

Preparation of Salt-Storage Aggregate using Magnesium Oxychloride Cement and Investigation on Salt-Release Performance

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Received 13 December 2023; Accepted 26 February 2024

Abstract

To investigate the salt-release performance of salt-storage aggregates, salt-storage aggregates were prepared using magnesium oxychloride cement as a carrier through the addition of chloride salts and sustained-release agents. The different types of salt-storage aggregates were compared and selected through compressive strength and immersion conductivity tests. In addition, the salt-release performance of salt-storage asphalt mixtures was investigated by immersion conductivity tests and simulated rain erosion tests. The results indicate that as a sustained-release agent, polyethylene glycol has a certain adverse effect on the compressive strength of salt-storage aggregates, whereas glycerol has an enhancing influence during 28 days of standard curing conditions. The salt-storage aggregates with sustained-release agents show a good sustained-release effect and a relatively stable conductivity growth. Compared with polyethylene glycol, glycerol attains a better sustained-release effect on salt-storage aggregates with CaCl₂ as a snow-melting agent. Under short-term immersion conditions, the conductivity of salt-storage asphalt mixture specimens increases rapidly with prolonged immersion. Under long-term immersion conditions, the conductivity changes under different replacement rates show two stages, namely, the early stage featuring a rapid increase and the later stage showing a gradual trend. The salt-dissolved regularity of salt-storage asphalt mixture conforms to the logarithmic model. After 5 years of equivalent rainwater erosion, the salt-storage asphalt mixture can still release salt. The study provides a good reference for the production and application of salt-storage aggregate in road maintenance.

Keywords: Road engineering, Magnesium oxychloride cement, Salt-storage aggregates, Salt sustained release performance, Conductivity

1. Introduction

Two-thirds of China's territory is located in the winter-snow accumulation region, with several areas experiencing snow cover for as long as 3–4 months. During winter, over 70% of roadways in these areas face icy and snowy weather conditions [1]. The road surface's antiskid performance deteriorates considerably under icy and snowy conditions, which poses a serious risk to vehicle braking and safety [2–5]. Salt-storage asphalt pavement refers to the incorporation of chloride salt components in the form of aggregates or mineral powders into asphalt concrete to achieve deicing and snow-melting effects through the release of chloride salt [6–8]. This technology holds great promise for winter road maintenance and has increasingly caught the attention of researchers.

Researchers from Switzerland and Germany conducted early studies on salt-storage asphalt pavement and developed Verglimit-260 (V260) as an additive to salt-storage asphalt pavement. This additive can reduce the freezing point to -20 °C [9]. Japanese researchers subsequently improved the moisture absorption and retarding effects of deicing materials based on this additive and developed Mafilon

(MFL). Li studied comparatively the road performance and precipitation patterns of V260 and MFL. The results showed the better road performance of V260 asphalt concrete than MFL asphalt concrete [9]. Giuliani et al. evaluated the effectiveness of a sodium chloride-based anti-icing filler in asphalt binders and analyzed the kinetics of sodium chloride release in water [10]. Hossain et al. assessed the effects of road salt on various pavement types for snow and ice control via an extensive field study and developed an optimized maintenance model [11]. Tan et al. researched the carrier and production process of sustained-release composite salt fillers, including hydrophobic agents and their composition ratios, and determined the components of sustained-release salt fillers with deicing and snow-melting functions [12]. Cao et al. prepared salt-storage asphalt mixture using styrene-butadiene-styrene (SBS) and highly elastic asphalt as binders and deicing salt as filler to experimentally study the influence of asphalt binder type and deicing salt content on performance of the mixture [13]. Wright studied the effects of the inclusion of ice retardant additives in pavement-surface course materials and concluded the feasibility of transferring deicing chemical formulations from within the asphalt mastic to the pavement surface [14]. Xu et al. assessed the water sensitivity of asphalt mixtures containing salt-storage additive and studied the migration

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doi:10.25103/jestr.171.10

characteristics and conducted natural leaching tests to determine the modes of chloride ions in these mixtures under various conditions [15]. Guo et al. investigated the effect of salt dissolution on the durability of asphalt and self-melting asphalt mixtures [16]. Wu et al. examined the influence of deicing salt types, concentrations, and exposure environments on the water sensitivity of asphalt mixtures [17]. Liu et al. investigated the salt-leaching patterns of salt-storage asphalt mixtures via conductivity tests and evaluated their anti-icing and anti-snow effects [18]. Zhong et al. established three-dimensional finite-element models of pavement structures under five different conditions to analyze the deicing mechanism of salt-storage high-elasticity pavement and discuss the sensitivity of ice-melting effect parameters, such as ice layer thickness, temperature, thickness, and modulus [19]. Zhu et al.'s research focused on the macroscopic snow and ice-melting mechanisms of salt-storage roads, development of a salt-storage material, and analysis of the road performance and antifreezing properties of salt-storage asphalt mixtures [20].

