



# Experimental Study of Asphalt Mixture with Acetate Anti-Icing Filler

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## Abstract

This study evaluated the effects of the types (MFL and acetate anti-icing filler) and the replacement rate of anti-icing additives on the performance of asphalt mixtures. Additionally, the road performance as well as ice inhibition capacity between environmental (acetate anti-icing filler) and conventional (MFL) anti-icing materials were compared. The equivalent volume displacement method was applied to determine the amount of anti-icing filler in asphalt mixture. In this paper, the high-temperature stability, low-temperature bending properties, water stability, ice inhibition properties and anti-icing persistence of the mixture were investigated in the laboratory. The results show that the ice inhibitor has negative effects on the rutting resistance, crack resistance and moisture sensitivity of the asphalt mixture. Additionally, the adverse effects become more obvious as the replacement rate increases. There was a similar trend in the mixtures with three kinds of ice inhibition filler in general. The tests also proved that the anti-icing fillers can effectively delay and inhibit the occurrence of road icing, weaken the bonding force between pavement surface and the ice layer and make the ice layer easy to be removed. It is found that the comprehensive performance of traditional ice inhibitors is better. Moreover, the high-temperature stability, anti-icing capacity and ice inhibition persistence of environmentally friendly anti-icing additives need to be improved.

**Keywords** Asphalt pavement · Environmentally friendly anti-icing filler · Road performances · Anti-icing property · Ice inhibition persistence · Performance comparison

## 1 Introduction

Road surface safety in the hazardous weather, especially in snowy weather, has always been a major management concern for the highway department in many countries. The snow or ice layer on the road will greatly reduce the skid resistance of pavement surface and brings driving safety threats contributing to severe road crashes and injuries [1, 2].

Mechanical equipment is firstly applied (snow sweepers, plows, etc.) to remove snow and ice layer effectively for safer driving in winter. However, relevant studies and practical application in engineering programs show that the utilization of mechanical equipment can only remove the snow

covering the road surface, and it is difficult to remove the ice layer closely sticking to the road surface [3]. More importantly, snow removal by mechanical equipment requires professional technicians and devices, which increases the economic cost of snow removal. Besides, excessive shoveling power of mechanical equipment may damage the road surface structure, thereby affecting the engineering performance of the road surface [4]. The idea of solving the problem has gradually shifted from snow removal to snow melting and ice suppression. The initial conventional method was to spread snow melting salt on the road surface [5], but this method was time-consuming and labor-intensive [6]. In regions with heavy snowfall, the methods of snow melting salt spreading and mechanical devices need to be applied in combination, which may cause traffic congestion. To improve the above snow removal methods, the energy conversion snow melting and deicing road surfaces [7] are proposed and implemented, consisting of the fluid heating road surface, hot water piping road surface and electric heating road surface. The technology means that a certain number of heat transfer pipelines or heat conductors are embedded in the pavement structure, converting the external energy

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into heat energy and inputting the heat energy into the interior of the road structure, and then heating the road surface through the internal heat of the road structure. The technology though improves the efficiency of snowmelt is still energy and cost-consuming.

Self-melting pavement has aroused much attention for its simple construction process and economic advantages [8, 9]. The basic working principle of self-melting snow pavement is to mix anti-icing materials into the asphalt mixture and substitute for all or part of the mineral powder in it. The salt is released from the mixture under the combined effects of wheel friction, vehicle load pumping, osmotic pressure, capillary, etc. In a wet environment, the ice inhibition components dissolve out, which decreases the freezing point of the road surface solution and weakens the adhesion between the road surface and ice layer [10]. The main point is to realize the direct transfer of ice melting capacity from deicing materials to pavement materials. Therefore, the self-melting snow pavement essentially represents a shift from deicing to anti-icing or preventive strategies [11].

Currently, the commonly used commercial anti-icing additives are Verglimit, Mafilon (MFL) and IceBane. Among them, the ice inhibitor "Verglimit" developed by Switzerland VERGLIMIT in 1973 can precipitate  $\text{CaCl}_2$  on the pavement surface and effectively delay the freezing time of pavement and weaken the bonding force between pavement and ice layer, so that the ice layer can be easily removed [12–14]. The MFL developed in Japan has similar characteristics to Verglimit. It is a snow-melting and ice inhibition composite filler wrapped with  $\text{NaCl}$ . The surface is hydrophobically treated and salt is released under the action of the external environment to achieve the snow-melting and anti-icing effects. IceBane containing  $\text{CaCl}_2$ ,  $\text{NaCl}$ , etc., have also undergone hydrophobic treatment. Numerous studies have been conducted on the snow-melting and ice-inhibiting effects and engineering performance of these three anti-icing additives at home and abroad. The test results showed that compared with ordinary asphalt pavement, the self-melting asphalt pavement has better snow melting and anti-icing ability under negative temperature condition [15–17]. However, most of the effective components of these anti-icing fillers are chloride salts, and the salt solution formed after precipitation is highly corrosive, causing some damage to the pavement structure. Therefore, the development of environmental and non-corrosive salt materials based on organic salts (formate, acetate, etc.) will be an important development trend in the future [18]. Zhang [19] produced calcium magnesium acetate through the direct reaction of glacial acetic acid and dolomite and systematically studied the influence of various process parameters on product quality. Additionally, obtained a calcium magnesium acetate production process with good ice melting effect. Xia [18] has developed a new type of composite salt with acetate

as the main component. The test results showed that the self-melting snow pavement mixed with the new type of composite salt could effectively inhibit icing in the early stage of snowfall and accelerate the melting of ice during the period of snowfall. After the snowfall, the ice layer is easy to be moved since the bond force between the ice layer and pavement is relatively weak. A previous study [20] has shown that acetate has a stronger ability to inhibit ice than chlorine salt, which can make snow more easily eradicated. It has been verified by experiments in Europe, the USA, Canada, Japan and other countries that acetate is an environmentally friendly chemical. It not only has ice suppression properties but also absorbs nitrides and sulfides produced by coal combustion, which is beneficial to alleviating environmental pollution problem. It can be seen that further research on environmentally friendly ice inhibitors with acetate as the main component is the development trend of self-melting snow pavement and ice inhibitor materials; nevertheless, there are few studies related to this, and its road performance and ice inhibition capacity have not been compared with the common anti-icing additives, which is not conducive to further optimizing the comprehensive performance of environmental friendly ice suppression additives.

The objectives of this research are to study the influence of the types (MFL, calcium acetate and magnesium acetate) and content of ice inhibition additives on the road performance of asphalt mixture through conventional test methods, such as rutting test, low-temperature bending test, immersion Marshall test as well as freeze-thaw splitting test. Besides, the anti-icing property was evaluated by British Pendulum Tester (BPT) and self-developed Walking Friction Tester (WFT). Additionally, ice inhibition persistence was evaluated by electrical conductivity test. Moreover, comparing the road performance and ice inhibition capacity of environmental and traditional anti-icing agents lays a foundation for the comprehensive performance optimization of environmental friendly ice inhibition additives. The research framework of this paper is schematically illustrated in Fig. 1.

