TRANSPORTES
www.revistatransportes.org.br
anpet
associação nacional de pesquisa e ensino em transportes
www.anpet.org.br

Evaluation of LD slag expansion effects on the mechanical behavior of asphalt mixtures subject to aging and moisture damage

Avaliação dos efeitos da expansão da escória LD no comportamento mecânico de misturas asfálticas sujeitas ao envelhecimento e dano por umidade

Sidineidy Izoton[1], Jamilla Emi Sudo Lutif Teixeira[2], Patrício José Moreira Pires[3], Viviane dos Santos Dias[4]

[1]Federal University of Espírito Santo, Espírito Santo - Brazil, sidineidy.izoton@hotmail.com
[2]Federal University of Espírito Santo, Espírito Santo - Brazil, jamilla.teixeira@ufes.br
[3]Federal University of Espírito Santo, Espírito Santo - Brazil, patricio.pires@ufes.br
[4]Federal University of Espírito Santo, Espírito Santo - Brazil, vivianeediaas@gmail.com

Recebido:
8 de dezembro de 2020
Aceito para publicação:
27 de janeiro de 2021
Publicado:
30 de agosto de 2021
Editor de área:
Kamilla Vasconcelos

Keywords:
Steel slag.
Expansion.
Aging.
Moisture damage.

Palavras-chave:
Escória de aciaria.
Expansão.
Envelhecimento.
Dano por umidade.

DOI:10.14295/transportes.v29i2.2442

ABSTRACT
Steel slag aggregates, such as Linz-Donawitz slag (LD), undergo volumetric expansions when subjected to heat and humidity. However, the asphalt binder might stabilize the LD expansion in hot mixture asphalt (HMA). This work aims to evaluate LD expansion effects on the mechanical behavior of HMA subject to aging and moisture damage. Four dense HMA were evaluated, one reference and three mixtures with 25% LD with different initial expansion. The mixtures were subjected to short- and long- term aging. Subsequently, compacted specimens were subjected to moisture induced damage and indirect tensile strength tests. Also, the expansion of aggregates extracted from HMA were evaluated. Mixtures with LD presented satisfactory mechanical results and even superior than mixtures produced without LD. It was also observed no significant difference in the expansion values between the aggregates extracted from the mixtures after conditioning indicating that LD expansion effects might have been stabilized.

RESUMO
As escórias de aciaria, como Linz-Donawitz (LD), sofrem expansões volumétricas quando submetidas ao calor e à umidade. Contudo, o ligante asfáltico pode estabilizar a expansão da LD em misturas asfálticas à quente (MAQ). Este trabalho objetiva avaliar os efeitos da expansão da LD no comportamento mecânico de MAQ sujeitas ao envelhecimento e dano por umidade. Foram avaliadas quatro MAQ, uma de referência e três misturas com 25% LD com diferentes expansões iniciais. As misturas foram submetidas ao envelhecimento de curto e longo prazo. Posteriormente, foram submetidas a ensaios de dano por umidade induzida e resistência à tração indireta. Também foi avaliada a expansão dos agregados extraídos das MAQs. As misturas com LD apresentaram resultados mecânicos satisfatórios e até superiores à mistura de referência. Não foi observada diferença significativa nos valores de expansão de agregados extraídos das misturas após condicionamento, indicando que os efeitos da expansão da LD podem ter sido estabilizados.

1. INTRODUCTION

In the search for new materials for paving application, different waste and residues generated by various industrial sectors have gain attention. Some residues are called co-products, due to the added value that they still present for other segments, such as in civil construction. One of the largest generation co-products in the country is the slag from the steelmaking process. Data from the sustainability report presented by the Centro de Coprodutos Aço Brasil show that the total generation of by-products and direct residues from the steel industry in 2015 was 19.8 million tons (IABR, 2019). Much research is carried out using Linz Donawitz steel slag (referred to in this study as LD slag), demonstrating its technical feasibility for use as aggregate in asphalt mixtures (Teixeira et al., 2019; Kim et al., 2018; Groenniger et al., 2017). However, most of the studies have been carried out proving its good performance as aggregate for asphalt mixtures considering the short term aging, and its effect and performance in the long term is not yet clear.

