

Evaluation of mechanical properties of graphite produced asphalt mixtures

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Abstract

This work evaluates the mechanical behavior of asphalt concrete embedded with natural graphite (CA-GRAFP) from 4 Point Bending Apparatus. The experimental models have submitted frequencies of 0.1 Hz, 0.2 Hz, 0.5 Hz, 1 Hz, 2 Hz, 5 Hz, 10 Hz and 20 Hz and temperatures from 0 °C to 40 °C in increments of 5 °C according to EN 12697-26. The objective was to evaluate the use of natural graphite in substitution of a traditional filler, as well as to evaluate the properties related to stiffness resulting from numerous loading combinations. The results showed that the samples of graphite-embedded asphalt concrete (CA-GRAFP) had an increase in their elastic modulus when compared to the reference asphalt concrete (CA-REF) as frequency and temperature increased.

Keywords: Four-point bending; pavement construction; asphalt composite; graphite.

1. Introduction

Historically, after the 1980s, waste became serious problems for society due to the process of urbanization and population growth which was directly reflected in the exponential increase of these materials, notably due to the difficulty of treatment and disposal in appropriate places.

At the same time, there has been a progressive growth in public awareness of the preservation of the environment with the reduction of the use of raw materials and management (reuse and recycling) of products in various industry sectors.

The discussion for the most used ways to dispose of waste is increasingly recurrent in the world context, once there is a global awareness that its mismanagement reflects in the environmental, economic and social categories. In Brazil, there is a large amount of waste that does not have a correct final destination. As a consequence, there is an increase in waste, contamination of natural resources, problems in public health that directly worsening the quality of life of society.

According to data from the solid waste panorama in Brazil published by the Brazilian Association of Public Cleaning and Special Waste Companies (ABRELPE) in 2016, the generation of urban solid waste approached 78.3 million tons in the country. Of this total, 7 million tons of waste was not collected and were improperly dumped [1].

After decades of unreasonable disposal, solid waste began to be used as an alternative material in different sectors of construction. In the current production model, the reuse of these byproducts has been increasing rapidly, and research shows the potential of these materials in the field of infrastructure engineering,

particularly on road transport. In general, the literature cites various types of industrial processes waste which has been used in civil construction to replace the usual materials, notably in asphalt paving. These residues include: polyethylene terephthalate (PET), mineral fillers, tire rubber, shale oil residue, folate quartzite residue, ceramic brick residue and Styrene-Butadiene-Styrene polymer (SBS) [2-9].

Other studies are also highlighted to evaluate the increase of mechanical and rheological performance, the technical and economic viability of asphalt composites.

Examples include case studies from the Geotechnical Group of the Federal University of Amazonas (UFAM), in which solutions have been applied to problems in the Amazon region by several authors [10-24].

More recently, with the advent of nanotechnology, the literature brings research with the application of nanomaterials as modifiers of asphalt binders and in asphalt mixtures, demonstrating positive effects.

In studies developed at the Federal University of Santa Catarina, it was found that the modification of petroleum asphalt cement (CAP) with the addition of small fractions of nanomaterials significantly improved the properties of the compositions as to rheological and mechanical performance. Among the studies carried out, the studies of E^* and fatigue resistance (8, 25-28] in the four-point apparatus stand out. Both have gained increasing acceptance in the mechanistic analysis of asphalt concrete, mainly due to their potential for the foundation of formulations of models constitutive of their structure, because it represents both the part of elastic behavior, resulting from the aggregates, as well as viscous behavior from the asphalt binder.

In this context, this work aims to evaluate the use of natural graphite in substitution of a traditional filler, as well as to evaluate the properties related to stiffness resulting from numerous loading combinations. On the other hand, the feasibility of using graphite as a substitute for Portland cement inserts this material in the sustainability principles, to minimize the exploitation of non-renewable materials, as well as favoring the implementation of maintenance and restoration pavement policies. Therefore, studies focused on the theme are essential to improve the use of graphite in asphalt concrete compositions, seeking to ensure the proper performance of the structural components executed with this material and, consequently, mitigating the environmental impact with the reduction of natural aggregate demand.

