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# Impact of Temperature Changes and Freeze—Thaw Cycles on the Behaviour of Asphalt Concrete Submerged in Water with Sodium Chloride

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**Abstract:** One of the main applications of salt in civil engineering is its use as a de-icing agent on roads in cold areas. The purpose of this research is to find out the mechanical behaviour of an asphalt concrete when it is subjected to temperature changes and freeze–thaw cycles. These temperature interactions have been carried out for dry specimens, specimens submerged in distilled water and specimens submerged in salt water (5% of sodium chloride, NaCl). An AC16 Surf D bituminous mixture was evaluated under three types of temperature interaction: three reference series remained at a controlled temperature of 20 °C, another three series were subjected to five freeze–thaw cycles and the last three series have been subjected to one year outside in Santander (Spain). The mechanical behaviour of the mixture was determined by Indirect Tensile Strength Test (ITS), Water Sensitivity Test (ITSR) and Wheel Tracking Test, Dynamic Modulus Test and Fatigue Tests. The results of the tests show that, although the temperature changes have a negative effect on the mechanical properties, salt water protects the aggregate-binder adhesive, maintains the mechanical strength, increases the number of load cycles for any strain range and reduces the time that the mixture is in contact with frozen water.

**Keywords:** salt; NaCl; asphalt concrete; freeze–thaw cycles; winter road

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**Highlights:** Specimens of hot mix asphalt were evaluated under different temperature changes, including freeze–thaw cycles. Temperature changes have a harmful effect on the behaviour of the mixture, but the amount of time that the mixture is submerged in contact with salt water is the main mechanism of damage. Salt water protects the aggregate-binder adhesive, maintains the mechanical strength, increases the number of load cycles for any strain range and reduces the time that the mixture is in contact with frozen water.

## 1. Introduction

Currently, the use of sodium chloride (NaCl) is widespread in a large number of countries, as a de-icing agent on winter roads. The impact that salt has on the environment has been extensively studied; the melting salt leads to the mobilization of heavy metals such as lead, cadmium, copper and zinc [1–4], along with increased chlorides ($Cl^-$) [5,6]. However, salt is still used, due to its low cost, versatility and physical properties [7,8]. These physical properties make their use propitious in temperatures up to −21 °C, but as García [7] indicated, from −5 °C, in order to be more effective, they can be used in combination with calcium chloride ($CaCl_2$).

Shi et al. [9] noted that the value of negative impacts on the environment and on vehicles must be taken into account when calculating the costs of de-icing agents, including NaCl. In this trend of reducing costs and negative impacts, Klein-Paste et al. [10] indicated that the amount of NaCl that is used in practice could be reduced by 40%, to prevent the pavement from being slippery, because the salt spread on the roads causes a weakening of the ice. Another aspect studied along the line of reducing the negative impact of NaCl is the creation of tools to decide more efficiently when salt should be applied to roads [11,12].

There are also studies on the behaviour of bituminous mixtures in contact with the different de-icing agents. Wang et al. [13] noted that, compared to other anti-icing agents, such as sand and quartz dust, salt does not produce a polished surface. Hassan et al. [14] indicated that the value of Indirect Tensile Strength (ITS) for asphalt mixtures exposed to salt after 25 freeze–thaw cycles is similar to other de-icing agents, such as potassium acetate, sodium formate or urea. However, after 50 cycles, the ITS result is even better for the mixtures exposed to salt than those submerged in distilled water.

Feng et al. [15] studied the impact of salt on the performance of bituminous mixtures when it remains submerged under seawater subjected to freeze–thaw cycles. They simulate this fact by adding salt to bitumen and subjecting various types of mixtures (AM-16, OGFC-19 and AC-16) to freeze–thaw cycles; their results show a decrease in the Water Sensitivity Test (ITSR), with results being lower in the case of the AC mixture.

Juli-Gándara et al. [16] investigated how NaCl influences the mechanical properties of three types of asphalt mixtures: a hot mix asphalt with conventional bitumen; and two porous mixtures, one manufactured with a conventional binder and another with a modified binder. The effect of the salt is analysed by three different processes: immersing the specimens in salt water; adding salt as aggregate into the mixture; and submerging the aggregate in water with a certain concentration of salt, drying it and then making the mixture with it. The results show that the hot mix asphalt is scarcely affected when it is submerged in salt water.

