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Valorization of vinasse as binder modifier in asphalt mixtures

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Abstract
The reutilization of waste generated by industrial processes has become a major environmental objective in scientific and technical research. In the construction sector, there is a broad range of techniques for the exploitation of different types of waste, which can then be used as a replacement for raw materials. This paper presents the results of a study of vinasse, a by-product of biomass ethanol, and analyzes its viability as a bitumen modifier in asphalt mixes. For this purpose, four AC-16S asphalt mixes were evaluated for moisture sensitivity, plastic deformation, stiffness, and fatigue. The mix formulas were the following: (Mix 1) 50/70 bitumen; (Mix 2) 50/70 bitumen modified with 10% vinasse; (Mix 3) rubber bitumen; (Mix 4) rubber bitumen modified with 10% vinasse. The results of this study showed that bitumen modified with vinasse improved the mechanical performance of the AC-16S mix and also contributed to the valorization of vinasse waste.

Keywords: Asphalt mixture, binder modifier, mechanical behavior, vinasse.

1. Introduction
Vinasse is a by-product that remains in the distillation column when ethanol is produced from biomass. It is the main waste material generated in alcohol distilleries and its release without previous treatment has an extremely negative impact on the environment. The composition of vinasse can vary depending on the nature of the distillation process. However, vinasse typically has a low pH (3-5) and a high content of organic matter (35,000-50,000 mg O₂/L BOD), which makes it a toxic substance[1].

The uncontrolled discharge of vinasse into the soil can negatively affect soil quality by increasing the salinity of the soil, thus deteriorating its structure, porosity, and fertility[2]. Furthermore, since the production temperature of vinasse is 50-80°C, its release into aquatic environments without previous cooling can increase water temperature and lower the oxygen content to a level at which fish cannot survive[3,4]. In addition, the turbidity and color of vinasse can trigger processes of photosynthesis that also endanger aquatic life forms [5].
Currently, more and more countries are enacting stricter laws that regulate the discharge of waste from alcohol distilleries. For example, in 2005, the Indian government decided to rapidly transform distillation industries into zero-discharge industries [6]. Europe is also implementing similar measures to achieve the same objective by processing waste in waste treatment plants as well as by reusing and recycling it[7].

The anaerobic digestion of vinasse is an interesting way to reduce vinasse waste, because, in addition to promoting the stabilization of organic matter, it also enables energy generation from biogas, as Bruna S. Moraes reflects in her study[8].

Another way to reuse waste is by incorporating it into bituminous mixes. A wide range of studies have analyzed how the characteristics of these mixes can be enhanced with the addition of recycled waste material or by using this waste instead of one of the mix ingredients. This is an excellent way of reducing waste deposits and discharges. It is also more cost-effective since there is less need to use raw materials. Glass, steelworks slag, plastic, and rubber are examples of different types of waste material that have been used in asphalt mixes[9]. However, in recent years, the main focus has been on rubber from end-of-life tires and polymers [10-14].

In this study, vinasse was the waste used to manufacture bituminous mixes. The objective was to study the mechanical performance of these mixes when vinasse binder was added to modify the bitumen. For this purpose, we analyzed an AC 16 S bituminous mix, manufactured with 50/70 bitumen modified with 10% vinasse, as well as a rubber bitumen mix also modified with 10% vinasse. The performance of the vinasse mixes was compared to that of the same mixes manufactured with unmodified bitumen and rubber bitumen.

2. Material and methods

For this study the asphalt mix used was an AC-16S mix, which is commonly used in wearing courses in countries throughout the world. It is a dense asphalt concrete mix with an air voids percentage of 4-6%, a continuous coarse grain size (maximum particle size of 22 mm) and a bitumen content of 3.5-5% of the total mix weight. The mix is usually spread in layers of 5-8 centimetres.

