# ANALYSIS OF THE MOISTURE SUSCEPTIBILITY OF HOT BITUMINOUS MIXES BASED ON THE COMPARISON OF TWO LABORATORY TEST METHODS

# ANÁLISIS DE LA SUSCEPTIBILIDAD AL AGUA EN MEZCLAS BITUMINOSAS EN CALIENTE MEDIANTE EL ESTUDIO COMPARATIVO DE DOS MÉTODOS DE ENSAYO DE LABORATORIO

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**ABSTRACT:** One of the most common road pavement pathologies is caused by water action. The presence of moisture causes a reduction in aggregate-binder adhesion in the mix as well as in the internal cohesion of the bitumen. This leads to problems such as pot holes, aggregate peeling, stripping, etc., which eventually lead to the structural failure of the pavement. Currently, there are numerous laboratory tests that analyze the susceptibility of bituminous mixes to moisture, providing a qualitative or quantitative evaluation.

This study analyzes the performance of bituminous mixes in different experimental conditions. For this purpose, a comparative study of two laboratory tests was carried out. The tests differed in mix compaction method, the conditioning of the test specimens, and the type of load applied. The results obtained showed that in these tests the conditioning temperature had a greater impact on mix performance than the temporal duration of the conditioning process. Furthermore, the application of tensile stress was found to be more suitable for studying moisture susceptibility since mixes were found to be more sensitive to this type of load.

Key Words: Hot bituminous mixes, road, mix design, moisture susceptibility.

**RESUMEN:** Unas de las patologías más comunes en los firmes flexibles de carretera de todo el mundo son las provocadas por la acción del agua. La presencia de éste elemento provoca una disminución en la adhesividad entre los áridos y el ligante de la mezcla, así como de la cohesión interna del betún, que se traducen en problemas de desprendimiento de áridos, baches, peladuras, etc., que terminan provocando el fallo estructural del firme. Actualmente existen numerosos ensayos de laboratorio que permiten analizar la susceptibilidad al agua de las mezclas bituminosas, proporcionando una medida cualitativa o cuantitativa. Así, desde esta investigación se ha pretendido analizar el comportamiento de las mezclas bituminosas ante diferentes condiciones de ensayo. Para ello se ha llevado un estudio comparativo entre dos de estos ensayos, los cuales difieren en la forma de compactación de la mezcla, el proceso de acondicionamiento de las probetas y el tipo de carga aplicado. Los resultados obtenidos pusieron de manifiesto que, para los ensayos estudiados, la temperatura de acondicionamiento ejerce mayor influencia en el comportamiento de la mezcla que el tiempo de acondicionamiento. Además, la aplicación de cargas de tracción resultan más adecuadas para el estudio de la susceptibilidad al agua de las mezclas bituminosas ya que las mezclas son más sensibles a éstas.

Palabras Clave: Mezcla bituminosa en caliente, carretera, diseño de mezcla, susceptibilidad al agua.

### **1. INTRODUCTION**

Bituminous mixes are some of the most frequently used materials in road construction all over the world [1]. This material is a conglomerate with a mineral skeleton composed of a coarse fraction with a diameter of more than 2 mm, a fine fraction with a diameter of 0.063 - 2 mm, and a mineral powder fraction with a diameter of less than 0.063 mm. It is also composed of a hydrocarbon binder that gives cohesion to the mineral skeleton (bitumen or one of its byproducts from the distillation of crude oil), and sometimes, a

series of additives (fibers, waxes, etc.) that improve the characteristics and mechanical performance of the mixes (greater resistance to plastic deformation, lower susceptibility to fatigue cracking, rutting, raveling, etc.) [2].

