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Preparation and properties of SBS / crumb rubber composite modified asphalt (SBS / CRCMA)

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Abstract

In view of the characteristics of drought, little rain and large temperature difference in Xinjiang, this paper takes SBS / CRCMA as the research object, and determines the optimal dosage of different components and their respective influence degree through orthogonal test. Taking SBS modified asphalt (SBSMA), crumb rubber modified asphalt (CRNMA) and SBS / CRCMA as the research objects, the storage stability of composite modified asphalt was analyzed by softening point difference method. The high and low temperature properties of SBS / CRCMA were evaluated by dynamic shear rheometer (DSR) and low temperature bending rheometer. The modification mechanism was analyzed by gel permeation chromatography (GPC) and Fourier transform infrared spectroscopy (FTIR). The test results show that : refining : towering = 6 : 4 ; sBS content 4% ; rubber powder content of 15% ; the optimum dosage of furfural oil is 4.5%, which has good storage stability and good high and low temperature performance. SBS, rubber powder and asphalt are mainly physically blended.

1 Introduction

With the further development of China 's economy and society, traditional pavement materials are difficult to meet the national development requirements of ' green, environmental protection, economy and conservation ' [1]. The commonly used modified asphalt in the market is mainly SBS modified asphalt and rubber powder modified asphalt, but there are also problems of high price and high viscosity [2].

In order to improve the performance of SBS asphalt, in recent years, many scholars have combined the characteristics of SBS and rubber to analyze its preparation process, material parameters, rheological properties and microscopic mechanism. He et al. [3, 4] determined the preparation and material parameters of modified asphalt by orthogonal design and grey correlation analysis. Yuan Dongdong et al. [5] studied the low temperature sensitivity of high viscosity asphalt, SBS modified asphalt and rubber asphalt through low temperature sensitivity test. The test showed that the high viscosity asphalt had the lowest temperature sensitivity and the best low temperature stability. Zhou Zhigang et al. [1, 6] analyzed the mechanism of rubber powder / SBS high viscosity composite modification by infrared spectroscopy (FTIR) and differential scanning calorimetry (DSC). The test results showed that the modifier, high viscosity agent and matrix asphalt were mainly physical blending. In order to further study the relationship between structure and performance of modified asphalt, Liang [7] used dynamic shear rheometer (DSR), bending beam rheometer (BBR) and fluorescence microscope (FM) to analyze and simulate the phase state, viscoelasticity and phase structure separation process of multiphase structure by phase field theory, phase field model and numerical simulation.

2 Raw materials and test methods

2.1 Raw materials and preparation process

This paper takes SBST6302 produced by Xinjiang Dushanzi Petrochemical Company as the research object. Asphalt is blended by Kelian 90 # asphalt and Tahua 60 # asphalt. The main indexes of asphalt materials are shown in Table 1. Other additives are : furfural oil, sulfur.

Table 1
Basic Properties of Asphalt

Asphalt Type	Penetration (25°C, 0.1 mm)	Softening Point (°C)	15° Elongation (cm)	RTFOF		
				Mass loss (%)	15° elongation (cm)	Penetration (25°C, 0.1 mm)
KL-90#	91.1	46.5	>100	0.08	26.7	62.6
TH-60#	64.3	57.8	>100	0.09	40.1	42
Through a large number of literatures and previous experimental studies[8–13], the preparation process was determined as follows :						
Step 1 : Mix a certain proportion of two kinds of asphalt, add a certain amount of SBS to preheat at 150°C for 15 min ;						
step 2 : Add rubber powder, quickly adjust the temperature knob to 180°C, use the glass rod to stir for 45 min, then move it to the stirrer for stirring, stirring time 2h ;						

step 3 : Place the oven and develop at 180°C for 2h.

2.2 Test Methods and Schemes

(1) Orthogonal design. Considering the complexity of the influencing factors, this paper takes refining : towering percentage, SBS, rubber powder and furfural oil as the research objects, establishes 4 × (3 − 1) + 1 = 9 combinations, and preliminarily determines the content and influence degree of each factor.

(2) Rheological test. DSR was used for temperature scanning test and multiple stress creep recovery (MSCR) test. When the temperature scanning is determined by consulting the literature and the previous test, the strain is set to 6%, the scanning temperature is 46°C ~ 82°C, and the step length is 6°C. The loading stress of MSCR was 0.1 kPa and 3.2 kPa, respectively. Among them, the cycle of loading stress 0.1 kPa applied to the asphalt sample is 20 times, and the cycle of subsequent stress is 10 times, a total of 30 times. Generally, the first 10 cycles are used as the pretreatment of asphalt binder, not as the final consideration. In a cycle, the stress time on the asphalt material applied by the instrument is 10 s, of which the initial stage is the loading time and the time is 1 s ; in the later stage, the unloading recovery stage is entered, the time is 9s, and the test temperature is set to 64°C.

The bending creep stiffness tester (BBR) was used to evaluate the modified asphalt according to the traditional evaluation method of low temperature performance. The test temperature was-18 ~ 36°C.

(3) Micro-test. Gel permeation chromatography (GPC) and infrared spectroscopy (FTIR) were used to compare matrix asphalt, SBSMA, CRMA and SBS / CRCMA, and the modification mechanism of

composite modified asphalt was analyzed.

