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Performance characterization and microscopic properties of asphalt modified with rapeseed heavy oil-activated waste rubber powder

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ABSTRACT

To improve the waste rubber powder modified asphalt viscosity is high, poor storage stability, and other shortcomings, 40 mesh, 60 mesh, and 80 mesh are three kinds of mesh rubber powder using rapeseed heavy oil pre-swelling activation way to prepare modified asphalt, improve the waste rubber powder modified asphalt storage stability and reduce the viscosity. The optimum blending amount of rapeseed heavy oil-activated waste rubber powder was determined by setting three oil-rubber ratios, and the unactivated waste gum powder modified asphalt was set as a control group, and the physical, rheological, and microscopic properties of the asphalt were evaluated accordingly. The optimum preparation parameters under the premise of setting the total dosage of modifier as 20% were: the mass ratio of rubber powder to rapeseed oil was 2:1, and the mesh size of rubber powder was 80 mesh. Rapeseed heavy oil-activated waste rubber powder modified asphalt storage stability and low-temperature performance improved, viscosity decreased, but high-temperature performance decreased at the same time. Compared with the unactivated waste rubber powder modified asphalt, the softening point decreased by an average of 16%, the needle penetration increased by an average of 19%, the ductility increased by an average of 44%, the viscosity decreased by an average of 22%, and the softening point difference decreased by an average of 44%. In the high-temperature rheological performance test set temperature interval, the rutting coefficient $G^*/\sin\delta$ decline, creep stiffness S, and creep rate m have increased. Therefore, rapeseed heavy oil activation can effectively improve the overall stability of waste rubber powder-modified asphalt and can reduce the overall viscosity, which is conducive to the uniform dispersion of rubber powder in asphalt.

KEYWORDS

Rapeseed heavy oil; Waste crumb rubber; Modified asphalt; Action mechanism; Microscopic properties

1. INTRODUCTION

Asphalt pavements are commonly used in road paving projects because of their significant advantages such as a smooth and seamless surface, smooth driving experience, reduced noise pollution, dust control, and simplified maintenance and repair processes. However, traditional petroleum asphalt is prone to brittle cracking at low temperatures, softening and deformation at high temperatures, and is susceptible to moisture erosion, coupled with its lack of weathering performance, which are defects that should not be ignored [1]. SBS-modified asphalt has been widely researched and applied because of its good basic properties, accounting for as much as 61% of global asphalt demand [2]. However, in general, SBS-modified asphalt mixtures are more expensive, occupying a higher cost in the huge volume of road projects in China. Rubber as a polymer with the same SBS, its modified asphalt and SBS properties have similar advantages [3]. From the perspective of environmental protection, the waste tires will be broken up and utilized also greatly alleviates the cost of disposal. As of the end of September 2023, China's motor vehicle ownership reached 430 million vehicles [4], huge car ownership has also brought great environmental pressure, a large number of discarded and piled up waste tires caused serious environmental pollution and waste of resources, the waste rubber centralized recycling is now recognized as harmless, resourceful way of treatment [5]. Existing research shows that the basic performance of waste rubber powder-modified asphalt has greater advantages, rubber powder‘s elasticity is excellent, and can improve the low-temperature flexibility so that the pavement is not easy to crack; secondly, rubber powder can interact with asphalt components, can significantly improve the performance of the high temperature; finally, its adhesion with the aggregate is better, can reduce the probability of pavement water damage, prolonging the service life of the road. With the increasing scarcity of natural resources, ecological and environmental protection problems are becoming more and more serious, and green and sustainable have become the themes of today's social development. While improving the performance of pavement, the research and development of waste rubber powder-modified asphalt can also largely solve the problem of disposal of waste rubber products, so it has good application prospects and environmental value. However, several major problems limit the application of waste rubber powder modified asphalt, one is the waste rubber powder modified asphalt system is not stable, asphalt and rubber powder two-phase segregation is likely to occur, is not conducive to long-distance transportation, increased construction limitations [6]. Another is the waste rubber powder-modified asphalt viscosity is large, in the preparation process requires high temperature, high temperature is easy to causes rubber powder carbonization and serious cracking, so that the elasticity of the rubber is almost lost, affecting the effect of rubber-modified asphalt [7]. At present, to cope with the shortcomings of powder modified asphalt is roughly the powder for all kinds of activation treatment, the effect of which is to increase the powder surface activity, reduce the sulfur content within the molecule, and so on.

Biomass energy is now widely recognized at home and abroad as the main solution to future energy shortages, biomass as raw materials, and ultimately can be converted into materials with or similar to the function of asphalt, not only to solve the short-term problem of petroleum asphalt in the construction and maintenance of the road construction, but also to achieve the efficient and comprehensive utilization of biomass and its residues, to improve the model of its circular economy [8]. The main principle of bio-oil activation of rubber powder is to accelerate the swelling of rubber in asphalt and improve the compatibility of rubber and asphalt. Given the above problems and advantages of bio-oil, this paper mainly uses rapeseed heavy oil to activate the waste rubber powder, to explore the changes in the basic properties of waste rubber powder modified asphalt after activation, to reduce the viscosity of rubber asphalt, improve its storage stability, and provide a reference for improving the defects of waste rubber powder modified asphalt.

2. MATERIALS AND METHODS

2.1. Raw Materials

The matrix asphalt is 70# road petroleum asphalt. Rapeseed heavy oil was provided by Xiangtan Zhaoshan Oleochemical Technology Co. (Xiangtan, China). 40, 60, 80 mesh waste rubber powder provided by Hebei Kexu Building Materials Co. atment. The essential properties of the raw materials are listed in Tables 1-3.

Table 1. Basic parameters of 70# matrix asphalt

Items	Units	Requirements	Results	Methods
Penetration (25℃)/(100 g, 5 s)	0.1 mm	60~80	65.6	ASTM D5
Penetration index	-	-1.5~1.0	-0.7	ASTM D5
Softening point	℃	≥46	48.2	ASTM D36
Dynamic viscosity (60℃)	Pa s	≥180	228	ASTM D4002
Ductility (15℃, 5 cm/min)	cm	≥100	140	ASTM D113
Flashpoint	℃	≥260	289	ASTM D92
Solubility (trichloroethylene)	%	≥99.5	99.74	ASTM D70

Table 2. Basic parameters of RHO

Items	Units	Results
pH	-	2.1
Density	g/cm3	0.96
Solubility(trichloroethylene)	%	87.4

Table 3. Rubber powder performance index

Items	Requirements	Results
Density	≤1.0	1.0
Carbon black content (%)	≥26	27
Ash content (%)	≤10	10
Rubber hydrocarbon content (%)	≥45	55
Acetone extractives (%)	≤8	7
Iron content (%)	≤0.05	0.002
Activation energy (100℃)	>1500	1540
Tensile strength (MPa)	≥15	22
Tensile elongation (%)	≥450	522
Heating reduction (%)	≤1.0	1.0

2.2. Activation Program' setting up

Rapeseed heavy oil pre-swelling has been proven to be an effective activation method. In the preparation of waste rubber powder modified asphalt, compared with the modified asphalt without pre-swelling, using the matrix asphalt pre-swelling method of modified asphalt performance is more stable, the rubber phase and asphalt phase do not easily occur separation [9]. Rapeseed heavy oil pre-swelling activation is through the rapeseed heavy oil on the waste rubber powder pre-mixing swelling treatment, to improve the surface activity of the waste rubber powder, and improve the compatibility between the waste rubber powder and asphalt. The specific activation scheme for this experiment is specifically named as shown in Table 4 below.

