

## EVALUATION OF LABORATORY PERFORMANCE OF ZYCOTHERM ADDITIVE FOR EXTENDED MOISTURE RESISTANCE OF HMA PAVEMENTS

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### Abstract

Moisture induced damage is one of the major distresses in asphalt concrete pavements. The failures occur when the asphalt binder strip off from the aggregate due primarily to the action of moisture, and thereby reducing the strength of asphalt concrete. In this study, the performance of ZycoTherm® -a nano-organosilane additive- was evaluated on various asphalt mixtures for moisture damage and stripping. The samples were prepared according to the Marshall mixture design method, using two types of basalt aggregates with and without ZycoTherm® additive. After the mixture design, a number of performance tests were conducted i.e., stripping, bottle rolling and boil tests. The study program mainly focused on testing the improvement of binder-aggregate bonding by ZycoTherm®, so that tests were repeated at extended conditioning times to compare with the normal (standard) testing conditions. Moreover, specimens were also tested in the sodium chloride (salt) solution to simulate the detrimental effect of de-icing agents on bitumen-aggregate bonding. In these tests, ZycoTherm® was evaluated for each aggregate type based on four test combinations as: normal condition, in 4% salt solution, normal condition with additive, with additive in 4% salt solution. In the Nicholson tests, the results showed that, the nano-organosilane additive significantly improved the stripping resistance of the mixtures of both aggregates at all extended conditioning times. The results were even better in the salt solution, almost no resistance loss for the mixtures with ZycoTherm®. In the boiling tests, ZycoTherm® was quite effective when the mixtures were tested in the normal conditions and was able to maintain the stripping resistance of mixtures up to 360 minutes of boiling. However, significant bitumen loss was observed when tested in the salt solution. In the bottle rolling tests, the mixtures showed significant bitumen loss when the conditioning time extended from 6 hours to 48 hours. However, the mixtures with ZycoTherm® showed improved resistance to bitumen loss especially in the salt solution.

**Keywords:** Moisture Induced Damage; ZycoTherm®; Stripping Test; Boil Test; Bottle Rolling Test.

### 1. Introduction

Water susceptibility has been a major concern in asphalt concrete pavements. The failure occurs when the water penetrates through pavement surface, causing the asphalt binder strip off from the aggregate particles, which will eventually lead to reduction in the strength of asphalt concrete. This moisture related damage can be seen in various forms and multiple mechanisms such as adhesion failure between asphalt and aggregate; moisture-induced cohesion failure; cohesion failures within the aggregate; emulsification of the asphalt; and freezing of entrapped water. Among those, the reduction of adhesion between asphalt and aggregates as the presence of water (stripping) and the deterioration of asphalt due to cohesive failure within the asphalt binder itself have been known as two primary driving mechanisms of moisture damage since the 1920s (Solaimanian et al. 2003).

Various studies confirmed that different factors, such as bitumen chemical and rheological characteristics, aggregate type and mineralogy, aggregate surface texture and environmental conditions contribute to moisture-induced pavement deterioration. Adherence and cohesion between binder and aggregate must be strong enough to resist stripping in the presence of water. Physical and chemical features of aggregate, play important role on this adhesion. It was stated in SHRP studies that mineralogy and chemical composition of aggregate are of

primary importance in stripping (Tarrer and Wagh, 1991). Additionally, compaction quality, the dynamic effects of moving vehicles and construction and design operations have considerable effects on moisture-induced damages.

One of the major methods in order to control and decrease the damaging effects of water, is to apply anti-stripping additives. Numerous studies indicate that anti-stripping additives can positively affect the binder-aggregate bonding characteristics (Hicks 1991; Kennedy and Ping 1991; Little and Epps 2001; McCann and Sebaaly 2003; Kim et al. 2004; Huang et al. 2005; Gandhi et al. 2009; Xiao and Amirkhanian 2010; Nazirizad et al. 2015). Application of ASAs, chemically improves the adhesion between the binder and the aggregates. In the literature, both liquid and solid forms of anti-stripping additives are available. One of the most common ASAs is hydrated lime. Considerable literatures emphasize the benefits of hydrated lime such as, improvement of moisture resistance, increased stiffness and resistance against rutting (Jones 1997; Little and Epps 2001; McCann and Sebaaly 2003; Esarwi et al. 2008; Niazi and Jalili 2009; Nazirizad et al. 2015; Das and Singh 2017).