2.3 Research method of modification mechanism

The gradation of fine grade material was optimized by orthogonal test design method, and the composite modification mechanism of asphalt was studied[14–19]. Chen Yuanzhong [20]used rubber powder, SBS and other polymer modified asphalt, which can effectively enhance the high temperature and low temperature stability of asphalt. Liu Pengfei[21]used silane coupling agent KH550 and diatomite to modify gneiss asphalt mixture. Combined with orthogonal test, the optimum content of KH550 and diatomite and the optimum oil-stone ratio in KH550 / diatomite composite modified gneiss asphalt mixture were determined. The high and low temperature and water stability of medium-acid gneiss asphalt mixture were studied, and its road performance was evaluated by dynamic modulus test, semi-circular bending test and freeze-thaw splitting test. Aiming at the activation grafting pretreatment of rubber-plastic alloy modified asphalt, He Chao[22]analyzed the influence of different pretreatment methods on the rheological behavior of rubber-plastic alloy modified asphalt. The microstructure and modification mechanism were analyzed by infrared spectrometer (FTIR) and fluorescence microscope (FM). Zhao Bin[23]used dynamic shear rheological experiment (DSR), bending beam rheological experiment (BBR), gel chromatography experiment (GPC), Fourier transform infrared spectroscopy (FT-IR) and other analytical methods to explore the high and low temperature rheological properties of SBS modified asphalt regenerated by bio-oil / biochar composite, the molecular weight distribution and chemical properties of each asphalt.

3 Performance characterization of modified asphalt

3.1 Rrthogonal Design

The results of orthogonal test are shown in Table 2.

Table 2
Orthogonal Design Table

No.	g/t Mixing Ratio	SBS content (%)	Rubber powder content (%)	Furfural oil content (%)
1	6:4	3	9	2.5
2	5:5	3.5	11	2.5
3	4:6	4	13	2.5
4	4:6	3.5	9	3.5
5	5:5	3	13	3.5
6	6:4	4	11	3.5
7	6:4	3.5	13	4.5
8	5:5	4	9	4.5
9	4:6	3	11	4.5

Table 3
Results of the orthogonal experiment

No.	Penetration	Softening Point	5°C Ductility	48-hour Segregation Value
1	75.7	63.3	25.6	4.2
2	57.6	85.8	29.6	1.6
3	51.4	95.8	26.3	0.7
4	63.3	86.2	34.5	0.9
5	62.8	82.8	32.2	1.3
6	69.9	72.8	38.9	1.1
7	75.1	73.4	26.7	0.6
8	73.1	79	47.7	1.4
9	73.5	84.1	28	0.6

It can be seen from the literature that the commonly used refined asphalt in Xinjiang has the disadvantages of poor high temperature performance and difficulty in modification. From the test results and range analysis, it can be concluded that the best formula of SBS / rubber powder CRCMA suitable for Xinjiang is : refining : towering = 6:4; SBS content 4% ; rubber powder content of 15% ; furfural oil 4.5%.

Range analysis is a commonly used analysis method in orthogonal design. Its basic principle is to calculate the average value K at different levels of a certain factor. The difference between the maximum

average value Kmax and the minimum average value Kmin reflects the degree of influence of this factor on specific indicators. The test results are shown in Fig. 1.

The storage stability of modified asphalt multiphase polymer has always been the focus of research at home and abroad. In China 's specifications[24–25], the difference of softening point between the upper and lower sections of the segregation tube is less than 2.5°C to evaluate the storage stability of multiphase polymer. It can be seen from Fig. 1 that the blending ratio has the greatest influence on the softening point and the segregation value, and the ductility is mainly affected by the SBS modifier and furfural oil, which is consistent with other scholars ' research. The blending ratio of the two asphalt binders and the furfural oil content have the same effect on the penetration.

A large number of studies have shown that it is difficult to evaluate the performance index of asphalt only by penetration system. Due to the complex composition of asphalt, in the process of practical application, asphalt pavement shows more viscoelastic properties. Therefore, the rheological test of asphalt is helpful to reflect the mechanical properties of asphalt more accurately. In this paper, the rheological tests of blended asphalt, SBSMA, CRMA and SBS / CRCMA were carried out by dynamic shear rheometer (DSR) and bending beam rheometer (BBR) based on viscoelastic principle.

The investigation shows that when the vehicle passes through the road surface, the road surface is subjected to a dynamic and changing load and undergoes a compression-tension process in a short time.

The working principle of the dynamic shear rheometer is to place the prepared asphalt sample between the oscillating plate and the fixed plate. After the instrument is started, the oscillating plate rotates, first from point A to point B, then through point B to point C, and finally back to point A is a cycle. The temperature scanning simulates the mechanical properties of the road surface at the same speed and different temperatures. The rotation frequency of the vibrating plate can be set according to the need. When the temperature scanning is carried out, the specific parameters are shown in Table 4.

Table 4
Temperature Scan Parameter Settings

Test Temperature	Temperature Gradient	Loading Strain	Parallel Plate Diameter	Specimen Thickness
46°C–82°C	6°C	6%	25 mm	2 mm

3.2 High Temperature Rheology

In this study, the test results of DSR were analyzed by rutting factor, complex modulus and phase angle. The test results are shown in Fig. 2.

Rutting factor represents the ability of asphalt binder to block unrecoverable deformation under temperature stress. The larger the value is, the better the high temperature performance is. It can be seen from Fig. 2(a) that when the temperature is low, the rutting factors of the four asphalts are relatively

close. With the increase of temperature, the four kinds of asphalt decreased linearly. Among them, SBS / CRCMA was always greater than the other three kinds of asphalt. The specific performance was SBS / CRCMA rut factor > CRMA > blended asphalt > SBSCRMA. With the increase of temperature, the composite modified asphalt decreased less than the other three. The reason is that under high temperature conditions, the ability of asphaltene to wrap aggregate is weakened, and the colloid attached to the asphaltene diffuses to the surrounding under high temperature conditions, which enhances the fluidity of asphalt.