In order for the mix to be used in the road surface course, it must pass a series of laboratory tests that certify its suitability and good performance under various types of load or stresses to which it will be subjected during its service life (traffic loads, extreme temperatures, water action, etc.) [3]. Of these stresses, those produced by water action are among the main causes of the pavement deterioration (loss of aggregate, peeling, pot holes, etc.). The presence of moisture in the mix reduces aggregate-binder adhesion (i.e. destruction of the chemical bonds between the binder and the aggregate) [4-10], as well as its cohesion (the emulsification of the binder due to water action separates its particles) [11-14]. This leads to a loss of aggregate, which in the medium and long term, and combined with other negative factors (traffic loads, ice formation, binder ageing, etc.) finally cause most road pathologies and the eventual failure of the road surface course [15-17].

As seen in Lu and Harvey [18], one of the key ways to mitigate the effect of water action on mixes is the design and application of laboratory tests capable of predicting the potential moisture damage during the service life of the pavement. This makes it possible for the mix to have an optimal design. Methods to predict the effect of moisture action on the bituminous mixes were initially developed in the 1930s, and there are now a wide range of such tests available [19-27]. A description of these methods can be found in Solaimanian et al. [28].

The objective of such methods is to reproduce mix performance in laboratory conditions when it is affected by water. They vary in reliability, depending on the characteristics of the test, the mix type, as well as the environmental conditions of the use case (cold or hot temperatures, dry or rainy weather, etc.). Most of these tests are performed with fixed temperatures and loads. This means that it is impossible to simulate the effect of moisture on the pavement when it is subjected to varying traffic loads and weather conditions [22]. Nevertheless, these experiments are often very complicated and timeconsuming to perform. Moreover, they are somewhat limited in scope when they try to reproduce the way that moisture attacks mixes. It is thus necessary to analyze and compare the effect of these laboratory tests on the mix in order to evaluate its response. Only in this way will it be possible to predict the performance of a mix when it is actually used in road construction.

In the laboratory, methods for testing moisture susceptibility differ in the mix compaction method, the type of conditioning, and the application of the load. Although there are many tests used for this purpose (e.g. Immersion-Compression, Hamburg Wheel Tracking, Moisture Vapor Susceptibility, Marshall Immersion, etc.), some are more effective than others. Today there is a division of opinion regarding which test is the most suitable for studying the susceptibility of bituminous mixes to moisture action. Consequently, in this study a comparative analysis was made of two of these methods, and the performance of the mix was analyzed in terms of characteristics, such as type of test specimen, compaction method, conditioning process, and load application. In this way it was possible to arrive at conclusions concerning the representativity of the results of each test.

The laboratory tests selected were two that are commonly used in Spain: a) the Immersion-Compression Test (regulated by the Spanish Technical Standard NLT-162); b) the Water Sensitivity Test (regulated by the European standard UNE-EN 12697-12 and implanted as a new reference test to analyze the susceptibility of bituminous mixes to moisture in all European Union countries). Both tests differ in so far as the type of test specimen used (dimensions and compaction methods), conditioning process, and applied load. However the result of both tests is a retained strength value, which makes it possible to compare them.

This article analyzes the results obtained after performing the Immersion-Compression Test and the Water Sensitivity Test on samples of the same mix type with an identical mineral skeleton and the same percentage of bitumen. The number of samples studied had to be sufficient to statistically validate the results obtained and thus extract representative conclusions. A total of 33 samples were used, and the central limit theorem was applied. The results of both laboratory tests were statistically analyzed, as well as the mix response in each.

#### 2. METHODOLOGY

#### 2.1. Materials

The semi-dense mix used in the study was regarded as representative, a generic asphalt concrete that is commonly found in all type of road surfaces (regardless of climate or traffic loads) throughout Europe. It had a continuous grain size with a strong mineral skeleton, and its bitumen content was 4-5% of the total mass. This made it resistant to plastic deformations and water action. More specifically, the mix used was an AC 16-S mix for the surface course [29], manufactured from ophite and limestone aggregate and penetration bitumen 40/60.

The samples were made in different batches though the characteristics of the mix did not vary. The mineral skeleton of each batch was within the grain-size limits specified in the Spanish regulations [29], which establishes a reference particle size for this type of mix. Nevertheless, in our samples, the mineral skeleton was slightly modified to obtain different air void contents so as to better study the effect of the water volume on the mix.

