SINTERED AGGREGATE OF CALCINED CLAY AS AN ALTERNATIVE FOR CONVENTIONAL AGGREGATE IN THE ASPHALT MIX PRODUCTION

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ABSTRACT

This study evaluated the mechanical behavior of asphalt mixture containing Sintered Aggregate of Calcined Clay (SACC) through Tensile Strength, Resilient Modulus, Dynamic Creep, and Fatigue Life tests compared to asphalt concrete with the pebble. The sample moldings used AC 50/70 asphalt binder, Portland cement (filler), and residual sand. Gradation follows the specifications of the Superpave and the National Transport Infrastructure Department of Brazil (Departamento Nacional de Infraestrutura de Transportes - DNIT). Overall, formulations with SACC outperformed those containing pebble as coarse aggregate. The conclusion suggests that the sintered aggregate of Calcined Clay could be an alternative material for asphalt overlay composition.

KEYWORDS: *Pavement, Tensile Strength, Resilient Modulus, Dynamic Creep, Fatigue Life*

I. INTRODUCTION

Researchers systematically study new road materials as environment-friendly alternatives. Replacing traditional materials with unconventional ones aims to find options that meet the engineering parameters and minimize environmental impacts [1-3]. In this context, the sintered aggregate of Calcined Clay (SACC) is a paving material that may replace pebble.

For construction purposes, some clay products, such as bricks and tiles, represent a new material alternative to asphalt mixtures and an option to dispose of these products. The literature mention works demonstrating this application, either as a replacement for coarse and fine natural aggregates or as an alternative filler [4-8]. These studies show that mechanical behavior was satisfactory when this alternative material partially replaced the natural aggregate, emphasizing increased tensile strength and creep modulus [4-8].

In this scenario, the literature presents the Sintered Aggregate of Calcined Clay (SACC), usually with the primary goal of replacing stone materials in asphalt mixtures. It could be a desirable alternative in areas where consolidated materials are scarce on the soil surface or when deposits are at long distances, increasing the cost of this preferred raw material for the obtention of coarse aggregate.

In Brazil, the research on this material started in the 1980s when the Institute of Road Research (Instituto de Pesquisas Rodoviárias - IPR), of the Brazilian National Transport Infrastructure Department

(Departamento Nacional de Infraestrutura de Transportes do Brasil - DNIT), proposed the first studies of the feasibility of a Sintered Aggregate production made with calcined clay. At the time, preliminary results indicated high production costs, and the studies were halted [9].

However, in the latest 20 years, SACC has received greater attention in Brazil. The Geotechnics Research Group – GEOTEC, from the Federal University of Amazonas, has investigated compositions with SACC [10,11,12,14,15,16]. Silva et al. [10] and Silva et al. [11] evaluated the dynamic modulus of asphalt concretes with SACC in coarse and fine fractions. The results showed higher values than pebble formulations, particularly at high temperatures and low load application frequencies. The authors also mention the lower susceptibility of the Sintered Aggregate to permanent deformations [10-11]. Silva et al. [10] particularly highlighted the performance of SACC in induced moisture (Lottman). The authors found that the mixture with pebble was more susceptible under high-temperature conditions than the composition with SACC, with approximately twice the loss of strength.

In the context of the GEOTEC studies, Silva and Frota [12] conducted a theoretical-experimental study of asphalt concretes with Sintered Aggregate of Calcined Clay (SACC). Using four-point bending tests, they investigated the viability of several models related to the dynamic modulus of asphalt composites made from this calcined material. They obtained an excellent fit of the master curve, allowing them to calculate $|E^*|$ values at any frequency and temperature without performing additional experiments. The authors showed that the Zeng et al. [13] equation for phenomenological models could satisfactorily fit the master curve of asphalt compositions containing SACC. With $R^2 = 0.999875$, they obtained an excellent correlation coefficient between the values of the master curve for 40 °C. It is worth noting that, in addition to representing the dynamic modulus in a continuous form, this equation allowed for data extrapolation and, as a result, the determination of the equilibrium value of the dynamic modulus when the frequency tends to infinity.

Cunha et al. [14] compared tensile tests (TS), resilient modulus (RM), and fatigue life (FL) of asphalt mixtures with the coarse aggregate of SACC type and asphalt concrete with pebble. Both compositions exceeded the minimum value required by the Brazilian standard (0.65 MPa) in tensile strength at 25°C. However, at temperatures above 40°C, as occurs in tropical zones, the composition with SACC performed better. Regarding RM and fatigue life, formulations with SACC performed better at temperatures above 30°C and in the temperature range of 25°C to 60°C, respectively, compared to pebble mixtures.