The rutting factor can be divided into complex modulus and phase angle. Complex modulus and phase angle are usually used to evaluate the high temperature ability of asphalt, as shown in Fig. 2 (b). The line and dotted line represent the complex modulus and phase angle of SBSMA, CRMA and SBS / CRCMA, respectively, as shown in Fig. 2. It can be seen from the figure that no matter what kind of asphalt, as the temperature rises, the complex modulus generally becomes smaller. At the same time, it can also be seen that the complex modulus of SBSMA is greater than that of blended asphalt, indicating that the addition of SBS reduces the high temperature performance of blended asphalt.

The phase angle is calculated by the storage modulus G ' and the loss modulus G ". The smaller the phase angle is, the smaller the proportion of the loss modulus is, the lower the viscosity proportion is, and the better the ability to resist high temperature is. It can be seen from the figure that as the temperature increases, the phase angles of the four modified asphalts increase continuously. The phase angles of blended asphalt and SBS modified asphalt are much larger than those of CRMA and SBS / crumb rubber composite modified asphalt, which are close to elastic materials, indicating that crumb rubber can effectively improve the elastic response of asphalt binders compared with SBS.

3.2.1 MSCR Test

It can be seen from Fig. 3 that (1) In a cycle, the stress is continuously ' loaded-unloaded '.Whether it is blended asphalt or modified asphalt, as the temperature increases, the strain continues to circulate in the form of ' creep-recovery ', but the overall strain continues to increase. (2) Under the two stress conditions, the strain from large to small is SBSMA, blended asphalt, CRMA, SBS / CRCMA, indicating that SBSMA has higher flow deformation characteristics, which is very unfavorable for asphalt pavement to resist high temperature deformation. (3) From the diagram, it can be seen that whether it is blended asphalt, SBSMA, CRMA, SBS / CRCMA, when 0.1 kPa enters 3.2 kPa, the strain changes at a certain point. At 0.1 kPa, CRMA and SBS / CRCMA have obvious creep recovery stage, while the recovery curve of blended asphalt and SBSMA is not obvious, which indicates that the creep recovery ability of CRMA and SBS / CRCMA is greater than that of blended asphalt and SBSMA.

3.2.2 Storage Stability

Cole-Cole diagram is composed of storage viscosity η " as abscissa, unrecoverable viscosity η " as ordinate, namely η " -η " relation curve. Research[26]shows that η " -η " relation curve can be used to

characterize the compatibility of polymer modified asphalt, and the more symmetrical the curve is, the better the compatibility is.

From Fig. 4, it can be seen that whether it is rubber powder modification or SBS / CRCMA, it rises in a straight line, and there is no arc shape. The reason is due to the addition of rubber powder, which greatly improves the elasticity of modification, that is, within the measured range, the sample is not completely relaxed. In the range of 60°C, the blended asphalt has only one arc, and there is a trend of 'small tail 'at the end of the arc. As the temperature increases, this trend becomes more obvious. It shows that the phase structure of the blended asphalt is uniformly dispersed at a lower temperature, and phase separation occurs as the temperature increases.

3.3 Low temperature Rheologic Behavior

The low temperature rheological properties of 3-minute modified asphalt were analyzed by creep stiffness and creep rate obtained by low temperature rheometer.

3.3.1Creep stiffness modulus and creep stiffness change rate

It can be seen from Fig. 5 that with the decrease of temperature, the stiffness modulus S increases and the change rate of creep stiffness decreases, whether it is blended asphalt or single modified asphalt or composite modified asphalt. When the test temperature is-24°C, the S value of the blended asphalt is 493, which is far greater than the requirement of the specification < 300. At the same time, it can be seen that whether it is S or m, SBSMA has the best low temperature performance, followed by SBS / CRCMA, and CRMA is the worst. Analysis of the reasons, as the temperature decreases, the chain segment molecules in the asphalt component will quickly freeze, the asphalt begins to harden, the elastic ratio in the asphalt becomes larger and larger, the viscous component decreases, and the internal ultimate tensile strength of the asphalt material decreases. Therefore, it is easy to break[27].

2.3.2 low-temperature Susceptibility

Research[28] indicates that the creep stiffness index can characterise the low-temperature sensitivity of bitumen, and its calculation formula is:

$$\lg S = S_{TS} T + C$$

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In the formula, STS— the creep stiffness index obtained by using the test equipment during the test ; T— test temperature (°C) ; C— Regression coefficient. The regression equation is shown in Fig. 6.

It can be seen from Fig. 6, (1)The creep stiffness indexes of SBSMA, CRMA, SBS / CR composite modified asphalt and blended asphalt are 0.04912, 0.5517, 0.06037 and 0.03574, respectively. The creep stiffness index of the four modified asphalts varies with temperature from fast to slow : CRMA, SBS / CR

composite modified asphalt, SBSMA, blended asphalt, which is similar to the results obtained by m / S. The larger the creep stiffness index, the stronger the low temperature sensitivity and the worst the low temperature stability. (2)From (1), it can be seen that the low temperature stability of blended asphalt is the best, and the stability of SBS / CR composite modified asphalt is the worst, which is opposite to the high temperature sensitivity, indicating that SBS and CR modifiers have strong sensitivity to low temperature and poor sensitivity to high temperature.

4 Mechanism analysis of modified asphalt

4.1 Gel permeation chromatography (GPC)

The separation principle of gel permeation chromatography (GPC) is as follows : when the sample solution passes through the chromatographic column, it is separated due to the different molecular size. The internal pore size of the gel in the chromatographic column is small, which is only suitable for the passage of molecules with small molecular weight. The speed is slow, and the macromolecules can only pass through the gap of each gel particle, so the leaching time is short. Studies have shown that[29], the molecular weight and distribution of asphalt have an important influence on the performance of asphalt, such as ductility, viscosity, softening point and temperature sensitivity. The test results are shown in the table.

