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Impact of nanoclay and carbon microfiber in combating the deterioration of asphalt concrete by non-chloride deicers

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- The stripping risk of asphalt concrete was assessed in presence of four non-chloride deicers.
- The incorporation of nanoclay (0.6–1.8% by mass of bitumen) produced a notably improved stripping resistance.
- The addition of carbon microfiber (1% by mass of bitumen) increased moisture susceptibility.
- The impact of non-chloride deicers on asphalt mastic was discussed based on their hydrophilic-lipophilic properties.

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This preliminary laboratory research aims to explore the impact of nanoclay and carbon microfiber in combating the deterioration of asphalt concrete by non-chloride deicers. Modified boiling water test, electrochemical impedance spectroscopy (EIS) and contact angle test were employed to investigate the effects of nanoclay and carbon microfiber on asphalt concrete in the presence of four non-chloride deicers, namely, dipotassium succinate, potassium formate, potassium propionate and potassium acetate. The results indicated that the incorporation of small amount of nanoclay in asphalt concrete produced a notably improved stripping resistance and reduced moisture susceptibility, while the addition of carbon microfiber increased moisture susceptibility. The deterioration effect of the four non-chloride deicers on asphalt mastic was discussed based on their molecule's hydrophilic-lipophilic properties.

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1. Introduction

In cold-climate regions, snow and ice control operations are of critical importance to winter roadway's safety and mobility. Large amounts of solid and/or liquid deicers as well as abrasives are applied to keep winter roadways clear of ice and snow and/or to provide them with a surface layer of good friction. Chloridebased chemicals such as sodium chloride (NaCl), magnesium chloride (MgCl₂) and calcium chloride (CaCl₂) are the most widely used deicers as freezing point depressants for winter road maintenance applications, due to their abundance and relative low cost $[1-3]$. However, growing concerns have been raised about high residual chloride levels and their adverse impacts on motor vehicles, transportation infrastructure and ecosystem [\[4–7\].](#page--1-0) In recent years, non-chloride deicers including mainly acetates (potassium acetate,

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sodium acetate, and calcium magnesium acetate), formates (potassium formate and sodium formate), succinates (potassium succinate), urea, glycerol/glycol and bio-based products gained much attention from winter maintenance agencies, because they tend to decompose quickly and thus pose less long-term environmental risk than chloride-based deicers [\[8–14\]](#page--1-0).

The deleterious effects of deicers on Portland cement concrete (PCC) and their corrosion to metals in transportation infrastructure are well documented $[8]$. In contrast, few studies were conducted to investigate the possible impacts of deicers, in particular, nonchloride deicers on asphalt pavement, likely due to the high chemical resistance of asphalt binder to chlorides. Such an unproven property, however, may have simply arisen from the fact that an asphalt pad is usually built as the storage bottom for various chloride-based highway deicers to prevent leakage [\[15\].](#page--1-0) Nonetheless, research dedicated to the interactions between asphalt and deicers has reported the loss of skid resistance [\[16\]](#page--1-0) and the damage within the asphalt pavement structure among other side effects of deicers $[10,17]$. It has been observed that damage to the asphalt mixture in the presence of deicers was typically greater

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than that in the presence of only water, especially when the asphalt pavement underwent freeze-thaw cycles [\[2,17\]](#page--1-0). The deicers seemed to reduce the strength and elasticity of asphalt mixture, exacerbating the freeze-thaw damage [\[18\]](#page--1-0). Premature deterioration of asphalt pavement due to the use of acetate/ formate-based deicers was found at some Nodic airports. Typical symptoms include degradation and disintegration of asphalt pavement, softening of asphalt binders, and stripping of asphalt mixes occurring together with loose aggregates on the runways [\[14,19\].](#page--1-0) A laboratory investigation also confirmed that the use of acetate/ formate-based deicers could accelerate the degradation of asphalt mixture through emulsification of the binder [\[11\].](#page--1-0) In light of the widespread use of deicers on asphalt pavements, in particular a preference for non-chloride deicers, a study of potential mitigation measures is essential.

Nanomodification could be a promising approach to enhancing the durability and global performance of asphalt mixtures [\[20–](#page--1-0) [22\]](#page--1-0). Nanoclays are nanoparticles of layered mineral silicates. They are widely used as functional modifiers for polymers to achieve remarkable enhancement in mechanical, thermal and barrier properties. Owing to their unique layered structure and variable chemical composition as well as large active surface area (700-800 m 2 /g), nanoclays have shown great promise in modifying asphalt mixtures [\[23\].](#page--1-0) Ghile [\[24\]](#page--1-0) demonstrated the benefits of nanoclay modification on the rheology of bitumen in addition to improved rutting resistance and fatigue mitigation of asphalt mixture. You [\[20\]](#page--1-0) suggested that if properly distributed, nanoclays could be an effective modifier to improve the mechanical properties of asphalt binders. The blending of two types of nanoclays at 4% by weight of asphalt was found to increase the complex shear moduli of the binder by 125% (for type A) and 196% (for type B), respectively $[20]$. Such nanomodification reduced the strain failure rate of the binder but increased its secant or direct tension moduli. Moreover, it was reported that the melt blending of sodium-montmorillonite (Na-MMT) or organophilic MMT (OMMT) into styrene-butadienestyrene (SBS) copolymermodified asphalt notably increased the softening point and viscosity of the asphalt and improved its aging resistance [\[25\]](#page--1-0). The nanomodified asphalt featured enhanced viscoelastic properties in terms of a higher complex modulus and lower phase angle, implying stiffer and more elastic asphalt. Thus, the nanoclay-modified asphalt exhibited better rutting resistance at high temperatures than the unmodified or SBS-modified asphalt. A further investigation revealed that the Na-MMT modified asphalt formed an intercalated structure, while the asphalt modified by OMMT featured an exfoliated structure [\[25,26\].](#page--1-0)

