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Experimental investigation on related properties of asphalt mastic with activated coal gangue as alternative filler

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Abstract

To evaluate the feasibility of using activated coal gangue (ACG) as replacement of mineral filler in asphalt mixture, some properties of asphalt mastic containing ACG were investigated in this paper, compared with the traditional limestone mineral filler (LMF). The investigated mastic consisted of asphalt and filler at a mass ratio of 1:1. Cone penetration test (CPT), Dynamic Shear Rheometer test (DSR), Brookfield viscosity test (BVT) and Dynamic Mechanical Thermal Analysis (DMTA) were applied to research the shear resistance rheological property, rutting resistance, high-temperature viscosity characteristics and low-temperature property of asphalt mastic. With the application of scanning electron microscope (SEM) and Infrared Spectroscopy (IR), the mechanism of ACG imposed on asphalt mastic was analyzed. Results indicate the possibility of ACG as inorganic filler derived from solid waste to improve the performance of asphalt. The ACG has better effect on improving the shear strength and temperature sensitivity of asphalt mastic than LMF. Besides, ACG has positive effect on high-temperature properties while some negative effect on low-temperature properties of asphalt mastic. The results also indicate that the physical adsorption between the ACG filler and asphalt binder is the main mode of action.

Keywords: Asphalt mastic; Activated coal gangue; Filler; Rheological properties; Dynamic Shear Rheometer (DSR); Dynamic Mechanical Thermal Analysis (DMTA)

1. Introduction

Asphalt pavements are the main paving type of highway because of its advantages such as low noise, good skid resistance, improved comfort, convenience of maintenance and recyclability. Asphalt mixtures have been widely used in road pavement [1–4]. The construction and maintenance of these pavements require large amounts of aggregates and mineral fillers, which typically account for nearly 95% of the asphalt concrete [5].

Natural limestone is processed into mineral filler used in asphalt mixture traditionally while the resource is being exhausted with the development of cement and construction industry in China [6]. The use of waste powders such as recycled waste lime, carbon black, fly ash, trass volcanic ash, recycled red brick powder and waste ceramic materials used as filler in asphalt mixture has been the focus of several research efforts over the past few years [7–11]. It was proved that these waste powders could be used in asphalt

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mixture and presented improved performance, being not only economically viable but environmental friendly.

At present, coal gangue produced in the process of coal mining and washing has become one of the largest industrial solid wastes of emissions and accumulated stock in China. So far, the accumulated amount of coal gangue has reached 4.5 billion tons and has a 300–400 million tons of annual production. But the comprehensive utilization of coal gangue is less than 20%. In this situation, precious land resources are occupied, surrounded environment is polluted and human health suffers from hazard for its variety of harmful trace elements [12–14]. Although in recent years, China has begun to attach importance to the comprehensive utilization of coal gangue, the overall level remains to be improved urgently. In particular, there is a big space to inspire coal gangue's activity to form ACG for road building materials recycling use [15]. Up to now, the utilization of ACG in asphalt mixture has rarely been reported. With the use of ACG, coal gangue will be recycled and utilized efficiently to reduce the pollution of solid waste and conserve land resources. The main focus of the present investigation was aimed to the related properties of ACG and asphalt mastic containing ACG.

Many tests including cone penetration test (CPT), Brookfield viscosity test (BVT) and Dynamic Shear Rheometer test (DSR) were applied to evaluate the related properties of asphalt mastic with ACG and the feasibility of ACG powder as mineral filler in asphalt pavement compared with conventional LMF.

2. Raw materials

2.1. Asphalt

The base asphalt used to prepare asphalt mastic is A-110 KLM asphalt provided by Kelamayi Petrochemical industry of China and its penetration is between 100 and 120 (0.1 mm) to be applied in cold regions. Table 1 shows the physical properties of asphalt binder following ASTM standards.

2.2. ACG and mineral filler

ACG is chosen as filler modifier for asphalt mastic and asphalt mixture. ACG derives from kaolin coal gangue in Lingshou county of Hebei province, China. The specific activation process is as follows. Firstly, kaolin coal gangue is collected and sorted, and then fragmented by jaw crusher. Secondly, the crushed coal gangue is dried to constant weight in 105 °C oven environment. Thirdly, it is levigated by ball-grinder to the size of No. 50 to achieve the similar percentage passing as LMF. Finally, the fine powder is calcined for 2 h in a 750 °C oven, seen in Fig. 1.