LD slag has an expansive nature, with volume variations of up to 10%, due to the hydration of chemical compounds such as free calcium and magnesium oxides present in the composition of this material (Beshears and Tutumluer, 2013). This volumetric variation, when it occurs on asphalt pavements, can result in internal stresses causing premature cracks and eruptions located in the surface layer of the pavement. In order to accelerate the expansive characteristics of LD slag, steel manufacturers perform a curing process. The Brazilian standard limits the maximum expansion value of steel slag to 3% for paving applications, according to the DNER - ME 262/94 specification. This value is obtained after performing the standard procedure PTM 130/78 (Pennsylvania Testing Method), developed in the American state of Pennsylvania. However, it is believed in this study that the expansive potential of LD slag can be stabilized by the asphalt binder film that covers the aggregate, minimizing the effects of the hydration process of the expansive oxides (Teixeira et al., 2019; Shen et al., 2009).

Although there are several studies on the evaluation of steel slag as aggregates, where it is possible to verify the satisfactory performance of asphalt mixtures containing steel slag, the effects of the expansion of this material in the long term in asphalt mixtures have not yet been clearly evaluated. It is known that part of the volumetric expansions occur in the long term. According to Tavares et al. (2012), the use of uncured slags within the limits established in standards can result in internal stresses and the consequent development of cracks in the pavement. In the short term, Teixeira et al. (2019) found that dense asphalt mixtures produced with partial replacement (25% by mass) of natural aggregate by LD slag can be viable for the production of asphalt mixtures, and that in most cases demonstrate a performance equal to or greater than those found for mixtures prepared entirely with natural aggregate. It was also found that the effects of the expansion of the material do not affect the volumetric and mechanical characteristics studied, even for LD slags with high expansion potential (greater than 3%), indicating a possible stabilization of the expansion mechanisms when it is covered by asphalt binder. However, the authors did not study the effects of expansion on the performance of asphalt mixtures considering the long-term aging mechanisms.

Aged asphalt mixtures have greater stiffness compared to non-aged ones, and this increase in stiffness associated with possible volumetric expansions of steel slag when exposed to conditions of humidity and heat can lead to the formation of cracks, thus causing less resistance and durability of mixtures. Thus, this study aims to verify the effects of expansion of LD slag on the mechanical behavior of asphalt mixtures aged in the long term and subjected to moisture induced damage (MID) in the laboratory.

2. MATERIALS AND METHODS

2.1. Aggregates (Natural and Slag) and Asphalt Binder

The materials used in this research were natural aggregates (AN), consisting of crushed stone no. 1, with Nominal Maximum Size (NMS) of 19.0 mm; crushed stone no. 0, with NMS of 12.5 mm; granulate, with 6.3 mm NMS; and stone dust. The steel aggregate samples (LD slag) were obtained from a steel plant located in the municipality of Serra - ES. Three different samples of LD slag at different expansion levels were intentionally collected in order to assess whether there is a different behavior for different expansion levels of this steel aggregate when used in the production of asphalt mixtures or if the asphalt binder can in fact stabilize the processes expansion of this material. The asphalt binder used was CAP 50/70 from the Petrobras/REDUC refinery. The binder was supplied by a local asphalt plant and its characteristics are shown in Table 1.

Table 1 – Characteristics of the CAP 50/70 used in this study

Characteristics	Method	Specification	Result	Unit
Penetration	D5	50 a 70	55	0,1 mm
Softening point	D36	46min.	50	°C
Brookfield Viscosity 135 °C -SP21 20RPM	D4402	274min.	302	cP
Brookfield Viscosity 150 °C -SP21	D4402	112min.	154	cP
Brookfield Viscosity at 177 °C SP21	D4402	57 a 285	58	cP
RTFOT retained penetration	D5	55min.	56	%
RTFOT softening point increase	D36	8 max.	5,4	°C
RTFOT - Ductility at 25 °C	D113	20min.	>150	cm
RTFOT Variation in mass%	D2872	-0,50 a 0,50	-0,31	%
Ductility at 25 °C	D113	60min.	>150	cm
Solubility in Trichlorethylene	D2042	99,50min.	100	% mass
Flash point	D92	235min.	314	°C
Thermal susceptibility index	X018	-1,50 a 0,70	-1,00	N/A (3)
Relative density at 20/4 °C	D70	Take note (1)	1,01	N/A
Heating at 177 ºC	X215	NESP (2)	NESP	N/A

Note: (1) Tests are not part of the specification.
Information for billing purposes. (2) NESP = No foam. (3) Not applicable.