Table 4. Rapeseed heavy oil activation program

Activation mode	Oil-rubber ratio	Rubber powder mesh	No.
Unactivated group	-	40	Y4
		80	Y8
		60	Y6
Rapeseed heavy oil pre-swelling	1:3	40	C43
		60	C63
		80	C83
	1:2	40	C42
		60	C62
		80	C82
	1:1	40	C41
		60	C61
		80	C81

2.3. Test Methods

The set amount of rubber powder is 20%, to study the influence of rapeseed oil on the performance of waste rubber powder modified asphalt, in 20% of the total mass of the premise of replacing part of the waste rubber powder with rapeseed oil, that is, rapeseed oil and the waste rubber powder of the mass and the total quality of the 20% of the total mass. Rapeseed oil is viscous at room temperature, and needs to be heated to the full flow state to be better and fully dissolved with the gum powder, then the rubber powder and bio-oil should first be put into the constant temperature drying oven at 70℃ hot baking for 1h, to remove the effect of water in the raw materials on the test results as well as baking the rapeseed oil to the full flow state. Subsequently, the rapeseed oil was slowly mixed into the gum powder, and the mass ratios of rapeseed oil to rubber powder were controlled to be 1/3, 1/2, and 1/1 for mixing. The rapeseed oil should be added while mixing with a glass rod so that the two are mixed evenly. When mixing, pay attention to controlling the speed of rapeseed oil mixing, too fast will lead to rapid agglomeration of rubber powder to affect the activation effect. To be added to the rapeseed oil, continue to stir until the mixture is no obvious rubber powder group, at this time with plastic wrap sealed on the sample dish to prevent evaporation of the oil. Finally, use the constant temperature oven that has been set at 55-60℃ for heating and insulation, and control the heating time for 5 h. After that, also take the matrix asphalt 400 g, put it into the oven at 150℃ constant temperature heating 0.5 h to the state of full flow, and then the matrix asphalt in a constant temperature heating furnace heated to 180℃ and maintain a constant, and then rapeseed heavy oil activation of the rubber powder poured a small number of times into the matrix asphalt, and use glass to activate the rubber powder in the asphalt. Asphalt, fully stirred with a glass rod, will be completely poured into the powder with a glass rod stirring to the asphalt surface is no obvious powder agglomeration phenomenon, and then in the shear 3000r/min shear 1h, during the period of every 5min with a thermometer to measure the temperature, so that the temperature of the asphalt shear to maintain the asphalt temperature of 180℃ ±5℃, and then into the rapeseed heavy oil activation of modified asphalt with rubber powder in the 180℃ oven swelling Development of 1h, you can get rapeseed heavy oil activated gum powder modified asphalt specimens, a total of 9 kinds.

3. RESULTS

3.1. The Three Indicators

The softening point is a basic index to evaluate the high-temperature performance of asphalt binding material. The instrument used for the test is SYD-2806F, the heating speed is 5.0℃ ±0.5℃, and the medium is distilled water. Needle penetration is generally used to indicate the degree of viscosity of asphalt, the smaller the needle penetration of asphalt is the thicker the greater. This paper adopts the test instrument for Jiangsu Muyang SYD-2801F type automatic needle penetration meter, in the test temperature of 25℃ when test. The ductility of asphalt is when the asphalt is subjected to external tensile force, the total ability to withstand plastic deformation, can be used to measure the size of asphalt cohesion. This test uses a LYY-7A type asphalt ductility tester, the test temperature is 10℃, and the tensile rate of 5cm/min. The test results are shown in Table 5 and Figure 1-3.

Table 5. Test results for the three main indicators

Oil-rubber ratio	Rubber powder mesh	No.	Softening point(℃)	Needle penetration(0.1mm)	Ductility(cm)
-	40	Y4	67.8	46.9	9.1
	60	Y6	66.5	57.2	9.6
	80	Y8	62.5	58.9	10.5
1:3	40	C43	64.5	59.9	14.4
	60	C63	62.8	64.7	14.9
	80	C83	59.7	69.3	15.8
1:2	40	C42	62.2	61.4	15.3
	60	C62	60.7	67.7	16.9
	80	C82	56.4	71.5	17.8
1:1	40	C41	59.4	61.9	15.5
	60	C61	56.6	69.4	17.3
	80	C81	52.5	70.7	18.0

