

Physical and Thermo-Oxidative Characterization of Asphalt Modified with High Density Polyethylene and Recycled Engine Oil

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Abstract

Modification of asphalt using polymers, oils and other additives has been an option to improve asphalt pavement performance and extend its lifespan. The present work aims to evaluate the influence of the addition of engine oil on the consistency and thermal properties of HDPE-modified asphalt. For this study, compositions containing asphalt, engine oil and high-density polyethylene (HDPE) were prepared, varying the concentration of engine oil by 2.5 wt%, 5 wt%, 7.5 wt% and 10 wt% and keeping the concentration of HDPE at 5 wt%. The samples were characterized by conventional tests of penetration, softening point and viscosity, aging in a Rotational Thin Film Oven (RTFO), Thermogravimetric Analysis (TGA). According to the results, the addition of HDPE to virgin asphalt causes an increase in the consistency of the virgin asphalt, which then decreases linearly as the engine oil is added into the matrix. Conventional tests showed improvements in the applicability of the asphalt in terms of resistance to cracks and permanent deformation. TGA showed a slight increase in stability for the modified asphalt samples at elevated temperatures. The RTFO showed mass gain and loss for samples with and without engine oil, respectively.

Keywords

Asphalt, Engine Oil, High-Density Polyethylene, Polymer Modified Asphalt

1. Introduction

Asphalt is a building material widely used in road paving. Currently, 95% of the approximately 100 million tons of asphalt produced worldwide are used in paving, where asphalt serves as a binder for mineral aggregates. Generally, paved

roads are designed to last 10 - 15 years. Many suffer degradation and need maintenance before 10 years of construction, as the asphalt ages due to factors such as sunlight, rain water, and repeated loads [1] [2].

The asphalt pavement degradation that occurs due to asphalt aging caused by load conditions and climatic factors gives rise to cracks and permanent deformation, considered the main problems of the asphalt pavement [3]. As a way to improve asphalt quality, the number of investigations focused on asphalt modification has increased. Thus, asphalt pavement performance is being improved by modifying the asphalt using virgin or recycled modifiers [4] [5].

As a way to guarantee the performance of the asphalt in different environments and improve or promote the asphalt properties, materials such as styrene-butadiene-styrene (SBS), styrene-butadiene rubber (SBR), crumb rubber (CR), polyphosphoric acid (PPA), polyethylene (PE), wood derived bio-oil, cooking oil and engine oil are being considered [6].

The addition of synthetic polymers such as thermoplastics and elastomers is the most common method to improve pavement performance. This approach offers solutions to overcome the inherent deficiencies of asphalt on pavement, where it is subject to excessive load requirements and extreme weather conditions [7] [8]. The improvement of the properties of polymer-enhanced asphalt include permanent deformation, thermal and fatigue cracking, wear and aging resistance [9]. However, despite the large number of polymers available, limitations such as compatibility and the ability of the selected polymer to maintain its main properties when mixed with equipment used in conventional mixing processes mean that there are relatively few suitable polymers for asphalt modification [9] [10].

Due to the urgent need to carefully control the use of hazardous materials and the current high demand for asphalt rejuvenators, the application of bio-based material residues and residues from other industries has recently also been investigated. These residues include recycled coal tar, coconut oil, engine oil, vegetable oil, orange oil and cooking oil, etc. as rejuvenating agents [2].

Considering the slow degradation rate and high toxicity of the residual engine oil, when not properly deposited or eliminated, it might threaten the human health, the environment, especially affecting aquatic life, polluting the soil [11] [12]. Recycling engine oil using conventional techniques such as vacuum distillation and refining with sulfuric acid can have a significant impact on the environment. Despite this, it has a disadvantage of requiring costly technology [13]. A study cited by Liu *et al.* (2018) showed that most residual engine oil is used as fuel. However, burning residual engine oil generates pollutants that can be airborne and can enter people's lungs and create adverse health effects. Therefore, the use of residual engine oil has gained wide attention in recent years in response to waste management, environmental concerns and economic benefits. Since engine oil is not fully recyclable, its incorporation into asphalt is an environmentally friendly solution and increases its commercial value. Research over the past decade indicates that engine oil from vehicles has the potential to improve asphalt performance [6] [14].