GEOTEC published a study conducted by Spínola et al. [15] in a more recent review based on a viscoelastic analysis and the four-point bending test. The study compared the mechanical behavior of unaged asphalt compositions containing SACC to 5 years aged samples. It was clear that aging increased the composition's stiffness, as measured by an increase in the dynamic modulus $|E^*|$. They also discovered that the compaction process of asphalt beams could significantly impact the magnitude of the dynamic parameter values. The four-point bending test applied the generalized Euler-Bernoulli model for viscoelastic materials with dynamic loading. The fitting confirmed the theoretical model's compatibility for the two studied compositions (unaged and aged).

Barbosa et al. [16] investigated Stone Matrix Asphalt (SMA) with the sintered aggregate (SACC) and fiber residues from typical Amazon plants - the curauá fiber (Ananas erectifolius). The authors determined the mechanical behavior using tensile strength (TS), resilient modulus (MR), and dynamic modulus, $|E^*|$. They discovered a significant percentage increase in these parameters in the studied mixtures, which corresponded to the data presented in the literature.

Other Brazilian institutions, such as the Military Institute of Engineering (Instituto Militar de Engenharia), in collaboration with the Federal University of Rio de Janeiro, also investigated the Sintered Aggregate of calcined clay (SACC). It has shown competitive production costs and the potential to compose the asphalt concretes with similar mechanical performance to asphalt mixtures made with natural aggregates [9].

The re-use of clayey matrix by-products and the synthetic aggregate of calcined clay (SACC) production leads to the development of new road construction materials. These aggregates have a lower environmental impact, avoid improper disposal of ceramic industry products, and reduce dredging of riverbeds.

Thus, this study compared the mechanical behavior of asphalt compositions containing SACC to formulations containing natural pebble aggregate. The second section presents the materials and methods adopted in this research. Then, there are the results, in which the chemical analysis of the synthetic aggregate of calcined clay, the physical characterization of the aggregates and the mechanical behavior of the asphalt mixtures made with this alternative aggregate are shown. In the fourth section is the conclusion, with the final considerations on the general performance of the sintered aggregate of calcined clay compared to the natural aggregate pebble.

II. MATERIALS AND METHODS

2.1. Materials

The asphalt concrete studied in this research is composed of petroleum asphalt cement, the sintered aggregate of calcined clay (SACC) and pebble (both as coarse aggregates), and also sand (as a fine aggregate).

The SACC material originated from typical clayey soil (NSOIL) of the Amazonas Capital, Brazil. For characterization, the Brazilian National Standards Organization (ABNT) standards analyzed the grains size (NBR-7181), liquidity limit (NBR-6459), and plasticity limit (NBR-7180). The calcination potential of *in natura* clay followed Brazil's National Transport Infrastructure Department specifications, DNIT (ME 222, ME 223, ME 225, and EM 230). This study also evaluated chemical composition, mainly the oxides concentrations foreseen by DNIT, and the possible changes in the soil before and after the burning process (Table 3). The SACC manufacturing process followed the steps: a) moisturizing and homogenization of the clayey soil; b) molding and extrusion of aggregates using standardized molds; c) air drying of the molded aggregates over 4 days; and d) calcination of the material at a rate of 3° C/min until it reached 950° C.

The coarse materials (SACC and pebble) were distinguished using DNIT (031-ES) and Superior Performance Asphalt Pavements – Superpave [17] grain size ranges. Three indexes distinguished it: "D" corresponding to C gradation of DNIT-031-ES, "S" referring to Superpave, and "DS" meeting the two specifications (DNIT and Superpave). As a result, the aggregates were named SACC-D, SACC-S, and Pebble-DS.

Grain size (ABNT-NBR 7181), Los Angeles Abrasion (ABNT-NBR NM 51), apparent specific gravity (Gsa), bulk specific gravity (Gsb), and absorption (ABNT-NBR-16917) parameters characterize all coarse aggregates. The fine – sand of residual origin – was evaluated by grain size (ABNT-NBR 7181) and according to ABNT-NBR-16916 for apparent specific gravity (Gsa), bulk specific gravity (Gsb), and absorption. Table 1 displays these findings.