2. Materials and methods

2.1 First stage of the experimental program

The first stage of this research consisted of the definition and capture of materials composed by asphalt mixtures of the present work. For the production of asphalt concrete, six materials were used: gravel 1, gravel 0, stone dust, sand, cement (Portland cement and graphite) and asphalt binder 50/70. The two materials participating in the research, except graphite, were donated by Construtora e Pavimentação LTDA (ARDO). Figure 1 presents the materials used in this study.

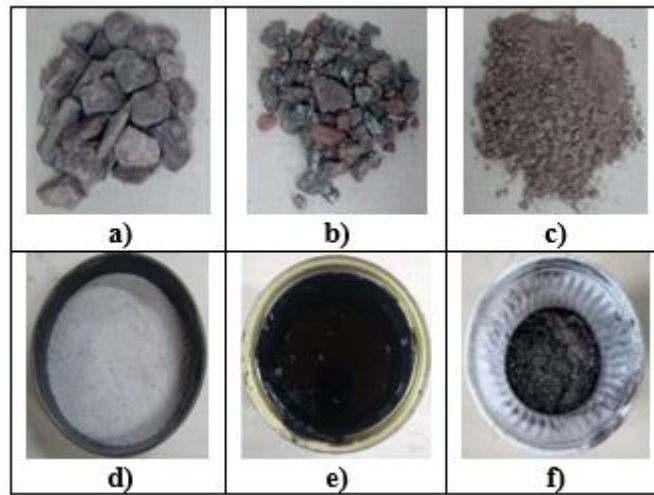


Figure 1. a) gravel 1; b) gravel 0; c) dust; d) sand; e) petroleum asphalt cement; f) graphite.

Table 1 shows the particle size distributions of mineral aggregates used in this research. The granulometry test was performed according to the American standard ASTM C136 / 2006 [29].

Table 1. Particle size distribution of materials.

| Sieve | Opening (mm) | % passing from total sample | | | | | |
|--------|--------------|-----------------------------|----------|-------|-------|-----------------|----------|
| | | Gravel 1 | Gravel 0 | Sand | Dust | Portland Cement | Graphite |
| 3" | 75 | 100 | 100 | 100 | 100 | 100 | 100 |
| 2" | 50 | 100 | 100 | 100 | 100 | 100 | 100 |
| 1 1/2" | 37.5 | 100 | 100 | 100 | 100 | 100 | 100 |
| 1" | 25 | 100 | 100 | 100 | 100 | 100 | 100 |
| 3/4" | 19 | 95.44 | 100 | 100 | 100 | 100 | 100 |
| 1/2" | 12.5 | 30.95 | 95.8 | 100 | 100 | 100 | 100 |
| 3/8" | 9.5 | 12.59 | 79.07 | 100 | 100 | 100 | 100 |
| N° 04 | 4.75 | 0.225 | 17.12 | 100 | 79.4 | 100 | 100 |
| N° 08 | 2.36 | - | 2.075 | 97.47 | 54.38 | 100 | 100 |
| N° 16 | 1.18 | - | 0.55 | 89.77 | 32.28 | 100 | 100 |
| N° 30 | 0.600 | - | 0.15 | 61.6 | 19.95 | 100 | 100 |
| N° 50 | 0.300 | - | 0.04 | 28.07 | 11.37 | 100 | 100 |
| N° 100 | 0.150 | - | 0.025 | 7.6 | 2.78 | 100 | 100 |
| N° 200 | 0.075 | - | - | - | - | 100 | 100 |

In this work was used petroleum asphalt cement, rated for penetration as 50/70, whose specifications are in accordance with the National Petroleum Agency (ANP) and following the technical guidelines of the American Society for Testing and Materials (ASTM) presented in Table 2.