When added to asphalt, residual engine oil increases penetration, lowers softening point temperatures and asphalt viscosity, thus lowering asphalt mix production and compaction temperatures. However, as a result of their incorporation, elastic recovery and resistance to permanent deformation can be compromised, which requires careful design for mixing these materials [15] [16]. The amount of engine oil to be incorporated into asphalt is limited in order to avoid hardening problems, but the use of polymers can be a solution to this limitation [17]. Considering that polymers are used to solve some of these problems, modifying asphalt with residual engine oil and polymers can be a promising, ecological and economical solution for the paving industry [15]. This work aims at evaluating the influence of the addition of engine oil on the consistency and thermal properties of HDPE-modified asphalt.

2. Materials and Methods

2.1. Materials

Asphalt, high-density polyethylene (HDPE) powder and recycled engine oil, designated as A, HDPE and O, respectively, were used to carry out the experiments. The asphalt is a 50/70 penetration grade, supplied by Puma Energy. Some physical properties of the asphalt are shown in **Table 1**. HDPE (0.95 g/cm³, 1.7 g/10min melting flow index) was supplied by Sasol, South Africa. The engine oil was collected from different auto repair shops located in Maputo City.

2.2. Methods

2.2.1. Preparation of Modified Asphalt Samples

A composition containing only asphalt and high-density polyethylene and four compositions containing asphalt, high-density polyethylene and engine oil were prepared. All the modified asphalt samples were prepared using an IKA brand high-shear mechanical mixer, model EUROSTAR 20. **Table 2** presents the description of the samples.

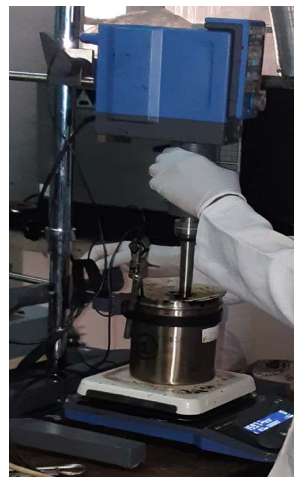
The sample preparation process began with the weighing step, where the asphalt was heated until it became fluid, placed in a stainless-steel container and allowed to cool down. The asphalt mass was obtained by calculating the difference between the mass of the container without asphalt and the mass of the container with asphalt. The samples were then transferred to the mixer and mixed in different conditions. **Figure 1** shows the mixing of samples.

Table 1. Physical Properties of the asphalt.

Property	Value
Softening Point, °C	45 - 52
Penetration at 25°C, dmm	35 - 100
Flash Point, °C	>230
Solubility	Soluble in most organic solvents
Density, kg/m ³	990 - 1300

Table 2. Description of the samples.

Sample name	HDPE, wt%	Oil, wt%
AH5	5	-
AH5O2.5	5	2.5
AH5O5	5	5.0
AH5O7.5	5	7.5
AH5O10	5	10.0

**Figure 1.** Sample preparation (mixing).

The sample containing asphalt and polymer only, was prepared in a metal container with asphalt that was heated in an oven to 170°C. The container was then transferred to the mixer and the polymer was gradually added at a speed of 500 rpm over 30 min. The rotation speed was gradually increased by 500 rpm every minute until it reached 5000 rpm. Stirring was continued for 90 min.

For the preparation of samples containing asphalt, HDPE and engine oil, the asphalt container was heated in an oven up to 170°C. The engine oil was added, at a speed of 500 rpm; then, the polymer was gradually added at the same speed for 30 min. The mixing speed was gradually increased by 500 rpm/min until it reached 5000 rpm and the stirring was continued for 90 min.

2.2.2. Characterization of Samples

Penetration

The penetration test measures the consistency of the asphalt. It was measured using a penetrometer applying a load of 100 g to the sample for 5 s at 25°C. The penetration test was carried out according to the specifications and procedures described in the ASTM D5 standard [18] [19].

Softening Point

The softening point is a measure of a temperature at which asphalt begins to show fluidity [20]. During the test, two asphalt samples were placed in metal rings, heated in a controlled manner in water placed in a beaker cup, with each

ring filled with asphalt supporting a steel ball. The softening point is reported as the average of the temperatures at which each asphalt-covered ball travels a distance of 25.0 mm. The test was performed according to the specifications and procedures described in the ASTM D36 standard [21].