Table 1. Aggregates Characterization

Tests	SACC-D	SACC-S	Pebble	Sand	Method	DNIT EM 230 requirement
Bulk specific gravity, Gsb (g/cm³)	1.806**	2.166**	1.917**	1.512*	NBR-16916* or NBR - 16917**	-
Apparent specific gravity, Gsa (g/cm³)	2.590**	2.584**	2.622**	2.625*		-
Absorption (%)	16.7**	19.2**	0**	0*		-
Los Angeles abrasion (%)	36	36	35	-	NBR-NM 51	≤35
Slaking test (%)	0.01	0.01	-	-	DNIT-ME 225	≤6

2.2. Methods

This research investigated three different asphalt concrete mix combinations. The samples named M1 and M2 used the coarse aggregates SACC-D and SACC-S from clayey soil (NSOIL), respectively. And the M3 mixture used natural aggregate Pebble-DS (Table 2).

2.2.1. Samples

According to the Marshall dosage method, the final samples had the following composition: M1 with 41% SACC-D, 55% sand, 4% Portland cement, and 7.25% AC50/70; M2 with 56% SACC-S, 38% sand, 6% Portland cement, and 9.5% AC50/70; and M3 with 50% pebble, 45% sand, 5% Portland cement, and 5.5% AC50/70. Following Marshall compaction, the samples displayed the parameters shown in Table 2, following the DNIT specifications.

Table 2. Volumetric parameters

Parameters	**M1**	**M2**	**M3**
Content of AC (%)	7.25	9.5	5.5
Air voids, Va (%)	3.8	4.5	3.5
Voids in mineral aggregates, VMA (%)	16.51	21.49	19.8
Voids filled with asphalt, VFA (%)	12.71	16.99	16.30
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Voids filled with asphalt, VFA (%)	12.71	16.99	16.30

2. 2. 2. Mechanical tests

The following tests evaluated the mechanical behavior of asphalt mixtures at a temperature of about 27 °C by the following experiments. The indirect tensile strength test (TS) follows the DNIT - 136 ME standard. The Resilient Modulus (RM) followed the ASTM D7369 recommendations, as well as the dynamic creep (DC) and fatigue life (FL) tests using repeated load under controlled stress, by ABNT NBR 16505 and DNIT – 183 ME requirements, respectively. All tests employed an IPC Global Universal Testing Machine (UTM), with a 14 kN capacity. Concerning dynamic tests, the transmitted load occurred in haversine form, that is, 0.1s of load and 0.9s of rest.

In the TS test, a static load was applied at 0.8 ± 0.1 mm/s until the specimens failed The RM test uses 10%, 20%, and 30% of the ultimate failure load found in TS test. In the DC test, the axial cyclic compression loading lasted 1h, totalling 3600 loading cycles. Respecting FL test, load pulses corresponding to 10%, 20%, and 30% of TS on the samples. The stopping criterion was the presence of several cracks on the sample's surface, a 50% decrease in RM initial value or both.

III. RESULTS AND DISCUSSION

3.1. Chemical analysis of SACC

A chemical analysis was performed on raw material to classify it as input for making calcined aggregates, and verify possible changes in the clayey soil before and after burning, according to the oxide values recommended by DNIT (Table 3). This analysis was carried out by the GEOSOL Laboratory (Belo Horizonte – MG/BR) using the lithium tetraborate fusion technique by X-ray fluorescence.

The samples presented high content of iron and aluminium oxides (Fe_2O_3 and Al_2O_3), responsible for the red-yellowish colour after burning. They also displayed low loss on heating at 950 ºC, which equals 2.4%, indicating a small amount of organic matter. The melting elements (Na_2O, K_2O) show low percentages despite their importance in producing low porosity aggregates [18], which could explain the high absorption rate of the produced aggregate (NBR-16917).

Table 3. Specified and obtained values from the chemical analysis

Oxides	DNIT Requirement (%)	NSOIL (%)	SACC (%)
SiO_2	50-65	59.1	62.9
Al_2O_3	15-20	24.1	25.7
CaO	1-5	0.22	0.3
Fe_2O_3	5-10	4.3	4.4
$Na_2O + K_2O$	1-5	2.73	2.53

Table 3 shows that oxides percentages are within or close to the limits set by the Brazilian agency (DNIT). It is worth noting that the chemical composition of the raw material analyzed did not change after the burning process. Possibly the burning temperature did not cause a change in the clay's original chemical composition.

