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MEASURING THE EFFECT OF DE-ICING SOLUTIONS ON THE ADHESION OF ASPHALT MIXTURES

ABSTRACT

De-icing chemicals are routinely used to improve the level of tire-pavement friction under cold weather conditions. However, their detrimental effect not only on vehicles and environment but also infrastructure has been recognized and documented by many studies. A time demanding testing method, based on the determination of the surface tensile strength of an asphalt sample after storage in de-icing fluid, is generally used to evaluate the deteriorative effect of the chemicals.

This study aims evaluate if it is possible to estimate the resistance to de-icing fluids of asphalt mixture through testing methods currently used to evaluate moisture sensitivity. The de-icing damage will hence be expressed in terms of adhesion of the bitumen-aggregate system evaluated as tensile strength of compacted specimens (measure with the indirect tensile strength, ITS, test) and of the degree of coverage of bitumen coated aggregates (rolling bottle test) after storage in the deicing solution. Four different mixtures will be immersed in fully saturated solutions containing sodium chloride and potassium formate.

A worsening of both tensile strength and degree of coverage of the aggregates are expected because the high storage temperature suggested in the ITS test that will enhance chemical reaction between the bitumen and the de-icer, and because of the infiltration of the chemical in the interface between bitumen and aggregate in rolling bottle test.

Keywords

De-icing Fluids, Adhesion, Sensitivity

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INTRODUCTION

In the last decades, the growing use of de-icers has raised concerns over their impact on the environment, vehicles and infrastructure. While a large amount of information is available on their performance as both preventive and reactive measure, de-icers' detrimental effect on asphalt pavements is often considered a secondary aspect (Akin and Shi, 2010). Although several test methods have been developed, they generally extend the investigation to the combined effect of de-icers and freeze-thaw cycles, therefore including freeze-thaw cycles' effects on the stiffness of the binder and the cohesion of the mixture (Hassan et al., 2002, Starck and Löfgren, 2007, Goh et al., 2011, Shi et al., 2009). Moreover, low temperatures decrease the rate of chemical reactions preventing any effect of the de-icing solution on the mixture.

In order to evaluate the effects of de-icers on asphalt pavements, the European Committee for Standardization recommends the use of the the European standard EN 12697-41 (Bituminous mixtures – Test methods for hot mix asphalt – Part 41: Resistance to de-icing fluids). The test consists in evaluating the surface tensile strength of a set of asphalt samples after storage in de-icing fluid. The asphalt samples, cored or laboratory prepared, are bonded to a metallic cylinder with a thin layer of epoxy adhesive and afterwards stored at 40°C for 70 days immersed in the de-icing fluid. After conditioning, the cylinder is pulled off with an increasing tensile force and the tensile force at failure and the mode of failure are recorded and compared it to the tensile force at failure of specimens stored in air (Figure 1).

Although the test is commonly used in the Nordic countries to evaluate the asphalt concrete resistance to de-icing chemicals, few studies investigated its performance.

Ekblad and Edwards (2008) report the results of a round-robin test with seven international laboratories participating whose purpose was to evaluate the precision of the method. Although the standard indicates the surface strength at failure should be reported to the nearest 0.1 N/mm², the standard deviation evaluated after the round-robin test for repeatability and reproducibility was reported to be respectively 130 N and 220 N. The analysis of the results indicated that although the method discriminates between harsh and neutral de-icing chemicals, it poorly distinguishes small differences between de-icers. Moreover the same study suggests a decrease of the test precision with an increase of the ratio between the largest particles in the mix and the volume or area tested.

The type of failure (superficial, deep within the asphalt specimen or de-bonding from the glue) also significantly affects the results. Figure 1b shows asphalt samples tested after storage in de-icing fluids. The three different failure types can be observed with a high percentage of the “de-bonding from the glue” type. Being these samples not considered in evaluation of the tensile strength of the mixture the standard advises to repeat the test until

“until the mean of both the dry and wet groups are the average of not less than four determinations” (CEN, 2003). Reduced number of samples because of the failure type was also experienced during the round-robin test (Ekblad and Edwards, 2008).

