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Mechanical performance of asphalt mixtures using polymer-micronized PET-modified binder

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The global engineering plastics market has been growing as it replaces materials such as glass and metal. It can be used in a wide variety of products and technologies in other sectors of the economy such as energy generation, automotive, maritime, construction, electronics, packaging, and others. However, this increase in global use presented problems with respect to the increase in plastic waste generation. The existence and destination of plastic waste in the soil and hydric resources has been recognised as an emerging issue in preserving the environment. In this research, Micronized Polyethylene Terephthalate (micronized PET) was used as an additive for asphalt mixtures. The polymer was mixed in amounts of 4%, 5%, and 6% by weight with an asphalt binder base and mechanical tests were performed. Two SUPERPAVE mix design methods were performed. The first one was made with pure binder and the optimum binder content was determined and the specimens were analysed according to the following parameters: Resilient Modulus (RM), Lottman, and Indirect Tensile Strength (ITS). A second mix design procedure was performed with the optimum PET content according to the best results obtained in the first one, and analysed according RM, ITS, Lottman, Fatigue, and Flow Number tests. The results show significant improvements on mechanical properties of the asphalt mixtures with micronized PET compared with asphalt mixtures without additive.

Keywords: polymers; waste; asphalt mixtures; PET

1. Introduction and background

Asphalt mixtures are composed of asphalt binder and aggregates. A pavement performance is dependent on the individual characteristics of its components or the interaction between them or their interface (Sultana, Bhasin, & Liechti, 2014). Likewise, improvements in a mixture are associated with a better interaction of these materials.

Therefore, a thorough analysis of the individual properties of constituent materials and the asphalt mixture is necessary in order to prevent premature damages to the pavement, and to ensure its satisfactory performance. High-traffic levels can result in permanent deformation (rutting) and deteriorations, such as fatigue cracking. On the other hand, change in temperatures and environmental factors can cause thermal cracking and ravelling on the pavement even without

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traffic loads. With these two factors, the pavement can have a decreased service life if not maintained properly. Polymeric additives are used in order to conduct pavement performance by enhancing quality of asphalt mixtures (Karakas, Kuloglu, Kok, & Yilmaz, 2015; Rahi, Fini, Hajikarimi, & Nejad, 2015; Zoorob et al., 2012).

Asphalt binder aging can occur by the oxidation process associated to oxygen demands and environmental temperature variations. Aging can change rheological properties of asphalt binder and cause physical hardening effects, which leads the mixture to become brittle. This phenomenon can be explained by melting paraffin wax, present in the composition of asphalt binders, and its surface crystallization (Pizzorno, 2010). Thus, polymers are used in asphalt mixtures to increase performance and pavement service life (Golalipour, 2011; Lu & Isacsson, 2000; Toraldo & Mariani, 2014).

The addition of polymers to asphalt mixtures can reduce permanent deformation, fatigue, and thermal cracking and increase cohesion with aggregates. Tarefder and Yousefi (2015) concluded that the addition of 3–5% of SB and SBS polymers to the asphalt binder promotes improvements in rheological properties and reduce aging susceptibility.

In this context, the use of polymeric waste may appear to be valuable as a construction material, particularly on roads, with some advantages such as high availability and low cost.

The deposition of polymeric waste is one of the greatest challenges faced not only in Brazil, but all over the world, by public administrations. There is no doubt that its management affects many aspects of a community such as health, social, economic, and even cultural aspects. The financial investment in polymeric waste’s correct disposal becomes a great ally to sustainable development, with short-, medium-, and long-run benefits for society.

Polyethylene Terephthalate (PET) is normally used in fibres for clothing, containers for liquids and foods, thermoforming for manufacturing, and in combination with glass fibre to produce engineering resins. Since late 1990s until 2011, the worldwide production of PET is around 14–60 million tons (Lepoittevin & Roger, 2011) and the annual production of it exceeded 6.7 million tons/year (Kumar, Reedy, & Sasidhar, 2014); therefore an equivalent amount of PET waste was generated (Geyer, Lorenz, & Kandelbauer, 2016). The consumption of PET is creating environmental problems due to its short service life and poor biodegradability (El Mejjati et al., 2014).

