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Influence of nanosilica and diatomite on the physicochemical and mechanical properties of binder at unaged and oxidized conditions

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HIGHLIGHTS

- Modification of binder with nanosilica and diatomite was proposed.
- Nanosilica and diatomite produced binders with largely different properties.
- The transformations occurring in the binder due to modification and aging were analyzed.
- The chemical interactions within the binder (cohesion) define its final performance.

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ABSTRACT

Modification of binders helps address common distresses in pavements by changing their essential properties. In order to further characterize the role of additives in these changes, a set of nanosilica and diatomite modified binders was analyzed to evaluate the performance under unaged and oxidized conditions. The results suggest that the mechanical and physicochemical behavior exhibited by binders is defined by the chemical interactions occurring within the material (cohesion). This study allowed understanding the chemical transformations occurring in the binder after modification and aging. Therefore, the results advance the achievement of materials with improved and engineered properties.

1. Introduction

Adequate performance of an asphalt mixture can be affected by common failure modes in pavements, such as permanent deformation and fatigue cracking [27]. Permanent deformation can be a consequence of the inability of asphalt concrete layer to resist the continuous traffic loading. It is a short term failure that appears as a depression along the tire path [21]. On the other hand, fatigue is the mechanical degradation of pavement caused by repetitive loading. The pavement accumulates damage and deflects until it cracks [23]. It is a long term failure which is intensified by oxidation of the binder [8]. Furthermore, these mechanical failures can be a catalyst to moisture-induced damage: one of the most severe types of distress in pavements. Moisture-induced damage deteriorates the essential properties of an asphalt mixture, such as adhesion and cohesion, causing stripping of binder and raveling of aggregates [7].

In order to address these distresses, it is imperative to design materials with improved properties. In this sense, modification of binder has been used as a suitable solution since it transforms the chemical environment of binder and consequently, its rheological and physicochemical responses. However, it is necessary to consider that, as a part of an in-service pavement, the binder is exposed to several transformations such as continuous oxidation, which weakens its mechanical properties and affect the compatibility of binder and aggregate [9,19]. These transformations will occur even if the asphalt binder has been modified. Therefore, it is important to understand the changes occurring in the binder

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due to modification and aging, and to relate them to the field performance during its service life. Consequently, the objective of this study is to characterize the effect of additives on the physicochemical and rheological properties of binders at unaged and oxidized conditions, with the aim of predicting its final field performance. To achieve this goal, the binder was modified with diatomite and nanosilica, which were chosen as additives because of promising performance improvements demonstrated in previous work [10,26,25,12,17]. The resulting binders were evaluated by means of deformation resistance, crack development and moisture-induced damage resistance.

2. Background

2.1. Surface Free Energy (SFE) of pavement materials

The Surface Energy (SE) is an inherent property to all materials related to the chemical interactions occurring along their surface. Mathematically, the total SE (γ^T) is the sum of these interactions, which are classified into dispersive (non polar) (γ^{LW}) and polar interactions (γ^{AB}). In turn, the polar component of SE is divided into the acidic (γ^+) and basic (γ^-) interactions (Eq. (1)) [22].

$$\gamma^T = \gamma^{LW} + \gamma^{AB} = \gamma^{LW} + 2\sqrt{\gamma^+\gamma^-} \tag{1}$$

The SE is an important property in pavement design since it allows quantification of the compatibility between binder and aggregate by means of work of adhesion, W_{AB} (Eq. (2)), as well as quantification of the stripping potential of a given binder-aggregate combination by means of work of debonding, W_{wet} (Eq. (3)) [18,1].

$$W_{AB} = \gamma^A + \gamma^B - \gamma^{AB} \tag{2}$$

$$W_{wet} = \gamma^{AW} + \gamma^{BW} - \gamma^{AB} \tag{3}$$

where γ^A is the total SE of aggregate, γ^B is the total SE of binder and γ^{AB}, γ^{AW} and γ^{BW} refer to the binder-aggregate, aggregate-water and binder-water interfaces, respectively.