Researchers currently focus on the development of salt-storage materials, evaluation of their road performance, and analysis of deicing and snow-melting mechanisms in salt-storage asphalt mixtures. However, the effectiveness of salt-storage asphalt pavement in snow and ice melting relies on the sustained-release properties of salt-storage materials. Therefore, to investigate the sustained-release patterns of salt-storage aggregates and improve the sustained-release performance of salt-storage materials, magnesium oxychloride (MOC) cement was employed as a carrier in this study, and it was incorporated with chloride salt and sustained-release agents to prepare sustained-release salt-storage aggregates. Compressive strength and immersion conductivity were used to compare the different sustained-release salt-storage aggregates. Furthermore, immersion conductivity tests and simulated rain erosion tests were conducted to investigate the salt-release performance of salt-storage asphalt mixture. This study aimed to provide a reference for the production and engineering applications of salt-storage aggregates.

2. Materials and test methods

2.1 Raw materials

The MgO used in the experiment was produced in Yingkou, Liaoning Province and had a MgO content of $\geq 75\%$ and a CaO content of $\leq 2.0\%$. MgCl₂ was prepared in Weifang, Shandong Province, with a magnesium chloride content of $\geq 45\%$. NaCl was obtained from Ji'an, Jiangxi Province, with a NaCl content of $\geq 98.5\%$. CaCl₂, with a content of $\geq 94\%$, originated from Quzhou, Zhejiang Province.

Limestone was used for coarse and fine aggregates in the salt-storage asphalt mixture. The coarse aggregate had an apparent density of 2.726 g·cm⁻³ and a crushing value of 18.7%. The fine aggregate had an apparent density of 2.782 g·cm⁻³ and a sand equivalent of 75%. Limestone powder was used as filler. SBS-modified asphalt was selected as the asphalt binder, with a penetration of 65 (0.1 mm) at 25 °C, a softening point of 80 °C, and a ductility of 35.2 cm at 5 °C.

2.2 Preparation of salt-storage aggregates

The snow-melting agents used in this study comprised three chloride salts: NaCl, CaCl₂, and MgCl₂. Polyethylene glycol (PEG1000) and glycerol (C₃H₈O₃) were added as sustained-release agents to improve the sustained-release performance

of salt-storage aggregates and the effects of deicing and snow melting. MOC cement was prepared at the MgO to MgCl₂ molar ratio of 7:1 and was used as the carrier of salt-storage aggregates. Fig. 1 shows the salt-storage aggregate preparation process. MgCl₂ solutions were prepared first. In accordance with the proportion requirements, the MgO powder, snow-melting agent, sustained-release agents, and the modifier (Table 1), were mixed with MgCl₂ solutions in a mixer to produce a paste. The paste was poured into the molds to obtain blocks. The prepared blocks were placed in a standard curing box at a temperature of 20 °C \pm 2 °C for 28 days of curing. After curing, the blocks were removed for drying treatment, cooled to room temperature, and crushed with a roll crusher. After crushing, the aggregates with particle sizes of 2.36–4.75 mm, which were obtained through sieving using a standard sieve, were selected as salt-storage aggregates for subsequent tests.

Table 1. Composition of different types of salt-storage aggregates

Type	Ingredients
Type 0	MgO, MgCl ₂ , and Modifier
Type 1	MgO, MgCl ₂ , NaCl, and Modifier
Type 2	MgO, MgCl ₂ , CaCl ₂ , and Modifier
Type 3	MgO, MgCl ₂ , CaCl ₂ , PEG1000, and Modifier
Type 4	MgO, MgCl ₂ , CaCl ₂ , C ₃ H ₈ O ₃ , and Modifier
Type 5	MgO, MgCl ₂ , CaCl ₂ , PEG1000, C ₃ H ₈ O ₃ , and Modifier