The effects of freeze–thaw cycles in the bituminous mixture are widely studied. Goh and You [17] used an image-processing technique to indicate that the removal of fine and coarse aggregates on the surface increases when the asphalt mixture undergoes more freeze–thaw cycles. Tarefder et al. [18] tested an AC mixture to ITS and Fatigue Test after 5, 10, 15 and 20 freeze–thaw cycles, and the results showed a reduction of 30.7% in the fatigue life and a small amount of reduction of ITSR after five cycles. Özgan and Serin [19] indicated through the void ratio (Vh), the void ratio filled with asphalt (Vf) and the void ratio inside mineral aggregate (VMA) parameters; the ultrasonic velocity test; and the Marshall Stability (MS) that the effect of the freeze–thaw cycles on the asphalt concrete is highly important, especially for the hot mix asphalt design. Islam and Tarefder [20], who investigated the stiffness and tensile strength degradation behaviour of asphalt concrete on long-term freeze–thaw samples in the laboratory, indicated that the flexural stiffness decreases with the number of freeze–thaw cycles, whereas the ITS does not change significantly with the number of freeze–thaw cycles. Teltayev et al. [21] investigated, in laboratory conditions, the effect of cyclic freezing and thawing on the characteristics of the neat bitumen and bitumens modified with different polymers, as well as stone mastic asphalt concretes. Their results of the SMA-20 Bit 100/130 show a 78% decrease in the value of ITS and a 270% increase in the Rut Depth (RD) after 50 freeze–thaw cycles.

Until now, no research has covered the results of mechanical behaviour such as mechanical strength, permanent deformations, dynamic modulus and fatigue life, when an asphalt concrete mixture is subjected to temperature changes, freeze–thaw cycles and the impact of salt. The purpose of this research is to fill this gap. The temperature interactions have been carried out for dry specimens, specimens submerged in distilled water and specimens submerged in salt water (NaCl).

## 2. Methodology

### 2.1. Materials

#### 2.1.1. Aggregate

The aggregate used in this research is an ophite with the following properties (Table 1):

**Table 1.** Properties of aggregate.

| Aggregate | Los Angeles Abrasion Test (%) (UNE-EN 1097-2:2010) [22] | Water Absorption (%) (UNE-EN 1097-6:2014) [23] | Flakiness Index (%) (UNE-EN 933-3:2012) [24] |
|---|---|---|---|
| Ophite | 16.0 | 1.0 | 9.0 |

The filler (mineral powder) is limestone, with a specific weight of 2.753 g/cm$^3$.

#### 2.1.2. Binder

A conventional bitumen B 50/70, which is frequently used in Spain, was used. Table 2 shows the principal properties of the bitumen:

**Table 2.** Properties of bitumen.

| Bitumen | Penetration (25 °C; 100 g, 5 s) (0.1 mm) (UNE-EN 1426:2015) [25] | Softening Point (°C) (UNE-EN 1427:2015) [26] | Frass Breaking Point (°C) (UNE-EN 12593:2015) [27] |
|---|---|---|---|
| B 50/70 | 65.0 | 47.2 | −9 |

#### 2.1.3. Mixture

An AC16 Surf B50/70 D mixture, which is frequently used on surface pavements in Spain, was studied. The composition is shown in Table 3. The density of the mixture, according to the Marshall Test (UNE-EN 12697-34:2013) [28], is 2.458 g/cm$^3$.

**Table 3.** Composition of bituminous mixture.

| | Passing Rate (%) | | | | | | | | Bitumen Content *s/m* (%) |
|---|---|---|---|---|---|---|---|---|---|
| Sieve size (mm) | 22 | 16 | 8 | 4 | 2 | 0.5 | 0.25 | 0.063 | |
| AC16 Surf D | 100.0 | 95.0 | 71.5 | 51.5 | 38.5 | 21.5 | 15.5 | 6.0 | 5.0 |

The manufacture of the AC16 Surf B 50/70 D is done at the SENOR S.A. asphalt plant.

#### 2.1.4. Salt

The salt used for this work is NaCl. Table 4 indicates the particle size of salt. The specific weight of the salt used is 2.165 g/cm$^3$.

**Table 4.** Particle size of salt.

| | Passing Rate (%) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Sieve Size (mm) | 22 | 16 | 8 | 4 | 2 | 0.5 | 0.25 | 0.063 |
| Salt | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 74.9 | 39.9 | 6.6 |

### 2.2. Experimental Plan

Nine different series have been evaluated. All the batches of bituminous mixture were manufactured at the same time. After this, the specimens were subjected to individual analysis (Table 5):

- Test Series A: remains at a constant temperature of 20 °C;
- Test Series B: subjected to five freeze–thaw cycles;
- Test Series C, subjected to one year outside storage;

whereby

- (A0) is dry specimens,
- (A1) is submerged in distilled water,
- (A2) is submerged in salt water,

in each case.