In the past decades, extensive research effort has been invested on the use of various natural or synthetic micro-sized fibers in cement-based materials, as fibers feature strain-hardening behavior associated with the enhancement of tensile/flexural strength and fracture toughness [\[27–29\]](#page--1-0). However, there are few studies on the use of carbon microfibers to reinforce asphalt pavement. In this context, there is also the need to explore the combined use of and potential synergism between micro- and nano-sized modifiers in improving the properties of asphalt materials, especially when they are exposed to deicer solutions. The objectives of this research are to modify an asphalt concrete with two materials: nanoclay and carbon microfiber and to examine the effects of the two modifiers in combating the deterioration of asphalt concrete (AC) by non-chloride deicers at various concentrations.

2. Experimental

2.1. Materials and mix design

The asphalt binder used to fabricate hot-mix AC mixtures in this research was a PG 58-28 binder. The asphalt binder content was set as 6% by weight, based on the air void requirements at 9 and 150 gyrations in the Superpave gyratory compactor. The aggregates, which were supplied by the Knife River Corporation in Belgrade, MT, consisted of 44% coarse aggregates (granites), 46% crushed fines (granites), and 10% concrete sand (siliceous sand) by weight. The combined aggregate gradation is shown in Fig. 1. The nanoclay used in this study was a polysiloxane-modified montmorillonite, with bulk density of 0.251 $g/cm³$ and aspect ratio of 200–400. The nanoclay was added at three dosages: 0.6%, 1.2% and 1.8% by mass of bitumen. The carbon microfiber was KRECA chop C-103T, 3 mm in length with a filament diameter of 18 µm, as obtained from Kureha (Tokyo, Japan), featuring a tensile intensity of 670 MPa and tensile elastic modulus of 30 GPa. The properties of the nanoclay and carbon microfiber can be found elsewhere [\[23,29\]](#page--1-0). The carbon microfiber content in the asphalt mixtures was 1% by mass of bitumen. The upper limits of nanoclay and microfiber dosage were chosen based on the fact that too much of them would cause poor dispersion and on our previous finding [\[35\]](#page--1-0) that nanoclay and carbon microfiber "(both at less than 2% by weight of asphalt binder) can enhance the tensile strength and fracture energy of asphalt concrete mixtures and reduce their moisture susceptibility and cracking risk", particularly in presence of chloride-based deicer solutions.

2.2. Modified boiling water test

The modified boiling water test was developed based on the ASTM standard method: standard practice for effect of water on bituminous-coated aggregate using boiling water [\[30\].](#page--1-0) The D3625-96 is normally used to study the moisture sensitivity of asphalt mixtures. It entails an accelerated procedure for visual registration of the loss of adhesion in asphalt concrete specimens subjected to the action of boiling water. In this modified boiling water test, a boiling deicer solution was used instead of boiling water. Four non-chloride deicers, namely, dipotassium succinate (DS), potassium formate (PF), potassium propionate (PP) and potassium acetate (PA) were tested. Each deicer aqueous solution was prepared at four mass concentration levels, 5%, 10%, 20%, and 40%. These deicer concentration levels were chosen based on the fact that PA is typically applied as a 50 wt% aqueous solution and there may be a certain level of dilution by snow and ice on the pavement. To examine the complicated interactions between various modified asphalt mix designs and deicer compositions, we constructed and followed a type of statistical experimental design to reduce the number of experiments needed for exploration of a relatively large domain of unknown factors and their potential interactions. Specifically, 16 combinations were run and replicated, thus totaling 32 runs. The 16 combinations represent a design scheme following the Taguchi orthogonal array L_{16} (4^5) to investigate the effect of three factors (each at four levels) on the percentage of stripping. The three factors denoted as A, B and C, each varying at four levels (1–4) are: A = type of deicer; $1 = PF$; $2 = PA$; $3 = PP$; $4 = DS$. $B =$ deicer mass concentration; $1 = 5\%$, $2 =$ 10%, 3 = 20%, 4 = 40%; C = dosage of nanoclay; 1 = 0%, 2 = 0.6%, 3 = 1.2%, 4 = 1.8% by mass of bitumen. The actual design of 16 combinations is shown in [Table 1,](#page--1-0) of which the results will be discussed in the following sections.

Prior to mixing, the asphalt binder, carbon microfiber and nanoclay were heated separately in an oven at 230 F (110 °C) for 8 min, while the aggregate was heated at the same temperature for 30 min. This lower-than-usual temperature (110 \degree C) was chosen to simulate poor construction practice that results in potentially insufficient adhesion between aggregate and binder. To improve distribution homogeneity, the asphalt binder, carbon microfiber and nanoclay were mixed by a high-shear mixer for 1 min before putting them back to the oven and heated for another 2 min. The heated aggregate was then poured into the asphalt mixture and mixed quickly by hand. The resulting mixture was subsequently heated at the same temperature for another 3 min and manually mixed again to achieve a uniform coating of aggregates by the asphalt. Then the hot mixture was poured immediately into a wood box with an inner length of 2.5 in (63.5 mm) and inner width of 3 in (76.2 mm). A material testing system was utilized to give a constant pressure of 200 psi (1.4 MPa and about 1500 lbf) on the asphalt-aggregate mixture for 30 min in order to

Fig. 1. Gradation of aggregates used to fabricate the asphalt concrete.

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