Before beginning the battery of laboratory tests, four AC-16 S mixes were manufactured. The first two were regarded as the reference mixes. The first mix was manufactured with conventional penetration grade bitumen B 50/70 and the second with rubber bitumen. The third and fourth mixes were also manufactured with the same types of bitumen as the first two, but they were modified with biopolymers obtained from vinasse. In other words, the third mix was made from penetration grade bitumen B 50/70 modified with 10% vinasse and the fourth mix was made from rubber bitumen, also modified with 10% vinasse.

The bitumen was supplied by the University of Huelva in southern Spain where it was characterized and modified with the previously mentioned percentage of vinasse. The laboratory tests in this research study start with the design of the asphalt mixes by determining the optimal bitumen content for four mixes with the same mineral skeleton. This bitumen content was ascertained with the Marshall Test, as described in the Spanish Standard NLT 159/00 and in article 542 (guidelines for hot bituminous mixes) of the General Technical Specifications for Road and Bridge Works (PG3).

As previously mentioned, the substance used to modify the bitumen was vinasse, the liquid waste remaining after alcohol distillation. The phase separation of vinasse effluent (pH = 4.58) is characterized by a precipitate with a higher concentration at the bottom and a supernatant with suspended solids on the surface. Before being mixed with the bitumen, the vinasse was first dehydrated at 70°C in a convection oven. Its grain size was reduced with a laboratory blade mill (IKA, Werke model) as well as a sieve with a mesh size of 3.0 mm. The final product was then added to the bitumen in a proportion of 10%.

Ophite was used as an aggregate in the mix for the coarse fraction, and limestone was used for the fine fraction. The filler was CEM II /B-L 32.5 N cement. The materials in the mixes were first characterized as described in article 542 (guidelines for hot bituminous mixes) of the General Technical Specifications for Road and Bridge Works (PG3).

The same set of tests was then applied to all four mixes in order to study their mechanical performance and to compare the mixes with modified bitumen with the mixes made of unmodified bitumen. The tests performed were the following:

- The moisture sensitivity test. Water action is one of the most common road pavement pathologies. The presence of moisture causes problems such as potholes, aggregate peeling or stripping which eventually lead to the structural failure of the pavement. There are numerous laboratory tests that analyze the susceptibility of bituminous mixes to moisture, providing a qualitative or quantitative evaluation [15]. In this work, the test performed was the moisture sensitivity test as described in the Spanish Standard UNE-EN 12697-12, which determines the response of mixes to water action.

- The wheel-tracking. Rutting causes the loss of road surface regularity, so it is necessary to analyze this damage to avoid a negative impact on the quality of service to users. In this work, the test performed was the wheel-tracking test as described in the Spanish Standard UNE-EN 12697-22 [16].

Determination of the dynamic modulus according to the Spanish Standard UNE-EN 12697-26 to calculate the stiffness of the samples. The modulus is a parameter used in the mechanical design of asphalt mixes. For this reason, it was interesting to discover whether the modulus value changed when the bitumen was modified [17].

The fatigue test characterizes the performance of bituminous mixes when they are subjected to repeated loads at a constant mode of loading. A mix’s fatigue resistance can be determined by different methods such as two, three, or four-point bending flexural tests on parallelepiped test specimens or the diametral compression test in which loads are applied to test cylinders. This test is described in Annex E of the Spanish Standard UNE-EN 12697-24 and was the one used in this research.

The experimental design is shown in Fig. 1:
3. Results and Discussion

The mineral skeleton of the mix had the following composition: (i) 5% ophite aggregate with a grain size of 12/18; (ii) 50% ophite aggregate with a grain size of 6/12; (iii) 40% limestone aggregate with a grain size of 0/6 (sand); (iv) 5% cement filler. The composition of the mineral skeleton is shown in Table 1.

The aggregate was mixed in the percentages shown in Table 1, and the following grain size of the combined aggregate was obtained. When represented in a graph, (Fig.2), the grain-size curve of the mineral skeleton is a continuous line that falls within the envelope of an AC mix.