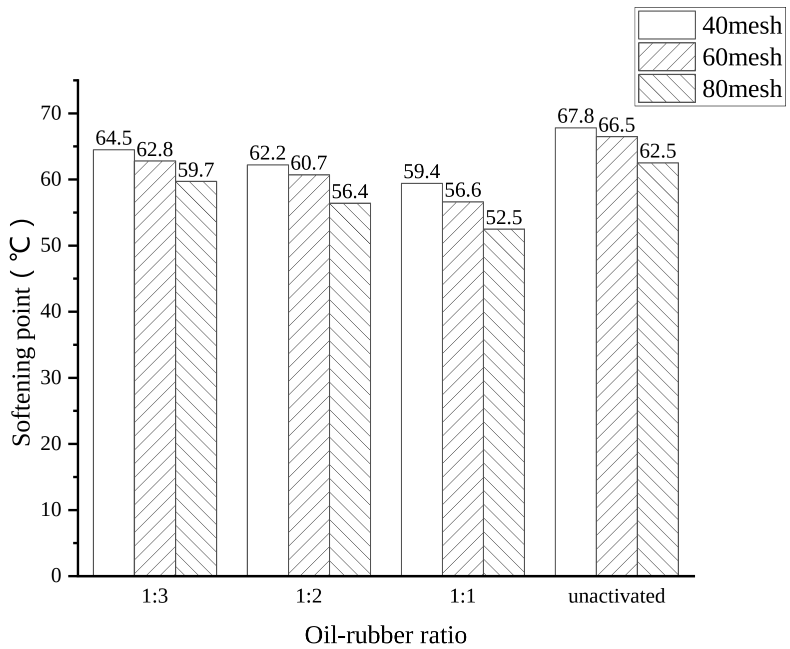

Figure 1. Softening point

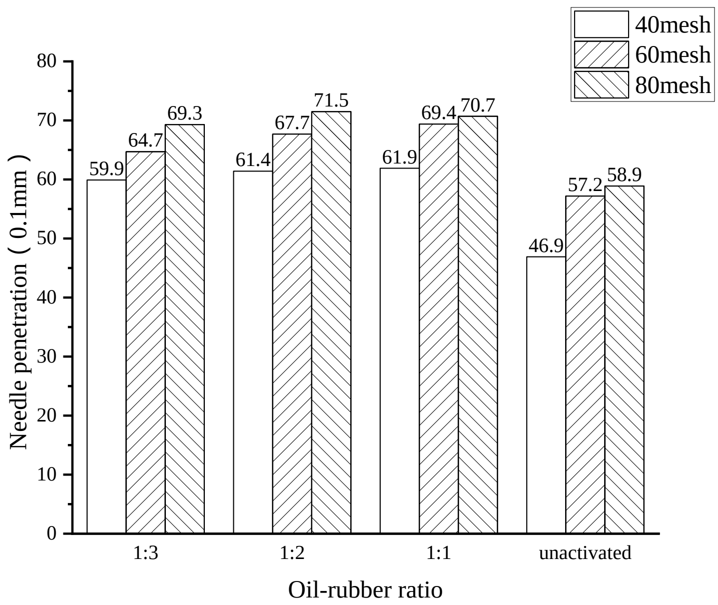

Figure 2. Needle penetration

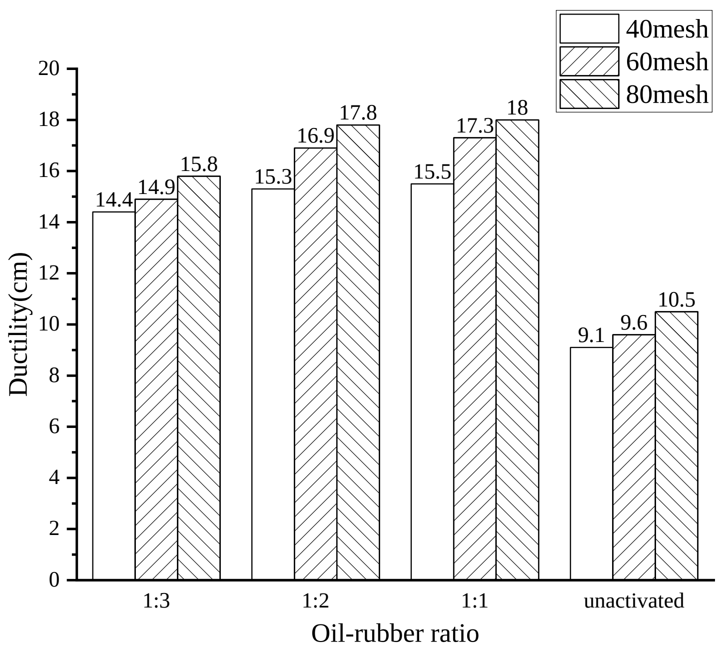

Figure 3. Ductility

From the data in Table 5, 40 mesh of waste rubber powder modified asphalt softening point is higher than 60 mesh and 80 mesh, this is because when the number of a mesh of waste rubber powder increases, the waste rubber powder and modified asphalt contact surface area increases at the same time, enhanced the role of waste rubber powder desulfurization and degradation of rubber powder particles resulting in the rubber powder particles become smaller in size, the rubber powder crosslinking between the weakening of the role of molecular force, and with the mesh number of the particles in the asphalt of waste rubber powder particles in the asphalt, it is difficult to form the structure of the backbone, so the softening point decreases [10]. Needle penetration with the increase in mesh and increase, and 40 mesh to 60 mesh growth rate is the largest, this is due to the absorption of lightweight components of the rate increases, making the waste rubber powder desulfurization and degradation rate increase, so that rubber asphalt rubber molecules in the chain of segments to increase the viscosity increases, asphalt soft, the needle penetration that is increased. Ductility also increases with the mesh number and improves, the reason is that with the further increase in the mesh number, the surface area increases, the waste rubber powder dissolution reaction becomes stronger, and desulfurization and degradation also becoming stronger, resulting in the waste rubber powder dissolved in asphalt increases the number of rubber asphalt, so that the rubber asphalt low-temperature flexibility enhancement, the larger the number of mesh dissolved in asphalt, the more the waste rubber powder, the ductility is to appear to increase [11].

Oil to rubber ratio of 1:3, with 40 mesh to 80 mesh softening point decreased by about 7%, the penetration rose by about 16%, and the degree of ductility rose by about 10%; oil to rubber ratio of 1:2, the softening point decreased about 10%, the needle penetration rose about 16%, the degree of ductility rose about 9%; oil to rubber ratio of 1:1, the softening point decreased by 12%, needle penetration rose by 14%, the degree of ductility rose by about 8%, indicating that with the increase in the number of mesh of the rubber powder, the group of rubber-oil ratio softening point decreased, the ductility increased, the needle penetration also increased and affected by the largest degree.

40 mesh powder with rapeseed heavy oil doping increases, softening point for the gum oil ratio of 1:3 were down 3%, 8%, the needle penetration of 5%, 3%, the ductility of 6%, 8%; 60 mesh powder softening point down 3%, 10%, the needle penetration of 5%, 7%, the ductility of 13%, 16%; 80 mesh softening point down 6%, 12%, the needle penetration of 3%, down 1%, the ductility of 13%, 14%. Up 13%, 14%. The reason is that when rapeseed heavy oil is added to the matrix asphalt system of lightweight components the proportion of the consequent increase in the mixing of rubber powder into the asphalt and lightweight components of the combination of more, in the asphalt dissolved more fully, reducing the overall consistency, so that the degree of penetration, elongation, but in the oil-rubber ratio added to 1:2, the proportion of lightweight components of the mixture to reach a certain amount of the rubber powder after the dissolution of the saturation, from the date of 1:1 oil-rubber ratio group of the needle penetration, ductility, and the degree of penetration. From the data of 1:1 oil-rubber ratio group penetration, ductility values and the oil-rubber ratio of 1:2 difference is small. Still, the softening point decreased more, so the oil-gum ratio of 1:2 for the optimal ratio.

In summary, to meet the basic performance requirements of the specification under the premise of 80 mesh rapeseed heavy oil activated waste rubber powder modified asphalt comprehensive compared to 40 mesh, 60 mesh softening point decreased less, and a small amount of needle penetration enhancement, the ductility of the enhancement of the more, the overall performance is better. Overall view with rapeseed heavy oil activated waste rubber powder selection of an oil-rubber ratio of 1:2 is appropriate [12].

3.2. Viscosity

Viscosity can not only be used as a metric to determine the amount of frictional resistance between internal molecules but also provide a corresponding macroscopic expression of the molecular weight of the material. When the temperature increases, the tendency of molecular movement increases, resulting in an increase in the free volume of internal molecules and a decrease in the intermolecular force, which ultimately leads to a decrease in the viscosity of asphalt. Generally speaking, the larger the molecular weight of the material, the greater the viscosity it has, making it more difficult to produce flow deformation, the material is less likely to change at high temperatures. In this paper, a 180℃ rotational viscosity test is chosen. The results are shown in Table 6.

Table 6. Viscosity test results

No.	Viscosity value at 180℃ (Pa·s)
Y1	1.94
Y2	2.63
Y3	3.05
C41	1.61
C61	1.91
C81	2.35
C42	1.69
C62	2.10
C82	2.86
C43	1.87
C63	2.47
C83	2.93

When the waste rubber powder is not activated, the unactivated rubber powder because of its poor swelling effect, the degree of dissolution is low, so the viscosity is at a high level. The smaller the particle size of the rubber powder-modified asphalt because of its larger surface area, the greater the viscosity. The average decrease in each mesh of waste rubber powder-modified asphalt after activation with rapeseed heavy oil was about 22%, and the highest average decrease was 55% compared with the unactivated waste rubber powder-modified asphalt.

In summary, in the total modifier for 20% of the dosage, the larger the mesh size of the rubber powder due to the larger specific surface area, the viscosity of the modified asphalt will be higher, and with the dilution of rapeseed oil, making the modified asphalt internal mobility is further improved, and ultimately fully dissolved rubber powder particles in the modified asphalt gradually formed a stable network structure.

3.3. Storage Stability

Waste rubber powder modified asphalt is a multi-phase mixing system composed of waste rubber powder and matrix asphalt, due to the large difference in molecular weight between the waste rubber powder and asphalt, so in the storage process, the waste rubber powder modified asphalt will be unstable, or even segregation and other phenomena.