LMF is levigated limestone, whose particle size ranges from 0 mm to 0.6 mm. The SEM-EPMA (S-4800) equipment manufactured by the Hitachi Group was utilized to observe the microstructure characteristic of ACG and LMF, seen in Fig. 2.

X-ray fluorescence spectrometer (XRF) was utilized to analyze the chemical constituents of ACG and LMF. Table 2 shows the test results.

3. Test methods

3.1. Preparation of asphalt mastic

For practical asphalt pavement engineering projects in China, AC-13 is the most widely used surface paving material. The 0.075 mm sieve passing percentage of mineral aggregate gradations is usually around 5% and the optimum asphalt content (OAC) is around 5% by the mass of mineral aggregates [7]. So the filler–binder ratio of asphalt mastic is 1:1. According to current technical specification requirements of China, oven-dried ACG and LMF were passed through 0.075 mm sieve respectively. Fillers passing the 0.075 mm sieve were used to prepared asphalt mastic samples at a mass ratio of 1:1. Take ACG asphalt mastic as example, the samples were prepared according to the following procedure. Firstly, asphalt was heated to the desired temperature (140 ± 5 °C) in a three-neck flask provided with stirrer and contact thermometer. Secondly, the temperature was held constant by an automatic control system while stirring lasting for 10 min after ACG was dispersed into the asphalt with different percentages. Then a series of samples could be prepared, the same as LMF asphalt mastic.

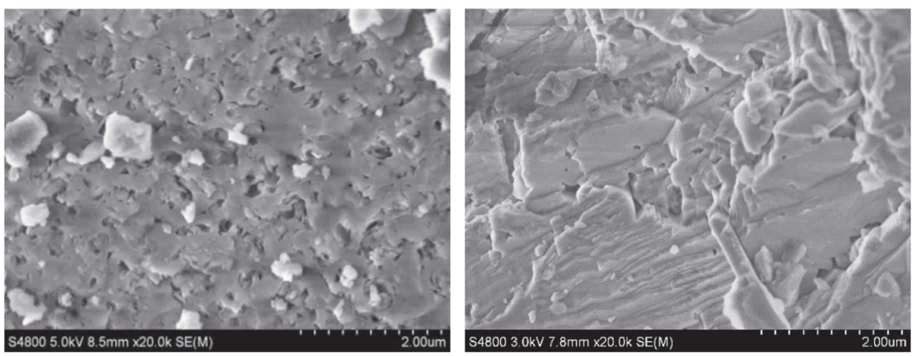
Table 1
Physical properties of asphalt binder.

Material	Test items	Unit	Value	Specification
Base asphalt binder	Penetration at 25 °C	0.1 mm	102	ASTM D5-97
	Ductility at 15 °C	cm	126	ASTM D113-99
	Softening point	°C	45.2	ASTM D36-06
	Wax content	%	1.78	ASTM D3344-90
	Flash point	°C	267	ASTM D92-02
	Specific gravity	Non	0.982	ASTM D70-76
RTFO binder*	Mass loss	%	−0.056	ASTM D2872-04
	Penetration ratio at 25 °C	%	62.3	ASTM D5-97
	Ductility at 10 °C	cm	15	ASTM D113-99

* Rolling thin film oven (RTFO) aged, according to ASTM D2872-04.

(a) Raw gangue block (b) ACG powder

Fig. 1. Macroscopic appearance of raw gangue block and ACG powder.



(a) ACG (b) LMF

Fig. 2. Microtopography of ACG and LMF (×20.0 K).

Table 2
The main chemical constituents of fillers.

Constituent/%	ACG	LMF
SiO_2	50.42	5.00
Al_2O_3	46.11	1.77
TiO_2	1.35	0.07
Fe_2O_3	0.56	0.57
P_2O_5	0.51	N/A
CaO	0.29	87.60
Na_2O	0.24	N/A
K_2O	0.23	0.28
MgO	0.10	4.48
SrO	0.06	0.15
SO_3	0.01	0.03
ZnO	0.01	N/A
Others	0.11	0.04

Besides, Table 3 shows the basic physical properties of ACG and LMF.

