Understanding the Rejuvenation Effect of Different Rejuvenators from Rheological Experiment

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ABSTRACT

The objective of this paper is to investigate the possibility of various self-developed compound rejuvenators on improving the physical, mechanical and aging properties of artificial-aged and RAP-aged bitumen, asphalt mortar as well as asphalt mixture. The results revealed that all kinds of compound rejuvenators could restore the physical properties, workability, temperature sensitivity, activation energy viscoelastic properties, performance grade and aging resistance of artificial-aged bitumen to the virgin binder level. From the perspective of balancing the high-and-low temperature properties and workability, the rejuvenators J4 and X3 are recommended. The restoration capacity of RJ-based rejuvenator on the low-temperature stiffness and high-temperature grade of aged bitumen is more significant. Moreover, the RX-based rejuvenator is beneficial to enhance the viscidity, flexibility, low-temperature grade and compatibility of rejuvenated binder even better than virgin bitumen. Compared to rejuvenator J4, the rejuvenator X3 exhibits better efficacy to improve the malleability, workability and aging resistance of RAP-aged binder. The compatibility between the RAP-aged bitumen and rejuvenator J4 is slightly superior to that of rejuvenator X3. Further, with the increase of curing temperature and time, the number and volume of clumps both increase dramatically. The aged bitumen is reactivated and its adhesion properties are strengthened.

Keywords: Rejuvenation efficiency; rejuvenated bitumen; Rejuvenators; Rheological properties

1. Introduction

Currently, asphalt pavement is one of the most important infrastructures because of its flexibility and versatility characteristics. However, due to the increasing traffic loading and environmental oxidation aging, the service life of asphalt roads is limited, which is generally designed in the region of 8-15 years [1, 2]. More distresses of asphalt pavement would occur because of the weakened mechanical performance, including the rutting, cracking, raveling, etc. [3]. Hence, the maintenance and reconstruction processes are necessary to repair the damaged asphalt roads, which would create large amounts of reclaimed asphalt pavement (RAP) materials.

The conventional method of addressing these waste materials would result in the environmental pollution and waste of resources [4]. At the same time, the reconstruction of asphalt roads would consume lots of bitumen and aggregates [5, 6]. From the viewpoint to environment protection, cost and resource-saving, the recycling technology of asphalt pavement are attributed to the attention of global researchers.

It was reported that the incorporation of RAP materials can not only reduce the consumption of binder and aggregate, but also improve the dynamic modulus and rutting resistance of asphalt mixture [7-9]. Nevertheless, the aged bitumen in RAP materials strongly weakened the low-temperature cracking, fatigue resistance and workability [10-13]. The aging process of bitumen would lead to the mutual transformation and proportion imbalance between different chemical components, which mainly shows the reduction of light-weight aromatic fraction and increase of heavy-weight asphaltene molecules [14]. To balance the distribution of the chemical fractions, the rejuvenators with characteristics of low-viscosity and light-molecular weight are developed and incorporated into RAP materials to reverse the inferior properties of aged bitumen. In general, the rejuvenator has the functions in the aspects of (1) coordinating the chemical fractions of aged bitumen; (2) adjusting the viscosity and improve the workability of aged bitumen; (3) enhancing the solubility and dispersing the asphaltene molecules in maltene matrix; (4) activating the molecular mobility and accelerating the mixing of aged bitumen with virgin binder and aggregates [15].

Different types of rejuvenators are proposed to restore the properties of aged bitumen, including cooking oil, engine oil, naphthenic oil, aromatic oil, bio-based extraction, and etc. [16-19]. Guo et al. [20] found that the rejuvenation influence of warm-mix rejuvenator on the complex modulus of aged binder was more significant, followed by the commercial rejuvenator, and the aromatic oil reduced it less obviously. In addition, Suo et al. [21] evaluated the aging behavior of vegetable oil (corn oil and soybean oil) rejuvenated bitumen and asphalt mixture, and proved that the rejuvenator type and dosage showed significant influence on the rheological properties of the rejuvenated binder. Moreover, the vegetable oil rejuvenated bitumen exhibited sufficient anti-aging properties, which was associated with the existed unsaturated carbon chains in vegetable oil. Recently, Bajaj et al. [22] studied the rejuvenation efficiency and mechanism of seven types of rejuvenators with rheological and chemical tools. According to the experimental results, the recycling agents were classified into three groups, including the softeners, replenishers and emulsifiers. Some rejuvenators (like the paraffinic oil) were only on a physical level, while others (such as the aromatic extracts and bio-oil) interacted with aged bitumen at a chemically level. Regarding the optimum rejuvenator content, Behnood [23] proposed a balance performance design method, in which the maximum rejuvenator dosage could be determined to satisfy the high-temperature performance, while the minimum concentration was related to the effective restoration of the intermediate and low-temperature properties of bitumen binder and asphalt mixture.

