Research Article

Preparation of long-lasting environment-friendly anti-icing agent suitable for snow and ice areas and its performance research

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Abstract

The addition of anti-icing agents to asphalt pavements as a way to prevent snow and ice residues should consider not only the environmental aspects of anti-icing agents, but also the water stability, high temperature stability and low temperature crack resistance of asphalt mixes. In this work, environmentally and friendly slow-release anti-icing agent called MAS was produced by using silica microspheres coated with magnesium acetate, and the performance of the mixes after adding anti-icing agents was analyzed by the water stability, high temperature stability and low temperature crack resistance tests The experimental results show that when the MAS replaces 6% of 0–0.075 mm aggregate by equal volume, the strength and performance of the mixture meet the requirements and have the best slow-release effect.

Keywords Slow release · Environmentally · Melting · Anti-icing agent

1 Introduction

Anti-icing agent is an admixture that replaces materials of different particle sizes in the asphalt mixture. Thus, the asphalt pavement has an automatic function of melting snow and ice. The salt of the main melting agent diffuses to the surface layer of the road through capillary pressure and traffic load, lowering the ice freezing point and playing the role of melting snow and ice [1].

Most of anti-icing agents are mainly chlorine salts. Switzerland was the first country to developed V-260, which is mainly composed of calcium chloride [2, 3]. Subsequently, Japan developed MFL, which is mainly composed of sodium chloride, magnesium chloride and calcium chloride [4]. In China, there are anti-icing agents prepared from calcium chloride, urea and heavy calcium superphosphate as raw materials [5] and anti-icing agents made by mixing calcium chloride with urea and sodium nitrite [6]. Although the application of anti-icing agents significantly reduces the amount of chlorine salt compared to direct seeding of chlorine salt anti-icing agent, there is still amount of harm to pavement and surrounding soil and water quality [7, 8]. It also affects the ion concentration of surface water, leading to an increase in other harmful substances [9, 10].

As a result, non-chlorine salt anti-icing agents are widely developed. Typical representatives include the CMA developed by the U.S. DOT with acetate as the main component [11]. Kris et al. [12] formulated a low-corrosive anticoagulant with ammonium succinate trihydrate, potassium succinate trihydrate, and sodium succinate trihydrate. In China, there are also anti-icing agents prepared from furfural wastewater [13] and nylon industrial by-products [14].

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However, organic anti-icing agents are generally more expensive, and non-chlorine salt anti-icing agents generally have poor slow-release properties [15], so they are not widely used. Therefore, it has become a priority to develop environmentally friendly anti-icing agents that are inexpensive and have slow-release properties.

In this work, we used silica microspheres to coat the magnesium acetate to obtain MAS anti-icing agent with slow-release properties and mixed it into the asphalt mixture in a certain proportion. Wang et al. [16] pointed out that salt accumulation asphalt reduces the water stability of asphalt mixture, and the silica microspheres in this study played a slow-release and supportive role, which not only prolonged the service life, but also reduced the effect of anti-icing agent on the asphalt mixture performance.

2 Experimental section

2.1 Materials

Acetic acid (99.9%, Sinoreagent), Ethyl orthosilicate (TEOS, 99.9%, Sinoreagent), Anhydrous ethanol (99.7%, Sinoreagent), Mineral powder (HAICHENG NO.2 TALC POWDER FACTORY CO.,LTD.), Aggregate (HAICHENG NO.2 TALC POWDER FACTORY CO.,LTD.), Magnesium acetate ($Mg(AC)_2$, 99%, Tianjin Ruijinte Chemicals Co., Ltd.), Sodium acetate (NaAC, 99%, Tianjin Ruijinte Chemicals Co., Ltd.), Calcium acetate ($Ca(AC)_2$,99%, Tianjin Ruijinte Chemicals Co., Ltd.), Ammonia (25%, Sinoreagent), SBS modified asphalt (AC-13, Shenyang Huasheng Water-Proof Engineering Co., Ltd.), Sodium chloride (99%, Tianjin Ruijinte Chemicals), Sodium hydroxide (99.5%, Tianjin Ruijinte Chemicals) are used without further purification.