PET is one of the most complex products to reuse or even recycle. Reusing in the same productive chain is unacceptable, since the containers used to create micronized PET can absorb contaminants. The interaction with asphalt mixture depends of the binder composition, and porosity and chemical nature of the aggregates (Qin, Schabron, Boysen, & Farrar, 2013). Micronized PET is obtained through a traditional friction-based micronization technique to reduce particle size.

Thermoplastic elastomers polymers are, nowadays, the most used modifier for binders, since it improves thermostability and aging resistance during mixing and construction (Mashaan, Ali, Karim, & Abdelaziz, 2014).

Physical and mechanical properties of PET are affected by crystallization and orientation (Chen, 2012; Wellen, 2014). Crystallinity is obtained by heating above the glass transition temperature (Tg) (Robertson, 1993). Below the glass transition temperature, polymer chains are rigid; after reaching the glass transition temperature, the chains become more flexible and are able to unfold under stress (Benning, 1983; Derimel, Yaras, & Elciçek, 2011).

When micronized PET is heated to a 72°C temperature, its form is slowly crystallized, and the material starts to become opaque, stiffer, and less flexible. Crystalline PET is able to resist higher temperatures and can be added to an asphalt binder to provide a better performance to the mechanical behaviour of asphalt mixtures. According to Collins, Bares, and Billmeyer (1973)
this material has higher elastic modulus, toughness, stiffness, tensile strength, hardness and more resistance to solvents, but less impact strength.

Silva, Lucena, Rodrigues, Carvalho, and Costa (2015) studied the influence of PET on asphalt binder properties. It was verified that the progressive addition of PET increased the consistency and physical properties of the modified binder and improves its elastic response.

The objective of this research is to verify if the addition of micronized PET waste in asphalt binder can be a viable alternative to improve mechanical properties of asphalt mixtures.

2. Materials and methods

2.1. Material’s characteristics

The materials for the HMA mix design (aggregates and asphalt binders) were collected and characterised according to ASTM testing procedure. This process was carried out according to ASTM standard for asphalt binder characteristics and the SUPERPAVE recommended evaluation tests.

The PET was collected from a local supplier in the city of Campina Grande, Paraiba, Brazil. The polymer was micronized through a physical process in which knives are used by an equipment for cutting PET bottles into small particles.

This research used a micronized PET, added in percentages of 4%, 5%, and 6% by weight with an asphalt binder base. For the mixing process, the base binder was heated at the temperature of 150°C. Then, it was stirred with a lab mixer with 60 rpm and 165°C, the amount of polymer was slowly added to the asphalt, in order to keep the PET in its natural state during the mix process, with minimum change in form and properties, and mixed for 2 hours. The specimens using modified binder were mixed at 165°C and compacted at 150°C.

2.1.1. Asphalt binders

Asphalt binder 50–70 penetration grade was selected for this research due to the most commonly used in the northeast region of Brazil. Its engineering properties are shown in Table 1.

The amount of micronized PET added to the asphalt binder was selected following the recommendations of Ahmadinia, Zargar, Karim, Abdelaziz, and Ahmadinia (2012). According to this author, the addition of contents between 4 and 6 wt% show acceptable mechanical tendencies within these limits, and satisfy the ASTM requirements.

The storage stability of asphalt binder with PET was determined according to DNIT ES 385/99. The samples were putted into an aluminium tube, and it was stored vertically in an oven for 5 days at 163°C. The difference of softening point between the top and the bottom sections of the tube was measured. Mixtures of asphalt binder with PET are heterogeneous; so it is

<table>
<thead>
<tr>
<th>Test</th>
<th>Result</th>
<th>Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Penetration 0.1 mm (100 g, 5 s a 25°C)</td>
<td>52.9</td>
<td>ASTM D946</td>
</tr>
<tr>
<td>Flash point (°C)</td>
<td>320.0</td>
<td>ASTM D3143</td>
</tr>
<tr>
<td>Softening point (°C)</td>
<td>39.6</td>
<td>ASTM D3461</td>
</tr>
<tr>
<td>Saybolt Furol viscosity (s)</td>
<td>310 (à 135°C)</td>
<td>ASTM D7496</td>
</tr>
<tr>
<td>Rotational viscosity (cP)</td>
<td>377.5 (à 135°C)</td>
<td>ASTM D4402</td>
</tr>
<tr>
<td></td>
<td>187.0 (à 150°C)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>68.5 (à 177°C)</td>
<td></td>
</tr>
</tbody>
</table>
Table 2. Aggregates’ physical properties.