Previous knowledge of W_{AB} and W_{wet} is useful to select the adequate binder and aggregate combination with improved moisture resistance. A higher value of W_{AB} indicates that the formation of the interface of a given binder-aggregate pair is thermodynamically favorable and difficult to break [16]. Conversely, a low value of W_{wet} indicates a lower susceptibility of the binder-aggregate interface to be breached by water. Since adhesion and debonding occur simultaneously in an asphalt mixture, the Energy Ratio (ER) parameter combines these processes into a single indicator (Eq. (4)): the higher the ER, the higher the moisture induced damage-resistance of the binder-aggregate interface [18].

$$ER = W_{AB}/W_{wet} \tag{4}$$

Also, SE allows quantifying the work of cohesion of asphalt binders, W_{BB}, by means of Eq. (5). Cohesion is recognized as the interactions binding the molecules of the same material. The higher the cohesion, the stronger the interactions.

$$W_{BB} = 2\gamma^B \tag{5}$$

The physicochemical parameters previously described have been related to the final performance of asphalt mixtures [18,4,11]. Therefore, such parameters are considered in this study to be appropriate for material selection and evaluation regarding moisture-induced damage resistance.

2.2. Rheological characterization of binders

In order to quantify the rutting and fatigue cracking resistance of asphalt binders, several rheological tests have been developed using the dynamic shear rheometer (DSR). The two tests used in this study are briefly described as follows.

2.2.1. Rutting resistance characterization

The Multiple Stress Creep Recovery test (MSCR) has been applied in order to characterize the rutting potential of binders. This test allows quantifying the compliance, J_{nr}, and the elastic recovery of binders subjected to several loading cycles. The J_{nr} is a parameter which estimates the deformation accumulated in the binder that impedes the return to its original state. By contrast, the elastic recovery is the capability of binder to return to its initial condition after continuous loading [14]. Then, a rutting resistant binder will exhibit low compliance and high elastic recovery values.

2.2.2. Fatigue resistance characterization

This test consists in applying controlled strain to the sample while monitoring several responses of the system, such as dynamic modulus. The failure is defined as the number of cycles at which a 50 percent loss in modulus is observed. Therefore, a fatigue resistant binder will resist a high number of cycles before the failure.

3. Materials and methods

3.1. Modification and aging of binder

The asphalt binder was modified with 2, 4 and 6% by weight of nanosilica and with 2, 4 and 6% by weight of diatomite, separately. The additives were incorporated with a low shear stirrer at 175 °C during 1.5 h. Fig. 1 shows the IR spectra of the binders modified with 6% additive.

From Fig. 1 it can be seen that the presence of the additives in the binder is evidenced by the bands located around 1100 cm^{-1} and 472 cm^{-1}, which correspond to the bending and stretching vibrations of O–Si–O groups. In the case of diatomite-modified binder the band at 503 cm^{-1} is attributed to inorganic compounds commonly found in the diatoms [15].

A sample of each binder was separated and treated to simulate short-term aging according to the Rolling Thin Film Oven (RTFO) procedure [5]. A second sample of the binders was oxidized as described in the Pressure Aging Vessel (PAV) procedure [6]. The rheology and contact angle measurements were performed on both the unaged or the aged samples as required.

3.2. Surface Free Energy (SFE) approach

The samples of asphalt binders were prepared by pouring a small amount of binder over a glass slide. The slide was heated at 100 °C until a homogeneous and smooth surface was obtained. Regarding the aggregate sample, an uncrushed boulder was saw-cut, polished, washed with distilled water and allowed to dry in an oven at 100 °C for at least 2 h. The bitumen and aggregate samples were allowed to reach 20 °C in a desiccator prior to testing.

