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Effect of aggregate–bitumen compatibility on moisture susceptibility of asphalt mixtures

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Stripping of an asphalt mixture is reached when the interaction between bitumen and aggregate weakens by effect of water. The identification of bitumen–aggregate pairs with higher adhesion is key to obtain pavements that are more resistant to stripping. In order to research the adhesion and debonding processes further, several bitumen and aggregate combinations were prepared and their compatibility was estimated by means of surface energy measurements. Additionally, the water susceptibility of the proposed bitumen–aggregate combinations was mechanically tested by means of loss in storage modulus of samples exposed to water. It was found that the compatibility of water to bitumen and aggregate describes the mechanical behaviour of the asphalt mixture. One of the features of this study is the evaluation of the effect of physicochemical properties of bitumen and aggregates on the stripping susceptibility of the given combinations and the corresponding correlation to the field performance of an asphalt mixture.

Keywords: bitumen–aggregate compatibility; moisture damage; asphalt mixture; dynamic modulus; surface energy

Introduction

Moisture damage is a failure mode that affects a large number of pavements throughout the world. The process starts in the bottom of the asphalt layer, by weakening the adhesive bond between bitumen and aggregate, and the cohesive bond of the asphalt mastic (Das, Baaj, Kringos, & Tighe, 2015). The progression of moisture damage in a pavement is reflected in the stripping of bitumen, ravelling of aggregates, proneness to deformation and cracking, among others (Canestrari, Cardone, Graziani, Santagata, & Bahia, 2010; Caro, Masad, Bhasin, & Little, 2008; Cho & Kim, 2010; Hicks, Santucci, & Aschenbrener, 2003; Meor, Muhammad-Rafiq, & Mohd-Rosli, 2015). Therefore, the consequences of moisture damage might be as severe as the total loss of the asphalt mixture (Airey & Choi, 2002). From a broader perspective, moisture damage can be considered as a distress affecting society in many ways. Economically, moisture damage costs millions of dollars in terms of required maintenance and rehabilitation of the affected pavements (Abuawad, Al-Qadi, & Trepanier, 2015). Furthermore, in many cases, additional investments are required to prevent damage such as the use of antistripping agents (Hicks et al., 2003). The resulting distresses from moisture damage reduce the durability of roads, which impacts the

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quality of life of users, and in the overall perception of the national infrastructure which can severely impact a country's competitiveness. Consequently, it is imperative to characterise the moisture susceptibility of asphalt mixtures, as well as to predict the sensitivity of a particular mixture to deteriorate in the presence of water. Therefore, the main objective of this study is to estimate the bitumen–aggregate compatibility in hot mix asphalt by means of physicochemical measurements, and to relate the results to the mechanical changes in the mixture associated with moisture-induced damage.

Background

Mechanical evaluation of moisture damage susceptibility

An asphalt mixture exposed to moisture experiences several physical and chemical processes that deteriorate its fundamental properties. Such processes occur simultaneously by means of mechanisms which are not fully understood. As a result, achieving a mechanical test that reasonably simulates these distresses has become a challenge to many researchers. In this sense, the dynamic modulus of the asphalt mixture has shown good correlation to the observed rutting and fatigue cracking in field tests (Witczak, Kaloush, Pellinen, El-Basyouny, & Von Quintus, 2002), and has been recommended as a primary material characterisation test in a hot mix asphalt (Solaimanian, Bonaquist, & Tandon, 2007). For such reasons, determination of dynamic modulus might represent an enhancement in moisture susceptibility evaluation of specimens subjected to severe humid conditioning (Vargas-Nordbeck, Leiva-Villacorta, Aguiar-Moya, & Loria-Salazar, 2016).

The loss in strength of an asphalt mixture due to the effect of water might be related to the weakening in adhesion and cohesion of the asphalt mastic. It has been observed that mixtures with poor adhesion characteristics show poor mechanical performance, and that the presence of moisture in the mastic reduces its flexibility and binding properties (Das et al., 2015). In addition, other mixture properties such as high void content and large water diffusion coefficients (Castillo, Caro, Darabi, & Masad, 2016), as well as the ageing of the asphalt mixture (Tong, Luo, & Lytton, 2015), have shown to increase the susceptibility of mixtures to moisture-induced damage. Therefore, the failure of an asphalt mixture by effect of water cannot be related to a singular process. However, by characterising the properties of materials involved, the water resistance of a certain mixture can be approximated and related to its final field performance.