In this paper, according to the regulation JTG E20-2011 T0661-2011 in the preparation of specimens, respectively, the asphalt injected into a diameter of 25mm, a length of 140mm aluminum tube, the quality of 50g, the open end of the aluminum tube pinched into a thin slice of folded twice and clamped with a clamp, and then the aluminum tube and the bracket together with the 163℃ oven static for 48h. After the end of the heating of the tube the bracket will be taken out, and then put into the refrigerator cooler static for 48 hours. After heating, the aluminum tube and holder were removed from the oven and placed in a refrigerator freezer for 4 hours to solidify. After taking them out of the freezer, when they were slightly softened, the upper and lower third of the aluminum tubes were taken as test specimens, and the softening points were tested and the differences were calculated. The results are shown in Table 7.

Table 7. Results of the segregation test

No.	Softening point difference (℃)
Y1	7.3
Y2	7.9
Y3	8.8
C41	2.9
C61	2.7
C81	3.5
C42	4.1
C62	3.8
C82	4.8
C43	5.8
C63	5.9
C83	6.4

From the experimental data in Table 7, it can be seen that there was a significant decrease in the softening point difference after the addition of rapeseed heavy oil, and the largest decrease in the softening point difference of 60 mesh waste rubber powder modified asphalt. In the segregation test, waste rubber powder modified asphalt in the material components will be slowly with the extension of time to the bottom of the sedimentation, resulting in the viscosity of the bottom specimen increasing, while the addition of rapeseed oil, waste rubber powder in the asphalt is more fully dissolved, the distribution of the more dispersed, the rate of sedimentation will be greatly reduced, the difference in the softening point is a substantial reduction.

In conclusion, after activation by rapeseed heavy oil, the separation softening point differences of the waste rubber powder modified asphalt were all significantly lower than those of the unactivated asphalt specimens. This phenomenon indicates that the activation treatment makes the swelling effect of waste rubber powder particles more adequate, and the compatibility between the asphalt is improved, thus improving the thermal storage stability of the modified asphalt, which is in line with the test expectations.

3.4. High-Temperature Rheological Properties

The rheological performance test of asphalt is mainly used to evaluate the physical properties of asphalt materials, especially their deformation and recovery ability under different temperatures and stress conditions. The results of asphalt rheological performance tests are of great significance for understanding the performance of various asphalt materials, predicting their performance in practical applications, and optimizing asphalt modification effects.

Research has shown that the anti-rutting performance of asphalt pavement is closely related to the high-temperature performance of asphalt materials, and conventional indicators cannot reflect the high-temperature mechanical properties of asphalt materials well. For example, the actual performance of matrix asphalt with the same grade may also have significant differences. Therefore, the famous American SHRP program combines performance indicators with the actual usage of asphalt materials to propose and develop a series of evaluation indicators based on rheological properties in response to this situation. In terms of high-temperature performance, a dynamic shear rheometer was used for testing. Due to the viscoelastic properties of asphalt, there is a certain delay in the strain force generated when loading the asphalt sample, which is called phase difference. The dynamic shear rheometer measures and calculates two important parameter quantities that can quantify this situation, namely complex modulus G^* and phase angle δ. By using a dynamic shear rheometer to measure, the behavior of asphalt at high working temperatures can be more accurately presented. This article uses the Anton Paar SamrtPave 102 dynamic shear rheometer as the experimental instrument. The experimental strain value is set to γ= 12%, the frequency of sinusoidal oscillation load as ω=10 rad/s, and a 25mm diameter flat plate was used to test each modified asphalt. The initial temperature is set to 58℃, with a temperature increase of 6℃/min, and a termination temperature of 82℃. Based on the experimental data from the previous chapter, select a group with 60 mesh and 80 mesh of waste rubber powder, and an oil-rubber ratio of 1:2 for rheological testing. The experimental data are shown in Table 8.

Table 8. Temperature scan results

No.	performance index	Temperature(℃)				
		58	64	70	76	84
Y2	G*(kPa)	25.824	12.171	6.975	4.581	2.69
	δ(°)	58.96	60.07	62.31	64.22	65.77
	G*/sinδ	30.140	14.044	7.877	5.087	2.950
Y3	G*(kPa)	18.334	9.21	4.762	2.918	1.676
	δ(°)	61.11	63.03	65.14	67.01	68.88
	G*/sinδ	20.940	10.334	5.248	3.170	1.797
C62	G*(kPa)	23.23	13.399	7.995	4.916	3.075
	δ(°)	59.79	61.86	64.12	66.73	68.08
	G*/sinδ	26.881	15.195	8.886	5.351	3.315
C82	G*(kPa)	15.914	7.239	4.389	2.714	2.072
	δ(°)	62.66	65.01	67.52	69.29	71.14
	G*/sinδ	17.915	7.987	4.750	2.901	2.190

Rubber-modified asphalt is a temperature-sensitive material, and its road performance changes with temperature, especially its high-temperature performance is more sensitive to temperature changes. The rutting factor is an important indicator to characterize the high-temperature resistance of modified asphalt to rutting. The rutting factor $G^*/\sin\delta$ can reflect the resistance of asphalt to high-temperature permanent deformation, and the larger its value, the stronger the asphalt's deformation resistance and better high-temperature performance.

According to the data in the table, the complex shear modulus values of each waste rubber powder-modified asphalt gradually decrease with the increase in temperature, and the complex modulus of each waste rubber powder-modified asphalt decreases significantly in the early stage of heating. After 70℃, the rate of decrease in the complex modulus of activated waste rubber powder-modified asphalt gradually slows down. Comparing the complex moduli of various modified asphalt, it can be found that the complex modulus of unmodified rubber powder modified asphalt is relatively large. However, as the complex modulus decreases with increasing temperature, the difference between the activated group and it gradually narrows. The G^* value of 80 mesh waste rubber powder modified asphalt is smaller than that of 60 mesh, indicating that the anti-flow deformation ability of 80 mesh waste rubber powder modified asphalt under load is slightly lower than that of 60 mesh. This is because too fine rubber powder makes it difficult to form a skeleton structure in the asphalt and is prone to flow deformation when the temperature rises [13]. After adding rapeseed heavy oil pre-swelling single activated waste rubber powder, the complex shear modulus value of the modified asphalt is slightly lower than that of the nonactivated waste rubber powder modified asphalt. This is because each rapeseed heavy oil contains oil and other light components, and the dilution and lubrication effect on the modified asphalt will also reduce its high-temperature deformation resistance. The composite shear modulus values of activated waste rubber powder modified asphalt are mostly lower than those of single activated waste rubber powder modified asphalt, but this difference gradually decreases with the increase of temperature; After adding rapeseed heavy oil, the activated rubber powder has better compatibility. At the same dosage, the solubility of the activated rubber powder is greater than that of the unactivated rubber powder. Therefore, the elastic content is reduced, resulting in an increase in the phase angle of modified asphalt at high temperatures compared to unactivated waste rubber powder modified asphalt. In other words, adding rapeseed heavy oil makes the modified asphalt more "sticky" at high temperatures and reduces its ability to resist deformation to a certain extent. After composite activation, the phase angle of waste rubber powder modified asphalt further increased, overall higher than that of nonactivated waste rubber powder modified asphalt. The rutting factor of modified asphalt after pre-swelling activated waste rubber powder with rapeseed heavy oil also decreased compared to that without activated waste rubber powder modified asphalt. This is because the solubility of the rubber powder after swelling treatment with rapeseed heavy oil is greater, and its ability to resist deformation is reduced, resulting in a decrease in the rutting resistance of the modified asphalt.

