asphalt (CHVMA) is compounded from SBS modified asphalt and high viscosity agent, the specific preparation process is as follows: firstly, SBS modified asphalt is put into the oven at 175 °C and heated to the flow state; secondly, 5% of the asphalt mass of high viscosity agent is added slowly, and in the process, the high viscosity agent is dispersed uniformly in asphalt by using a glass rod; then, at 160 °C, the asphalt and high viscosity agent are fully stirred for 30min by using the dispersion mixer; then, at 170 °C, it is sheared for 45min by using a high-speed shear emulsifier at 2000r/min; then, at 5000r/min, it is sheared for 45min. Fully stirred for 30 min; then, at 170°C, the asphalt was sheared for 30 min at a speed of 2000 r/min using a high-speed shear emulsifier, and then sheared for 45 min at a speed of 5000 r/min; finally, the sheared asphalt was then placed in an oven at 170°C to develop for 1h.

2.3 Aging test

In order to simulate the short-term aging of asphalt mixtures during production and paving, the asphalt was subjected to a rolling thin film oven test (RTFOT) according to JTG E20-2011 (Chinese Standard 2011). Then, the asphalt after RTFOT aging was further subjected to pressurized aging vessel (PAV) test according to JTG E20-2011 in order to simulate the long-term aging of the pavement during its service life. Thus, SBS, FHVMA and CHVMA have three aging states: Original asphalt (OA), RTFOT and PAV.

2.4 Viscosity test

The viscosity test was conducted according to JTG E20-2011 to test the viscosity of three different asphalts at 135°C, 155°C, 165°C and 175°C. Rotor speeds of 2, 5, 10, 20 and 50 r/min were used respectively.

2.5 Contact angle test

The contact angle test was used to detect the adhesion performance between asphalt and aggregate before and after aging. Firstly, the asphalt was heated to a molten state using an oven, and then a dry slide was completely immersed into the asphalt, and the slide was slowly removed after full adhesion, and then trimmed around the slide after cooling. The limestone aggregate was cut and processed into cubes of 10 × 10 × 10 mm, and the surface of the cubes was ground and polished using coarse and fine sandpaper, and was cleaned by distilled water several times before being put into the oven to dry for more than 6h. According to ASTM D7490-13 (ASTM 2013), the contact angle of asphalt and limestone was measured by using HARKE-SPCA contact angle meter. The test temperature was chosen to be 25°C, and the reagents were chosen to be three materials with known surface energies: distilled water, propanetriol, and ethylene glycol, and their surface energy parameters are shown in Table 3.

Table 3. Surface energy of liquid.

Liquid	γ_{LV}	γ_{LV}^{P}	γ_{LV}^{d}
Distilled water	72.8	51.0	21.8
Ethylene glycol	48.3	19.0	29.3
Glycerol	64.0	30.0	34.0

3 Results and analysis

3.1 Asphalt Flow Characterization

Since the rotor and rotation speed of the cloth viscometer have little effect on the viscosity of the asphalt, the corresponding shear stress can be obtained by changing the rotation speed of the viscometer rotor, and the antiflow deformation ability of the asphalt at different shear rates can be described phenomenologically. It is generally believed that for most quasi-Newtonian fluids, there is an exponential function relationship between the shear force S and the shear rate D:

$$K = \mathrm{d}\gamma / \mathrm{d}t = a \cdot S^{M} \tag{1}$$

Where, a and M are material parameters, where M can characterize the dependence of shear stress on shear rate. Therefore, equation (1) can be rewritten as:

$$K = M \cdot D^{N} \tag{2}$$

Where, N is called the rheological index of the fluid, which is used to describe the flow degree of the fluid. When N is closer to 1.0, the fluid is closer to the Newtonian fluid state.

The relationship between shear rate and shear stress of asphalt under different aging degrees was fitted by equation (2), and the results are shown in Figure 1.