Compatibility between bitumen and aggregate

The bitumen and the mineral aggregates are two different materials which do not tend to adhere spontaneously at room temperature. The reason for such behaviour goes beyond the high viscosity of bitumen, which impedes its ascension by capillarity into the pores of the aggregate. More accurately, it is a matter of chemical compatibility. During hot mix asphalt production, the bitumen and the aggregate are forced to combine by increasing the temperature. Once the mix returns to typical ambient temperatures, the adhesive bond between bitumen and aggregate is defined by their compatibility: a bitumen–aggregate pair with good compatibility is believed to have better adhesion properties and to be less prone to collapse in the presence of water. Therefore, it is imperative to improve the quality of the interaction between bitumen and aggregate by improving their compatibility. This could be achieved by the proper selection of materials to be used in the asphalt mix design.

By accounting for the physicochemical surface characteristics of bitumen and aggregate (i.e. surface energy), it is possible to estimate the blend's compatibility. The surface energy is a

property inherent to all materials and is defined by the nature and magnitude of the chemical interactions occurring at the surface. By definition, the total surface energy (γ^T or simply γ) is the sum of dispersive (γ^{LW}), acidic (γ^+) and basic (γ^-) interactions (Equation 1) (Van Oss, Chaudhury, & Good, 1988).

$$\gamma^T = \gamma^{LW} + 2(\gamma^+\gamma^-)^{1/2}. \quad (1)$$

The surface energy of the individual components such as bitumen, aggregate and water is also useful to estimate the interactions taking place in a hot mix asphalt, such as adhesion (denoted as work of adhesion, W_{AB}) (Equation 2) and stripping potential of the bitumen in the presence of water (denoted as work of debonding, W_{wet}) (Equation 3).

$$W_{AB} = \gamma_A + \gamma_B - \gamma_{AB}, \quad (2)$$

$$W_{wet} = \gamma_{AW} + \gamma_{BW} - \gamma_{AB}, \quad (3)$$

where the subscripts A, B and W refer to aggregate, bitumen and water, respectively; γ_{AB} , γ_{AW} and γ_{BW} are the energies (in mJ/m^2) of the aggregate–bitumen, aggregate–water and bitumen–water interfaces, respectively.

A bitumen–aggregate pair with good compatibility will exhibit higher values of work of adhesion. However, simultaneous to the bitumen–aggregate adhesion process, water is also competing to reach a thermodynamic favourable condition in detriment of the adhesive bond between bitumen and aggregate. In order to avoid the latter, the work of debonding must be as low as possible. Since the adhesion–debonding processes occur simultaneously, the energy ratio parameter (ERP) (Equation 4) acts as a convenient parameter to estimate the resistance of the bitumen–aggregate interface to collapse in the presence of water. The higher the ERP of a given blend, the more resistant it will be to develop moisture-induced damage (Bhasin, Little, Vasconcelos, & Masad, 2007).

$$\text{ERP} = \left| \frac{W_{AB} - W_{BB}}{W_{WET}} \right|, \quad (4)$$

where W_{BB} in Equation (4) refers to the internal cohesion of bitumen, which can be defined as per Equation (5) (Little & Bhasin, 2006).

$$W_{BB} = 2\gamma_B. \quad (5)$$

Materials and methods

Binder modification and mixture designs

A PG 70-22 bitumen source was modified with the following different materials: 2% SBS (styrene-butadiene-styrene polymer), 2% SBS + 1% Lime, 1% Lime and 0.5% liquid antistrip (LAS). The percentages are given by weight of bitumen, and in the case of Lime, the weight is given by weight of dry aggregate. During modification, the bitumen was heated to 185°C and the corresponding additive was incorporated with a shear stirrer for 1.5 h or until homogeneous.

