Center for Environmentally Sustainable Transportation

A New Sustainable Additive for Anti-Icing Asphalt



Control HMA Sample after Fog Freezing

Anti-icing HMA Sample

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EXECUTIVE SUMMARY

This project was designed to develop a new asphalt additive for *in-situ* anti-icing, without compromising the durability of the asphalt pavement. We first conducted a review and synthesis of the state-of-the-art literature on anti-icing asphalt pavement with salt-storage additives, which mainly focused on the type of salt-storage additives and their effects on the anti-icing performances and mechanical properties of asphalt pavement. Anti-icing asphalt pavement through salt-storage additives is an effective strategy that not only prevents ice formation or weakens the bond of snow/ice to pavement but also reduces the use of salt chemicals for winter road maintenance. However, this strategy still faces a host of challenges in real practice, such as the longevity of anti-icing ability, low-temperature effectiveness, and potentially adverse impacts to the mechanical properties of asphalt pavement.

Thus, we developed an anti-icing asphalt pavement for snow and ice control by incorporating innovative laboratory-prepared salt-storage additives with a low salt-release rate. These additives were prepared through a surface treatment approach, in which zeolite containing CaCl₂ was coated by a porous epoxy layer. The additives had three different sizes: sieve #4 - #8, sieve #8 - #16 and sieve #16 - #30. The CaCl₂ content in additives #8, #16, and #30 was 20.1%, 20.5%, and 21.1% respectively, determined based on the mass difference between the additives with and without CaCl₂. The dosage of the additives in asphalt mixture was 100% by the mass of asphalt binder. Laboratory evaluation of the asphalt mixture with the additives consisted of fog-freezing friction tests at -3.9 °C (25°F) and -9.4 °C (15°F) for anti-icing capacity, anti-icing longevity tests at 15 °C (59°F) and 0 °C (32°F) for estimation of effective anti-icing period, Hamburg wheel-track tests in water at 50 °C (122°F) for assessing the moisture susceptibility

and rutting resistance of asphalt mixture, and indirect tensile tests for mid-temperature (25 °C, 77°F) and low-temperature (-10 °C, 14°F) cracking resistances of asphalt mixture.

The results indicated the laboratory-prepared additives showed remarkable benefits to the anti-icing performances of asphalt pavement. The anti-icing capacity of asphalt mixture significantly improved through the addition of the additives at both testing temperatures, although the friction coefficient at 60 min after moisture spray decreased from 0.75 at -3.9 °C to 0.55 at -9.4 °C. Reducing the size of the additive further improved the anti-icing capability. Under simulated conditions, the estimated effective anti-icing period of asphalt pavement with additives #8, #16, and #30 were 5.8 years, 9.9 years and 15.3 years respectively. The testing results demonstrated a strong potential for using the laboratory-prepared salt-storage additives in asphalt pavement for snow and ice control.

The results revealed these novel additives had positive effects on the rutting resistance, mid-temperature and low-temperature cracking resistance of asphalt mixture and negligibly increased the moisture susceptibility of asphalt pavement. The addition of the additives slightly reduced the moisture damage resistance, but almost all the mixtures passed the WSDOT specification (except sample #8-3 that had 14143 passes at SIP) as well as the Wisconsin and Iowa specifications. The rutting resistance, mid-temperature cracking resistance, and low-temperature cracking resistance of asphalt mixture improved by the addition of the additives to various extents. The rutting resistance was higher but the mid-temperature cracking resistance was lower than the acceptable limit based on the WSDOT specification. Future investigation should focus on further improvements in the engineering performances and mechanistic understanding of this type of anti-icing pavement.

CHAPTER 1.0 INTRODUCTION

1.1 Background

Asphalt pavement is widely used in engineering applications such as highways, roads, streets, and airfields, because it features high strength and outstanding durability. However, in cold regions, the accumulation of snow and the formation of ice on asphalt pavement during winter weather causes slippery pavement, which raises the risk of vehicle accidents and property damage (Lépy et al. 2016). Many traffic accidents occur due to the reduction of tire-pavement friction resistance in the presence of snow and ice. Research has indicated the rate of accidents increased by 84% and the rate of injuries increased by 75% because of snow and ice on roads (Qiu and Nixon 2008). Pan et al. reported more than 15% of traffic accidents occurred every year due to wet, icy, snowy and slippery road conditions (Pan et al. 2015). The statistical data reported by Federal Highway Administration (FHWA) pointed out, on average over ten years (from 2005 to 2014), the weather-related crashes with due to snow/sleet road were 17% of total crashes; the weather-related crashes due to icy pavement were 13%; and the weather-related crashes resulting from snow/slushy pavement were 14% (FHWA 2015). Moreover, Call et al. indicated 25% of total crashes occurred under conditions of 'snow/blowing snow' (Call et al. 2018). The high rate of traffic accidents caused by inclement winter conditions calls for the developments of better and more timely methods for snow and ice control on pavements.

To keep asphalt pavement clear of ice and snow during winter weather, many methods have been developed and employed, including mechanical removal (such as snowplowing), the use of abrasives (such as sanding), and the application of salt chemicals before and after snow

precipitation (Shi et al. 2018). Such snow and ice control operations have direct benefits to the public including, safer driving conditions, fewer traffic accidents, better road mobility, shorter travel time and lower travel cost. However, there are concerns over their adverse impacts. For instance, abrasives pose risks to the natural environment due to the presence of suspended particles in air, which can present more risks than road salts in terms of human health (Fay and Shi 2012). Mechanical removal is the main method for snow and ice removal, but the consumption of vehicle fuel and the efforts to remove snow and ice can be substantial in light of the strong bond between ice/snow and asphalt pavement.

To reduce the use of abrasives and to improve the efficiency of mechanical removal, large amounts of deicing and anti-icing chemicals (a.k.a., deicers) are applied every year (Ramakrishna and Viraraghavan 2005), of which benefits have been widely witnessed. Nonetheless, there are growing concerns over the short-term and long-term risks associated with the use of deicers, including the potential adverse impacts of salt chemicals on the natural environment, motor vehicles and transportation infrastructure(Fay and Shi 2012; Shi et al. 2009).

1.2 Problem Statement

Although deicing and anti-icing chemicals effectively facilitate snow and ice control on pavements, large amounts of the application of deicer products, including chloride-based chemicals, acetate-based chemicals, formate-based chemicals, urea and glycols, have raised serious concerns over their potential risks to the natural environment, motor vehicles and transportation infrastructure.

Based on the results of a survey of road maintenance agencies, chloride-based chemicals were the most frequently used in winter road maintenance, less than 25% of the responded

agencies selected acetate-based and/or formate-based chemicals, and a small number of agencies used other chemical deicers (Fay et al. 2008). While readily available and relatively low in their initial costs, chloride-based chemicals have major disadvantages with respect to corrosiveness to metals as well as detrimental effects on cement concrete, water quality and soil (Fay and Shi 2012; Shi et al. 2009, 2011, 2013).

On the airfield pavements, the use of traditional chemicals, urea and glycols for example, is limited, due to their very high oxygen demand posing a considerable risk to water bodies receiving the deicer-laden runoff. Instead, acetate-based and formate-based chemicals have been commonly applied over the last two or three decades (Edwards et al. 1999; Shi 2008). However, laboratory studies revealed that acetate-based and formate-based chemicals might result in degradation and disintegration of asphalt pavement, through softening effects on asphalt and stripping effects on asphalt pavement with loose aggregates. These deterioration mechanisms have caused significant moisture damages of asphalt pavement in Europe, Canada and United State (Edwards et al. 1999; Pan et al. 2008).

Several types of anti-icing asphalt pavements such as the asphalt pavements modified by salt additives (Peng et al. 2015a) and electrically conductive asphalt pavements (Pan et al. 2015) have been developed for decades. These smart pavements can not only prevent ice formation or weaken the bond strength between snow/ice and pavement but also respond to freezing conditions on pavement in a more timely fashion. The use of anti-icing pavements can also reduce or avoid the use of deicing and anti-icing chemicals. Nevertheless, the anti-icing effectiveness and durability of these anti-icing pavements are yet to be determined for widespread application in practice.

1.3 Scope of Work

This study consisted of three tasks. In Task 1, the emphasis was on the review of literature relevant to research and technological development of salt additives incorporated into anti-icing asphalt pavement (i.e., slow release of freezing point depressants). In Task 2, the focus was on the development of innovative laboratory-prepared salt-storage additives to blend into asphalt pavement for snow and ice control. In Task 3, we conducted a systematic laboratory evaluation of the anti-icing performances and common engineering properties of the asphalt pavement with the laboratory-prepared additives.

1.4 Outline of Report

This report is divided into four chapters. Chapter 1 provides an introduction to background and the problem of interest and then outlines the scope of work. Chapter 2 provides a literature review based on both national and international sources. Chapter 3 describes the sample fabrication, laboratory test methods, results and data analyses. Chapter 4 summarizes the work of this report and gives conclusions.

CHAPTER 2.0 LITERATURE REVIEW

2.1 Introduction

Black ice, a thin and slippery sheet of ice, tightly bonds with both pavement and snow, resulting in difficult snow removal during winter maintenance operations. Black ice is the most difficult type of ice to detect and remove, and is responsible for many vehicle accidents. The formation of black ice is usually caused by moisture remaining on the surface of pavement which subsequently suffers freezing or sub-freezing temperatures (Liu et al. 2017). With snow precipitation, moisture is produced by the melted snow on pavement with a temperature above 0°C. When the pavement temperature drops to the freezing point, the moisture becomes black ice when a tight bond forms with the pavement and accumulated snow. Black ice can form under other conditions as well, such as freezing rain and sleet. Snow precipitation and the subsequent ice on roads greatly increases potential traffic hazards.

A combination of strategies involving mechanical removal, chemical applications and other technologies are often adopted for snow and ice control operations, based on different weather conditions, locations and resources (Shi 2010). Any single operation may not achieve the goal of winter road maintenance effectively and efficiently, especially for storm events. Mechanical removal is still the main means to clear the pavement during heavy snow events in cities, highways and airports. In the case of black ice, the strong bond between black ice, pavement and snow makes it very difficult to remove the snow and ice in a single mechanical removal, increasing the costs of fuel and labor and influencing the service life of equipment. Moreover, the aggregates on the surface of asphalt pavement are often damaged due to improper operation of mechanical removal (Ma et al. 2018). As such, it is desirable to prevent the

formation of black ice by releasing anti-icing chemical directly from the asphalt pavement. If a thicker layer of compacted snow or ice has tightly bonded to the pavement, then deicing becomes necessary. Deicing aims to break the bond between snow/ice and pavement. Both anti-icing and deicing facilitate the removal of snow and ice by mechanical means and maintain high friction resistance against the pavement surface by vehicle tires.