The total surface energy and its individual components were determined with contact angle measurements by means of a goniometer. The calculation regarding the estimation of surface energy, adhesion and cohesion values was carried out based on the methods described in the literature [13,3,24,20].

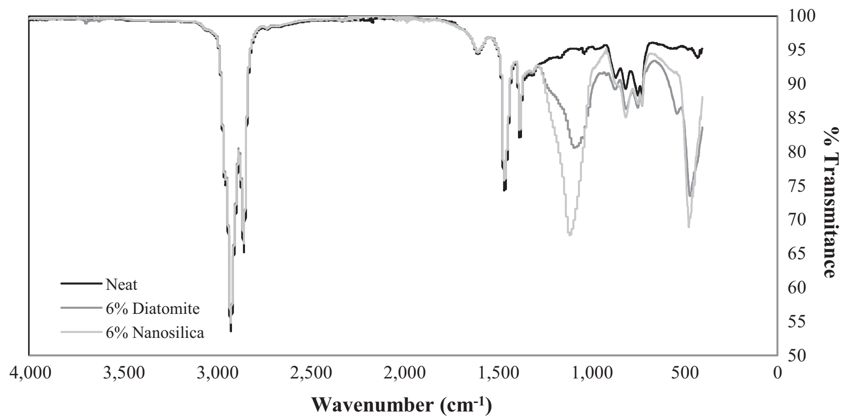

Fig. 1. Infrared spectra of 6% diatomite and 6% nanosilica modified binders. The spectra of neat binder is shown for comparison.

3.3. Rheological characterization

The MSCR test and fatigue test were carried out in a dynamic shear rheometer. The MSCR test was performed to the RTFO-aged samples at the high service temperature for each binder. The test was carried out using the 25 mm geometry at a frequency of 10 rad/s and 10% strain. Additionally, the fatigue test was performed to the PAV-oxidized samples at the low service temperature of each asphalt binder, using an 8 mm geometry. A controlled strain of 1% and a frequency of 10 rad/s were applied.

4. Results and discussion

4.1. Effect of additives and aging on surface energy of asphalt binder

As per Table 1, the presence of additives, as well as the oxidation processes occurring on the binder, generates immediate changes on its surface energy characteristics.

It was noted that nanosilica and diatomite decreased the dispersive component (γ^{LW}) and increased the polar components of SE in the unaged binder. As a result, a higher polarity contribution to the total surface energy was observed. The previous is expected since both nanosilica and diatomite are polar particles that should provide polarity to the material. The overall effect of the additives is the reduction of total SE of the binder, and consequently, the cohesion is diminished. It was found that the magnitude of these changes is linearly related to the additive content (Fig. 2).

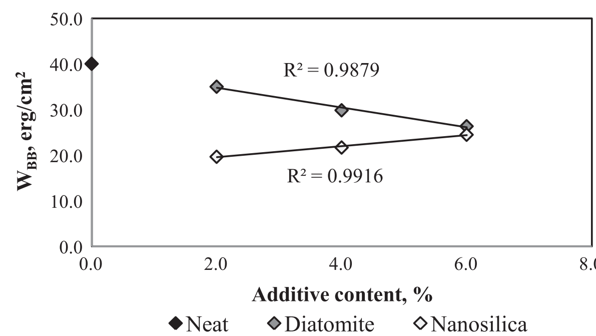

Fig. 2. Influence of additive content on the cohesion of unaged asphalt binders.

As per Fig. 2, the additives have the opposite effect on cohesion: a linear increase in the nanosilica content causes a linear increase in cohesion. By contrast, the linear increase in the diatomite content causes a linear reduction in cohesion. Regardless of the trends observed in Fig. 2, the results suggest that the addition of both additives reduces the chemical interactions that originally occur in the neat binder, which is reflected in the resulting cohesion values. The addition of 2% of nanosilica seems to drastically break the interactions between molecules of the binder. As the content of nanosilica is increased, these interactions appear to be reestablished, increasing the cohesion. Conversely, the continuous addition of diatomite seems to be negatively affecting the interactions within the binder; therefore, higher contents of this additive reduce the cohesion.