Two sets of mixtures were designed and produced in the laboratory, based on two different aggregate sources (gravel). Ten mixtures were designed with aggregate source 1, and nine mixtures with aggregate source 2. The mixtures were produced using the previously mentioned modified bitumens, and the neat bitumen was used as control. Each set of mixtures was also designed using two different gradations having a nominal maximum aggregate size (NMAS) of 9.5 and 12.5 mm. The specific gradations, as well as the volumetric properties of the mixtures, are listed in Tables 1 and 2, respectively.

Table 1. Mixture gradations.

Sieve size (mm)	% Passing			
	Aggregate source 1		Aggregate source 2	
	9.5 NMAS	12.5 NMAS	9.5 NMAS	12.5 NMAS
19	100	100	100	100
12.7	100	95.4	100	95.9
9.51	95.0	78.3	94.7	84.7
4.76	60.0	43.3	49.0	48.5
2.38	40.0	28.9	32.7	32.6
1.19	25.0	20.0	22.5	22.4
0.595	17.0	14.8	16.0	16.0
0.297	10.0	10.9	11.4	11.4
0.149	7.0	8.0	7.8	7.9
0,074	5.0	5.8	5.6	5.6

Table 2. Mixture volumetric properties.

Aggregate source	Mixture	% Design AC ^a	%VMA ^b	% Air voids	% VFA ^c	Dust ratio
1 (9.5NMAS)	Control	6.5	15.5	4.0	73.7	1.0
	2% SBS	6.5	15.8	4.1	74.3	1.0
	2% SBS + 1% Lime	6.6	16.0	4.1	74.4	1.0
	1% Lime	6.7	16.3	4.2	74.4	0.9
	0.5% LAS	6.5	15.9	4.0	72.7	1.0
1 (12.5NMAS)	Control	7.0	15.8	4.0	74.5	1.1
	2% SBS	6.5	15.1	4.0	73.3	1.2
	2% SBS + 1% Lime	6.3	14.3	4.0	72.0	1.3
	1% Lime	6.0	14.0	4.0	71.5	1.3
	0.5% LAS	6.5	14.7	4.0	72.7	1.2
2 (9.5NMAS)	Control	6.6	15.6	4.0	74.5	1.1
	2% SBS	6.6	15.6	4.0	74.5	1.1
	2% SBS + 1% Lime	6.5	15.5	4.0	74.0	1.1
	1% Lime	6.5	15.6	4.0	74.4	1.1
	0.5% LAS	6.4	15.2	4.0	73.8	1.1
2 (12.5NMAS)	Control	6.2	14.9	4.0	73.3	1.2
	2% SBS	6.2	14.7	4.0	72.9	1.2
	2% SBS + 1% Lime	5.9	14.4	4.0	72.6	1.3
	1% Lime	5.9	14.5	4.0	72.2	1.3
	0.5% LAS	6.1	14.5	4.0	72.1	1.2

^aAC: Asphalt content.^bVMA: Voids in the mineral aggregate.^cVFA: Voids filled with asphalt.

Dynamic modulus testing

In order to simulate aggressive moisture-induced damage, the mixtures were exposed to 1, 3 and 6 freeze/thaw conditioning cycles. Each conditioning cycle consisted of keeping the vacuum-saturated specimens at -18°C for 16 h, followed by immersion in a water bath at 60°C for 24 h as per AASHTO T 283 (2014). The dynamic modulus (E^*) of the conditioned and unconditioned mixtures was measured based on AASHTO TP 79 (2012) by means of an asphalt mixture

performance tester. The results allowed estimation of the ratio between E^* on the conditioned specimens with respect to E^* on the dry specimens (E^*R).

The multiple conditioning levels, as well as the use of E^*R as a moisture-induced damage potential of asphalt mixtures indicator, have been found to be appropriate to more accurately predict the performance of such mixtures after being exposed to severe moisture conditions (Vargas-Nordbeck et al., 2016). Therefore, the same criteria were selected in this study to evaluate the mechanical response of the proposed mixtures after being exposed to water.