2. Materials and methods

In this study, the virgin bitumen with the PEN-70 grade was used to prepare the artificial aged bitumen and compound rejuvenators. The physical properties and chemical components of virgin

bitumen are listed in **Table 1**. Moreover, two pure rejuvenators, vacuum distilled oil (rejuvenator J) and aromatic oil (rejuvenator X), were both obtained from Jiangsu Province, China. **Table 2** displays the conventional properties of two rejuvenators, including the density, viscosity, saturate-aromatic-resin-asphaltene (SARA) fractions, mass loss and viscosity ratio after the Rolling Thin Film Oven (RTFO) short-term aging procedure [25].

Table 1 Physical properties and chemical components of virgin bitumen

Properties	Measured Value	Test standards
25 ℃ Penetration (0.1mm)	67	ASTM D5 [26]
Softening point (℃)	48.2	ASTM D36 [27]
10℃ Ductility (cm)	84.8	ASTM D113 [28]
15℃ Ductility (cm)	>150	ASTM D4124 [29]
Saturate (wt%)	13.33	
Aromatic (wt%)	17.36	
Resin (wt%)	39.71	
Asphaltene (wt%)	29.60	

Table 2 Conventional properties of pure rejuvenators

Properties		Rejuvenator J	Rejuvenator X
20℃ Density (kg·m-3)		0.985	0.997
60℃ Viscosity (Pa·s)		0.137	0.219
SARA analysis	Saturate S (wt/%)	73.24	56.84
	Aromatic A (wt/%)	22.85	36.13
	Resin R (wt/%)	3.64	6.96
	Asphaltene As (wt/%)	0.27	0.07
RTFOT aging	Mass change (wt/%)	-0.6	-0.4
	60℃ Viscosity-ratio	1.01	1.25

The physical properties, including penetration, softening point, ductility and viscosity, of rejuvenated artificial-aged binders are measured and compared with virgin bitumen to evaluate the rejuvenation efficiency of different compound rejuvenators. The corresponding results are listed in **Table 3**. It can be found that the basic properties of all rejuvenated binders are close to that of virgin bitumen, which fulfills the performance requirements of AH-70 bitumen according to JTB F40-2004 standard. Thus, all kinds of compound rejuvenators can restore the physical properties of artificial-aged bitumen to the virgin binder level. The difference in conventional properties of various rejuvenated binders is also observed. The rejuvenated bitumen with smaller 60℃ viscosity would show the higher ductility and lower softening point. Overall, although their penetration grades are the same, the high-and-low temperature properties and workability of various rejuvenated artificial-aged binders are significantly different, especially for the ductility and viscosity indicators.

Table 3 Physical properties of virgin and rejuvenated artificial-aged bitumen

Bitumen samples	J1RAB	J2RAB	J3RAB	J4RAB	J5RAB	J6RAB
Rejuvenator dosage (wt%)	24	27	36	43	54	68
25℃ penetration (0.1mm)	73	63	65	66	69	70
Softening point (℃)	50.5	51.3	52.2	50.8	49.7	48.1
15℃ ductility (cm)	103.3	109.9	103.3	113	115.6	128.5
60℃ viscosity (Pa·s)	437.5	335.8	591.3	501.0	420.5	310.3
Bitumen samples	X1RAB	X2RAB	X3RAB	X4RAB	X5RAB	VB
Rejuvenator dosage (wt%)	26	36	43	60	69	-
25℃ penetration (0.1mm)	67	68	68	67	69	67
Softening point (℃)	48.5	50.3	49.3	48.6	47.7	48.2
15℃ ductility (cm)	109.8	108.2	126.6	150	150	150
60℃ viscosity (Pa·s)	378.3	397.5	343.1	298.1	276.5	307.0