**Table 5.** Series description.

| | | Water Interaction | | |
|---|---|---|---|---|
| | | **Dry** | **Submerged in Distilled Water** | **Submerged in Salt Water** |
| Temperature interaction | Constant temperature at 20 °C | A0 | A1 | A2 |
| | Five freeze–thaw cycles | B0 | B1 | B2 |
| | One year outside storage | C0 | C1 | C2 |

The amount of salt by water weight in A2, B2 and C2 series is five percent (5.0%). For submerged series, the temperature and time that the specimens are submerged change according to the test requirements.

### 2.2.1. Temperature Interaction

Three different temperature interactions were analysed. The A-Series are for reference; the temperature remains constant at 20 °C.

#### Freeze–Thaw Cycles

The B-Series were subjected to five freeze–thaw cycles between 38 and −33 °C. These temperatures have been chosen because 38 °C is approximately ten degrees higher than the summer air temperature average in Santander (Spain), and −33 °C is ten degrees lower than the freezing point of water with 5.0% salt content.

The temperature was measured by introducing a thermo-sensor inside the specimens at the time of manufacturing the mixture (Figure 1). With these thermo-sensors, it is possible to determine the internal temperature of the specimens (logging interval one minute). Knowing this, the time of each freeze–thaw cycle is established so that both phases, hot and cold, have enough time to reach the defined temperatures. Each freeze–thaw cycle requires 48 h to be completed, 24 h for the hot time period and 24 h for the cold time period (Figure 2).

**Figure 1.** Thermo-sensor inside the specimens.

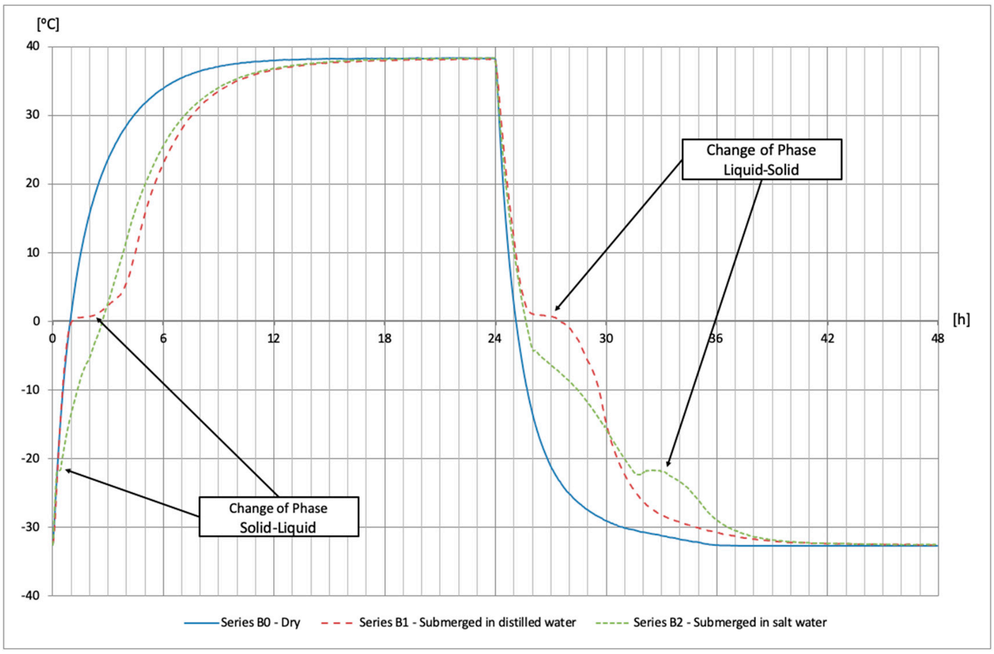


**Figure 2.** Freeze–thaw cycle.

The time that each submerged series remained in frozen water was defined by the steps of solid–liquid and liquid–solid changes. In the case of the B1 series, the time submerged in frozen water is 22 h, and in the B2 series, it is approximately 15 h.