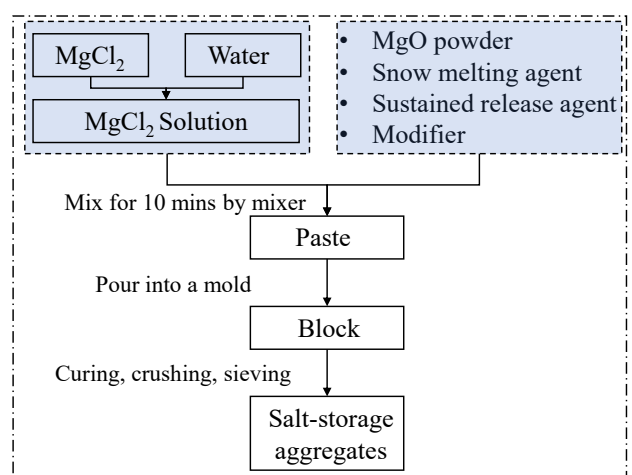


Fig. 1. Salt-storage aggregates preparation

2.3 Preparation of salt-storage asphalt mixture

The coarse and fine aggregates in the salt-storage asphalt mixture comprised limestone aggregates, and the mineral powder was limestone powder. The studied gradation was AC-13 (Table 2), and the asphalt-stone ratio was 5.5%. Type 4 salt-storage aggregates were selected for the preparation of salt-storage asphalt mixtures. The salt-storage aggregates replaced the aggregates with sizes below 4.75 mm in the gradation of asphalt mixtures based on the volume ratios of 0%, 10%, 15%, and 20%. The AC-13 asphalt mixture was mixed in accordance with the Technical Specification for the Construction of Asphalt Pavement of Highway (JTG F40-2017), and Marshall specimens were formed.

Table 2. Gradation of salt-storage asphalt mixtures

Gradation type	Sieve size (mm)									
	0.075	0.15	0.3	0.6	1.18	2.36	4.75	9.5	13.3	16
Upper limit	8	15	20	28	38	50	68	85	100	100
Lower limit	4	5	7	10	15	24	38	68	90	100
Studied gradation	5	8	11	16	24	34	58	81	98	100

2.4 Salt-storage aggregate performance test

2.4.1 Compressive strength test

The poured MOC cement block specimens were cured for 15 and 28 days under standard curing conditions. The cured MOC cement block specimens were polished and tested for compressive strength in accordance with the Cement Mortar Strength Test Method (ISO Method) GB/T 17671-2021. To evaluate the influence of soaking water on the compressive strength of salt-storage aggregates, the MOC cement specimens with 15-day standard curing were soaked in distilled water at $25\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for 8 days. After retrieval, the specimens were dried in a drying oven at $70\text{ }^{\circ}\text{C}$, polished, and tested for compressive strength. Three specimens in each group were tested in parallel, and the average values were considered the compressive strength.

2.4.2 Immersion conductivity test on aggregates

At 30:1 the mass ratio of distilled water to salt-storage aggregates, the salt-storage aggregates were soaked in water, and changes in the conductivity of water were continuously recorded using a DDS-11C conductivity meter. The recording frequency, which was recorded once a day for 13 days, was higher in the first 270 min.

2.5 Testing of sustained-release performance of salt-storage asphalt mixtures

2.5.1 Immersion conductivity test of the mixtures

The salt-storage asphalt-mixture specimens with different replacement rates were immersed in a beaker filled with 1200 ml distilled water, and changes in the conductivity of water were continuously recorded via the same method as before. Fig. 2 shows the conductivity test of salt-storage asphalt-mixture specimens.



Fig. 2. Immersion conductivity test of salt-storage asphalt-mixture specimens

2.5.2 Simulated rain erosion test

In the simulated rain erosion test, the time of a year was reduced to 12 h, and the water flow rates of the shower corresponding to different rainfall intensities were as follows: light rain, 1.2 L/h; moderate rain, 4.3 L/h; heavy rain, 9.2 L/h. With the assumption that the average annual rainfall in a certain area was 800 mm, the volume content was converted into volume content ratios of heavy, medium, and light rain (1:3.5:7.5) for the simulated rain erosion test. The loss of salt in salt-storage asphalt mixtures was measured through measurement of the quality differences in Marshall specimens before and after rain erosion. The simulated rainfall period was 5 years. The Marshall specimens under the same replacement rate were tested thrice in parallel. To

measure the salt-release potential of the salt-storage asphalt-mixture specimens after rain erosion, conductivity tests were performed to measure the changes in the conductivity of salt-storage asphalt-mixture Marshall specimens after the simulated rain erosion test. Fig. 3 shows the schematic of the simulated rain erosion device.