In addition, for RJ-based rejuvenator, the rejuvenated bitumen with the rejuvenator J4 presents the lowest softening point and viscosity, while its ductility value is the largest. The softening point and viscosity of J3 rejuvenated bitumen are the highest, and the corresponding ductility value is the lowest. Meanwhile, for RX-based rejuvenator, the X2RAB binder with the highest viscosity has the highest softening point and lowest ductility, while the X5RAB binder with the lowest viscosity shows the lowest softening point and largest ductility. Therefore, from the perspective of balancing the high-and-low temperature properties and workability, the rejuvenators J4 and X3 are recommended for the rejuvenation of artificial-aged bitumen. Furthermore, the rejuvenation efficiency of compound rejuvenators on the physical properties of rejuvenated binders significantly depends on the type of pure rejuvenator. Compared to the RJ-based rejuvenators, the rejuvenated binders with RX-based rejuvenators exhibit higher ductility and lower softening point and viscosity. The rejuvenated bitumen with RX-based rejuvenator presents a better low-temperature cracking resistance and workability.

3. Results and discussion

3.1. Viscosity and activation energy

The influence of compound rejuvenators on the viscosity and temperature sensitivity of rejuvenated bitumen is evaluated, and the results are displayed in **Fig.1**. As expected, with the temperature increase, the viscosity values of all samples decrease dramatically, and there is a linearly increasing relationship between the logarithm value of viscosity and the reciprocal of temperature. The aged bitumen shows the largest viscosity, which is associated with the limited free volume and molecular mobility. The addition of a rejuvenator remarkably reduces the viscosity of artificial-aged bitumen. Interestingly, the viscosity values of all rejuvenated binders are all lower than that of virgin bitumen. It implies that the rejuvenated binder with the same penetration grade

of virgin bitumen has a lower viscosity and better workability.

In addition, the viscosity-temperature correlation equations of the virgin, aged and rejuvenated binders are also shown in **Fig.1**, and the slope values in correlation equations represent the temperature sensitivity. It can be found that the aged bitumen shows the highest slope value, indicating that its temperature susceptibility is strongest. Meanwhile, virgin bitumen has the lowest slope value and minimal temperature sensitivity. The slope values of rejuvenated binders are all close but larger than that of virgin bitumen, denoting that the addition of compound rejuvenator could restore the workability and temperature sensitivity of aged bitumen to the virgin binder level simultaneously. Further, the compound rejuvenator type affects the viscosity and temperature sensitivity of rejuvenated binders, especially for the RJ-based rejuvenators. As the viscosity-grade of the compound rejuvenator increasing, the viscosity of the rejuvenated binder decreases.

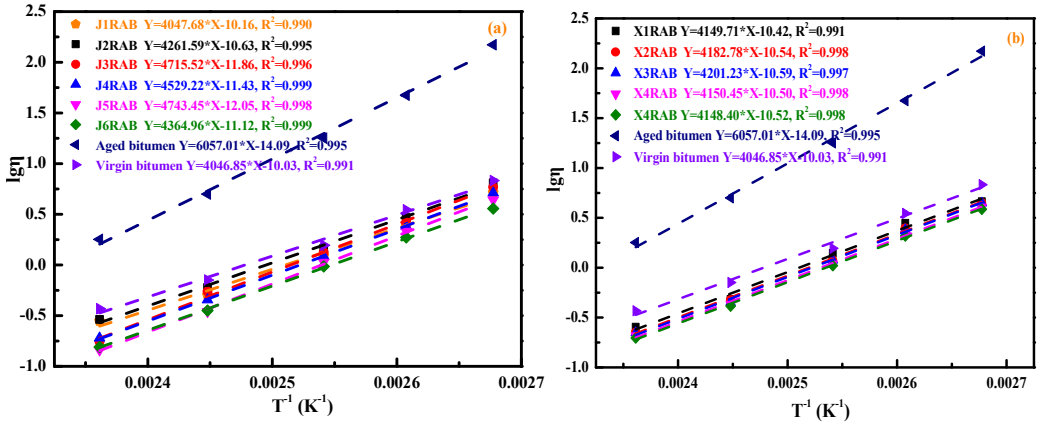

Fig.1. The correlation of viscosity and temperature for aged and rejuvenated bitumen

