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DCLR Modified Petroleum Asphalt Optimization and Mixture Road Performance

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ARTICLE INFO

Article history

Received: 24 October 2022

Revised: 31 October 2022

Accepted: 9 November 2022

Published Online: 16 November 2022

Keywords:

Orthogonal design

DCLR modified asphalt

Asphalt mixture

Coal liquefaction residue

Microscopic composition

ABSTRACT

In recent decades, modified asphalt materials have been used in enhancing the traffic load on the roads. The main objective of this paper is to explore the modification effect of direct coal liquefaction residue (DCLR) on asphalt binders and investigate the effectiveness of DCLR in improving the performance of asphalt road. This paper prepared modified petroleum asphalt under different process conditions and tested its penetration, softening point and ductility index. Based on the experimental data, according to gray correlation degree, the performance for the asphalt was compared. The performance for the modified asphalt is simulated and predicted using polynomial functions. The modified asphalt was analyzed by FT-IR, TGA, SEM and HPLC. The results show that the optimal process conditions for DCLR modified asphalt are shear mixing time of 45 min, shear mixing temperature of 150 °C and shear mixing rate of 4000 r/min. The predicted fit with the experimental data of 0.993 further demonstrates the effectiveness of the method. The characterization results show no significant chemical change between the DCLR and the asphalt. DCLR can significantly improve the high temperature performance and water stability of the asphalt, but it has little impact on its low temperature performance.

1. Introduction

In recent decades, the paving of highway roads has gained rapid development to satisfy the increasing number of trucks, cars, and other vehicles all over the world. Especially in China, the huge traffic flow causes increasingly serious damage to the road, which pushes urgent demands

for high-quality asphalt materials for paving high-grade highways. In addition, the outdoor ambient temperature in north China changes variably at different seasons, exerting a great influence on road performance. Hence, the usage of fluxes is necessary to mix with the base asphalt to improve the road properties, such as the asphalt durability, adhesion, deformation resistance, etc. ^[1]. The main mod-

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DOI: <https://doi.org/10.30564/frae.v5i1.5196>

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ifiers used at present are coal tar pitch, tire rubber, and polymers. Although the road property could be improved to some degree, some environmental and economic problems appeared concomitantly [2].

The Trinidad Lake Asphalt (TLA) modifier is the most effective flux for improving the relative properties of paving asphalt. TLA modifier has been widely used in China due to its excellent modification performance, including Jiangyin Bridge, capital international airport, Chengdu-Chongqing expressway, etc. By analyzing the TLA composition, it was found that modifiers with multiple aromatic structures and more polar functional groups would show better modifying ability [3]. Namely, strong adsorption capacity on the surface of quartz and limestone and good anti-stripping performance could be presented. After preliminary analysis, it has been found that coal direct liquefaction residue (DCLR) has a similar structure to TLA and is a potential alternative asphalt modifier [4].

Typically, DCLR is rich in organic compounds with high molecular weight, varied heteroatomic species, rich aromatic cyclic compounds and strong polarity. These organic macro-molecules doped with polar groups (i.e. N, S) have strong adsorption ability on the mineral surface, such as silicate, limestone, quartz and so on [5]. The excellent adsorption performance could enhance the anti-peeling property of the asphalt mixture obviously. In recent studies, it was shown that the DCLR can improve the high-temperature performance and durability of road asphalt. However, lots of researches focused on the binary modifying effect of DCLR coupled with other polymer compounds, while rarely probed into the single DCLR modifier. Hence, it was necessary to determine the optimal condition in the preparation of high-quality DCLR modified asphalt [6].