Table 1
Surface Energy and cohesion values of aggregate and binders.

Asphalt binder	W_{BB} erg/cm^2	γ^{TOTAL} erg/cm^2	γ^{LW} erg/cm^2	γ^{AB} erg/cm^2	γ^{+} erg/cm^2	γ^{-} erg/cm^2
Neat	40.2	20.1	19.00	1.03	0.13	2.07
Neat aged	35.0	17.5	15.10	2.41	0.72	2.02
2% Diatomite	35.0	17.5	16.70	0.84	0.04	4.59
2% Diatomite aged	25.8	12.9	5.04	7.87	4.78	3.24
4% Diatomite	30.0	15.0	11.80	3.15	0.52	4.76
4% Diatomite aged	24.0	12.0	2.58	9.44	6.83	3.26
6% Diatomite	26.4	13.2	6.94	6.27	2.93	3.35
6% Diatomite aged	24.2	12.1	2.95	9.12	6.96	2.99
2% Nanosilica	19.7	9.86	2.02	7.84	7.84	1.96
2% Nanosilica aged	24.8	12.4	4.28	8.12	4.74	3.48
4% Nanosilica	21.8	10.9	3.9	6.98	6.31	1.93
4% Nanosilica aged	25.0	12.5	4.2	8.32	4.77	3.63
6% Nanosilica	24.6	12.3	5.7	6.60	4.14	2.63
6% Nanosilica aged	38.0	19.0	18.4	0.60	0.03	2.81
Limestone	–	42.1	39.4	2.77	0.226	8.49

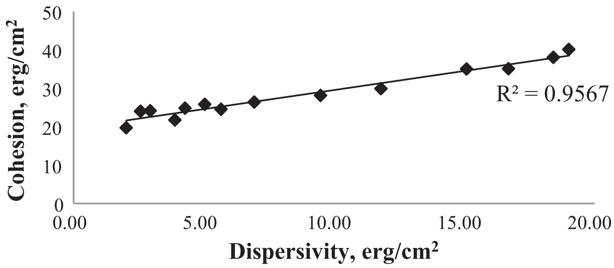

Fig. 3. Cohesion as a function of dispersivity of asphalt binders.

Regarding the changes in surface energy due to aging, it can be seen from Table 1 that in the case of neat binder, the total SE decreases after aging. It was observed that the dispersive component is reduced and the polar component, particularly the acidic component, increases slightly, probably due to the oxygenated molecules produced during oxidation. The diatomite-modified binders follow the same trend; however, the changes observed in dispersive and polar components are significantly higher. Based on this observation, it can be concluded that the presence of diatomite causes a more drastic oxidation by abruptly increasing the polarity of binder. On the other hand, the nanosilica-modified binders showed the opposite trend. After aging, the total SE increases due to an increase in the dispersive component. The polar component also increases, but in this case, the increase is perceived in the basic component, while the acidic decreases.

As discussed, the changes in the individual components of surface energy introduced by the additives influence the resulting cohesion of the binder. To illustrate this, Fig. 3 shows a linear relationship between cohesion and dispersivity of the unaged and oxidized binders: it can be noticed that the binders with larger dispersive component exhibit larger cohesion values. It is important to highlight that no relationship was found between cohesion and basicity or acidity of binders, which suggests that the dispersive component seems to have a more important role in defining the interactions within the asphalt binder. This is reasonable since the asphalt binder is mainly composed by hydrocarbons, roughly considered as non-polar molecules that interact with each other by means of dispersive forces.

4.2. Effect of additives and aging over moisture damage-resistance

The quality of the binder-aggregate interface formed during mixing in HMA production is influenced by the surface energy characteristics of both materials, and defines the behavior of the binder-aggregate combination in the presence of water. By means of surface energy measurements several moisture damage indicators, such as work of adhesion, work of debonding and Energy Ratio, were calculated (Fig. 4).