The function of salt chemicals on pavement is to prevent the formation of black ice or the ice-pavement bond or to weaken or destroy the ice-pavement bond. In other words, it is not intended to melt the snow and ice on the pavement. The mechanism involved is that salts firstly dissolve in the moisture existing in snow or on the pavement to produce the salt solution with a lower freezing point (known as freezing point depression) than pure water, and then the salt solution turns more snow and ice to liquid, causing more salts to dissolve in water. This cycle continues until one of the following conditions occurs: 1. all salts dissolve in water and the concentration of the initial salt solution reaches a level that fails to melt any additional snow or ice; or 2. there is no longer a source of moisture. Therefore, the ability of salt chemicals to prevent or break bonds and to melt snow and ice depends greatly on the application rate (reported in tons/lane-mile for solids, or gallons/lane-mile for liquids) if moisture is adequate. It is unrealistic to use salt chemicals to melt all the snow and ice accumulated on the pavement during a winter storm event, to do so would require the use of considerably higher amounts of salt chemicals than normal, which would be cost-prohibitive and environmentally disastrous.

Even with the best practices of anti-icing, deicing and mechanical removal, the snow and ice control operations on roads and highways still use substantial amounts of chemicals (mainly chloride salts) every year (Ramakrishna and Viraraghavan 2005; Shi 2010). Anti-icing is a

proactive strategy that uses various technologies against the formation of ice that tightly bonds with the surface layer of asphalt pavement, such as spreading chemical deicers before the snow event. Relative to deicing, anti-icing helps to reduce the usage of chemicals without reducing the level of service on the pavement. In addition, other anti-icing technologies include physical modification of pavement surface, high-friction anti-icing polymer overlays, anti-icing asphalt pavement with salt-storage additive, internal heating (e.g., geothermal heating and electrical resistive heating), external heating (e.g., energies from wind, solar, microwave and infrared), and other advanced technologies (e.g., ice-phobic pavements and thermochromic asphalt pavements) (Shi et al. 2018). All of these have been investigated as alternatives to reduce the use of salt chemicals for snow and ice removal or even eliminate their application (in the case of black ice). More importantly, these anti-icing technologies may provide more timely response to potentially slippery condition on the pavement surface, before any maintenance vehicle (snowplow or salt spreader) is deployed to the site.

Anti-icing asphalt pavement with salt-storage additive is the technology that blends a salt-storage additive into an asphalt mixture to achieve anti-icing by slowly releasing salt chemicals from the interior to the surface of asphalt pavement under traffic loading. Although some field-performance evaluations have shown the beneficial effect of this novel asphalt pavement on snow and ice control, the anti-icing effectiveness and durability are still a challenge in practice. The limitations of the anti-icing asphalt pavement with salt-storage additives include:

- 1. Difficulty controlling the release rate of encapsulated salts to extend the anti-icing service life (usually one to three years).
- 2. Difficulty reducing the risk of degradation to mechanical performance, especially moisture damage resistance, in the presence of salts.
- 3. Difficulty making anti-icing effective at low temperatures (below 20 °F) (Shi et al. 2018).

2.2 Anti-icing Asphalt Pavement with Salt-Storage Additives

This section discusses anti-icing asphalt pavement containing salt-storage additives found in previous studies, including the classification and characterization of salt-storage additives, their impacts on the anti-icing and mechanical performance as well as anti-icing durability.

2.2.1 Salt-Storage Additives

Various salt-storage additives used to promote anti-icing in asphalt pavement have been studied for decades. Calcium chloride (CaCl₂)-based and sodium chloride (NaCl)-based additives are the two main types of salt-storage additives. Other additives are less investigated, including sodium formate (NaFm) and sodium silicate (Wright et al. 2016), magnesium-aluminum/chloride layered double hydroxide (Mg-Al/Cl⁻ LDH) (Peng et al. 2015b), magnesium-aluminum/acetate layered double hydroxide (Mg₂/Al-Ac⁻ LDH) (Peng et al. 2015a) and a laboratory-prepared additive produced by mixing cementitious materials, water resistant modifier and NaCl powders together (Wang et al. 2017). These less-investigated additives are not commercial products and only studied in a laboratory.

Some additives have been commercialized for many years, with laboratory and field test reports available, including Verglimit, Mafilon, IceBane, IceGuard and WinterPave (Shi et al. 2018). Verglimit, featuring 0.1–5 mm flake particles with 95% CaCl₂ and 5% sodium hydroxide (NaOH) packaged in linseed oil or polyvinyl acetate, has been investigated since the 1980s.

Verglimit is fabricated by grinding a mixture of salt chemicals and synthetic thermoplastic materials (Zheng et al. 2015). Generally, 5-6 wt% Verglimit is blended into asphalt mixture paving used on bridge decks, steep grades, sharp curves, heavily shaded roads, and roads adjacent to water. Mafilon containing 56 wt% NaCl is another commercial additive available (Li and Wang 2012). Mafilon particles are less than 0.6 mm and contain other components including SiO₂, MgO and CaO. IceBane mainly consists of NaCl and CaCl₂ modified by a special hydrophobe, and 99% of the particles are less than 0.6 mm, (Liu et al. 2014a, 2015a). WinterPave contains CaCl₂ (20~30 wt%) and SiO₂ (Sargand and Williams 2016). The main component of Iceguard with the particle size from 0.1 mm to 4 mm is 58.8 wt% CaCl₂, and its cost is 60% of Verglimit (Zheng et al. 2017). Other commercialized additives are NaCl-based but unnamed. The Harbin Institute of Technology produced an unnamed additive consisting of 34 wt% NaCl, Carrier C, polysiloxane A and ethyl alcohol, and the particle size is less than 0.6 mm (Tan et al. 2014). Giuliani et al. studied an unnamed additive containing 73 wt% NaCl, CaMg(CO₃)₂, MgCO₃ and SiO₂ (Giuliani et al. 2012). One unnamed additive is composed of 60 wt% NaCl, CaCl₂, SiO₂, CaO, Fe₂O₃, and Al₂O₃, and 90% of the particles are less than 0.075 mm (Ma et al. 2018). This additive is fabricated by immersing porous pyrolith aggregates in a saline solution. Other similar NaCl-based additives have been studied as well (Xu et al. 2015; Zheng et al. 2016). Table 1 shows the summary of commercialized and laboratory-prepared additives.

Salt Type	Anti-icing Additive	Active Ingredie nt	Active Ingredient Concentratio n by Mass	Particl e Size	Other Ingredients	Commercialize d / Laboratory- prepared
	WinterPav e	CaCl ₂	20~30%	N/A	SiO ₂	Commercialize d
	Verglimit	CaCl ₂	94.6%	0.1-5 mm	N/A	Commercialize d
	Iceguard	CaCl ₂	58.8%	0.1-4 mm	N/A	Commercialize d
	Mafilon	NaCl	56%	Less than 0.6 mm	SiO ₂ , MgO, CaO, and others	Commercialize d
Chlorid e based	IceBane	NaCl, and CaCl ₂	56%	99% < 0.6 mm	CaO, Al ₂ O ₃ , SiO ₂ , Na ₂ O, K ₂ O, Fe ₂ O ₃	Commercialize d
	Harbin Institute of Technolog y, ZFSY	NaCl	34%	< 0.6 mm	Carrier C, polysiloxan e A, ethyl alcohol	Commercialize d
	N/A	NaCl	73%	Less than 4.75 mm	CaMg(CO ₃) 2, MgCO ₃ and SiO ₂	Commercialize d
	N/A	NaCl	60%	90% < 0.075 mm	CaCl ₂ , SiO ₂ , CaO, Fe ₂ O ₃ , and Al ₂ O ₃	Commercialize d
	N/A	NaCl	55%	Less than 0.6 mm	CaCl ₂ , MgCl ₂ and SiO ₂	Commercialize d
	N/A	NaCl	55%	Less than 0.3 mm	SiO ₂ , CaCl ₂ , CaCO ₃	Commercialize d
	Salt- Storage Aggregate	NaCl	N/A	0.075 mm - 4.75 mm	MgCl ₂ , MgO, silicone powder, glass	Laboratory- prepared

Table 1 Summary of commercialized and laboratory-prepared salt-storage additives	Table 1 Summar	y of commercialized	and laboratory-prepa	ared salt-storage additives
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Salt Type	Anti-icing Additive	Active Ingredie nt	Active Ingredient Concentratio n by Mass	Particl e Size	Other Ingredients	Commercialize d / Laboratory- prepared
					powder and maifanite powder	
	NaFm	NaFm	100%	Ave 0.055 mm	N/A	Commercialize d
Non- chloride	Sodium Silicate	Sodium Silicate	100%	Ave 0.075 mm	N/A	Commercialize d
based	Mg-Al/Cl ⁻ LDH	Mg- Al/Cl ⁻ LDH	100%	98.5% < 0.6 mm	N/A	Laboratory- prepared
	Mg ₂ /Al- Ac ⁻ LDH	Mg ₂ /Al- Ac ⁻ LDH	100%	N/A	N/A	Laboratory- prepared

2.2.2 Anti-Icing Characterization of Salt-Storage Additives

Direct and indirect testing approaches adopted to measure the anti-icing and deicing capacities of salt-storage additives included ice melting test, penetration test, differential scanning calorimetry (DSC) test and freezing point test. Ice melting test, penetration test and DSC test are usually used to evaluate the deicing performance of salt chemicals (Fay and Shi 2011).

2.2.2.1 Freezing point test

Nonstandard freezing point test measures the freezing time and the freezing temperature of a salt solution. The freezing time and the freezing temperature of the solution indirectly reflect the potential of the capacity that salt-storage additives prevent water from freezing.

The testing procedures of freezing point tests reported in previous studies are similar. The salt solution, prepared by admixing salt-storage additives with de-ionized (DI) water, is placed

into a cold chamber with a certain cooling rate, followed by monitoring the freezing process, the freezing time and the freezing temperature. Zheng et al. reported Iceguard and Verglimit had lower freezing temperatures (-18 °C and -19 °C respectively) than that of Mafilon (-7 °C) as the solution concentration was 5 wt%, and this phenomenon might be explained by the lower freezing point of CaCl₂ than that of NaCl (Zheng et al. 2017).