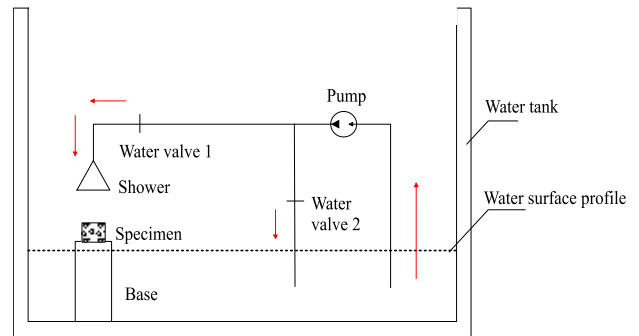


Fig. 3. Schematic of the simulated rain erosion device (The red arrow indicates the direction of water flow)

3. Results analysis and discussion

3.1 Performance of salt-storage aggregates

3.1.1 Compressive strength of salt-storage aggregates

Fig. 4 displays the compressive strength of different salt-storage aggregates. According to the results on compressive strength, the compressive strengths under standard curing for 15 days were ordered as follows: Type 1 > Type 0 > Type 3 > Type 2 > Type 4 > Type 5. The compressive strengths under standard curing conditions of 28 days were observed in the order of Type 1 > Type 0 > Type 5 > Type 4 > Type 2 > Type 3. Comparison of the compressive strengths at 15 and 28 days and calculation of the growth rate indicated that the strength of Type 1 salt-storage aggregate increased rapidly, and those of Types 4 and 5 increased slowly. Under standard curing for 15 days and soaking for 8 days, the compressive strengths were ordered as follows: Type 1 > Type 0 > Type 3 > Type 2 > Type 4 > Type 5. Compared with that under standard curing for 15 days, the compressive strength increased, and the order did not change under standard curing for 15 days and soaking for 8 days. Comparison and analysis of the composition and compressive strengths of Types 2, 3, 4, and 5 salt-storage aggregates at 28 days revealed that the sustained-release agent of polyethylene glycol had a certain adverse effect on compressive strength, and glycerol had an enhanced effect.

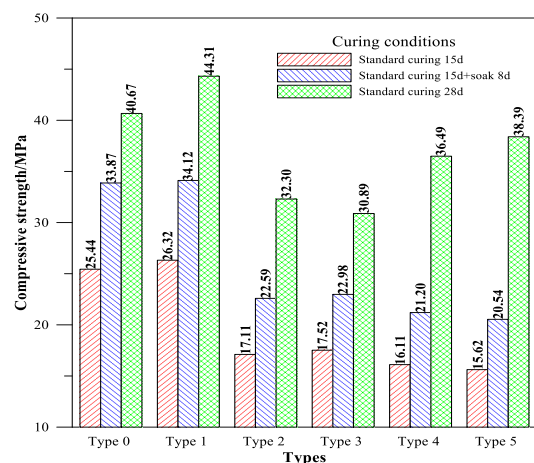


Fig. 4. Compressive strength of different salt-storage aggregates

3.1.2 Conductivity of salt-storage aggregates immersed in water

The conductivity of salt-storage aggregates immersed in water for 270 min was recorded as a short-term immersion conductivity. Fig. 5 shows the short-term immersion conductivities of different salt-storage aggregates. The conductivity of salt-storage aggregates immersed in water for 13 days was recorded as a long-term immersion conductivity. Fig. 6 displays the long-term immersion conductivities of different salt-storage aggregates.