2.2. Experimental Methodology

In order to achieve the objectives, this study was conducted in three stages, the first being the characterization of LD slags with different levels of expansion as well as the AN to verify the differences in the properties of the aggregates. In the second stage, the compositions of the asphalt mixtures and the production of compacted specimens (CSs) were defined, which were subjected to different aging procedures (natural and in oven, in the short- and long- term conditions) and MID according to the standard DNIT 180/2018. In the third stage, indirect tensile strength tests (IDT) of asphalt concretes and expansion tests in the composition of aggregates extracted from each mixture studied after the aging and MID stages were carried out.

2.2.1. Step I: Characterization of the aggregates

For the characterization of the materials, granulometric analysis (ABNT NBR 7181/1984), specific mass and absorption (ABNT NBR 6458/1984), unit mass (DNER-ME 152/1995), abrasion Los Angeles (ABNT NBR NM 51/2001), sand equivalent (DNER-ME 054/1997), durability to sodium sulfate (DNER-ME 089/94), adhesion to the asphalt binder (DNER-ME 078/1994), and expansion potential tests (DNIT- ME 113/2009) testing protocols were used.

In addition, X-Ray Fluorescence (FRX) analysis was performed for quantitative analysis of oxides present in LD samples based on ABNT NBR 11303/1990. For this analysis, a Malvern Panalytical diffractometer (Model X'Pert PRO MPD) using copper radiation was used. Representative samples of kiln-dried steel slag were ground until a powder passed through the No. 200 sieve (opening 0.075 mm). The powder samples were scanned from 4 to 70 ° (2θ) in increments of 0.04 ° in 1 second of counting time.

2.2.2. Stage II: Preparation of CSs and conditioning methods

For the production of asphalt mixtures, it was adopted the mix design results found by Teixeira et al. (2019), since the same aggregates and proportions of materials were used. The authors adopted the limits of DNIT Range C for the aggregate combined gradation. The binder content was determined according to the Marshall mix design methodology, with a value of 5.14%. 25% LD slag and 75% natural aggregates were used. After verifying the fulfillment of the criteria established for the volumetric parameters, i.e., air voids (AV) of 4 ± 1% and voids filled by asphalt (VFA) of 75-82%, CSs were produced with dimensions of 63.5 ± 1.3 mm following the DNER 043/95 - ME standard.

Four aging procedures were used, i.e., i) short-term aging of loose mix in oven, ii) long-term aging of loose mix in oven, iii) long-term aging of CS in oven and iii) natural aging of CS. Figure 1 shows the loose mix and the long-term aged CSs.

(a) (b) (c)

Figure 1. (a) Aging of loose mixture in oven, (b) Aging of CSs in oven and (c) Aging outdoors (natural aging)

All mixtures were subjected to short-term aging, being conditioned for 2h in an oven at 145 °C before compaction. For the preparation of CSs that suffered aging from the loose mixture in the long-term, the aggregates and the asphalt binder were first mixed. This mixture was spread on trays so that a thin layer was formed and subsequently placed in an oven for a period of 9 days at 95 ° C (Figure 1a). The aging period of 9 days at 95 ° C was chosen based on the report by the National Academy of Science (2018), which states that it would take up to 9 days at 95 °C to represent an 8-year aging on a 20-film mm in the coating layer in a place with similar climate and humidity to the location of this study. After this period, the mixtures were aged for another 2 hours at a temperature of 145 °C for compaction.

For the aging of CSs, the mixtures were aged for another 2 hours at a temperature of 145 °C and compacted in sequence. After compacting, they were placed in an oven for a period of 9 days at 95 ° C (Figure 1b), only after this period they were subjected to IDT test.