Nevertheless, the experimental studies would hardly give a consistent conduction for all kinds of DCLR species since it was difficult to test the road performance of the whole DCLR modified asphalts. Noticeably, different kinds of DCLR had the same aromatic structure and similar chemical or physical properties, which gave a possibility to use scientific predictions for evaluating the road performance. BP neural network is the core part of the forward neural network and the essence of the entire artificial neural network system [7]. In network applications, about 80% of neural networks adopt BP networks or improved. The asphalt model can be optimized on the basis of self-learning, which is very suitable for the establishment and prediction of DCLR modified asphalt road performance model [8]. Zhao et al. used BP neural network to study the asphalt pavement compaction prediction model in 2020, the results showed that it was feasible to use

this model to predict asphalt pavement compaction [9]. Xie (2017) used BP neural network to predict the fatigue performance of asphalt mixture. It was suggested that all the statistical indexes obtained by the neural network model were obviously better than that of fatigue equation model [10]. And the neural network also had the function of index weight analysis. Dou et al. (2016) used BP neural network to predict the ductility of asphalt, the result showed that the trained BP neural network had a good prediction ability for the ductility of blending asphalt [11].

Generally speaking, DCLR is a promising flux to improve the high temperature and sensitive properties of road asphalt. While the modified process needed to be further investigated. The preparing condition is vital to the road performance of modified asphalt. Hence, this paper established basic research for the physical engineering promotion of DCLR modified asphalt. In addition, to broad the applicability of the preparation method of the DCLR modified asphalt, BP artificial intelligence was used to predict relative road properties and optimize the modifying condition. Through the combination of experimental research and mathematical simulation and prediction, the preparation method and performance evaluation of DCLR modified asphalt can be further improved, and its popularization and application can be realized.

2. Experimental

2.1 Raw Materials

The DCLR modifier used in this experiment was the residue of Shenhua coal direct liquefaction demonstration plant in Inner Mongolia. The 90# base petroleum asphalt was derived from Shenhua Group. The basic road properties and compositions of base petroleum asphalt and DCLR were listed in Table 1 and Table 2.

2.2 Preparation of Modified Asphalt

The 90# base petroleum asphalt was heated to liquid status by oil bath in a 0.5 L uncovered vessel at 125 °C. Then the DCLR or TLA modifier was added into the container and mechanically stirred for 90 min to prepare DCLR modified asphalt. The base asphalt mass was kept in the same quality to decrease the system deviation during the preparation process.

2.3 Optimization of DCLR Modified Petroleum Asphalt Process

2.3.1 Orthogonal Test

Generally, the road performance of modified asphalt would be affected directly by the blending process and

Table 1. Composition and properties of base asphalt

The basic road properties			
Items	Softening point/°C	Penetration /0. 1mm, 25 °C	ductility(25 °C)/cm
Test result	49.9	72. 1	>140
Proper range	≥46	60~80	>140
Chemical composition proportion / %			
Saturation point	Aromatics	Asphaltene	Colloid
11.2	50.8	12.3	25.7

Table 2. Composition and properties of DCLR

Properties of DCLR									
Items	Softening point/°C			Penetration /0. 1mm, 25 °C			Ductility(25°C)/cm		
Test result	190			5			6		
Composition of DCLR / %									
Saturation point	Aromatics			Asphaltene			Colloid		
11.2	50.8			12.3			25.7		
Composition content analysis of DCLR family/%									
heavy oil	asphaltene		Pre-asphalt		organic detritus		inorganic ash		
8.04	14.97		10.37		36.44		30.18		
DCLR ash composition and content/%									
SiO ₂	Al ₂ O ₃	FeO ₃	CaO	MgO	SO ₃	TiO ₂	K ₂ O	Na ₂ O	P ₂ O ₅
20.3	8.65	33.69	17.06	1.01	15.34	0.84	0.07	1.79	1.25

preparing condition. It was necessary to study and analyze the reaction conditions systematically. The optimal incorporation of DCLR was 8%. The orthogonal design method was used to investigate the effects of temperature, shear time and shear rate on the road properties of modified petroleum asphalt, such as penetration, softening point and ductility.

2.3.2 Grey Correlation Analysis

Grey correlation analysis was applied to analyze the correlation degree among different factors, which could combine qualitative analysis with quantitative analysis effectively [12]. The main calculation steps of grey correlation analysis were listed below.