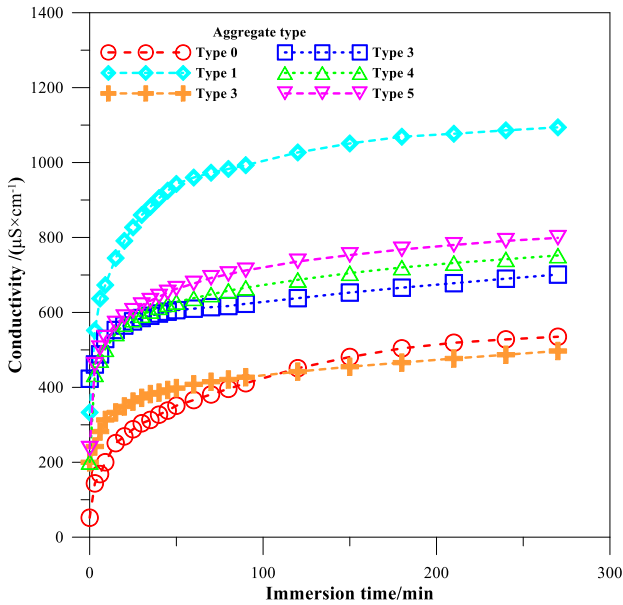


Fig. 5. Short-term immersion conductivity of different salt-storage aggregates

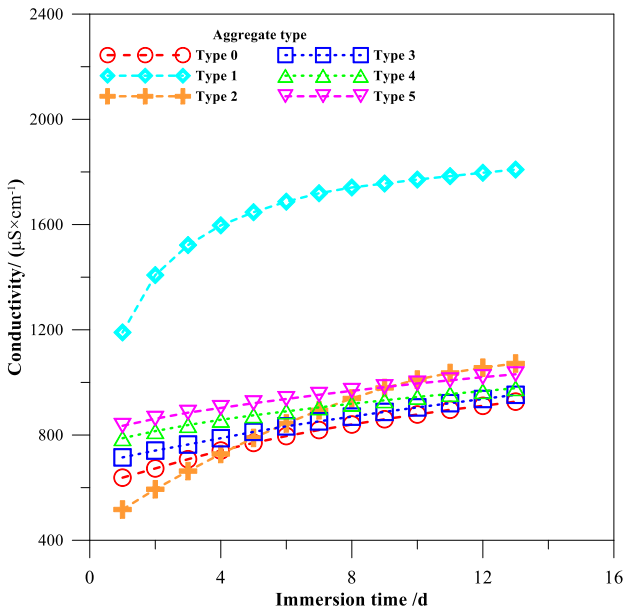


Fig. 6. long-term immersion conductivity of different salt-storage aggregates

As shown in Fig. 5, under short-term immersion, the immersion conductivity of different types of salt-storage aggregates grew rapidly in the early stage and gradually slowed down in the later stage due to the fast dissolution rate of salt in each salt-storage aggregate in the early stage. With the continuously prolonged immersion, the salt content of salt-storage aggregates continued to decrease, and the dissolution gradually penetrated the core of aggregates. The

conductivity of Type 1 salt-storage aggregates containing NaCl increased the fastest and the largest. On the contrary, the conductivity of Types 0 and 2 increased the slowest and were the smallest, and those of types 3, 4, and 5 salt-storage aggregates were in the middle. The analysis showed the slower dissolution rate of CaCl₂ than MgCl₂ and NaCl, which resulted in low immersion conductivity and slow growth of the corresponding salt-storage aggregates.

The polar groups in alcohol molecules have a strong adsorption effect and form a dense and complete adsorption film on the surface of salt molecules to protect the salt in salt-storage aggregates, which plays a sustained-release role. Fig. 6 shows that the conductivity of Types 1 and 2 salt-storage aggregates without sustained-release agents increased at fast rates, with values increasing by 52.02% and 107.35%, respectively, compared with those at 13 days and the first day. By contrast, the conductivity of Types 3, 4, and 5 salt-storage aggregates with sustained-release agent increased relatively slowly by 33.28%, 24.11%, and 23.47%, respectively. This finding indicated that Types 3, 4, and 5 salt-storage aggregates presented good sustained-release performance and relatively stable release growth. Compared with the test results on Types 3 and 4 salt-storage aggregates, glycerol showed a better sustained-release effect than polyethylene glycol for the salt-storage aggregate using CaCl₂ as snow-melting agent.

According to the results of compressive strength and immersion conductivity test under different curing conditions, Type 4 salt-storage aggregate was selected to prepare salt-storage asphalt mixture, as will be discussed in the following sections.

3.2 Sustained-release performance of salt-storage asphalt mixture

3.2.1 Immersion conductivity of mixtures

Figs. 7 and 8 show the short- and long-term immersion conductivities of salt-storage asphalt mixtures prepared at different replacement rates using Type 4 salt-storage aggregate, respectively.