To assess outdoor aging, mixtures were prepared and aged in the short-term and then compacted. Subsequently, the CSs were placed on the top of a container attached to the research laboratory as shown in Figure 1c, so that they were exposed to the weather, i.e., variation in heat and humidity in the local region, for three different periods (3 months, 6 months and 12 months). Figure 2 shows the flowchart of tests performed in the research.

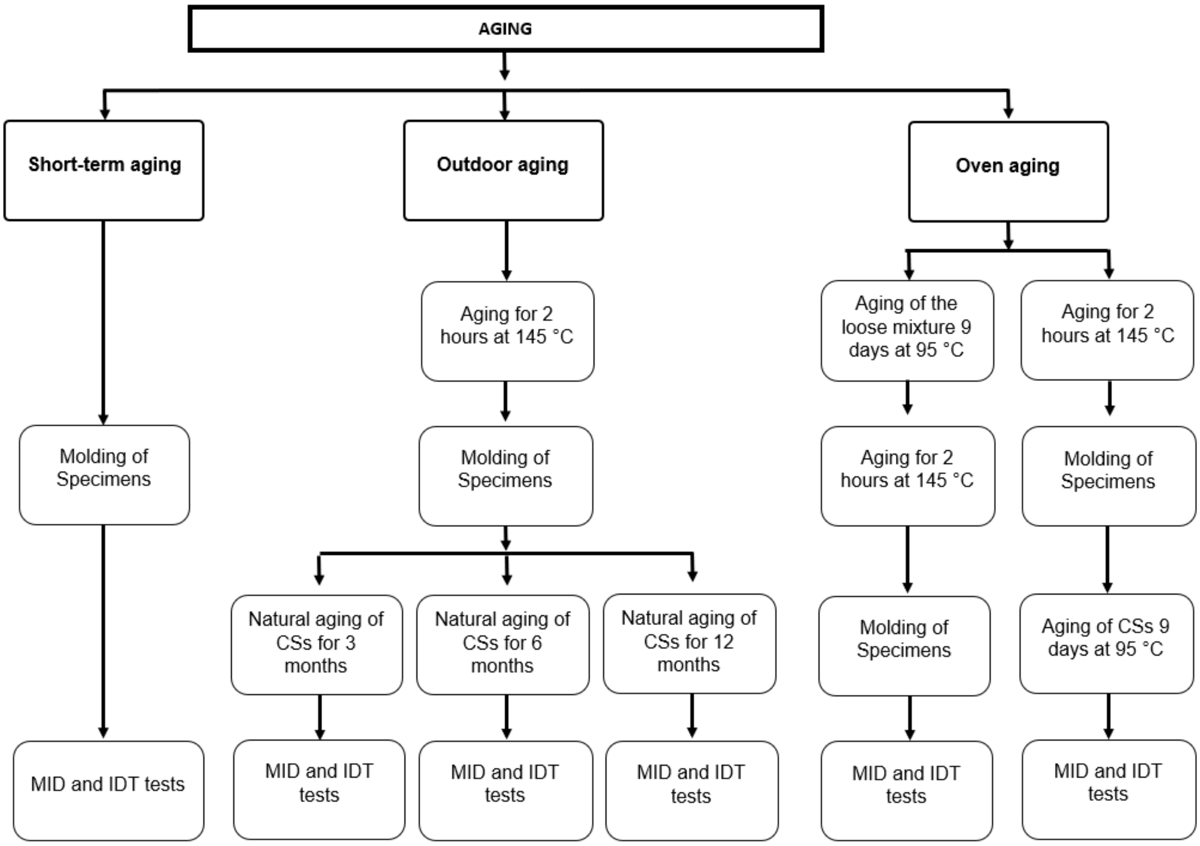


Figure 2. Experimental tests

Mechanical tests

Firstly, IDT tests were carried out according to DNIT 136/2017-ME in CSs with AV of 4.0 ± 1.0% and tested at a temperature of 25 ± 1ºC. Results from three CSs were obtained per mixture, and averaged values and the standard deviation bar are shown in the resulting graphs. For the MID test, the DNIT 180/2018 standard procedure was used. 6 CSs were produced with AV of 7.0 ± 1.0% and divided into 2 sets of 3 CSs for IDT tests. The first set is the control group (IDT without conditioning) and the second set is the group subjected to saturation and freezing and thawing cycles (IDT_C with conditioning), as described in the DNIT 180/2018 standard procedure.