Inadequate preparation, storage or handling of the samples and effect of the chemicals on the adhesion between the glue and the cylinder are among the causes of de-bonding from the epoxy adhesive. Even though no specific study has analysed the degree of occurrence of the different failure modes, the large costs connected long conditioning time and possible limited number of samples, the likelihood of the de-bonding from the glue failure type, and the poor repeatability and reproducibility suggest the need of a more practical method.

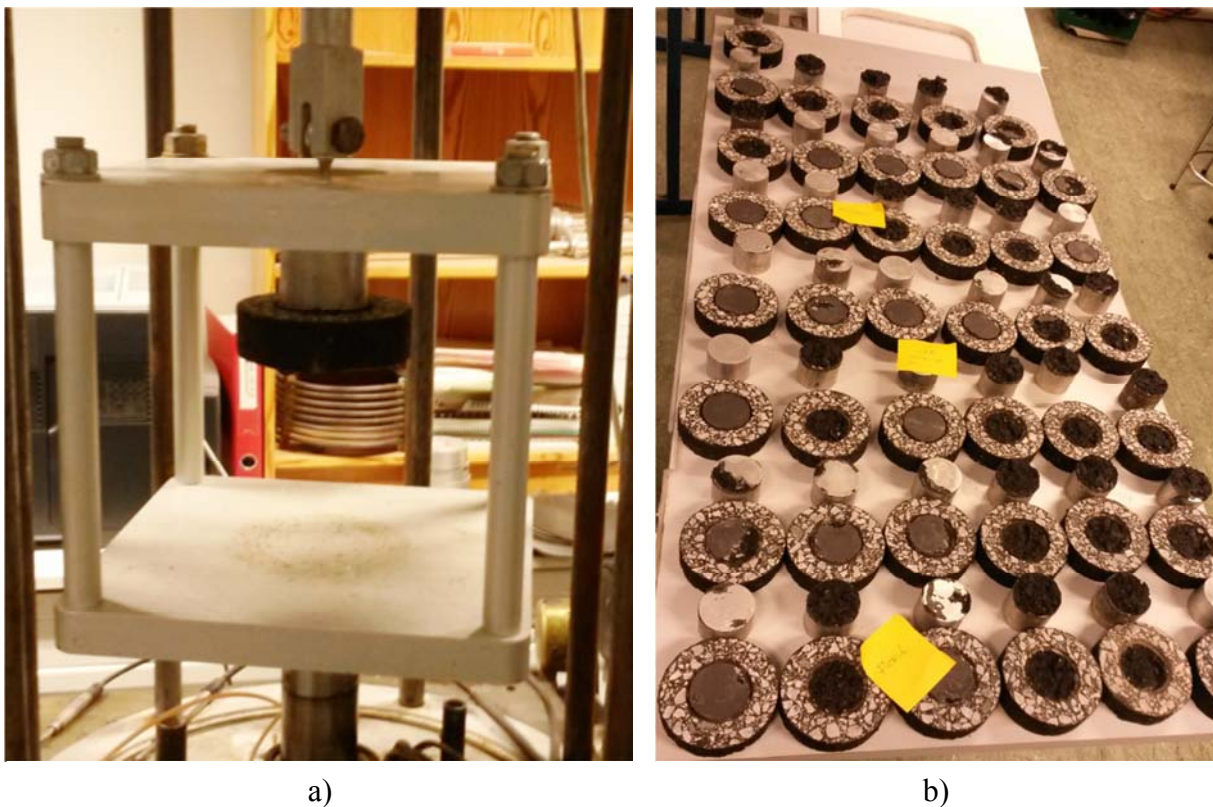


Figure 1 De-icing test: sample before testing attached to the tensile test machine (a); samples after testing with the different failure mode visible (b).

Loose aggregates and aggregates from which the bitumen has been washed away, and loss of strength were found to be among the damaging effects of different de-icers (Alatypö and Valtonen, 2007, Kalevi and Salminen, 2011, Shi et al., 2009). Above all loose particles were found to be particularly dangerous for flight safety on airport runways (Starck and Löfgren, 2007). These damages, consisting in adhesive and cohesive failures, suggest a possible use of test methods intended for moisture damage.

This study aims therefore to evaluate if test methods used to evaluate water sensitivity of asphalt mixtures, such as the rolling bottle and the indirect tensile strength (ITS) test can also identify the effects of de-icing solutions.