## Outside Storage

The outside storage series, the C-Series, remained outside in Santander (Spain) for one year, from 20 December 2016 to 20 December 2017, and was protected from precipitation. The maximum and minimum daily temperatures during this period were collected by the Spanish Meteorological Agency AEMET (Figure 3).

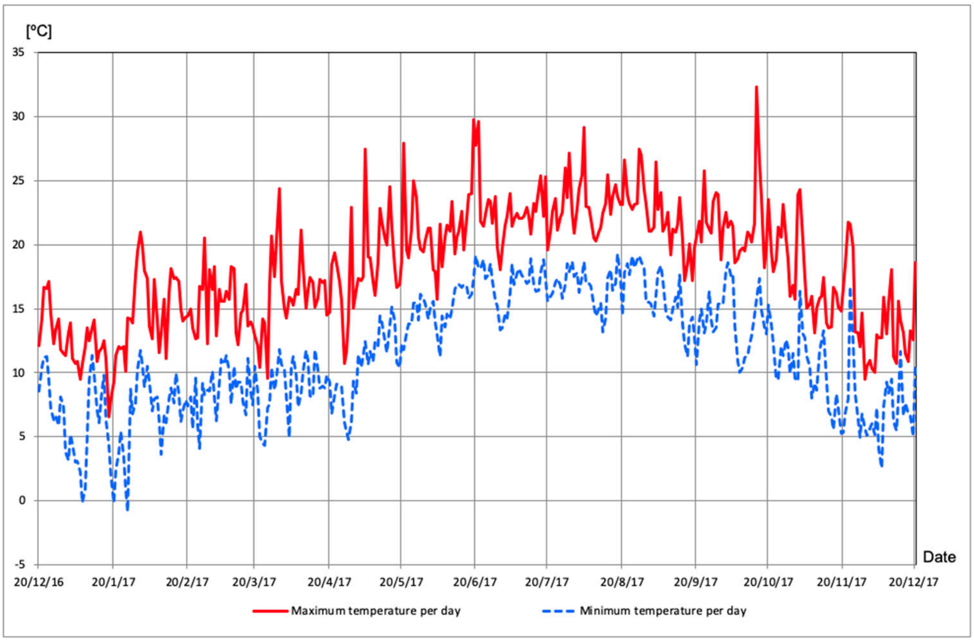


**Figure 3.** Maximum and minimum temperature per day in Santander for the year 2017.

### 2.3. Tests Programme

#### 2.3.1. Indirect Tensile Strength (ITS) and Water Sensitivity Test (ITSR)

These tests have been done to determine the mechanical strength and how the aggregate-binder adhesive is influenced by the action of water. As established by Spanish Standard PG-3 [29], the test procedure was the "Method A" of the UNE-EN 12697-12: 2009 standard [30], with a compaction of the specimens by impact (UNE-EN 12697-30: 2013) [31], with 50 blows per side. Four dry specimens and another four wet specimens for each series were evaluated.

The dimensions of the specimens are 63.5 mm in height and 101.6 mm in diameter. This test is done for all specimens.

#### 2.3.2. Wheel Tracking Test

This test has been carried out to determine the susceptibility of the specimen to deformation when a moving vertical load is applied. The test is done in accordance with the standard UNE-EN 12697-22: 2008 + A1 "Procedure B in air" [32], compacted by roller compactor (UNE-EN 12697-33: 2006 + A1) [33]. The limits established by PG-3 depending on the heavy traffic categories are 0.07 and 0.10 mm/$10^3$ cycles of Wheel Track Slope (WTS).

The specimens are 410 mm long by 260 mm wide by 50 mm high. The Wheel Tracking Test is only done for the A-Series and C-Series. For these series, two specimens of each one were tested.

#### 2.3.3. Dynamic Modulus Test

Since bituminous mixtures are a viscous material, this test was done to provide insight into the viscosity properties of the mixtures. The test was carried out as established by the standard UNE-EN 12697-26: 2012 "Annex B" four-point bending test [34]. Six specimens have been evaluated for each series, except for the C-Series, where eight specimens have been tested due to the long period that these specimens have been outdoors.

The specimens are 410 mm long by 60 mm wide by 60 mm high. The Dynamic Modulus Test was done for all specimens.

#### 2.3.4. Fatigue Test

This test provides an estimate of durability of the bituminous mixture by load–unload cycles. The test procedure has been the UNE-EN 12697-24: 2013 "Annex D" four-point bending test [35]. The same number of specimens, as in the case of the Dynamic Modulus Test, was evaluated.

