REGENERATION PERFORMANCE OF AGED ASPHALT BASED ON RESPONSE SURFACE METHOD

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The influence of various factors on the high and low temperature performance of aged asphalt in RAP mixture was analyzed in this paper. Taking the preheating temperature of aged asphalt and the addition of regenerative agent as the influence factors, rutting factor, stiffness modulus and creep rate as the index, the influence rules of various test factors on different indexes were analyzed to optimize the optimum conditions of asphalt regeneration test and test for verification. The results show that the best regenerative condition of asphalt is 143 ℃ and addition of the regenerative agent is 3.2%; the effect of regenerative agent content on each index is much greater than the preheating temperature of old asphalt. The research results have some guiding significance for the production of hot recycled mixture.

Keywords: RAP mixture, Regeneration test, response surface methodology, rutting factor, low temperature performance

1. Introduction

With the rapid development of economy, the highway industry of China is also developing rapidly. By the end of 2018, the total mileage of national highway has reached 4846500km [1, 2]. However, the early stage of asphalt pavement in China has entered a large maintenance stage. During this process, a large number of waste asphalt mixture appeared. In order to save resources, protect the environment and respond to the green development idea of "13th Five-Year" highway maintenance management development program, the thermal regeneration technology of asphalt pavement is highly valued by domestic and foreign researchers for saving funds, resources, environmental protection and more[4-6]. Bennert and Maher analyzed the durability of recycling pavement and ordinary asphalt pavement. Zhao Yalan from Chang'an University indirectly evaluated the low temperature performance of recycled asphalt mixture by gel liquid chromatography analysis [i]; Xu Zhiyuan et al. used laboratory tests to simulate the aging of asphalt mixture and analyzed sensitivity and correlation of bending fatigue on the aging of asphalt mixture [3]; Chen Zining studied the optimum blending ratio of RAP in the hot recycled mixture based on the rheology

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principle. In terms of the regeneration of aged asphalt, three-index are generally adopted to evaluate the performance of recycled asphalt in China. This evaluation method has some limitations [7-10]. In this regard, the principle of rheology [11-15] is introduced, and the high and low temperature performance of the regenerated asphalt are evaluated by the rutting factor G*/sin δ, the creep stiffness S and the creep rate m respectively. The response surface method [16-18] is used to optimize the analysis of the test results, which will provide some references for the regeneration test of aged asphalt.

2. Response Surface Methodology

The response surface methodology obtains data through reasonable test design and fits the function relation between the test factor and response value by using multiple two regression equation. It is generally used to guide multivariable optimization problems and has been widely used in chemical, biological and mechanical analysis [19-20].

For example, the regression model established for two regression design is:

$$\hat{y} = \mathrm{a} + \sum_{\mathrm{j=1}}^{\mathrm{m}} b_j x_j + \sum_{\mathrm{k<j}} b_{kj} x_k x_j + \sum_{\mathrm{j=1}}^{\mathrm{m}} b_{jj} {x_j}^2, k = 1,2,...m-1(j \neq k) \quad (1)$$

Or the regression model established in the standard variable space

$$\hat{y} = a' + \sum_{j1}^{m} b'_j z_j + \sum_{k\,j} b'_{kj} z_k z_j + \sum_{j1}^{m} b'_{jj} {z_j}^2, k = 1,2,...m-1(j \neq k) \quad (2)$$

In order to find x_j or Z_j（j=1,2,…m）making prediction value $\hat{y}$ be optimal, according to the extremum necessary condition of, $\frac{\partial y}{\partial x_j} = 0$，$\frac{\partial y}{\partial x_j} = 0(j = 1,2,...m)$ can solve the possible extreme point of $\hat{y}$, which is generally called the "stable point". A stable point is a possible extreme point of a multivariate function, and the predicted value $\hat{y}$ at the point may take the maximum or minimum value; when the stability point $\hat{y}$ cannot get the maximum and minimum, the stable point is called saddle point. In response surface methodology, Central composite design (CCD) is a test design method developed based on full factorial two-level test and partial experimental design. A level test is added to the two-level test to evaluate the nonlinear relationship between the index and factor time. It is often used to test the nonlinear effects of factors on indicators. In this paper, CCD is used to evaluate the regeneration performance of regenerated agents and optimize the best regeneration conditions.