2.2 Preparation of MAS

Add ethanol to the main melt for stirring first, add deionized water, and ammonia drop by drop to make dispersion, adjust the PH between 8 and 10. Different masses of TEOS are weighed and quickly added to the dispersion. Quickly stir (1800 rpm) for 1 min, then slowly stir (900 rpm). Filter at the end of stirring, wash and dry with anhydrous ethanol.

2.3 Characterization

Observation of sample morphology using scanning electron microscope (HITACHI S-4800).

2.4 Preparation of anti-icing asphalt mixes

Replace different proportion of 0–0.075 mm particle size aggregates with equal volume of anti-icing agent. Add anti-icing agent in the mixing process of asphalt mixture, design asphalt mixture according to the specification and adjust it according to the actual situation [17]. Cylindrical specimens of asphalt mixture were prepared and the optimum oil to stone ratio was obtained by Marshall experiment.

2.5 Performance evaluation of anti-icing asphalt mixes

Conductivity meter (DDS-11A) is used to test electric conductivity (EC). The more salt ions that are dissolved, the greater the EC value of the aqueous solution. The EC test involves weighing the same mass of the target sample into deionised water and using a conductivity meter to determine the EC value of the sample over a certain period of time. The value reflects the solubility of the anti-icing agent and thus establishes the relationship between the retarding effect and EC.

Marshall tests are carried out using the Marshall stability meter (LWD-3A) to calculate the optimum Oil-stone ratio. The test process involves standard compaction of the specimens under specified conditions of temperature and humidity to determine certain indicators of the asphalt mixture, which are then calculated to determine the optimum Oil-stone ratio.

Digital display hydraulic universal testing machine (WES-1000D) is used to measure creep deformation amount. The procedure is to apply a transient load to the specimen in the axial direction and to hold the load of this magnitude constant so that the specimen remains in a deformed state for a certain period of time. When the load is unloaded, some of the deformation of the specimen is recovered and the trend in creep deformation is obtained, allowing the high temperature stability of the asphalt mixture to be evaluated.

Bending and tensile strain and splitting strength, using automatic rutting meter (ZCZ-10A) to measure ruttin depth (RD) and dynamic stability (DS). DS is an index for the study of rutting deformation and refers to the number of trips of a standard axle load for every 1 mm of deformation of the asphalt mix under high temperature conditions.

3 Results and discussion

Snow melting and ice melting capacity experiments refer to the operation of *Road deicing anti-icing agent (GB/T23851-2009)* [18], in which the same mass concentration of anti-icing agent solution is used. The initial mass and

Table 1 Volume of liquid melted by different acetate salts

Acetate salts	Volume of melted liquid (ml)
NaAC	3.5
Mg(AC)2	4.6
Ca(AC)2	4.2

Table 2 Table of factor levels

Level	Factors		
	A (Temperature, °C)	B (TEOS dosage, ml)	C (pH)
1	20	10	8
2	30	15	9
3	40	20	10
T	3.63	2.24	7.57

the mass of liquid poured out after 30 min are recorded, and the experimental data are brought into the following equation to derive the corresponding snow melting and ice melting capacity ω of the anti-icing agent solution of this mass concentration.

$$\omega = \frac{m_0 - m_1}{m_0^1 - m_1^1} \times 100\% \tag{1}$$

where m_0 and m_0^1 are the masses of the beaker and ice; m_1 is the mass of the beaker and ice to which the anti-icing agent solution was added; and m_1^1 is the mass of the beaker and ice to which the sodium chloride solution was added. All weight units are in grams.

The relationship between the resulting acetate and the melted liquid is shown in Table 1. $Mg(AC)_2$ have the highest volume ofliquid dissolved at 4.2 ml, followed by $Ca(AC)_2$, and NaAC have the smallest volume of liquid dissolved at 3.5 ml. From the molecular formula of NaAC, $Mg(AC)_2$ and $Ca(AC)_2$, it can be seen that the number of particles of $Mg(AC)_2$ and $Ca(AC)_2$ in the same concentration is larger than that of NaAC, so the ice melting effect of $Mg(AC)_2$ and $Ca(AC)_2$ is better than that of NaAC. The solubility of $Mg(AC)_2$ and $Ca(AC)_2$ at 0 degrees in water are 56.7 g/cm^3 and 37.4 g/cm^3 respectively. The solubility of $Mg(AC)_2$ is greater than $Ca(AC)_2$, the number of ions dissolved in water is more than that of $Ca(AC)_2$, and the freezing point is reduced more significantly. Therefore, $Mg(AC)_2$ is chosen as the main melting agent.