Table 2. Asphalt Binder Properties.

| Features | Unity | Oil Asphalt Cement | |
|-----------------------------------|--------|--------------------|--------|
| | | Result | Limits |
| Penetration -5s, 25 °C | 0,1 mm | 69 | 50-70 |
| Softening Point, min | °C | 49,7 | > 46 |
| Saybolt Furol Viscosity at 135 °C | s | 283 | > 141 |
| Saybolt Furol Viscosity at 150 °C | s | 140,7 | > 50 |
| Saybolt Furol Viscosity at 177 °C | s | 50,8 | 30-150 |
| Brookfield Viscosity at 135 °C | cP | 539 | > 274 |
| Brookfield Viscosity at 150 °C | cP | 279,8 | >112 |
| Brookfield Viscosity at 177 °C | cP | 96,8 | 57-285 |
| Flash point, min | °C | 318 | > 235 |
| Solubility in Trichloroethylene | % | 99,5 | > 99,5 |
| RTFOT Bulk Variation | % | 0,04 | < 0,5 |
| Ductility at 25 °C | cm | > 100 | 60 |
| RTFOT softening point increase | °C | 7,1 | < 8 |
| RTFOT retained penetration | % | 63 | > 55 |

2.2. Second stage of the experimental program

The second stage of the experimental program aimed to perform the physical characterization of the materials, according to the methods of the American standardization body (ASTM). For coarse aggregates (gravel 1 and gravel 0), G_{sa} (Apparent Specific Gravity), G_{sb} (Bulk Specific Gravity) and absorption assays were performed using ASTM C127 / 2015 [30]. Fine aggregates (stone dust and sand) were evaluated only for G_{sa} (Apparent Specific Gravity) specified by US Standard ASTM C128 / 2015 [31]. The fillers (Portland cement and natural graphite) were characterized by the Apparent Specific Gravity (G_{sa}) test following ASTM C188 / 2017 [32].

2.3. Third stage of the experimental program

This step corresponded to the process of making the prismatic specimens of reference asphalt concrete and modified with pulverized graphite to perform the mechanical tests through the four-point bending apparatus. As for mineral dosage, a formulation used in the construction of urban roads was employed. The asphalt mixtures were kept in the greenhouse for two hours at the compaction temperature for the aging process, as recommended by ASTM D 4867/2014 [33]. This period aims to simulate the process of preparation and application of asphalt concrete in the field.

For the fabrication of beams, the same methodology adopted by several authors was adopted [34-36]. For this purpose, a metal mold consisting essentially of an assembly with a locking device and a base was used. Depending on the compaction, a 30-ton hydraulic press was used in which the mold was subjected to constant pressure. Figure 2 illustrates the molding and compacting system of the prismatic beams.

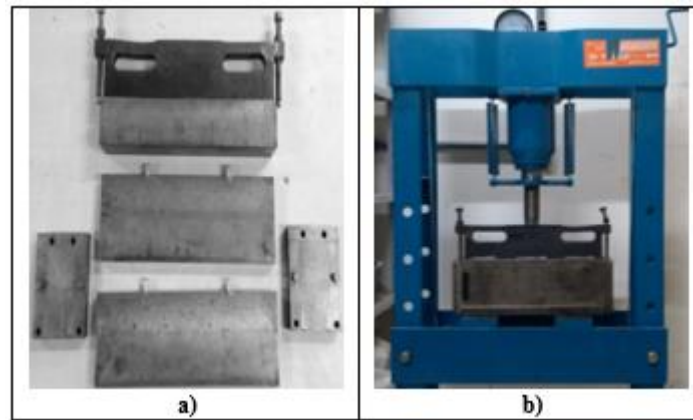


Figure 2. a) metal mold for beam molding and compaction; b) hydraulic press 30 tons.

2.4. Fourth stage of the experimental program

The fourth step was to study the rheological characteristics of asphalt samples, based on the specified four-point flexural testing methods [37]. The prismatic beams were examined based on the four-point bending test to determine the complex modulus and phase angle. A climate camera (Figure 3a) and the 4-point apparatus of the Australian company IPC Global[®] - Model CS 7800 (Figure 3b) were used and belong to the Geotechnical Research Group's Mechanical Behavior Laboratory (GEOTEC / UFAM). The equipment applies pulses of consecutive sinusoidal loads (tensile / compression) in the middle thirds of the beam span, with frequency varying from 0.01 to 30 Hz. The methodology adopted followed the recommendations of the European standard EN 12697-26 [37], with application of uniaxial sinusoidal compression loading, strain amplitude of $50 \mu\text{m} / \text{m}$ under the frequencies of 0.1 Hz, 0.2 Hz, 0.5 Hz, 1 Hz, 2 Hz, 5 Hz, 10 Hz, and 20 Hz and test temperatures from 0°C to 40°C in increments of 5 by 5°C .