The aggregate used in the mix was ophite for the coarse fraction and limestone for the fine fraction as well as mineral dust. The characteristics of this aggregate are in accord with the PG-3 General Technical Specifications for Roads and Bridge Works in Spain[29].

The filler fraction chosen for the mix was limestone, given its good response to water action. This is due to the fact that the adhesion of limestone with the binder is greater since its alkaline surface reacts better to the acid composition of the bitumen [30]. The limestone had an apparent density in toluene of 0.67 g/cm3 in accordance with the NLT-176.

The manufacturing temperature of the mix was 160-165°C, whereas the compaction temperature of the test specimens was 150-155°C. The optimal bitumen content used to make the specimens was 4.5% of the total weight of the mix (based on the Marshall Test results of the job mix formula).

### 2.2. Experimental design

The experimental design was based on the comparison of the Immersion-Compression Test (NLT-162) and the Water Sensitivity Test (UNE-EN 12697-12).

Both tests offer a similar result (retained strength). Nevertheless, test conditions (compaction method, specimen conditioning, and load applied) are not the same, so they can be used to analyze and compare the performance of the mix to water action

In this study 33 samples of the same mix type (AC 16 - S) were tested. The specimens had identical characteristics but belonged to different batches. They were all subjected to the Immersion-Compression Test and the Water Sensitivity Test. The number of samples was based on the central limit theorem. According to this theorem, when the number of samples of a population is sufficiently large (i.e. more than 30 [31-33]) it is assumed to have a normal distribution. It is thus possible to derive scientifically valid conclusions from the results obtained. The mix response in the two tests was statistically analyzed, and it was possible to observe the representativity of each regarding the mix's susceptibility to moisture action.

The Immersion-Compression Test involves the manufacturing of 10 test cylinders with a diameter of 101.6 mm and a height of 101.6 mm. These cylinders are compacted by means of a static load produced by a double plunger. There is an initial pre-load of 1 MPa (in order to settle the mix) after which the load applied steadily increases for 2.5 minutes until reaching 21 MPa. Then the density of the samples is determined by the saturated surface dry method. As part of the conditioning process before the test, the specimens are divided into two sets of five. One set is submerged in a container with water at 60°C for 24 hours, whereas the other set is left at room temperature. After 24 hours, both sets of specimens are subjected to a simple compression load at a constant deformation velocity of 5.08 mm/min until they break. In this way it is possible to obtain a mean retained strength value for each set. The result of this test, which measures the specimens' sensitivity to water action, this is known as the retained strength index. This index (expressed in %) is calculated by dividing the strength obtained by the group immersed in water at 60°C by the strength obtained by the dry set.

The Water Sensitivity Test involves manufacturing 6 specimens with a diameter of 101.6 mm and a height of 60 mm. The specimens are compacted with an impact compactor by applying 50 blows on each

face. Then the density of the samples is determined by the saturated dry surface method. As part of the conditioning process, the specimens are divided into two sets: a wet set and a dry set. The dry set is stored at room temperature in the laboratory ( $20\pm5$  °C), whereas a vacuum is applied to the wet set for 30±5 minutes until a pressure of 6.7±0.3 KPa is reached. Then, the specimens in this set are left in immersion conditions of 40°C for 72 hours. Afterwards, the indirect tensile fracture of each specimen (of the dry set as well as the wet set) is performed at a temperature of 15°C after the specimens have previously been acclimated to this temperature for 120 minutes. The division of the tensile strength obtained by wet specimens, by the tensile strength obtained by dry specimens, results in the retained indirect tensile strength (expressed in %).

In this study, the relation between the variables in each test and the mix response was obtained by using a simple linear regression.