While using $Mg(AC)_2$ as the main melting agent, the orthogonal experiments are carried out according to $L_9(3^4)$ orthogonal table with the reaction temperature, TEOS dosage and pH as factors. The factor levels and results are presented in Table S1 and Table 2.

As shown in Table 2, it can be obtained that $T_C > T_A > T_B$. That means the order of the magnitude of the influence of these three factors on the experimental index is pH > temperature > TEOS. By the magnitude of δ value, it is known that the optimal solution is $A_3B_2C_3$, i.e., the solution with the reaction temperature of 40, TEOS dosage of 15 ml and pH of 10. The more ammonia is used as a basic catalyst, the higher the amount and the higher the pH, the faster the reaction. However, it will be more difficult to produce silicon dioxide in the form of microsphere at higher pH. The higher the temperature, the faster the reaction. But high temperature condition will lead to a decrease in the agglomeration stability of the microsphere forming nuclei. The amount of TEOS is too low to coat the main melt. High dosage increases the particle size of microsphere [19].

Therefore, a reaction temperature of 40 °C, a TEOS dosage of 15 ml and a pH of 10 are the best preparation conditions. The SiO_2-coated $Mg(AC)_2$ anti-icing agent prepared under these conditions is called MAS. Observations before and after MAS synthesis using SEM are shown in Fig. 1.

From Fig. 1a, the surface is smooth before coating. After coating treatment, at the same magnification, the silicon dioxide microspheres encapsulated on the surface of the master melt are visible by Fig. 1b. With further magnification, a few microspheres are visible from Fig. 1c. Because TEOS first forms nuclei under alkaline conditions and gradually grows to form nanoscale particles, with a small amount of aggregation in the process, forming a microsphere buildup. From Fig. 1d, the size of microspheres is between 900 nm and 2 μm, and the size is relatively uniform. Most of them are micron level microspheres.

EC of MAS and $Mg(AC)_2$ were tested at different times after addition to water to provide a comparative analysis of the retardation of the two anti-icing agents, the results of which are shown in Fig. 2.

From Fig. 2, the EC starting points of MAS and $Mg(AC)_2$ are different. $Mg(AC)_2$ diffuses directly in the water without coating, there is no slow-release effect. Resulting in a high EC starting point and a fast rise rate. The EC no longer changes after $Mg(AC)_2$ completely dissolved. Because of the presence of the membrane, MAS dissolves at a much lower rate, the starting point of EC is lower than that of $Mg(AC)_2$, and the rate of EC rise is smoother than that of $Mg(AC)_2$.

The above tests established that MAS has good retarding properties compared to $Mg(AC)_2$. It was added to the mix to prepare anti-icing asphalt mixes. Different proportions of aggregates of 0 to 0.075 mm particle size were replaced by equal volume of anti-icing agent. The gradation design calculation is shown in Table S2 and the Marshall test results in Table S3. According to the Marshall test results, the Oil-stone ratio a_1 for the density maximum is