2.2.3 Effects on Anti-Icing Performances of Asphalt Pavement

There is no standard testing method to evaluate the anti-icing performance of asphalt pavement with salt-storage additives. The role of salt-storage additives is to prevent the formation of the strong bond of snow/ice to asphalt pavement or to directly impede ice formation. Thus, to evaluate the anti-icing performance, measurements related to the bond of snow/ice to asphalt pavement or the friction condition between pavement surface and vehicle tire have been developed, including but not limited to frost formation simulation test (Giuliani et al. 2012), water freezing point test (Giuliani et al. 2012), skid resistance test (Giuliani et al. 2012; Xu et al. 2015), ice-mixture bond test (Chen et al. 2018; Luo and Yang 2015; Ma et al. 2016, 2018; Zheng et al. 2017), and snow-traffic-plowing-friction test (Akin et al. 2018a; b). Deicingrelated testing methods (such as snow/ice melting test) are not involved in the research.

2.2.3.1 Frost formation simulation

Giuliani et al. simulated the process of the frost formation on the surface of asphalt mastics film with 50 wt% salt additives to evaluate the anti-icing characteristic as shown in Figure 1 (Giuliani et al. 2012). The authors continuously monitored the frosting process with a digital video camera as the samples were cooled from room temperature to -15 °C with a cooling rate of 15 °C/min. The result showed that after the 10-min cooling period, frost totally covered

the surface of the control sample whereas frost only formed at the edge of the sample with NaClbased additive. Moreover, the time it took for frost to cover the whole surface of the sample with the additive was $30 \sim 60$ min. They concluded the released NaCl from asphalt mastic greatly delayed the formation and evolution of the frost.

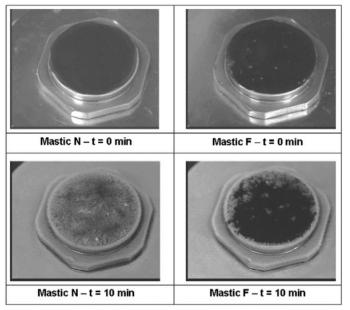


Figure 1 Frost formation test with relative humidity of 20% (Giuliani et al. 2012)

2.2.3.2 Water freezing point test

Similar to a freezing point test, a water freezing point test is used to measure the freezing time and the freezing temperature of a thin water film on the surface of an asphalt mixture by continuously monitoring the temperature of the interface between water and sample surface and the corresponding time with a certain cooling rate (Giuliani et al. 2012). The result confirmed that the freezing point of the water film on the sample with 5.7 % NaCl-based additives was slightly lower than that of the control sample (-2 °C vs 0 °C), as shown in Figure 2. The negative peaks could be explained by supercooling processes, and the freezing point depression after the peaks was attributed to the presence of the NaCl ions dissolved in the water film.

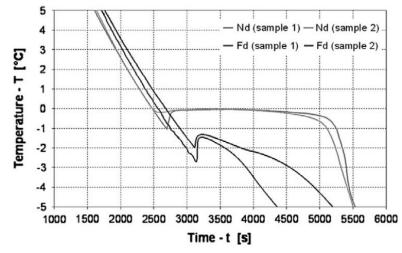


Figure 2 Freezing point determinations (Giuliani et al. 2012)

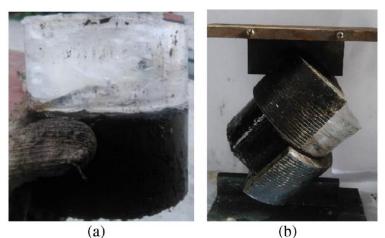
2.2.3.3 Skid resistance test

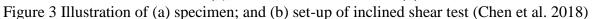
Skid resistance is an index presenting the friction condition of pavement surface, which can be used to evaluate the anti-icing performance of asphalt pavement. A British Pendulum Tester is usually used to measure skid resistance by following the procedure from UNI EN 13036-4:2005 (Giuliani et al. 2012). The output is expressed in terms of British Pendulum Number (BPN), and a greater BPN suggests a better friction condition.

Giuliani et al. conducted the skid resistance test using slab samples, which were previously conditioned at -22 °C for 12 hours then spread with water at 5.0 ± 0.5 °C and finally transferred in a room at 5.0 ± 0.5 °C (Giuliani et al. 2012). The authors concluded the presence of 5.7 wt% NaCl-based additives slightly increased the BPN of dense-graded samples and significantly increased the BPN of open-graded samples after 10 minutes spreading water, which was interpreted as more NaCl located at the interface between sample surface and water due to more air voids on the surface of open-graded samples. This indicated the presence of the additives prevented and delayed ice formation. Some researchers used the decreasing rate of BPN to evaluate the skid resistance durability of asphalt pavement (Xu et al. 2015). The result indicated the decreasing rate of BPN reduced by 37% in the presence of NaCl-based additives that 100% replaced mineral fillers.

2.2.3.4 Inclined shear test, hammer load test, ice layer rupture test and ice layer pull-out test

The magnitude of the bond of ice to asphalt pavement can be quantitatively evaluated by similar tests, including but not limited to inclined shear test (Figure 3), hammer load test, horizontal bond shear test (Figure 4), ice layer rupture test (Figure 5) and ice layer pull-out test. The range of the testing temperatures for these tests were from -1 °C to -15 °C.





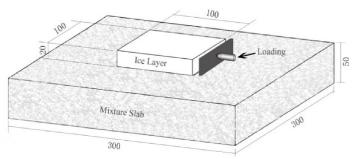


Figure 4 Horizontal bond shear test (Luo and Yang 2015)



Figure 5 Ice layer rupture test (Ma et al. 2016)

A research group used the 100 kN load produced by the free fall of a hammer to impact the ice formed on mixture samples with the impact frequency of 30 times per minute until the ice was completely cleared (Zheng et al. 2017). The control sample required 61 impacts whereas the sample with 5 % Verglimit by the mass of asphalt mixture required 46 impacts. The authors explained the salt solution formed at the interface between ice and mixture surface reduced the adhesion of ice to sample, leading to easier removal of ice than for the control samples.

An ice-layer-rupture test uses a constant load to push a loading head with a certain speed into an ice layer on mixture sample until the ice is ruptured. Ma et al. reported the rupture pressure used to break a control sample was 1.7 times of that for the sample with 6 % NaClbased additives by the mass of aggregates (Ma et al. 2016). The rupture strength of the sample with NaCl-based additives used to replace 100% mineral fillers was half of that of the control sample.

An ice layer pull-out test is when a pull-out head is frozen in an ice layer on mixture sample followed by measuring the pull-out strength with a constant speed. The pull-out strength of the sample with 6 wt% additives was about half of that of control sample (Ma et al. 2016), and a similar result was obtained in another study (Ma et al. 2018).

2.2.3.5 Snow-traffic-plowing-friction test

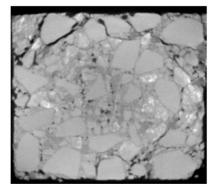
A comprehensive and sophisticated test named 'snow-traffic-plowing-friction test' not only evaluates deicing and anti-icing performances in terms of friction measurement and snowpavement bond measurement but also simulates real traffic conditions on roads (Akin et al. 2018a; b). Sieved snow was compacted onto a slab sample followed by trafficking, snowpavement bond measurement, plowing and friction measurement. Static friction measurements were conducted throughout the test beginning on clean, bare pavement, after snow compaction, after trafficking and after plowing. Figure 6 shows the sequence of steps. Salt chemicals could be blended into an asphalt mixture or applied before or after snow compaction for anti-icing and deicing evaluation.



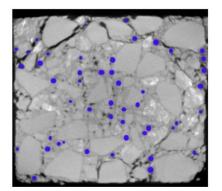
Figure 6 Sequence of steps during snow-traffic-plowing-friction tests (Akin et al. 2018b)

2.2.3.6 Factors influencing anti-icing performances

Although the anti-icing performance of asphalt pavement mainly depends on the content of salt-storage additives, other aspects including salt categories, additive distribution, additive properties and environmental conditions have varying degrees of impact on anti-icing. The distribution of additives in asphalt pavement can be observed by the cross-sectional images acquired by X-ray CT scanning technology, followed by a digital image processing technique, as shown in Figure 7 (Zheng et al. 2017). Better distribution of additives may show potential to prevent ice formation on pavement surface. The particle size of additives can influence the antiicing property as well. A smaller particle size showed more potential to higher hydrophobicity (Liu et al. 2014b), which means smaller particles may have a lower salt-release rate. On the other hand, the testing results also revealed a better anti-icing performance occurred with a larger particle size, which was explained by the larger surface area contacting with water. Furthermore, it has been shown that the additives with low surface energy reduced the adhesion between pavement and ice (Wright et al. 2016). Snow precipitation and environmental temperature may greatly limit the anti-icing performance of asphalt pavement with salt-storage additives. A better anti-icing performance can be achieved with a lower rate of snow precipitation and a higher temperature condition (more than 20 °F, possibly even 27 °F) (Liu et al. 2014b; Shi et al. 2018).



(a) Before image processing



(b) After image processing

Figure 7 Cross-sectional images by CT scanning technology (Zheng et al. 2017)

2.2.4 Anti-Icing Durability Evaluation

A long anti-icing service life is a critical criterion to determine the success of an antiicing asphalt pavement. Few studies reported on the anti-icing durability evaluation for asphalt pavement with salt-storage additives. However, a conductivity test (also called an immersion test) is usually adopted to evaluate the anti-icing durability.

Although different apparatuses have been used for the conductivity test in previous studies, the testing procedures are similar (Giuliani et al. 2012; Ma et al. 2016; Zheng et al. 2015, 2016). Unsealed or partially sealed mixture samples are immersed in DI water with different temperature conditions followed by the measurements of the conductivity or density of the solution with dissolved salts over time, and the whole system is sealed to avoid any vapor evaporation, as shown in Figure 8. The conductivity or density can be converted to be the release rate of the salts escaping from asphalt pavement, and they have a positive relationship. Giuliani et al. reported NaCl ions were still releasing from mixture samples after a one-month testing period even with fresh water replacement and mechanical abrasion on sample surface (Figure 9), indicating the high anti-icing durability (Giuliani et al. 2012). They explained it as due to the slow diffusion of NaCl from the interior to the surface through the micropores inside the asphalt mixture.