In recent years, the use of nano-organosilane as anti-stripping additive in asphalt pavements have become popular. Arabani et al. in 2011 focused on the evaluation of the moisture susceptibility of warm mix asphalt (WMA) with and without Zycosoil. The results of the surface free energy (SFE) method have indicated that the Zycosoil increased the surface energy of adhesion between aggregate and asphalt in dry and wet condition. Consequently, the energy released in Zycosoil modified mixtures were lower than those without it, and thus their stripping resistance were higher (Arabani et al. 2011). Ameri et al. (2013) investigated the moisture susceptibility of hot mix asphalt with and without Zycosoil as a nano-organosilane additive. Results showed that the Zycosoil additive increased the adhesion bond between the aggregates and asphalt binders and in turn improved the moisture resistance. Behbahani et al. (2015) also studied the effects of nanotechnology Zycosoil and found out that, the application of Zycosoil as an anti-stripping agent improved mechanical properties and moisture sensitivity of mixtures. The effects of Zycotherm on water susceptibility of warm mix asphalt (WMA) mixtures were investigated by Mirzababaei in 2016. The results indicated that Zycotherm significantly improves water susceptibility performance of asphalt mixtures with all aggregate types and gradations (Mirzababaei 2016). Several other studies have also confirmed the improvement of the resistance of asphalt mixtures against water damage with the application of Zycotherm as an anti-stripping additive (Ziari et al. 2016; Ayazi et al. 2017; Ameri et al. 2017)

In this study, the performance of ZycoTherm® against moisture damage and stripping was investigated. The samples were prepared according to Marshall mixture design method with two different types of aggregates, i.e., low and high absorptive. The samples were prepared with and without the nano-organosilane additive for the 4% of design air void content. After that, a number of performance tests were conducted to evaluate the performance of ZycoTherm®. The study program mainly focused on testing the improved binder-aggregate bonding by ZycoTherm®, so that many of the selected tests were repeated at extended conditioning times to compare with the normal (standard) testing conditions. In the study program, laboratory specimens were also tested in the sodium chloride (salt) solution to simulate the detrimental effect of de-icing agents on bitumen-aggregate bonding.

## **2. Materials and method**

### ***2.1. Aggregate***

In the study program, two types of basalt aggregates were used; Yapracik basalt is a highly porous aggregate with an absorption of around 2.7% and known by its stripping problem from past experiences, and Kayseri basalt is, however, a low porosity aggregate with an absorption of around 1.8% and rated as a good aggregate for wearing courses by the local contractors. In terms of other properties, i.e., LA abrasion, sand equivalence, flatness index, freeze-thaw resistance, methylene blue test etc., both aggregates comply with the specifications of the Turkish General Directorate of Highways (TGDH). The measured aggregate properties and the testing standards used for both aggregate types are given in Table 1. It can be seen that the measured properties of the two aggregates are quite similar except that LA abrasion is higher (23%) for Kayseri aggregate than for Yapracik (16%); however, the average absorption of Yapracik is 2.7% (2.4% coarse portion and 3.0% fine portion) while it is measured 1.8% for the Kayseri basalt (1.8% for both coarse and fine portions).

**Table 1.** Aggregate properties

Properties	Yapracik Basalt	Kayseri Basalt	Test Standard
Los Angeles abrasion loss (%)	16	23	TS EN 1072-2
Flatness index (%)	13.6	17.3	TS EN 933-3
Coarse aggregate apparent specific gravity (g/cm <sup>3</sup> )	2.853	2.841	TS EN 1097-6
Coarse aggregate bulk specific gravity (g/cm <sup>3</sup> )	2.669	2.706	TS EN 1097-6
Fine aggregate apparent specific gravity (g/cm <sup>3</sup> )	2.844	2.907	TS EN 1097-6
Fine aggregate bulk specific gravity (g/cm <sup>3</sup> )	2.621	2.760	TS EN 1097-6
Mineral filler apparent specific gravity (g/cm <sup>3</sup> )	2.856	2.842	TS EN 1097-6
Coarse aggregate absorption (%)	2.4	1.8	TS EN 1097-6
Fine aggregate absorption (%)	3.0	1.8	TS EN 1097-6

## 2.2. Asphalt binder and additive

In the experiments, 50/70 penetration graded asphalt binder was provided by the Turkish General Directorate of Highways (TGDH). The properties of the bitumen are presented in the Table 2. The anti-stripping additive used in this study, ZycoTherm®, is an organosilane additive to bitumen binder, to be added before bitumen is mixed with aggregates. Physical properties of the silane-based additive are presented in the Table 3. The additive provides moisture resistance to pavements, as it promotes chemical bonding at the aggregate interface, and also reduces the oxidation, by providing complete coating. The manufacturer guidelines states that the mixing temperature of the additive and bitumen is 150 °C by a mixer that can create a vortex with a depth of 2-3 cm. In order to provide a homogeneous distribution, nanomaterials must be added to asphalt binder in drops using a syringe over a span of 10 min. The silane-based additive consists of both organic and inorganic parts and establishes covalent bonding to bitumen and aggregates. While mixing the additive and binder at high temperatures, the inorganic part solves and the hydrogen bonds break leading to production of H<sub>2</sub>O molecules. The inorganic part of anti-stripping agent establishes Si-O-Si covalent links with the presence of H<sub>2</sub>O molecules and thus a hydrophobic layer that prevents the penetration of moisture in the aggregate-bitumen interface is created (Mirzababaei 2016).