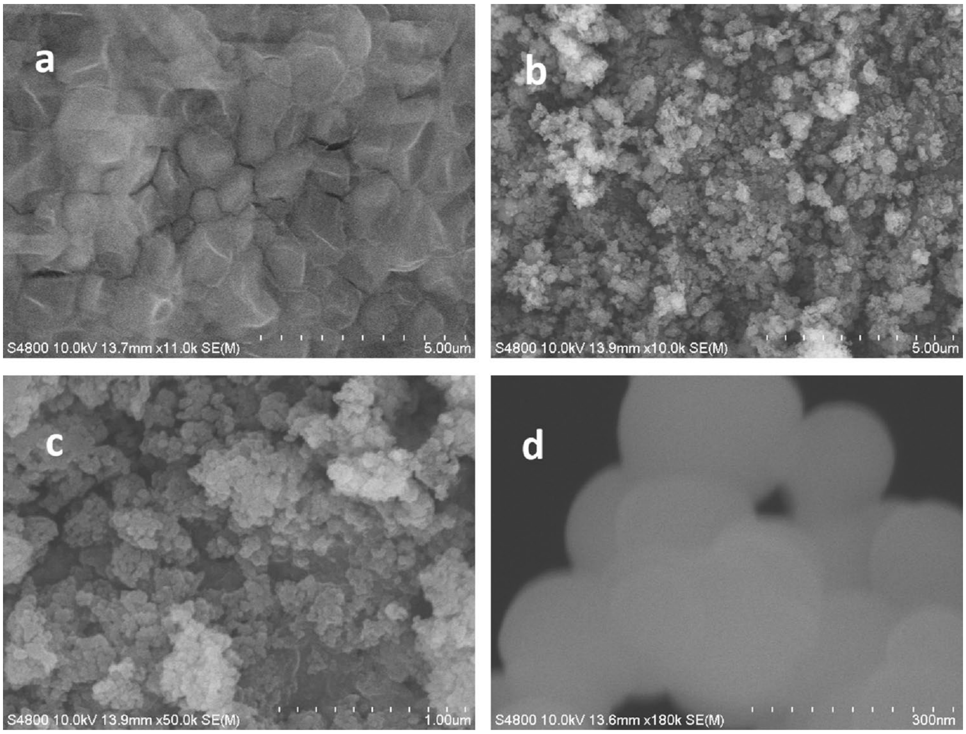

Fig. 1 **a** SEM images of $Mg(AC)_2$, **b**–**d** SEM images at different magnifications of MAS

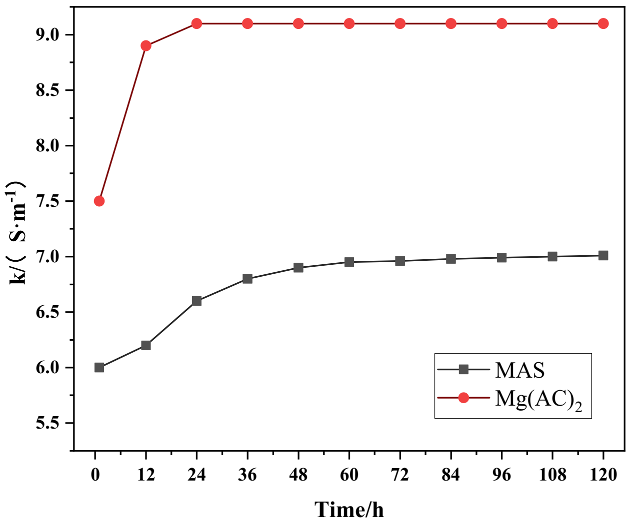

Fig. 2 Relationship between EC value and time, Temperature = 60 °C. Sample mass = 10 g. Volume of deionised water = 400 ml

5.6%, the Oil-stone ratio a_2 for the stability maximum is 4.95%, the Oil-stone ratio a_3 for the median void ratio is 5.79%, and the Oil-stone ratio a_4 for the median asphalt saturation is 4.97%.

This leads to:

$$OAC_1 = \left(a_1 + a_2 + a_3 + a_4\right)/4 = 5.33\%$$

$$OAC_{min} = 4.52\%,\ OAC_{max} = 5.46\%$$

$$OAC_2 = \left(OAC_{min} + OAC_{max}\right)/2 = 4.99\%.$$

In summary, $OAC = (OAC_1 + OAC_2)/2 = 5.16\%$, i.e., the optimal Oil-stone ratio is 5.16%.

Marshall dip rutting test using optimum Oil-stone ratio. SBS modified asphalt and the measured gross bulk density of standard Marshall specimens was used to make 36 pieces of 300 mm × 300 mm × 50 mm rutting plates by wheel mills, which were divided into four groups of nine pieces each, keeping the same number of pre- and post-milling. Eight of the rutted slabs had MAS and $Mg(AC)_2$ added to the asphalt mixture, and the other one served as a blank control group without anti-icing agents.

The group 1 is not pre-soaked. Except for the group 1, the rutting plates of group 2–4 were pre-soaked in a container filled with aqueous solution at 298 K before the formal start of the experiment. The pre-soak time for each group was increased by 48 h compared to the previous group. At the beginning of the experiment, the specimens were placed in a constant temperature water bath at 60 °C for 6 h, weighed and recorded, and then the rutting experiments were carried out at the same temperature of 60 °C. At the end of the experiment, the specimens were weighed and recorded again, and the DS values were obtained according to the number of walks and RD.