3. Evaluation Index of Regenerated Asphalt

At present, penetration, ductility and softening point are used to evaluate the quality of asphalt in China. The United States carried out a large number of experimental studies through SHRP program and obtained the results "Specification for Superpave Asphalt Binder", i.e.“Specification for American performance grading asphalt binder”. This specification puts forward a new method of asphalt mixture design based on the performance of asphalt. This method has a clear viscoelastic mechanics theory basis instead of the present experience method design, such as the Marshall Test method and more. For this, SHRP program developed a Dynamic Shear Rheometer (DSR) to determine the high temperature performance and fatigue performance of asphalt; the Bending Beam Rheometer (BBR) was used to determine the anti cracking performance of asphalt at low temperature. In this regard, according to the local temperature conditions of Lanzhou, the rutting factor G*/sin δ by DSR test at 64℃ was used to evaluate the high temperature performance of recycled asphalt. The stiffness modulus S and creep rate m by BBR test at -12℃ are used to evaluate the low temperature performance of recycled asphalt.

4. Design and Optimization of Regenerated Asphalt Test

4.1 Raw Materials

The regenerative agent used in this paper is domestic regenerator. The aged asphalt is extracted from the marathon road in Lanzhou riverside road through centrifuge separation [20]. The properties of the asphalt are tested as shown in Table 1.

Table 1

Three-index for recycled asphalt

Test index	Unit	AH-90 asphalt	Recycled asphalt
Penetration(25℃)	0.1mm	80~100	38.4
Softening point	℃	≮44	52.6
Ductility(15℃)	cm	>100	15.5

According to the results of Table 1, it is not difficult to find that the aging of recycled asphalt is serious. It is necessary to add regenerant to restore its performance.

The regenerant used in the test is XT-2 produced by Changzhou Xintuo Refined Materials Incorporation. The technical property indexes are shown in Table 2:

Table 2.

XT-2 Regenerant index

Test items	Performance index	
	Index range	Measured value
Viscosity at 60℃, cSt	50～175	165
Contents of saturates,%,≤	30	28.5
Contents of Sweet fragrance, %,＞	60	71.5

Flash point, ℃, ＞	220	223
Viscosity ratio before and after film oven test,%≤	3	1.2
Mass change before and after film oven test,%,≤	3	-0.79
Densityat 15℃,g/cm³	0.95～1.05	1.01

4.2 Design and Analysis of Regenerative Test

The preheating temperature (℃) interval of old asphalt in this experiment is [130,150] and the regeneration agent (%) interval is [2, 4]. According to the design principle of CCD, the test design and results are shown in Table 3.

Table 3.

The mixed design and results of the test

NO.	Preheating temperature of old asphalt/℃	Regenerativeagent content /%	G*/sinδ/MPa	S/MPa	m
1	150.00	4.00	0.59	215	0.372
2	140.00	3.00	1.68	238	0.341
3	130.00	2.00	1.92	276	0.309
4	125.86	3.00	1.42	250	0.330
5	140.00	3.00	1.73	242	0.337
6	140.00	1.59	2.36	309	0.296
7	140.00	4.41	0.48	198	0.409
8	130.00	4.00	0.52	219	0.373
9	140.00	3.00	1.69	238	0.340
10	140.00	3.00	1.72	239	0.340
11	150.00	2.00	2.06	281	0.307
12	154.14	3.00	1.83	242	0.333
13	140.00	3.00	1.76	240	0.339

4.2.1 Analysis of rutting factor G*/sinδ

Design-Expert software was used to analyze the variance of the regression model. The analysis of variance is shown in Table 4.

Table 4.