In order to obtain a binder-aggregate interface with high resistance to moisture there must be a good adhesion between the materials: when the W_{AB} is high, the binder-aggregate interface is more resistant and difficult to break. However, since water easily reaches the binder-aggregate interface, it is required that the stripping process be less favorable, reflected as low values of W_{wet}. The Energy Ratio (ER) is obtained after combining these parameters and therefore, it is expected that a moisture-resistant interface will exhibit high ER [2].

According to Fig. 4a, the adhesion of the combination neat binder-limestone is superior when compared to those combinations resulting from the modified binders. This suggests that adding either nanosilica or diatomite into the matrix of the binder reduces its compatibility with the aggregate. However, the additives reduced the propensity of binder to be displaced by water, by reducing the W_{wet} (Fig. 4b). As a result, is it generally observed that the modification increases the ER of the binder (Fig. 4c): the continuous addition of nanosilica gradually increases the ER of the binder; on the other hand, the resulting ER of the diatomite-modified binders is not influenced by the additive content. It is important to highlight that modification with diatomite produces binders with higher moisture resistance, as noticed in the larger ER obtained with the diatomite-modified binders.

The trends observed in Fig. 4 are related to the physicochemical characteristics of the binder surface, as shown in Fig. 5.

As per Fig. 5 the moisture damage indicators are highly related to the magnitude of the surface energy components of the binder: higher dispersivity and lower polarity cause the binder to adhere strongly with the aggregate (Fig. 5a). This suggests that the dispersive forces have a higher contribution to the adhesion process, which in turn can be related to the dispersive nature of the binder given by its chemical composition. Conversely, lower dispersivity and higher polarity causes the debonding process be less favorable (Fig. 5b). These observations suggest that the dispersive and polar components of the surface energy of binder must be balanced in order to produce an interface with good adhesion and low proneness to debonding. The previous explains the trends followed by ER when related to the surface energy components of binders (Fig. 5c).

These moisture damage indicators are affected by the aging of binder, as shown in Fig. 6.

Once aged, the adhesion of neat and diatomite-modified binders decreases (Fig. 6a). It is also noted that the adhesion for the diatomite-modified binders drops more sharply after oxidation, compared to the neat binder. Such behavior is related to the increase in polarity observed for the aged diatomite-modified binders, which would weaken the adhesion with aggregate, as stated previously. These observations indicate that these modified

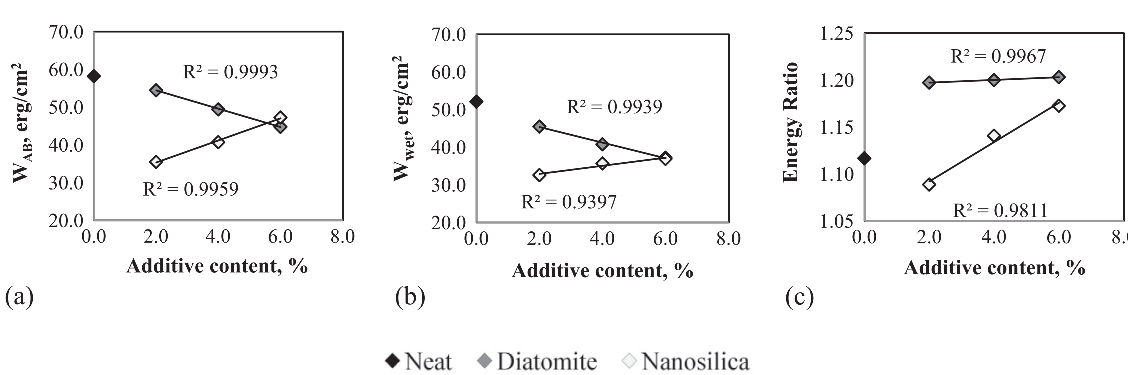

Fig. 4. Influence of additive content on a) work of adhesion, b) work of debonding and c) Energy Ratio of the binder-limestone combinations.