In order to verify whether there was expansion or stabilization of the slags after the mixtures were subjected to aging and MID procedures, PTM tests were performed on samples of aggregates extracted from asphalt mixtures after these conditions. These mixtures (Figure 3a) were subjected to extraction of the binder in the rotarex equipment (Figure 3b, c and d). The samples of extracted aggregates (Figure 3e) were used to perform the PTM tests. In addition, a sample of aggregates without being pre-mixed with binder was subjected to solvent washing in the rotarex so that the effect of the solvent used (kerosene) could be considered in the analysis of the results. Thus, PTM tests were performed on the following mixtures:

- Mixing aggregates without binder and without extraction (without the effect of the solvent).
- Mixture of aggregates without binder but with extraction (with solvent effect).
- Mixture of aggregates extracted from asphalt mixtures aged outdoors in the long term and MID.
- Mixture of aggregates extracted from asphalt mixtures aged in the laboratory in the long term and MID.
- Mixture of aggregates extracted from asphalt mixtures aged in the laboratory in the short term and MID.

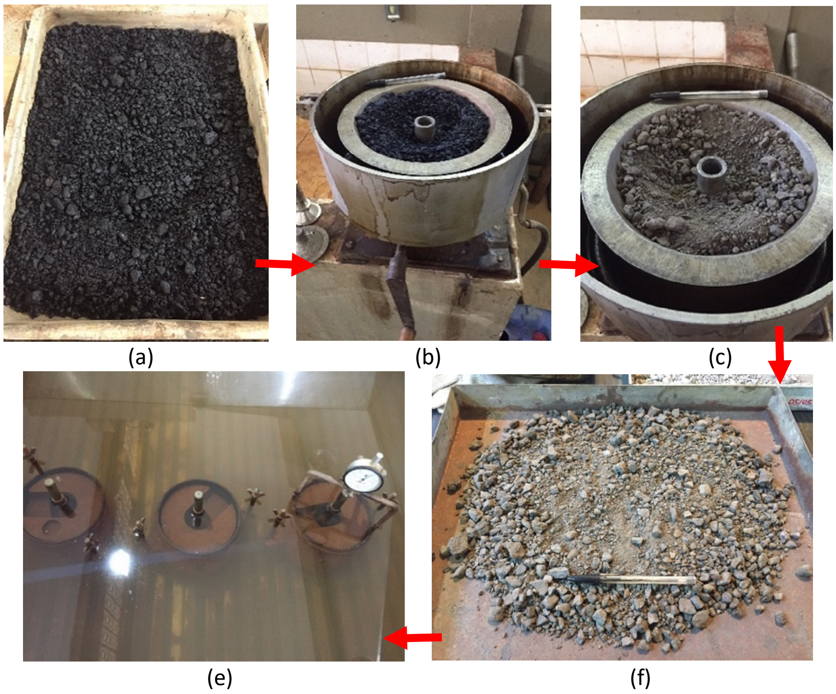


Figure 3. Binder extraction scheme for PTM tests: a) asphalt mixtures after undergoing aging and MID, b) Mix inside rotarex equipment, c) Appearance of the mixture after initial binder extraction, d) Aspect of the mixture after complete binder extraction, e) sample inside mold for PTM test

3. RESULTS AND DISCUSSION

3.1. Characterization of Aggregates

Table 2 shows the characterization results.