Table 5
GPC test results for different modified bitumens

Asphalt Type	Number-average molecular weight Mn	Weight-average molecular weight	Peak molecular weight Mp	Coefficient of Polydispersity (d)
SBSMA	525	1624	565	2.9689
CRMA	548	1626	624	2.9689
SBS/CRCMA	588	1743	597	3.0302
Blended bitumen	493	1493	548	3.0959

It can be seen from the above table that the number-average molecular weight and the peak molecular weight have the same change rule. The number-average molecular weight of rubber powder is the largest, followed by SBSMA and SBS / CRCMA, and the blending asphalt is the smallest. Analysis of the reasons, studies have shown that[13], the greater the relative mass of the molecule, the greater the resistance generated by the external load, indicating that its high temperature performance is better. This is consistent with the data obtained from conventional indicators, that is, CRMA and composite modified asphalt are the best, and SBSMA and blended asphalt have the worst high temperature performance. According to the literature, the smaller the dispersion coefficient, the more concentrated the molecular weight, that is, the more asphalt components in a certain range, indicating that there is a agglomeration

phenomenon inside the asphalt structure. When the temperature increases, the interval absorbs more energy, and the components in the asphalt are easy to deform, so the temperature sensitivity is poor and the high temperature resistance is poor. The larger the dispersion coefficient is, the smaller the molecular weight is in a certain range, the less the asphalt component is, the less the number of molecular phase changes is, and the lower the temperature sensitivity is. This also shows from another perspective that the addition of modifier is beneficial to improve the high temperature performance of blended asphalt.

4.2 Infrared Spectrum

It can be seen from Fig. 7 that there is no characteristic peak in SBSMA, CRMA and SBS / CRCMA, only the area of some characteristic peaks is different. At the same time, it can be seen from the figure that when the composite is modified, both the functional group area and the fingerprint area are smaller than the single modified absorption peak, which is caused by the addition of modifier to reduce the relative content of asphalt[30]. The results show that whether it is SBSMA, CRMA, or SBS / CRCMA. The modification mechanism is similar to that of physical blending reaction. The properties of SBS / CRCMA are closely related to the swelling degree of modifier.

In order to further evaluate the modification of four kinds of asphalt, based on Lambert-Beer 's law, OMNIC software was used to preprocess them, including data smoothing, baseline correction and so on. Then, the peak area was calculated according to the preprocessed spectrum ; finally, the index of functional groups is obtained. There are four commonly used benchmarks : A1.650 ~ 1400cm-1 ; A2.1400 ~ 4000cm-1 ; A3.650 ~ 4000cm-1 ; A4.2920 and 2850[31].

Through research[31], it is found that in the evaluation of asphalt aging, it is more accurate to use 4000 ~ 650cm-1 to evaluate asphalt aging. Considering that the peak area calculation is less used in asphalt modification, this paper mainly selects A1, that is, the peak area of the whole spectrum as a benchmark for calculation. The results are shown in Table 6.

Table 6
Summary of functional group indices for different bitumens

IC = O	**SBSMA**	**CRMA**	**SBS/CRCMA**	**Blended Bitumen**
	0.001715	**0.01209**	**0.006349**	**0.008621**
IS = O	0.068611	0.010363	0.012698	0.068966
IB	0.500858	0.55095	0.507937	0.52931
IB,a	0.248714	0.11399	0.266667	0.284483
IAr	0.015873	0.001727	0.005146	0.000862

Studies have shown that carbonyl I_C=Oand sulfoxide I_S=Oindicate the change of oxygen-containing functional groups, so they are used to evaluate the aging performance of asphalt. The aromatic index IAr

represents the content of aromatic ring components, such as aromatics, saturates, and asphaltenes. The higher the content, the greater the ductility, and the better the low temperature performance. As shown in Table 6 in the aromatic index, SBSMA > SBS / CRCMA > CRMA > blended asphalt.

5 Conclusions

(1) The optimum formula of SBS / CRCMA was determined by orthogonal design : refining : towering = 6 : 4 ; sBS content 4% ; rubber powder content of 15% ; furfural oil 4.5%.

(2) Temperature scanning and multiple stress scanning were carried out by dynamic shear rheometer. The results show that SBS / CRCMA has the best high temperature performance, followed by CRMA, SBSMA and blended asphalt. The compatibility of four kinds of asphalt was evaluated by rheological method. From the Cole-Cole diagram, it can be seen that the symmetry of the blended asphalt is the best, which also explains from another perspective that the blended asphalt is a homogeneous system before modification.

(3) Through the analysis of stiffness modulus S and creep rate m, the order of low temperature performance is determined as follows : SBSMA > SBS / CRCMA > SBS / CRCMA > blending asphalt. At the same time, the low temperature sensitivity is studied. It is found that the creep stiffness index of SBS / CRCMA is the largest, indicating that it has strong low temperature sensitivity, which is opposite to high temperature sensitivity. The reason needs further study.

(4) From the molecular weight curve, it can be seen that the molecular weight of SBSMA, CRMA and their complexes at 103 after modification of asphalt is larger than the molecular weight of matrix asphalt. The reason for the formation of the status quo is that the modifier absorbs the light components in the asphalt during the shear development process. Swelling, crosslinking, grafting and other reactions form macromolecules. It can be seen from the infrared spectrum that no characteristic peaks appear in SBSMA, CRMA and SBS / CRCMA, indicating that the modification mechanism is similar to physical blending. Therefore, SBS / CR composite modified asphalt mainly depends on the swelling degree of modifier and asphalt.

Declarations

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Author contributions

Writing-original draft preparation, J.C.; data curation, J.C.; Investigation, W.C. ,H.Y. ,S.O.and H.X.;All authors have read and agreed to the published version of the manuscript.