The main calculation steps were conducted as follows: Determining the reference; comparing and equalizing each sequence; finding the correlation coefficient, the maximum and minimum difference between the two poles; calculating the grey correlation degree and sorting by gray correlation size [13].

Table 3. Orthogonal experimental design

Level factor	Shear temperature/A	Shear time/B	Rate of shear/C
1	150 °C	30 min	2000 r/min
2	160 °C	45 min	4000 r/min
3	170 °C	60 min	6000 r/min

Table 4. Orthogonal experiment scheme

Number	Shear temperature/A	Shear time/B	Rate of shear/C	Combination
1	150 °C	30 min	2000 r/min	A ₁ B ₁ C ₁
2	150 °C	45 min	4000 r/min	A ₁ B ₂ C ₂
3	150 °C	60 min	6000 r/min	A ₁ B ₃ C ₃
4	160 °C	30 min	4000 r/min	A ₂ B ₁ C ₂
5	160 °C	45 min	6000 r/min	A ₂ B ₂ C ₃
6	160 °C	60 min	2000 r/min	A ₂ B ₃ C ₁
7	170 °C	30 min	6000 r/min	A ₃ B ₁ C ₃
8	170 °C	45 min	2000 r/min	A ₃ B ₂ C ₁
9	170 °C	60 min	4000 r/min	A ₃ B ₃ C ₂

3. Results and Discussion

3.1 Determination of the Optimal Condition

The parameter of penetration represents the hardness of the asphalt, the low penetration value indicates the strong hardness of road materials. Normally, this value was basically between 50 and 70 (25 °C, 5 s, 0.1 mm) according to the latest TLA modified asphalt standard. (JTG E20-2011). PI is an important index to evaluate asphalt temperature sensitivity. PI values can also be used to evaluate

the colloidal structure of asphalt in addition to evaluating the temperature sensing properties of asphalt. $PI < -2$ shows that asphalt is a sol-shaped structure and a pure viscous fluid, such as coal tar pitch. $-2 < PI < 2$ shows that asphalt is a sol-gel structure, have elastic effect; $PI > 2$ shows that the asphalt is a gel structure, showing typical thixotropy, such as oxidized asphalt (Ge, 2016).

From Table 5, compared with the base asphalt, the penetration value was decreased from 52 mm to 71 mm. Hence, the penetration index was enhanced by the additive of DCLR, which was extremely vital for preparing the high-quality road asphalt. In addition, the penetration

property of DCLR modified asphalt meted the current standard and the high value of R (19.00) indicated shear time was the main factor to affect the asphalt hardness. For the parameter of ductility, it can be observed that the ductility value was decreased with the addition of DCLR flux. It was attributed that the DCLR and petroleum asphalt were not completely melted, forming a sol-based structure with macro molecular particles and leading to the decrease of shear strain resistance. The analysis of the range R value showed that the main indicator affecting the modified asphalt was the shear time.

Table 5. Orthogonal experiment results analysis

Surveillance project		Shear temperature A/°C	Shear time B/min	Rate of shear C/r/min
Penetration /0. 1mm, 25 °C	\bar{K}_1	56.1	52.0	57.2
	\bar{K}_2	56.7	64.4	57.5
	\bar{K}_3	62.1	71.0	68.4
	R	6.0	19.0	11.2
	Factor sort		B > C > A	
	Optimal group		A ₂ B ₂ C ₃	
Ductility(25°C)/cm	K_1	8.9	13.7	9.6
	K_2	9.5	10.6	8.1
	K_3	9.9	10.7	8.9
	R	1.0	3.1	1.5
	Factor sort		B > C > A	
	Optimal group		A ₂ B ₁ C ₂	
Softening point/°C	K_1	49.9	51.3	50.4
	K_2	50.9	51.1	49.3
	K_3	49.5	50.6	48.9
	R	1.4	0.7	1.5
	Factor sort		B > C > A	
	Optimal group		A ₂ B ₂ C ₃	
PI	K_1	1.032	1.039	0.996
	K_2	1.012	1.067	1.083
	K_3	1.066	1.115	1.047
	R	0.054	0.076	0.09
	Factor sort		C > B > A	
	Optimal group		A ₃ B ₂ C ₁	