Surface energy measurements

The surface energy measurements of the materials used in this study were obtained by means of a goniometer. The samples of bitumen were prepared as follows: the bitumen was heated until fluid and then poured over a glass slide. The glass slide was introduced to an oven at 100°C and heated for 5 min, until a homogeneous and smooth surface of bitumen was formed. The aggregate samples were obtained from uncrushed river gravel which was later saw-cut, polished and washed with distilled water. The samples were allowed to dry in an oven for at least 2 h. The bitumen and aggregate samples were allowed to reach 20°C in a desiccator prior to testing.

For both bitumen and aggregate samples, a uniform solid surface was produced. Then, the contact angle formed between the solid surface and a drop of a probe liquid was measured (Aguar-Moya, Salazar-Delgado, Baldi-Sevilla, Leiva-Villacorta, & Loria-Salazar, 2015; Arabani & Hamedi, 2011; Little & Bhasin, 2006; Wei & Zhang, 2012). The mathematical transformation from contact angle to surface energy is given by Young–Dupré’s equation.

$$\gamma_S = \gamma_{SL} + \gamma_L(\cos \theta_{SL}), \quad (6)$$

where γ_S is the surface energy of the solid surface, γ_{SL} is the energy of the solid–liquid interface, γ_L is the surface energy of the probe liquid and θ_{SL} is the contact angle between the solid surface and a drop of the probe liquid.

The relationship between Young–Dupré’s equation and the individual components of the surface energy of materials is given by Equation (7) (Van Oss et al., 1988).

$$\gamma_L(\cos \theta_{LS}) = 2(\gamma_S^{LW} \gamma_L^{LW})^{1/2} + 2(\gamma_S^+ \gamma_L^-)^{1/2} + 2(\gamma_S^- \gamma_L^+)^{1/2}. \quad (7)$$

In the surface energy components of Equation (7), the subscripts L and S refer to liquid and solid, respectively. In order to obtain the three surface energy components of the bitumen and aggregates, a minimum of three probe liquids (for which their surface energy components are fully known) are required to compute a set of three linear equations, which can be solved from a matrix (Hefer, Bhasin, & Little, 2006). In this study, the surface energies of the proposed materials (bitumens and aggregates) were calculated using the values of the surface energy of probe liquids assigned by Della Volpe (Della Volpe & Siboni, 1997). The surface energy characterisation of the probe liquids and the data obtained for the binders and aggregates are listed in Table 3.

In addition to Equation (2), the work of adhesion between aggregate (A) and bitumen (B) can be estimated from their individual components of surface energy, based on Equation (8).

$$W_{AB} = 2(\gamma_A^{LW} \gamma_B^{LW})^{1/2} + 2(\gamma_A^+ \gamma_B^-)^{1/2} + 2(\gamma_A^- \gamma_B^+)^{1/2}. \quad (8)$$

Table 3. Surface energy of materials.

Material	Identification	γ^T (mJ/m ²)	γ^{LW} (mJ/m ²)	γ^+ (mJ/m ²)	γ^- (mJ/m ²)
Probe liquid ^a	Water	72.8	21.8	65.0	10.0
	Glycerine	64.0	34.4	16.9	12.9
	Ethylenglycol	48.0	31.4	1.58	42.5
	Formamide	58.0	35.6	1.95	65.7
Bitumen	Control	15.6	11.3	4.17	1.14
	2% SBS	15.9	12.8	3.69	0.669
	2% SBS + 1% Lime	15.9	12.1	3.29	1.12
	1% Lime	24.4	24.0	0.536	0.0829
	0.5% LAS	28.7	28.7	0.0213	0.00
Mineral aggregate	Source 1	27.9	15.0	6.91	5.99
	Source 2	14.9	6.35	7.26	2.51

^aBased on Della Voipe scale.