## 2 Materials

### 2.1 Asphalt and Aggregate

The salt storage asphalt pavement is mainly used in low temperature and cold areas, and the pavement is required to have excellent low-temperature cracking resistance. Styrene–butadiene–styrene (SBS) modified asphalt has good low-temperature stability and strong adhesion to aggregates. The asphalt binder used in this study is Karamay 70# asphalt (KLM-70) modified with SBS. Its physical properties were investigated, as shown in Table 1.

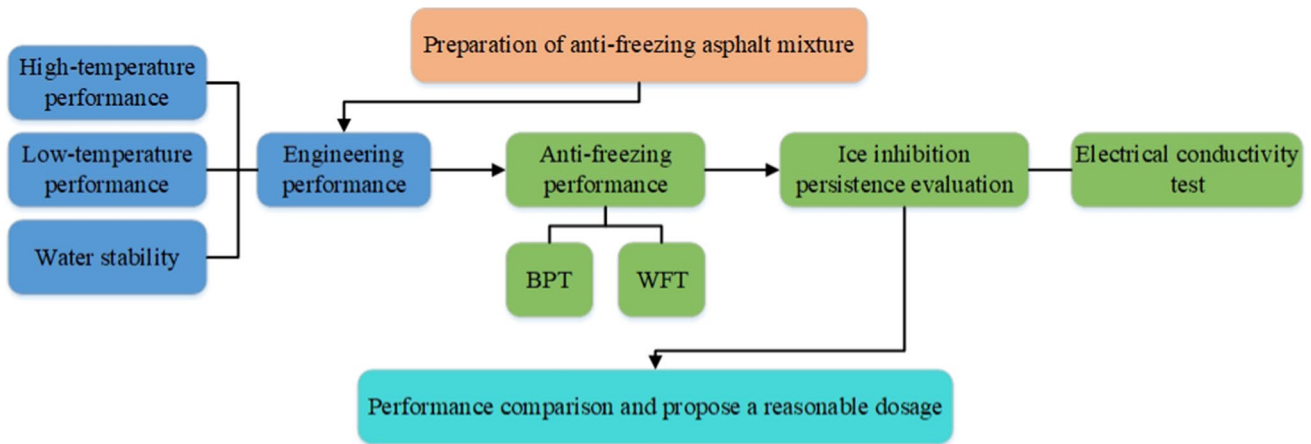


Fig. 1 Research framework

Table 1 Technical properties of SBS modified asphalt

Test items	Specification requirements	Test results
Penetration (25 °C, 5s, 100g) (0.1mm)	40–60	57.5
Penetration index	≥ 0	0.72
Ductility (5 °C, 5 cm/min) (cm)	≥ 20	34
Softening point (°C)	≥ 60	85.8
Kinematic viscosity (135°C) (Pa·s)	≤ 3	2.24
Recovery of elasticity (25° C) (%)	≥ 75	89
Flash point (°C)	≥ 230	286

The selected aggregate is diorite, which is clean, dry, rough surface, no weathering and no impurities. Additionally, the water absorption rate is less than 1.0% and adhesion to the aggregate is up to level 5. Therefore, sufficient strength and wear resistance are guaranteed. Other technical indexes all meet the relevant requirements of the Chinese corresponding specification [21]. The selected mineral powder is dry and free of impurities. The main technical indicators are shown in Table 2.

## 2.2 Antifreezing Fillers

### (1) Basic properties

MFL, calcium acetate and magnesium acetate were used to prepare anti-icing asphalt mixtures. The basic

Table 2 Physical properties of miner filler

Index	Appearance	Hydrophilicity	Apparent density (g/cm <sup>3</sup> )	Moisture content (%)	Percent passing (%)			
					< 0.6	< 0.3	< 0.15	< 0.075
Value	No lumps	< 1	2.72	0.45	100	100	100	98.6

properties of the anti-icing additives are shown in Tables 3 and 4.

### (2) Working mechanism of antifreezing fillers

The action mechanism of the three kinds of ice inhibition materials is similar, shown as in Fig. 2, since all of them are added to the asphalt mixture to replace all or part of the mineral powder in the asphalt mixture. Due to the existence of the voids in the pavement structure, water gradually enters the mixture and makes the ice inhibition material dissolve. Under the combined action of environmental temperature and humidity changes, road compression, vibration and abrasion, the soluble salt solution gradually moves from the internal structure of mixture with higher concentration to the road surface with lower salt concentration, which reduces the liquid vapor pressure of road surface water. However, in order to achieve the state of the equal solid–liquid vapor pressure of the ice–water mixture, the solid vapor pressure of ice remains constant. Thus, the effect of delaying road icing can be realized.

## 2.3 Mix Design

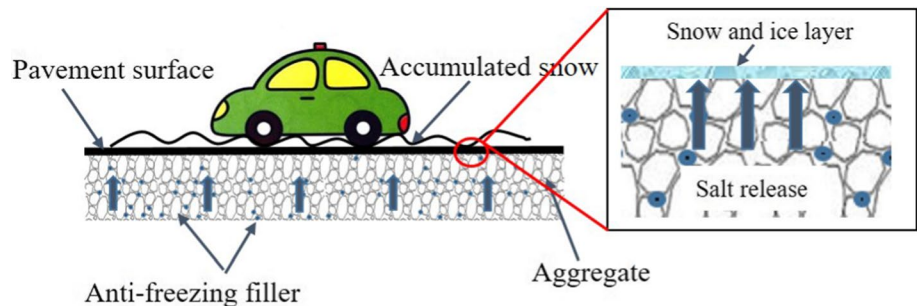
According to the upper and lower limits of AC-13 type gradation stipulated in “*Technical Specifications for Construction of Highway Asphalt Pavements*” (JTG F40-2004) [21], and considering the requirements of pavement performance and void ratio, the gradation is adjusted properly to make the grading curve close to the middle gradation.