For the parameter of softening point, it showed that the value of DCLR modified asphalt was 49.2 °C, which was 0.7 lower than the base asphalt. The additive of DCLR had a significant influence on softening point property. This was attributed to the heterogeneity of the mixture, which was prepared by physical blending method. Although this index was decreased, the softening point index of modified asphalt still meted the current standard. The main factor was the shear rate to affect softening point according to the R value (1.50). For the PI value, the result indicated that the structure of DCLR modified petroleum asphalt belonged to sol-gel structure (PI= 1.047). Then the analysis of the range R results showed that the main indicator affecting the modified asphalt is the shear rate (R=0.90). With the comprehensive consideration of all parameters, the optimum technological conditions of DCLR modified petroleum asphalt were prepared at 45 min (shear time), 4000 r/min (shear rate) and 150 °C (shear temperature).

3.2 Grey Correlation Analysis

The grey association coefficient of DCLR modified asphalt was presented in Table 6. The result indicated that the four parameters had different influence degree on the road performance of modified asphalt. Specifically, the factor proportion was 25.03%, 25.02%, 25.01% and 24.92% respectively for the penetration, PI, ductility and softening point.

Table 6. The grey association analysis of modified asphalt

Group	Penetration/ 0.1mm, 25 °C	Grey correlation degree		
		Ductility (25°C)/cm	Softening point/°C	PI
ξ_1	0.985	0.977	0.987	0.997
ξ_2	0.959	0.962	0.976	0.965
ξ_3	0.973	0.966	0.939	0.971
ξ_4	0.884	0.985	0.914	0.908
ξ_5	0.926	0.925	0.917	0.916
ξ_6	0.917	0.898	0.887	0.896
ξ_7	0.963	0.966	0.975	0.963
ξ_8	0.982	0.952	0.989	1.000
ξ_9	0.97	0.925	0.938	0.942
Association degree	0.951	0.9506	0.9469	0.9509
Factor proportion / %	25.03	25.01	24.92	25.02

4. DCLR Modified Asphalt Performance Test and Mixture Test

4.1 DCLR Modified Asphalt Performance Test

The FT-IR spectra of base asphalt and DCLR modified asphalt was shown in Figure 1. Typically, the peak wave numbers of 2918.19 cm^{-1} and 2848.18 cm^{-1} indicated the asymmetric and symmetric stretching vibration of methylene. 1592.47 cm^{-1} and 1453.2 cm^{-1} meant aromatic ring skeleton vibration, 2362.61 cm^{-1} was C=C or C=N stretching vibration peaks. And the 700-900 cm^{-1} absorption peaks were mainly caused by hydrocarbon bonds of substituted aromatic rings. From Figure 1, the characteristic absorption peak of the sample at 3100-3300 cm^{-1} is caused by alcohol, carboxylic acid and ester C=O; 3000-3100 cm^{-1} is the absorption vibration peak of aromatic hydrocarbon C-H; 2800-2900 cm^{-1} is caused by saturated fatty hydrocarbon-CH₂-expansion vibration [14]. The result suggested that DCLR has more influence on asphalt, and the strength and width of all absorption peaks are weakened.