The dimensions of the specimens are the same as the ones of the Dynamic Modulus Test. The Fatigue Test is done for all specimens.

## 3. Results and Discussion

### 3.1. Indirect Tensile Strength (ITS) and Water Sensitivity Test (ITSR)

Table 6 shows the results of Indirect Tensile Strength and Water Sensitivity Test for all the series. The same as Hassan et al. [14] indicated, the values of ITS are similar or even better for mixtures exposed to salt than those submerged in distilled water.

The results obtained in the A-Series for ITS and ITSR are the regular ones for a hot mix asphalt. There are no noticeable differences between the series submerged in distilled water (A1) and the series submerged in salt water (A2). This result was expected due to the fact that the A-Series was not submitted to temperature changes.

In the series subjected to five freeze–thaw cycles (B-Series), although the result of ITS for dry specimens (B0) shows no significant difference when compared to the dry reference specimens (A0), that difference exists for the wet series. This variation is lower between specimens submerged in salt water (B2 and A2) and greater between specimens submerged in distilled water (B1 and A1). These results show that the greatest damage to the hot mix asphalt is not the temperature changes but the amount of time that the mixture is submerged in frozen water. The B2 series obtained a value of ITRS, which is close to the limit established by PG-3 (Spanish Regulation) for surface layers (85%).

**Table 6.** Indirect tensile strength and water sensitivity.

| | Series | AC16 Surf B 50/70 D | | |
|---|---|---|---|---|
| | | **Maximum Load (kN)** | | **ITSR (%)** |
| Constant temperature at 20 °C | A0 | 2340 | – | – |
| | A1 | – | 2075 | 89 |
| | A2 | – | 2053 | 88 |
| Five Freeze–Thaw Cycles | B0 | 2385 | – | – |
| | B1 | – | 1540 | 63 |
| | B2 | – | 1975 | 83 |
| One year outside storage | C0 | 2189 | – | – |
| | C1 | – | 1418 | 65 |
| | C2 | – | 1690 | 77 |

For the series subjected to a year outdoors (C-Series), the results of ITS for dry and submerged specimens are lower than the ones of the reference series (A-Series). However, in the case of submerged specimens (C1 and C2), especially for the series submerged in distilled water, the values of the ITS test are lower than in dry specimens (C0). This result is due to the fact that remaining submerged for a year is a more damaging process for the mixture than remaining dry for the same period.

### *3.2. Wheel Tracking Test*

The values for the Wheel Track Slope and the Ruth Depth at 10,000 cycles (RD) for all the series are shown in Tables 7 and 8. The results obtained from these tests comply with the Spanish regulations for most climate zones and traffic.

**Table 7.** Wheel track slope.

| | Series | AC16 Surf B 50/70 D |
|---|---|---|
| | | **WTS (mm/$10^3$ Cycles)** |
| Constant temperature at 20 °C | A0 | 0.08 |
| | A1 | 0.07 |
| | A2 | 0.05 |
| One year outside storage | C0 | 0.06 |
| | C1 | 0.04 |
| | C2 | 0.08 |

Although the results for all series are similar, the series that have remained a year outdoors have obtained WTS and RD values lower than the reference series, except for the series whose specimens were submerged in salt water (C2). These low values (C0 and C1) can be due to the fact that, after one year in outside storage, the binder has become stiffer.

**Table 8.** Rut depth.

| | Series | AC16 Surf B 50/70 D |
|---|---|---|
| | | RD (mm) |
| Constant temperature at 20 °C | A0 | 3.2 |
| | A1 | 3.6 |
| | A2 | 2.4 |
| One year outside storage | C0 | 2.6 |
| | C1 | 2.1 |
| | C2 | 3.0 |

### 3.3. Dynamic Modulus Test

As Table 9A,B indicate, the phase angles have no significant variation between the different series. Only in the case of those series which have been subjected to freeze–thaw cycles, the variation is slightly greater. This fact may be due to the larger temperature differences between the B-Series and the other series.