Once the straight regression line is fit to the cloud of data points, it is crucial to have a measurement of the goodness of fit, which shows whether the fit is sufficient. The determination coefficient was used for this purpose. When a simple linear regression is performed, this coefficient coincides with the square of the linear correlation coefficient. The determination coefficient is interpreted as the percentage variation of the dependent explained by the model. It is denoted by  $R^2$ , and since  $0 < R^2 < 1$ , this measurement is usually expressed as a percentage. In this type of study, the tests were very heterogeneous, and this meant that the results of the tests were, as well. Consequently, the determination coefficients of the linear fit lines were not very high.

Apart from the simple linear regressions, and given that both tests had different characteristics, the retained strength values were studied according to a normal distribution in order to reinforce the comparative analysis.

#### 3. ANALYSIS OF RESULTS

The study described in this paper was the result of twelve months of laboratory work and shows the results obtained for the 33 samples in the ImmersionCompression Test and the Water Sensitivity Test in relation to the mean values of the wet and dry sets of samples for fracture strength, density, and air void content of the test specimens.

Coefficient of variation (CV) is calculated for the Immersion-Compression Test and the Water Sensitivity Test for fracture strength, density, and air void content for both wet and dry sets obtaining in all cases similar deviations (see tables 1 and 2). Similar results in retained strength values (%) were obtained in both tests as is resumed in table 3.

Table 1.CV Immersion-Compression test.

Compression Strength		Density		Air Void Content	
Dry Set	Wet Set	Dry Set	Wet Set	Dry Set	Wet Set
0.1844	0.1809	0.0222	0.0226	0.3107	0.3190

Table 2. CV Water Sensitivity test.

Indirect Tensile Strength		Density		Air Void Content	
Dry Set	Wet Set	Dry Set	Wet Set	Dry Set	Wet Set
0.1790	0.2065	0.0251	0.0243	0.3508	0.3387

 Table 3.CV Retained strength in the tests.

Retained strength				
Immersion-Compression Test	Water Sensitivity Test			
0.0279	0.0239			

Since the samples in the study were manufactured with the same materials (aggregate and bitumen), there is a close relation between the air void content and the density of the specimens in both tests (despite the fact that they differ in compaction method). Accordingly, the samples with a lower density have a greater content of air voids in the mix.

As can be observed in the results, in the Immersion-Compression Test, the air void content and the density of the specimens do not have a significant effect on the simple compressive strength (Figure 1). Therefore, a dispersed cloud of data points, and the extremely low determination coefficients of the regression lines, reflect the independence of both characteristics in the mix response to compression forces. This is due to the fact that in bituminous mixes, compression forces are endured mainly by the internal friction of the mineral skeleton. Consequently, the strength of the specimen mainly comes from the aggregate's capacity to withstand the transmitted load, and is independent of the moisture content.

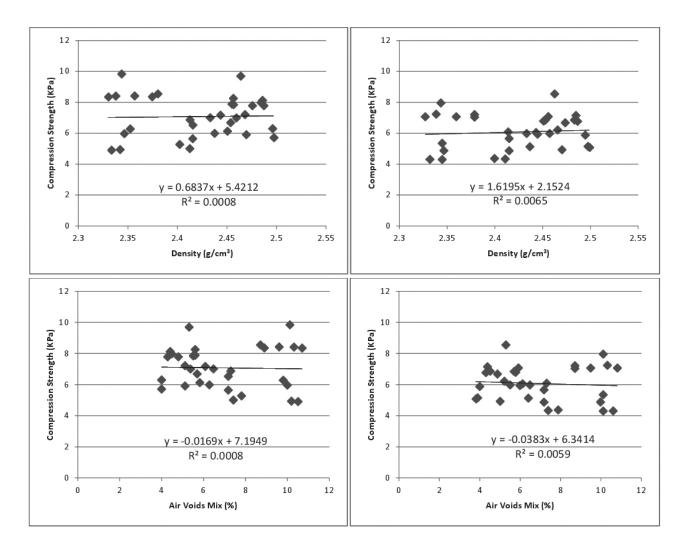


Figure 1.Simple compression strength based on the density and air void content of the mix: dry set (left) and wet set (right)