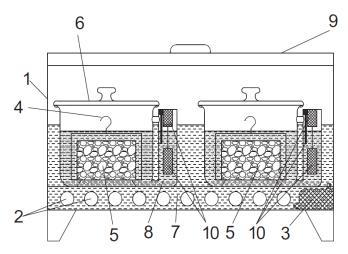


Figure 8 Schematic diagram of conductivity test (1) incubator; (2) heating tube; (3) water recycle system; (4) support; (5) specimen; (6) volumetric flask; (7) water bath; (8) solution; (9) cap; (10) density test device (Zheng et al. 2015)

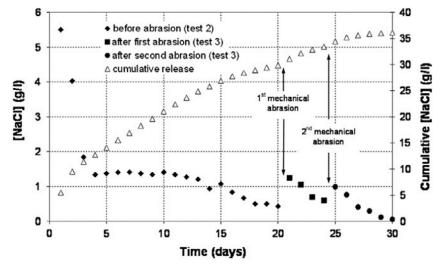


Figure 9 NaCl releasing process of open-graded sample (Giuliani et al. 2012)

Anti-icing durability varies with the content of additives in asphalt pavement and can be influenced by other factors as well, including but not limited to particle size, testing temperature, mixture category, external load, and environmental condition. A smaller particle of IceBane led to a lower conductivity of the solution with dissolved salts, due to smaller particles having a higher hydrophobicity than larger particles (Liu et al. 2014b). The salt-release rate gradually increased with the rise of the testing temperature owning to the higher activities of salt molecules (Zheng et al. 2015) as well as the softer asphalt binder that had a higher permeation coefficient (Zheng et al. 2016). Liu et al. suggested dense-graded mixtures tend to reduce the loss rate of the active ingredients in additives thus increasing the long-term anti-icing durability, compared to open-graded mixtures (Liu et al. 2015a). Moreover, a conductivity test was conducted with a dynamic load on mixture samples (Figure 10) to simulate traffic on roads (Liu et al. 2015b). The results (Figure 11) indicated dynamic loadings facilitated salt release, compared to static loadings. Meanwhile, salt-release characteristics were positively influenced by the content of additive, wetting-drying cycle, solution temperature and immersion time. Based on the obvious difference between the first cycle and the second cycle, researchers inferred that the first rainfall might significantly weaken the anti-icing performance of asphalt pavement with additives after construction.

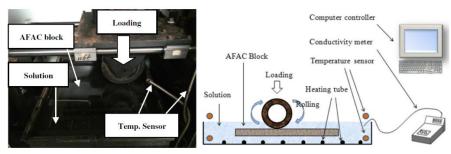


Figure 10 Dynamic loading and conductivity test system (Liu et al. 2015c)

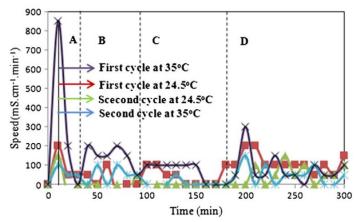


Figure 11 Salt-release rate of AFAC under dynamic loading conditions (Liu et al. 2015c)

2.2.5 Effects on Mechanical Performances of Asphalt Pavement

The importance of the mechanical properties of asphalt pavement is indubitable, because these properties reflect the durability and the serviceability and relate to the subsequent cost of the maintenance. There are many indices that can be used to evaluate the mechanical performances of asphalt pavement such as tensile strength, flexural strength, splitting strength, cracking resistance, rutting resistance, moisture susceptibility, Marshall flow and stability, fatigue potential, etc. They can be classified into three categories: low-temperature performance, intermediate-temperature performance and high-temperature performance. In cold regions, the low-temperature performance of asphalt pavement seems to account for a greater proportion of importance on the engineering properties than the intermediate-temperature and hightemperature performance as pavement temperature is commonly below 0 °C during winter. On the other hand, the presence of salts in additives has high potential risk to reducing the moisture damage resistance of asphalt pavement whatever the temperature is. However, salt-storage additives have been proven to impact the mechanical performance of asphalt pavement (Li and Wang 2012; Liu et al. 2015b; Ma et al. 2018; Zheng et al. 2017). The following sections focus on moisture susceptibility, rutting resistance and lowtemperature cracking resistance, which are usually used to evaluate the mechanical performances of asphalt pavement.

2.2.5.1 Moisture susceptibility

Moisture damage, expressed as the loss of strength and durability due to moisture (Wright et al. 2016), is a primary distress in asphalt pavement. Moisture susceptibility is commonly used to predict the potential for damage caused by moisture (Li and Wang 2012). Indirect tensile (IDT) test, Marshall test and freeze-thaw splitting test are usually used to evaluate moisture susceptibility, which can be expressed as wet-dry tensile strength ratio, freezethaw splitting strength ratio and immersion Marshall strength ratio, etc. The Hamburg wheeltrack test can be utilized to measure moisture susceptibility (expressed as stripping inflection point, SIP) however; this method has not been mentioned in the literature to evaluate the moisture susceptibility of anti-icing asphalt pavement with salt-storage additives.

Generally, salt-storage additives have adverse impacts on moisture susceptibility. Zheng et al. blended 5 wt% Verglimit and 5 wt% Iceguard into asphalt mixtures respectively, in which mineral fillers were not replaced and remained unchanging (Zheng et al. 2017). The results showed the moisture susceptibility reduced by 7% for both. The adverse effect of Verglimit on moisture susceptibility was also confirmed by wet-dry Marshall strength ratio and wet-dry tensile strength ratio (Zheng et al. 2015). Mafilon replacing 6.2% mineral fillers by the mass of total aggregates led to a reduction of residual stability by 6.5% and freeze-thaw splitting residual strength ratio by 7.5% respectively (Li and Wang 2012). The results from binder affinity test and water sensitivity test in accordance with BS EN 12697-11:2005 and BS EN1297-12:2003 respectively showed sodium silicate was highly sensitive to water even with a small amount

whereas 100% NaFm replacement had a similar effect on moisture susceptibility with limestone fillers (Wright et al. 2016). Other NaCl-based additives have negative effects on moisture susceptibility as well (Ma et al. 2016, 2018; Wang et al. 2017). It has been commonly accepted that the weak adhesion (including chemical absorption and physical adhesion) between binder and aggregate accounts for the moisture damage of asphalt pavement, and that chemical absorption plays a dominant role (Zheng et al. 2016).

In some cases, the addition of salt-storage additives improves moisture susceptibility. Liu et al. demonstrated 1~6% IceBane by the mass of aggregates slightly improved moisture susceptibility, owing to the existence of a special alkaline modifier in IceBane (Liu et al. 2016). The smaller particles of IceBane seemed to have less impact on the moisture susceptibility of asphalt pavement than the large particles, based on the result of freeze-thaw splitting test (T0729-2000, national standard of China) (Liu et al. 2014b).

2.2.5.2 Rutting resistance

Wheel tracking dynamic stability test (Figure 12a) and asphalt pavement analyzer (APA) wheel-tracking test (Figure 12b) are two tests commonly used to measure the rutting behavior of asphalt pavement. The wheel-tracking test (D0719-1993, national standard of China) usually used in China, generates an output index of dynamic stability (DS) with the unit of times/mm. DS can be defined as the number of repeated cycles that result in 1-mm permanent deformation of asphalt pavement under a dynamic wheel load at a specific high-temperature condition. The APA wheel-tracking test conducted to evaluate rutting resistance measures the permanent deformation of asphalt pavement under a dynamic wheel load with repeated cycles at a specific high-temperature condition.

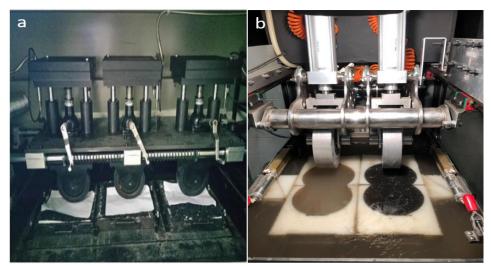


Figure 12 Device for a) wheel tracking dynamic stability test (Ma et al. 2016) and b) asphalt pavement analyzer wheel-tracking test

The rutting resistance of asphalt pavement can be impacted detrimentally with varying levels by different salt-storage additives. For example, the DS of asphalt mixture decreased with the increase of the content of Mafilon (Figure 13), and the DS reduced by 45% if 100% mineral fillers were replaced (Li and Wang 2012). The authors also concluded the rut depth gradually increased with the increase of repeated cycles and reached the largest value after 8000 times of cyclical loading in the case of replacing 100% mineral powder. Other NaCl-based additives have been studied and similar results reported (Ma et al. 2016, 2018).

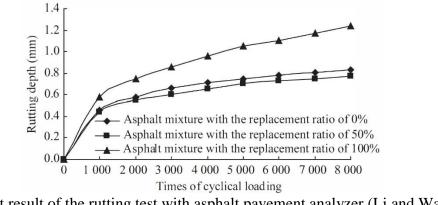


Figure 13 Test result of the rutting test with asphalt pavement analyzer (Li and Wang 2012)

On the other hand, some salt-storage additives enhanced the rutting resistance of asphalt pavement. Zheng et al. demonstrated the addition of 5% Verglimit by the mass of asphalt mixture led to a DS that increased by 22%, and this might be due to the accessory components in Verglimit (Zheng et al. 2015). Wang et al. confirmed 20 vol% laboratory-prepared NaCl-based SSAs replacing limestone aggregates (LASs) improved the DS of asphalt mixture by 16%, which was explained by the higher mechanical strength of SSA than that of LAS (Wang et al. 2017).

2.2.5.3 Low-temperature cracking resistance

Low-temperature cracking resistance reflects the ability of asphalt pavement to resist cracking at low temperatures. The testing methods include thermal stress-restrained specimen test, three-point bending beam test, IDT test, and so on. Through these tests, the variations of the interior stress and strain of asphalt pavement with a static or dynamic temperature condition can be measured under a static or dynamic load. The variation curve of the stress with the drop of temperature and the failure stress of asphalt pavement can be obtained as shown in Figure 14 (Li and Wang 2012).