3.2. Physical characterization of materials

Figure 1 depicts the grain size distribution for natural soil (NSOIL), coarse aggregates (SACC-D, SACC-S, and Pebble-DS), and sand. The NSOIL consistency indicated values equals to LL = 53.5 %, PL=28%. Including the granulometry analysis, these results classified the material as a silty clay of medium to high plasticity according to the Unified Soil Classification System (USCS).

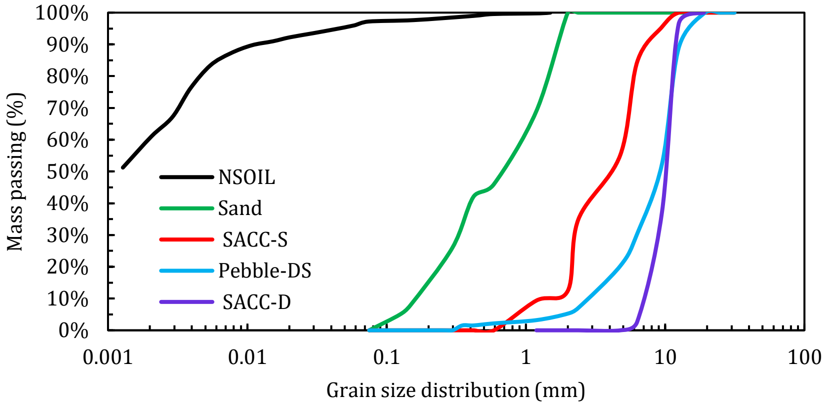

Figure 1. Granulometry of natural soil (NSOIL), residual sand, sintered aggregates of calcined clay (SACC-S and SACC-D) and pebble (Pebble-DS).

The AC 50/70 petroleum asphalt classification was PG 64-22. In compliance with the DNIT – ME 085 and DNIT – EM 367 standards, respectively, the results for the Portland cement filler showed an apparent specific gravity of 3.15 g/cm³ and a 96% passing mass percentage in the 0.075 mm sieve.

3.3. Tensile Strength

As presented in table 4, TS values for M1 and M2 formulations ranged from 0.78 MPa to 0.92 MPa, respectively, and from 0.58 MPa to 0.64 MPa for M3 composition. In terms of average values (Avg.) and standard deviations (SD), it recorded 0.84 MPa (M1) and 0.07 (M1), 0.88 MPa (M2) and 0.05 (M2), and 0.64 MPa and 0.06 for M3. It is essential to note the lower results for pebble formulations due to their smooth surface texture and lack of angularity.

The TS test (Figure 2a-c) validated the behaviour of the aggregate in the mixtures. There was a shear failure in specimens made with SACC (M1 and M2), while in the M3 specimen, the pebble structure prevailed, and the asphalt mastic was more compromised. Segregation in M1 mixture is also notable (Figure 2a). The higher aggregates concentration laterally and on the lower specimen region likely contributed to further dispersion in the results.

Table 4. Tensile strength of asphalt mixtures

Samples								
M1			**M2**			**M3**		
Tensile strength (MPa)	SD	Avg. (MPa)	Tensile strength (MPa)	SD	Avg. (MPa)	Tensile strength (MPa)	SD	Avg. (MPa)
0.83	0.07	0.84	0.82	0.05	0.88	0.58	0.06	0.64
0.78			0.87			0.58		
0.83			0.91			0.69		
0.78			0.92			0.59		
0.84			0.92			0.64		

Figure 2. Specimens after failure: a) M1 mixture (SACC-D), b) M2 (SACC-S) and c) M3 (Pebble-DS).

It is important to emphasize that the M3 mixture, made with coarse pebble aggregate, has a smoother texture than SACC, which may have contributed to more significant deformation than other formulations. Furthermore, more angular aggregates contribute to interlock particles, promoting an increase in strength composition (FHWA, 2000). Such a characteristic is uncommon in pebble. Concerning M1 and M2, the SACC used to produce M2 was manually crushed to fit into the Superpave gradation. This process may have increased the material angularity, contributing to its improved performance when incorporated into the asphalt mixture.

3.4. Resilience Modulus

The average values of RM, shown in Figure 3, display a decline as the load increases. In all loading conditions, mixtures M1 (SACC-D) and M3 (Pebble-DS) performed worse than composition M2 (SACC-S). Comparing those two mixtures, M1 (SACC-D), in the C grain size range (DNIT) presented lower values in all tests, as compared to M2 (SACC-S), made under Superpave standards, especially for the stress level referring to 30% of TS (Figure 4).