**Table 3** Main technical indexes of MFL

Test items	Specification requirements	Test results
Appearance	No lumps	Rust red powder
Apparent density ( $\text{g}/\text{cm}^3$ )	2.25–2.35	2.32
Packing density ( $\text{g}/\text{cm}^3$ )	0.93–0.99	0.95
pH	8–8.5	8.1
Moisture content (%)	$\leq 0.5$	0.2
Salt content (%)	45–65	59
Passing percent (%)	< 0.6mm	100
	< 0.15mm	85–95
	< 0.075mm	70–80
Main chemical components	Sodium chloride, calcium oxide, magnesium oxide, silicon dioxide, etc. The active ingredient is sodium chloride, accounting for about 56%	

**Table 4** Main technical indexes of acetate anti-icing filler

Test items	Calcium acetate		Magnesium acetate	
	Specification requirements	Test results	Specification requirements	Test results
Appearance	No lumps	White uniform powder	No lumps	Colorless monoclinic crystal
Bulk density ( $\text{g}/\text{cm}^3$ )	1.3–1.7	1.45	1.42–1.75	1.454
Moisture content (%)	$\leq 7$	5.3	–	–
Salt content (%)	$\geq 98$	99	$\geq 98$	99
pH	6–8	6.4	6–8	6.1
Sulfate content (%)	$\leq 0.1$	0.06	–	–
Fluoride content (%)	$\leq 0.005$	0.003	–	–

**Fig. 2** Working mechanism of antifreeze asphalt concrete (AFAC) pavement containing antifreezing fillers

The gradation of the AC-13 asphalt mixture with ice inhibition materials is shown in Fig. 3. The optimal asphalt content of salt-storage asphalt mixture is 5% determined by the Marshall test. Due to the difference of density between ice inhibition materials and mineral powder, the equivalent volume replacement method was adopted in the process of replacing mineral powder, and four replacement ratios (0%, 35%, 70% and 100%) were set for each of the three types of anti-icing materials. The equivalent volume replacement method means that a certain volume

of mineral powder is replaced by the same volume of ice inhibition filler. In theory, the equivalent volume replacement method is the most ideal grading design method for AFAC [22].

## 2.4 Specimen Preparation

Firstly, the aggregates heated at 165–170 °C were mixed for 30–40 s, and asphalt binder heated at 170 °C was added and stirred for 90s. Secondly, the mineral filler or the antifreeze

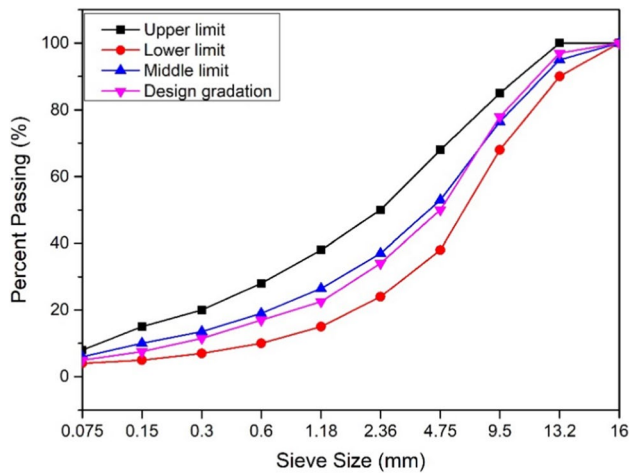


Fig. 3 Grading curve of AC-13

filler was added into the mixture and blended another 90 s. Finally, the hot mixtures were poured into the hot test mold and compacted according to the national standard “Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering” (JTG E20-2011) [23]. Slab specimens (300 mm × 300 mm × 50 mm, 500 mm × 500 mm × 50 mm), Marshall specimen (diameter 101.6 mm, height 63.5 mm) and prismatic beams (250 mm × 30 mm × 35 mm) were obtained. Then, rutting test (T0719-2011), low-temperature bending test (T0715-2011), immersion Marshall test (T0709-2011), freeze-thaw splitting test (T0729-2000), BPT (ASTME303-13), WFT (walking frictional tester, antiskid equipment independently developed by the laboratory) as well as electrical conductivity test were carried out.

### 3 Experiments

#### 3.1 Pavement Performance Tests

##### 3.1.1 High-Temperature Performance

Rutting test is used to simulate the effect of actual vehicle load on pavement structure under high temperature, so as to evaluate the permanent deformation resistance of asphalt pavement. The mixture was compacted into slab specimen of 300 mm × 300 mm × 50 mm with a wheel roller forming machine. The test was carried out in accordance with T0719-2011 of the Chinese specification [23]. The test temperature was 60 °C and the wheel pressure was 0.7 MPa. The dynamic stability applying to evaluate the ability of asphalt pavement to resist high-temperature deformation was calculated according to equation (1) [23]:

$$DS = \frac{(t_2 - t_1) \times N}{d_2 - d_1} \times C_1 \times C_2 \tag{1}$$

In equation (1):  $t_2$  = test time, take 60 (min)  $t_1$  = test time, take 45 (min)  $N$  = the round-trip rolling speed of the test wheel, take 42 (times/min)  $d_1$  = the deformation when the test time is 45 min  $d_2$  = the deformation when the test time is 60 min  $C_1$  = the type coefficient of the testing machine, take 1.0  $C_2$  = the specimen coefficient, take 1.0

##### 3.1.2 Low-Temperature Performance

Salt storage asphalt pavement is mainly used in low temperature and cold areas. Excellent low-temperature crack resistance is indispensable in order to guarantee its durability. The low-temperature bending test is used to simulate the deformation of the pavement under the repeat action of wheel load under the condition of low temperature, as well as evaluate its low-temperature cracking resistance. The mixture was compacted by a wheel roller forming machine and cut into prismatic beam of 250 mm × 30 mm × 35 mm after cooling for 24 h. The test temperature was −10 °C and the loading rate was 50 mm/min. The low-temperature crack resistance of the mixture is evaluated by the maximum tensile strain  $\epsilon_B$  during failure, and the calculation is carried out according to equation (2) [23]:

$$\epsilon_B = \frac{6 \times h \times d}{L^2} \tag{2}$$

In equation (2):  $\epsilon_B$  = the maximum tensile strain of the specimen before failure ( $\mu\epsilon$ )  $h$  = the height of mid-span section of the beam (mm)  $d$  = the deflection in the middle of the span when the specimen is damaged (mm)  $L$  = the span of the specimen (mm)

##### 3.1.3 Water Stability Performance

At present, the research on the influence of various ice-inhibiting materials on the water stability of asphalt mixtures has different laws. In this paper, the immersion Marshall test and freeze-thaw splitting test were used to study the effect of three kinds of ice-inhibiting materials on the water stability of the asphalt mixture. A Marshall compactor was used to compact the salt-storage asphalt mixture into a standard Marshall specimen with a diameter of 101.6 mm and a height of 63.5 mm, and 50 blows on both sides. For the immersion Marshall test, all the specimens in each group are kept in a constant temperature water bath at 60 °C. Among them, four specimens are kept for 30 min, and the other four pieces are kept for 48 h. After the heat preservation time is reached, the Marshall stability of all

specimens was tested, and the anti-stripping ability of the mixture was evaluated by the immersion residual stability  $MS_0$  calculated according to equation (3) [23]:

$$MS_0 = \frac{MS_1}{MS} \times 100 \quad (3)$$

In equation (3):  $MS_0$  = residual stability of the specimen immersed in water (%),  $MS_1$  = Marshall Stability of the specimen after being immersed in water for 48 h (kN),  $MS$  = Marshall Stability after 30 min of immersion (kN)