In contrast, in the Water Sensitivity test, there is a closer relation between the air void content and density of the specimen and its indirect tensile strength. This is reflected by a higher determination coefficient in the linear regression lines (see Figure 2). This tendency implies that when a specimen has a lower density or a higher air void content, its strength is reduced. In this case, the application of the load varied, and the specimen was subjected to tensile stresses such that the internal cohesion of the mix (the sum of binder cohesion and the aggregate-binder adhesion) was the basis of the specimen's fracture strength. This strength decreased as the air void content of the specimen increased. This was because the quantity of air voids (spaces without aggregate or bitumen) on the plane where the break occurred meant that there was less surface to withstand the strain. The test specimen was thus weaker.

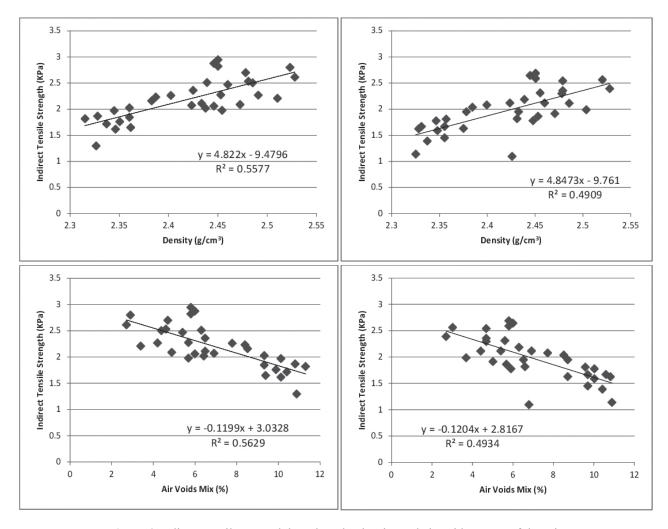


Figure 2.Indirect tensile strength based on the density and air void content of the mix: dry set (left) and wet set (right)

Moreover, it should be noted that the indirect tensile strength of the specimens (in the Water Sensitivity Test) was less than the simple compression strength (in the Immersion-Compression test). This response is logical since concrete-type stone materials, such as bituminous mixes, perform much better under compression loads than under tensile stresses. This is due to the fact that the mineral skeleton makes up about 90% of the weight of the bituminous mix, whereas the binder, which is the source of tensile strength, is only about 5% of the weight of the mix.

Furthermore, the retained strength values (%) obtained in both tests are shown in Figure 3.

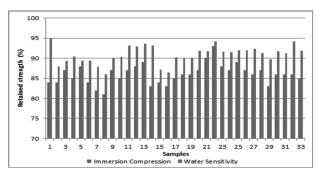
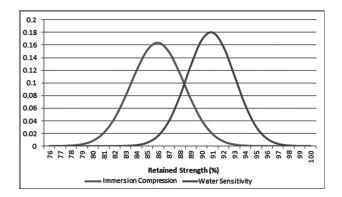
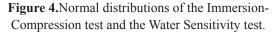


Figure 3.Retained strength of the samples studied in the tests.

As can be observed, the retained strength in the Water Sensitivity Test was greater than in the Immersion-Compression Test. In fact, it was 5% higher, based on the mean value of the 33 samples. When the retained strength values obtained in each test were compared by using a normal distribution (based on the central limit theorem), this difference in the values can be observed clearly (Figure 4). Thus, in the Water Sensitivity Test, this effect was less than in the Immersion-Compression test. The analysis also showed that the Gaussian bell curve in the Water Sensitivity Test is sharper, which means that the results are more clustered. The samples thus have a lower variability, which signifies that the test is more reproducible (and thus its results are more representative).