3.2. Testing physical and mechanical properties of asphalt mastic cone penetration test

In order to evaluate the shear strength of asphalt mastic, the cone penetration test (CPT) was developed [1], as shown in Fig. 3. During the test, 250 g asphalt binder and 250 g filler (No. 200) were mixed at (140 ± 5 °C). Subsequently, the uniform asphalt mastic was filled in a funnel and then was slightly oscillated into an iron vessel. The prepared sample was kept at room temperature for 40 min until it cooled and solidated. Then it was immersed in water at 30 °C for more than 1 h. Afterward, the sample was taken out and an iron cone (195 g) was released from the mix surface. The cone would gradually penetrate into the asphalt mastic until a stable situation. During the

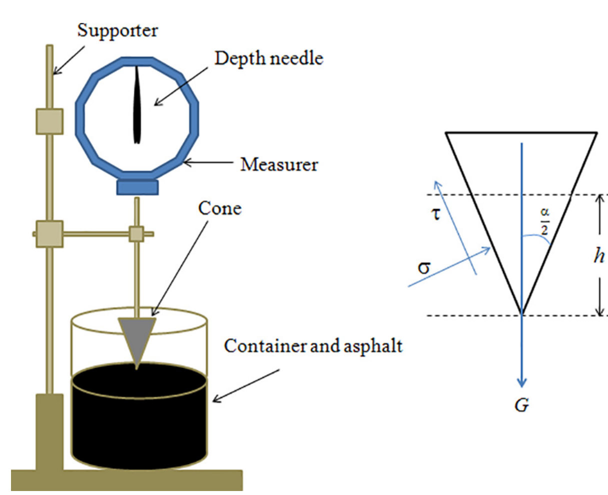


Fig. 3. Cone penetration test.

experiment, the sink depth was measured and recorded. It should be noted that the cone would not completely penetrate into the mix. Based on the force balance, the shear stress τ(kPa) of asphalt mastic at the direction tangential to the cone surface can be determined as follows:

$$\tau = \frac{981Q\cos^2(\frac{\alpha}{2})}{\pi h^2 \tan(\frac{\alpha}{2})} \quad (1)$$

where Q is the cone weight (195 g); h is the sink depth (0.1 mm); α is the cone angle (30°). Three replicate specimens were measured for each filler type. Here, the cone sink depth and calculated shear stress, according to the force balance, are used to evaluate the resistance to shear force of asphalt mastic.

3.3. Dynamic Shear Rheometer test (DSR)

Rheological measurement is the basic method to investigate the viscoelasticity of materials. In this paper, the Dynamic Shear Rheometer (DSR) test was conducted to measure the shear modulus and phase angle of asphalt binder. According to ASTM D7175, the rheological properties and rutting resistance of asphalt mastic were evaluated at 30 °C, 40 °C, 50 °C, 60 °C and 70 °C. Strain sweep test was firstly carried out to fix the value of shear strain. Under the strain-controlled mode, complex shear modulus (G^*) and phase angle (δ) of asphalt mastic samples were obtained at each temperature. In this experiment, the asphalt film sample with a diameter of 25 mm and a thickness of 2 mm was sandwiched between a fixed steel plate and an oscillating plate with an angular frequency of 10 rad/s under a constant torque. The shear modulus G^* and phase angle δ (shear lag of strain to stress) can be determined as follows:

$$G^* = \frac{\sigma(t)}{\gamma(t)} = i\omega \int_0^\infty \varphi(\xi)\exp(-i\omega\xi)d\xi = G' + iG'' \quad (2)$$

$$tg\delta = \frac{G''}{G'} \quad (3)$$

where i is the imaginary unit; ω is the angular frequency; σ is the shear stress; γ is the shear strain; δ is the phase angle; G' is the elastic part and G'' is the viscous part of G^*. In addition, $G^*/\sin\delta$ is the performance index to evaluate asphalt mastic's rutting resistance. A higher $G^*/\sin\delta$ value indicates a higher elasticity and rutting resistance of filler asphalt mastic.