Temperature Susceptibility

The thermal susceptibility of asphalt is evaluated using the penetration index (PI). The PI is a measure of the asphalt's temperature sensitivity and was obtained by correlating the penetration and softening point values using the Equation (1), as described by [22].

$$PI = \frac{1952 - 500 \log Pen - 120SP}{50 \log Pen - 50 \log Pen - 120} \quad (1)$$

where PI is penetration index, Pen is the penetration at 25°C, and SP is the ring and ball softening point temperature.

Viscosity

The viscosity was determined at 165°C using a rotational viscometer. This consisted of a thermostatically controlled chamber containing a sample of hot asphalt. The torque on the apparatus-measuring geometry, rotating in a thermostatically controlled sample holder containing a sample of asphalt, was used to measure the relative resistance to rotation. The test was performed according to the specifications and procedures described in the ASTM D4402 standard [23].

Short Term Aging (RTFO)

This test indicates the approximate change in asphalt properties during mixing with aggregate. During the test, a film of asphalt material in a moving glass container was heated in an oven for 85 min at 163°C. The effects of heat and air were determined from changes in the values of the physical tests before and after the treatment in the oven. The test was performed according to the specifications and procedures described in the ASTM D2872 standard [24].

Thermal Analysis

The thermal analysis were performed using a TA instrument, model SDT Q600. The samples were weighed and subsequently heated on the instrument in an air atmosphere with a flow of 100 mL/min, from 25°C to 600°C, at a heating rate of 10°C/min.

3. Results and Interpretation

3.1. Penetration

Figure 2 shows the penetration test results for virgin and modified asphalts. It can be seen that the addition of HDPE decreases virgin asphalt penetration from 52 to 23 dm, indicating an increase in asphalt stiffness, making it more resistant to permanent deformation at high temperatures. However, this increase in stiffness might make the asphalt more fragile, and more susceptible to thermal cracking under severe temperature conditions [25] [26]. This disadvantage appears to have been reduced with the addition of engine oil. Accordingly, the

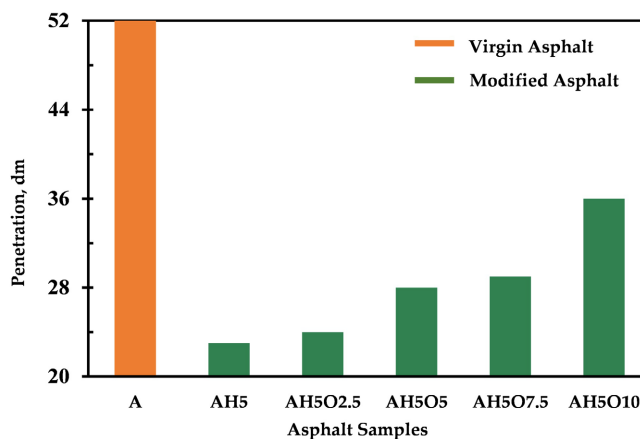


Figure 2. Penetration test results.

penetration values increased linearly as the concentration of engine oil increases, showing a reduction in the stiffness of the asphalt modified by HDPE, which makes it less susceptible to thermal cracks.

The observed results can be explained by the fact that the HDPE particles are absorbed by the lighter fraction of the asphalt (the maltenes), making the composite matrix deficient in maltenes and enriched in asphaltenes, resulting in an increase in the consistency of the asphalt modified by HDPE [27]. On the other hand, the main components of engine oil resemble the light fraction of asphalt [28]. Thus, it can be suggested that the engine oil acts by replacing the light components of the asphalt formerly absorbed by HDPE in the matrix of the HDPE-modified asphalt.

3.2. Softening Point

According to **Figure 3**, the addition of HDPE causes an increase in the softening point of pure asphalt from 47°C to 63°C, while the addition of engine oil over HDPE-modified asphalt linearly reduces the softening point from 63°C down to 50°C. Similar to what was observed in the penetration test, the addition of HDPE to virgin asphalt causes a deficiency of maltenes molecules in the composite which results in an increase in the consistency of the virgin asphalt. The latter, linearly decreases as the engine oil is increasingly added, without, however, reaching the softening point of the virgin asphalt.

These results show that the asphalt modified by either HDPE or HDPE and engine oil is less thermally susceptible, more resistant to hot climates and has a lower tendency to soften in hot conditions.