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Data availability statement

All data generated or analysed during this study are included in this published article.

Consent for publication

Not applicable.

Declaration of competing interestst

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethics statement

Not applicable.

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Figures

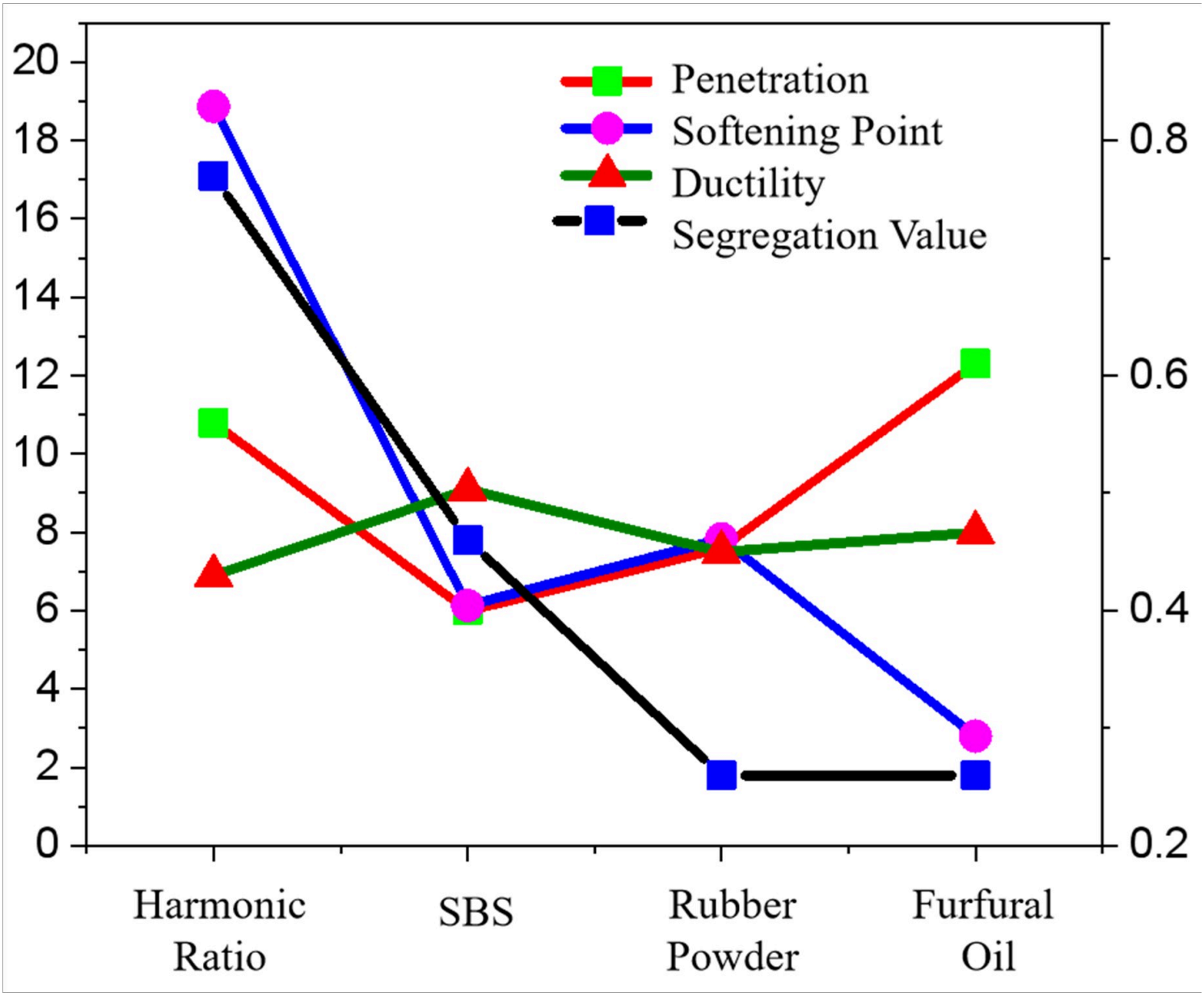
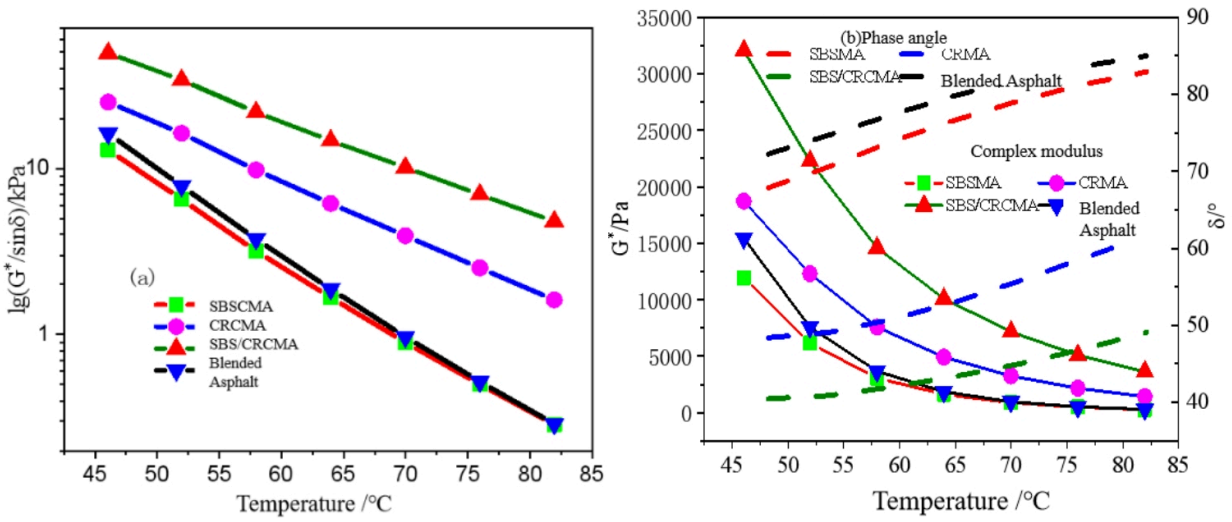