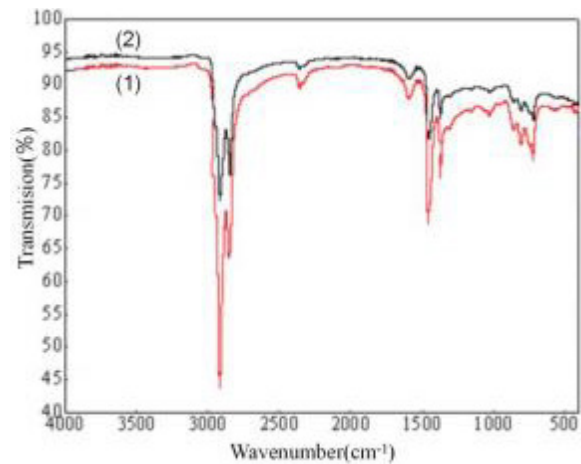


Figure 1. FT-IR spectra of DCLR and modified asphalt

The TG curves of base asphalt, modified asphalt and DCLR feedstocks were depicted in Figure 2. The results showed that the initial decomposition temperature of DCLR is about 230 °C and the maximum weight loss rate occurred at 520 °C. While the initial decomposition temperature of base asphalt and modified asphalt was around 350 °C, and the maximum weight loss rate occurred at 500 °C. The maximum residual weight of the DCLR was 67%, which indicated that the DCLR contained more macro-molecular substances. With the increasing temperature, DCLR volatiles released and the weight decreased gradually. In addition, the maximum weight loss of modified asphalt was less than 80% while it was more than 80%

for base asphalt. This indicated that the modified asphalt contained more high temperature resistant substances than the base asphalt. Therefore, the additive of DCLR to base asphalt could improve the high temperature properties.

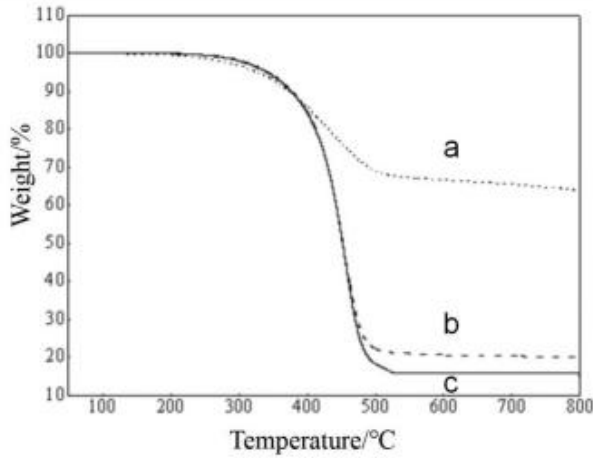


Figure 2. TG analysis of base asphalt, modified asphalt and DCLR

The HPLC test was applied to further investigate the components contained in the road asphalt, which was conducted with the flow phase of THF at 60 °C. The results were shown in the Figure 3 and Figure 4. The compositions were divided into heavy group area (5.12 min < t < 10.92 min), medium group section (10.92 min < t < 11.67 min), and light group section (11.67 min < t < 14.67 min) according to the difference of the complex macro molecular mixture [15]. The proportion of each peak area in the whole sample was calculated by normalizing the spectral line area. It can be obtained that the proportion of heavy, medium and light components in base asphalt were 20.82%, 21.65%, 57.53%, and the values were 22.36%, 21.67%, 55.97% for DCLR modified asphalt. This indicates that the heavy components become higher and less light components during the modification process.