3.5. Low-Temperature Rheological Properties

The use of the low-temperature bending beam rheology test (BBR) as a test method for evaluating the low-temperature rheological properties of asphalt was proposed under the U.S. SHRP program. The BBR is used to determine the creep strength S and creep rate m of asphalt, which can be used to derive the performance of asphalt subjected to pavement stresses in a low-temperature environment. Creep strength S is a characterization of asphalt flexibility at low temperatures, the smaller the value of S, the better the asphalt flexibility at low temperatures, and the less likely to crack. Creep rate m is the asphalt stress dispersion capacity of the characterization, the larger the value of m, the better the asphalt stress dispersion capacity. For this experiment, an aluminum mold of 127mm×6.35mm×12.70mm was used to prepare the asphalt beamlet specimens required for the test, and the instrument model was PAVETEST Instrument Company Model B216 Bending Beam Rheometer. The test data are shown in Table 9.

Table 9. Low temperature test results

No.	Creep strength S(Mpa)			Creep rate m		
	-12℃	-18℃	-24℃	-12℃	-18℃	-24℃
Y2	73	158	271	0.388	0.330	0.259
Y3	68	149	265	0.382	0.322	0.263
C62	46	94	159	0.431	0.392	0.321
C82	39	90	157	0.442	0.395	0.332

From the data in the table, the bending strength of each modified asphalt with the decline in temperature and gradually increase, unactivated waste rubber powder modified asphalt bending strength from -12℃ to -18℃ an average of 117%, from -18℃ to -24℃ an average of 75%, and in each temperature is higher than the activation of the waste rubber powder modified asphalt, the bending strength of the rapeseed heavy oil single activation of the waste rubber powder modified asphalt an average of 68%, the composite activation of modified asphalt, an average of 134% higher. The bending strength is 68% higher than that of rapeseed heavy oil single activated waste rubber powder modified asphalt, and 134% higher than that of composite activated modified asphalt, which is due to the waste rubber powder in the asphalt not sufficiently dissolved without activation, the specimen rubber powder particles dispersed in the less parts of the cracking is prone to occur, which affects the overall bending strength value. After activation, the degree of desulfurization and degradation of the rubber powder is higher, the swelling effect in the asphalt is more adequate, and the rubber powder particles in the asphalt are more uniformly dispersed, so that the whole modified asphalt system is more flexible and stable at low temperatures.

Each waste rubber powder-modified asphalt with the decline in temperature creep rate is showing a downward trend. Unactivated waste rubber powder modified asphalt creep rate than activated waste rubber powder modified asphalt as a whole is smaller than the rapeseed heavy oil single activated waste rubber powder modified asphalt as a whole is about 20% lower, and unactivated 60 mesh and 80 mesh waste rubber powder modified asphalt at temperatures up to -24℃, the creep rate of similar. rapeseed heavy oil single activation of waste rubber powder than the composite way of activation of waste rubber powder modified asphalt as a whole is about 4% lower, indicating that the microwave activation at -12℃ and above the temperature of the creep rate of the influence of the smaller, lower temperatures, the use of rubber powder as a modifier could have been to improve the substrate asphalt stress relaxation capacity, reducing the possibility of low-temperature cracking [14]. The decrease in temperature decreases the creep rate of each activated waste rubber powder modified asphalt, but the creep rate of each modified asphalt is still larger compared to the unactivated waste rubber powder modified asphalt, i.e., their low-temperature stress relaxation ability is better than that of the matrix asphalt.

3.6. Micromechanical Studies

3.6.1. Infrared spectrum(IFTR)

Infrared spectroscopy is a commonly used method for chemical structure analysis, which combines the characteristics of clear characterization and high efficiency and convenience. Through infrared spectroscopy test, the distribution of functional groups of canola heavy oil activated waste rubber powder modified asphalt can be viewed, and then the effect of activation treatment on the internal structure composition can be investigated. The test results are shown in the figure4-6.

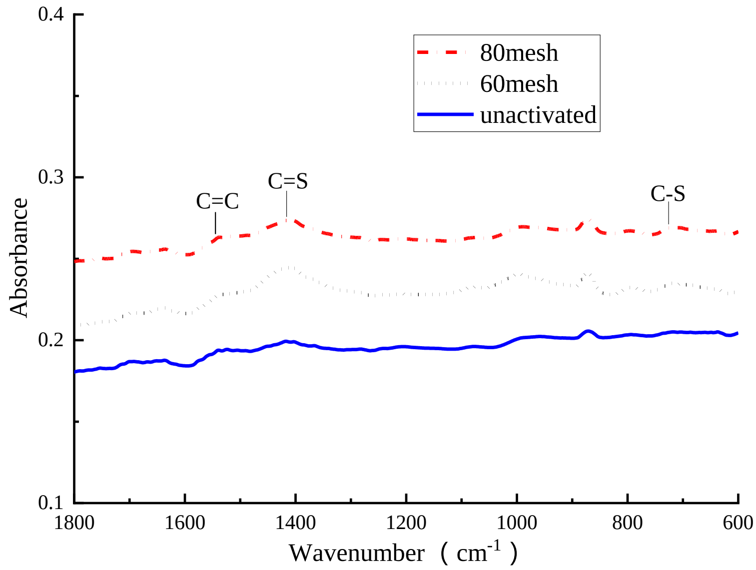

Figure 4. Infrared spectra of rubber powder

The infrared spectra of waste rubber powder treated by different activation methods are shown in 4. The spectra were plotted for the convenience of observation by intercepting the 1800~700 cm-1 wavelengths, in which the C=C bond was located near 1637 cm-1, the absorption peak of the C=S bond was located near 1350 cm-1, and the absorption peak of the C-S bond was located near 723 cm-1 [15].