Figure 1

The influence degree of different factors on different indexes



(a) (b)

Figure 2

Analysis of test results

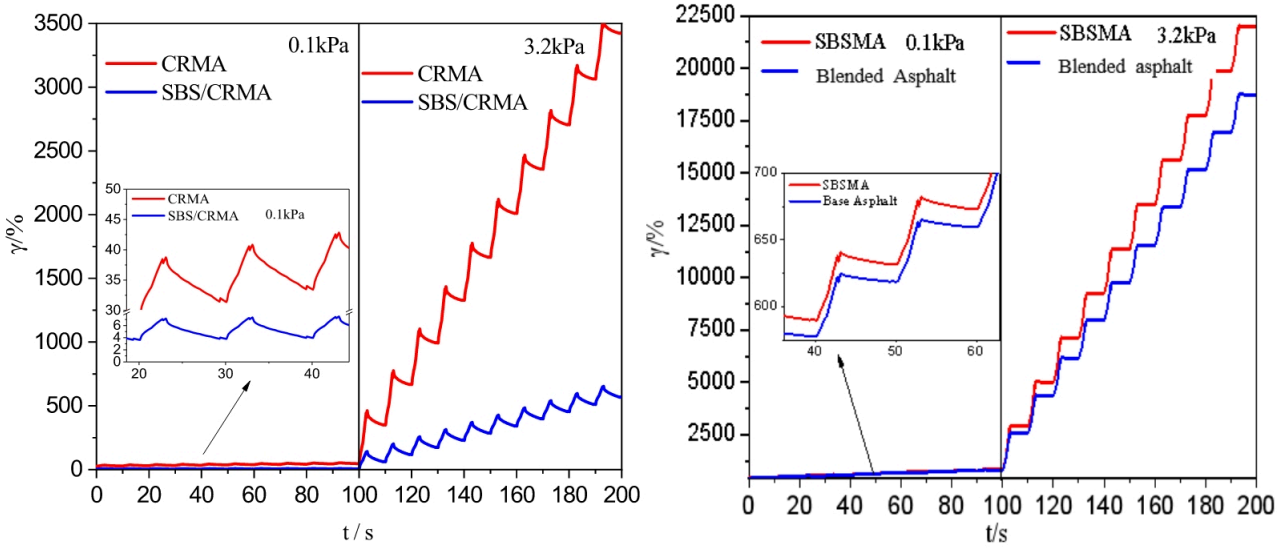


Figure 3

Asphalt strain diagram