In addition to the immersion Marshall test, the freeze-thaw splitting test was also applied to evaluate the water stability of the salt storage mixture. In each group of specimens, four specimens were stored at room temperature for later use. The other four specimens were vacuum-saturated and bathed in water for 30 min. Then, the specimens were wrapped in a plastic bag and 10 ml of water was added to the bag. After conditioned at  $-18^\circ\text{C}$  for 16 h, the specimens were taken out and put in a constant temperature water tank at  $60^\circ\text{C}$  for 24 h. Finally, the specimens at room temperature and the specimens after water bath were kept in a constant temperature water bath at  $25^\circ\text{C}$  for not less than 2 h, and the splitting tensile strength was tested after the specimens were taken out. The water stability of the salt storage mixture was evaluated by the freeze-thaw splitting tensile strength ratio TSR calculated by equation (4) [23]:

$$TSR = \frac{ITS_2}{ITS_1} \times 100 \quad (4)$$

In equation (4): TSR = freeze-thaw splitting tensile strength ratio (%),  $ITS_1$  = freeze-thaw splitting strength under dry condition (MPa),  $ITS_2$  = freeze-thaw splitting strength under wet condition (MPa)

### 3.2 Antifreezing Performance Evaluation

The salt storage asphalt pavement is to add a certain amount of salt storage snow melting and ice inhibition materials to the asphalt pavement in advance to constitute for all or part of the mineral powder in it. In a wet environment, the snow

melting components are actively dissolved and resolved to reduce the road surface water solution freezing temperature to achieve the effect of inhibiting freezing and delaying road icing. In this paper, the ice inhibition performance of AFAC with three kinds of antifreeze agents was evaluated indirectly through measuring the salt storage asphalt mixture friction coefficient with BPT and WFT.

WFT is a walking low-speed antiskid tester suitable for indoor and field testing, and its appearance is shown in Fig. 4b. The previous research [24] shows that WFT is as reliable as or better than BPT and dynamic friction tester (DFT) in measuring pavement skid resistance at low speed, and there is a good correlation between WFT and BPT data. The correlation coefficient between WFT and BPT test data is 0.80. Besides, the measured data of WFT have less variability than those of BPT and DFT. The field test on the same road section showed that WFT is more efficient than the field measurement mode of BPT or DFT. At the same time, the test results of WFT are not vulnerable to the changes in operator walking speed and the thickness of water film applied.

The skid resistance of pavement is evaluated by WFT friction coefficient (WFC) calculated according to equation (5) [24]:

$$WFC = \frac{M}{R \times N} \quad (5)$$

In equation (5): WFC = longitudinal friction coefficient.  $M$  = torque measured by the torque sensor (N·m).  $R$  = radius of the test wheel (m).  $N$  = vertical load applied to the test wheel (N)

The BPT test is carried out in accordance with the test procedure of ASTM E303-93 (2018). After the test piece was kept at a temperature of  $-4^\circ\text{C}$  for 4 h, the initial BPN and WFC were tested. 15 g water was sprinkled evenly on the surface of the specimen. Thereafter, the specimen was rolled with a wheel tracking tester for 10 min and then was taken out for the measurement of BPN and WFC. The above procedure was repeated until BPN and WFC no longer decrease, indicating that the surface of the specimen has frozen at this time. The anti-icing stability (AIS) [25] and change rate of WCF were applied to characterize the anti-icing performance. Under negative temperature

**Fig. 4** Test devices for evaluation of antifreezing performance: **a** British pendulum tester, **b** walking frictional tester



conditions, the lower the BPN and WFC reduction rate, the longer the time required for icing, that is, the better the ice inhibition effect. AIS was obtained as equation (6) [25]. The size of slab specimens used in BPT and WFT tests was 500 mm × 500 mm × 50 mm [24]. For the mixture specimens containing different kinds of anti-icing agents, four specimens were prepared for each dosage, a total of 16 specimens.

$$AIS = \frac{BPN_0 - BPN_t}{t} \tag{6}$$

In equation (6):  $t$  is the total testing time (min)  $BPN_0$  = initial BPN value  $BPN_t$  = final BPN value

### 3.3 Ice Inhibition Persistence Evaluation

The persistence of ice inhibition performance refers to the ability of AFAC to maintain the function of snow melting and ice inhibition for a long time with the increase of service time. Whether the salt remains in the pavement material is the main factor that determines whether the salt-storage asphalt pavement can continue to play the role of ice suppression [17]. At the same time, the electrical conductivity of the solution increases with the increase of ion concentration. Therefore, this section measures the solution conductivity of the Marshall specimen at different immersion time and indirectly evaluates the ice inhibition persistence of AFAC through the change of electrical conductivity.

The Marshall sample was completely immersed in 500 mL deionized water at 25 °C and placed in a stainless steel container. Then, the electrical conductivity of the immersion solution was measured by a conductivity tester (DDS-11D), and the use of the conductivity meter can refer to the literatures [26, 27].

## 4 Experimental Results and Discussion

### 4.1 Evaluation of Pavement Performance

#### 4.1.1 High-Temperature Performance

Rutting test evaluated by dynamic stability (DS) is a practical engineering test method to simulate the actual wheel load applied on the pavement surface and form rutting. The test results are shown in Fig. 5. As detailed, Fig. 5 indicates that the DS values reduced with an increase of replacement ratio of all these three types of antifreeze filler. Following results could be obtained: (1) The DS values of asphalt mixtures with three kinds of antifreeze filler all decreased obviously with the increase of antifreeze filler replacement ratio. In

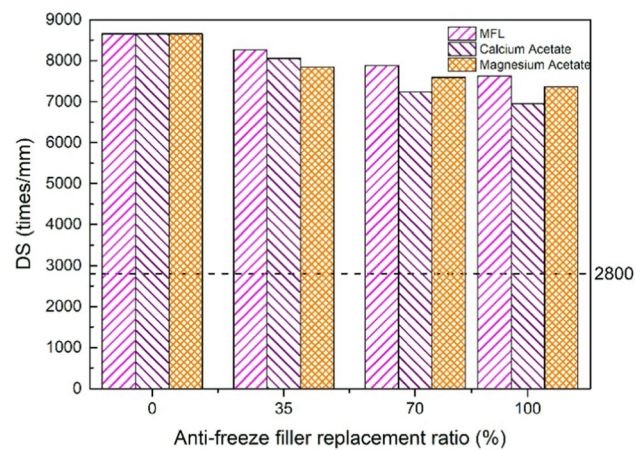


Fig. 5 Result of rutting test

the case of the 100% mineral powder replacement in the bituminous mixture by calcium acetate, the asphalt mixture retained its excellent high-temperature stability, and its DS value was up to 6954 times/mm, which satisfied with the current China specification requirements (not less than 2800 times/mm). (2) The high-temperature stability of antifreeze asphalt concrete (AFAC) is listed as: MFL>magnesium acetate>calcium acetate.