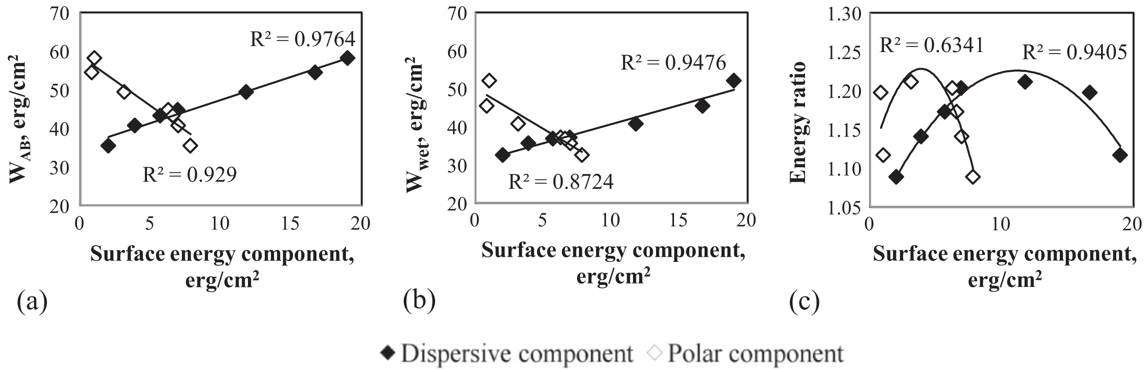

Fig. 5. Influence of surface energy components of binder on a) work of adhesion, b) work of debonding and c) energy ratio.

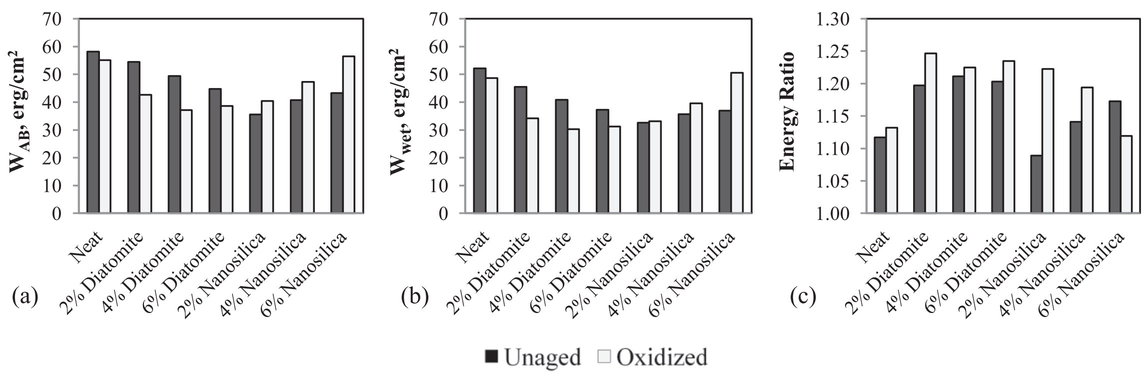

Fig. 6. Effect additives on a) work of adhesion, b) work of debonding, and c) energy ratio of unaged and oxidized binders.

binders are more prone to detach from the aggregate surface in dry condition during the service life. On the other hand, the adhesion of the nanosilica-modified binders increased after aging (Fig. 6a), due to the increase in the dispersive forces occurring in the aged nanosilica-modified binder. This suggests that the quality of the interaction between the aggregate and these modified binders might not be compromised with aging. Consequently, the resulting nanosilica modified binder-aggregate combinations are more resistant to debond in dry conditions through time. In the case of the neat and diatomite-modified binders, the changes in adhesion are accompanied by a reduction in the work of debonding, indicating that the compatibility between these binders and water is reduced with oxidation. The opposite is observed in the case of nanosilica-modified binders (Fig. 6b). As a result, these trends alter the ER of the binder-aggregate combinations (Fig. 6c): the addition of either nanosilica or diatomite produces interfaces for which their moisture damage resistance is physicochemically improved through time.