According to these results, the simple compression strength of the bituminous mix was potentially more affected by moisture action than its indirect tensile strength was. However, the simple compression strength mainly depends on the internal friction of the aggregate, and the indirect tensile strength on the internal cohesion of the mix, which is the sum of binder cohesion and aggregate-binder adhesion. Both factors are significantly more sensitive to moisture action than the internal friction of the aggregate (i.e. the presence of water can break the chemical bonds in the bitumen as well as reduce its adhesion to the aggregate) [3-13]. As a result, the retained strength of the Water Sensitivity Test should be lower. This means that the conditioning undergone by the wet set of specimens in the Immersion-Compression Test (24 hours at 60 °C) was more aggressive than that undergone by the wet set in the Water Sensitivity Test (72 hours at 40 °C). The temperature of the conditioning had a greater influence on mix performance than the temporal duration of the conditioning.

The compaction method was the other variable in which the tests differed, apart from the conditioning

of the specimens and the type of fracture. However, it did not seem to have a significant influence on the results obtained. In this respect, the densities of the 33 samples in both studies were very similar, though they were slightly higher in the case of the Immersion-Compression Test (Figure 5).

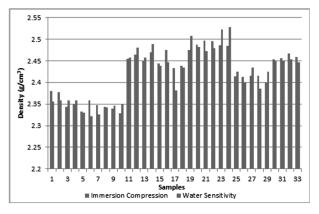
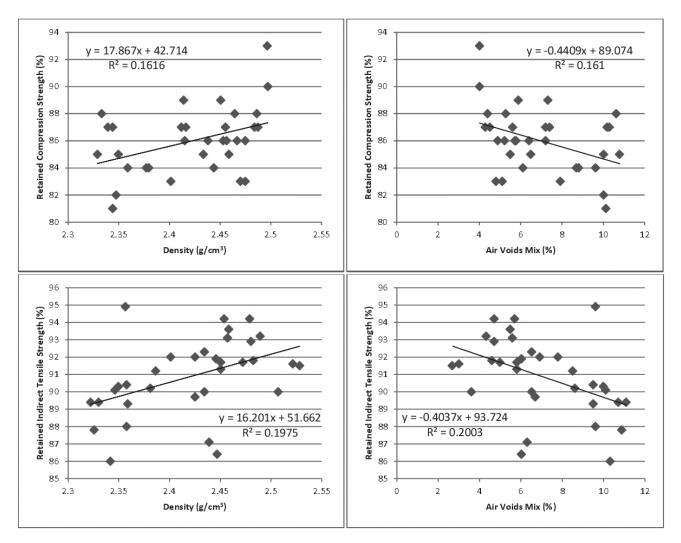


Figure 5.Mean density of the sample specimens studied in each test.

Finally, in the analysis of the influence of the air void content and the density, on the retained strength, it was observed that both tests showed the same tendency. The greater the density (i.e. lower air void content) of the specimens, the greater was the retained strength of the samples (Figure 6). In other words when the air void content of the mix increased, the volume of water that penetrated it was greater. This significantly affected mix performance since it reduced simple compression strength as well as indirect tensile strength.

In the Immersion-Compression Test, the relation between the air void content and the retained strength of the mix was greater than that between the air void content and the simple compression strength (larger determination coefficient). The higher percentage of air voids caused the mix not to respond as well to water action. However, the simple compression strength remained similar to that of a mix with a lower percentage of air voids.

In the Water Sensitivity Test, the relation between the air void content and the indirect tensile strength of the mix was greater than that between the air void content and the retained strength (larger determination coefficient). This shows that an increase in the air void content of the mixes had a greater impact on its capacity to resist indirect tensile stresses than on



its susceptibility to water action. Despite this, the percentage of air voids affected the retained strength of the mix in the Water Sensitivity Test more than in the Immersion-Compression Test. Consequently, it seems that it could be more sensitive to moisture action.