Table 2 – Result of the characterization of LD slag at different levels of expansion

Test	Limits (DNIT-ES 031/2006)	LD < 3%	LD ≈ 3%	LD > 3%	AN
Specific mass (g/cm³)	-	3.12	3.12	3.26	2.84
Absorption (%)	-	3.40	2.44	1.95	0.49
Los Angeles Abrasion (%)	Max. 50%	18	20	19	42
Durability (%)	Max. 12%	0.70	1.62	2.36	0.60
Adhesiveness (%)	-	S*	S*	S*	S*
Unit mass (kg/dm³)	-	2.02	2.01	2.14	1.57
Sand Equivalent (%)	> 55 %	85	79	70	74
Expansion (%)	Max. 3%	1.63	2.31	5.92	-

It is observed that there was a change in the characteristics of the slag due to the variation in expansion, mainly in the absorption and durability results, which can cause changes in both the volumetric and mechanical parameters of the asphalt mixtures. Note that absorption tends to increase when the level of expansion is reduced, which can lead to increases in the binder content in the mix design. Observing the Los Angeles abrasion resistance values of the studied aggregates, it is possible to observe that the abrasion loss of LD slags is less than that obtained for AN, confirming the greater mechanical resistance of this material compared to AN.

This result may be related to the chemical composition of the steel aggregates, due to the presence of metallic elements from the steel process. Moura (2020) presented the results of nanoindentation, where it was verified that LD slags have an elasticity module varying from 80 to 170 GPa and hardness from 6 to 14 GPa, while the natural aggregates presented an elasticity module, varying from 70 to 95 GPa and hardness of 8 to 12 GPa, which corroborates the high mechanical resistance of the slag compared to the AN found in this study. The results of the chemical composition regarding the presence of oxides determined in the FRX are shown in Table 3.

Table 3 – Percent mineralogical composition of the oxides present in the LD samples

Oxides	LD<3% (1.63%)	LD≈3% (2.31%)	LD>3% (5.92%)
Al_2O_3	1.84	3.49	7.14
CaO	41.31	36.99	38.16
Cr_2O_3	0.13	0.15	0.19
Fe_2O_3	21.33	17.54	17.10
K_2O	<0.01	<0.01	0.11
MgO	9.17	11.19	9.99
MnO	2.03	1.93	2.25
Na_2O	<0.10	<0.10	<0.10
P_2O_5	1.06	0.78	0.76
SiO_2	8.31	8.05	8.00
TiO_2	0.27	0.28	0.33
ZrO_2	-	-	-
SO_3	0.18	0.40	0.24
ZnO	<0.01	<0.01	<0.01

The chemical composition of steel slag is formed by a heterogeneous structure, composed of a variety of elements. In the percentage composition, the oxides are the oxides of calcium, magnesium, iron, silica and aluminum. Regarding the changes in chemical compounds as a function of the slag curing time, it is possible to verify that there was a decrease in Al_2O_3 with a decrease in the slag expansion (longer curing time), and a decrease and then stabilization of the result MnO. This is consistent with the decrease in expansion since MnO is one of the responsible for the expansion of LD. However, CaO, which is also a major contributor to the expansion of LD, remained without much change for the three samples studied. However, it is noteworthy that there may have been a change in free CaO levels, which was not verified in this study. The decrease in Al_2O_3 with the decrease in expansion and the little variation in CaO, are consistent with what was presented by Teixeira et al. (2019). Considerable values of Al_2O_3, Fe_2O_3, MnO and MgO are highlighted. According to Cala et al. (2019a), the presence of these oxides in the mineralogical composition of the aggregates contributes to a better adhesion of the binder to the aggregate, and consequently, a greater resistance to moisture damage of asphalt mixtures containing these materials.

3.2. Indirect Tensile Strength (IDT)

Figure 4 shows the IDT results of samples compacted with AV of 4,0 ±1,0% and submitted to different aging processes.