As previously mentioned, the mixes in this study all had the same mineral skeleton in which the combined aggregate fit within the grain-size envelope corresponding to that of an AC 16S mix.

The optimal binder content was determined by means of the Marshall Test as described in Spanish Technical Standard NLT 159/00. The parameters used for this purpose were the following: Design Marshall stability, Design Marshall deformation, Maximum Marshall stability, Maximum Marshall deformation, Density, Air voids and aggregate voids.

Regarding the first two mixes, Mix 1 was made from 50/70 bitumen, and Mix 2 was made from 50/70 bitumen modified with 10% vinasse. Both mixes had the mineral skeleton shown in Table 1. A set of test specimens were then manufactured, each with a different binder content (3.5%, 4%, 4.5%, and 5% b/m). The manufacturing temperatures were 160°C for the 50/70 bitumen mix (Mix 1) and 170-175°C for the bituminous mix modified with vinasse (Mix 2). The compaction temperatures were 155-160°C for the first mix and 165-170°C for the second.

The specimens were compacted with 75 blows to each side by a Marshall hammer. The density of each specimen was determined according to Spanish Standard UNE-EN 12697-6 Procedure B, Dry Saturated Surface. Air voids were calculated by determining the maximum density (UNE-EN 12697-5).Procedure A: Volumetric of the mix for each of the specimens).

To obtain the stability and deformation values, the specimens were fractured in the Marshall stability breaking head. Based on the results of this test, the optimal bitumen content was 4.0% of the mix weight. This same process was also followed to determine the bitumen content for the second mix with bitumen modified with 10% vinasse. In this case, the optimal bitumen content was 4.2% of the mix weight. Regarding the mixes made of rubber bitumen, and of rubber bitumen modified with 10% vinasse, the optimal binder contents were 4.5% and 4.6%, respectively.

As can be observed, the optimal bitumen content was slightly higher when the bitumen binder was modified with vinasse. This occurred because the addition of this biopolymer also increased binder viscosity. Consequently, more binder was needed to guarantee a good mixture of the aggregate.

After the optimal bitumen content was calculated, the moisture sensitivity of the mix was tested. Table 2 shows the values obtained:

<table>
<thead>
<tr>
<th>Test Description</th>
<th>AC-16</th>
<th>AC-16 S</th>
<th>AC-16</th>
<th>AC-16 S</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Bitumen</td>
<td>50/70</td>
<td>50/70+10%</td>
<td>50/70</td>
<td>50/70+10%</td>
</tr>
<tr>
<td>% Vinasse</td>
<td>Mix</td>
<td>Ref. Mix</td>
<td>Mix</td>
<td>Ref. Mix</td>
</tr>
<tr>
<td>Number of specimens tested</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>Time elapsed between mixing and testing</td>
<td>4 days</td>
<td>4 days</td>
<td>4 days</td>
<td>4 days</td>
</tr>
<tr>
<td>Temperature and immersion time of the wet specimens</td>
<td>40 °C</td>
<td>40 °C</td>
<td>40 °C</td>
<td>40 °C</td>
</tr>
<tr>
<td>Testing temperature</td>
<td>15 °C</td>
<td>15 °C</td>
<td>15 °C</td>
<td>15 °C</td>
</tr>
<tr>
<td>Mean ITSd (KPa)</td>
<td>1679.4</td>
<td>2083.3</td>
<td>1786.0</td>
<td>1860.8</td>
</tr>
<tr>
<td>Mean ITSR (%)</td>
<td>90.9</td>
<td>84.6</td>
<td>80.2</td>
<td>87.9</td>
</tr>
</tbody>
</table>

Source: The authors
Table 3. Results of the Wheel-tracking Test

<table>
<thead>
<tr>
<th></th>
<th>AC-16 S Ref. Bitumen 50/70</th>
<th>AC-16 S B 50/70 + 10% Vin.</th>
<th>AC-16 S Ref. Rubber Bitum.</th>
<th>AC-16 S Rubber Bitumen + 10 % Vin.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean WTS (mm/103 load cycles)</td>
<td>0.088</td>
<td>0.07</td>
<td>0.076</td>
<td>0.087</td>
</tr>
<tr>
<td>Test temperature (°C)</td>
<td>60</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Source: The authors