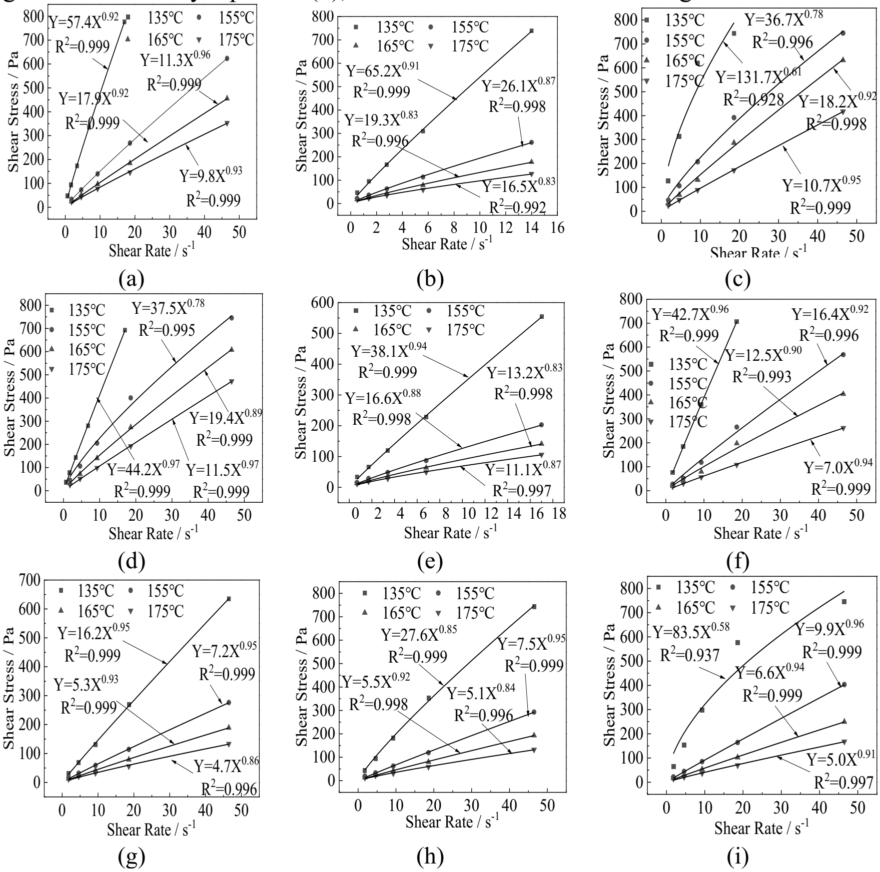

Fig. 1. Relationship between shear rate and shear stress of asphalt at different temperatures: (a) OA of FHVMA; (b) RTOFT of FHVMA; (c) PAV of FHVMA; (d) OA of CHVMA; (e) RTOFT of CHVMA; (f) PAV of CHVMA; (g) OA of SBS; (h) RTOFT of SBS; (i) PAV of SBS.

As can be seen from Figure. 1, the exponential functions can accurately describe the dependence of shear stress on shear rate under three different aging degrees of three different asphalts, with the correlation coefficients reaching a maximum of 0.999 and a minimum of 0.937, which are overall greater than 0.9, indicating that the fitting results have good reliability. In order to further quantitatively analyze the flow characteristics of different asphalts under different aging degrees, the M and N values were statistically analyzed, and the results are shown in Figure. 2 and Table 4.

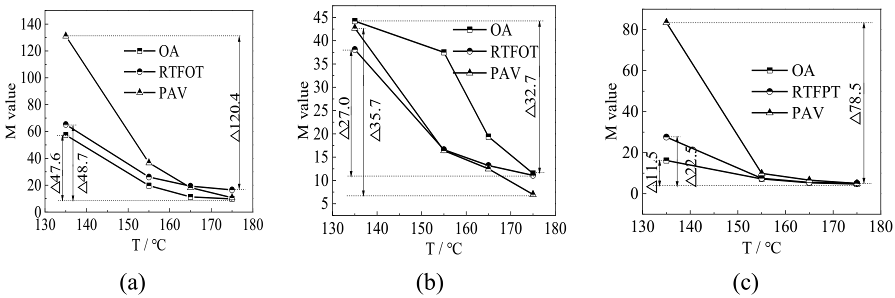

Fig. 2. Statistical results of M value of asphalt: (a) FHVMA; (b) CHVMA; (c) SBS.

Table 4. Statistical results of asphalt rheological index N.

Asphalt Tpye	T(°C)	OA	RTFOT	PAV
FHVMA	135	0.92	0.91	0.61
	155	0.92	0.87	0.78
	165	0.96	0.83	0.92
	175	0.93	0.83	0.95
Average		0.93	0.86	0.82
CHVMA	135	0.97	0.94	0.96
	155	0.78	0.88	0.92
	165	0.89	0.83	0.9
	175	0.97	0.87	0.94
Average		0.90	0.88	0.93
SBS	135	0.95	0.85	0.58
	155	0.95	0.95	0.96
	165	0.93	0.92	0.94
	175	0.86	0.84	0.91
Average		0.92	0.89	0.85

As observed in Figure. 2, with the increase of temperature, the M values of FHVMA, CHVMA, and SBS-modified asphalts basically show a decreasing trend whether in OA, RTFOT, or PAV aging state, which indicates that the increase of temperature reduces the dependence of asphalt shear stress on shear rate. Meanwhile, the decreases of different asphalts at different aging levels are different, such as 120.4, 35.7, and 78.5 for FHVMA, CHVMA, and SBS, respectively, at the PAV aging level, which is supposed to be related to the type of asphalt. Except for CHVMA, the M-values of both FHVMA and SBS asphalts show an overall increasing trend with the aging degree, demonstrating that aging increases the sensitivity of shear stress to shear rate for these two types of asphalts.