<table>
<thead>
<tr>
<th>Aggregate</th>
<th>Specific gravity</th>
<th>Apparent specific gravity</th>
<th>Absorption (%)</th>
<th>Los Angeles abrasion (%)</th>
<th>Index form (%)</th>
<th>Sand equivalency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coarse aggregate 25 mm</td>
<td>2742</td>
<td>2719</td>
<td>0.32</td>
<td>36.1</td>
<td>0.90</td>
<td>–</td>
</tr>
<tr>
<td>Coarse aggregate 19 mm</td>
<td>2747</td>
<td>2719</td>
<td>0.39</td>
<td>34</td>
<td>0.85</td>
<td>–</td>
</tr>
<tr>
<td>Coarse aggregate 12.5 mm</td>
<td>2755</td>
<td>2722</td>
<td>0.45</td>
<td>23.0</td>
<td>0.73</td>
<td>–</td>
</tr>
<tr>
<td>Fine aggregate</td>
<td>2435</td>
<td>2430</td>
<td>0.04</td>
<td>–</td>
<td>–</td>
<td>64.90</td>
</tr>
<tr>
<td>Sand</td>
<td>2543</td>
<td>2540</td>
<td>0.08</td>
<td>–</td>
<td>–</td>
<td>80.96</td>
</tr>
</tbody>
</table>

recommended to use agitators in storage tanks in order to correct possible temperature differences of softening point.

2.1.2. Aggregates

The aggregates for research are from granitic origins, and show maximum diameters of 9.5, 19, and 25 mm. The physical properties of the aggregate samples were evaluated. These evaluations included coarse and flat/elongated particles, sand equivalency, coarse and fine aggregate specific gravity and absorption, and Los Angeles abrasion (Table 2). The selected aggregate gradation was carried out according to ASTM specifications. The recommended gradation for the national HMA mix design relied on the SUPERPAVE Mix design protocol.

2.2. HMA mix design

SUPERPAVE mix design protocols were used for the HMA mix design. The specifications of SUPERPAVE, which are the recommended gradations for wearing courses, were followed by optimising the aggregate grading characteristics. The Fuller curve (Figure 1) presents the asphalt mixture gradation.

Figure 1. Fuller curve for the project.
The compaction of specimens were performed in a SUPERPAVE Gyratory Compactor (SGC), following the procedures described in ASTM D 6925/08 (Preparation and Determination of The Relative Density of Hot Mix Asphalt – HMA Samples by Means of The SUPERPAVE Gyratory Compactor). All tested samples were prepared following the same compaction procedure of the gyratory compactor with 4% air void.

As stated previously, two SUPERPAVE designs were applied. The first one was performed with asphalt binder without additive to determine the optimum binder content of the SUPERPAVE mix design method. In the second one, polymer was added to the optimum asphalt binder in the amounts of 4, 5, and 6 wt%, and mechanical tests were performed. The mix design procedure (Mixture A) was carried out for the mixture without additives; so the effect of the additives could be analysed separately. Analysing the results from this stage, the sample which provided the best results was chosen, and an optimum binder content determination was performed following the SUPERPAVE method (Mixture E).

2.3. Mechanical tests
In order to observe the influence of the modified binder on mechanical properties, four asphalt mixtures were produced and characterised: Mixture A – without additives; Mixture B – with 4 wt% of micronized PET; Mixture C – with 5 wt% of micronized PET; and Mixture D – with 6 wt% micronized PET. The best composition for the HMA mixture was thus obtained using the SUPERPAVE design method.

The samples were tested regarding Indirect Tensile Resistance (ASTM D6931), Susceptibility to Moisture Damage (AASHTO T 283-02), and Resilient Modulus (ASTM 4123-82). It was done at least 3 replicates in each test.