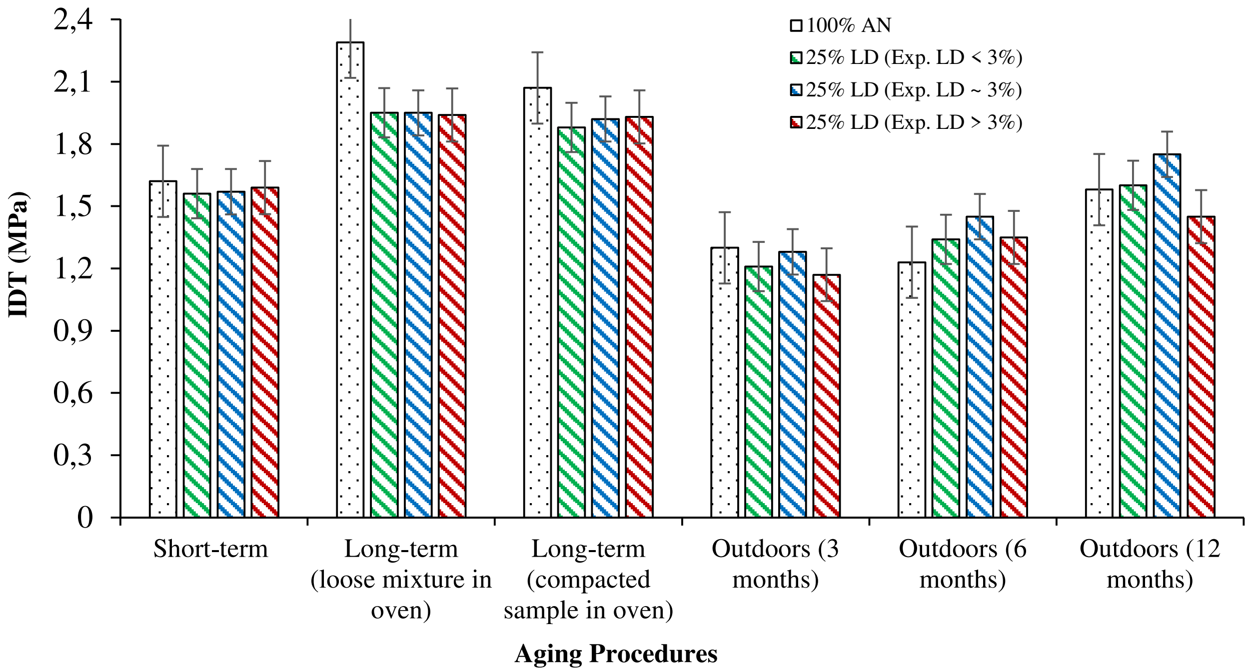


Figure 4. IDT Results considering different aging procedures

It appears that all studied asphalt mixtures presented IDT values above the minimum limit of 0.65 MPa, required by DNIT 031/2006 ES. Comparing mixtures with substitution of AN for LD, it is possible to infer about the effects of the level of expansion on mechanical performance. It is observed that there was no considerable difference in the tensile strength of mixtures produced with slag at different levels of expansion. However, a greater increase in IDT was observed for aged mixtures without the use of steel aggregate. This result can be associated with two reasons: i) the slag may have chemically reacted with the asphalt binder in order to delay the effects of aging, or ii) the slags have undergone expansion, resulting in a decrease in tensile strength due to microcracks and/or adhesive fractures on the binder-aggregate interface.

To evaluate this hypothesis, expansion tests were performed on samples of aggregates after they had been subjected to different aging processes. These will be presented later. For mixtures aged outdoors, no significant differences were found in the IDT values. This indicates that the expansive effects were not manifested in a way to decrease IDT in the first 12 months, but they can be manifested for ages older than 12 months.

3.2. Moisture Induce Damage (MID)

The results of the MID tests on samples submitted to 1 cycle and 5 cycles of thermal conditioning are shown in Table 4.

As can be seen, the percent of AV of the samples aged outdoors (natural aging) are 4.0 ± 1.0%, as these were initially compacted according to the Marshall mix design methodology for checking the IDT requirements. The MID tests were later incorporated into the experimental plan, being not feasible due to the research time, to allow the natural aging of new samples with AV of 7.0 ± 1.0%. Because of this, the results are analyzed separately from samples that have AV specified in the MID test standard, i.e. 7.0 ± 1.0%. Figure 5 shows the TSR results of the mixtures in the form of a clay graph with the respective standard deviations.