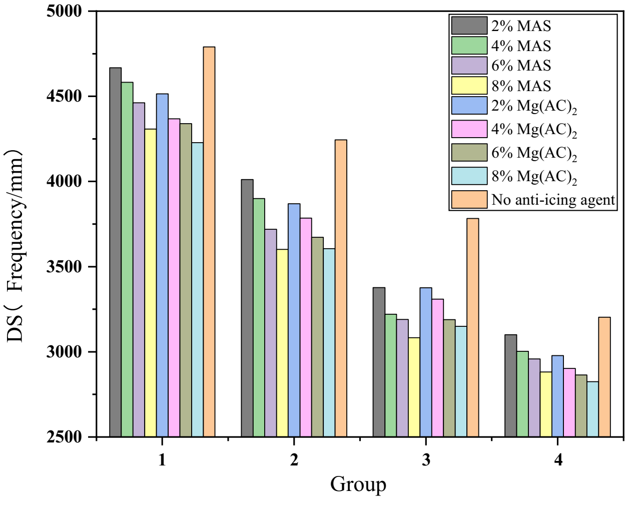

Fig. 3 DS value. From group 1 to group 4, the pre-soak times were 0 h, 48 h, 96 h and 144 h respectively

The values of DS in each group of the blank control group in Fig. 3 are the largest, which indicates that the admixture of anti-icing agent has some effect on the DS of the mixture. After the specimen is immersed in water, the active ingredient in the anti-icing agent gradually dissolves in water, resulting in more gaps and voids in the mixture, making the specimen more susceptible to deformation under dynamic vehicle loading. The greater the admixture, the lower the DS. The reason for the decrease in asphalt adhesion is that after the asphalt is soaked in aqueous solution, the aqueous solution penetrates into the asphalt through displacement and adsorption, resulting in a decrease in the adhesion of the asphalt and the aggregates. At the same time, acetate also emulsifies the asphalt, leading to a further reduction in adhesion [20]. The DS of Mg(AC)$_2$ doped is lower compared to the same amount of MAS doped.

SiO$_2$ microspheres in MAS not only play a slow-release effect, but also play a role in supporting the void space, so that the active ingredients of MAS dissolved while still maintaining the asphalt mixture void ratio remains unchanged or slowly increases and there is a higher DS than Mg(AC)$_2$. An obvious curve of gentle decrease can be seen in Fig. 3.

The Technical Specification for Asphalt Pavement Construction (JTGF40-2004) [21] stipulates that the DS of modified asphalt mixes is not less than 2800 in the hot summer zone, 2400 in the hot summer zone and 1800 in the cool summer zone. All the asphalt mixes in this experiment meet the technical requirements of the rutting test.

The uniaxial static load creep test was used to analyze the high temperature deformation characteristics of asphalt mixture. The standard Marshall specimen is prepared and the RD is measured. The results are shown in Table 3.

From Table 3, with the increase of admixture dosage, the RD of the specimen is also greater. Because the amount of admixture affects the skeletal embedding of minerals in the asphalt mixture, which also plays an important role in the rutting resistance of the asphalt mixture. Too much mixture will weaken the embedding force between minerals and make the mixture easy to flow deformation and form rutting. The deformation of the specimen with Mg(AC)$_2$ is larger than MAS, because the active ingredient in the Mg(AC)$_2$ is only acetate, while the active ingredient in the MAS is not only acetate but also SiO$_2$ microspheres, which play a supporting role for the mineral skeleton and make the asphalt mixture relatively more rutting resistant.

According to *Standard for Evaluation of Technical Condition of Highways (JTJH20-2007)*, [22] RD of asphalt pavement is divided into five grades: excellent, good, moderate, inferior and poor, with RD ≤ 5 mm, 5–10 mm, 10–15 mm, 15–20 mm and > 20 mm respectively. All the admixture mixes in this experiment meet the specification requirements.

The low-temperature bending test was selected to test and evaluate the low-temperature crack resistance of asphalt mixture. The results of the experiment are shown in Table 4.