As mentioned before, the increased temperature would accelerate the reduction of bitumen viscosity. From the viewpoint of the atomic level, the molecular thermal movement is promoted as the temperature rising. It would result in the increase of intermolecular distance and decrease of molecular interaction. That's why the viscosity of the whole bitumen system decreases at high temperatures []. The Arrhenius equation was used to quantitively assess the viscosity-temperature correlation, which is as follows:

$$\lg(\eta(T)) = \frac{E_\eta}{2.303RT} + \lg(A) \quad (2)$$

where η(T) refers to the bitumen viscosity at temperature T, Pa·s; R is the gas constant, 8.314 J/(mol·K); E_η represents the viscous activation energy, kJ/mol; and A is the pre-reference parameter.

Table 4 lists the viscous activation energy of virgin, aged and rejuvenated binders. It can be seen that the aging process increases the activation energy from 77.49 to 115.98 kJ/mol. The addition of compound rejuvenator significantly decreases the activation energy of aged binder closely to the virgin binder level. It is interesting to note that most of the rejuvenated binders present a higher activation energy than virgin bitumen. It implies that these compound rejuvenators could not restore the activation energy completely. Additionally, compared to the RJ-based rejuvenators, the RX-based rejuvenators can restore the activation energy of aged bitumen to the virgin binder level. Thus, considering the reversible degree of activation energy, the RX-based compound

rejuvenators are better than the RJ-based ones.

Table 4 Viscous activation energy of aged and rejuvenated binders

Samples	VB	J1RAB	J2RAB	J3RAB	J4RAB	J5RAB	J6RAB
E_η (kJ/mol)	77.49	77.50	81.59	90.29	86.72	90.83	83.58
Samples	AB	X1RAB	X2RAB	X3RAB	X4RAB	X5RAB	-
E_η (kJ/mol)	115.98	79.46	80.09	80.45	79.47	79.43	-

3.2. Complex modulus and phase angle

The complex modulus and phase angle of the virgin, aged and rejuvenated binders are characterized with frequency sweep test, and the results are illustrated in **Fig.2.** It is expected that with the loading frequency increases, the complex modulus and phase angle values of all samples increase and decrease, respectively. It indicates that the bitumen shows more elastic characteristics under the high vehicle speed condition. The aged binder has the highest G* and lowest δ values due to the largest stiffness and elasticity. Adding the compound rejuvenators can distinctly reduce the complex modulus and increase the phase angle of the aged binder, which is close to the virgin bitumen level. It means that the compound rejuvenators can significantly restore the viscoelastic properties of the aged binder.