**Table 2.** Asphalt binder properties

Properties	Asphalt
Penetration (0.1 mm)	54
Specific gravity (g/cm <sup>3</sup> )	1.025
Viscosity (cP at 135 °C)	361
Ductility (cm)	>100
Softening Point (°C)	52

**Table 3.** Properties of Zycotherm

Properties	Zycotherm
Specific gravity (g/cm <sup>3</sup> )	1.04
Physical state	Liquid
Color	Pale yellow
Viscosity (cP at 25 °C)	100-500
Flash point (°C)	90

## 2.3. Mixture design method

The mixture designs for all specimens were performed according to the standard Marshall mixture design methodology. Since ZycoTherm® affects the wetting characteristics of bitumen, separate mixture designs were

conducted for the same aggregate sources when the bitumen was modified with ZycoTherm®; hence two designs for Kayseri basalt (with/without additive) and two designs for Yapracik basalt (again with/without additive) were conducted.

The trial samples were compacted to determine the optimum bitumen contents using 75 blow counts. The optimum bitumen contents were calculated for the design air voids of 4%. The design mixture gradations, shown in Figure 1, were selected based on the Turkish General Directorate of Highways (TGDH) gradation requirements for wearing courses. It can be seen from the figure that the design gradations for both basalt aggregates are quite similar with minor deviation from No.3/8 sieve size, hence in comparison of the both aggregate types with ZycoTherm®, the effect of gradation will not be a major factor on the performance test results.

The results of Marshall mix designs for both basalt aggregates are shown in Table 4. It can be realized that Yapracik basalt, due to its high absorptivity, resulted in slightly higher optimum bitumen content (5.72%) with ZycoTherm® as compared to its control (normal) mixture, which required 5.55%. This is because of the fact that ZycoTherm® improves the wetting properties of bitumen and helps to increase the amount of absorbed bitumen in the aggregate. It is also worth noticing that the stability of Yapracik aggregate mixtures with ZycoTherm® are slightly lower, hence yielding higher flow values even though the specification requirements are satisfied. ZycoTherm®, however, showed different results on the design values of the mixtures with Kayseri aggregate. Even though the optimum bitumen contents were not changed, the stability of mixtures with ZycoTherm® increased significantly in spite of the increase in the flow values. This can be perhaps explained by the improved bonding between bitumen and aggregate when ZycoTherm® is used, leading increased stability of the mixtures.