From Table 4, compared with the blank control group, the bending and tensile strains of the specimens gradually decreased with the increase of the anti-icing agent admixture. Since the active ingredients of the anti-icing agent added in the specimens would be dissolved and precipitated during the process of cutting the beams and low-temperature freeze–thaw cycle test. Therefore, when the loading was carried out, the bending and tensile strains of the beams of the blank control group were larger and less likely to break, while the bending and tensile strains of the beams with the addition of the anti-icing admixture were

Table 3 Creep deformation of specimens with different amounts of anti-icing agent

Specimen type	RD (mm)
No anti-icing agent	1.1
4% MAS	1.3
6% MAS	1.7
8% MAS	2.5
10% MAS	3.2
4% Mg(AC)$_2$	1.4
6% Mg(AC)$_2$	1.9
8% Mg(AC)$_2$	3.0
10% Mg(AC)$_2$	3.8

Loading stress = 0.7 MPa, Temperature = 60 °C, Loading time = 60 min, Unloading time = 20 min

Table 4 Low temperature bending test results for standard Marshall specimens with different admixtures

Specimen type	Bending and pulling strain (με)
No anti-icing agent	3122
4% MAS	2993
6% MAS	2911
8% MAS	2748
10% MAS	2687
4% $Mg(AC)_2$	2898
6% $Mg(AC)_2$	2812
8% $Mg(AC)_2$	2682
10% $Mg(AC)_2$	2490

Loading rate = 50 mm/min, temperature = – 10 °C

smaller. Compared with MAS, $Mg(AC)_2$ makes the bending and pulling strain smaller because MAS has a slow-release effect, and the low-temperature experiment does not allow the active ingredients to be released quickly for dissolution. $Mg(AC)_2$ has no slow-release effect, the active ingredients quickly dissolve, the loaded beam bending and pulling strain is smaller, the asphalt mixture of low-temperature flexibility is reduced.

According to *The Technical Specification for Construction of Asphalt Pavements on Highways (JTGF40-2004)* [21], the technical requirements for the low temperature bending test of modified bitumen mixes in China are: the breaking strain in the severe winter zone is not less than 3000, in the freezing winter zone is not less than 2800, in the cold winter zone and warm winter zone is not less than 2500. With the exception of the last group, all the admixture mixes in this experiment met the specification requirements.

Test the change of salt dissolution of anti-icing asphalt mixture by conductivity meter [23]. The next step was to test the snow and ice melting and retarding properties of the anti-icing asphalt mix. The relationships between sample EC and time are shown in Fig. 4.

As shown in Fig. 4, the starting EC sample 2 is greater than sample 1.Because the same volume of sample 2 is denser than sample 1 and the main melt content is greater than sample 1 when the equal volume of substituted aggregates, the starting EC is greater than sample 1. With the change of time, the main melt in sample 2 does not have a slow-release effect, due to rapid dissolution resulting in a rapid increase in EC, which has been completely dissolved at 36 h, and thereafter EC no longer changes. The change in EC over time for sample 1 can be divided into three stages because of the silica microsphere coating.

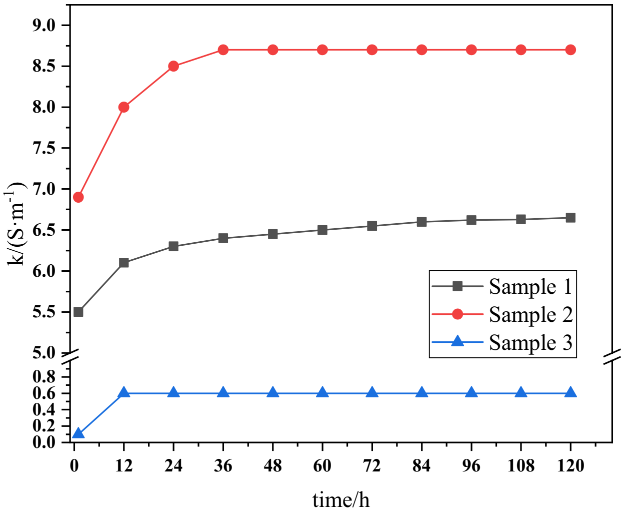

Fig. 4 Relationship between sample EC and time. Sample 1 (replacing 6% of 0–0.075 mm aggregate with equal volumes of MAS), Sample 2 (replacing 6% of 0–0.075 mm aggregate with equal volumes of $Mg(AC)_2$), Sample 3 (without anti-icing agent). The rise in EC of sample 3 was due to the inevitable presence of a small amount of impurities

Because of the percussive method of forming the specimen, a small portion of the primary melt precipitates from the surface of the specimen and rapidly forms a salt solution when it meets water. Therefore, the first stage of DC growth is more obvious. In the second stage, the DC increases steadily as the primary melt continues to dissolve on both the surface and the interior of the specimen. In the third stage only the slow dissolution of the primary melt in the internal MAS leads to a plateauing of the DC.