EXPERIMENTAL PROGRAM

For this study, four dense graded AC 11 mixtures (hereinafter A a metasandstone, B a metagabbro, C a rhyolite and D a greywacke) with different aggregate types but same filler and binder, were designed according to the Norwegian Pavement Design Guide (NPRA, 2014). The samples, prepared according to the respective European standard (EN 12697-11 and EN 12697-12), were immersed in solutions prepared with distilled water and conditioned according to the test method.

Two types of de-icers, sodium chloride (NaCl) and potassium formate (KCHO₂), used respectively on road and runways, were chosen for the study. Since both the rolling bottle and the ITS test include different temperatures between the preparation/conditioning phase and the test and since potassium formate solubility in water highly varies with temperature, the same molar concentration, corresponding to a saturated water-NaCl solution, was used for both de-icers. This choice also allows the same damaging potential to both the solutions being the same amount of the de-icer molecules dispersed in water.

Rolling bottle test

The rolling bottle test is commonly used to determine the affinity between aggregates and binder, and to evaluate the effect of moisture on the binder-aggregate combination. The affinity is determined as visual observation of the degree of coverage of bitumen coated aggregates subjected to a mechanical stirring action. The test does not require earlier conditioning and the observations are taken after 6, 24, 48 and 72 hours from the beginning of the test. However, the Norwegian Pavement Design Guide recommends 25% as minimum degree of coverage of the aggregates after 48 hours (NPRA, 2014).

Although the test is highly dependent on the observers, the results are considered valuable indication of the long term performance of the bitumen-aggregate combination in presence of moisture.

Indirect tensile strength test

The ITS test is used to evaluate water susceptibility of asphalt mixture. Differently from the rolling bottle procedure this test is carried out on compacted mixtures. The indirect tensile strength of at least three samples is measured per each conditioning type. The samples are

subjected to accelerate conditioning in the solution at 40°C for 72 hours followed by two hours conditioning, still in the water bath, at the testing temperature, 25°C.

In this study, an additional set of control samples was stored in water and conditioned as the samples in solution.

ANALYSIS OF THE RESULTS

The results of the ITS test are given in Figure 2. The average indirect tensile strength of each set and their standard deviation do not show particular differences in the results except for the samples D conditioned in sodium chloride. Although the difference of the average bulk densities of the sets did not exceed 30 kg/m^3 , the mixture behaviour could have been caused by the average lower air voids content (0.5% less than the other D sets). In all cases water seems to affect the cohesion of the mixture more than the de-icing solutions. The general worse performance of the mixture containing metasandstone aggregates (A) compared to the others should be attributed to the high sensitivity to water of the rock type (Hicks, 1991).

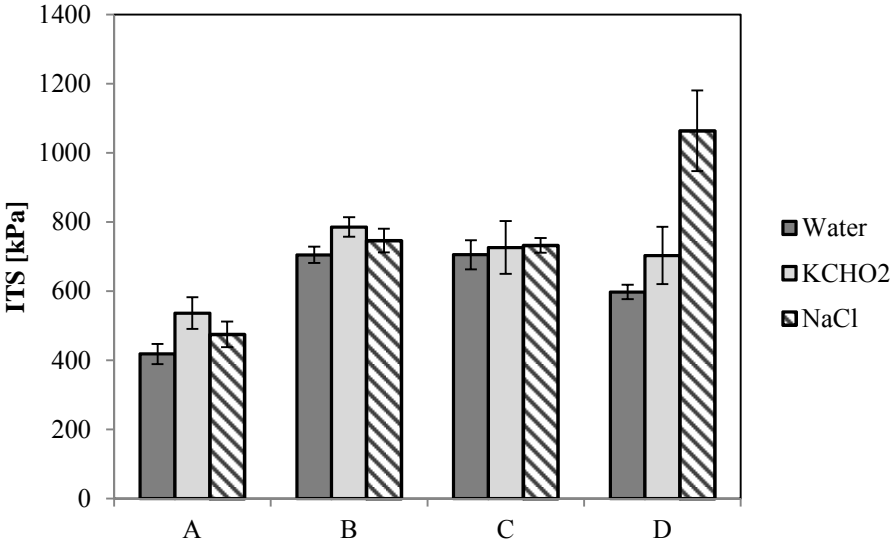


Figure 2 Maximum indirect tensile strength of the four mixtures (A, B, C and D), after conditioning in dry conditions, in water, in a potassium formate and in a sodium chloride solution. The standard deviation of the results is represented by the error bars.