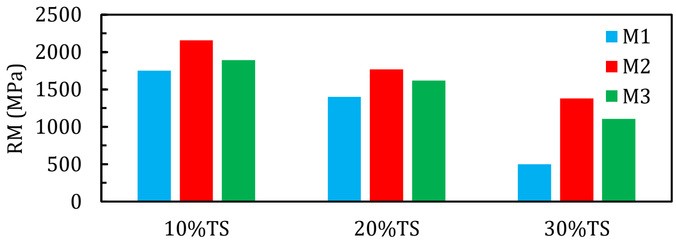

Figure 3. Resilient modulus of asphalt mixtures M1 (SACC-D), M2 (SACC-S) and M3 (Pebble-DS).

The RM/TS ratio combines the resilient modulus (RM) and tensile strength test (TS) results. Studies show that the lower the RM/TS ratio, the longer the fatigue life of the asphalt mixture [19-21]. Figure 4 depicts the outcomes of this relationship for M1, M2, and M3 compositions. At all stress levels, it is possible to observe that the mixtures made with the sintered aggregate of calcined clay (M1 and M2) have lower values than the formulations with pebble (M3).

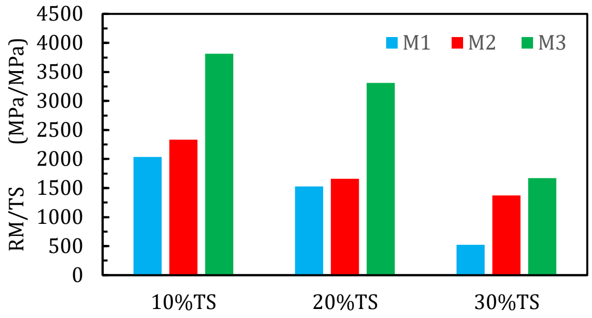

Figure 4. RM/TS ratio for M1 (SACC-D), M2 (SACC-S) and M3 (Pebble-DS) mixtures.

3.5. Dynamic Creep

Figure 5 depicts the results of the non-recoverable axial strain. Strains increase with the load addition in all evaluated mixtures. It is worth noting that the composition containing pebble (M3) had the highest values. It is worth highlighting that the formulation behavior with SACC-S (M2) differs significantly for the first 100 cycles, with more significant strains. However, after about 400 cycles, the values decrease, reaching stability at 2000 load cycles. M1 and M3 mixtures had the highest strain values, at about 0.0025 mm/mm and 0.0040 mm/mm, respectively, besides not showing a tendency toward stability.

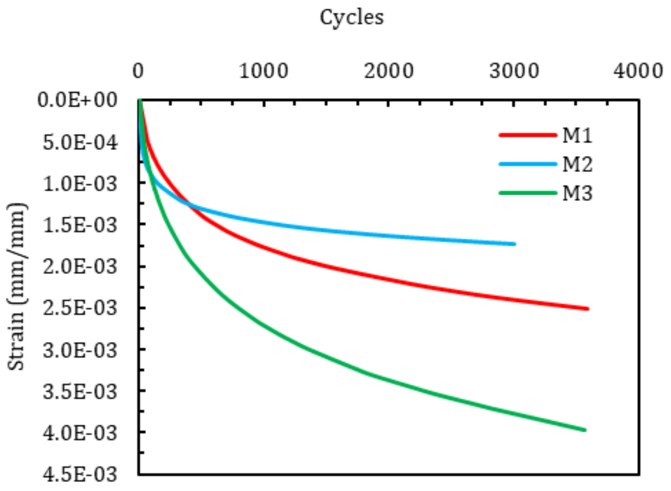

Figure 5. Dynamic creep test results for mixtures M1 (SACC-D), M2 (SACC-S) and M3 (Pebble-DS).

Table 5 shows the maximum axial strain (MSAS) and the corresponding average (Avg.) for all evaluated mixtures, as well as the Creep Modulus (CM) results. The M3 mixture exhibited the most significant average strain (0.0039 mm/mm) and the smallest creep modulus (50.38 MPa) and, compared to the M2 mixture, had 55% lower CM and 54% higher strain. M1 produced the second-best results, with 27% lower average strain and 57.5% higher CM than M3.

