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Use of frequency sweep and MSCR tests to characterize asphalt mastics containing ornamental stone residues and LD steel slag

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HIGHLIGHTS

- Rheological characteristics of asphalt mastics have been measured.
- Ornamental stone fine by-products and steel slag residues effects in asphalt mastic is assessed.
- Frequency sweep and MSCR tests are performed.
- Microstructure and chemical analysis of studied residue materials are evaluated.

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ABSTRACT

This study presents an evaluation of ornamental stone industry fine by-products (OSR) and LD steel slag residue (SLR) as a potential paving material, more specifically, as filler in hot mix asphalt mixtures. In order to accomplish that, a rheological study of mastics produced by mixing an asphalt binder PG70-28 with OSR, SLR, and a mixture of both residues in different filler proportions (0.36, 0.54 and 0.72) was performed. First, the materials were characterized physically, chemically and mineralogically. Also, their microstructure was observed by image analysis using scanning electron microscopy. For the rheological parameters analysis, complex modulus ($|G^*|$), phase angle (δ) and multiple stress creep and recovery (MSCR) tests were carried out and compared. In general, the use of residues improved rheological behavior of the pure binder, especially SLR residue which contributed to the increase in the material stiffness since it provided greater elasticity compared to binder or OSR waste, and also showed better results of recovery and creep compliance, and reduced sensitivity to tension.

1. Introduction

The ornamental stone industry has a large potential market worldwide and it is an important industrial sector in Brazil, which is the 5th largest producer in the world according to the XXIV Report Marble and Stone in the World [1]. The steel production industry is also significant for the Brazilian economy, in which the country is ranked as the second largest exporter of iron ore and fifteenth largest exporter of steel, according to the TWI Report [2]. Even though the exploration of those materials has great benefits to the local economy, during their production large quantities of fine by-products/waste (such as stone sludge and steel slag) are generated. The handling and disposal of these fine by-products pose severe environmental problems since they greatly contribute to the accumulation and harmful dispersion of fine solid particles in the air, water and soil [3,4].

The processing of ornamental rocks, which refers to the unfolding of the blocks and superficial polishing of the surfaces, generates a significant amount of reject material, which takes the form of mud. For many years, this mud was deposited in lakes in Brazil, which represented a significant environmental threat. However, nowadays companies collect this residue through tubes and channels, which are directed to collection wells. Many companies have adopted the press filter to reduce moisture in the material before it is sent on, trying to reuse the water from the processing. The main uses of rock residue have been as a fine aggregate in asphalt mixtures, in the production of cement and in ceramic matrixes [5].

The use of metallurgical by-products, such as LD steel slag, blast furnace slag, fly ash, and others can also be alternative materials in concrete mix design not only to minimize the costs on concrete production but also to reduce the amount of residue disposal in

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open areas. The use of steel slag has been shown to be beneficial due to its lower heat of hydration, higher sulphate and acid resistance, better workability, lower permeability and higher corrosion resistance [6,7].

Although there are many references regarding the use of those materials in Portland Concrete systems, the studies incorporating this material in asphaltic mixtures are not many. Considering that OSR and SLR are very fine powders after being dried and pre-treated (crushed and sieved to eliminate large particles), they could be added as filler in asphalt mixtures. However, to be used as filler, it is important to understand the possible interactions that can occur among filler particles and asphalt binders, as well as with other asphalt concrete components.

The rheological properties of mastics are a result of the combination of the elastic, viscoelastic or viscous characteristics of the asphalt binder and the elastic nature of the mineral filler, which affects the mechanic properties of the asphalt mixture made up of these materials. As such, the study of the rheological properties of mastic enables the assessment of how this compound can affect the properties of the asphalt mixture [8].

Many rheological studies have been carried out to try to understand the internal structure of asphalt mastic. Tests are performed mainly under linear viscoelastic conditions (LVE) to obtain viscoelastic properties such as complex modulus (G^*) and phase angle (δ) under a wide range of temperature and/or frequencies through the construction of master curves. The laboratory equipment to determine these rheological properties is the dynamic shear rheometer (DSR). The rheological parameter G^* is related to the rigidity of the material, being used as indicator to pavement susceptibility to permanent deformation when an asphalt road is subjected to shear stress. The phase angle (δ) is a function of a viscoelastic response of the material. When a material is pure elastic, the δ is zero degrees, and the viscoelastic response to deformation is immediate. However, when δ is 90 the material behaves like a purely viscous material. High values of G^* represent an increase in stiffness of material, while low values of δ represent improvement in elastic response [9–11].

This paper presents an analysis of rheological properties of asphalt mastics produced with different residues (OSR and SLR) as fillers and different filler/binder proportions. The properties analyzed through laboratory tests are: complex modulus ($|G^*|$), phase angle (δ) under linear viscoelastic region, and multiple stress creep and recovery (MSCR) parameters such as recovery percentage (%R) and non-recovery compliance (Jnr).

2. Experimental procedure

2.1. Materials

To produce the asphalt mastics, an asphalt binder PG 70-28, made of Brazilian oil from Campo Fazenda Alegre, located in Espirito Santo was used. Two types of residues were used as filler: ornamental stone residue (OSR) and steel slag residue (SLR). Both residues were obtained from local companies. They were characterized by sieve analysis testing for particle dimensions, as shown in Fig. 1. Also, X-ray diffraction was performed to assess the main chemical components, as presented in Fig. 2 and Table 1.