From Table 4, it can be concluded that with the increase in temperature, the N values of the three kinds of asphalt without aging did not show a clear pattern of change. It is mainly manifested in the N value of FHVMA asphalt increased first and then decreased, the N values of CHVMA asphalt decreased first and then increased, while SBS-modified asphalt gradually decreased. This indicated that there was a certain volatility in the fluidity index of asphalt in the test temperature range of 135℃~175℃. In order to visually analyze the effect of different aging degrees on asphalt fluidity, the N values at different temperatures were averaged. By comparing the average value of unaged asphalt, it was found that the order of N value was: FHVMA>SBS>CHVMA, which means that FHVMA asphalt has better workability in the mixing, paving and rolling stages of asphalt mixtures. CHVMA asphalt has the smallest N value, which means that CHVMA has the worst fluidity. This was because CHVMA was compounded from SBS-modified asphalt and high viscosity agent. SBS-modified asphalt is inherently high viscosity, and high viscosity agent usually contains more thermoplastic rubber and plasticizing resins or other plasticizing ingredients, when the two are fused, the asphalt will become viscous, and the fluidity is reduced. It was also observed that the N value of both FHVMA and SBS asphalts decreased gradually with aging, for example, the N value of FHVMA asphalt decreased by 7.7% and 12.6% after RTFOT and PAV aging, respectively, and those of SBS-modified asphalt decreased by 3.5% and 8.1%, respectively, in comparison with those of the as-existing asphalts, which was consistent with the trend of the M value. However, the N value of CHVMA asphalt decreased and then increased. In summary, it can be concluded that thermo-oxidative aging affects the Newtonian fluid state of asphalt, but the degree and trend of the effect are related to the type of asphalt. It can also be seen that the N values of both FHVMA and SBS asphalts decreased gradually with the aging degree, for example, compared with the as-received asphalts, the N values of FHVMA asphalt decreased by 7.7% and 12.6% after RTFOT and PAV aging, respectively, while those of SBS-modified asphalt decreased by 3.5% and 8.1%, which was consistent with the trend of the M values. However, the N value of CHVMA asphalt decreased and then increased. In summary, it can be concluded that thermo-oxidative aging affects the Newtonian fluid state of asphalt, but the degree and trend of the effect are related to the type of asphalt.

3.2 Adhesion analysis of asphalt to aggregates

The water stability of asphalt mixture mainly depends on the adhesion between asphalt and aggregate. This adhesion can be expressed by the adhesion work W_a. The larger W_a is, the stronger the adhesion between asphalt and aggregate is. The values of asphalt and limestone before and after thermo-oxidative aging were calculated according to the equations in the literature [14], and the results are presented in Figure 3.

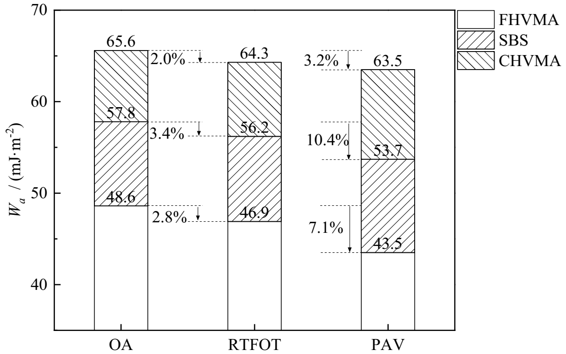

Fig. 3. Adhesion work of asphalt at different aging degrees.

From Figure. 3, it is noticed that all values between asphalt and limestone decreased after thermo-oxidative aging, which illustrates that thermo-oxidative aging weakens the interfacial adhesion between asphalt and limestone. This is since after thermo-oxidative aging, the light groups in the bitumen will volatilize, the proportion of heavy components will increase, and the chemical bonds in the SBS modifier and the high viscosity agent will be destroyed to some extent through thermal decomposition, which deteriorates the wetting properties of the bitumen molecules and affects the adhesion between the bitumen and the limestone. Further analysis shows that FHVMA-limestone adhesion is stronger than CHVMA-limestone and SBS-limestone adhesion before and after aging, implying that the asphalt compounded with SBS modifier has the best adhesion properties to the aggregate. This was attributed to the fact that the high binder significantly increased the resins content in the SBS-modified bitumen and reduced the proportion of asphaltenes. Meanwhile, the SBS modification itself has a reticulated structure, and the addition of the high binder produces cross-linking between the high binder and SBS modifier to form a more stable structural body, which makes the adhesion of FHVMA-limestone higher than that of CHVMA-limestone and SBS-limestone.