The DC still had a slow upward trend at 120 h, which also indicates that there is still a slow release effect at this point. Experimental result of replacing 6% of 0–0.075 mm aggregate with equal volumes of MAS was the best of all options in this experiment. The above series of experiments demonstrate that the optimum retarding effect and duration of use is achieved while maintaining the basic properties of the asphalt mix.

In general, the mechanism of MAS is shown in Fig. 5. When the ambient humidity and temperature of the atmosphere differs from the depth of the road surface layer and the pavement layer, and the asphalt pavement is subjected to compression, vibration and friction loads from moving vehicles, the MAS mixed into the asphalt mix is slowly pumped and migrated from different depths to the surface layer of the pavement to be released under the action of pore penetration pressure and capillary pressure.

4 Conclusion

In summary, $Mg(AC)_2$ was identified as the main melting agent for the anti-icing agent. SEM showed that silica microspheres with particle sizes ranging from 900 nm to

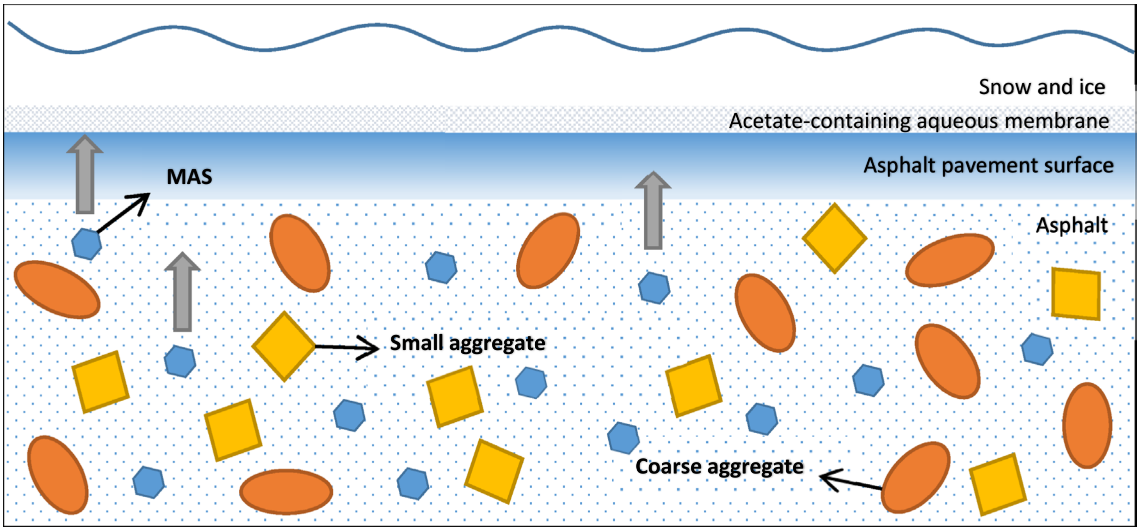

Fig. 5 Snow and ice melting mechanism of MAS added to asphalt mixes

2 μm were successfully wrapped around the surface of magnesium acetate. The water stability, high temperature stability and low temperature crack resistance tests showed that the best results were achieved in terms of slow release of the anti-icing agent and the performance of the asphalt mixture when 6% volume of 0 to 0.075 mm aggregate was replaced by MAS and it complies with standards for asphalt pavements which means the cladding does not significantly reduce the performance of the asphalt mix. Conversely, silica microspheres can provide the framework support property. The salt solubility test shows that the asphalt mixture of 120 h still has retarding effect, it demonstrates the retardation and snow-melting properties of anti-icing asphalt mixes.

It is worth noting that the frequency with which a road is crushed is closely related to the length of time the MAS is used. It is necessary to design asphalt pavements with different MAS additions depending on the volume of traffic.

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Data availability and code availability All data generated or analysed during this study are included in this published article and its supplementary information files.

Declarations

Conflict of interest No potential conflict of interest was reported by the authors.

Ethical approval Ethics approval was not required for this research.

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