Variance analysis of rutting factor

Project	Sum of squares	Freedom	Mean square	F-value	P-value (Prob＞F)	Remarks
Model	4.25	5	0.85	39.24	< 0.0001	Significant
A-temperature	0.078	1	0.078	3.60	0.0997	
B-Regenerative agent content	3.82	1	3.82	176.20	< 0.0001	
AB	1.225×10^{-3}	1	1.225×10^{-3}	0.056	0.8189	
A^2	0.081	1	0.081	3.74	0.0943	
B^2	0.31	1	0.31	14.21	0.0070	
Residual	0.15	7	0.022			
Unintended term	0.15	3	0.049	47.79	0.0014	Significant
Pure error	4.120×10^{-3}	4	1.030×10^{-3}			
The sum	4.41	12				

Notes: R^2=0.9655; Adj R^2=0.9409; Pred R^2=0.7602; Adeq Precision=19.539; C.V.=9.69%

The F-value is 39.24 in the equation, p-value(Prob＞F)is less than 0.0001, which presents that there is a significant nonlinear relationship between each factor and response value. The experiment method is accurate and reliable. The regression equation represented by factor coding is as follows:

$$G^*/\sin\delta = 1.72 - 0.099X_1 - 0.69X_2 - 0.017X_1X_2 - 0.11X_1^2 - 0.21X_2^2 \tag{3}$$

The value of actual factor is expressed as:

$$G^*/\sin\delta = -21.3904 + 0.3175A + 0.8169B + 1.75\times10^{-3}AB - 1.08\times10^{-3}A^2 - 0.2105B^2 \tag{4}$$

(Among them, A and B respectively represent old asphalt preheating temperature / ℃ and regenerated agent content /%).

At the same time, the complex correlation coefficient (R^2) from Table 4equals0.9655, and the modified complex correlation coefficient (Adj R^2) equals0.9409, indicating that this model can explain the 94.09%of response value change. The model has optimal regression and can be used to analyze and predict the rutting factor of regenerated asphalt. Adeq Precision measured the signal of signal-to-noise ratio,which can be used for simulation when the value is larger than 4. Adeq Precision is 19.539, greater than 4 in this experiment, indicates the model has enough signals to response to the test design. The value of Pred R^2 is 0.7602, which is greater than 0.7,and the value of C.V.% is 9.69, less than 10, F-value of the unintended term is 47.79, greater than 0.1. The difference between Adj $R^2$and Pred R^2 is 0.0246, could meet the requirements being less than 0.2.The results show that all the coefficients are in conformity with the standard.

Based on the above data, the RT diagram and contour map of rutting factor with the relationship between the preheating temperature of old asphalt and the dosage of reclaimed agent are drawn in Figure 1.