3.3. Thermal Susceptibility

The addition of HDPE to virgin asphalt increased the PI (**Figure 4**). When the engine oil was added over HDPE-modified asphalt the PI decreased with increasing engine oil concentration until it became lower than virgin asphalt for 10% engine oil concentration. Asphalts with a higher PI value have lower thermal susceptibility as well as a better resistance to cracks and permanent deformation

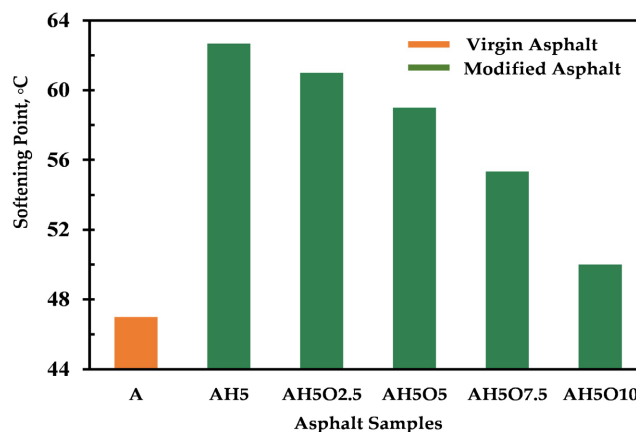


Figure 3. Softening point test results.

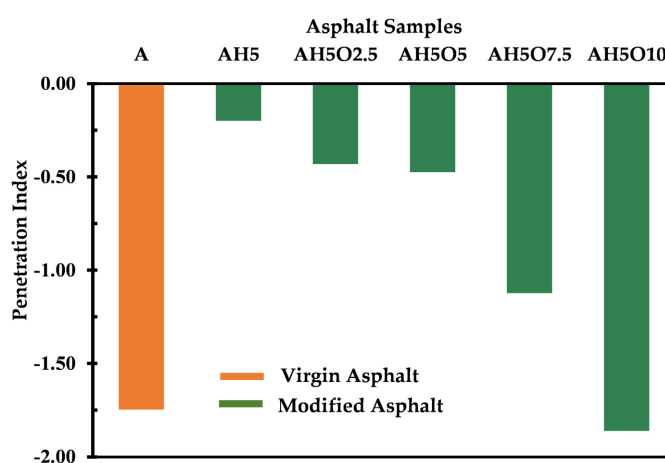


Figure 4. Penetration index of asphalt and its modified forms.

[29]. Thus, these results suggest that the modification of asphalt with 5% HDPE presents a good resistance to cracks and permanent deformation. Furthermore, the addition of engine oil on asphalt with 5% HDPE showed a reduction in its resistance to cracks and permanent deformation as the concentration of engine oil increased.

For paving applications, asphalt typically has to have PI values between -2 and $+2$ [30]. According to the present results, all samples have tolerable PI values for paving applications.

The PI was considered a good indicator of the colloidal structure of the asphalt, with PI values > 2 being indicators of a sol-type asphalt and PI values < 0 being indicators of a gel-type asphalt [27]. According to these results, virgin asphalt has a gel-like structure that does not change even when modified by HDPE and engine oil.

3.4. Viscosity

Figure 5 shows the results of dynamic viscosity at 165°C . It can be observed that the addition of HDPE to virgin asphalt causes an increase in viscosity from 0.08

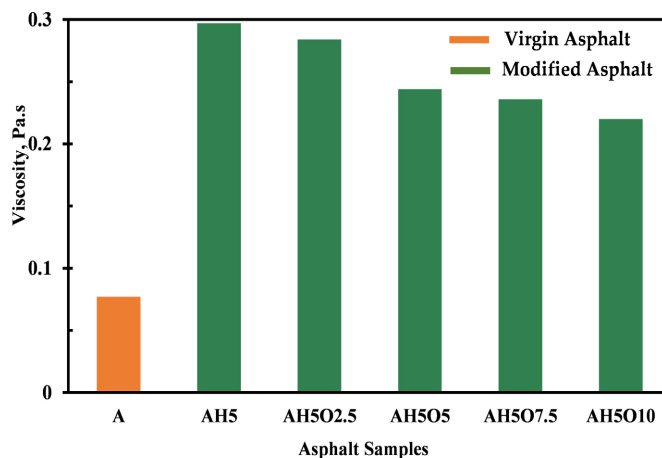


Figure 5. Dynamic Viscosity results at 165°C.

to 0.30 Pa.s. It is worth noting that the addition of engine oil over HDPE modified asphalt causes a decrease in viscosity from 0.30 to 0.22 Pa.s. This behavior agrees with the results obtained by the penetration and softening point tests which showed an increase in asphalt consistency with the addition of HDPE to virgin asphalt and a decrease with the addition of engine oil.