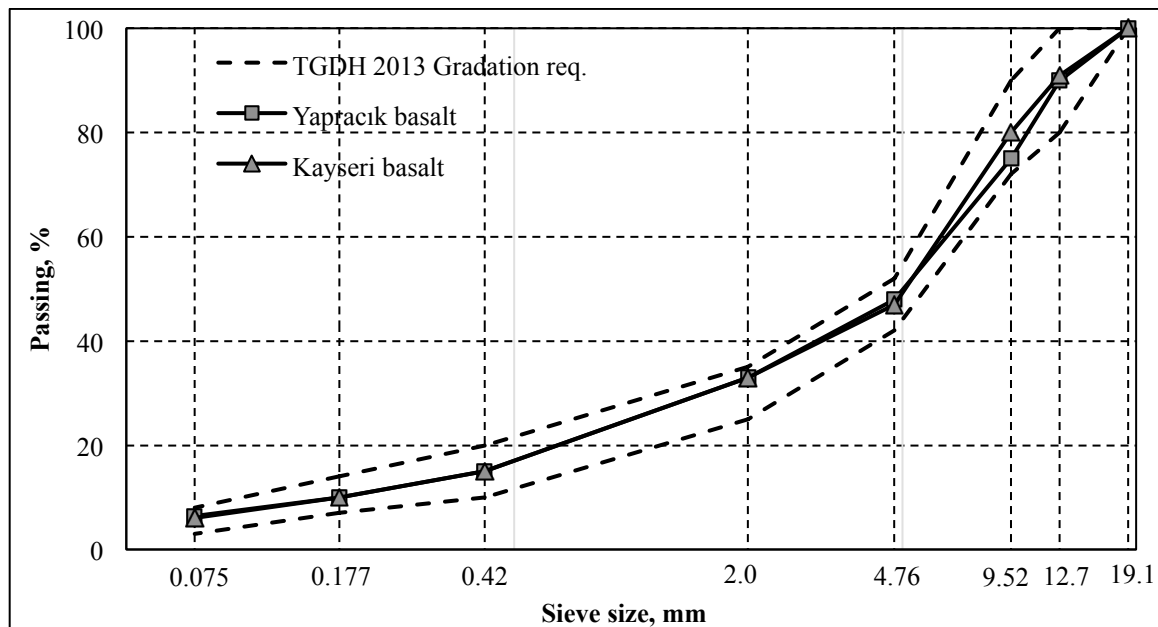


Figure 1. Design gradations for both aggregates with/without modification

Table 4. Mixture design properties

Design property	Yapracik aggregate		Kayseri aggregate		TGDH req.
	Without ZycoTherm®	With ZycoTherm®	Without ZycoTherm®	With ZycoTherm®	
Optimum bitumen ratio (%)	5.55	5.72	5.70	5.70	4 - 7
Bulk specific gravity(g/cm <sup>3</sup> )	2.409	2.409	2.475	2.479	-
Air void (%)	4.03	4.03	4.09	4.05	3-5
VMA (%)	14.2	14.3	14.4	14.3	≥ 14
VFA (%)	71.6	71.9	71.6	71.6	65 - 75
Stability (kg)	1490	1220	1380	1745	≥ 900
Flow (mm)	2.50	4.00	2.87	3.21	2 - 4
Filler / Bitumen ratio	1.15	1.12	1.05	1.05	-

### **3. Laboratory testing program**

After the mixture designs, a number of performance tests were conducted to evaluate the performance of the ZycoTherm®. The study program mainly focused on testing the improved binder-aggregate bonding by the organosilane additive, so that the selected tests were repeated at extended conditioning times to compare with the normal (standard) testing conditions. Additionally, the specimens tested in the sodium chloride (salt) solution to simulate the detrimental effect of de-icing agents on bitumen-aggregate bonding.

#### ***3.1. Stripping test***

In these tests, ZycoTherm® was evaluated for each aggregate type based on four test combinations as: normal condition, in 4% salt solution, normal condition with ZycoTherm®, with ZycoTherm® in 4% salt solution. All the stripping tests were performed according to the Nicholson method as described in AASHTO T182. During the tests, the specimens were also subjected to extended conditioning of 5, 7 and 14 days to validate ZycoTherm® performance.

#### ***3.2. Boil test***

The same test combinations as for the stripping test were used except that the extended conditioning times were selected 10 min, 1 hour, 3 hours, and 6 hours. All the tests were conducted according to the guidelines as specified in ASTM D3625 standard.

#### ***3.3. Bottle rolling test***

Bottle rolling tests were also conducted with the same test combinations as with the boiling and the stripping tests, i.e., normal condition, in 4% salt solution, normal condition with ZycoTherm®, with ZycoTherm® in 4% salt solution. These tests were carried out for the coating retention times of 6, 24, 72, and 96 hours according to EN 12697-11 standard.

### **4. Test results**

The performance of ZycoTherm® was evaluated based on a number of laboratory performance tests using two different basalt aggregates, one with high absorption and the other with low absorption. The test outcomes were analysed and the results are presented in the below sections.

#### ***4.1. Stripping test with & without salt solution (NaCl)***

Results of Nicholson stripping tests for extended time periods for Yapracik basalt and Kayseri basalt aggregates are given in Figure 2 and 3, respectively. The results in Figure 2 for Yapracik aggregate shows that the extended conditioning time has only a minor effect on the stripping resistance of the mixtures whether tested at normal or in 4% salt solution without ZycoTherm®. Even though there was slight decrease in the stripping resistance after 168 and 360 hours of conditioning, no pronounced effect of the extended conditioning was detected. The reduction in the stripping resistance between 24 and 360 hours in around 5% for the control specimen of Yapracik basalt. However, when ZycoTherm® is used, a pronounced improvement in the stripping resistance can be observed from testing both at normal and in salt solution. The stripping resistance of the mixtures with ZycoTherm® after 360 hours of conditioning is even higher than that of control mixtures conditioned for only 24 hours. For these mixtures, even though the stripping resistance decreased around 15% between 24 hours and 360 hours, this effect dissipates when the tests were run in the salt solution. Hence, the test results clearly indicate that the salt solution, in fact, increased the stripping resistance as opposed to the expectation for all the test combinations. The other outcome is that even though Yapracik basalt is a highly absorptive aggregate, ZycoTherm® is able to double the stripping resistance in spite of extended conditioning times.

The trends in Figure 3 for Kayseri aggregate are similar to those of Yapracik aggregate; however, the improvement in the stripping resistance seems even better for Kayseri basalt, which represents the low absorptive aggregate in the test mixtures. The other interesting results is that the mixtures with ZycoTherm® do

not seem to be affected at extended conditioning times whether tested at normal or in salt solution even though there is nearly a 40-50% loss of resistance between 24 hours and 360 hours of conditioning for its control mixtures without ZycoTherm®.

