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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** highlights are the control of the c

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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### ARSTRACT

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. 2016 Elsevier Ltd. All rights reserved.

#### 1. Introduction

Adequate performance of an asphalt mixture can be affected by common failure modes in pavements, such as permanent deformation and fatigue cracking [\[27\].](#page--1-0) 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 [\[23\]](#page--1-0). It is a long term failure which is intensified by oxidation of the binder [\[8\].](#page--1-0) Furthermore, these mechanical failures can

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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 [\[7\]](#page--1-0).

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 [\[10,26,25,12,17\]](#page--1-0). 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  $(\gamma^{\text{T}})$  is the sum of these interactions, which are classified into dispersive (non polar) ( $\gamma^{\text{LW}}$ ) and polar interactions ( $\gamma^{AB}$ ). In turn, the polar component of SE is divided into the acidic ( $\gamma^{\text{+}}$ ) and basic ( $\gamma^{\text{-}}$ ) interactions (Eq. (1)) [\[22\].](#page--1-0)

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

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 binderaggregate combination by means of work of debonding,  $W<sub>wet</sub>$ (Eq. (3)) [\[18,1\]](#page--1-0).

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

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

where  $\gamma^A$  is the total SE of aggregate,  $\gamma^B$  is the total SE of binder and  $\gamma^{AB}$ ,  $\gamma^{AW}$  and  $\gamma^{BW}$  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 Wwet 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 [\[18\]](#page--1-0).

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

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 [\[18,4,11\]](#page--1-0). 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,  $I_{nr}$ , 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  $\degree$ C during 1.5 h. [Fig. 1](#page--1-0) shows the IR spectra of the binders modified with 6% additive.

From [Fig. 1](#page--1-0) it can be seen that the presence of the additives in the binder is evidenced by the bands located around  $1100 \text{ cm}^{-1}$ 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 [\[15\].](#page--1-0)

A sample of each binder was separated and treated to simulate short-term aging according to the Rolling Thin Film Oven (RTFO) procedure [\[5\]](#page--1-0). A second sample of the binders was oxidized as described in the Pressure Aging Vessel (PAV) procedure [\[6\]](#page--1-0). 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 sawcut, polished, washed with distilled water and allowed to dry in an oven at 100  $\degree$ C for at least 2 h. The bitumen and aggregate samples were allowed to reach 20 $\degree$ 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 [\[13,3,24,20\].](#page--1-0)

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