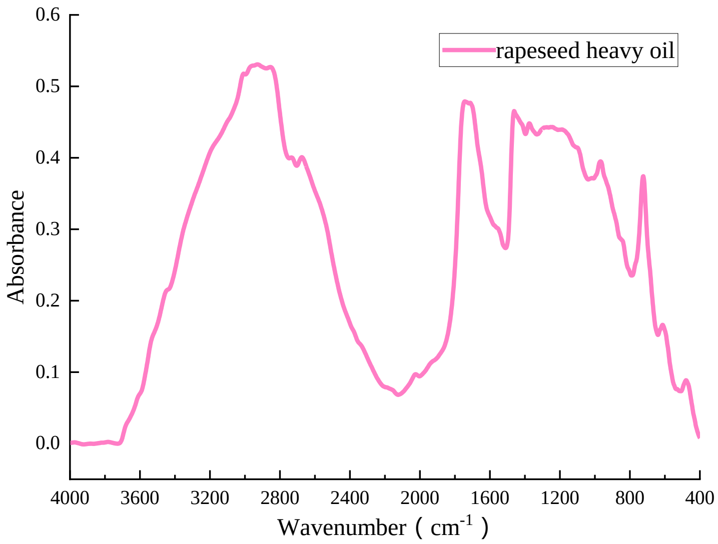

Figure 5. Infrared spectra of rapeseed heavy oil

The infrared spectral diagram of rapeseed heavy oil is shown in Figure 5, rapeseed heavy oil is a vegetable fat with fatty acids as its main component. Chemically, the oil consists of fatty acids and glycerides, and its molecular structure contains methyl, methylene, carbon-carbon double bonds, ester groups, and other groups, all of which have their characteristic absorption peaks in the infrared spectra. The ester group is the predominantly represented group in the lipid structure, which contains C=O and C-O-C structures. The absorption peak of the unsaturated C=C-linked unsaturated C-H stretching vibration near 3020 cm^{-1}, its vibration intensity, and its frequency range can reflect the structural information of the C=C and partially C=C-linked groups in the oils and fats. The absorption peaks between 2960 and 2840 cm^{-1} are symmetric stretching vibrations of saturated C-H. The absorption peaks near 1739 cm^{-1} are symmetric stretching vibrations of saturated C-H. The absorption peaks appear near 1739 cm^{-1} for the stretching vibration of C=O, 1465 cm^{-1} for the absorption peak of the aromatic compound N=O, 1395 cm^{-1} for the asymmetric bending vibration of $-CH_3$, and the distribution of the in-plane swaying vibration of $-CH_3$ is around 724 cm^{-1}, 965 cm^{-1} for the absorption peak of C=C, and 605 cm^{-1} for the absorption peak of C-H, suggesting the structure information of saturated C=C and some groups connected with C=C in rapeseed heavy oils. Peak around 965 cm^{-1}, and C-H absorption peak around 605 cm^{-1}, indicating that rapeseed heavy oil contains more light components.

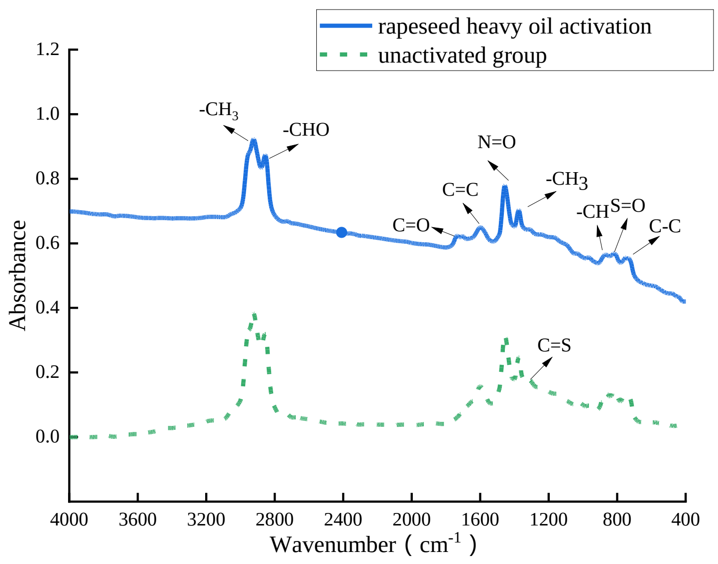

Figure 6. Infrared spectra of each modified asphalt of waste rubber powder

As shown in the figure, each modified asphalt has two strong absorption peaks located near 2945 cm^{-1}-2890 cm^{-1}. The absorption peaks generated in this interval belong to the absorption peaks of methyl and methylene groups, which are formed by the symmetric telescopic vibration of the C-H bond and the antisymmetric telescopic vibration of the C-H bond, indicating that all three kinds of asphalt contain $-CH_2$ and $-CH_3$. The absorption peaks appearing in the region near 2850 cm^{-1} are O=C-H functional group vibrational absorption peaks of the aldehyde group [16]. The absorption peaks appearing near 1600 cm^{-1} are the C=C stretching vibrations of olefins in thick ring aromatic hydrocarbons, indicating that unsaturated bonds are contained in each asphalt. The C-H absorption peaks corresponding to aromatic fractions and the N=O absorption peaks existed at about 1477 cm^{-1}-1356 cm^{-1}. This indicates that the complex activation is favorable for C=S fracture. At 871.5cm^{-1}, 754.7cm^{-1} is the formation of a C-C single bond with a carbon chain greater than or equal to 4. This absorption peak is the alkane portion of the bio-oil modified asphalt, and it can be known that the rapeseed heavy oil activation of the waste rubber powder modified asphalt contains the carbon long chain structure and benzene ring structure. The absorption peak near 817 cm^{-1} is the S=O stretching vibration of alkyl sulfoxide and aryl sulfoxide.

In the vicinity of 1320cm^{-1} for the C=S absorption peak, from the figure can be seen that the unactivated waste rubber powder modified asphalt in this peak is more obvious, a single activation group in the place of the absorption peak appears to decline, the composite way of activation group in the absence of a significant absorption peak, indicating that the composite activation of the C=S double bond absorption peak area of the reduction of a significant role. A new absorption peak appeared near 1735 cm^{-1}, and it only existed in the activation group with the addition of rapeseed heavy oil, which was the C=O vibration absorption peak in the lipid structure after the addition of rapeseed heavy oil. This functional group is mainly derived from fatty acids, so the activation group without rapeseed heavy oil has no absorption peak in this wave number range.

In conclusion, after the activation of the rubber powder with rapeseed heavy oil, the positions of the main absorption peaks in the infrared spectra of each modified asphalt are roughly the same, with different changes in intensity. The new emergence of several more obvious characteristic peaks is caused by the addition of components contained in rapeseed oil, i.e., there are no new functional groups generated by the activation treatment. Therefore, it can be judged that the activation of vegetable oil is physical activation, while microwave radiation activation interrupted part of the C=S. The modification mechanism of the activated rubber powder and matrix asphalt is still based on physical blending, and the process of canola heavy oil activation can be summarized as follows: in the pretreatment stage, the rubber powder absorbed a large number of components of vegetable oil, and some pre-dissolution occurred, and the sulfide structure was initially destroyed, and the surface has a certain degree of activity.

3.6.2. Scanning electron microscope (SEM)

Scanning electron microscopy (SEM) is a common method to observe the micromorphology of modified bitumen. The instrument used in this experiment is produced by FEI Company, Model: INSPECT F50. The specific manifestation of the activation effect can be obtained by observing the microscopic morphology of modified asphalt with unactivated waste rubber powder and modified asphalt with activated rapeseed heavy oil. The activation effect is shown in the figure7-8.