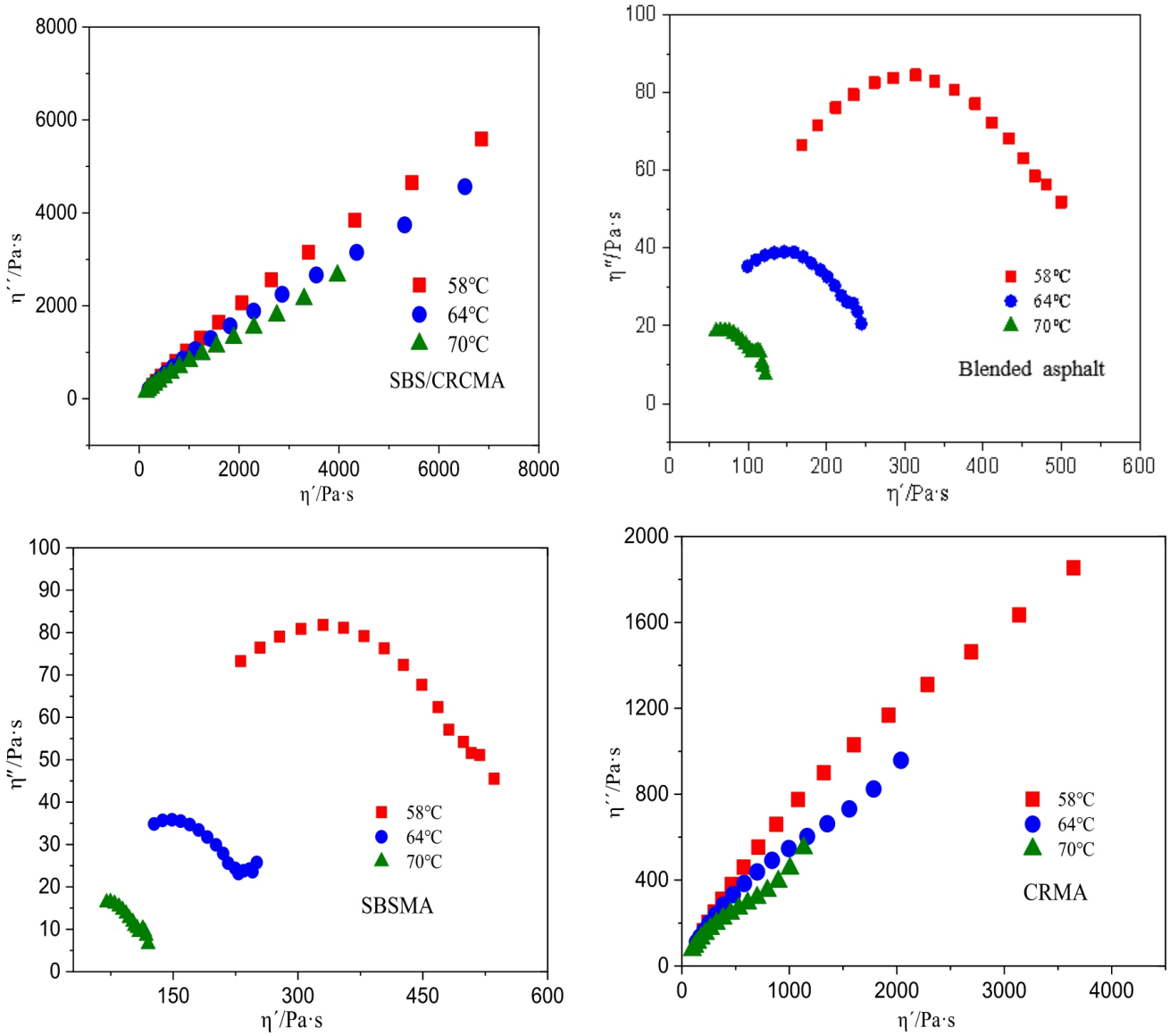


Figure 4

Co-Co curves of different asphalts

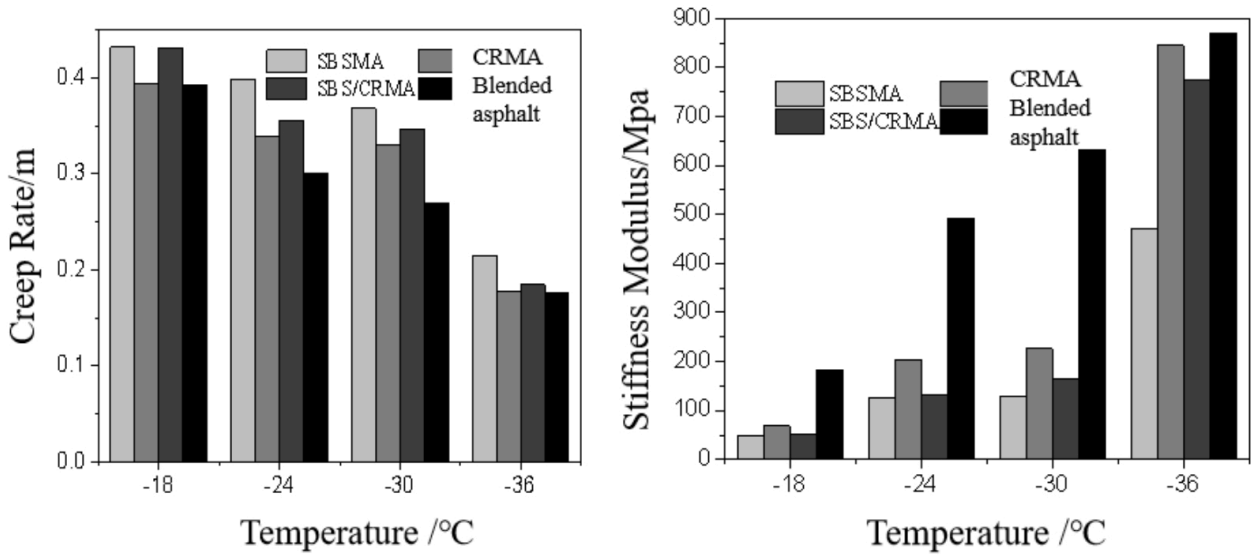


Figure 5

Rigidity modulus and creep stiffness change rate diagram