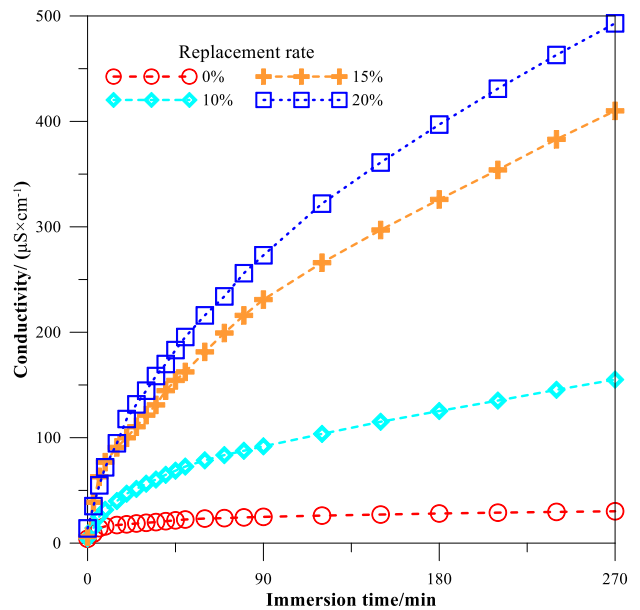


Fig. 7. Short-term immersion conductivity of salt-storage asphalt mixture with different replacement rates

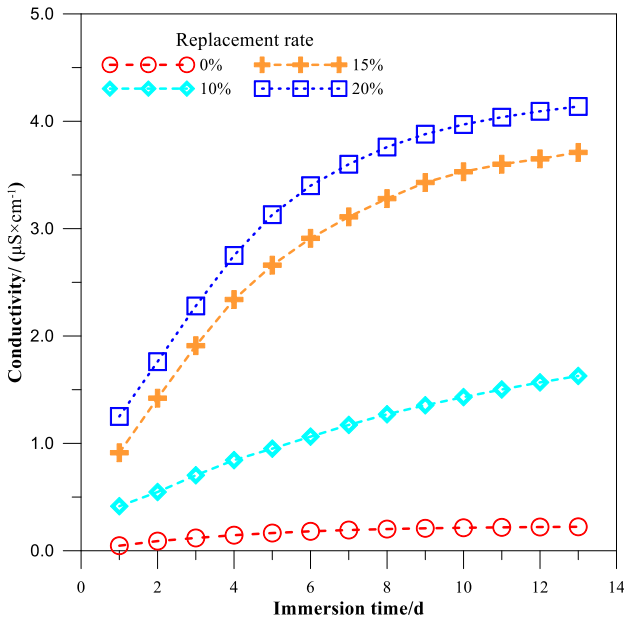


Fig. 8. Long-term immersion conductivity of salt-storage asphalt mixture with different replacement rates

Figs. 7 and 8 reveal that in the conductivity test, given the salt-storage aggregate content of the Marshall specimens, the salt in salt-storage aggregates was released, and the conductivity increased rapidly with prolonged immersion under short-term water immersion conditions. Under long-term immersion, the conductivity changes in different replacement rates presented two stages: the early stage that increased rapidly and the late stage that tended to be flat. At 10% replacement rate, the conductivity and conductivity growth rate were considerably lower than the replacement rates of 15% and 20%, respectively. At the end of the 13th day of immersion, the conductivity increased by 127.91% and 154.23% with the increase in replacement rate from 10% to 15% and from 10% to 20%, respectively.

3.2.2 Salt-dissolved regularity

The conductivity of solutions has a certain relationship with the content of contained inorganic salts. At relatively low concentrations, conductivity increases with the increase in concentration. Therefore, conductivity is often used to predict the total concentration of ions or salt content in water. Given the main salt-dissolved component of Type 4 salt-storage aggregates, that was, CaCl_2 in this study, CaCl_2 solutions with different mass concentrations were prepared, and their conductivity was measured. The standard curve of concentration and conductivity of CaCl_2 solution was drawn through data fitting (Fig. 9). According to the results of long-term immersion conductivity test on Marshall specimens at different replacement rates, the change curves of solution concentration were obtained through calculation of the long-term immersion curve of salt-storage asphalt mixture (Fig. 10).

A logarithmic model was used to fit the solution concentrations in the different soaking times displayed in Fig. 10, and the fitting results are shown in Table 3.