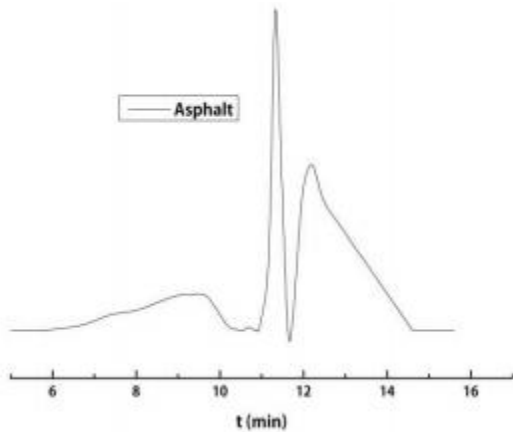


Figure 3. HPLC of base asphalt

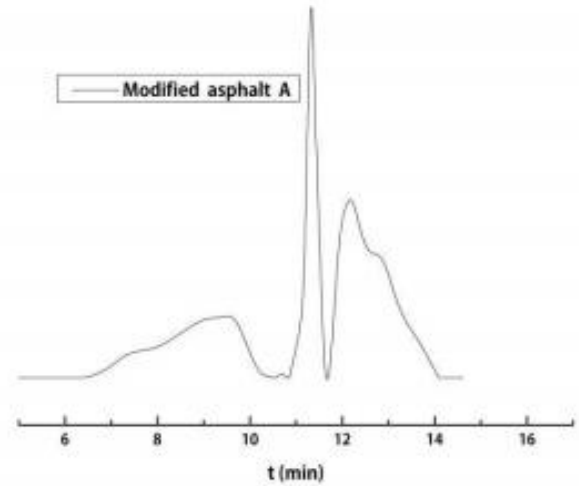


Figure 4. HPLC of modified asphalt

4.2 Mixture Test

The asphalt mixture was prepared by a certain graded synthetic method with modified asphalt served as materials. Then the asphalt mixture was usually used as surface layer of asphalt pavement, which went through the direct damage from a variety of vehicle loads and environmental factors. Hence the performance of asphalt mixture greatly affected the service performance and service life of the pavement [16].

The AC-20 asphalt mixture was selected to test the properties of DCLR modified asphalt. The mineral grading process was implemented according to the test code of asphalt and asphalt mixtures of ministry of communications (JTG E20-2011). The mineral material grading design used in this article was listed in Table 7.

In order to obtain the optimum asphalt ratio of asphalt mixture, the interval of 0.5% and take 5 different asphalt ratios were used to proceed Marshall test. For the measurement index and relevant mechanical indicators, the test results were shown in Table 8.

Considering the relevant and mechanical indexes of asphalt mixture, the optimum asphalt stone ratio was 4.38%.

4.2.1 Evaluation on the High-temperature Performance of the Asphalt Mixture

If the stress is applied to the asphalt mixture for a long time, the strain will increase over time, and the deformation of the asphalt will gradually recover after the stress disappears, but it will still undergo permanent deformation, especially under high temperature conditions. Rut test is used to evaluate the high temperature stability of the asphalt mixture. Table 9 shows the test results.

Table 7. Asphalt AC-20 gradation

Mesh size (mm)	19	16	13	9.5	4.75	2.36	1.18	0.6	0.3	0.15	0.075
Specification upper limit	100	92	80	72	56	44	33	24	17	13	7
Lower specification	90	78	62	50	26	16	12	8	5	4	3
Synthetic grading	95	88	78	65.5	41	28.5	17.6	11.3	7.4	6.1	5.1

Table 8. Marshall stability experiment result of bitumen blended stock

Bitumen aggregate Ratio (%)	3.3	3.8	4.3	4.8	5.3
Porosity (%)	6.7	5.7	3.9	2.6	1.7
Mineral material clearance ratio (%)	13.3	13.5	13.3	12.8	13.0
Asphalt Saturation (%)	49.8	57.7	70.3	79.7	87.2
Stability (KN)	10.27	10.77	15.16	10.45	9.89
Flow value (mm)	30.9	27.4	27.7	33.5	34.3

Table 9. Rutting test result of bitumen blended stock

Type of mixture	45 min deformation (mm)	60 min deformation (mm)	Dynamic stability (secondary/mm)
Matrix-based asphalt mixture	6.031	7.016	3546
DCLR modified asphalt mixture	6.013	6.925	3714

If the stress was applied to the asphalt mixture for a long time, the strain would increase over time, and the deformation of the asphalt would gradually recover when the stress was removed. However, the asphalt would undergo the permanent deformation, especially in the high temperature conditions. Generally, rut test was used to evaluate the high temperature stability of the asphalt mixture. From Table 9, the dynamic stability of the base asphalt mixture met the specification requirements (dynamic stability ≥ 2400 /mm). Compared with the latter, the value increased from 3546/mm to 3714/mm, having 4.8% increment. The deformation degree of the asphalt mixture increased with the time extended, and the value was 6.031 mm and 6.013 mm at 45 min respectively for the base asphalt mixture and DCLR modified asphalt mixture. While it was 7.016 mm and 6.925 mm at 60 min. DCLR modi-

fied asphalt mixture decreased by 2% and 1.3% compared with the base asphalt mixture. The result showed that the high temperature performance of DCLR modified asphalt mixture is better than the base asphalt mixture.