It may be contributed to three reasons: (1) the asphalt binder in asphalt mixture consists of two parts, one is structural asphalt that provides bond strength and mechanical strength, and the other is free asphalt which has not interacted with mineral aggregate and is harmful to the friction in structures. Although the antifreeze filler replaces the equivalent volume of mineral filler, the difference in the particle size, especially the size lower than 0.075 mm, leads to the increase of free asphalt which is not beneficial to the structural strength and cohesion of AFAC. (2) The mixture applied in this paper is AC-13, in which the coarse aggregates are suspended in fine aggregates. After the replacement, the coarse particle of antifreeze filler increased the distance between mineral aggregates and more content of antifreeze filler maybe change the microstructure of AC, leading to the decreased of DS. (3) The interaction between antifreeze filler and asphalt is much lower than that between mineral powder and asphalt, resulting in the decrease of the cohesion of asphalt mortar, leading to the reduction in shear strength of mixture and high-temperature performance.

It could be conducted that the addition of antifreeze filler has a negative effect on the high-temperature performance of the asphalt mixture. Therefore, the placement ratio of anti-freeze filler plays crucial role in determining the DS value of AFAC.

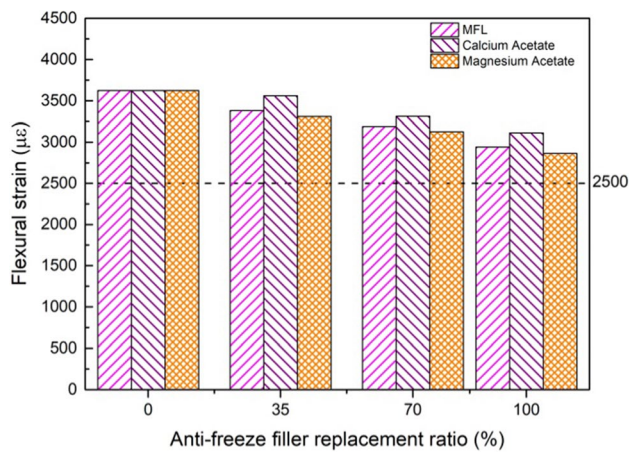


Fig. 6 Result of low-temperature flexural test

#### 4.1.2 Low-Temperature Performance

Figure 6 shows the low-temperature cracking resistance property of AFAC using the flexural strain value, measured by flexural test at  $-10 \pm 0.5$  °C. The flexural strains of specimens are found to decrease to a certain extent with the increase of replacement ratio of three types of anti-freeze filler. In most cases, a larger flexural strain indicates a better low-temperature performance. The low-temperature cracking resistance properties of three types of anti-freeze filler are in the arrangement as follows: calcium acetate > MFL > magnesium acetate. The maximum drop values of flexural strain were 14.1%, 18.82% and 20.94% for calcium acetate, MFL and magnesium acetate, respectively. The calcium acetate generally has the best cracking resistance, while the magnesium acetate has the worst one.

With the increase in the replacement ratio of antifreeze filler, the structural asphalt in the AFAC decreases. In other words, the antifreeze filler weakens the bond between aggregate and binder, thus reducing the self-healing capacity of concrete, resulting in the reduction in the low-temperature cracking resistance. Based on the standard requirement that the flexural strain of asphalt mixture at low temperature should not be less than 2500 μϵ in the cold region, the bending strain of the antifreeze asphalt mixture satisfied the standard requirement.

#### 4.1.3 Water Stability Performance

Figure 7 exhibits that the water stability of specimens containing anti-icing additives degrades with the replacement ratio. The more the anti-icing filler content is, the larger the reduction extends. The three types of anti-icing filler have a similar trend, and the addition of antifreezing filler has the same effect on the  $MS_0$  and TSR. It can be clearly seen

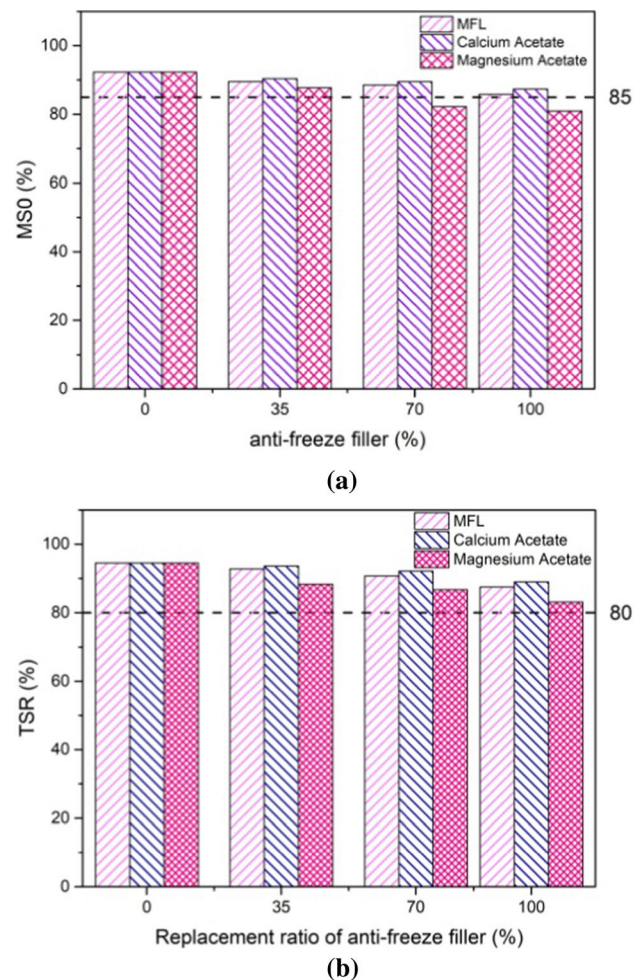


Fig. 7 Results of water stability tests: **a** Residual Marshall stability ratio; **b** tensile strength ratio

from Fig. 7 that whether it is MFL or calcium acetate, the values of  $MS_0$  and TSR are less affected by the increase in replacement ratio, and the decrease is relatively small compared to the control specimen. However, with the addition of magnesium acetate, the decrease of  $MS_0$  and TSR was more obvious than that of the other two additives. When the replacement amount is 70%, the value of  $MS_0$  has been lower than the requirements of the relevant specifications, but when all the mineral powder is replaced with magnesium acetate, the value of TSR still meets the requirements of relevant specifications (Figs. 8, 9).