Traffic loading is a dynamic loading and is over a wide range of frequency and temperature conditions [7]. Due to the characterization of the viscoelastic properties of the asphalt mastic system, it is difficult to obtain the viscoelastic parameters of the asphalt mastic by the conventional method. The dynamic viscoelastic test has become one of the most effective means for simulating the deformation characteristics under the dynamic load of the pavement and characterizing the asphalt mastic performance. Previous research results show that the shear frequency can be correlated with the traffic volume and high frequency simulates heavy traffic while low frequency simulates light traffic. The shear frequency of 0.01 rad/s–100 rad/s (0.0016 Hz–16 Hz) can simulate the normal road traffic [16,17]. In this paper, the filler asphalt mastics were frequency-scanned by DSR. The temperature range is 30 °C, 40 °C, 50 °C, 60 °C, 70 °C, and the load frequency range is 0.0016 Hz–16 Hz.

3.4. Brookfield viscosity test (BVT)

Brookfield viscosity test (BVT) is often applied to investigate the apparent viscosity of asphalt in the temperature range above 45 °C. Based on the viscosity curves at different temperatures, the mixing temperature and compaction temperature of various asphalt mixes can be determined. In this experiment, the viscosity of asphalt mastic at 135 °C, 150 °C, 165 °C, 180 °C was conducted in accordance with JTG E20-2011.

Before the test, it is important to choose a suitable rotor according to estimated asphalt viscosity and applicable rate for different type of rotors. In order to ensure the measurement accuracy, the torque reading should be between 10% and 98%.

Conventionally, the sensitivity of the viscosity to temperature changes reflects the temperature sensitivity of the asphalt material. The excellent asphalt material should be viscous at high temperatures to resist deformation under load and at the low temperature with sufficient flexibility to enhance cracking resistance [18]. Viscosity index V_{TS} put forward by Saal is applied to evaluate the viscosity characteristics of asphalt mastic according to the following formula [19]:

$$V_{TS} = \frac{\lg\lg(\eta_1 \times 10^3) - \lg\lg(\eta_2 \times 10^3)}{\lg(T_1 + 273.13) - \lg(T_2 + 273.13)} \quad (4)$$

where T is absolute temperature, K; η_1 and η_2 are viscosities at different temperatures, Pa·s. The greater $|V_{TS}|$ means the more obvious viscosity change of asphalt mastic affected by temperature.

3.5. Dynamic Mechanical Thermal Analysis (DMTA)

Dynamic mechanical behavior refers to the mechanical response of the material under the vibration condition of alternating stress (alternating strain). The relationship between mechanical properties (modulus, internal friction) and temperature and frequency can be obtained. Dynamic Mechanical Thermal Analysis (DMTA) is often applied to measure the variation of dynamic mechanical properties of the material in a certain temperature range. DMTA is sensitive to the glass transition temperature (T_g), molecular crosslinking, phase separation and molecular chain movement of viscous polymer materials. The main parameters of the instrument are the storage modulus (E'), the loss modulus (E'') and the loss factor (tanδ), where E' reflects the elasticity of the material, characterizing the stiffness of the material; E'' reflects the damping nature. Tanδ is the ratio of E' to E'' which can also characterize the damping properties of the material.

When the temperature is low, the polymer material appears in a glassy state, where the molecular segments of the material are frozen. At this moment, only small deformation due to the change of bond angle and bond length happens which is elastic deformation and the internal friction is little. When the temperature is further increased, the molecular segments of the polymer material are free to move. When the molecular segments within the polymer material are transformed from thawing to free, the segments need to overcome a large frictional force during the movement and so the internal friction is large. Meanwhile, when the internal friction reaches the maximum, the material is at vitrification transition state. The glass transition temperature (T_g) is the characteristic temperature that characterizes the motion of the polymer material. In this paper, T_g is applied to evaluate the low-temperature property of asphalt mastic.

The DMTA instrument and sample preparation is shown in Fig. 4.

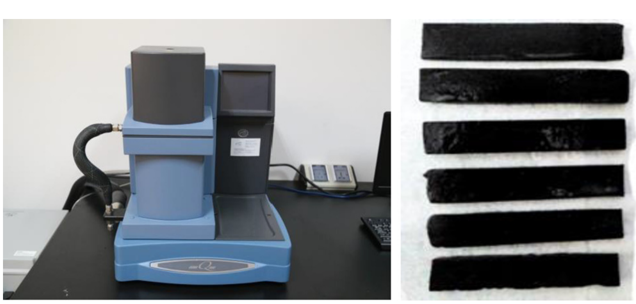


Fig. 4. DMTA (Q800, TA instrument) and sample preparation (60 mm × 12 mm × 3mm).