Similarly, the aggregate–water (AW) and bitumen–water (BW) interactions can be calculated by Equations (9) and (10), respectively.

$$W_{AW} = 2(\gamma_A^{LW} \gamma_W^{LW})^{1/2} + 2(\gamma_A^+ \gamma_W^-)^{1/2} + 2(\gamma_A^- \gamma_W^+)^{1/2}, \quad (9)$$

$$W_{BW} = 2(\gamma_B^{LW} \gamma_W^{LW})^{1/2} + 2(\gamma_B^+ \gamma_W^-)^{1/2} + 2(\gamma_B^- \gamma_W^+)^{1/2}. \quad (10)$$

Results and discussion

Mechanical behaviour of asphalt mixtures

The E^*R results for the analysed mixtures are shown in Figure 1. The values correspond to E^*R calculated at 20°C and 10 Hz to simulate typical pavement operating conditions, and also to establish a point of comparison between the mechanical response of mixtures with the surface energy results obtained at 20°C, as will be explained in the subsequent sections.

It was considered that an asphalt mixture with a minimum E^*R value of 0.8 will perform adequately under high moisture conditions. The value of 0.8 is used in this study as criteria to evaluate the good or poor performance of the proposed mixtures (20% decrease in a material property is considered as acceptable). Based on this requirement, it can be observed from Figure 1 that the mixtures produced with aggregate source 2 exhibited E^*R values higher than 0.8, including the mixture produced with neat bitumen. On the other hand, when the aggregate source 1 is combined with either neat or SBS modified bitumen, the resulting E^*R values are under the requirement. In general, the additives have a positive effect on almost all of the blends, since the resulting E^*R are higher than 0.8 for most of the samples. It can also be noted that the modified bitumens, particularly LAS-modified and Lime-modified, produce mixtures with higher resistance to loss of modulus by effect of water, even after repeated freeze–thaw conditioning cycles.

Regarding the conditioning cycles, it can be concluded from Figure 1 that the mixtures of aggregate source 2 are more resistant to the successive freeze/thaw cycles than those of aggregate source 1, for which 9.5 NMA mixtures experiment a higher modulus loss. These results suggest that aggregate source 2 is more appropriate than source 1 to produce mixtures that are resistant to the detrimental effects of water. However, it is important to highlight that observed differences in sample behaviour are not only due to the different mix designs, but that they might be explained by means of the chemical compatibility of the materials involved, as will be later discussed.