**Table 9.** Dynamic modulus test.

(A)

| | Series | AC16 Surf B 50/70 D | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Frequency (Hz) | | | | | | | | | |
| | | 0.1 | | 0.2 | | 0.5 | | 1.0 | | 2.0 | |
| | | E (GPa) | Phase Angle (°) | E (GPa) | Phase Angle (°) | E (GPa) | Phase Angle(°) | E (GPa) | Phase Angle (°) | E (GPa) | Phase Angle (°) |
| Constant temperature at 20 °C | A0 | 2.4 | 32.8 | 2.9 | 30.5 | 3.7 | 27.0 | 4.4 | 24.9 | 5.2 | 22.6 |
| | A1 | 2.2 | 33.8 | 2.7 | 31.2 | 3.5 | 27.5 | 4.1 | 25.1 | 4.9 | 23.2 |
| | A2 | 2.1 | 34.7 | 2.6 | 32.1 | 3.5 | 28.4 | 4.2 | 26.1 | 5.0 | 24.0 |
| Five Freeze–Thaw Cycles | B0 | 2.2 | 35.9 | 2.7 | 33.2 | 3.5 | 30.0 | 4.3 | 27.4 | 5.1 | 25.2 |
| | B1 | 1.7 | 34.8 | 2.2 | 32.1 | 2.9 | 28.9 | 3.5 | 26.8 | 4.1 | 24.6 |
| | B2 | 2.0 | 33.0 | 2.5 | 30.5 | 3.3 | 27.1 | 4.0 | 25.0 | 4.7 | 23.0 |
| One year outside storage | C0 | 2.4 | 31.3 | 2.9 | 28.9 | 3.7 | 26.0 | 4.4 | 23.9 | 5.2 | 21.9 |
| | C1 | 2.2 | 33.1 | 2.6 | 30.6 | 3.4 | 27.5 | 4.1 | 25.4 | 4.8 | 23.3 |
| | C2 | 2.2 | 34.0 | 2.8 | 31.2 | 3.6 | 28.0 | 4.3 | 25.8 | 5.1 | 23.6 |

(B)

| | Series | AC16 Surf B 50/70 D | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Frequency (Hz) | | | | | | | | | |
| | | 5.0 | | 8.0 | | 10.0 | | 20.0 | | 30.0 | |
| | | E (GPa) | Phase Angle (°) | E (GPa) | Phase Angle (°) | E (GPa) | Phase Angle (°) | E (GPa) | Phase Angle (°) | E (GPa) | Phase Angle (°) |
| Constant temperature at 20 °C | A0 | 6.2 | 20.3 | 6.9 | 19.0 | 7.1 | 18.5 | 8.1 | 17.0 | 8.9 | 16.5 |
| | A1 | 6.0 | 20.7 | 6.6 | 19.2 | 6.9 | 18.6 | 7.9 | 17.6 | 8.7 | 16.6 |
| | A2 | 6.1 | 21.3 | 6.7 | 20.0 | 7.0 | 19.3 | 8.0 | 17.8 | 8.9 | 17.6 |
| Five Freeze–Thaw Cycles | B0 | 6.3 | 22.4 | 7.0 | 21.1 | 7.3 | 20.5 | 8.5 | 18.8 | 9.2 | 17.8 |
| | B1 | 5.1 | 21.9 | 5.7 | 20.6 | 5.9 | 19.9 | 6.9 | 18.6 | 7.5 | 17.7 |
| | B2 | 5.8 | 20.3 | 6.4 | 19.2 | 6.6 | 18.6 | 7.8 | 17.7 | 8.5 | 16.6 |
| One year outside storage | C0 | 6.4 | 19.6 | 6.9 | 18.4 | 7.2 | 17.6 | 8.5 | 16.8 | 9.2 | 15.9 |
| | C1 | 5.9 | 20.8 | 6.5 | 19.5 | 6.9 | 19.1 | 7.9 | 18.6 | 8.4 | 16.2 |
| | C2 | 6.2 | 21.1 | 6.9 | 19.7 | 7.2 | 19.1 | 8.3 | 17.9 | 8.9 | 16.8 |

In the A-Series, very similar values were obtained for all the specimens. However, there exists a trend in which the dry series has higher modulus values and lower phase angles, due to the fact that it is more elastic than the two others.

However, for the series subjected to freeze–thaw cycles, a clear tendency appears; the dry specimens (B0) have greater modulus and phase angles for all of the test frequencies. In the case of the submerged series, B2 is more elastic than B1.

As in the case of the B-Series, for the C-Series, C0 has greater values of modulus, followed by C2 and finally by C1. However, in the case of the phase angles, the trend is different; the greatest angle is C2 and the lowest is C0. For the C-Series, the values of the modulus are greater, and the values of phase angles are lower compared with the A-Series and B-Series, and this indicates that the specimens have suffered a stiffening process.