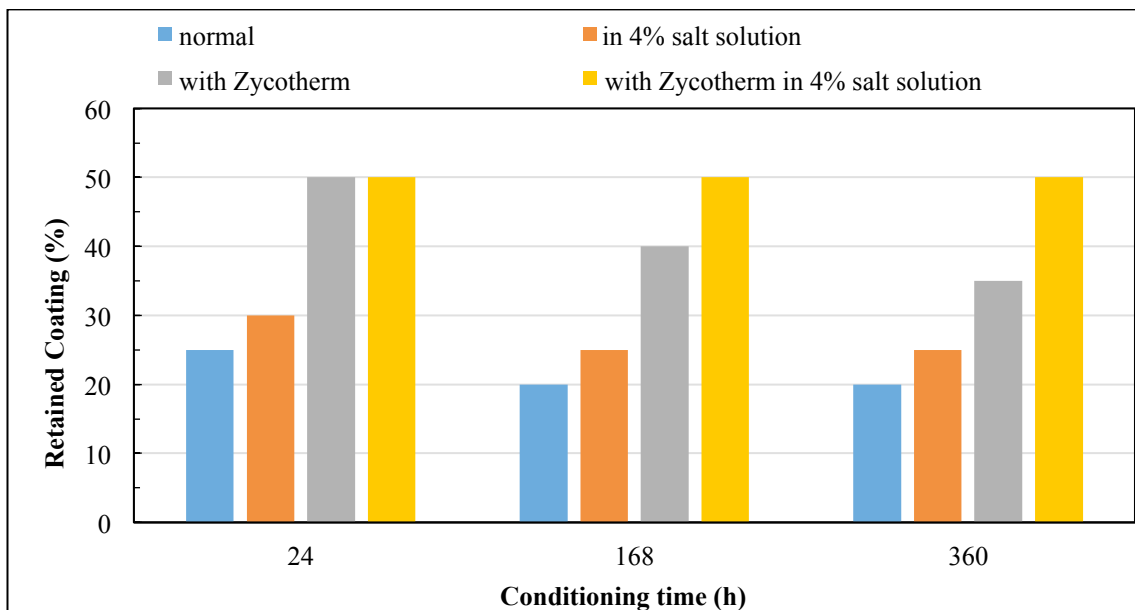


Figure 2. Results of Nicholson stripping tests for Yapracik aggregate

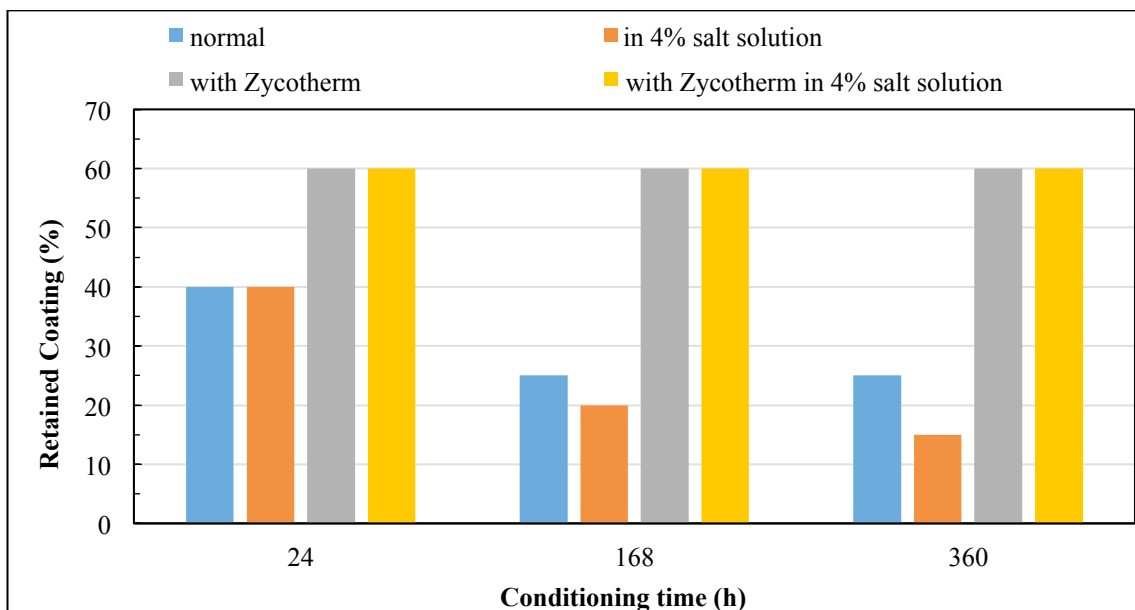


Figure 3. Results of Nicholson stripping tests for Kayseri aggregate

#### 4.2. Boil test with & without salt solution (NaCl)

The results for Yapracik aggregate in Figure 4 shows that except for the mixtures with ZycoTherm® tested without salt solution, the effect of conditioning time is dramatic, in that the stripping resistance was nearly zero and no bitumen remained on the aggregate surface when the tests were finished. When mixtures were tested in the salt solution, again the contribution of ZycoTherm® was clear, maintaining a stripping resistance of around 25%. However, the best performance of ZycoTherm® was obtained from the mixtures tested without salt solution; there was only 10% of loss in resistance (from 90% to 80%) between 10 min and 360 min of

conditioning times. This shows that ZycoTherm® at normal testing conditions performs quite better in the boiling test even for high absorptive aggregates by successfully maintaining the bitumen-aggregate bonding.