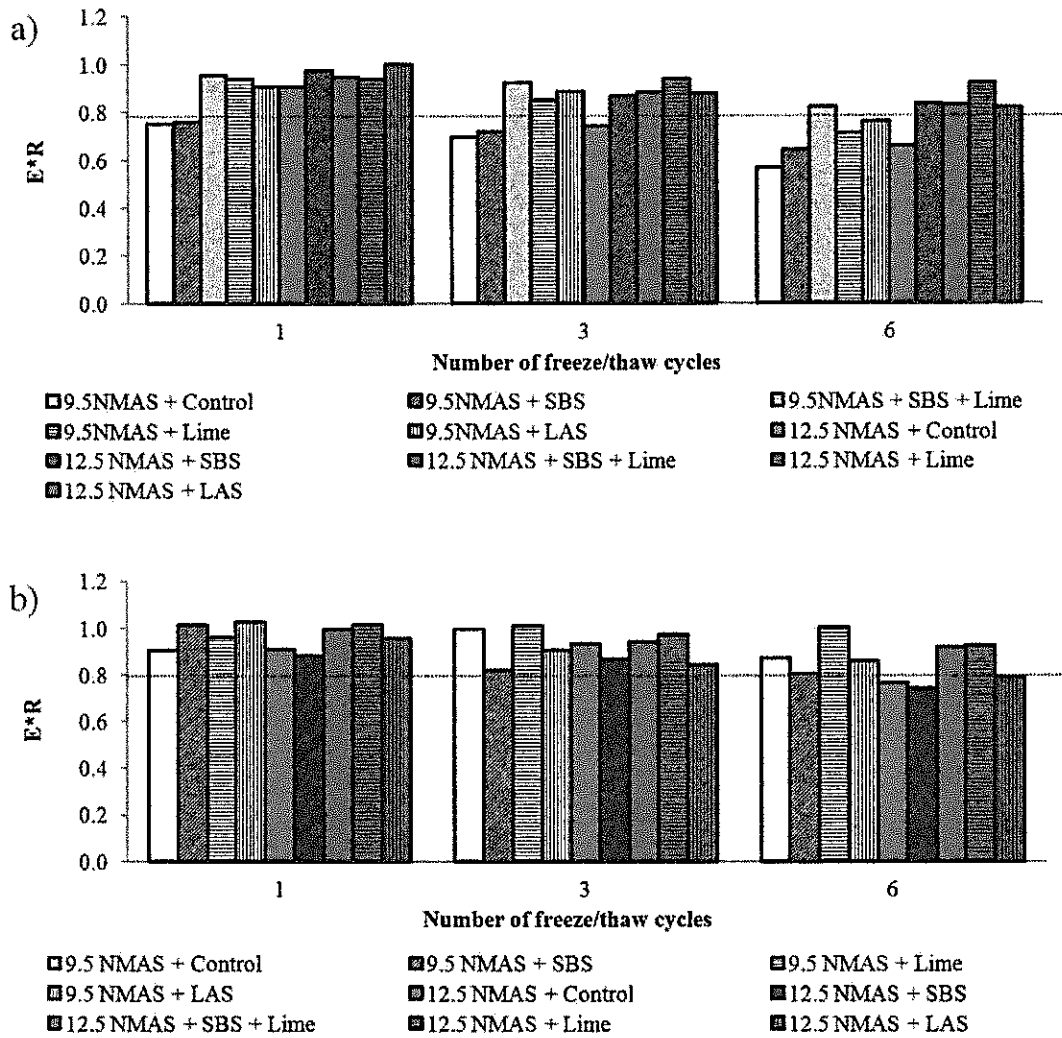


Figure 1. E^*R results for (a) mixtures designed with aggregate source 1 and (b) mixtures designed with aggregate source 2.

Physicochemical parameters related to moisture damage

The data in Table 4 show the results of the physicochemical parameters calculated from surface energy measurements.

Table 4. Physicochemical parameters of the binder–aggregate combinations.

Binder type	W_{BB} (mJ/m ²)	Aggregate source 1			Aggregate source 2		
		W_{AB} (mJ/m ²)	W_{wet} (mJ/m ²)	ERP	W_{AB} (mJ/m ²)	W_{wet} (mJ/m ²)	ERP
Control	31.3	41.7	33.5	8.3	29.2	42.7	0.05
2% SBS	31.8	41.4	36.0	8.3	28.5	44.8	0.07
2% SBS + 1% Lime	31.9	41.4	33.7	7.8	29.0	43.0	0.07
1% Lime	48.9	43.1	41.4	5.5	28.6	48.6	0.42
0.5% LAS	57.4	42.4	44.3	15.6	27.7	51.3	0.58

As previously mentioned, in order to consider a bitumen–aggregate interface as less susceptible to moisture-induced damage, its value of W_{AB} must be high, and its value of W_{wet} must be low. More importantly, the resulting ERP value must be as high as possible. From Table 4, it can be generally seen that the work of adhesion (W_{AB}) seems not to be significantly influenced by the presence of additives in the bitumen. However, the work of debonding (W_{wet}) seems to be more sensitive to the presence of the additives, and it is noticed that this value increases compared to the neat bitumen, mainly for the combinations involving Lime-modified bitumen and LAS-modified bitumen. Although the previous might suggest that those additives are increasing the compatibility of bitumen to water, the resulting ERP values increased for most of the combinations, which means that the proposed additives will produce bitumen–aggregate pairs less prone to collapse in the presence of water. It is noticed that the combinations involving Lime-modified and LAS-modified bitumen exhibited the higher ERP values. In fact, the LAS-modified bitumen significantly improves the resistance of the resulting interface in the presence of water, by increasing the ERP value of control bitumen by two and ten times, when combined with aggregate sources 1 and 2, respectively. The reason for this can be attributed to the higher internal cohesion (W_{BB}) of these bitumens: strongly bonded molecules which are expected to block the path of water more efficiently. Conversely, it is important to note that the use of SBS as additive has no significant change in the moisture-resistant behaviour of bitumen, which is expected since this additive is not recognised as an antistrip.