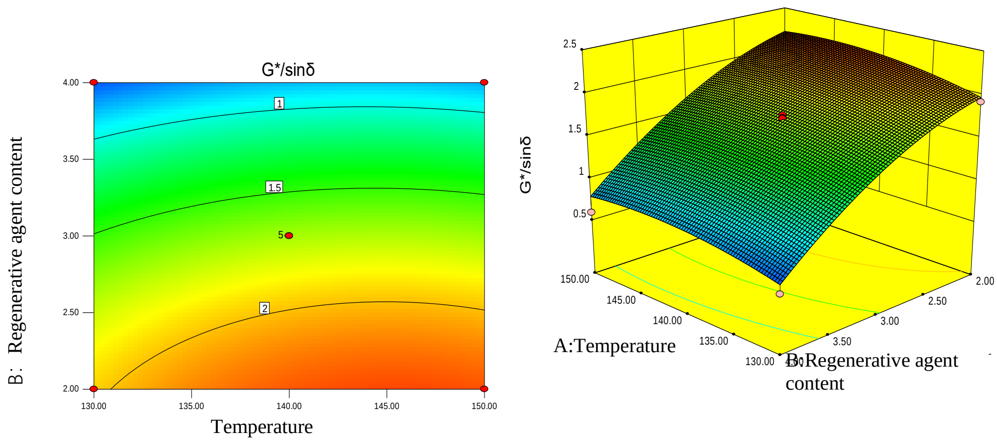

Fig.1 Response surface and contour map of rutting factor

Fig. 1 reflects the influence of regenerative agent content and the preheating temperature of old asphalt on rutting factor of recycled asphalt. The results show that the rutting factor of regenerated asphalt increases gradually with the increase of the preheating temperature of old asphalt under the conditions of regenerative agent. The constant increase of preheating temperature may cause the slight aging of asphalt again, which causes the asphalt to be hardened and the stability of high temperature increases. Under the certain temperature conditions, the rutting factor of recycled asphalt decreases rapidly with two order termas the increase of regenerative agent content.The influence of regenerative agent content on rutting factor is more significant than that of old asphalt preheating temperature. From the results of Table 3, it can be found that the rutting factor G*/sin δ satisfies the requirement of greater than 1.1kPa when the dosage of reagents is less than 4%. When the regeneration agent reaches 4% and above 4%, rutting factor is less than 1.1kPa, which can no longer meet the requirements. Therefore, the appropriate amount of regenerative agent and the appropriate preheating temperature of old asphalt are particularly important for the high temperature performance of recycled asphalt.

4.2.2 Analysis of Stiffness Modulus S

Design-Expert software was used to analyze the variance of the regression model. The analysis of variance is shown in Table 5.

Table 5

Stiffness modulus variance analysis table

Item	Sum of squares	Freedom	Mean square	F-value	P-value(Prob＞F)	Remarks
Model	10157.23	5	2031.45	77.93	< 0.0001	Significant
A-temperature	13.30	1	13.30	0.51	0.4982	
B-Regenerative agent content	9798.44	1	9798.44	375.90	< 0.0001	
AB	20.25	1	20.25	0.78	0.4073	
A^2	54.54	1	54.54	2.09	0.1913	
B^2	298.45	1	298.45	11.45	0.0117	
Residual	182.46	7	26.07			
Unintended term	171.26	3	57.09	20.39	0.0069	Significant
Pure error	11.20	4	2.80			
The sum	10339.69	12				

Note: R^2=0.9824; Adj R^2=0.9697; Pred R^2=0.8805; Adeq Precision=28.539; C.V.=2.08%

It is known that F value is 77.93 in the equation, p-value(Prob＞F)is less than 0.0001 , which presents that there is a significant nonlinear relationship between each factor and response value. The experiment method is accurate and reliable. The regression equation represented by factor coding is as follows:

$$S = 239.40 - 1.29X_1 - 35.00X_2 - 2.25X_1X_2 + 2.80X_1^2 + 6.55X_2^2 \tag{5}$$

The value of actual factor is expressed as:

$$S = 875.6906 - 7.2939A - 42.7972B - 0.225AB + 0.028A^2 + 6.55B^2 \quad (6)$$

(Among them, A and B respectively represent preheating temperature / ℃ of old asphalt and regenerated agent content /%).

Meanwhile, the complex correlation coefficient (R^2)is0.9824andcorrection of complex correlation coefficient (Adj R^2)is0.9697, indicating that the model can explain 96.97% of the response value change. The model is well regressive and can be used to analyze and predict the stiffness modulus of recycled asphalt. Adeq Precision measures the signal-to-noise ratio and specifies that it can be used for simulation when the value is greater than 4. The value of Adeq Precision is 28.539, which is greater than4 in this experiment, indicates that the model has enough signals to response to test design. Pred R^2 is 0.8805, greater than0.7, and C.V.% is 2.08, less than10. F-value of the unintended term is 20.39, greater than 0.1.The difference between Adj R^2 and Pred $R^2$is 0.0127 in the experiment, could meet the requirements being less than 0.2. The results show that all the coefficients are in conformity with the standard.

Contact the above data and use software to make the response surface 3D map and contour map of the relationship between stiffness modulus, and the old asphalt preheating temperature and regeneration agent dosage, as shown in Figure 2.