Stripping resistance for Kayseri aggregate in the boiling tests proves similar behavior of ZycoTherm®, as shown in Figure 5; at normal test conditions without salt solution, it was able to maintain the bitumen resistance to strip even for 360 min of boiling. The mixtures with ZycoTherm®, after 10 min of boiling almost no loss of bonding especially when tested in normal water. The other mixtures tested at normal or in salt solution showed a clear loss of bonding with the extended boiling times. In these tests, however, salt solution resulted in a reduction of the bonding strength whether ZycoTherm® is used or not. In summary, the mixtures with ZycoTherm® showed a superior performance over the control mixtures even at extended boiling times, and yielded minor or no bonding loss between bitumen and aggregate.

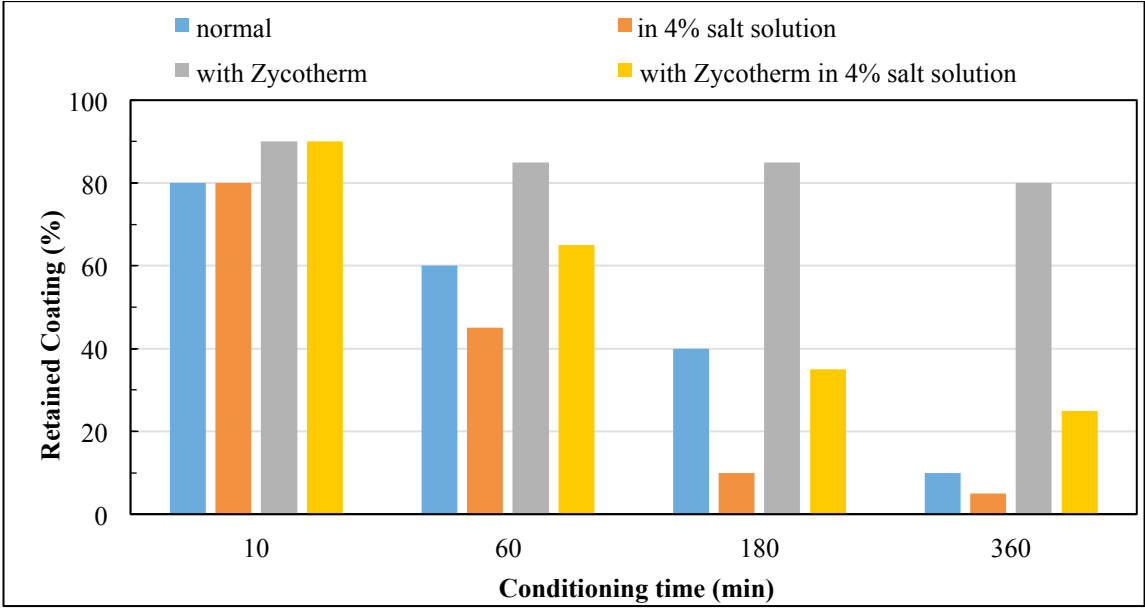


Figure 4. Results of stripping tests according to boiling water method for Yaprak aggregate