The rolling bottle test, used to evaluate the affinity between aggregates and bitumen, was also used to determine the impact of de-icers on the aggregate-bitumen system. Figure 3 shows the loss of adhesion between the two materials at different intervals when immersed in different solutions. It is possible to distinguish between the B-D group, with a degree of coverage higher than 40% after 72 hours (except the samples immersed in water) and the A-C group, whose coverage at the end of the test is lower than 20%. Very small differences are visible in A, C and D between the samples immersed in the de-icers. The mixture C also appears to be the only one whose performance worsened when de-icing agents were added to the mixture.

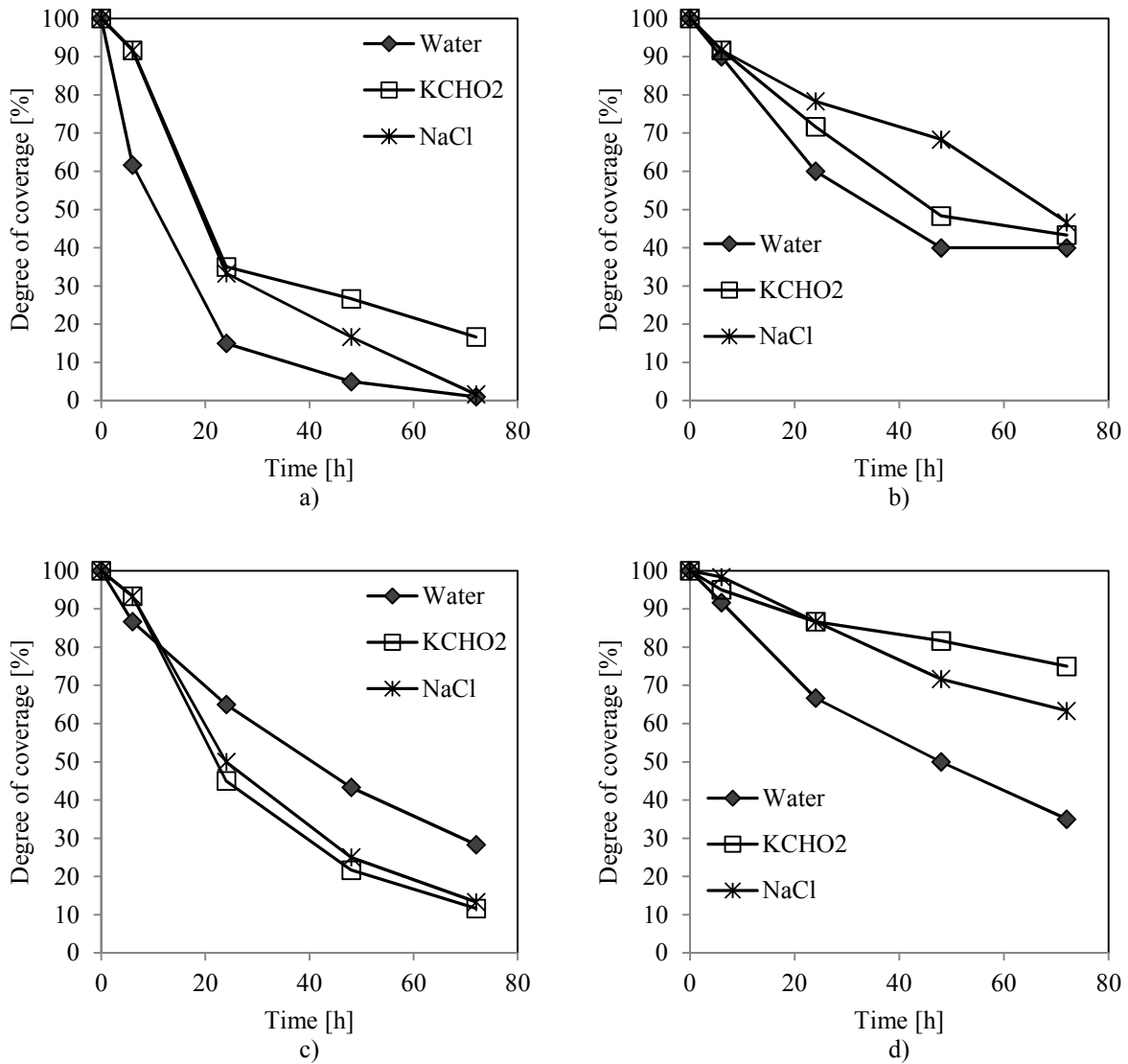


Figure 3 Degree of coverage of the aggregates after 24, 48 and 72 hours for A, B, C and D (a, b, c, and d respectively) during immersion in water, a potassium formate and a sodium chloride solution.

In order to evaluate if the test methods can distinguish between the different solutions, a statistical analysis was performed to test whether the group population means are all equal or not. Since the observations are based on several set of measurements, the analysis of variance (ANOVA) test at 95% confidence level was preferred to a multiple t-tests to reduce the probability of Type 1 errors.

A summary of the statistical analysis is reported in Table 1 divided depending on mixture and test type. The results considered for the rolling bottle test (RB in the table) corresponds to the observations after 48 hours as recommended by the Norwegian Pavement Design Guide.

According to ANOVA, there is no difference between the conditioning solutions when the samples are tested with the ITS test. Significant differences occur for the D mixture when conditioned in sodium chloride compared to water or potassium formate (Table 2).

When the rolling bottle results are analysed, significant differences are observed and confirmed in almost all cases by the Fisher's least significant difference (LSD) post-hoc analysis, suggesting that the de-icers affect the stripping resistance of the mixture.

Table 1 ANOVA test for significant effects with $\alpha=0.05$.

	A		B		C		D	
	F-statistic	p-value	F-statistic	p-value	F-statistic	p-value	F-statistic	p-value
ITS	4.91	0.055	3.87	0.083	0.148	0.87	17.13	0.003
RB (48h)	25.40	0.001	114.50	<0.001	29.40	0.001	141.50	<0.001