4. Results and discussion

4.1. Shear behavior

Fig. 5 shows cone penetration test (CPT) results of asphalt mastic including the calculated shear strength according to measured cone sink depths. The shear strength of asphalt mastic ranked in a decreased order as follows: ACG asphalt mastic, LMF asphalt mastic and A-110 base asphalt. Compared with base asphalt, fillers increase the shear strength of asphalt binder. This is because fillers can perform the absorption and stabilization of asphalt (especially light components) and increase the viscosity and stiffness. ACG improving the shear strength more than the LMF is because ACG is much finer than LMF, as indicating by Table 3 which shows that ACG has 85.4% finer than 0.075 mm whereas LMF only has 75.3%. Besides, the shear strength of each type of asphalt mastic decreases with the increase in temperature. Like unmodified base asphalt, filler asphalt mastic is also viscoelasto-plastic material, affected greatly by the temperature. When the temperature increases, the stability of asphalt mastic deteriorates, causing asphalt mastic shear strength becomes smaller.

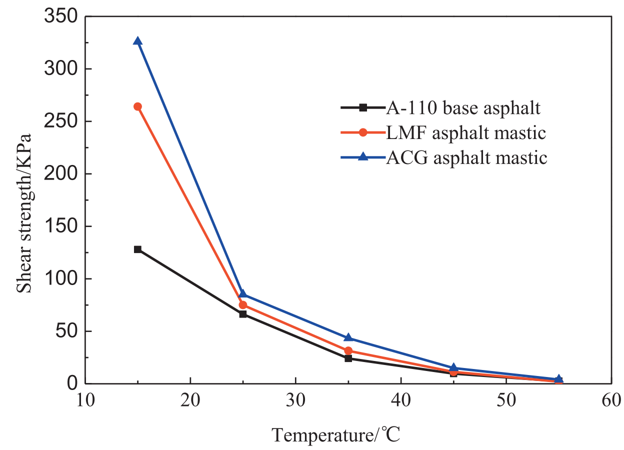


Fig. 5. Results of CPT.

4.2. Dynamic Shear Rheometer analysis

The test results are shown in Fig. 6. It can be clearly seen that rutting factor $G^*/\sin\delta$ of base asphalt and asphalt mastic increases with the increase in load frequency. Under the action of external load, deformation of asphalt and asphalt mastic occurs, including elastic deformation, recoverable viscoelastic deformation and irreversible viscous deformation. The greater the load frequency is, the shorter the load time of the samples at each cycle becomes, resulting in the smaller amount of deformation of asphalt materials and an increase in modulus, and vice versa.

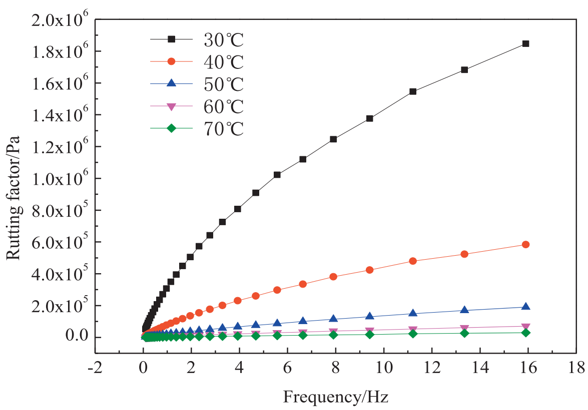
Furthermore, a marked increase for $G^*/\sin\delta$ is obtained by the addition of filler. The addition of ACG results in more significantly increased $G^*/\sin\delta$ than LMF under load frequency range, meaning that ACG asphalt mastic has better high temperature property than LMF especially within the test temperature range of 40 °C–60 °C. For the same asphalt or asphalt mastic, the rutting factor $G^*/\sin\delta$ at low temperature or high frequency is much larger than that at high temperature or low frequency.