As for the reasons, on the one hand, the increase of anti-icing materials makes the free asphalt in the mixture increase relatively, which weakens the adhesion between mortar and aggregate, affects the overall structural strength and leads to the decrease of DS. On the other hand, it is well known that the air void has a vital influence on the strength of the mixture structure. The solution of anti-icing materials leads to the increase of air void in AFAC, which brings a



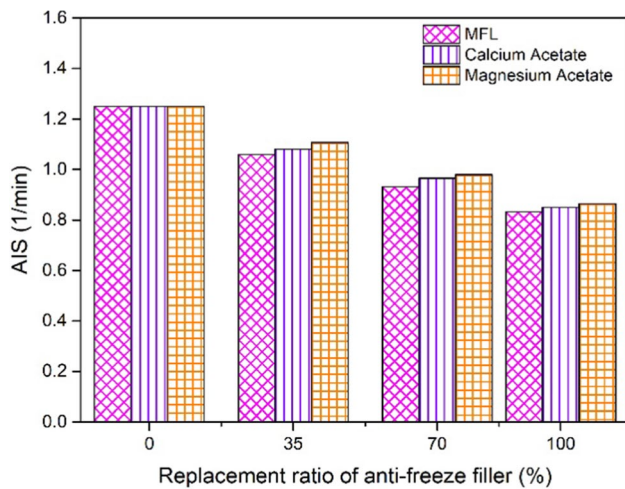


Fig.8 AIS of AFAC with different types of antifreezing filler

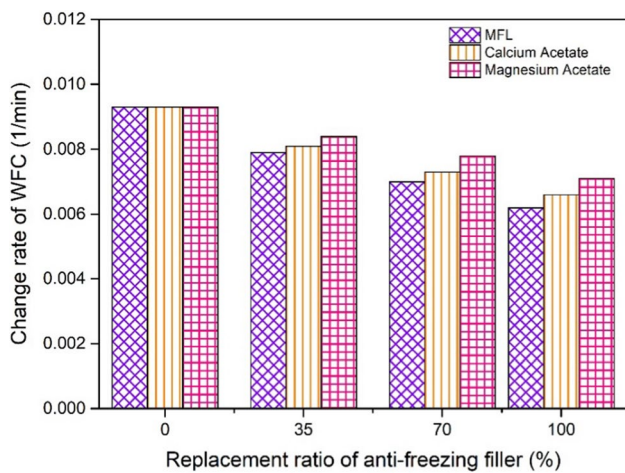


Fig.9 Change rate of WFC of AFAC with different types of anti-freeze agent

certain negative effect on the Marshall stability of the mixture. Besides, the sample was preserved in a water bath at 60 °C, which promoted the migration of antifreezing filler from the mixture to water to form salt solution, intensified the weakening of the bond between aggregate and binder, and destroyed the bond between asphalt mortar and aggregates. As a result, the asphalt spalling and other phenomena change the internal structure of the mixture, so that the Marshall stability significantly decreased. In addition, the salt solution may transfer from the high concentration area to the low concentration region, and the capillarity in this process will also weaken the adhesion between the mortar and the aggregate, resulting in the decrease of Marshall stability. During freeze-thaw cycles, the anti-icing filler may recrystallize and expand, thereby exerting additional stress on the pore surface. Especially when the replacement rate is 100%,

all sample groups already gave results below the values of the control samples.

### 4.2 Antifreezing Performance

When the pavement texture is filled and covered with dirt, the friction of the pavement surface is weakened, so that the road surface is covered with snow and ice. Therefore, to some extent, the ice resistance of pavement is related to the road surface texture, so the BPN and WFC reflecting the pavement texture can be used to characterize the anti-icing performance of pavement indirectly. The change rate of BPN and WFC per unit time can be used to characterize the ice resistance of different asphalt mixtures. Under negative temperature conditions, the lower the BPN and WFC reduction rate, the better the ice inhibition property of the road surface.

The test results illustrate that the attenuation rate of ice inhibition performance of AFAC pavement is obviously lower than that of ordinary pavement (asphalt mixture without antifreeze filler). With the increase in replacement rate of anti-icing fillers, the attenuation rate of BPN and WFC decreased significantly, indicating that the increase of anti-icing fillers in AFAC can effectively improve the antiskid and ice inhibition performance of the road surface. Under the same replacement ratio, for both BPN and WFC, the icing rate of AFAC is listed as: MFL < calcium acetate < magnesium acetate, which implies that the ice inhibition performance of MFL is the best, while that of magnesium acetate is relatively weak. BPN stabilized at about 23 (Table 5) and WFC stabilized at about 0.26 (Table 6), indicating that the surface of the specimen has frozen at this time, and the final BPN and WFC represent the ice resistance of the road surface at the moment. Continued water spraying and rolling will only increase the thickness of ice film on the surface of the specimen, but will not affect BPN and WFC.

If the AFAC mixture containing calcium acetate is paved on roads, the ice on pavement surface is easier to be removed by snow shoveling trucks compared to conventional asphalt mixture. Obviously, the mixture containing anti-icing filler can delay the freezing of rain and snow on the road surface under negative temperature and improve driving safety in winter. It is indirectly proved that the anti-icing filler can improve the driving safety of asphalt pavement during the snowy period in winter. The mixture containing anti-icing filler on the pavement surface can keep good antiskid performance in winter.

### 4.3 Ice Inhibition Persistence Evaluation

The persistence of ice inhibition performance refers to the ability of AFAC to maintain the function of snow melting and ice inhibition for a long time with the increase of service time. The anti-icing components embedded in the asphalt