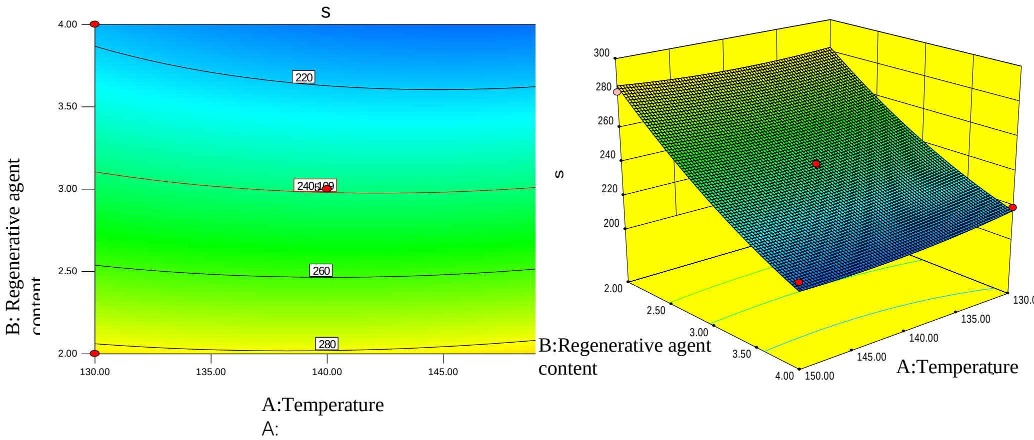

Fig.2 Response surface and contour map of stiffness modulus

Fig. 2 reflects the relationship between the regenerative agent content and the preheating temperature of old asphalt on the modulus of elasticity of recycled asphalt S. The results show that under the conditions of regenerative agent, the stiffness modulus of regenerative asphalt gradually decreases with the increase of preheating temperature of old asphalt, decreasing to a stable rate at 140 ℃. Under the condition of a certain temperature, the stiffness modulus of regenerated asphalt decreases with the increase of regenerative agent content, and it is easy to find that the influence of regenerative agent on the modulus of stiffness is more significant than the temperature. From the test results of Table 3, it is found that all the stiffness modulus meet the requirement of 300MPa when the regenerator content is larger than 2%;S is 309MPa when regenerator content is less than 2%, which cannot meet the requirements. With the increase of the amount of regenerative agent and increase of the preheating temperature of old asphalt, the stiffness modulus of the recycled asphalt has been reduced in varying degrees, and the low temperature crack resistance is enhanced. Therefore, it is more important to appropriately increase the amount of regenerative agent and select the suitable preheating temperature of old materials for low temperature performance of the recycled asphalt.

4.2.3Analysis of Creep Rate M

Design-Expert software was used to analyze the variance of the regression model. The analysis of variance is shown in Table 6.

Table .6

Variance analysis table of creep rate

Item	Sum of squares	Freedom	mean square	F-value	P-value(Prob＞F)	remarks
Model	0.011	5	2.175×10^{-3}	108.37	< 0.0001	Significant
A-temperature	1.930×10^{-7}	1	1.930×10^{-7}	9.616×10^{-3}	0.9246	
B-Regenerative agent content	0.010	1	0.010	519.42	< 0.0001	
AB	2.500×10^{-7}	1	2.50×10^{-7}	0.012	0.9143	
A^2	1.339×10^{-4}	1	1.339×10^{-4}	6.67	0.0363	
B^2	2.599×10^{-4}	1	2.599×10^{-4}	12.95	0.0088	
Residual	1.405×10^{-4}	7	2.007×10^{-5}			
Unintended term	1.313×10^{-4}	3	4.377×10^{-5}	19.03	0.0079	Significant
Pure error	9.200×10^{-6}	4	2.300×10^{-6}			
The sum	0.011	12				

Note: R^2=0.9872; Adj R^2=0.9781; Pred R^2=0.9139; AdeqPrecision=33.547; C.V.=1.32%