4.3. Brookfield viscosity test (BVT)

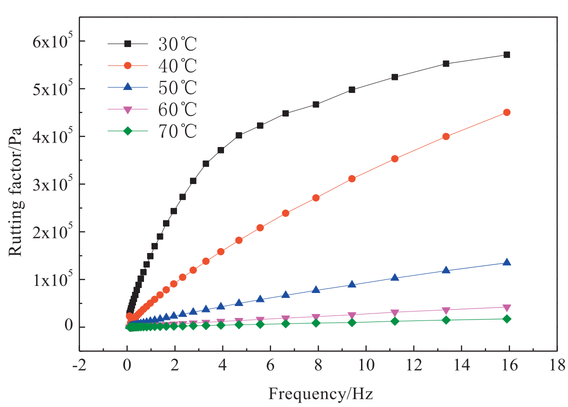
In general, there is a close relationship between the viscosity of asphalt or asphalt mastic and the mixing and compacting temperatures of asphalt mixture. The effect of ACG on high temperature viscosity of asphalt mastic is discussed here compared with LMF. The high temperature viscosity test was conducted by a Brookfield Viscometer at 135 °C, 150 °C, 165 °C and 180 °C. There is a linear relationship between the common logarithm of the viscosity values (lgη) and temperature (T).Table 4 illustrates the viscosity-temperature index V_{TS} of asphalt mastic under different temperatures.

Table 3
Physical properties of ACG and mineral powder.

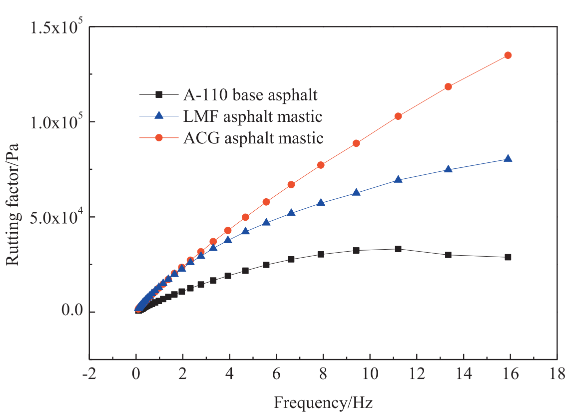
Items	ACG	LMF	Specification
0.6 mm sieve size percent passing/wt.%	100	100	ASTM C136
0.15 mm sieve size percent passing/wt.%	96.6	90.2	
0.075 mm sieve size percent passing/wt.%	85.4	75.3	
Density/(g/cm^3)	2.626	2.723	ASTM D3800
Specific surface area/(m^2/g)	0.29	0.27	ASTM D3037



(a) Rutting factor $G^*/\sin\delta$ of LMF asphalt mastic



(b) Rutting factor $G^*/\sin\delta$ of ACG asphalt mastic



(c) Rutting factor $G^*/\sin\delta$ of different asphalt mastic at 50 ℃ as an example

Fig. 6. Frequency sweep test results of base asphalt and asphalt mastic.

Table 4 shows that the $|V_{TS}|$ of asphalt mastic ranked in an increased order as follows: ACG asphalt mastic <LMF asphalt mastic <A-110 base asphalt. Compared with the base asphalt, fillers decrease the Viscosity-temperature index of asphalt binder. That means the addition of filler can effectively improve the high-temperature sensitivity of asphalt binder, and the ACG has a better effect than LMF.

4.4. Dynamic Mechanical Thermal Analysis (DMTA)

Fig. 7 shows the DMTA typical curve of A-110 base asphalt. With the increase in temperature, the storage modulus E' gradually decreases and finally tends to be gentle. The loss modulus increases first and then reaches the peak and subsequently decreases. Meanwhile, the loss factor E'' keeps increasing. The T_g of base asphalt can be achieved from the DMTA curve and its value is −17.19 °C. Similarly, T_g of ACG and LMF asphalt mastic are −12.42 °C and −10.89 °C, respectively. It indicates that ACG may have some negative effect on low-temperature property of asphalt mastic. So when ACG is used in extremely cold areas, the low-temperature performance of asphalt mixture needs paying attention to.