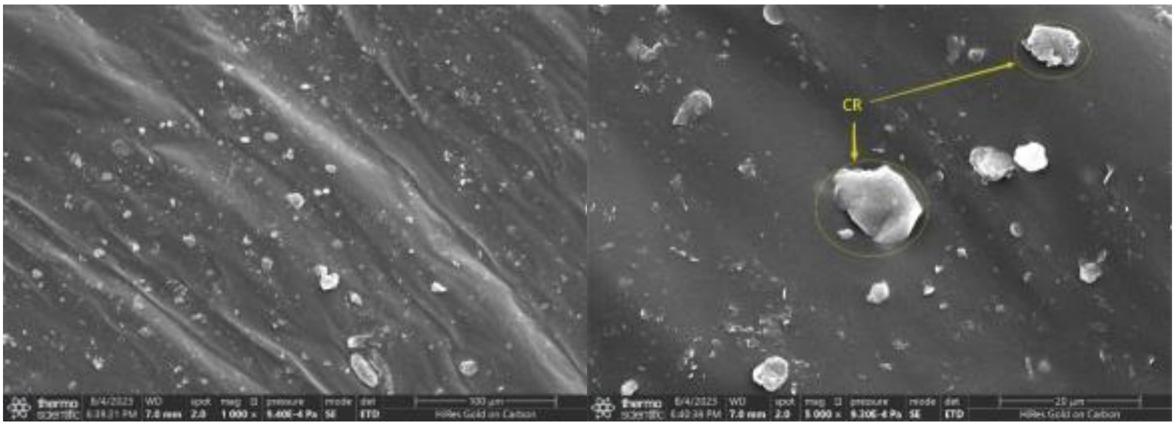

Figure 7. Unactivated group

Taking 80 mesh unactivated waste rubber powder modified asphalt as a representative, CR (crumb rubber) for rubber powder abbreviation. As can be seen from the left figure, the waste rubber powder has a large number of sheared small particles in the asphalt surface, not fully integrated into the asphalt, indicating that the rubber powder in the asphalt is not fully dissolved, rubber powder and asphalt poor combination of research shows that the increase in the folds will limit the mobility of asphalt, improve the viscoelasticity of the interface of asphalt, to prevent it from cracking or rutting [17]; from the right figure, we can see, the surface of the unactivated rubber powder is smoother, there is no obvious From the figure on the right, it can be seen that the surface of the unactivated rubber powder is smooth, without obvious open pore structure, and most of it maintains the original structure and volume before entering the asphalt, which is not conducive to the effective dissolution and dispersion of the rubber powder in asphalt [18], and part of the rubber powder is also adhered together to form a larger rubber powder cluster, resulting in a higher viscosity at the macroscopic level and easy to segregation problems.

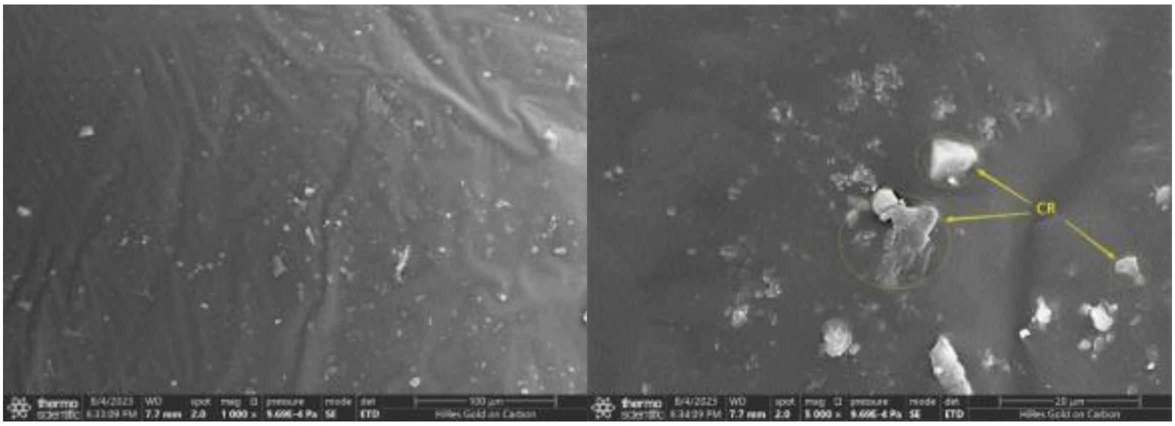

Figure 8. Rapeseed heavy oil activation group

Compared with the left figure and the unactivated group, the waste rubber powder is better compatible with the asphalt after being wrapped in rapeseed heavy oil, and the undissolved part can also be covered by the asphalt, this is because the proportion of the light component of the system is increased after the mixing of asphalt and rapeseed heavy oil, which indicates that the addition of rapeseed heavy oil makes the waste rubber powder more uniformly distributed in asphalt, which means that the modified asphalt has a lower viscosity. The right figure can be seen compared the surface of the modified asphalt without activation of the rubber powder, adding rapeseed heavy oil after the compatibility of the rubber powder and asphalt increased significantly, and some not completely dissolved in the rubber powder particles are not intact on the contact surface, and the contact area of the matrix asphalt increased significantly, which is advantageous for the waste rubber powder adsorption with the asphalt phase, and by the activation of the heavy oil of rapeseed rubber particles on the surface of the flocculent edge of the softer, the rubber powder surface is more The surface flocculent edge of rubber particles activated by rapeseed oil is softer, and the surface of rubber powder is more fluffy, which indicates that after adding rapeseed oil for pre-swelling, the newly formed numerous internal voids can absorb more lightweight components and enhance the swelling effect.

3.6.3. Fluorescence microscope

Fluorescence microscopy is more commonly used in rubber asphalt research, the technique is to use the rubber particles and asphalt in the fluorescent light source irradiation under the principle of displaying different colors, in the fluorescence microscope can be clearly distinguished between the rubber phase and the asphalt phase, so that you can observe the morphology and structure of the rubber phase in the asphalt phase. Rubber asphalt belongs to a strictly immiscible binary system, through the fluorescence microscope can be seen that the rubber particles are dispersed in the continuous asphalt phase, and these microstructures are often different [19]. The activation effect is shown in the figure9-10.

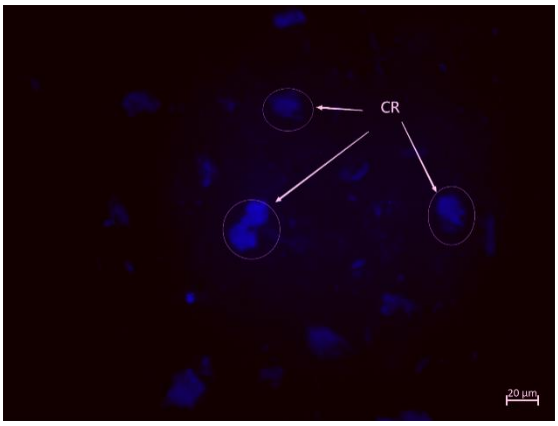

Figure 9. Unactivated group

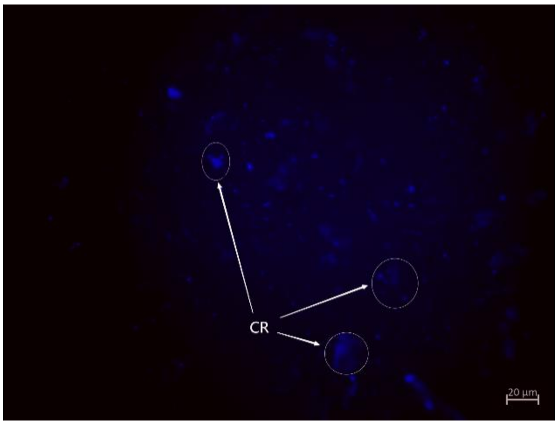

Figure 10. Rapeseed heavy oil activation group

As can be seen in Figure 9, the unactivated waste rubber powder modified asphalt in the rubber powder particles are larger, and part of the agglomeration, uneven distribution in the asphalt phenomenon, which indicates that the degree of dissolution of the waste rubber powder in the asphalt is low, the stability of modified asphalt has an impact. After the activation of rapeseed oil pre-swelling, as shown in Figure 10, compared to the unactivated group of waste rubber powder particle size reduction, and no longer agglomerated together, the distribution is more dispersed, after the swelling of the rubber powder surface area increases, and the asphalt contact area is larger, which is conducive to the fusion with the asphalt.