Figure 3. a) climate camera; b) 4-point test apparatus for complex module assay

For each test temperature, the minimum acclimatization time established by EN 12697-26 [37] was adopted. For testing at temperatures of 0°C , samples were placed in an environmental chamber for a minimum of 2 hours before the start of the test, minimum acclimatization time for test temperatures from 5°C to 20°C required interpolation, from 2 hours and 1 hour. And for test temperatures above 20°C , the specimens remained for at least 1 hour in the climate chamber. To avoid excessive aging of the samples,

the maximum acclimatization period did not last more than 6 hours. To evaluate the rheological behavior of the mixtures, the isothermal curves, isochronous curves and Black spaces were plotted.

3. Results and discussion

Table 3 presents the physical properties of the aggregates used in this research. It is observed that the real and apparent density values of the large aggregates presented similar values. The coarse aggregates (gravel 1 and gravel 0) also indicated low absorption potential (less than 2%), which results in low consumption of asphalt binder.

Table 3. Routine test results for mineral aggregates.

| Test | Result | Standard |
|--|--------|------------|
| Gsa, g/cm ³ (gravel 1) | 2,58 | ASTM C 127 |
| Gsb, g/cm ³ (gravel 1) | 2,55 | |
| Absorption, % (gravel 1) | 0,51 | |
| Gsa, g/cm ³ (gravel 0) | 2.74 | ASTM C 127 |
| Gsb, g/cm ³ (gravel 0) | 2,62 | |
| Absorption, % (gravel 0) | 1.66 | |
| Gsa, g/cm ³ (sand) | 2.21 | ASTM C 128 |
| Gsa, g/cm ³ (dust) | 2.75 | |
| Gsa, g/cm ³ (Portland cement) | 2.94 | ASTM C 188 |
| Gsa, g/cm ³ (graphite) | 2,64 | |

To systematically show the kinetic susceptibility of the compositions (stiffness), the isothermal curves of Figure 4 that correlate the complex modules (ordered) as a function of the loading frequencies (abscissa) for different temperatures were elaborated. Thus, Figure 4 confronts the isothermal curves of the asphalt mixtures of the present study.