According to the results, the optimum asphalt mixture has a micronized PET content of 5 wt%, because of its better performance on mechanical properties. With this amount of micronized PET, Mixture E was produced and tested for Resilient Modulus at different temperatures (25°C and 40°C), Fatigue, and Flow Number, besides for the three tests described in the paragraph above. These results were compared to new samples of Mixture A.

In Brazil, the most used fatigue test is the repeated load controlled force indirect tensile test, for which there is still no standard procedure (Babadopulos, Soares, & Branco, 2015). The Fatigue Cracking test was performed with applied pulse frequency of 1 Hz, in which 0.1 s was for the sinusoidal load application and 0.9 s were for rest. The test was carried out in the same equipment used for the Resilient Modulus tests (UTM-25), under controlled temperature of 25°C. The criterion for the end of the test was the occurrence of axial deformation of 1 mm. For this test, three levels of tension were used (10% ITS, 20% ITS and 30% ITS), which were defined after Indirect Tensile Strength tests results, following the procedure described by Babadopulos et al. (2015).

Flow Number tests were performed with the application of a load with a loading time of 0.1 s and rest time of 0.9 s, until a maximum of 10,000 cycles was reached according to AASHTO TP79. Load was applied in a level of 600 kPa, while the test was performed under a controlled temperature of 60°C.

3. Results
3.1. Mix design and mechanical properties
The results shown in Tables 3 and 4 are the average values of the parameters with addition of contents of micronized PET of 4, 5, and 6 wt% to the asphalt binder. Based on the analysis of
Table 3. Volumetric properties of neat binder and addition of 4%, 5%, and 6% of micronized PET.

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Air voids (%)</th>
<th>VMA (%)</th>
<th>VsFA (%)</th>
<th>Binder content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3.93</td>
<td>14.07</td>
<td>72.06</td>
<td>4.25</td>
</tr>
<tr>
<td>B</td>
<td>3.95</td>
<td>14.12</td>
<td>72.00</td>
<td>4.25</td>
</tr>
<tr>
<td>C</td>
<td>4.01</td>
<td>14.14</td>
<td>71.66</td>
<td>4.25</td>
</tr>
<tr>
<td>D</td>
<td>4.13</td>
<td>14.25</td>
<td>71.01</td>
<td>4.25</td>
</tr>
<tr>
<td>Criteria</td>
<td>4.00</td>
<td>≥ 13.0</td>
<td>65% and 75%</td>
<td></td>
</tr>
</tbody>
</table>

Table 4. Mechanical properties with the neat binder and addition of 4%, 5%, and 6% of micronized PET.

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Indirect tensile resistance (MPa)</th>
<th>Susceptibility to moisture damage (%)</th>
<th>Resilient modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1.22 ± 0.3</td>
<td>81 ± 3</td>
<td>8.138 ± 272</td>
</tr>
<tr>
<td>B</td>
<td>1.29 ± 0.5</td>
<td>80 ± 2</td>
<td>8.710 ± 310</td>
</tr>
<tr>
<td>C</td>
<td>1.34 ± 0.3</td>
<td>83 ± 3</td>
<td>10.704 ± 350</td>
</tr>
<tr>
<td>D</td>
<td>1.51 ± 0.4</td>
<td>93 ± 4</td>
<td>6.720 ± 235</td>
</tr>
</tbody>
</table>

volumetric and mechanical properties, the optimum content was chosen as the one which allowed the asphalt mixture to show the best performance, as shown in Section 3.2.

Tables 3 and 4 show an increase in the values of the mechanical properties of the mixtures with addition of micronized PET. The improved susceptibility to moisture damage can be explained by the partially of the PET acting as filler. So PET decreases the air voids and water absorption, and improves the adhesion and viscosity.

Despite resilient modulus having increased from Mixture B to mixture C, it appears that there was a decrease for mixture D. In mixtures with higher fine content (6%), the coarse aggregates are floating on the surface of asphalt mixtures without the contact of particles, which makes it less resilient with less rough surface.

The chosen criteria for optimum amount of PET added to the asphalt mixtures followed the volumetric parameters proposed by the SUPERPAVE method – 4% of air voids. In this case, Mixture C satisfying the volumetric parameters and susceptibility to moisture damage according to AASHTO, besides presenting the highest resilient modulus. Thus, the addition of 5% of PET was used in the second SUPERPAVE mix design (Section 3.2).