Table 4. Stiffness results

<table>
<thead>
<tr>
<th>Stiffness Modulus 20°C (MPa)</th>
<th>AC-16 S Ref. Bitumen 50/70</th>
<th>AC-16 S B 50/70 + 10% Vinasse</th>
<th>AC-16 S Rub. Bitumen</th>
<th>AC-16 S Rub. Bitumen + 10% Vinasse</th>
</tr>
</thead>
<tbody>
<tr>
<td>4462</td>
<td>8807</td>
<td>4212</td>
<td>8237</td>
<td></td>
</tr>
</tbody>
</table>

Source: The authors

The results of the moisture sensitivity test for reference Mix 1 with bitumen 50/70 were very satisfactory with an ITSR value of 90.6%. When this bitumen was modified with vinasse (Mix 2), the ITSR value was somewhat less. This less satisfactory performance could be due to a slight reduction in aggregate-binder adhesion caused by the incorporation of the biopolymer. Reference Mix 3 with rubber bitumen obtained an ITSR of 80.2%. However, in the case of Mix 4, rubber bitumen modified with vinasse, the addition of the biopolymer improved mix performance by 7.7% with an ITSR value of 87.9%.

Another result worth highlighting is the increase in indirect tensile strength in the four groups of test specimens, the dry specimens as well as the wet ones. This shows that the vinasse-modified binder improved mix cohesion.

Results obtained of the Wheel-Tracking Test are shown in Table 3.

**Figure 3. Fatigue laws for bitumen B 50/70, and vinasse-modified bitumen**

Source: The authors

**Figure 4. Fatigue laws for rubber bitumen and vinasse-modified rubber bitumen**

Source: The authors

The analysis of the fatigue lines shows that in the initial cycles, the performance of the reference mix with bitumen B 50/70 was more satisfactory than that of the other mixes. However, at a stress less than 400 KPa, the vinasse-modified bitumen became more resistant to fatigue. As can be observed, the slope of the line representing the fatigue life of this mix becomes considerably gentler when compared to that of the reference mix.

In Fig. 4, the performance of the mixes manufactured with rubber bitumen and vinasse-modified rubber bitumen shows a similar tendency. At stresses less than 700KPa, the fatigue life of the vinasse-modified rubber bitumen mix was found to be more satisfactory.

**4. Conclusions**

Based on the results obtained, it was found that the use of vinasse-modified bitumen influenced the mechanical performance of the bituminous mix used in the study. Generally speaking, the modified bitumen had a positive impact since its addition produced an overall enhancement of the characteristics of the mix. The following conclusions can be derived from this study:
1. Vinasse enhances mix cohesion. This was evident in the increase in indirect tensile strength, as reflected in the values obtained in the fracture of the specimens with the Marshall stability breaking head as well as in the results of the indirect tensile test to perform the moisture sensitivity test.

2. The variations obtained in the permanent deformation tests were not very significant. Nevertheless, in one of the cases, there was a slight improvement in mix performance, possibly due to the lesser influence of the temperature on the mix manufactured with vinasse. These results complied with the requirements for the most demanding traffic loads. Furthermore, the deformation values obtained were lower than that of the reference mix.

3. Regarding the dynamic tests, the stiffness of the mix increased to almost double its value.

4. The expressions for the fatigue laws reflected an improvement in the performance of the mixes modified with 10% vinasse, when subjected to repeated loads.

5. The results obtained in this research study show that when vinasse was used to modify the bitumen in the AC-16 S bituminous mixes studied in this work, the addition of this bioplastic improved their mechanical performance and at the same time, revalued a highly-polluting industrial waste that is otherwise very difficult to eliminate.

Acknowledgments

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References


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