Table 2 LSD post-hoc analysis. At the bottom left (in italic) the mixtures (A, B, C or D) whose test results were statistically different depending on the conditioning solution according to the ITS test, at the top right according to the rolling bottle test, after 48hours.

	Water	KCHO2	NaCl
Water	-	A, B, C, D	A, B, C, D
KCHO2		-	A, B, D
NaCl	<i>D</i>	<i>D</i>	-

CONCLUSIONS

The study presented in this paper aimed to evaluate the possibility to use alternative test methods to evaluate asphalt mixture resistance to de-icing fluids than the EN 12697-4. Although the European standard is commonly used in the Nordic countries to estimate the effect of chemicals on the pavement surface, several factors such as its scarce repeatability and cost, suggest the need of an easier test method. Since previous studies suggest loss of cohesion and adhesion as main damage mechanisms, this study tried to evaluate if the ITS and rolling bottle test, generally used to measure moisture damage, can identify the effects of de-icing chemicals. Potassium formate and sodium chloride were chosen for the study.

Both tests, carried out on four different mixtures, suggest a lower damage potential of the de-icing solutions compared to water. Although small differences in the average result are visible, the ITS test does not seem to be able to recognize the different types of conditioning. Conversely the rolling bottle test appears to clearly identify the presence of the de-icers showing a clear improvement of the performance for three out of four mixtures with very small differences between $KCHO_2$ and $NaCl$. These results are confirmed by the statistical analysis.

These tests provide useful information on the effects of de-icers. They seem to positively affect the adhesion between aggregate and bitumen reducing the stripping potential of the mixture. This effect could be due to the different surface tension of the solution or to the larger size of the water molecule when it bonds with de-icing chemical. Affecting the first the wettability of the aggregate by the solution and the second the spontaneous emulsification of the water within the bitumen layer. The lack of effects on the cohesion of the mixture, evaluated with the ITS test, could suggest that the high conditioning temperature (40°C) affects the results more than the solution itself.

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