4.3. Effect of additives on the deformation and fatigue resistance of asphalt binder

Modification of binder will affect its mechanical properties (eg. deformation resistance). The compliance and recovery for the proposed materials were estimated by means of the MSCR test. The results are shown in Fig. 7.

According to Fig. 7 it is observed that the response of the binder is related to the additive content: higher contents of either diatomite or nanosilica gradually reduce the compliance and increase the recovery under loading. However, it is noticed that the modification with diatomite seems to be worsening the rutting potential of the asphalt binder. On the other hand, the addition of 4% and 6% of nanosilica significantly reduces the deformation rate and improves the recovery capacity of asphalt binder.

Regarding the fatigue tests (Fig. 8), it can be observed that the nanosilica-modified binders showed superior resistance compared to the neat binder. In the case of the diatomite-modified binders,

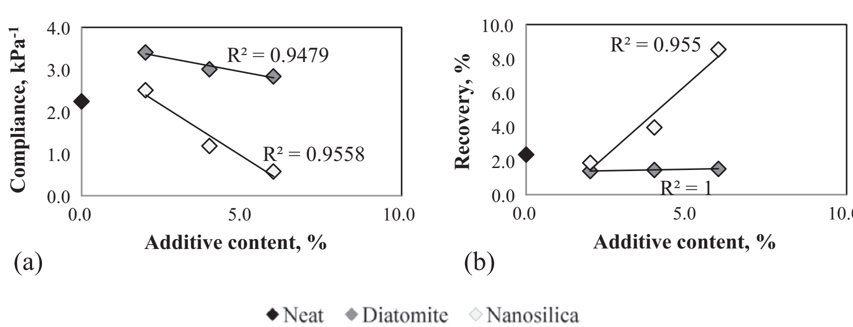

Fig. 7. Influence of additive content on a) compliance and b) elastic recovery of asphalt binders.

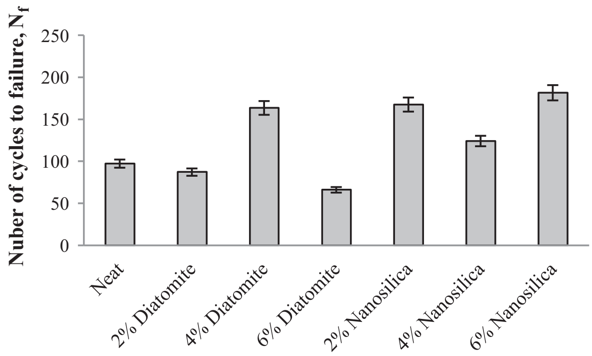

Fig. 8. Number of cycles to failure for neat and modified binders.

only the 4% diatomite exhibited an improvement, the remaining two binders presented a decrease in the fatigue resistance, compared to the neat binder. The trend shown by the binders regarding fatigue behavior is independent of the percentage of additive used.

The explanation regarding the rutting and fatigue behavior exhibited by the asphalt binders will be discussed in the next section.

4.4. Relationship between the cohesion and performance of binder

It was found that the influence of the additives on the cohesion of the binder is highly related to its resulting performance. To illustrate this, Energy Ratio, compliance, recovery and fatigue were related to the cohesion for each asphalt binder (Fig. 9).