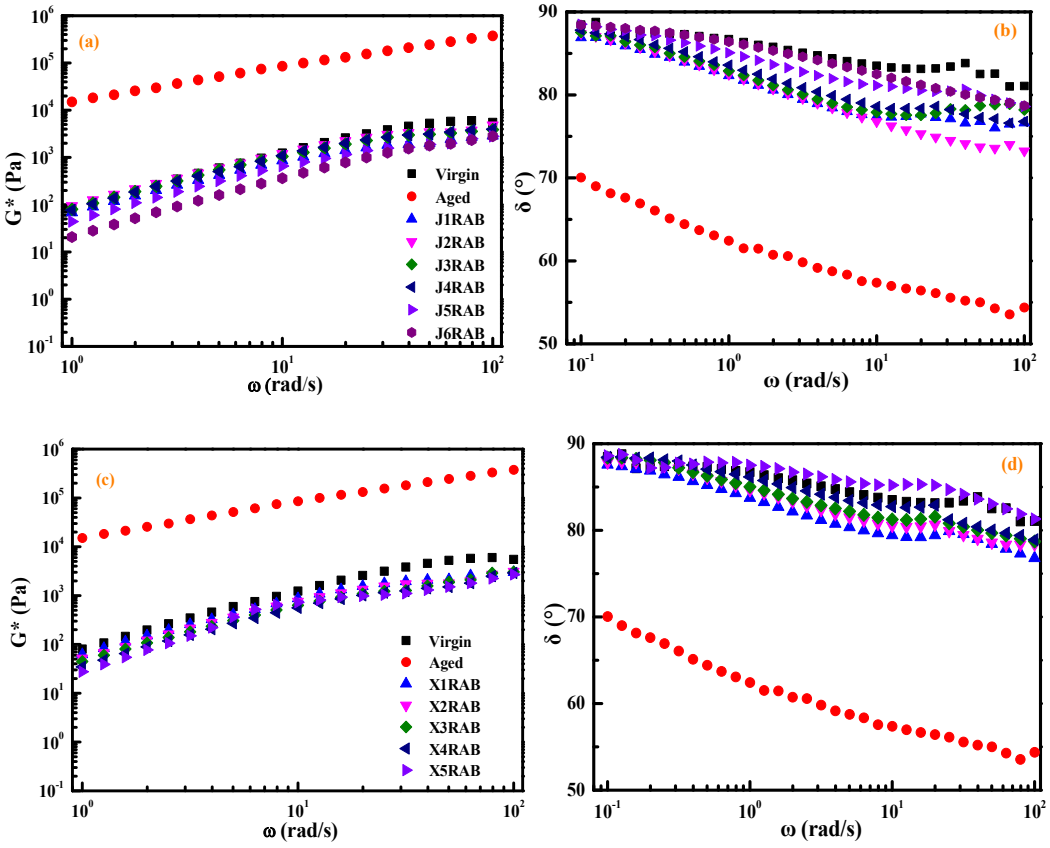

Fig.2. The complex modulus and phase angle of virgin, aged and rejuvenated bitumen

Interestingly, the complex modulus and phase angle of RJ-based rejuvenated bitumen are both lower than that of the virgin binder. It implies that although the RJ-based compound rejuvenators can revert the complex modulus of aged bitumen, but still have limitations regarding the complete restoration of phase angle. In other words, the elastic proportion in the rejuvenated binder is higher than that in the virgin binder. Hence, the viscidity of aged bitumen cannot be restored to the virgin binder level with these different types of compound rejuvenators. However, the phase angle of X5RAB binder is higher than virgin bitumen, which illustrates that the RX-based rejuvenator is beneficial to improve the viscidity of the aged binder even better than virgin bitumen. The rheological properties of rejuvenated binders remarkably depend on the rejuvenator type, especially for the RJ-based rejuvenated binders. It can be seen that with the increase of pure rejuvenator dosage in compound rejuvenators, the complex modulus of rejuvenated bitumen decreases gradually, which is due to the softer characteristic of pure rejuvenator than that of virgin binder.

3.3. Low temperature performance

In this study, the bending beam rheometer (BBR) tests were conducted to estimate the low-temperature cracking resistance of virgin and various rejuvenated artificial-aged binders. **Fig.3** illustrates the stiffness S-values of bitumen binders at different temperatures of -6, -12 and -18℃, respectively. As expected, as the temperature dropping down, the s-values of all bitumen increase dramatically. The high stiffness of bitumen at low temperatures is related to the reduction of free volume and enhancement of molecular interactions. Regarding the RJ-based rejuvenated binders, with the viscosity grade of compound rejuvenator rises, the stiffness of the rejuvenated binder increases gradually. It indicates that the RJ-based rejuvenated binder would show the better low-temperature flexibility with the pure rejuvenator dosage increases. For RX-based rejuvenated binders, the difference in stiffness values is very small, especially when the testing temperature is lower than -18℃.