By comparing the ERP data for the two aggregate sources, it can be observed that source 1 is expected to produce the bitumen–aggregate pairs that are more resistant to debond in the presence of water. On the other hand, the source 2 aggregate produces weaker interfaces. It is necessary to clarify that the parameters shown in Table 4 are mainly related to the feasibility with which the stripping process might occur at the bitumen–aggregate interface. Also, it must be noted that stripping is only one of numerous processes occurring in an asphalt mixture when subjected to water. Therefore, other physicochemical parameters must be taken into account to better approximate the behaviour of certain mixture regarding moisture damage. In this sense, a few suggestions are made in the following section.

Relationship between physicochemical approach and field performance

In the mechanical test performed in this study, failure occurs because of the deterioration of the mastic due to water absorption. In this sense, the manner in which water interacts with the components of an asphalt mixture appears to be key in determining the proneness to fail under wet conditions.

The loss in modulus of the proposed mixtures was found to be higher for the mixtures designed with aggregate source 1 than for the aggregate source 2, as per Figure 1. On average, the loss of modulus for mixtures of aggregate sources 1 and 2 is 33.7% and 16.8%, respectively. In spite of this behaviour, the physicochemical data suggested that aggregate 1 is expected to have a better performance regarding stripping, compared to aggregate 2. The opposing findings in these tests suggest that stripping might not be the main mechanism by which failure is observed in the samples. Then, in addition to stripping, other processes might be occurring in the mixtures that can be probably related to compatibility of materials.

Assuming that the water absorption of mastic is related to the failure of mixture, it is expected that the materials more compatible with water will be more likely to fail. Thus, physicochemical parameters such as W_{AW} and W_{BW} can be used as indicators of compatibility of aggregate and bitumen to water, respectively. The calculated W_{AW} and W_{BW} for the materials used in this study are listed in Table 5.

Table 5. Compatibility indicators W_{AW} and W_{BW} .

Material	W_{BW} (mJ/m ²)	W_{AW} (mJ/m ²)
Control	61.5	–
2% SBS	58.7	–
2% SBS + 1% Lime	61.0	–
1% Lime	55.0	–
0.5% LAS	51.4	–
Aggregate source 1	–	92.3
Aggregate source 2	–	66.2

As per Table 5, the value of W_{AW} for source 1 is significantly higher than that for source 2, which means that source 1 has a higher compatibility with water. Such compatibility can be related to the lower water resistance shown by mixtures of source 1 as captured thru E^*R , and it can be concluded that the aggregate compatibility to water is dictating the final mechanical response of the mixtures regarding moisture-induced damage. This observation is consistent with previous studies that found that the moisture damage is more related to the aggregate than to bitumen, or even to mastic (Apeageyi, Grenfell, & Airey, 2015; Baldi-Sevilla, 2015).

On the other hand, it is necessary to differentiate between the observed performances of mixtures produced with the same aggregate source. Such behaviour can also be explained by the compatibility of bitumen to water, W_{BW} . In Table 5, it is noticed that the Lime-modified bitumen and LAS-modified bitumen are the ones with lower compatibility to water. This is consistent with the fact that the mixtures involving these modified binders exhibited higher resistance to the successive conditioning cycles in the mechanical test.

Conclusions

The exposure of an asphalt mixture to water triggers several deteriorating mechanisms that occur simultaneously. It was found that the resulting loss in modulus of the mixtures by action of water cannot be associated with an individual process, such as stripping or debonding. The moisture-induced damage will take place by a complex combination of physical and chemical processes. Also, the contribution of each process to the final performance of the mixture is expected to be specific for every particular blend of bitumen and aggregate. In this sense, the compatibility indicators, W_{AW} and W_{BW} , were successfully applied to relate the physicochemical behaviour to the field performance of asphalt mixtures.

Additionally, it was found that by selecting the proper materials it is possible to design asphalt mixtures that are more resistant to moisture-induced damage. Furthermore, by means of surface energy measurements, as well as mechanical tests (to measure fundamental material properties), it was possible to distinguish between different blends and choose the aggregate source and bitumen that better perform under high levels of moisture.

Disclosure statement

No potential conflict of interest was reported by the authors.

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