Figure 6.Retained strength based on the air void content and the density of the mix. Immersion-Compression Test (upper graph) and Water Sensitivity Test (lower graphs)

## 4. CONCLUSIONS

This paper shows the results obtained in a research study that analyzed the moisture susceptibility of hot bituminous mixes based on two different test methods. For this purpose, a comparative study of two laboratory tests was performed, which evaluated the effect of water action on the mechanical performance of the mix. The tests selected for this study were the Immersion-Compression Test and the Water Sensitivity Test. Although these tests differ in the size and compaction method of the test specimens (static load/compaction by blows), their wet conditioning (24 hours at 60°C/72 hours at 40°C), and the type of load applied (simple compression/indirect tensile stress).Both evaluated the same parameter, the retained strength value of the wet set and dry set of specimens. Thus, in order to guarantee the reliability of this analysis, we used the central limit theorem (which defines whether the sample population is sufficiently large to assume the normality of the results). Accordingly, the test was carried out on a total of 33 specimens made from bituminous mixes of identical characteristics. The type of mix selected was a standard semi-dense mix that is widely used in many

European countries. Based on the results obtained in our research, the following conclusions were derived:

- For the type of mix analyzed, the simple compression strength of the bituminous mixes was not affected by the density and air void content, but rather by the strength of the mineral skeleton and internal friction. Thus, the fact that the mix has voids where water could be stored was not a determining factor that affected its compression strength. Consequently, the type of load used in the Immersion-Compression Test was not suitable to measure the susceptibility of a bituminous mix to water since the presence of air voids did not affect its response.
- The indirect tensile strength was found to be directly related to the density and air void content of the mix. This means that the lower the density of the mix (or the greater its air void content), and the lower its indirect tensile strength. When the density of the mix became lower, this reduced the cohesion of the mix. The cohesion is what withstands the tensile stresses on the mix. Moreover, since the mix is a stone-like material, its strength against this type of load is not as great as its strength against simple compression loads. As a result, the mechanical performance of the mix is much more sensitive to this type of stress. Thus, indirect tensile stress is better for evaluating the possible moisture damage to the mixes.
- Even though the tests used different compaction methods for the specimens, this did not influence the characteristics of the mix since the samples in both tests produced similar results regarding their density and air voids.
- The type of conditioning given to specimens in the Immersion-Compression Test significantly affected their performance since the retained strength values obtained in this test were lower than those obtained in the Water Sensitivity Test. This seems to indicate that conditioning at high temperatures is more aggressive than conditioning for a longer duration. It was thus found that the effect of the temperature on the mix was greater than that of the duration of the conditioning process.
- The normal distribution obtained from the retained strength results of the Water Sensitivity Test had a

sharper peak than that obtained from the Immersion-Compression Test. This indicated that the results had a lower dispersion, and thus, the reproducibility of the test was greater (which points to the fact that the results are more reliable).

- Both tests showed that when the mix had a greater air void content (and thus was less dense), its retained strength was also lower.
- The Immersion-Compression Test showed that these characteristics of mix did not affect its simple compression strength, but did have an impact on its retained strength. This was due to the fact that the increased air void content in the mix caused a larger volume of water to enter. This affected the mechanical performance of the mix and reduced its strength.
- The results of the Water Sensitivity Test showed that the air void content had a greater effect on the indirect tensile strength of the mixture than on its retained strength. Despite this, the retained strength was more closely related to the air void content than the retained strength of the Immersion-Compression Test. This was because the tensile stresses in bituminous mixes are withstood by their cohesion, which is the sum of the binder cohesion and of aggregate-binder adhesion. Both factors are directly affected by the presence of moisture (which breaks the chemical bonds in the bitumen, thus reducing its cohesion as well as its adhesion with the aggregate). For this reason, a larger number of air voids in the mix permits the presence of moisture that reduces the strength of the mix.
- The results obtained in this study point to the fact that the Water Sensitivity Test is more suitable for analyzing the susceptibility of bituminous mixes to water than the Immersion-Compression test. For this reason, the results obtained with the Water Sensitivity Test were more representative of the performance of the mix during its service life.

As a continuation of this research, it would be interesting to make a more in-depth study of the design of more effective laboratory tests for determining the susceptibility of bituminous mixes to moisture. For this purpose, it would be useful to perform further comparative analyses with other laboratory tests and other types of bituminous mixes.

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