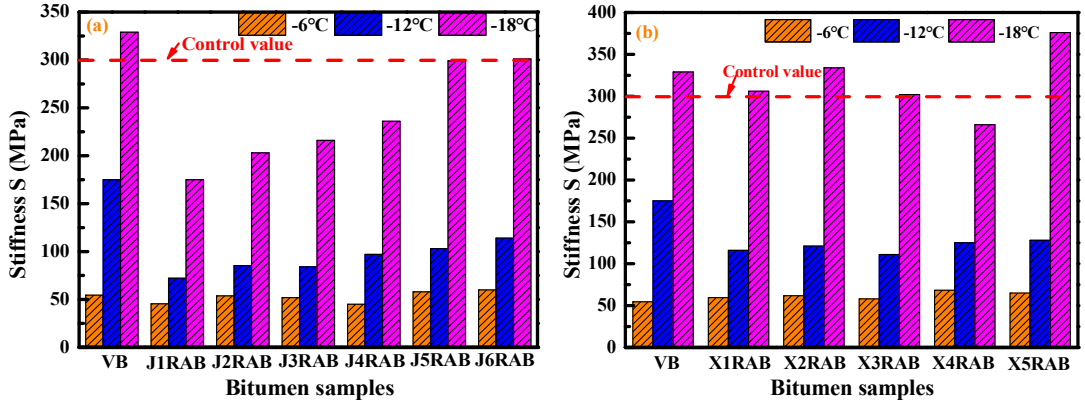

Fig.3. The stiffness values of rejuvenated bitumen at different temperatures

In addition, it can be found that the stiffness values of rejuvenated binders are close or even lower than that of virgin bitumen. Hence, these compound rejuvenators can significantly restore and improve the low-temperature cracking resistance of aged bitumen to the virgin binder level. According to the SHARP standard, the control stiffness point is 300MPa, and the bitumen would have the cracking potential when its S-value exceeds the value. From **Fig.3**, the stiffness values of

RJ-based rejuvenated binders at -18℃ are all lower than 300MPa, which of virgin binder is higher than the control value. It means that the RJ-based rejuvenators have a great influence on decreasing the stiffness of aged bitumen. On the other hands, the RX-based rejuvenated binders show a larger stiffness value than the control value at -18℃ except for the X4RAB binder. Overall, the restoration capacity of RJ-based rejuvenators on the low-temperature stiffness of aged bitumen is more obvious than that of RX-based rejuvenators.

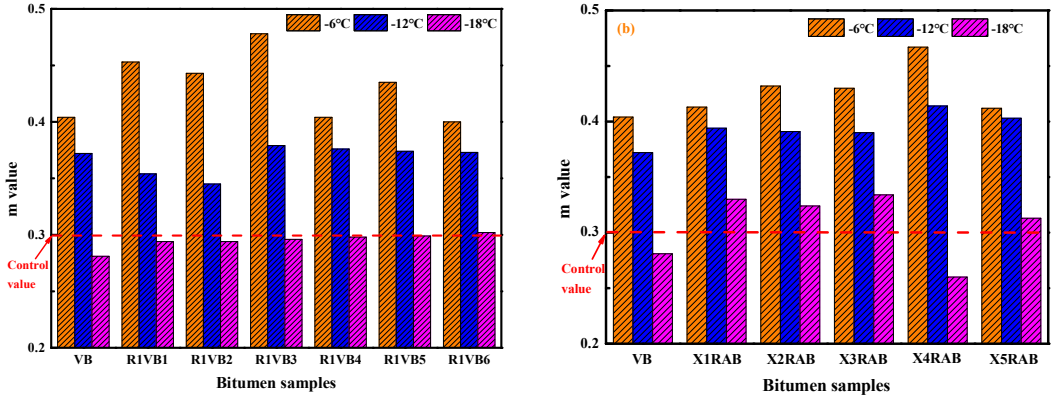

Fig.4. The m-value of rejuvenated bitumen at different temperatures

The creep rate m-values of virgin and rejuvenated binders are displayed in **Fig.4.** It can be seen that the m-value of bitumen reduces as the temperature decreasing. The stiff bitumen molecules would need more time to accomplish the creep stage with the external force at low temperature. Compared with the virgin binder, the m-values of most rejuvenated binders are larger regardless of the testing temperature. The RJ- and RX-based compound rejuvenators can significantly retrieve and improve the flexibility of aged binder better than virgin one. Interestingly, the m-values of RX-based rejuvenated binders are higher than that of RJ-based rejuvenated bitumen, which is contrary to the previous stiffness results. In other words, although the RJ-based rejuvenators can remarkably reduce the stiffness value, their effects on improving the m-value and flexibility of rejuvenated binders are weaker than the RX-based rejuvenators. Moreover, the m-values of virgin and rejuvenated binders are all higher than the control value of 0.3, while the m-value of virgin and all RJ-based rejuvenated bitumen are all lower than the control point at -18℃. However, the m-values of all RX-based rejuvenated binders at -18℃ all meet the control requirement except for the X4RAB binder. Considering the standard requirements of S and m-value, the compound rejuvenators of J4 and X3 are recommended.