Fig. 1(a) shows that 90% of the OSR particles used in this study have dimensions bellow 25 μm, which infer that this residue can work as active filler, changing properties of the binder in addition to its filling effect on the mineral skeleton of mastic. From Fig. 1(b), the analysis of SLR showed particles dimensions varying from 150 μm to bellow 25 μm. Thus, one can suppose that part of this material can act as inert filler, which works more likely filling the voids in the composite, and part can act as active filler, changing binder properties due to chemical interactions.

As far as the OSR is concerned, results indicate predominance of calcium oxide (CaO) and silicon dioxide (SiO_2); the high concentration of calcium oxide is good for asphalt mixtures, considering that this material reduces wheel track rutting, as well as aging and stress-related cracking [12]. The high percentage of SiO_2 can affect negatively the asphalt concrete performance, since the presence of this chemical compound can reduce the adhesion of the mastic into the aggregates [13]. Regarding the SLR, there is predominance of calcium oxide (CaO) and iron oxide (Fe_2O_3), followed by silicon dioxide (SiO_2). The Fe_2O_3 in the steel slag contributes to increased hardening of the asphalt mixture, but in lower temperatures it may contribute to the formation of inner cracks and fissures in the asphalt coating. The percentages of SiO2 were lower than the ones found in the OSR, but since considerable percentages are still found, a careful analysis needs to be further taken to evaluate its effect.

The residue micrographs in Fig. 3(a and b) show that the OSR particles do not have a definite form. They usually have a lamellar shape due to the rock sawing process; grain size ranges between 2 and 25 μm, with some larger grains of about 50 μm. Although particle shape contributes to the interlocking of grains, the residue has a smooth texture, which, together with the considerable concentration of impurity (SiO_2), might harm the adhesiveness with the binder. It can be concluded, based on the micrographs and the particle size analysis, that the OSR residue will act more as active filler than as influence to the makeup of the mixture's mineral framework.

As for the SLR residue, through the images showed on Fig. 3(c and d) it can be concluded that part of the residue will act as an inert filler, which contributes to the formation of the mineral skeleton of mixture, filling in empty spaces between the small and large aggregates, binding to the large aggregates, and acting in many instances as an adhesiveness improvement; part will act as an active filler, which is responsible for increasing the viscosity of the binder, altering the softening point, reducing thermal susceptibility and increasing the resistance to deformation. The micrographs of the residue in Fig. 3 show that the grains have a generally angled shape and their size ranges between 2 and 25 μm, with some slightly larger grains around 50 μm. The shape of the grain contributes to the interlocking of grains. The residue has a smooth texture, which, together with a decrease in concentration of impurity (SiO_2), might lead to better adhesiveness than the OSR residue.

2.2. Studied mastics

A total of nine asphalt mastics were designed using OSR and SLR residues in different blends (100% OSR, 100% SLR, 50%OSR + 50%SLR) and three filler residue/binder proportions in mass (0.36, 0.54 and 0.72).

The mastics were aged using the Rolling Thin Film Oven Test (RTFOT), according to ASTM D2872 [14]. This testing method indicates changes in the asphalt properties that may occur during machining at 150 °C, verified by variations in the rheological measures. Samples of approximately 35 g were weighed in glass containers. These samples were heated at 163 °C for 85 min.

2.3. Test methods

Oscillatory tests such as frequency sweep (FS) and Multiple Stress Creep Recovery Test (MSCR) were performed using a Dynamic Shear Rheometer (DSR).

2.3.1. Frequency sweep tests

A series of frequency sweep tests were performed in 8 mm diameter by 2 mm thick samples using the DSR. Test frequencies varied between 0.1 and 10 rad/s at five temperatures, ranging from 10 and 70 °C. A strain level of 0.1% was applied based on the results of preliminary amplitude strain sweeps carried out at 10 rad/s checking the extension of the linear viscoelastic domain.

The rheological behavior of the binders and mastics at midrange service temperature was described by applying the time-temperature superposition principle. Master curves were constructed based on Williams-Landel-Ferry (WLF) formulation to express the temperature-shift factor, considering 40 °C as a reference temperature.

2.3.2. Multiple stress creep and recovery test

The multiple stress creep and recovery test was developed by the Federal Highway Administration (FHWA) by improving the repeated fluency and recovery test as an alternative to the Superpave in oscillatory regimen for the characterization of the resistance of asphalt binders to permanent deformation. Both recovery percentage (R) and non-recoverable compliance (Jnr) properties are obtained with the MSCR. The calculations of the recovery percentage and the non-recoverable compliance are obtained through equations prescribed as per ASTM D7405 [15]. Regarding the recovery percentage, Eq. (1) is used.

$$R(\sigma, N) = \frac{[(\varepsilon_c - \varepsilon_0) - (\varepsilon_r - \varepsilon_0)].100}{\varepsilon_c - \varepsilon_0} \quad (1)$$

where R (σ, N) is the recovery percentage for tension σ (for σ = 100 Pa or 3200 Pa) for the cycle of fluency and recovery number N (where $1 \leqslant N \leqslant 10$). For non-recoverable compliance, Eq. (2) is used:

$$J_{nr}(\sigma, N) = \frac{\varepsilon_r - \varepsilon_0}{\sigma} \quad (2)$$

where Jnr (σ, N) is the non-recoverable compliance for tension of σ Pa and for the cycle of fluency and recovery number N, for σ and N with the same values previously mentioned (σ = 100 or 3200 Pa and $1 \leqslant N \leqslant 10$).