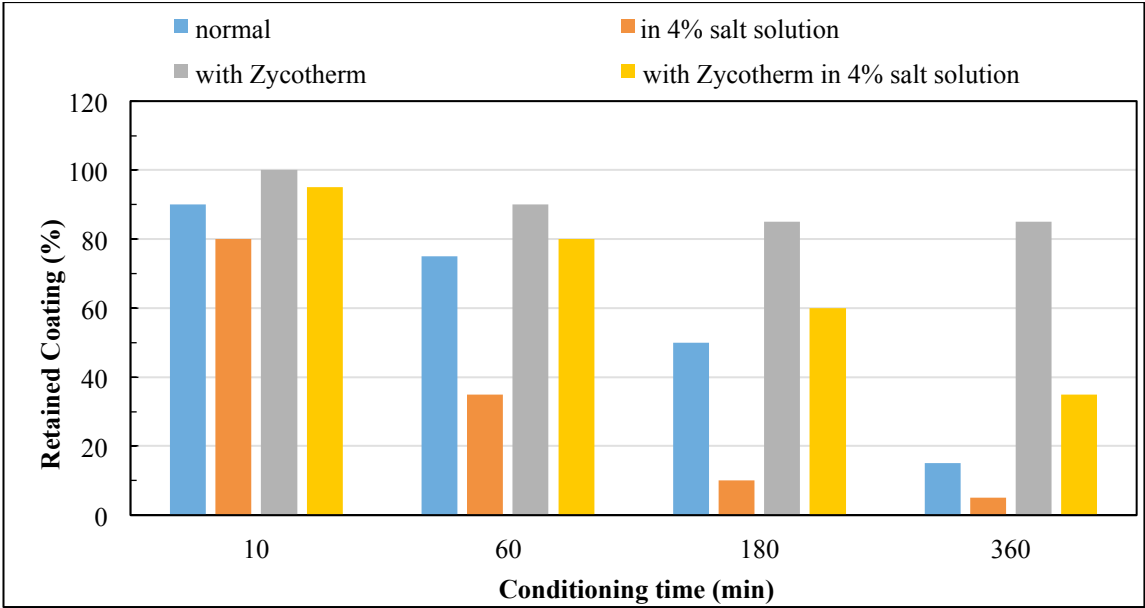


Figure 5. Results of stripping tests according to boiling water method for Kayseri aggregate

4.3. Bottle rolling test with & without salt solution (NaCl)

Bottle rolling tests results for Yapracik aggregate are given in Figure 6 for the test combinations with/without ZycoTherm® and normal/in 4% salt solution. In these tests, the mixtures were also subjected to extended conditioning of 6, 24 and 48 hours. The test results reveal that, as in the Nicholson tests, salt solution results in a positive contribution to bitumen-aggregate bonding, yielding better stripping resistance for all the extended conditioning periods. However, all of the test mixtures showed a decrease in the stripping resistance as the testing time was increased from 6 hours to 48 hours. The mixtures with ZycoTherm® showed improved resistance against stripping over the control mixtures; however, the improvement was better when tested in the salt solution.

The results for Kayseri aggregate are shown in Figure 7. The stripping resistance for this aggregate seems slightly higher due to its low absorptivity; however, the effect of extended testing time is similar to Yapracik aggregate. Again, testing the mixtures in the salt solution increased the bonding resistance against stripping in spite of degradation with the extended testing time from 6 to 48 hours. As for the Yapracik aggregate, mixtures with ZycoTherm® showed higher resistance against stripping as compared to the control mixtures.

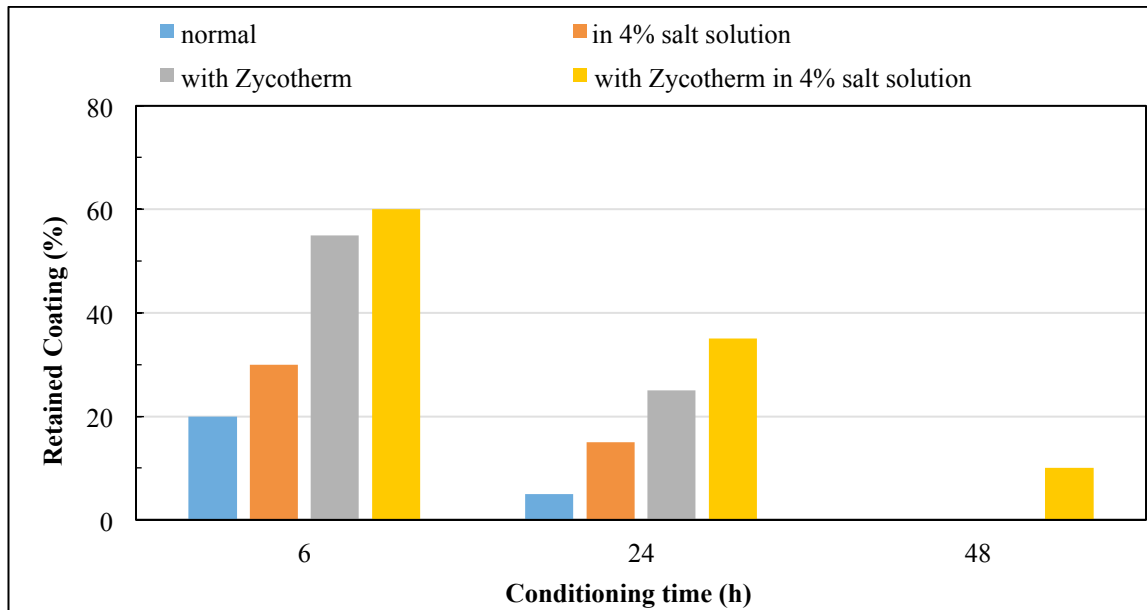
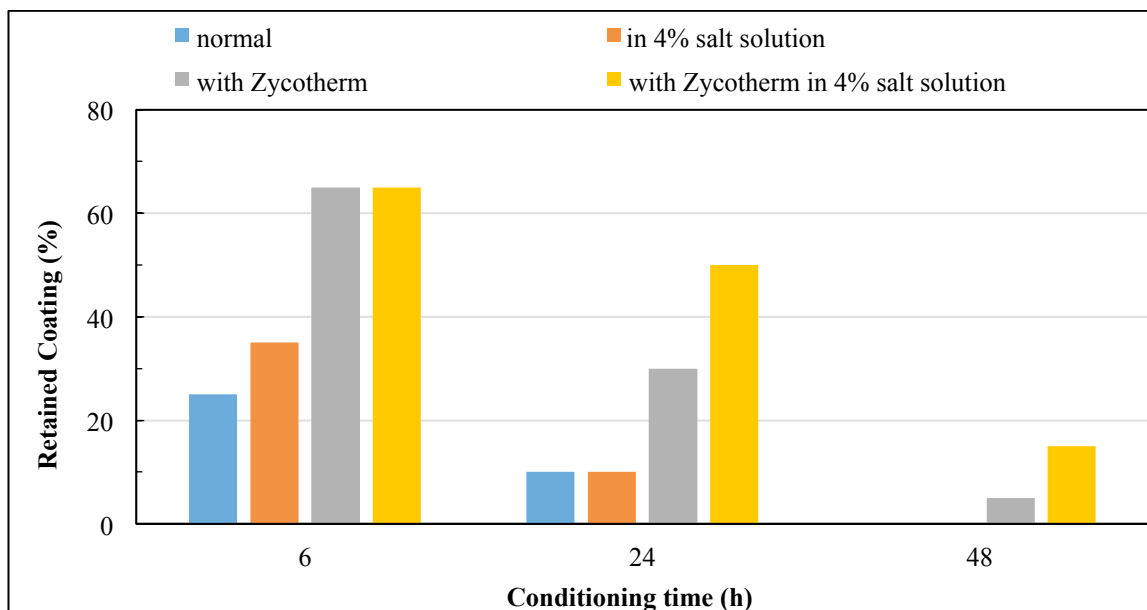