4.2.2 Evaluation on the Low-temperature Performance of the Asphalt Mixture

The low temperature crack resistance of asphalt mixture was directly related to the low temperature service performance of pavement. Then it would affect the service life of pavement. The low temperature bending experiment of small beam was used to evaluate the crack resistance of modified asphalt mixture. In Table 10, the curved tensile strain of DCLR modified asphalt mixture was 3% lower than the base asphalt mixture, which was failed to meet the current requirements (greater than 2300 $\mu\epsilon$ in the JTGE20-2011). The result indicated that DCLR modified asphalt mixture had a deficient problem on the low temperature crack resistance. This was attributed to the poor mutual solubility between DCLR and base asphalt. Then a solvent structure was generated to form in the form of macro-molecular particles. Therefore, the heterogeneous mixture making the mixture material, resulting in the general improvement of low temperature performance.

Table 10. At low temperature bending test results

Type of mixture	Bending- tensile strength/ MPa	Rupture strain / $\mu\epsilon$	Failure strength measure modulus/ MPa
Matrix-based asphalt mixture	6.56	2385	2845
DCLR modified asphalt mixture	6.69	2373	2913

4.2.3 Evaluation of Water Stability of Asphalt Mixture

Water stability of asphalt mixture referred to the water entering the asphalt pavement into the asphalt pavement. Under the repeated action of the dynamic wheel load, water gradually penetrated into the asphalt and decreased the asphalt adhesion, resulting in the final asphalt stripping from the ore surface. In this case, the asphalt mixture started to drop grain, loose, and damage the overall

performance. Frozen-melt cracking strength was used to evaluate the water stability energy of the asphalt mixture. From Table 11, the DCLR modified asphalt mixture improved the split tensile strength of the test piece. The maximum load of the test piece after the freeze-melting cycle increased from 8.9 kN to 9.6 kN, and the freeze-melting cracking strength ratio increased from 83.1% to 87.1%. The result indicated that the DCLR could improve the water stability of the asphalt.

Table 11. Freeze-thaw split test results

Type of mixture	Splacking tensile strength of unfrozen-thaw circulating test piece		Splitting-resistant tensile strength of test specimen after freeze-thaw cycle		Frozen-melt splitting strength ratio (%)
	Maximum load (kN)	Splitting strength (MPa)	Maximum load (kN)	Splitting strength (MPa)	
Base asphalt mixture	9.2	0.894	8.1	1.15	83.3
DCLR modified asphalt mixture	9.61	1.00	8.9	1.36	87.1

5. Conclusions

Based on the obtained data, the following conclusions were made:

1) This study through the design of orthogonal experiment, the optimum technological conditions of DCLR modified petroleum asphalt are as follows: shear time 45 min, shear rate 4000 r/min, shear temperature 150 °C ; Through the analysis of grey correlation degree, the maximum correlation degree of needle entry degree is calculated;

2) The FT-IR spectra of base asphalt and DCLR modified asphalt was shown that DCLR has more influence on asphalt, and the strength and width of all absorption peaks are weakened. The TG curves of base asphalt, modified asphalt and DCLR indicated that the modified asphalt contained more high temperature resistant substances than the base asphalt.

3) Testing on the asphalt mixture has found that DCLR can significantly improve its high temperature performance and water stability, but it has little impact on its low temperature performance.

Data Availability Statement

Some or all data, models, or code generated or used during the study are available from the corresponding author by request.

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