It can be known from Table 6 that F-value in the equation is 108.37, p-value (Prob＞F)is less than0.0001, which presents that there is a significant nonlinear relationship between each factor and response value. The test method is accurate and reliable. The regression equation represented by factor coding is as follows:

$$m = 0.34 + 1.533\times10^{-4}X_1 + 0.036X_2 + 2.5\times10^{-4}X_1X_2 - 4.387\times10^{-3}X_1^2 + 6.112\times10^{-3}X_2^2 \quad (7)$$

The value of the actual factor is expressed as:

$$m = -0.5655 + 0.01223A - 4.0742\times10^{-3}B + 2.5\times10^{-5}AB - 4.3875\times10^{-5}A^2 + 6.1125\times10^{-3}B^2 \quad (8)$$

(Among them, A and B represent old asphalt preheating temperature / ℃ and regenerated agent content /% respectively.)

Meanwhile, from Table 6 that complex correlation coefficient (R^2) equals to 0.9872; correction of complex correlation coefficient (Adj R^2) is 0.9781, indicating that the model can explain 96.97% of the response value change. The model is well regressive and can be used to analyze and predict the creep rate of recycled asphalt. Adeq Precision measures the signal-to-noise ratio and specifies that it can be used for simulation when the value is greater than 4. Adeq Precision is 33.547, greater than 4 in this test, illustrates that this model has enough signals to respond to this test design. Pred R^2 is 0.9139, greater than0.7,and C.V.% is 1.32, less than 10; F-value of the unintended term is 19.03, greater than 0.1; the difference between Adj R^2 and Pred R^2 is 0.0091could meet the requirements being less than 0.2. The results show that all the coefficients are in conformity with the standard.

Contact the above data, use software to make the response surface 3D map and contour map of the creep rate and the old asphalt preheating temperature and regeneration agent dosage, as shown in Figure 3.