Table 3. Salt dissolution equation of salt-storage asphalt mixtures

Replacement rate	Fitting equation	Fitting coefficient
10%	$Y=0.00702\ln(x+6.14982)-0.01203$	$R^2 = 0.99927$
15%	$Y=0.00750\ln(x+0.45909)+0.00143$	$R^2 = 0.99111$
20%	$Y=0.00751\ln(x+0.30472)+0.00404$	$R^2 = 0.98485$

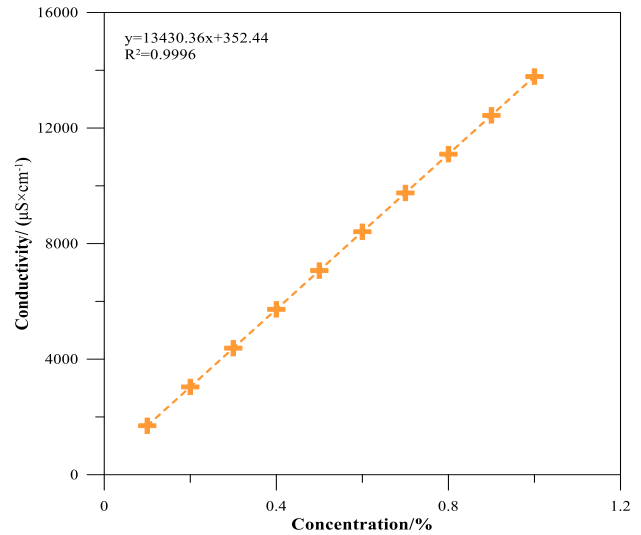


Fig. 9. Standard curve of concentration and conductivity of CaCl_2 solution

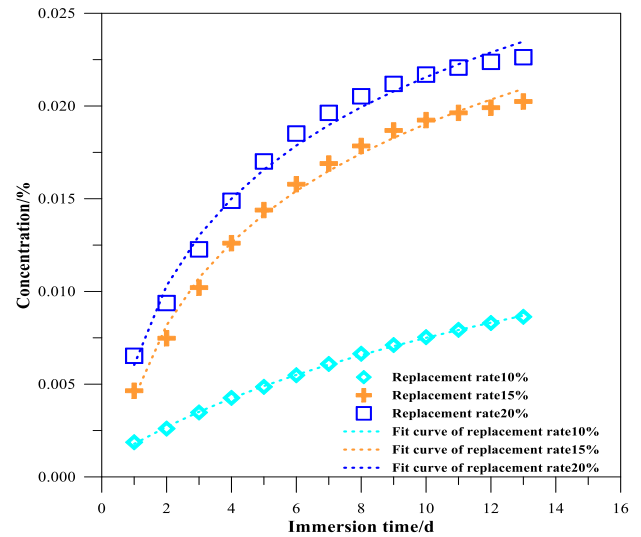


Fig. 10. Solution concentration under different immersion duration

The findings in Table 3 revealed that under the three replacement rates, the salt-dissolved regularity of the salt-storage asphalt mixture conformed to the logarithmic model:

$$Y = a\ln(x + b) + c \tag{1}$$

where Y refers to the solution concentration, x denotes the immersion time, and a , b , and c are regression parameters. The fitting coefficients were 0.99927, 0.99111, and 0.98485, respectively, with a good fitting effect. According to the relationship model of concentration and immersion time, the amount of salt dissolution equation of salt-storage asphalt mixture can be estimated and can provide a reference for the design and engineering applications of salt-storage asphalt mixtures.

3.2.3 Mass loss after rain erosion

After the simulated rain erosion test, the percentage of mass loss of salt-storage asphalt mixtures was measured after rain erosion (Fig. 11).

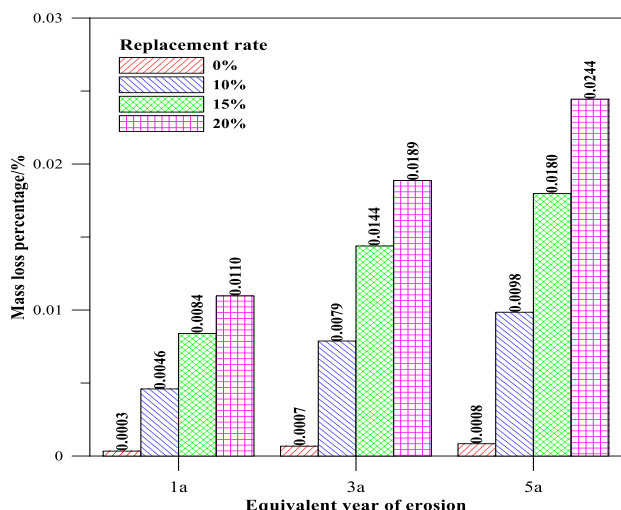


Fig. 11. Mass loss percentage of salt-storage asphalt mixtures after rain erosion