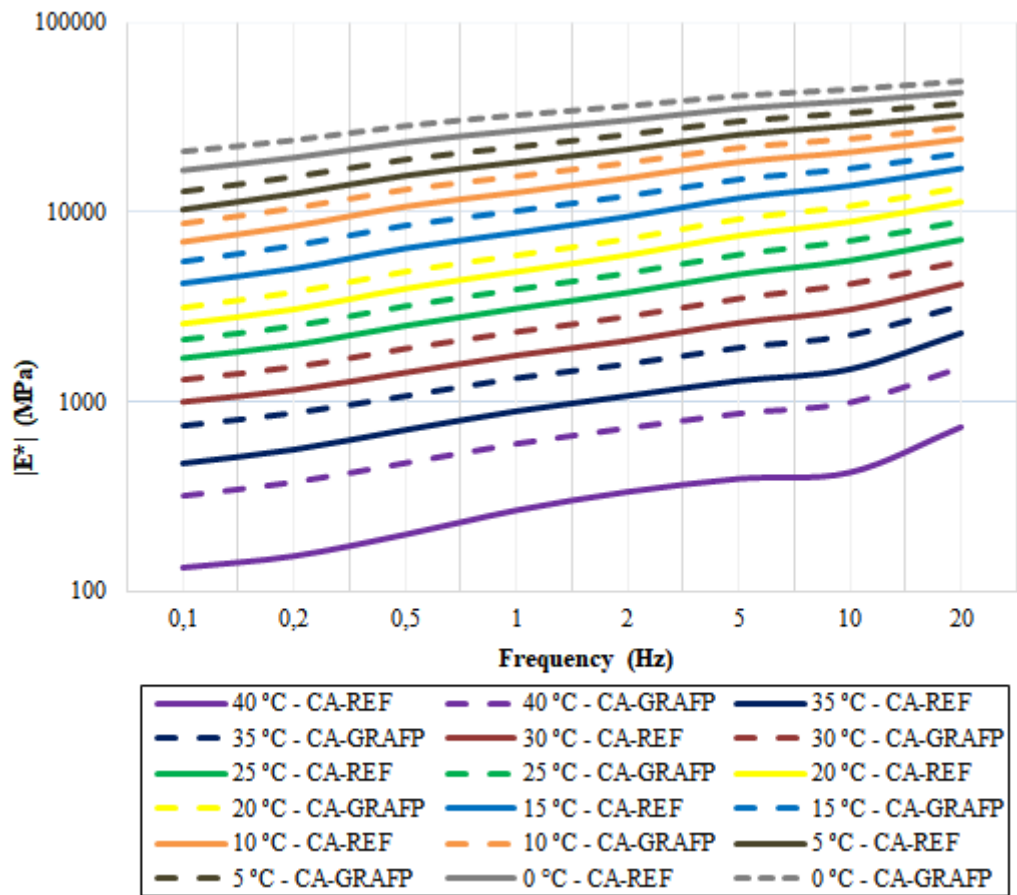


Figure 4. Comparison of isothermal curves for the asphalt mixtures studied.

Figure 4 shows the gains in the composition of asphalt concrete with embedded graphite (CA-GRAFP) over the formulation of reference asphalt concrete (CA-REF). The increase in stiffness resistance must be associated with the combination of different physicochemical properties of graphite, such as cleavage, plasticity, low friction coefficient and high melting temperature (3650 °C), as well as being highly refractory, lubricant, compressible, flexible and have great chemical, thermal and oxidation resistance [38]. Analyzing the behavior of the mixtures about the different frequencies the CA-GRAFP mixture had greater stiffness than the conventional mixture (CA-REF).

Moving forward in the analysis of the results, two isothermal curves were selected for evaluation (Figure 5), corresponding to the temperature of 25 °C and another of 40 °C that represents the daily conditions of the asphalt pavement surface of Manaus (AM). From these curves, it can be seen that the kinetic behavior of asphalt concretes from the inclination of the isotherms demonstrates an increase in stiffness as the loading frequency increases. In particular, this increasing inclination characterizes the vertical displacement of alternative compositions relative to the conventional formulation. It is also found that the lower the temperature, the greater the stiffness gains by incorporating natural graphite. Also, all mixtures showed greater kinetic sensitivity as temperature increased.