3.4. PG grade

The low-and-high temperature performance grades of virgin and various rejuvenated binders are illustrated in **Fig.5.** The PG-grades of all rejuvenated binders are close to that of virgin bitumen, which indicates that the PG grade of the aged binder could be restored to the virgin bitumen level with the different types of RJ- and RX-based compound rejuvenators. The difference in the PG-grade of rejuvenated binders can also be observed, especially for the RX-based ones. Compared to the virgin bitumen, all rejuvenated binders present a lower low-temperature grade, especially for the X4RAB. At the same time, most the RJ-based rejuvenated binders possess the

larger high-temperature grade except for the J5RAB and J6RAB binders, while all RX-based rejuvenated binders show a smaller high-temperature grade than that of virgin bitumen. Hence, the RJ-based rejuvenated binders exhibit superior high-temperature grade, while the RX-based rejuvenators are more beneficial to improve the low-temperature grade of the aged binder. In conclusion, to ensure the high-and-low temperatures grade of rejuvenated binder simultaneously, the compound rejuvenators J2, J4, X1 and X3 are recommended.

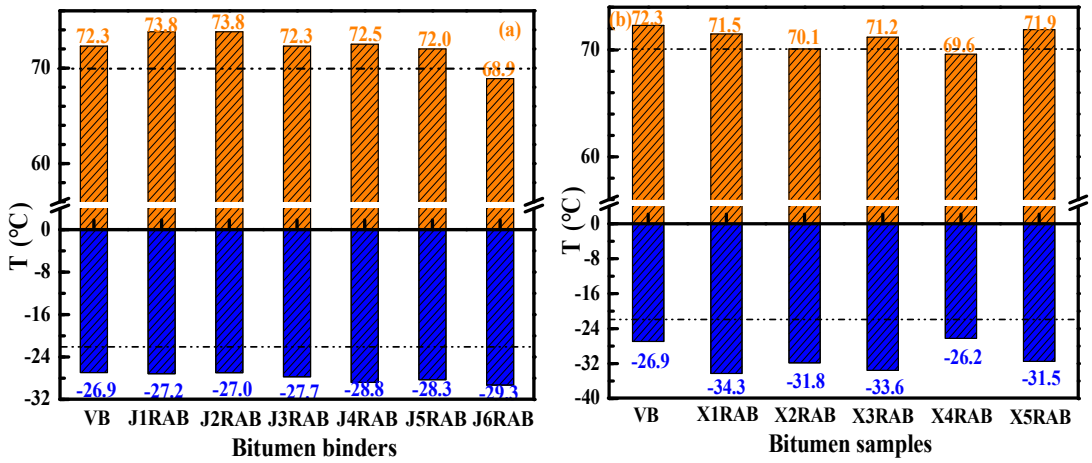

Fig.5. The Performance grade (PG) of rejuvenated binders

3.5. Compatibility

It was reported that there is a difference between the measured and predicted viscosity values of rejuvenated bitumen due to the incomplete compatibility between rejuvenator and aged binder. The difference parameter is applied to evaluate the compatibility of rejuvenated bitumen, and the predicted viscosity can be calculated as follows:

$$\lg\mu = x_1\lg\mu_1 + x_2\lg\mu_2 + x_1x_2G_{12} \quad (3)$$

where μ, μ_1 and μ_2 refer to the measured viscosity of rejuvenated bitumen, aged bitumen and rejuvenator at the same temperature, respectively; x_1 and x_2 are the mass fractions of aged bitumen and rejuvenator; G_{12} is the correction factor, which represents the difference between the measured and predicted viscosity of rejuvenated binder. The larger the G_{12} value is, the worse the compatibility between the rejuvenator and aged bitumen.