**Table 5** BPN and AIS of AFAC with different types of antifreezing filler

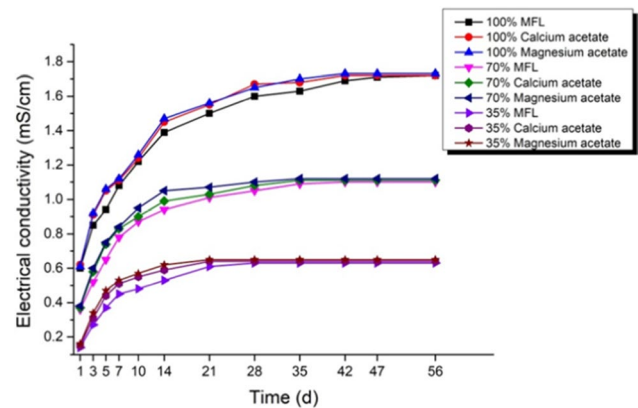
Antifreezing filler replacement ratio and types	Freezing time (min)										AIS
	0	10	20	30	40	50	60	70	80		
0% MFL	85.7	71.9	51.8	38.8	25.3	23.2	23.2	23.2	23.2	23.2	1.250
35% MFL	86.4	70.5	59.5	48.0	39.5	26.4	22.8	22.8	22.8	22.8	1.060
70% MFL	88.3	76.2	63.8	52.9	45.8	36.7	28.3	23.1	23.1	23.1	0.931
100% MFL	89.7	77.7	63.2	58.3	47.2	45.7	40.5	25.6	23.1	23.1	0.832
0% calcium acetate	85.3	72.1	52.8	38.6	25.9	22.8	22.8	22.8	22.8	22.8	1.250
35% calcium acetate	87.9	75.7	60.3	45.1	31.6	25.6	23.1	23.1	23.1	23.1	1.080
70% calcium acetate	90.5	82.2	65.2	57.2	45.5	30.8	26.3	22.9	22.9	22.9	0.966
100% calcium acetate	90.8	78.7	64.9	59.3	50.2	45.3	39.2	30.9	22.8	22.8	0.850
0% magnesium acetate	85.5	71.3	51.2	39.2	25.3	23.0	23.0	23.0	23.0	23.0	1.250
35% magnesium acetate	89.5	73.9	60.9	42.9	31.5	24.5	23.1	23.1	23.1	23.1	1.107
70% magnesium acetate	91.6	81.7	64.8	53.3	42.4	30.8	23.4	23.0	23.0	23.0	0.980
100% magnesium acetate	92.3	78.1	64.9	58.2	49.6	42.7	31.3	25.1	23.2	23.2	0.864

**Table 6** WFC and change rate of WFC of AFAC with different types of antifreeze agent

Antifreezing filler replacement ratio and types	Freezing time (min)										Change rate of WFC
	0	10	20	30	40	50	60	70	80		
0% MFL	0.720	0.582	0.422	0.331	0.285	0.255	0.255	0.255	0.255	0.255	0.0093
35% MFL	0.731	0.618	0.499	0.437	0.399	0.274	0.257	0.257	0.257	0.257	0.0079
70% MFL	0.748	0.759	0.536	0.470	0.460	0.391	0.261	0.258	0.258	0.258	0.0070
100% MFL	0.752	0.655	0.539	0.483	0.479	0.405	0.354	0.281	0.256	0.256	0.0062
0% calcium acetate	0.723	0.591	0.444	0.324	0.288	0.258	0.258	0.258	0.258	0.258	0.0093
35% calcium acetate	0.742	0.652	0.515	0.454	0.400	0.262	0.256	0.256	0.256	0.256	0.0081
70% calcium acetate	0.766	0.690	0.548	0.480	0.406	0.322	0.270	0.255	0.255	0.255	0.0073
100% calcium acetate	0.784	0.661	0.545	0.498	0.455	0.392	0.322	0.281	0.256	0.256	0.0066
0% magnesium acetate	0.724	0.599	0.430	0.323	0.291	0.259	0.259	0.259	0.259	0.259	0.0093
35% magnesium acetate	0.761	0.621	0.512	0.443	0.322	0.276	0.257	0.257	0.257	0.257	0.0084
70% magnesium acetate	0.802	0.686	0.544	0.473	0.457	0.323	0.284	0.256	0.256	0.256	0.0078
100% magnesium acetate	0.825	0.756	0.638	0.525	0.475	0.354	0.321	0.263	0.257	0.257	0.0071

pavement structure are dissolved, migrated and diffused to the road surface under the combined effect of chemistry and mechanics [28], leading to the reduction in freezing point of the road surface water solution and the delay of road surface icing in snowfall weather. Therefore, the change of electrical conductivity of salt solution produced by immersion of Marshall specimen over time can reflect the release law of ice inhibition components in AFAC, so as to further evaluate the anti-icing persistence of AFAC.

Three rules can be found from Fig. 10 illustrating the change of the electrical conductivity of the solution with the immersion time of the samples. Firstly, the change of electrical conductivity with time is clearly divided into three stages. The first stage is the rapid precipitation stage of ice inhibition materials, the second stage is that the stage during which the precipitation rate is slowed down, and the third stage is the period in which ice inhibition materials almost no longer precipitate. The reason for the phenomenon in



**Fig.10** Electrical conductivity of AFAC

the first stage is that part of the ice inhibition materials are directly exposed on the specimen surface under compaction

during the process of specimen forming, and some of the ice inhibition materials are wrapped in the mortar on the specimen surface. When the specimen is immersed in deionized water, these two parts of ice inhibition materials can dissolve quickly, resulting in a rapid increase in the electrical conductivity of the solution. In the second stage, the growth rate of electrical conductivity is slower since the salts exposed on the surface of the specimen have been almost completely dissolved, and the ice inhibition material wrapped inside the specimen gradually precipitates from the inside to the surface through the capillary action. Therefore, the growth rate of electrical conductivity slows down. In the third stage, the electrical conductivity almost stops growing, and the reason is that all the anti-icing materials on the surface of the specimen have been dissolved, and only part of the ice inhibition material inside the structure has not been dissolved and precipitated, which was affected by the compactness of the AC structure. This part of ice-inhibiting material is difficult to precipitate [29], and thus, the electrical conductivity remains basically unchanged.

The second rule found is that the conductivity increases obviously with the increase of replacement rate under the same immersion time, since the physical meaning of conductivity is the ability to transfer electrons in the solution. In AFAC, the increase of the replacement rate of anti-icing material means that the more ion content in the solution under the same immersion time, resulting in higher conductivity.

The third rule observed is that under the same immersion time and the same replacement rate, the conductivity of MFL is relatively small, and it takes a longer time for MFL to reach a stable conductivity value, which means that MFL can play an ice inhibition role continuously for a long time. The conductivity growth rate of calcium acetate and magnesium acetate is not much different. More specifically, the conductivity growth rate of calcium acetate is slightly lower than that of magnesium acetate. Therefore, the anti-icing persistence of MFL is better. In addition, the time for the electrical conductivity to reach the stable value is prolonged with the increase of the ice-inhibiting filler replacement rate, and the time for the electrical conductivity of the immersion solution mixed with MFL to reach the stable value is slightly longer. For example, for magnesium acetate and calcium acetate, the replacement rate is 35%, 70% and 100%, the corresponding stabilization time is 21 d, 35 d and 42 d, respectively. For MFL, the stabilization time is 28 d, 42 d and 47 d. This corresponds to the conclusions of the previous section.

In a conclusion, experimental studies show that adding ice-inhibiting fillers into asphalt mixtures could effectively improve the anti-icing ability of pavement, but part of the road performances will be sacrificed. In particular, compared with MFL, acetate ice-inhibition fillers have more

high-temperature stability losses, besides, they have some room for improvement in the anti-icing capacity and ice inhibition persistence. In order to balance road performance and anti-icing capacity, the radar graph shown in Fig. 11 were applied. In a radar chart, the larger the area, the better the overall performances. Therefore, it can be seen that when MFL is applied as the ice inhibitor for self-melting snow pavements, 35% is recommended as a reasonable replacement rate. When acetate is chosen as the anti-icing additive, a reasonable replacement rate of 35% for calcium acetate is recommended.