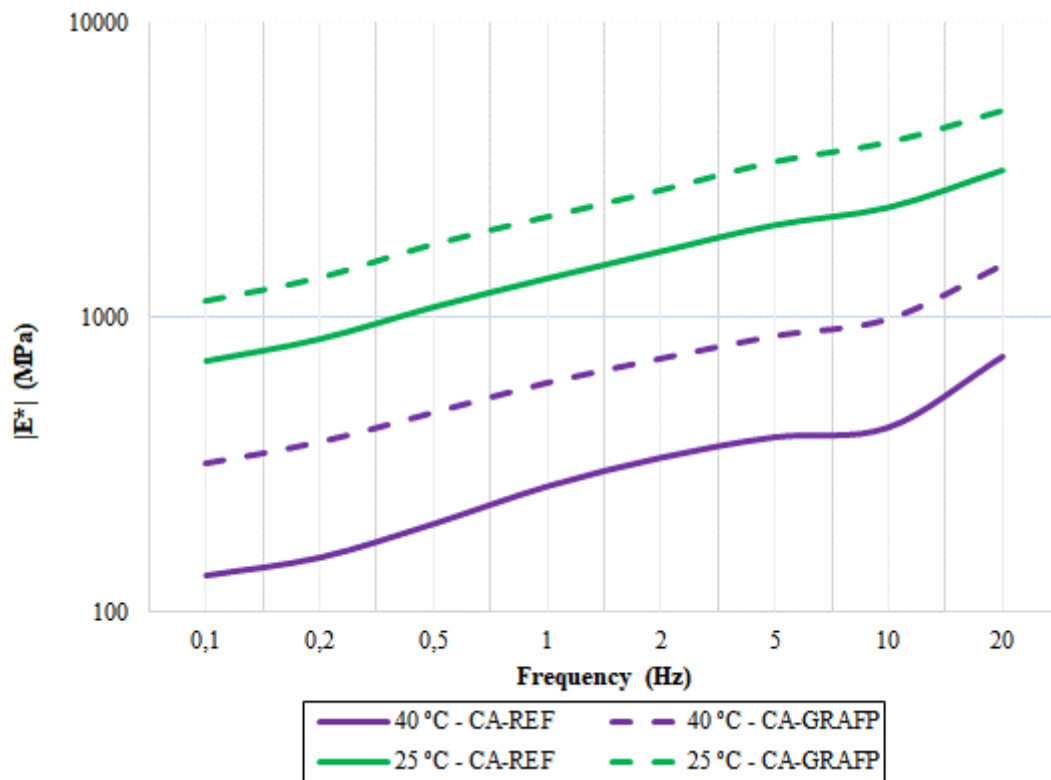


Figure 5. Curvas isotérmicas, temperaturas 25 °C e 40 °C

Regarding the increase in stiffness provided by the replacement of the traditional filler, we also found logical similarity in the tendency to increase stiffness with the insertion of alternative materials [8, 25, 26, 28].

Table 4 presents, in percentage terms, the increase of stiffness, showing the gains of the complex modulus of the CA-GRAFP asphalt formulations compared to the CA-REF asphalt mixture.

Table 4. Increase in the Complex Modulus for the asphalt mixtures produced with graphite to the Conventional Asphalt Mixture (in Percentage Terms).

| | 0 | 5 | 10 | 15 | 20 | 25 | 30 |
|--------|-------------|-------|-------|-------|-------|-------|-------|
| f (Hz) | Temperature | | | | | | |
| 0,1 | 15,05 | 46,59 | 14,93 | 27,51 | 31,64 | 10,93 | 22,30 |
| 0,2 | 17,10 | 36,48 | 15,58 | 39,46 | 34,90 | 13,31 | 32,54 |
| 0,5 | 18,67 | 32,77 | 11,06 | 40,38 | 29,22 | 14,32 | 35,60 |
| 1 | 18,10 | 27,53 | 8,08 | 37,28 | 25,93 | 11,81 | 36,21 |
| 2 | 19,94 | 23,74 | 5,52 | 33,21 | 23,48 | 9,29 | 36,79 |
| 5 | 18,44 | 15,79 | 0,36 | 23,56 | 19,43 | 11,01 | 31,94 |
| 10 | 19,77 | 10,25 | 3,97 | 15,87 | 12,42 | 14,08 | 35,47 |
| 20 | 22,75 | 8,75 | 14,49 | 8,24 | 1,59 | 13,34 | 36,29 |

Depending on the results for the CA-GRAFP composition, and taking as an example the 10 Hz frequency representative of a speed corresponding to 72 km / h [39], the addition of the complex module is on the

order of 35.47%, 14.08 %, 12,42% and 15,87% for the temperatures of 30 °C, 25 °C, 20 °C and 15 °C, respectively. It is emphasized that, in practice, increased stiffness means a higher angular coefficient in the stress-strain curve [25]. This indicates that the stiffness of CAG-GRAFP mixtures in the field under the same stress state would be less sensitive to tensile deformations in the bottom fiber of the asphalt coating layer.

The graphical representation of the results in the Black Diagram for all mixtures is shown in Figure 6. The Black space aims to indicate the elastic behavior of asphalt mixtures and to avoid possible discrepancies in experimental results [40].