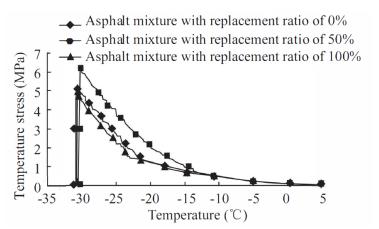


Figure 14 Temperature-stress curve of asphalt pavement (Li and Wang 2012)

The low temperature (thermal) cracking resistance of asphalt pavement usually reduces with the addition of salt-storage additives. Mafilon (Li and Wang 2012) and another commercialized NaCl-based additive (Xu et al. 2015) showed little impact on the cracking temperature of asphalt pavement even if 100% mineral fillers were replaced, but the cracking stress reduced by about 8% (Li and Wang 2012). Ma et al. reported the low temperature cracking resistance of asphalt pavement degraded as the content of salt-storage additives increased (Ma et al. 2018). Meanwhile, the failure flexural strain derived from low-temperature flexural test decreased by 36% when the total mineral fillers (7% by the mass of aggregates) were replaced. The reduction of the low-temperature cracking resistance might result from the micro-fissures produced by the dissolution of salt particles in asphalt pavement (Liu et al. 2015b).

Some of salt-storage additives may improve low-temperature cracking resistance, due to the other ingredients beyond salts inside additives. With 5% Verglimit by the mass of asphalt mixture, the flexural strain at failure increased by 19% based on low temperature bending test (Zheng et al. 2015). The cementitious hydration produced on the surface of laboratory-prepared NaCl-based SSAs helped improve the interfacial adhesion between asphalt binder and aggregates, enhancing the low-temperature cracking resistance of asphalt pavement (Wang et al. 2017).

2.2.5.4 Contributions to deterioration

The adverse effects of salt-storage additives on the mechanical performances of asphalt pavement relates to their physical and chemical properties and the components, as well as the interaction between additive and asphalt binder.

Previous studies have reported 1 - 2 wt% solid deicers relative to NaCl, NaFm or KAc caused serious detrimental effects in terms of the weight loss of the mixture samples collected

from fields when subjected to freeze-thaw cycles (Hassan Y. et al. 2002). The potential deterioration mechanism of the asphalt pavement exposed to chloride-based, acetate-based and formate-based deicers seems to be a combination of chemical reactions and emulsifications, as well as the extra stresses produced in the samples (Shi et al. 2013).

Ma et al. indicated the reduction in the moisture susceptibility, the rutting resistance and the low-temperature cracking resistance of asphalt pavement may attribute to the porous structure, the weak strength and the smooth-surface character of coarse-aggregate-like NaCl-based additives, compared to real aggregates (Ma et al. 2016).

Liu et al. indicated the particle size of IceBane significantly influenced the rutting resistance of asphalt pavement (Liu et al. 2014b). The authors found the rutting resistance of the samples with coarser IceBane particles was slightly greater than that of control samples, whereas finer IceBane particles made the rutting resistance significantly lower than control samples, as shown in Table 2 and Figure 15. Meanwhile, finer IceBane particles resulted in greater reduction in the rutting resistance.

Passing rate (%)				
0.30	0.15	0.075		
89.17	51.25	21.39		
92.82	62.24	43.95		
92.49	72.38	55.17		
95.11	90.12	78.50		
95.51	90.24	78.33		
	89.17 92.82 92.49 95.11	89.17 51.25 92.82 62.24 92.49 72.38 95.11 90.12		

Table 2 Fineness of anti-freeze filler and mineral powder (Liu et al. 2014b)

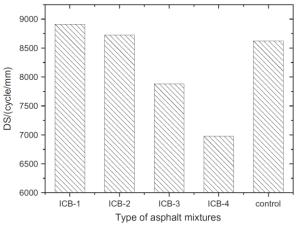


Figure 15 Result of rutting test with different IceBane particle sizes (Liu et al. 2014b)

Moreover, salt-storage additives caused the higher air void in asphalt pavement and the poorer physical adhesion as well as weaker chemical absorption between asphalt binder and aggregate, which may account for the decline in the mechanical performances as well (Luo and Yang 2015; Xu et al. 2015; Zheng et al. 2016). The capillary action of the dilute salt solution and the recrystallization of NaCl occurring in asphalt pavement may generate additional internal stress, leading to the reduction of the mechanical performances (Xu et al. 2015). However, there is no direct evidence to interpret the mechanisms of the pavement deteriorations related to various salt-storage additives.

2.2.5.5 Approaches to compensation

Although most of salt-storage additives resulted in depression of the mechanical performance of asphalt pavement, blending other advanced materials into the pavement compensated for the impact. Ma et al. demonstrated the addition of 0.3% polyester fibers by the weight of aggregates with 6 mm in length and 0.2 mm in diameter significantly improved the moisture susceptibility, the rutting resistance and the low-temperature cracking resistance of the asphalt pavement with commercialized NaCl-based additives (Ma et al. 2016). An advanced

granular modifier with 2-3 mm in diameter and mainly composed of thermoplastic elastomer, adhesive resin, plasticizer and other stabilizing agents has been investigated for the mechanical improvement of asphalt pavement with commercialized NaCl-based additives (Ma et al. 2018). The addition of 15% modifiers by the mass of asphalt binder compensated for the adverse impacts of salt-storage additives (100% replaced mineral fillers) to the mechanical performance of asphalt mixture. Similar studies can be found in the references (for example Luo and Yang 2015; Min et al. 2017).

2.3 Summary and Conclusions

For anti-icing asphalt pavement with salt-storage additives, the researchers drew the following conclusions based on the review of relevant literature:

- Salt-storage additives classify into two categories: Cl-based additives and non-Cl-based additives. CaCl₂-based and NaCl-based additives are the two main types of commercialized Cl-based additives, including WinterPave, Verglimit, Iceguard, Mafilon and IceBane. Some additives like NaFm, sodium silicate, Mg-Al/Cl⁻ LDH and Mg₂/Al-Ac⁻ LDH were less studied. The salt content usually ranged from 55 wt% to 75 wt%. Generally, the particle size was less than 5 mm, and most of them were similar with the size of mineral fillers. In addition, SiO₂, MgO, CaO, Al₂O₃, Na₂O, K₂O, Fe₂O₃, MgCO₃, CaCO₃ and MgCl₂ were often blended into the additives as auxiliary ingredients.
- 2. There was no standard method to evaluate the anti-icing performance of asphalt pavement with salt-storage additives. To this end, the measurements related to the bond of snow and ice to pavement or the friction condition between pavement and vehicle tire have been developed, including inclined shear test, hammer load test, ice layer rupture test, ice layer

pull-out test and snow-traffic-plowing-friction test. Water freezing point test and skid resistance test were used less. The efficiency of salt-storage additives depended on the physical and chemical properties of additives, the content and distribution in asphalt pavement, the air void of asphalt pavement, as well as environmental condition, etc.

- 3. The conductivity test (also called immersion test) was often adopted to evaluate the antiicing durability of asphalt pavement with salt-storage additives. The anti-icing durability varied with different additives, influenced by the particle size of additive, environmental condition, the air void of asphalt pavement and external load, etc.
- 4. Moisture resistance, rutting resistance and low-temperature cracking resistance were the main mechanical performance features of anti-icing asphalt pavement with salt-storage additives studied in literature. Generally, salt-storage additives resulted in detrimental impacts on moisture susceptibility, rutting resistance and low-temperature cracking resistance. Nevertheless, blending advanced materials minimizes the degradation. The adverse effects of additives on the mechanical performance of asphalt pavement relates to their physical and chemical properties and the components, as well as the interaction between additive and asphalt binder.

Even though the asphalt pavement with those commercialized or laboratory-prepared salt-storage additives has been proven to have many advantages for snow and ice control, there are still problems to be considered. Typically, 5 - 6 wt% or greater salt-storage additives are used to prepare the asphalt pavement that has an effective anti-icing function, which greatly reduces the application of salt chemicals compared to traditional application methods. However, such an amount of salt chemicals blended in asphalt pavement is still massive and not cost-effective. The

addition of 5-6 wt% or even less salt chemicals may result in significant adverse effects on the mechanical performances of asphalt pavement.

The limitations of the anti-icing asphalt pavement with salt-storage additives also include that it is difficult to control the release rate of salt chemicals in additives; difficult to reduce the risk of degrading the mechanical performance in the presence of salts; and difficult to make antiicing effective at low temperatures (below 20 °F). The anti-icing effectiveness and durability of this novel type of asphalt pavement requires more in-depth investigations.

CHAPTER 3.0 DEVELOPING ANTI-ICING ASPHALT PAVEMENT WITH LABORATORY-PREPARED SALT-STORAGE ADDITIVES

3.1 Introduction

Chapter 2 provided a review and synthesis of the state-of-the-art literature on anti-icing asphalt pavement with salt-storage additives, and focused on the type of salt-storage additives, anti-icing testing methods and performance evaluation, and mechanical testing methods and performance evaluation. This chapter will focus on the development of an anti-icing asphalt mixture incorporating laboratory-prepared salt-storage additives, featuring a sustained saltrelease rate. The researchers evaluated the anti-icing performance and mechanical properties of asphalt mixture with the additives. The innovative salt-storage additives were prepared through a surface treatment approach, in which zeolite containing $CaCl_2$ was coated by a porous epoxy layer (CaCl₂-zeolite/p-epoxy). The microporous structure of the epoxy coating on zeolite particles was designed to extend the anti-icing service life of the additives by controlling the releasing rate of CaCl₂ from the asphalt mixture. Fog-freezing friction test and anti-icing longevity test investigated the anti-icing capability and determined the effective anti-icing period of asphalt pavement containing the additives. The mechanical properties including moisture damage resistance, rutting resistance, mid-temperature cracking resistance, and low-temperature cracking resistance were investigated by Hamburg wheel-track test and IDT test.

3.2 Experimental

3.2.1 Materials

Western State Asphalt, Spokane, Washington, supplied the PG 64-22 asphalt binder used in this research. Poe Asphalt Paving Inc, Pullman, Washington, provided basalt aggregates.

Granular CaCl₂ was purchased from Thermo Fisher Scientific (C79-500). Porous zeolite particles were purchased from KMI Zeolite. The angular zeolite particles with 15% in pore volume and 4.0 - 7.0 angstroms in pore diameter mainly compose of 96.9% Clintoptilolite-Ca, 2.3% Montmorillonite and other ingredients, and the crushing strength and the hardness is 176 kgs/cm² and 4.0 - 5.0 Mohs respectively. Technical data about zeolite particles is on the website of the company (https://www.kmizeolite.com/technical-data/). The epoxy used for coating zeolite was Dura-Plate 235 Multi-Purpose Epoxy (B67W235 Mill White Part A and B67V235 Hardener Part B) purchased from Sherwin-Williams. The epoxy provides exceptional performance in saltwater and freshwater immersion resistance and can be applied at temperatures as low as -17.7 °C (0 °F). Halloysite nanoclay purchased from Sigma-Aldrich was used to avoid undesired defects and pores forming in epoxy coating and to increase the affinity and compatibility of asphalt binder and epoxy coating. Acetone (AR® ACS) was purchased from Macron Fine ChemicalsTM.