In general, for all of the series and frequencies tested, the modulus values are greater for the dry series. This was expected due to the fact that submerging specimens in water is more harmful for the mixture than keeping them dry during any temperature interaction. Likewise, for all of the temperature interactions, there exists a trend which indicates that the series that are submerged in salt water have greater values of modulus than their corresponding pair submerged in distilled water, even more for the series subjected to freeze–thaw cycles. This trend supports the idea that the largest damage to the mixture that is submerged in water is the amount of time that it remains in frozen water, and the specimens that are submerged in salt water remain in frozen water for a shorter period of time.

## 3.4. Fatigue Test

The fatigue lines (Strain—Number of Load Cycles) obtained for all of the series are very similar, reaching notably high $R^2$ values; although, in the case of the C-Series, it is slightly lower. This may be due to the fact that the temperature interaction process is longer than in the A-Series and B-Series.

In the A-Series, for low values of strain, the submerged series, especially A1, obtain more load cycles than the dry specimens (Figure 4).

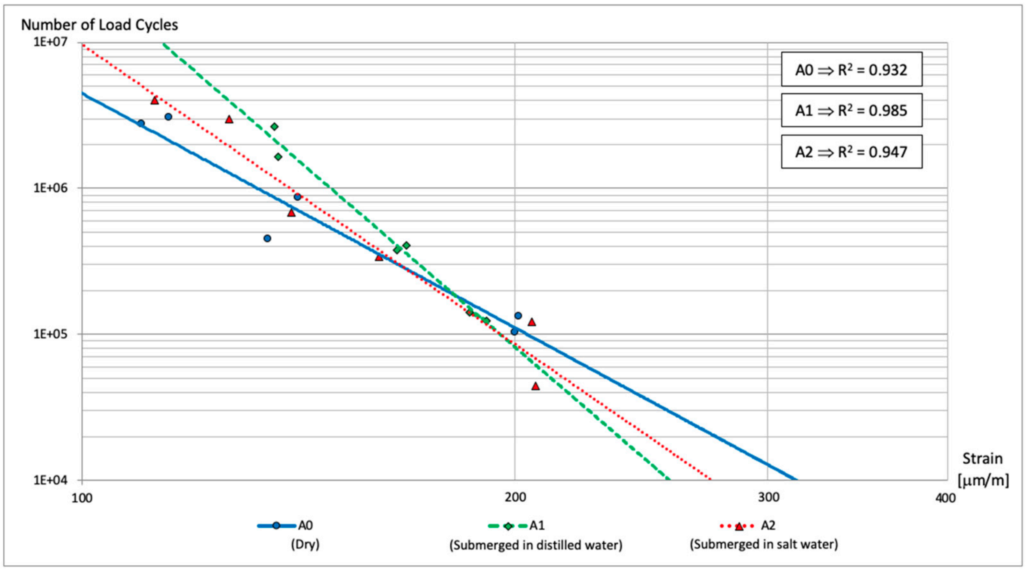


**Figure 4.** Fatigue test: specimens at constant temperature of 20 °C.

In the case of the series subjected to five freeze–thaw cycles (B-Series), the one that is submerged in salt water (B2) obtains more load cycles for any strain range than the other two series (Figure 5). This fact may be due to two main reasons. The first is that salt water offers a cushion effect on the temperature variations due to its thermal conductivity, which is smaller than that of distilled water [36]. The second is that the specimens of this series are in contact with frozen water for less time, which, as corroborated by the rest of the tests, is one of the most harmful effects for the mixture.