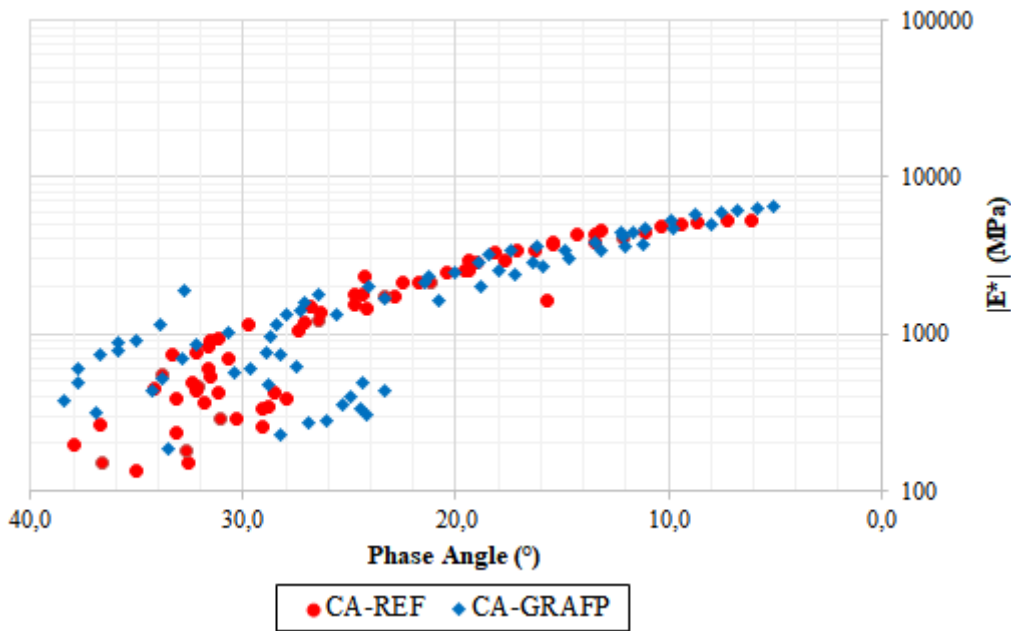


Figure 6. Black space, CA-REF × CA-GRAFP

Figure 6 shows a shortening of Black space for alternate blending (CA-GRAFP) compared to reference blending (CA-REF). This result reflects the reduction of the phase angle as a function of the incorporation of pure graphite. Still using the Black diagram, it was possible to verify that, for temperatures higher than 32°C, the results were non-standard for lower temperatures, possibly due to the phase angles indicating the creep of the asphalt material at the interface of the measuring instruments. Therefore, the positive influence of graphite on asphalt samples is noticeable. This can be noted by the fact that the elasticity of asphalt concrete increases with the replacement of conventional filler (Portland cement) with natural graphite, which is observed by the reduction of phase angle values.

5. Conclusion

In this work, the mechanical behavior of 3 asphalt concrete prismatic specimens submitted to the four-point test under the frequencies of 0.1 Hz, 0.2 Hz, 0.5 Hz, 1 Hz, 2 Hz, 5 Hz, 10 Hz and 20 Hz was studied from test temperatures from 0 °C to 40 °C, in increments of 5 to 5 °C. In this sense, asphalt concrete traces were elaborated with the incorporation of graphite (CA-GRAFP) and compared with the reference asphalt concrete (CA-REF) to increase the knowledge on the use of natural graphite to replace the traditional filler

(Portland cement), as well as to evaluate the properties related to stiffness when subjected to loads resulting from numerous combinations.

Finally, the main conclusions of this study are aligned below:

- a) It was observed that all samples of asphalt concrete with graphite incorporation (CA-GRAFP) had an increase in its elastic modulus when compared with the reference asphalt concrete (CA-REF);
 - a) It was observed that all samples of asphalt concrete with graphite incorporation (CA-GRAFP) had an increase in its elastic modulus when compared with the reference asphalt concrete (CA-REF);
 - b) Regarding the resistance contribution of asphalt concrete subjected to temperature variation, there was a greater elastic gain in the specimens subjected to lower temperature for asphalt concrete with the incorporation of natural graphite, which shows a kinetic sensitivity for higher temperatures;
 - c) Finally, although a larger number of tests can better validate the behavior of the models studied, it was observed that the experimental results proved to be conservative.

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