Table 5. Dynamic creep test results

Samples								
M1			**M2**			**M3**		
MSAS (mm/mm)	**Avg. (mm/mm)**	**CM (MPa)**	**MSAS (mm/mm)**	**Avg. (mm/mm)**	**CM (MPa)**	**MSAS (mm/mm)**	**Avg. (mm/mm)**	**CM (MPa)**
0.00237	0.00284	79.34	0.0016	0.00178	112.3	0.0043	0.0039	50.38
0.00414			0.0019			0.0039		
0.00252			0.0022			0.0038		
0.00262			0.0013			0.0042		
0.00257			0.002			0.0036		

3.6. Fatigue

Figure 6 shows the average number of cycles (NC) as a function of stress levels for the compositions studied. This graph demonstrates that the M2 mixture produced the best results across all stress levels. The M3 mixture (Pebble) had the lowest values even when subjected to a lower load (TS around 0.64 MPa), as compared to formulations M1 and M2, which had similar TS results, about 0.86 MPa.

At the first stress level (10% of TS), the NC of mixture M2 was 20% and 35% higher than the values of compositions M1 and M3, respectively. At the second stress level (20% of TS), the NC obtained by the M2 formulation was approximately 35% higher than the values obtained by the M1 and M3 mixtures. Also, when the tests reached 30% of the TS, all compositions displayed cracks for an NC close to 1500.

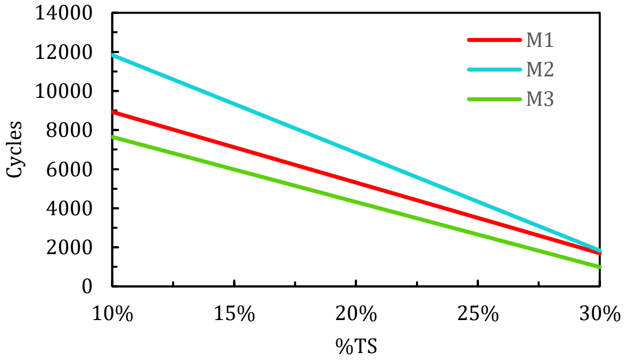

Figure 6. Comparison of M1 (SACC-D), M2 (SACC-S) and M3 (Pebble-DS) mixtures for tension levels of 10%, 20% and 30% referring to TS.

Figure 6 confirm the Figure 4 results: a lower value of the RM/TS ratio for the formulations with SACC than the mixture with pebble. As previously stated, a lower value for this ratio indicates a longer fatigue life. This finding is consistent with Figure 6 because the compositions M1 and M2, as predicted by the RM/TS ratio, had the most extended fatigue life than the M3 mixture. It demonstrates that, by the RM/TS ratio, such analysis can provide a preliminary indication of fatigue life.

IV. CONCLUSIONS

The soil chosen to produce the Sintered Aggregate of calcined clay (SACC) demonstrated satisfactory technical characteristics for the composition of asphalt mixtures following the requirements of the National Transport Infrastructure Department. Even though SACC exhibited a result above the 35% wear limit for Los Angeles Abrasion (DNIT-EM 230/94), the material produced satisfactory results when added to asphalt formulations.

The M2 mixture (SACC-S) had the highest binder content among the formulations studied. It possibly happens because the lamellar aggregate particles (SACC) formed during the crushing process, resulting in a larger specific surface and the material's noticeable vitrification caused by the high burning temperature. This aggregate was also more prone to fracturing.

The M3 (Pebble-DS) mixture performed the worst in the tensile strength test (TS) as compared to the M1 (SACC-D) and M2 (SACC-S) formulations, most likely due to its smooth texture. The SACC mixtures, even in different grain size ranges (C of DNIT and Superpave), showed similar results.

Specifically regarding MR determination, it was observed that, among the mixtures under study, M2 (SACC-S) was the most rigid mixture, with the highest value for that parameter. Therefore, it is more prone to absorbing higher stresses and less deformability.

Regarding permanent deformations, the presence of pebble resulted in the highest values (M3). The SACC formulations (M1 and M2) did not show significant variations. The M2 composition (Superpave) demonstrated slightly less deformability and a greater tendency to stabilize deformations up to 3600 cycles. This asphalt mixture had the highest Creep Modulus (CM), which explained the tinier permanent deformations for the same stress level.

The mixture dosed in the Superpave gradation design (M2) has a longer fatigue life than Range C - DNIT (M1), most likely due to the better aggregate distribution observed during the compaction process.

The behavior of asphalt mixtures made with Sintered Aggregates (M1 and M2) suggested that it could be an alternative material for asphalt coating composition. Notably, the formulation containing SACC in the crushed condition (M2) outperformed the other compositions' mechanical behavior.

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