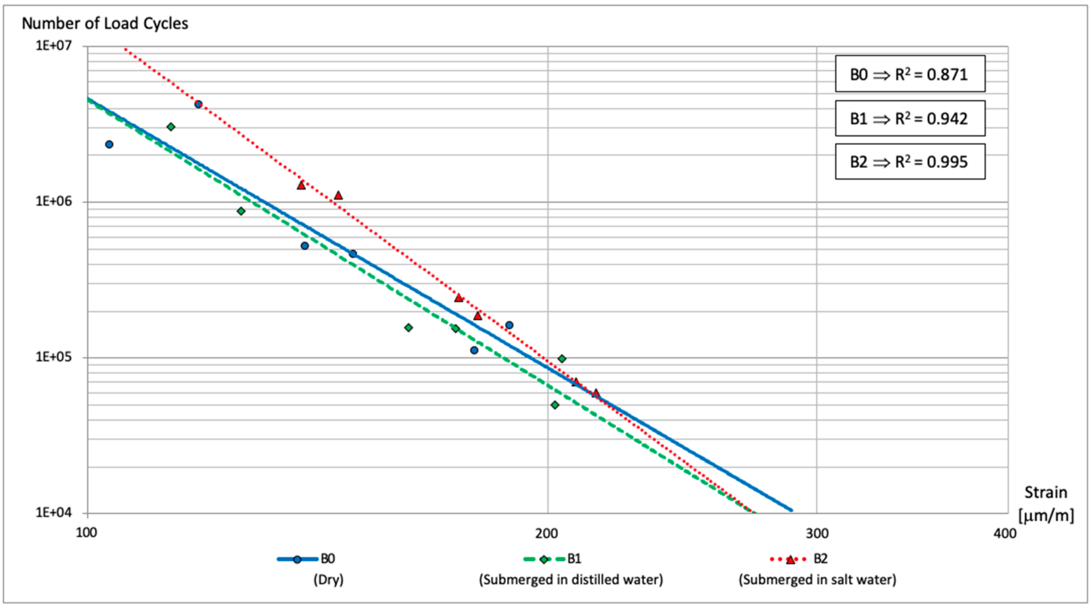


**Figure 5.** Fatigue test: specimens subjected to five freeze–thaw cycles.

The series that have been subjected to a year outdoors (C) obtain similar values for all specimens, being for C1 the number of load cycles slightly lower for high strain values (Figure 6).

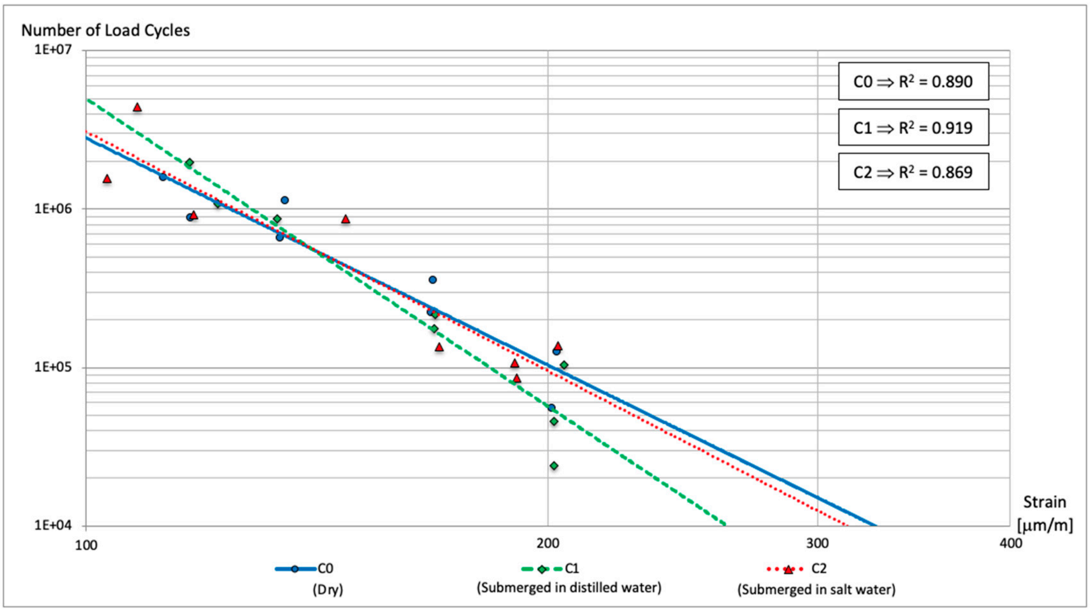


**Figure 6.** Fatigue test: specimens subjected to one of year outside storage.

## 4. Conclusions

Salt water reduces the time that the mixture is in contact with frozen water, which, as corroborated by the results, is one of the most harmful effects for the mixture.

When the bituminous mixture is subjected to freeze–thaw cycles, salt water has a protective effect on the specimens that remain submerged in it. The salt in the water protects the aggregate-binder adhesive, maintains the mechanical strength and increases the number of load cycles for any strain range.

The results of the Wheel Track Slope and the Rut Deep for reference mixtures and those that remained outdoors for one year are suitable for most climates zones and traffic.

The results of the Fatigue Test and Dynamic Modulus Test have no significant variation between the different series analysed.

The results show that, although the temperature has an injurious effect on the mechanical properties, the specimens submerged in salt water obtain better results than their analogs that are submerged in distilled water.

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