The ER trend shown (Fig. 9a) suggests that there must be an optimum level of cohesion in the binder that produces binder-aggregate interfaces with improved moisture damage resistance. This observation is related to the balance of the dispersive and polar components of surface energy required to produce the optimum adhesion and debonding values, as discussed previously. Regarding the compliance and recovery results (Fig. 9b and c), it is generally observed that the binders with lower cohesion exhibited lower deformation rate and higher recovery capability. The previous can be explained in terms of molecular mobility: a lower cohesion is an indicative of weaker (or lower in number) interactions within the asphalt binder. Therefore, it is expected that the molecules that are not strongly bound with each other will rearrange more easily and recover more efficiently when loaded. This behavior can also be related to the fatigue life of the material: the binders with higher cohesion exhibited lower efficiency to resist the load cycles and therefore, failed more easily (Fig. 9d), probably due to a hindered mobility that causes molecules to be less efficient in supporting stresses.

5. Conclusions

1. Modification of asphalt binder with chemically similar additives allowed obtaining materials with different physicochemical and mechanical responses. This supports the use of modification in obtaining asphalt binders with improved and engineered properties that can be adjusted according to the need.
2. The physicochemical and mechanical responses of binder can be explained in terms of the intermolecular interactions occurring in the material: cohesion. It was found that a highly cohesive binder is more prone to rutting and fatigue cracking. Regarding the moisture induced damage resistance, it is necessary to achieve an optimum level of cohesion in order to balance the adhesion and debonding processes taking place in the binder-aggregate interface.
3. Given that the cohesion is an important property defining the final performance of the binder, the design of additives must be performed considering the changes in cohesion introduced after modification.
4. The physicochemical transformations taking place during aging influence the resulting performance of the aged binder. These transformations must be considered in order to select additives for modification, such that the essential requirements of binders would not be significantly deteriorated in time.

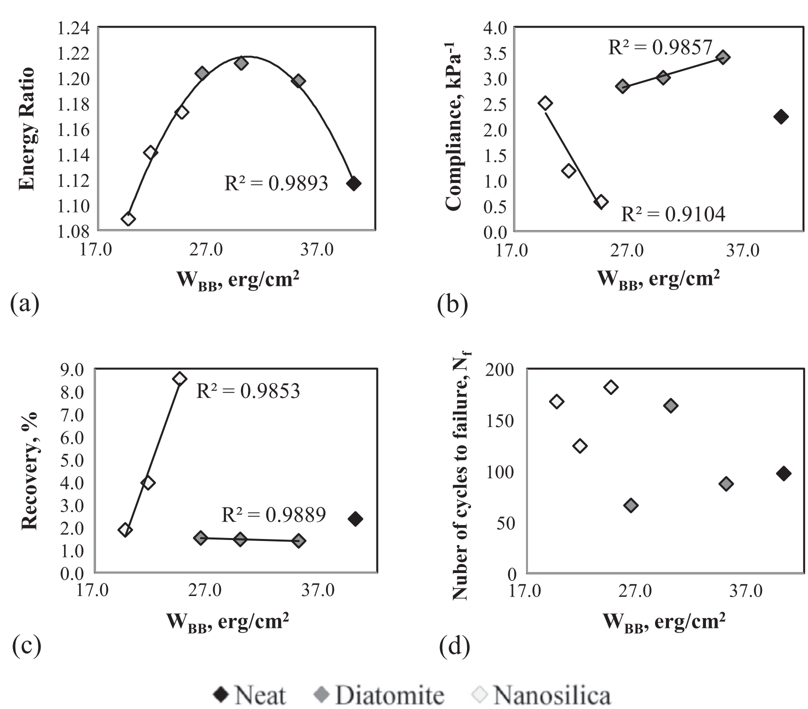

Fig. 9. Relationship between cohesion of the binders and a) energy ratio, b) compliance, c) elastic recovery and d) fatigue.

In summary, the introduction of additives into the binder produces materials with different mechanical and physicochemical responses. Then, in order to successfully improve the quality of asphalt binders, a designed selection of additives must be performed, considering all the changes that occur during the service life of pavements. To obtain a complete characterization, it is highly recommended that a mechanical evaluation of the resulting materials be performed.

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