The results of the viscosity test are important for determining the mixing and compaction temperatures of asphalt mixtures. The equiviscous method, the most recommended for the determination of mixing and compaction temperatures, establishes that the temperatures to produce asphalt mixtures are those at which the viscosity values are between 0.17 ± 0.02 Pa.s and the compaction temperatures of asphalt mixtures are those in which the viscosity values are between 0.28 ± 0.03 Pa.s. However, the application of the equiviscous method may not present a realistic approach regarding the mixing and compaction of modified asphalt. Therefore, alternative methods that should be reliable and more applicable are needed [31] [32]. Yildirim *et al.* (2006) [33] proposed a method that couples a shear rate of 500 s^{-1} and a new range of viscosity values between 0.275 and 0.550 Pa.s for the determination of mixing and compaction temperatures. According to the viscosity results at 165°C, the modified asphalt did not present viscosity values within the ranges established by the equiviscous method for mixing temperatures of unmodified asphalts. However, all the modified asphalt samples showed viscosity values within the ranges established by the proposed method by Yildirim *et al.* (2006) [33] for mixing temperatures of modified asphalts. These results show that the modification of asphalt with HDPE and/or engine oil improves the applicability of virgin asphalt, at least, in terms of suitability at the mixing temperatures.

3.5. Short Term Aging

Figure 6 shows the short-term aging results obtained for the pure and modified asphalt samples. Positive mass change values are mostly due to the occurrence of reactions with oxygen, while negative mass change values are due to evaporation

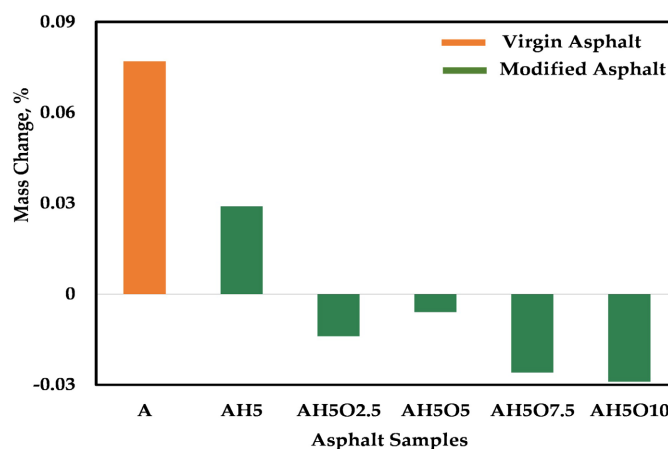


Figure 6. Short term aging results.

of volatile components. The virgin asphalt and asphalt modified only by HDPE showed positive mass change values, while all samples of asphalt modified by HDPE and engine oil exhibited mass losses. Among the samples that contain engine oil, the one with 5 wt% of oil revealed less mass loss, suggesting that in this composition there might be a greater degree of absorption of maltene molecules by the polymer matrix enriched by oil. It is well known that samples with a very low percentage of volatile components generally exhibit a mass gain, while with a high percentage of volatile components exhibit mass loss [24]. According to the results, the addition of HDPE to virgin asphalt improves its thermal stability at high temperatures; on the contrary, the addition of engine oil to HDPE-modified asphalt reduces overall thermal stability. This mass loss can be ascribed to the relatively lower degradation temperature of the compounds present in the added engine oil. However, these values, with a mass loss of less than 1%, are still within the required ranges [34] [35].

3.6. Thermal Analysis

It is important that the onset degradation temperature of the selected modifier is above the asphalt modification temperature or the asphalt mix production temperature. Otherwise, it would lose its initial properties by the time the modification process is complete [36]. From **Figure 7** and **Figure 8**, where TGA results are shown, it can be seen that the degradation process of the modifiers starts above the asphalt modification temperature and that the selected modifiers maintain their integrity above the asphalt modification process temperature.

The degradation process of the recycled engine oil occurs in three stages in the range of approximately 85°C - 511°C (**Figure 7**). The slight mass loss that occurs at temperatures close to 100°C is due to the evaporation of the unbound water present in the recycled engine oil [37] [38]. Below 250°C occurs the elimination of the most volatile components: The mass loss in the range of 170°C - 360°C is probably due to the elimination of low molecular mass degradation products, while in the region of 380°C - 530°C the observed mass loss corresponds to the decomposition of higher molecular mass hydrocarbons [39].