4.5. Mechanism analysis of ACG reinforcing asphalt binder

Fig. 8 shows the infrared spectrogram of ACG, A-110 base asphalt and ACG asphalt mastic. The characteristic peak at 3427 cm^{-1} is the broad absorption peak caused by the hydroxyl group, and the characteristic peak near 2800 cm^{-1} is the absorption peak of methylene "—CH_2—". Meanwhile, the broad absorption band near 1200 cm^{-1} is caused by the expansion of the aliphatic amine C—N, and the absorption peak near 750 cm^{-1} is caused by the N—H out-of-plane bending vibration absorption. After the interaction between the ACG and the asphalt, the most absorption peaks are the superposition of the respective

Table 4
Viscosity-temperature index V_{TS} of asphalt mastic under different temperatures.

Type	135 °C/150 °C	150 °C/165 °C	165 °C/180 °C
A-110 base asphalt	−2.467	−4.098	−2.992
LMF asphalt mastic	−2.325	−3.651	−2.507
ACG asphalt mastic	−1.836	−2.937	−2.098

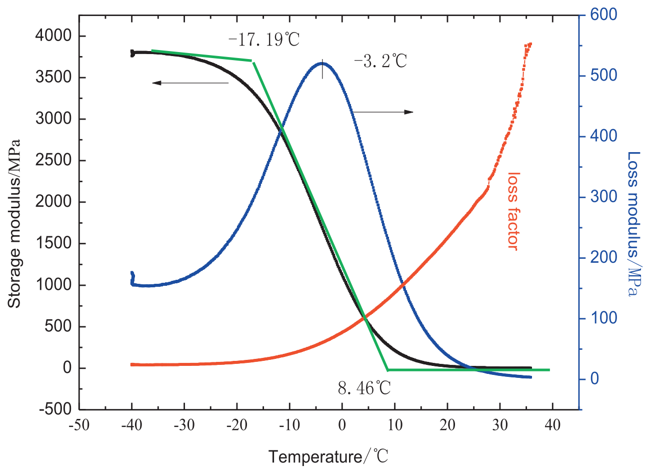


Fig. 7. Temperature scanning mechanics curve of A-110 base asphalt through DMTA.

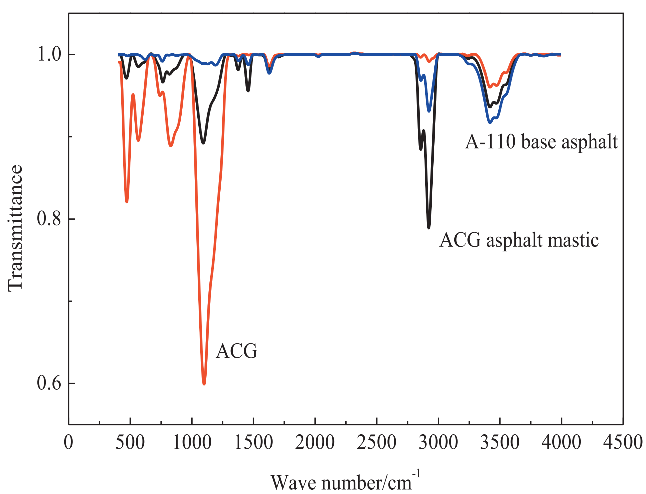


Fig. 8. IR test results.

absorption peaks of the ACG and asphalt binder. There is no obvious new stretching vibration or deformation vibration peak, which indicates that physical adsorption between the ACG filler and asphalt is the main mode of action.

5. Conclusions

The related properties of asphalt mastic with activated coal gangue as alternative filler were investigated through laboratory experiments in this paper. The following conclusions can be drawn.

(1) Compared with LMF, the ACG has porous surface and larger specific surface area, indicating the possibility and feasibility of ACG as inorganic filler derived from solid waste to improve the performance of asphalt. In addition, the physical adsorption between the ACG filler and asphalt is the main mode of action.
(2) The ACG has the better effect on improving shear strength of asphalt binder than LMF. In asphalt mastic, filler can perform the absorption and stabilization of asphalt (especially light components) and increase the viscosity and stiffness.
(3) The addition of ACG resulted in more significantly increased $G^*/\sin\delta$ than LMF under load frequency range, so ACG has better effect on high-temperature property of asphalt mastic.
(4) ACG and LMF can effectively improve the high-temperature sensitivity of asphalt binder, and ACG is the better one.
(5) ACG may have some negative effect on low-temperature properties of asphalt mastic. So when ACG is used in extremely cold areas, it is necessary to consider carefully the low-temperature performance of asphalt mixture.

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