Fig. 11 shows that the percentage of mass loss of salt-storage asphalt-mixture specimens under different replacement rates all showed increased trends with the increase in equivalent years of erosion. The higher the replacement rate of salt-storage aggregates, the greater the percentage of mass loss. Under continuous erosion of water, the loss of quality became more evident.

3.2.4 Variation in conductivity after rain erosion

To evaluate the salt-release potential of salt-storage asphalt mixtures after rain erosion, conductivity tests were conducted to measure the conductivity of salt-storage asphalt-mixture specimens under different replacement rates after the simulated rain erosion test. The test results are shown in Fig. 12.

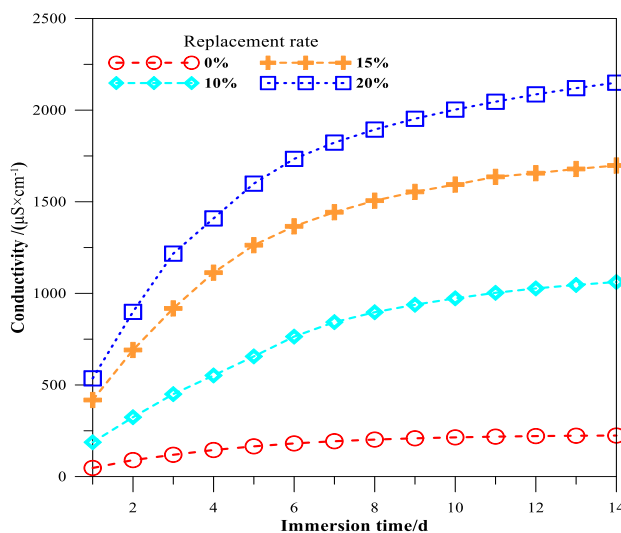


Fig. 12. Variation of immersion conductivity after simulated rain erosion test

Fig. 12 illustrates that under different replacement rates after the simulated rain erosion test, the immersion conductivity of salt-storage asphalt-mixture specimens showed an increasing trend with the increase in time. However, compared with that before rain erosion, the conductivity decreased considerably. The test results revealed that after 5 years of equivalent rain erosion, the salt-storage asphalt mixtures can still release salt, but the release rates were slow.

4. Conclusions

To investigate the salt-release performance of salt-storage aggregates, sustained-release salt-storage aggregates were prepared using MOC cement as a carrier through the addition of chloride salts and sustained-release agents. Immersion conductivity tests and simulated rain erosion tests were conducted to study the salt-release performance of salt-storage asphalt mixtures. The following conclusions were drawn:

- (1) Under the standard curing condition of 28 days, the sustained-release agent polyethylene glycol had a certain adverse effect on the compressive strength of salt-storage aggregates, whereas glycerol exhibited an enhancing effect.
- (2) The salt-storage aggregates with sustained-release agent showed a better salt-release effect, and the conductivity growth was relatively stable. For the salt-storage aggregates with CaCl₂ as the snow-melting agent, glycerol addition attained a better sustained-release effect than that of polyethylene glycol.
- (3) During short-term immersion, the immersion conductivity of the salt-storage asphalt-mixture specimens increased rapidly with the continuous prolongation of the immersion time. Under long-term immersion, the conductivity changes at different replacement rates presented two stages: the early stage increased rapidly and the late stage tended to be flat. The salt dissolution regularity of salt-storage asphalt mixtures with different replacement rates conformed to the logarithmic model, and good fitting effects were obtained.
- (4) After 5 years of equivalent rain erosion, the salt-storage asphalt mixtures can still release salt, but the release rates were slow.

This study provides a feasible method for the preparation of salt-storage aggregates and describes their sustained-release pattern. However, the capability of salt-storage asphalt pavement for ice and snow melting can be affected by the production and construction processes in engineering application. The composition of salt-storage aggregates must be further optimized for production and construction.

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