3.2.2 Fabrication of innovative salt-storage additives

The innovative salt-storage additives with a sustained CaCl₂-release rate had three gradations (sieve #4 - #8, sieve #8 - #16 and sieve #16 - #30, hereinafter referred to as #8, #16, and #30) and were prepared in the laboratory. First, adequate 60 wt% CaCl₂ solution was pumped into a vacuumed container that contained 200 g porous zeolite particles. The vacuum condition was maintained by a vaccum pump for 2 hours. The wet zeolite particles containing CaCl₂ (CaCl₂-zeolite) then dried in an oven at 110 °C overnight and sieved. The coating material was a nanoclay modified acetone-epoxy mixture. First, 20 mL epoxy A was mixed with 5 mL epoxy B using a magnetic stirrer for 10 min. Second, 0.3 g nanoclay and 5 mL acetone were added into the epoxy and mixed for 10 min. The mixture then was sonicated for 10 min. Subsequently, 23 g dry CaCl₂-zeolite particles were coated using 2.4 mL, 3 mL, and 3.6 mL

mixture for the particle #8, #16, and #30 respectively. Finally, the coated particles dried in an oven at 50 °C for 24 hours. Figure 16 shows the final products. The CaCl₂ content in additives #8, #16, and #30 was 20.1%, 20.5%, and 21.1% respectively, based on the mass difference between the additives with and without CaCl₂.

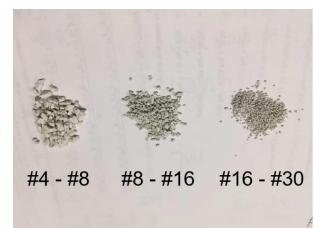


Figure 16 Laboratory-prepared CaCl₂-zeolite/p-epoxy

3.2.3 Mixture preparation

The optimum binder content of asphalt mixture was 5.1 wt% designed with the Superpave method. The gradation of the aggregates used for HMA mix design is shown in Table 3. Three kinds of asphalt mixture with the diameter of 150 mm were prepared using the Superpave gyratory compactor, including the samples with 100% CaCl₂-zeolite/p-epoxy by the mass of asphalt binder, the samples with 100% CaCl₂-zeolite by the mass of asphalt binder, and the samples without additive (control). CaCl₂-zeolite/p-epoxy or CaCl₂-zeolite replaced the same-size traditional aggregates in asphalt mixture.

Table 3 Aggregate gradation used for HMA mix design

Sieve opening (mm)	19	12.5	9.5	4.75	2.36	1.18	0.6	0.3	0.15	0.075
Percent passing (%)	100	93	79	50	33	24	17	13	8	4.8

3.2.4 **Testing methods**

The researchers used the fog-freezing friction test to evaluate the anti-icing capability of asphalt mixture with CaCl₂-zeolite/p-epoxy or CaCl₂-zeolite. Static friction measurements were performed on clean, bare mixture samples at the test beginning and at 15, 30, 45 and 60 min after 10-time moisture spray on sample surface. The project purchased an All-Purpose sprayer from Ace Hardware and used it to spray moisture. Figure 17 illustrates the testing procedure. All the mixture samples were conditioned overnight at testing temperatures (-3.9 °C and -9.4 °C).



Friction on dry sample

Figure 17 Testing procedure of fog-freezing friction test

The effective anti-icing period of asphalt payement with these laboratory-prepared additives was predicted by Equation 1 (Zhou et al. 2019):

$$T = \frac{m}{24vt} \tag{1}$$

Where T is effective anti-icing period in year, m is mass per square meter of $CaCl_2$ in asphalt pavement with additives in g/m^2 , v is the releasing rate per square meter of CaCl₂ from asphalt pavement with additives in $g/(m^2 \cdot h)$, and t is days of local precipitation (rainfall and snowfall) per year in days/year. v was calculated based on the Cl^{-} concentration that was determined by soaking method, in which a sample with 63.5 mm in height and 150 mm in diameter was soaked in DI water in a sealed container. The Cl⁻ concentration under a certain temperature was measured at intervals. The test simulated the local climatic conditions in Pullman, WA. Because v depends highly on temperature, 15 °C and 0 °C were chosen as two testing temperatures. 15 °C simulates the temperature when it was raining during summer, and obtained by averaging the max and min temperatures on rainy days in 2018. 0 °C resembles the temperature when snow or ice was melting during winter. Thus, Equation 1 converts to Equation 2:

$$T = \frac{m}{24(v_s t_s + v_w t_w)} \tag{2}$$

Where v_s and t_s are v and t simulated during summer respectively, and v_w and t_w are v and t simulated during winter respectively. t_s and t_w for Pullman in 2018 were 47 days/year and 59 days/year respectively, which were counted from the website of U.S. Climate Data.

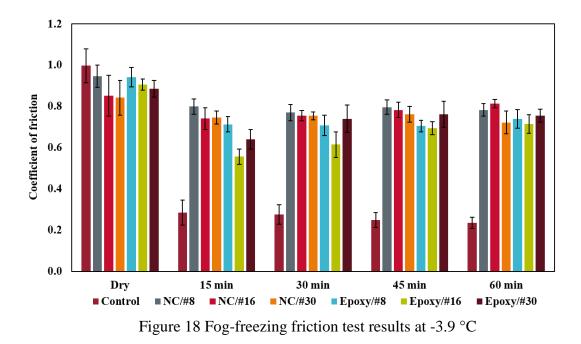
The Hamburg wheel-track test evaluated the rutting resistance and moisture damage resistance of asphalt mixture with CaCl₂-zeolite/p-epoxy in water at 50 °C. The testing procedure followed AASHTO T283, and the air void of the samples was $7\% \pm 0.5\%$. The tests stopped when tests reached a 12.5-mm rut depth or at the end of 20,000 passes, whichever came first.

IDT test evaluated the low-temperature (-10 °C) and mid-temperature (25 °C) cracking resistances of asphalt mixture with CaCl₂-zeolite/p-epoxy in terms of IDT strength and fracture work density. Samples were monotonically loaded to failure along the vertical diametric axis at the constant rate of 12.5 mm/min for -10 °C and 50 mm/min for 25 °C. The testing procedure followed AASHTO T322 for -10 °C and ASTM D6931 for 25 °C. The air void of the samples was 7% \pm 0.5%. Researchers ran the tests in triplicate at least, and reported averaged results.

3.3 Results and Discussions

3.3.1 Anti-icing capability

Error! Reference source not found. shows the results of the fog-freezing friction tests at -3.9 °C. In the figure, NC/# (e.g., NC/#8) represents the samples with CaCl₂-zeolite #8, and Epoxy/# (e.g., Epoxy/#16) represents the samples with CaCl₂-zeolite/p-epoxy #16.



The friction coefficient of dry samples varied with the change of additive category at the beginning of a test, although all the values were greater than 0.80. The friction coefficient of control samples (1.0) was slightly greater than that of samples with CaCl₂-zeolite/p-epoxy or CaCl₂-zeolite due to trace amounts of moisture attracted to the surface of samples by the presence of CaCl₂. The presence of moisture made sample surface more slippery. CaCl₂-zeolite seemed to result in a lower friction coefficient, compared to CaCl₂-zeolite/p-epoxy. Uncoated CaCl₂-zeolite exposed more CaCl₂ to the air than coated CaCl₂-zeolite/p-epoxy, resulting in more moisture on sample surface. Furthermore, the friction coefficient tended to decrease with reduction of the average particle size of the additive for both CaCl₂-zeolite and CaCl₂-zeolite/p-epoxy. The decrease might be related to the shape of the additives. A larger angular additive (e.g., #8) produced a rougher asphalt surface, whereas a smaller round additive (e.g., #30) resulted in a smoother surface.

The friction coefficient of control samples stayed low, less than 0.3 at 15, 30, 45 and 60 min after moisture spray, while the friction coefficients of samples with the additives were greater than 0.55. Meanwhile, the friction coefficient of control samples decreased continuously over time, whereas the friction coefficients of samples with the additives tended to increase. For example, the friction coefficient of Epoxy/#16 increased from 0.56 to 0.72. Moisture froze after being sprayed. As time passed, ice started to melt due to the existence of the high-concentration CaCl₂ solution on the surface of samples with the additives. At 15 min, the CaCl₂ solution melted the frozen moisture that contacted the surface asphalt mixture, and the rest of the frozen moisture remained frozen. The melting process kept going until all ice melted or the concentration of the CaCl₂ solution diluted to the eutectic point (Klein-Paste and Dalen 2018) at -3.9 °C. Moreover, contrary to the results at the beginning of testing, uncoated CaCl₂-zeolite seemed to give rise to higher friction coefficients after moisture spray, compared to CaCl₂-zeolite/p-epoxy. This was because uncoated CaCl₂-zeolite exposed more CaCl₂ than CaCl₂-zeolite/p-epoxy to the air, resulting in a higher concentration of the CaCl₂ solution following a faster melting. Nevertheless, CaCl₂-zeolite/p-epoxy had a similar anti-icing performance to CaCl₂-zeolite at 60 min as all the friction coefficients were around 0.75. It is worth noting that throughout the time after moisture spray, CaCl₂-zeolite/p-epoxy #30 showed better anti-icing capability, compared to CaCl₂zeolite/p-epoxy #8 and #16.

Figure 19 shows the results of the fog-freezing friction tests at -9.4 °C. The friction coefficients of samples at -9.4 °C had similar behaviors with that at -3.9 °C, except samples with CaCl₂-zeolite/p-epoxy. The friction coefficients of dry samples were greater than 0.8 at the beginning of testing. The friction coefficient of control samples stayed low, measuring less than 0.4 at 15, 30, 45 and 60 min after moisture spray. The friction coefficients of samples with

CaCl₂-zeolite were greater than 0.60. The friction coefficient of control samples decreased over time, whereas the friction coefficients of samples with CaCl₂-zeolite slightly increased. Throughout the time after moisture spray, CaCl₂-zeolite/p-epoxy #30 showed better anti-icing capability, compared to CaCl₂-zeolite/p-epoxy #8 and #16. The above results were consistent with the results at -3.9 °C.