3.6.4. Thermogravimetric experiment(TGA)

Bitumen has a complex composition with no fixed melting point, and its viscoelastic properties are characterized by a physical state that changes with temperature. This will lead to the internal components of asphalt at different temperatures, the state will be different, that is, the aggregation state changes. This change affects the composition and macroscopic physicochemical properties of the bitumen, resulting in changes in its properties, especially its thermodynamic stability. Differential calorimetric scanning test can be tested in the warming process, asphalt specimens due to temperature changes in the physical state of the changes generated to assess the degree of change in the internal components and physical properties of asphalt, to evaluate the thermodynamic stability of asphalt. In this paper, the test uses PerkinElmer's STA 600 thermogravimetric analyzer test samples, the test temperature of 30 ~ 800℃, the heating rate of 20℃/min, and the atmosphere gas for nitrogen. The test results are shown in Figures 11-12.

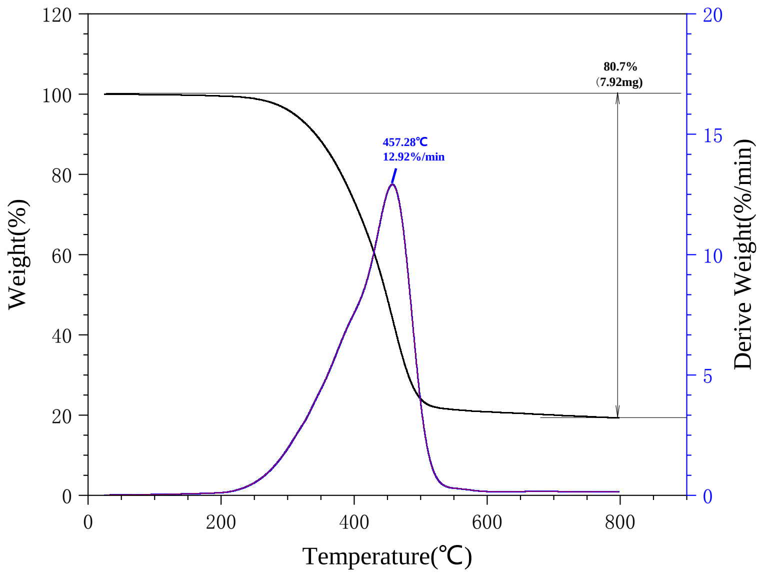

Figure 11. Unactivated group

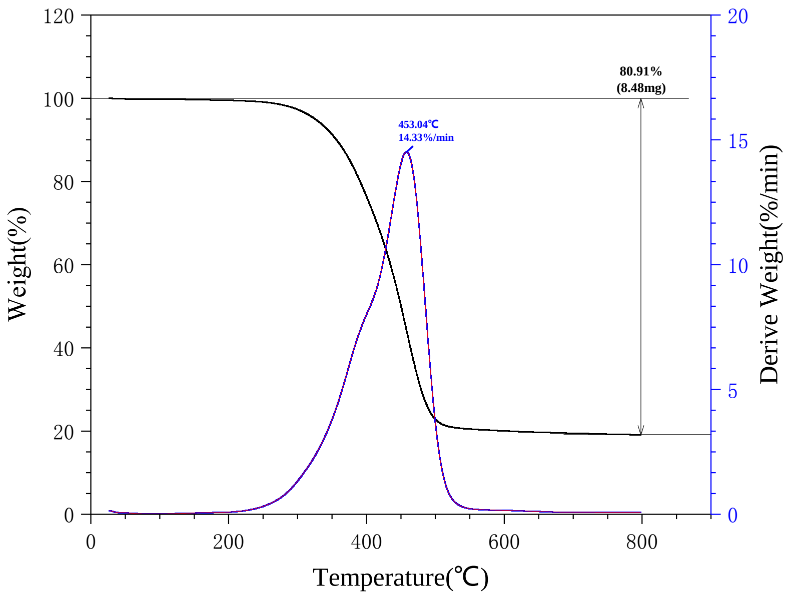

Figure 12. Rapeseed heavy oil activation group

From the above results, it can be seen that there are three main stages of zero pyrolysis, vigorous pyrolysis, and slow pyrolysis in the pyrolysis process of asphalt [19]. From room temperature to 250℃

is the zero pyrolysis stage, when the heating rate is slow, the TGA curve and DTG curve are roughly horizontal, the asphalt quality change is small, and the weight loss rate is almost zero. The intense pyrolysis stage occurs between 250℃ and 500℃, where the TGA curve decreases sharply, the DTG curve rises gradually with localized heat absorption and exothermic peaks, and the weight loss rate increases sharply.

The maximum rate of weight loss was reached at 457.28℃ for the unactivated waste rubber powder-modified asphalt and at 453.04℃ for the single activated waste rubber powder-modified asphalt with rapeseed heavy oil. The maximum weight loss rate indicates that the asphalt is extremely unstable in this temperature interval, and the quality loss is large. The slow pyrolysis stage occurs at 500℃ ~ 800℃, the TGA curve and DTG curve gradually tend to flatten, and the thermo-oxidative decomposition is also gradually stabilized. Reach the maximum weight loss rate of the temperature size ranking for the unactivated group> rapeseed heavy oil activation, indicating that the addition of rapeseed heavy oil on the asphalt high-temperature performance has a negative impact on the modified asphalt to reach the maximum weight loss rate of the temperature drop; and the residual percentage of the size ranking for the unactivated group > rapeseed heavy oil, a part of the rapeseed heavy oil with the increase in temperature and gradually volatilized. Before the weight loss of 20%, activated waste rubber powder modified asphalt at the same weight loss percentage of the temperature is higher than the unactivated waste rubber powder modified asphalt, indicating that the modified asphalt system in this temperature range of temperature sensitivity is lower, the overall more stable.

4. SUMMARY

In this paper, rapeseed heavy oil was used to activate the waste rubber powder pretreatment, and its activated modified asphalt performance was studied in depth, respectively, from the macro-micro level to investigate the effect of different activation methods on the performance of modified asphalt. The main conclusions are as follows:

(1) A rubber-oil ratio of 2:1 and a rubber powder mesh of 80 mesh were identified as the preferred activation group. After activation by rapeseed heavy oil, the ductility of waste rubber powder-modified asphalt was significantly increased, but the softening point was reduced, which had a greater effect on reducing the viscosity of rubber powder-modified asphalt in each mesh and also helped to reduce the segregation softening point difference.

(2) The results of rheological property analysis showed that the rutting factor and stress recovery ability of waste rubber powder-modified asphalt decreased after activation by rapeseed heavy oil, indicating that the high-temperature performance of asphalt was weakened to some extent. However, this activation can significantly improve the low-temperature flexibility of asphalt and increase its low-temperature deformation resistance.

(3) In the microcosmic level of the waste rubber powder modified asphalt microcosmology and physical and chemical indexes of the multi-angle observation, rapeseed heavy oil activation can significantly enhance the degree of rubber powder in the asphalt solubility, so that both activation modes contribute to the improvement of the rubber powder dispersion in the asphalt. Thermogravimetric analysis showed that the addition of rapeseed heavy oil improved the stability of the bitumen before 20% weight loss, while microwave activation reduced the mass residue percentage of the modified bitumen.

CONFLICTS OF INTEREST

The brand names mentioned in this paper were for the reader's convenience only and they do not suggest any endorsement by the authors. The authors declare no conflict of interest.

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