3.2. **SUPERPAVE mix design with modified asphalt binder**

The SUPERPAVE method with the modified binder was performed similarly to the one without PET on this research. The criterion for the chosen curve was the one that represents the best performance for the method with the asphalt binder without additive. In this process, 5wt% of micronized PET is mixed with the binder, and a SUPERPAVE mix design with the modified binder was carried out.

Tables 5 and 6 show a synthesis of the medium value of volumetric and mechanical properties of Mixture E.

Mixture E showed significant improvements on the mechanical properties of mixtures in comparison to the results obtained with Mixture A, since there was an increase in 16% on Indirect Tensile Strength, 6.5% on the percentage of susceptibility to moisture damage, and 43% on resilient modulus. Besides, Mixture E has a reduced binder content from 4.25% to 4.00%, which could imply a more economic project.
In case of design methods for flexible paving, it is reasonable to evaluate the deformation compatibility between the layers of the paving’s structure. On the other hand, it is expected that the deformation compatibility is beneficial to the fatigue cracking of the structure with the use of the modified binder.

The increase observed on the mechanical parameters of the mixtures using modified binder can be associated to the fact that micronized PET is a semi crystalline material inside the asphalt mixture. Micronized PET, when added to the asphalt mixture, forms a layer around the aggregate and probably improve an interaction between the carbon chains from PET and asphalt binder, decreasing the susceptibility to moisture damage.

The temperature of 25°C has been commonly used as reference for the resilient modulus test. However, it is possible to perform this test under lower or higher temperatures to analyse the thermal effect on the asphalt mixtures’ performance.

Results presented in Table 7 shows the resilient modulus results at temperatures of 25°C and 40°C. Results indicate that the mixtures perform differently at different temperatures, achieving better performances when the parameter of 25°C was used. Results for the flow number tests performed on Mixtures A and E are shown in Table 7 as well.

In Table 7 it is seen that the permanent deformation of asphalt mixtures decreased with the addition of PET, once the flow number value decreased around 24%. The flow number, performed at 60°C on plastic region, shows that Mixture E is more susceptible to permanent deformation than the pure binder (Mixture A). In other words, although there is an increase in stiffness of 75% on asphalt mixtures with polymer addition, PET addition might not be beneficial in a plastic region.

The results of the fatigue cracking tests are shown in Figure 2. It shows the number of cycles regarding the stress level, in logarithmic scale, for mixtures A and E.

It is clear that the mixture prepared with the micronized PET modified binder has a better behaviour than the mixture prepared with the pure binder. The effect of polymer on asphalt
mixtures increased the mixture’s fatigue resistance, besides being possible to obtain a better statistic correlation, with $R^2$ in the order of 0.92.

4. Conclusions

The objective of this research was to evaluate the mechanical properties of modified asphalt mixtures with micronized PET waste. According to the results, it is possible to conclude that micronized PET waste improves asphalt mixtures’ mechanical behaviour. The addition of micronized PET to the asphalt binder increased the parameter of indirect tensile strength, resistance to moisture damage, fatigue life and resilient modulus. However, in this case, an increase in permanent deformation with PET addition was observed.

The resistance to moisture damage is the least in mixtures with PET, probably because PET improves cohesion in a mastic, by the influence of the filled binder’s rheology, surface energy, and mechanical adhesion.

The asphalt mixtures using the micronized PET modified binder went through improvements of 25% in fatigue life compared to the conventional mixtures, which is beneficial for increasing the service life by reducing fatigue cracking.

PET modified binders have had proved to be successful in laboratory level, and a continuing effort should be made to develop a correlation between results from laboratory tests and field performance.

Researchers plan to evaluate the individual properties of each fraction of asphalt mixtures with PET and the influence of different times, rotation and temperature in the mixtures with asphalt binder and PET. Assimilating more information in this area can eventually lead to a knowledge base that can be used to engineer better asphalt binders and asphalt binder modifiers.

Disclosure statement

No potential conflict of interest was reported by the authors.
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