Figure 6. Results of stripping tests according to rolling bottle method for Yapracik aggregate





**Figure 7.** Results of stripping tests according to rolling bottle method for Kayseri aggregate

## 5. Conclusions

In this study, the performance of ZycoTherm® was evaluated on various asphalt mixtures for moisture damage and stripping properties. A detailed experimental program was scheduled to evaluate how ZycoTherm® affects these important mixture properties. In the testing program, two types of basalt aggregates that are commonly used in Turkey were selected, representing a low absorptive aggregate by Kayseri source basalt and high absorptive aggregate by Yapracik source basalt. In the stripping and the moisture damage tests, the test conditions were also revised by extending the conditioning time and altering the conditioning environment by using sodium chloride (salt) solution. Based on the analyses of the data from the experimental program, the following conclusions can be drawn:

ZycoTherm® did not cause any change in the optimum bitumen content for mixtures with low absorptive aggregate, however, increased the Marshall stability and improved the flow value as compared to its control mixture without ZycoTherm®. For high absorptive aggregate mixtures, however, the optimum bitumen content increased slightly from 5.55% to 5.72%. The Marshall stability decreased and the flow value increased as compared to its control mixture without ZycoTherm®.

In the Nicholson tests, ZycoTherm® significantly improved the stripping resistance of the mixtures of Yapracik and Kayseri aggregates at all extended conditioning times. The resistance of both mixtures was even better in the salt solution, showing only a slight decrease with extended conditioning of the normal mixtures and almost no resistance loss for the mixtures with ZycoTherm®. Comparing the two sources of basalt aggregates, mixtures of Kayseri aggregate showed overall a slightly better resistance to stripping than that of Yapracik.

In the boiling tests, ZycoTherm® was able to maintain the stripping resistance of both mixtures, i.e., with Yapracik and Kayseri aggregates, up to 360 minutes of boiling when tested in the normal conditions. However, significant bitumen loss was observed in both mixtures when tested in the salt solution even though ZycoTherm® was used. It is, therefore, concluded that ZycoTherm® is quite effective in the boiling test when mixtures are tested in the normal conditions as opposed to the case where mixtures are conditioned at extended times in the salt solution.

In the bottle rolling tests, all the test mixtures, i.e., with Yapracik and Kayseri aggregates together, showed significant bitumen loss when the conditioning time is extended from 6 hours to 48 hours. However, the mixtures with ZycoTherm® showed improved resistance to bitumen loss especially in the salt solution. On average, mixtures of Kayseri basalt showed slightly better stripping resistance than that of Yapracik basalt.

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