3.3 Correlation analysis of asphalt fluidity and adhesion

In this paper, the grey correlation analysis theory is introduced to analyze the correlation between fluidity and adhesion of asphalt before and after aging. Although the gray correlation theory has achieved a wide range of applications in different research fields, not many studies have been conducted on the fluidity and adhesion of asphalt before and after thermo-oxidative aging. The basic steps of gray correlation are as follows:

(i) Identification of reference and comparison sequences and variable indicators.

$$X_0(i)=\left\{x_0^1,\ x_0^2,\cdots,\ x_0^k\right\},\ i=1,2,\cdots,\ k \tag{3}$$

$$X_j(i)=\left\{x_j^1,\ x_j^2,\cdots,\ x_j^k\right\},\ i=1,2,\cdots k,\ j=1,2,\cdots,m \tag{4}$$

Where, $X_0(i)$ is the reference sequence, $X_j(i)$ is the comparison sequence, k is the number of factors in the constituent reference and comparison sequences of the sequence, and m is the number of comparison sequences.

(ii) Dimensionless data processing.

$$X_0(i)=\left\{\frac{x_0^1}{\overline{X_0(i)}},\frac{x_0^2}{\overline{X_0(i)}},\cdots,\frac{x_0^k}{\overline{X_0(i)}}\right\},\ i=1,2,\cdots,\ k \tag{5}$$

$$X_j(i)=\left\{\frac{x_j^1}{\overline{X_j(i)}},\frac{x_j^2}{\overline{X_j(i)}},\cdots,\frac{x_j^k}{\overline{X_j(i)}}\right\},=1,2,\cdots k,\ j=1,2,\cdots,m \tag{6}$$

Where, $\overline{X_0(i)}$ and $\overline{X_j(i)}$ are the mean values of the reference and comparison sequences, respectively.

(iii) Calculate the grey correlation coefficient.

Denote the reference sequence after quantization as $\{X_0(t)\}$ (t is the time) and the comparison sequence after dimensionless as $\{X_j(t)\}$, then the grey correlation coefficient $W_{oj}(i)$ between $\{X_0(t)\}$ and $\{X_j(t)\}$ at the moment t = i can be calculated by equation (7):

$$W_{oj}(i) = \left| \frac{\Delta_{\min} + \lambda\Delta_{\max}}{\Delta_{oj}(i) + \lambda\Delta_{\max}} \right| \tag{7}$$

(iv) Calculate the correlation.

The grey correlation R_{0j} is calculated as follows:

$$R_{0j} = \frac{1}{m}\sum\nolimits_{j=1}^{m} W_{0j}(i) \tag{8}$$

Where, R_{0j} is the correlation between the reference sequence X_0(i) and comparison sequence $X_j(i)$.

Among them, the value of M value and N of asphalt can represent asphalt fluidity, and the adhesion work W_a represents asphalt adhesion. Taking asphalt fluidity as the comparison sequence and asphalt adhesion as the reference sequence, the correlation degree is calculated. The results are shown in Table 5.

Table 5. Correlation results.

Temperature (°C)		135	155	165	175
Comparison sequence	M	0.77	0.62	0.67	0.73
	N	0.79	0.68	0.81	0.76

It can be observed that the correlation between W_a and the N value of 165°C is the highest at 0.81, and the lowest value is with the M value of 155°C at 0.62. Thus the overall correlation of the asphalt M and N values with W_a fluctuates from 0.6 to 0.85. From the gray correlation theory, when the correlation between two parameters is greater than 0.6, it indicates that these two parameters are significantly correlated [15]. It can be concluded that the M and N values of asphalt are significantly correlated with A, which means that the fluidity of asphalt is closely related to the adhesion of asphalt. In practical engineering, asphalt M and N values are usually easier to test and obtain, while the value will pay more test cost. The research results of this paper can show that the use of asphalt flowability index can predict the adhesion of asphalt, and then guide the design and construction of asphalt mixtures and other related work.

4 Conclusions

(1) Thermo-oxidative aging significantly affects the Newtonian fluid state of asphalt, and the trend and degree of influence vary with the type of asphalt. With the deepening of aging, the rheological index of FHVMA and SBS modified asphalt showed a decreasing trend, while the rheological index of CHVMA asphalt decreased first and then increased.

(2) Aging weakens the adhesion between asphalt and aggregate, but the combination of high viscosity agent and SBS modifier can effectively slow down this aging effect. The high-viscosity / SBS compound modified asphalt is recommended as an open-graded anti-skid wear layer pavement material.

(3) There is a good correlation between the fluidity of asphalt and the adhesion of asphalt. The adhesion ability of asphalt can be predicted by using the fluidity of asphalt, so as to guide the design and construction of asphalt mixture.

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