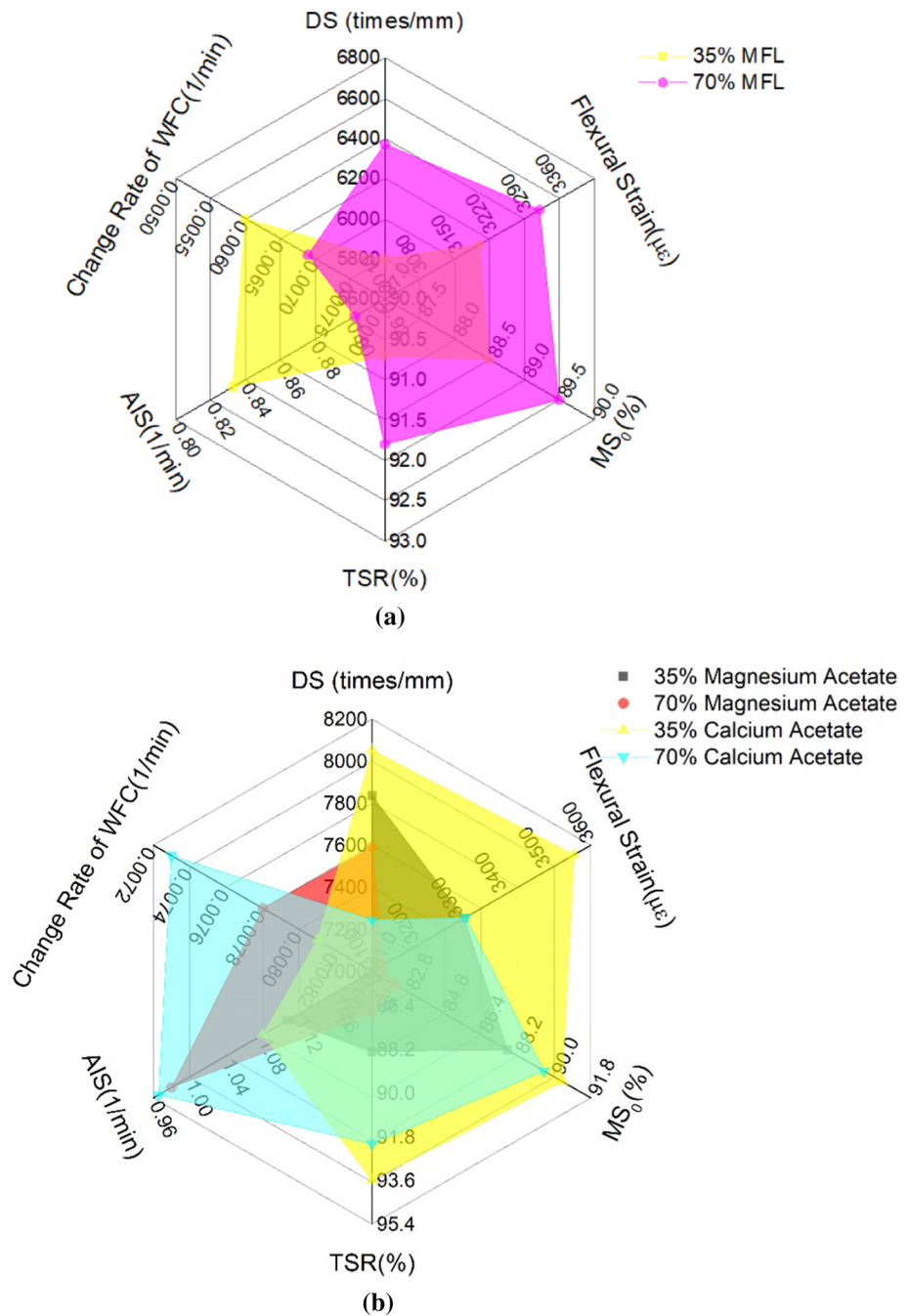
## 5 Conclusions

In this paper, the equivalent volume replacement method is applied to replace the mineral filler in the mixture with ice inhibition filler MFL, calcium acetate, and magnesium acetate, and the replacement ratio is set to 0%, 35%, 70% and 100% for each type of deicing agent. The engineering performance and anti-icing capacity of salt storage asphalt mixture containing these three kinds of antifreeze agents with different dosages were investigated. The performance investigated include anti-rutting performance, thermal cracking resistance, water damage resistance, anti-icing capacity as well as ice inhibition persistence. Findings and conclusions can be summarized as follows:

- (1) Partially replacement of mineral fillers by anti-icing additives leads to the compromised road performance of asphalt mixture, especially in high-temperature stability. This phenomenon is not caused by a single factor, but is the integrative result of the bond weakening caused by the reduction of effective mortar, the change of mixture structure and the void varying caused by the salt dissolving.
- (2) Asphalt mixtures added with anti-icing additives would achieve a significant improvement in ice inhibition property of asphalt pavement, since they effectively restrain and delay the icing speed of the pavement. Besides, the weakened bonding force between the road surface and ice layer make the ice layer easy to be removed and improve the driving safety in snowy weather.
- (3) By comparing the effects of three kinds of ice inhibition fillers with different dosages on the road performance and anti-icing capacity of asphalt mixture, it is found that MFL has the best comprehensive performance. MFL performs best in terms of high-temperature performance, anti-icing capacity and ice inhibition persistence, while calcium acetate performs best in low-temperature performance and water stability. Thus, improving the overall performance of acetate can be



**Fig. 11** Comprehensive performances evaluation: **a** Overall performances evaluation of MFL with different replacement ratio, **b** overall performances evaluation of acetate ice-inhibition fillers with different replacement ratio



considered in terms of high-temperature performance, anti-icing capacity and ice inhibition persistence.

- (4) It can be found from the conductivity test that the electrical conductivity remained almost stable in the later stage of the test. In fact, there are still anti-icing fillers in the mixture; however, the dense structure of AC makes it interlocked inside the structure, preventing the anti-icing additives from exerting its ice inhibition

effect. Therefore, it can be considered from the mixture design aspect to improve the functional persistence of the salt storage asphalt pavement.

- (5) Future research will pay more attention to the mechanism investigation of AFAC engineering performance and the comprehensive performance optimization of environment-friendly anti-icing additives.

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## Declarations

**Conflict of interest** On behalf of all authors, the corresponding author states that there is no conflict of interest. We declare that we do not have any commercial or associative interest that represent a conflict of interest in connection with the work submitted.

**Data Availability and Material** All data are fully available without restriction.

**Consent for Publication** Written informed consent for publication was obtained from all participants.

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