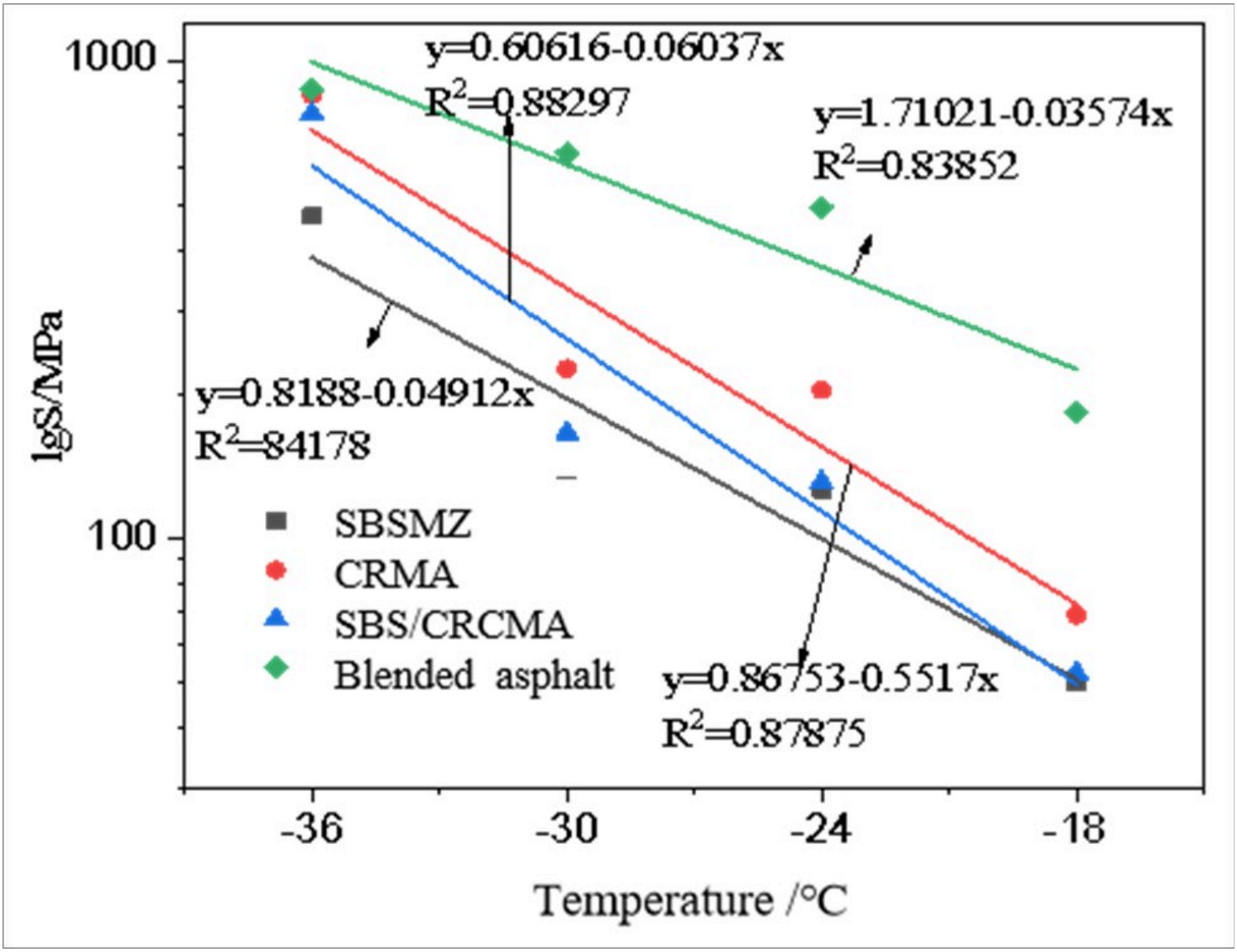


Figure 6

S_{TS} index of different asphalts

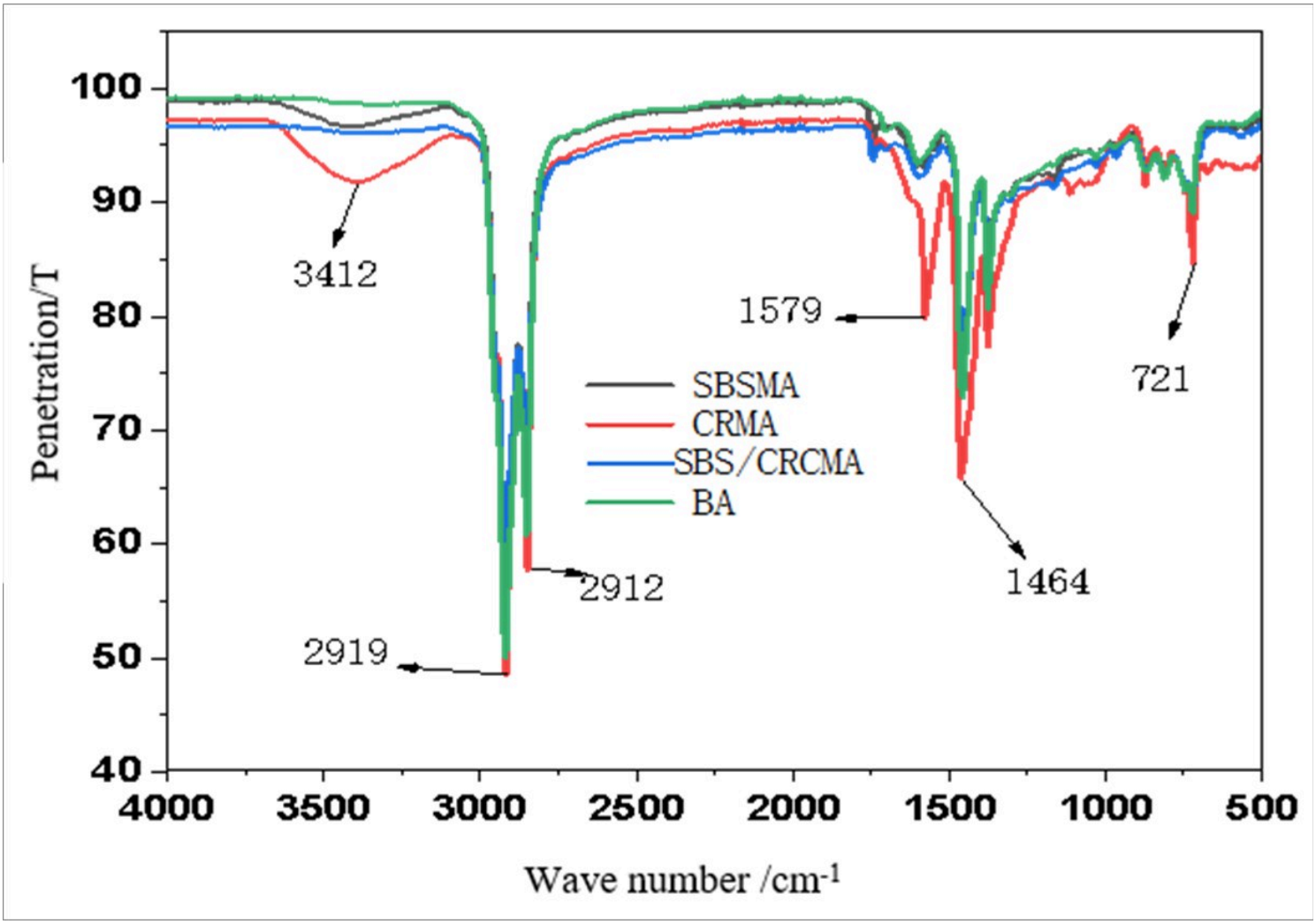


Figure 7

Infrared spectrogram