Table 5 lists the 60°C viscosity and G_{12} parameter values of RJ-based rejuvenated binders. As the viscosity of the rejuvenator decreases, the absolute G_{12} value decreases dramatically. The J1RAB binder shows the highest absolute G_{12} value of 2.807, while the J6RAB binder has the lowest value of 0.417. The compound rejuvenator with a high pure rejuvenator dosage and low viscosity would show better compatibility with the artificial-aged bitumen. Moreover, **Table 6** shows the G_{12} parameter of RX-based rejuvenated binders. Similarly, the X1RAB and X5RAB has the largest and smallest G_{12} value, respectively. Compared to RJ-based rejuvenators, the absolute G_{12} values of RX-based rejuvenated binders are higher, but the difference is not significant. Therein,

the RJ-based rejuvenators show better compatibility with aged bitumen than RX-based rejuvenators.

Table 5 G_{12} parameter of rejuvenated binders with RJ-based rejuvenators

Rejuvenated binders	Complex rejuvenators			Aged bitumen			Rejuvenated binders		
	μ1 (Pa·s)	lgμ1	x1 (wt%)	μ2 (Pa·s)	lgμ2	x2 (wt%)	μ (Pa·s)	lgμ	G12
J1RAB	0.147	-0.83	24	25800	4.41	76	437.5	2.64	-2.807
J2RAB	0.505	-0.29	27			63	335.8	2.52	-1.017
J3RAB	2.570	0.41	36			64	591.3	2.77	-0.865
J4RAB	6.213	0.79	43			57	501.0	2.69	-0.636
J5RAB	21.38	1.33	54			46	420.5	2.62	-0.498
J6RAB	52.71	1.72	68			32	310.3	2.49	-0.417

Table 6 G_{12} parameter of rejuvenated binders with RX-based rejuvenators

Rejuvenated binders	Complex rejuvenator			Aged bitumen			Rejuvenated binders		
	μ_1 (Pa·s)	lgμ_1	x_1 (wt%)	μ_2 (Pa·s)	lgμ_2	x_2 (wt%)	μ_0 (Pa·s)	lgμ_0	G_{12}
X1RAB	0.515	-0.28	26	25800	4.41	74	397.5	2.59	-3.068
X2RAB	2.811	0.44	36			64	378.3	2.57	-1.767
X3RAB	6.463	0.81	43			57	343.1	2.54	-1.337
X4RAB	24.95	1.39	60			40	298.1	2.47	-0.536
X5RAB	53.02	1.72	69			31	276.5	2.44	-0.541

4. Conclusions

This study hierarchically investigated the effects of different compound rejuvenators on the physical, rheological and mechanical properties of artificial-aged and RAP-aged bitumen binder, asphalt mortar as well as asphalt mixture. Some main conclusions are as follows:

(1) All kinds of compound rejuvenators could restore the physical properties, workability, temperature sensitivity and activation energy of artificial-aged bitumen to the virgin binder level. However, although the penetration grades are the same, the high-and-low temperature properties and workability of various rejuvenated artificial-aged binders were different, especially for the parameters of ductility and viscosity. From the perspective of balancing the high-and-low temperature properties and workability, the rejuvenators J4 and X3 were recommended to be used

in the rejuvenation of artificial-aged bitumen.

(2) The compound rejuvenators could restore the viscoelastic properties, performance grade and aging resistance of rejuvenated binder. The restoration capacity of RJ-based rejuvenator on the low-temperature stiffness and high-temperature grade of aged bitumen was more significant, which exhibited better compatibility with aged bitumen. Moreover, the RX-based rejuvenator was beneficial to enhance the viscidity, flexibility, low-temperature grade and aging resistance of rejuvenated binder even better than virgin bitumen.

(3) Two rejuvenators, J4 and X3, could restore the physical and rheological properties of RAP aged binder to the virgin bitumen level. Although the addition of rejuvenator improved the low-temperature cracking and aging resistance properties of aged bitumen, it had an adverse influence on the high-temperature rutting resistance. Compared to rejuvenator J4, the rejuvenator X3 showed better efficacy to improve the malleability, workability and aging resistance of rejuvenated RAP-aged binder. The compatibility between the RAP aged bitumen and rejuvenator J4 was slightly superior to that of rejuvenator X3.

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