The friction coefficients of samples with CaCl₂-zeolite/p-epoxy at 15, 30, 45 and 60 min after moisture spray greatly decreased at -9.4 °C, compared to the values at -3.9 °C. This occurred because epoxy coating limited the connection of CaCl₂ in CaCl₂-zeolite with moisture or ice. However, the friction coefficients of samples with the additive (e.g. CaCl₂-zeolite/p-epoxy #30) gradually increased from 0.4 at 15 min to 0.55 at 60 min, which were still significantly greater than the friction coefficient of control samples.

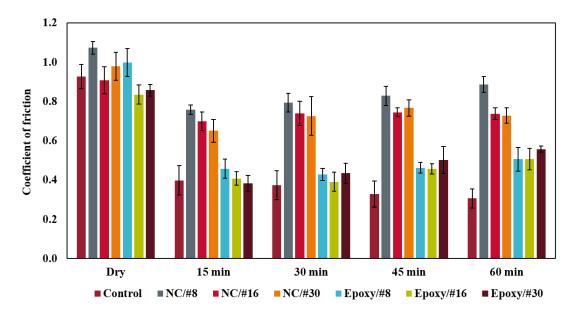


Figure 19 Fog-freezing friction test results at -9.4 °C (Note: the friction coeffcient > 1 was caused by experimental errors, within acceptable limits)

3.3.2 Anti-icing longevity

The temporal variation of the mass of CaCl₂ that leached out of the asphalt mixture in 1.5L DI water at intervals is shown in Figure 20. The mass of CaCl₂ in the water increased fast during the first 48 hours for all the samples, which can be attributed to the quick dissolution of the CaCl₂ not well encapsulated in the CaCl₂-zeolite/p-epoxy (as the outcome of the fabrication process of the salt-storage additives). Subsequently, the releasing rate of CaCl₂ suddenly decreased and became nearly steady (as indicated by a stable slope of chloride concentration increase over time). The inflection point indicated the encapsulated CaCl₂ in CaCl₂-zeolite/pepoxy inside asphalt mixture started to leach out of the coated zeolite.

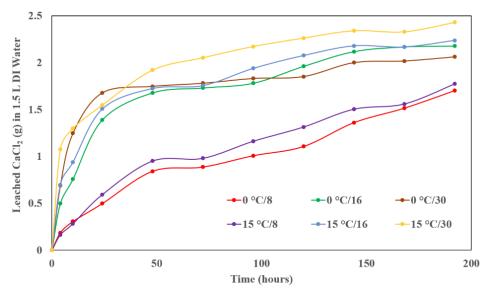


Figure 20 Anti-icing longevity test results

v in terms of the slope of the curve after 48 hours and T are shown in Table 4. 0 °C and 15 °C did not result in a great difference in *v*. However, *v* varied greatly as a function of the particle size of CaCl₂-zeolite/p-epoxy. When reducing the size of CaCl₂-zeolite/p-epoxy, *v* decreased from the maximum value of around 0.10 g/(m²·h) to the minimum value of around 0.04 g/(m²·h). Based on Equation 2, the estimated effective anti-icing period of asphalt pavement

containing CaCl₂-zeolite/p-epoxy #8, #16, and #30 was 5.8 years, 9.9 years, and 15.3 years, respectively.

Temperature/Sample #	Slope of Curve after 48 hours (g/h)	v (g/(m²·h))	T (year)	
0 °C/#8	0.0070	0.107	5 0	
15 °C/#8	0.0064	0.098	5.8	
0 °C/#16	0.0042	0.064	0.0	
15 °C/#16	0.0038	0.058	9.9	
0 °C/#30	0.0025	0.038	15.2	
15 °C/#30	0.0029	0.044	15.3	

3.3.3 Moisture damage resistance and rutting resistance

Figure 21 illustrates the results of Hamburg wheel-track tests, and Figure 22 shows an example of the data analysis of the results. The outputs used to evaluate moisture susceptibility and rutting resistance include creep slope, stripping slope, number of passes at stripping inflection point (SIP), number of passes at failure, as well as the rut depth at 5000th, 10000th and 15000th pass. Creep slope is the number of passes required to produce a 1-mm rut depth, which primarily derives from plastic flow. Number of passes at SIP is the number of passes at the intersection of the creep slope and stripping slope. Stripping activity starts to dominate performance from this point. Stripping slope is the number of passes required to produce a 1-mm rut depth after the SIP, and it is a measure of the accumulation of rutting primarily from moisture damage (Izzo and Tahmoressi 1999).

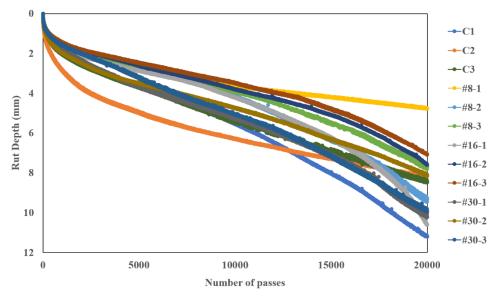


Figure 21 Hamburg wheel-track test results

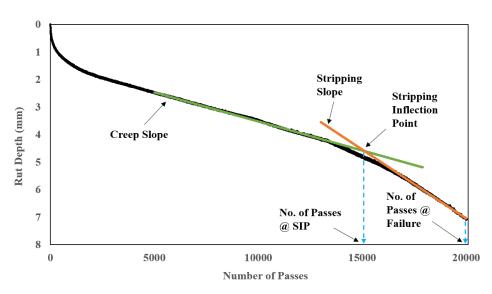


Figure 22 An example of data analysis for Hamburg wheel-track test results

The number of passes at SIP as shown in Table 5 evaluated the moisture damage

3.3.3.1 Moisture damage resistance

resistance of asphalt mixture. No control samples, sample #8-1, sample #30-2, and sample #30-3 revealed a stripping phenomenon before 12.5 mm rut depth or 20000 passes, demonstrating their high moisture damage resistances. Although sample #8-2, sample #8-3, all the samples #16 and sample #30-1 presented stripping behaviors, the number of passes at SIP were greater than 15000 passes according to the standard specifications of Washington Department of Transportation

(WSDOT) (WSDOT 2018), excepting sample #8-3 that had 14143 passes at SIP. The SIP is invalid and the mixture is considered passing if the ratio of stripping slope and creep slope is less than 2.0 according to Wisconsin and Iowa specifications (Chaturabong and Bahia 2017). All the ratios of the samples that had a stripping behavior were greater than 2.0, indicating the samples passed Wisconsin and Iowa specifications.

The addition of CaCl₂-zeolite/p-epoxy slightly reduced the moisture damage resistance of asphalt mixture. The asphalt mixture that has a larger number of passes at SIP is considered to have a better moisture damage resistance. Based on an overall consideration to the number of passes at SIP from Table 5, the order of the magnitude of the moisture resistance from high to low was control > #30 > #16 > #8.

Sample #	Number of Passes at SIP	Creep Slope (Passes/mm)	Stripping Slope (Passes/mm)	Ratio of Stripping Slope (mm/Pass) to Creep Slope (mm/Pass)
Control 1	NA	NA	NA	NA
Control 2	NA	NA	NA	NA
Control 3	NA	NA	NA	NA
#8-1	NA	NA	NA	NA
#8-2	15127	3106	1377	2.25
#8-3	14143	4274	2058	2.08
#16-1	16464	4525	683	6.63
#16-2	15807	4219	1776	2.38
#16-3	15110	4739	1996	2.37
#30-1	15272	3226	1337	2.41
#30-2	NA	NA	NA	NA
#30-3	NA	NA	NA	NA

Table 5 Number of Passes at SIP, creep slope, stripping slope and stripping-creep slope ratio from Hamburg wheel-track tests

3.3.3.2 Rutting resistance

The rut depth at 5000th, 10000th and 15000th pass as shown in Figure 23 were used to evaluate the resistance of asphalt mixture to rutting, i.e., permanent deformation. The asphalt mixture that has a lower rut depth at a specific point has a better rutting resistance. The addition of CaCl₂-zeolite/p-epoxy improved the rutting resistance, whatever the number of passes and the particle size were. The WSDOT requires the rut depth of asphalt mixture to be less than 10 mm at 15000 passes (WSDOT 2018). Thus, all the samples passed the WSDOT specification. At the 5000th pass, the rut depth decreased by 34%, 36% and 17% for sample #8, #16, and #30 respectively. At the 10000th pass, the rut depth decreased by 32%, 33% and 13% for sample #8, #16, and #30 respectively; and at the 15000th pass, the rut depth decreased by 28%, 27% and 8% for sample #8, #16, and #30 respectively. This suggests that additives #8 and #16 had the similar effect on the rutting resistance of the asphalt mixture and were better than additive #30. Moreover, all the tests stopped after 20000 passes as shown in Figure 21, which indicates the high rutting resistance of the asphalt mixtures.

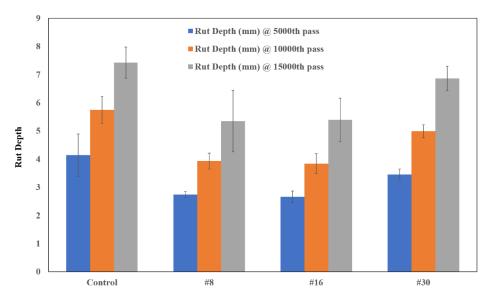


Figure 23 Rut depth at 5000th, 10000th and 15000th pass from Hamburg wheeltrack tests

3.3.4 Mid-temperature and low-temperature cracking resistances

IDT strength and fracture work density (FWD) evaluated the mid-temperature and lowtemperature cracking resistances of asphalt mixture. IDT strength was calculated using Equation 3:

$$S_t = \frac{2000 \times P}{\pi \times t \times D} \tag{3}$$

Where $S_t = IDT$ strength in kPa; P = maximum load in N; t = specimen height immediately before test in mm; and D = specimen diameter in mm. The FWD was calculated using Equation 4:

$$FWD = \frac{W_f}{V} \times 10^6 \tag{4}$$

Where FWD = fracture work density in kPa; W_f = fracture work in N ·m, which was the area under a load-deformation curve; and V = specimen volume in mm³.