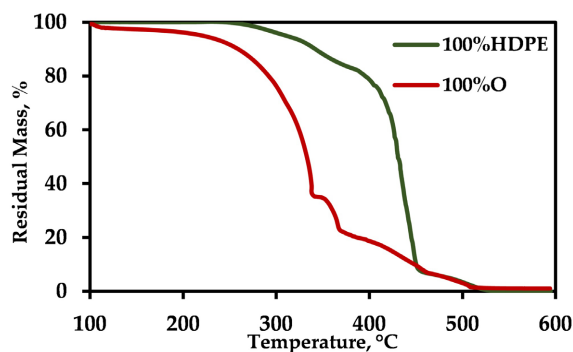


Figure 7. TGA results for HDPE and recycled engine oil.

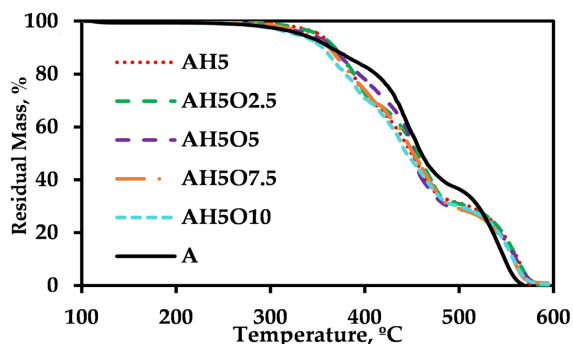


Figure 8. TGA results for modified asphalt samples.

Virgin HDPE degraded in two stages that occur between approximately 255°C - 527°C (**Figure 7**). The first stage of the mass loss corresponds to the thermal cracking of the hydrocarbon chains and the production of oxygenated hydrocarbons, including CO, CO₂ and H₂O [36]. The second stage of the mass loss may be due to the decomposition of the degradation products formed in the first stage [40]. The thermal oxidation of HDPE, in air, takes place through the chain-radical mechanism that occurs in four steps: 1) initiation of the chain's thermal oxidation; 2) propagation of the chain; 3) chain branching and chain termination [41]. Bolbukh *et al.* (2008) [42] attributed the changes between 252°C - 340°C to the first two stages of HDPE degradation, with a prevalence of the chain propagation stage. Changes between 323°C - 435°C correspond to the chain branching step. They also attributed the changes above 460°C to the chain termination step, degradation of the high molecular mass products formed in the previous steps and to the end of the chain termination step.

The virgin and modified asphalt decomposition took place in three steps in range of approximately 240°C - 570°C (**Figure 8**). The first step is around 250°C - 360°C, the second around 360°C - 470°C and the third step around 470°C - 570°C. At temperatures lower than 350°C the mass loss was due to the decomposition of the saturates and aromatics through radical polymerization reactions, whereby components of low molecular mass were released [43]. In the temperature range between 380°C - 500°C resins, asphaltenes and polymer residues are volatilized. At temperatures higher than 500°C asphaltenes are decomposed [36]

[37].

According to **Figure 8**, the onset degradation temperature of all modified asphalt samples is above that of neat asphalt, suggesting a slight gain of thermal stability of the asphalt after modification.

4. Conclusion

This work aimed at evaluating the influence of the addition of engine oil on the consistency and thermal properties of HDPE-modified asphalt. According to the results, it can be concluded that there is a linear reduction in the consistency of asphalt modified by 5% of HDPE as the concentration of engine oil increases in concentration from 2.5% to 10%. There was an improvement in susceptibility to thermal cracks and mixing conditions. On the other hand, a reduction in resistance to permanent deformation was observed. Both modifiers maintained their thermal properties during the modification process. The addition of engine oil in concentrations between 2.5% and 10% on asphalt modified by 5% HDPE exerts influence on its thermal properties causing an increase on its thermal stability. In general, the engine oil acts as a softening agent of the asphalt modified by the polymer. It improves the stability of the asphalt mixes by maintaining the colloidal balance and the viscosity during the processing and application.

Recommendations

For further studies, it is recommended to carry out rheological tests and morphological analysis of the different compositions studied in the work to better understand their viscoelastic behavior and variations in the colloidal structure of the asphalt after being modified, in order to have a broader view of the applicability of this type of asphalt.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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