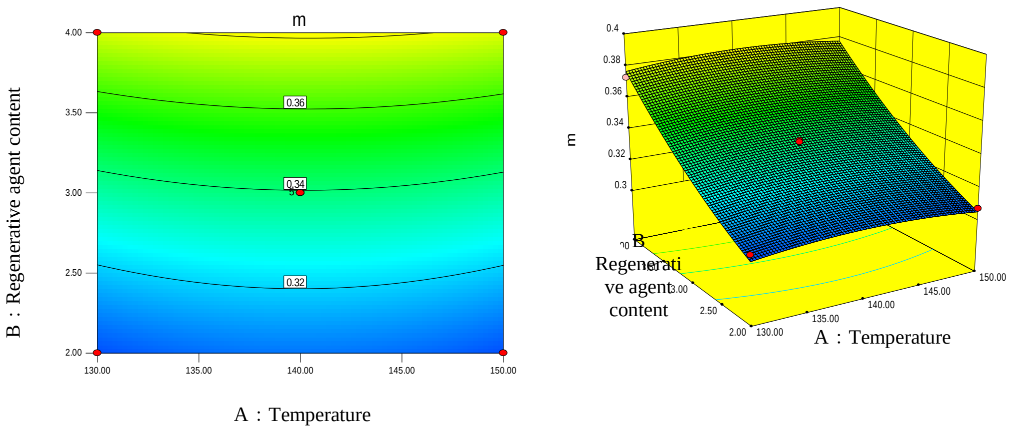

Fig.3 The response surface and contour map of creep rate

Fig. 3 reflects the relationship between the regenerative agent content and the preheating temperature of old asphalt on the creep rate of recycled asphalt. The results show that the creep rate of recycled asphalt increases with the increase of the preheating temperature of old asphalt under certain conditions. When the temperature is about 140℃, it tends to be stable and m-value decreases at 150℃. The reason may be that the higher temperature makes the regenerated asphalt slightly aging again. Under the certain temperature conditions, the creep rate of recycled asphalt increases rapidly with the increase of regeneration agent content. It is not difficult to find that the regenerative agent has a more significant influence on the creep rate than the temperature, and the relation between the creep rate and the regenerative agent content and preheating temperature of the old asphalt is the opposite of the stiffness modulus and the two. From the test results of Table 3, it is found that when the regenerator content is more than or equal to 2%, the entire creep rate meets the requirement of more than 0.3. When the regenerator content is below 2%, the m value cannot meet the requirements. With the appropriate increase of preheating temperature, the creep rate of regenerated asphalt increased in varying degrees with the increase of the amount of regenerative agent, and the low temperature cracking resistance of the regenerated asphalt is enhanced. Therefore, when other conditions were certain, proper increase of regenerative agent dosage and selection of suitable preheating temperature of suitable old asphalt could improve the low temperature performance of asphalt.

4.3 Optimization of Test Results

Through the analysis and optimization of the test results, the response value reaches the optimal value when the preheating temperature of old asphalt is 143℃ and the regeneration agent dosage is 3.2%. The predictive response value isG*/sin; S is 232.5MPa and m is 0.346 at this moment。For this, the recycled asphalt is reproduced in accordance with the optimized results and verified by tests. The high temperature rheological properties of the recycled asphalt are tested after aging of the film oven and PAV aging. It is stipulated that the rutting factor G*/ sin δ of the regenerated asphalt should be more than 2.2kPa after the aging of film oven; the fatigue factor G*sin δ should be less than 5000KPa after the aging of PAV, and the three-index of regenerated asphalt are detected at the same time. The results of the test are shown in Table 7 and table 8 respectively.

Table 7

Test verification of optimal regenerative condition stability model test

No.	Preheating temperature of old asphalt(°C)	Regenerative agent content (%)	(G*/sinδ) /MPa	S /MPa	m	After TFOT (G*/sinδ) /MPa	19°C (G*sinδ)/MPa
1	143	3.20	1.56	230	0.347	3.23	4723
2	143	3.20	1.63	241	0.342	3.34	4749
3	143	3.20	1.59	233	0.349	3.25	4730

Table 8

Test results of three-index of regenerated asphalt

No.	Preheating temperature of old asphalt/°C	Regenerative agent content /%	Penetration (25°C)	Softening point /°C	Ductility(15°C)
1	143	3.20	93.3	47	>100
2	143	3.20	92.8	48	>100
3	143	3.20	93.5	48	>100

From table 7 and table 8 that the regeneration performance of old asphalt is optimal under the conditions that the preheating temperature of old asphalt at 143℃ and the amount of regenerative agent is 3.2%. The dynamic shear rheology test and the low temperature bending test data both meet the requirements. Three-index of the regenerated asphalt all meet the requirements of 90# asphalt specification, indicating the regeneration effect of old asphalt under this regeneration condition is optimal.

5. Conclusion

(1) Using the CCD design method, the asphalt regeneration test is designed, analyzed and optimized by Design-Expert8.0 software. The response surface model is established with the rutting factor, the stiffness modulus and the creep rate as the influencing factor, which greatly reduces the number of tests and calculation. The regression analysis shows that the model is stable and calculated; the result is accurate. This method can be used for the design and optimization of asphalt regeneration test.

(2) Based on the response surface design, the optimization results of old asphalt regeneration test are designed. The optimal regeneration agent dosage is 3.2% and the preheating temperature of old asphalt is 143℃. The recycled asphalt has optimal performance in all aspects.

(3) Through the analysis on the significance of the test factors, it is found that the rutting factor has a negative correlation with the modulus of stiffness and the amount of regenerator and the creep rate is positively correlated with the amount of regenerative agent. With the increase of regenerative agent dosage, the high temperature performance of recycled asphalt decreases, and the low temperature performance increases gradually. With the increase of the preheating temperature of old asphalt, the rutting factor and stiffness modulus of the asphalt increases, and the creep rate decreased because of aging of the asphalt again after heating. The influence of reagents on the test results is much greater than that of the asphalt preheating temperature on the test results.

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