3.3.4.1 Mid-temperature cracking resistance

Figure 24 presents the IDT strength and the FWD of asphalt mixture at 25 °C. The addition of CaCl₂-zeolite/p-epoxy slightly improved both the IDT strength and the FWD. The IDT strength increased from 733 kPa for control sample to 852 kPa, 789 kPa and 819 kPa for the sample with CaCl₂-zeolite/p-epoxy #8, #16, and #30 respectively. The FWD increased from 61 kPa for control sample to 63 kPa, 67 kPa and 64 kPa for the sample with CaCl₂-zeolite/p-epoxy #8, #16, and 64 kPa for the sample with CaCl₂-zeolite/p-epoxy #8, #16, and 64 kPa for the sample with CaCl₂-zeolite/p-epoxy #8, #16, and 64 kPa for the sample with CaCl₂-zeolite/p-epoxy #8, #16, and #30 respectively. These increases suggest that the addition of CaCl₂-zeolite/p-epoxy enhanced the mid-temperature cracking resistance, regardless of the particle size of the salt-storage additive. On the other hand, the WSDOT requires the IDT strength of asphalt mixture to be greater than 1207 kPa (175 psi) at 25 °C (WSDOT 2018). Therefore, the IDT strength of

asphalt mixture with CaCl₂-zeolite/p-epoxy needs to be improved in future research, likely by incorporating another type of additive.

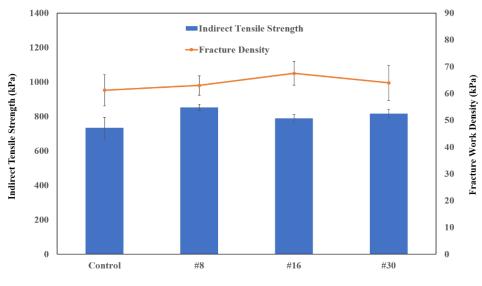


Figure 24 IDT strength and FWD from IDT tests at 25 °C

3.3.4.2 Low-temperature cracking resistance

Figure 25 presents the IDT strength and the FWD of asphalt mixture at -10 °C. Like the results at 25 °C, both the IDT strength and the FWD improved slightly by the addition of CaCl₂-zeolite/p-epoxy, except additive #8. The IDT strength increased from 3631 kPa for control sample to 3648 kPa, 3762 kPa and 3897 kPa for the sample with CaCl₂-zeolite/p-epoxy #8, #16, and #30 respectively. The FWD decreased negligibly from 40 kPa for control sample to 39 kPa for CaCl₂-zeolite/p-epoxy #8 but increased to 44 kPa and 46 kPa for the sample with CaCl₂-zeolite/p-epoxy #16 and #30 respectively. These data suggest that the low-temperature (thermal) cracking resistance improves with the addition of CaCl₂-zeolite/p-epoxy except in the case of additive #8.

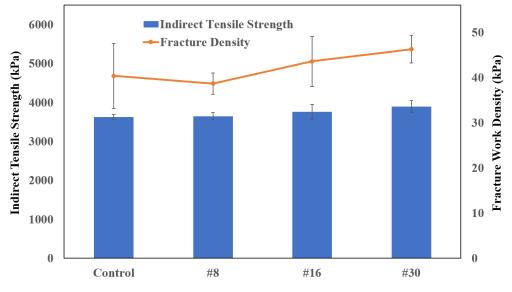


Figure 25 IDT strength and FWD from the IDT test at -10 °C

3.4 Summary and Conclusions

In this chapter, the researchers developed innovative salt-storage additives with a sustained salt-release rate for anti-icing asphalt pavement. The laboratory-prepared additives had three different gradations, including sieve #4 - #8, sieve #8 - #16 and sieve #16 - #30. The CaCl₂ content in additives #8, #16, and #30 was 20.1%, 20.5%, and 21.1% respectively, determined based on the mass difference between the additives with and without CaCl₂. Three types of asphalt mixtures including samples with CaCl₂-zeolite/p-epoxy, samples with CaCl₂-zeolite, and control samples were prepared to evaluate the anti-icing performances and the mechanical properties of anti-icing pavements.

The anti-icing capacity of asphalt mixture was evaluated by fog-freezing friction test at - 3.9 °C and -9.4 °C. At -3.9 °C, the friction coefficient of control samples (which was 1.0) was slightly greater than that of samples with the additives at the beginning of the test. Meanwhile, all the samples with the additives exhibited a friction coefficient greater than 0.80. However, after moisture spray, the friction coefficient of control samples reduced to a value less than 0.3 at

15, 30, 45, and 60 min, while all the friction coefficients of samples with the additives were greater than 0.55 at different times. CaCl₂-zeolite/p-epoxy exhibited a similar anti-icing capacity with CaCl₂-zeolite at 60 min, since the friction coefficients were around 0.75 for all the samples with either additive. Samples at -9.4 °C had similar behaviors in friction coefficient with those at -3.9 °C, excepting samples with CaCl₂-zeolite/p-epoxy. The friction coefficient of samples with CaCl₂-zeolite/p-epoxy #30 gradually increased from 0.4 at 15 min to 0.55 at 60 min, which was significantly greater than that of control samples, although reduced compared with the values at -3.9 °C. At both temperatures, the friction coefficient of control samples decreased continuously, whereas the friction coefficients of samples with the additives showed an increasing trend. Throughout the time after moisture spray, CaCl₂-zeolite/p-epoxy #30 showed a better anti-icing capability, compared to CaCl₂-zeolite/p-epoxy #8 and #16 at both testing temperatures.

Under simulated conditions, the estimated effective anti-icing period of asphalt pavement with additives #8, #16, and #30 were 5.8 years, 9.9 years and 15.3 years respectively, as evaluated by anti-icing longevity test. The testing results demonstrated a strong potential for using the laboratory-prepared salt-storage additives in asphalt pavement for snow and ice control.

The mechanical properties of asphalt mixture were slightly influenced by the addition of CaCl₂-zeolite/p-epoxy. The effect of CaCl₂-zeolite/p-epoxy on the reduction of the moisture damage resistance was negligible. Almost all the samples passed the WSDOT specification (except sample #8-3 that had 14143 passes at SIP) as well as the Wisconsin and Iowa specifications. The rutting resistance, mid-temperature cracking resistance, and low-temperature cracking resistance of asphalt mixture improved with the addition of CaCl₂-zeolite/p-epoxy. The

rutting resistance was higher but the mid-temperature cracking resistance of asphalt mixture with CaCl₂-zeolite/p-epoxy was lower than the acceptable limit based on the WSDOT specification.

CHAPTER 4.0 CONCLUSION AND RECOMMENDATIONS

4.1 Introduction

Anti-icing asphalt pavement through salt-storage additives is an effective strategy for winter road maintenance. However, this strategy still faces multiple challenges in real practice, such as the longevity of anti-icing ability, low-temperature effectiveness, as well as potentially adverse impacts to the mechanical properties of asphalt pavement. The innovative laboratoryprepared salt-storage additives developed and blended by the researchers into asphalt pavement made a novel type of anti-icing pavement. The anti-icing performance and mechanical properties of asphalt mixture with the obtained additives were evaluated in the laboratory.

4.2 Summary and conclusions

The fog-freezing friction test of asphalt pavement validated the effectives of the prepared salt-storage additives for anti-icing. At -3.9 °C (25°F), the friction coefficient of control samples (1.0) was slightly greater than that of pavement samples with the additives at the beginning of the test. Meanwhile, the asphalt pavement samples with the additives exhibited friction coefficients greater than 0.80. However, after moisture spray, the friction coefficients of control samples reduced to values less than 0.3 at 15, 30, 45, and 60 min, respectively, whereas the friction coefficients of the samples with the additives were greater than 0.55 at the different times. The CaCl₂-zeolite/p-epoxy exhibited a similar anti-icing capacity with CaCl₂-zeolite at 60 min, since the friction coefficients were around 0.75 for all the samples with either additive.

Pavement samples at -9.4 °C ($15^{\circ}F$) exhibited similar behaviors in friction coefficients to those at -3.9 °C ($25^{\circ}F$), the only exceptions were the samples with CaCl₂-zeolite/p-epoxy. The friction coefficient of samples with CaCl₂-zeolite/p-epoxy #30 gradually increased from 0.4 at 15

min to 0.55 at 60 min, significantly greater than that of control samples, although it was greatly reduced compared with the values at -3.9 °C. At both temperatures, the friction coefficient of control samples decreased continuously, whereas the friction coefficients of samples with the additives showed an increasing trend. Throughout the time after moisture spray, CaCl₂-zeolite/p-epoxy #30 showed a better anti-icing performance, relative to CaCl₂-zeolite/p-epoxy #8 and #16 at both testing temperatures.

Under simulated conditions, the estimated effective anti-icing period of asphalt pavement with additives #8, #16, and #30 were 5.8 years, 9.9 years and 15.3 years respectively, as evaluated by an anti-icing longevity test. The testing results showed a strong potential for using the laboratory-prepared salt-storage additives in asphalt pavement for snow and ice control.

The mechanical properties of asphalt mixture were slightly influenced by the addition of CaCl₂-zeolite/p-epoxy. The effect of CaCl₂-zeolite/p-epoxy on the reduction of the moisture damage resistance was negligible. It is worth noting that all the samples passed the WSDOT specification (except sample #8-3 that had 14,143 passes at SIP) as well as the Wisconsin and Iowa specifications. The rutting resistance, mid-temperature cracking resistance, and low-temperature cracking resistance of asphalt mixture benefited by the addition of CaCl₂-zeolite/p-epoxy. The rutting resistance was higher but the mid-temperature cracking resistance of asphalt mixture with CaCl₂-zeolite/p-epoxy was lower than the acceptable limit based on the WSDOT specification.

The researchers concluded that the innovative laboratory-prepared additives had positive effects on the properties of anti-icing, rutting resistance, mid-temperature and low-temperature cracking resistance of asphalt pavement and had negligible impacts on the moisture damage resistance of asphalt pavement. It is necessary to use advanced materials (e.g., carbon fiber) to

improve the mid-temperature cracking resistance of asphalt pavement with the additives because the asphalt mixture failed to pass the WDOT specification.

4.3 Directions for future research

Further investigations into the asphalt pavement with these innovative laboratoryprepared additives should focus on improving the engineering performances (especially midtemperature cracking resistance and moisture resistance) of the asphalt mixture and exploring the anti-icing and degradation mechanisms caused by CaCl₂ at a micro- and nano- scales.

CHAPTER 5.0 REFERENCES

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