Table 4 – IDT values for CSs submitted to 1 or 5 cycles of MID

Mixture	Aging	One MID cycle				Five MID cycles			
		AV (%)	IDT (MPa)	IDTc (MPa)	TSR (%)	AV (%)	IDT (MPa)	IDTc (MPa)	TSR (%)
100% NA	Natural (12 months)	3.50	1.58	2.18	137.97	3.71	1.58	1.06	67.09
	Natural (6 months)	4.01	1.22	1.36	111.17	3.88	1.22	1.54	125.61
	Natural (3 months)	3.91	1.30	1.83	140.15	2.54	1.30	1.41	108.44
25% LD<3%	Natural (12 months)	4.08	1.60	1.75	118.37	3.79	1.60	1.39	87.28
	Natural (6 months)	3.40	1.34	1.71	127.24	4.27	1.34	1.85	138.06
	Natural (3 months)	5.54	1.21	1.73	142.70	5.24	1.21	1.68	138.84
25% LD≈3%	Natural (12 months)	3.97	1.75	1.86	106.48	3.83	1.75	1.77	101.14
	Natural (6 months)	3.25	1.45	1.72	118.66	3.49	1.45	1.77	122.35
	Natural (3 months)	4.52	1.28	1.49	116.15	4.72	1.28	1.43	111.72
25% LD>3%	Natural (12 months)	3.80	1.45	1.67	115.67	4.18	1.45	1.70	117.28
	Natural (6 months)	3.22	1.36	1.88	138.33	3.30	1.36	1.79	131.57
	Natural (3 months)	4.03	1.17	1.76	150.86	4.39	1.17	1.56	134.00
100% NA	Short-term	7.23	1.24	0.87	70.43	7.28	1.24	0.71	56.85
25% LD<3%		7.37	1.15	1.06	92.15	7.57	1.15	0.95	82.56
25% LD≈3%		7.47	1.24	1.11	89.78	7.74	1.24	1.05	84.95
25% LD>3%		7.22	1.31	0.97	74.23	7.12	1.31	0.88	67.35
100% NA	Long-term (aging of CS)	6.35	1.77	1.31	74.34	7.00	1.77	1.00	56.79
25% LD<3%		7.42	1.49	1.31	88.14	6.77	1.46	0.92	62.89
25% LD≈3%		6.78	1.79	1.39	78.06	6.77	1.79	1.23	74.80
25% LD>3%		7.24	1.77	1.38	78.19	6.82	1.77	1.09	61.57

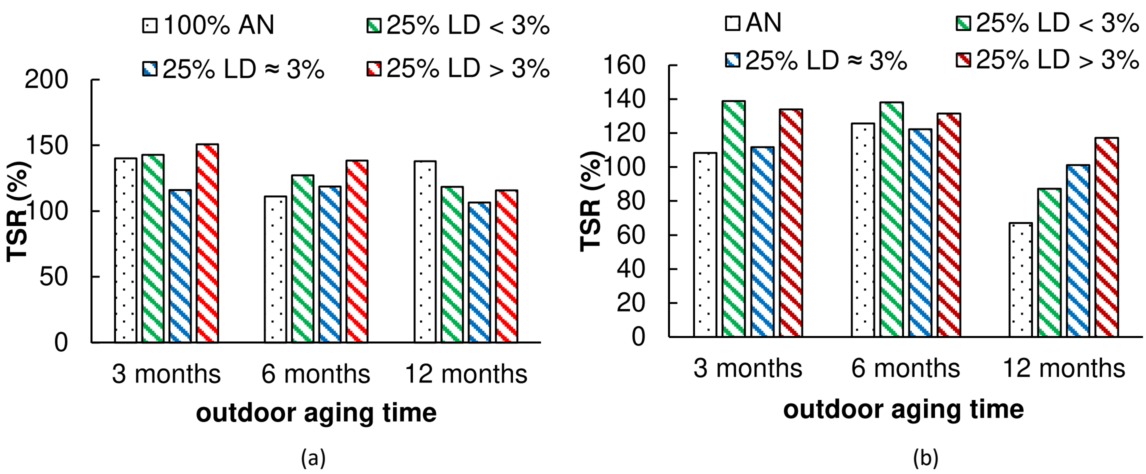
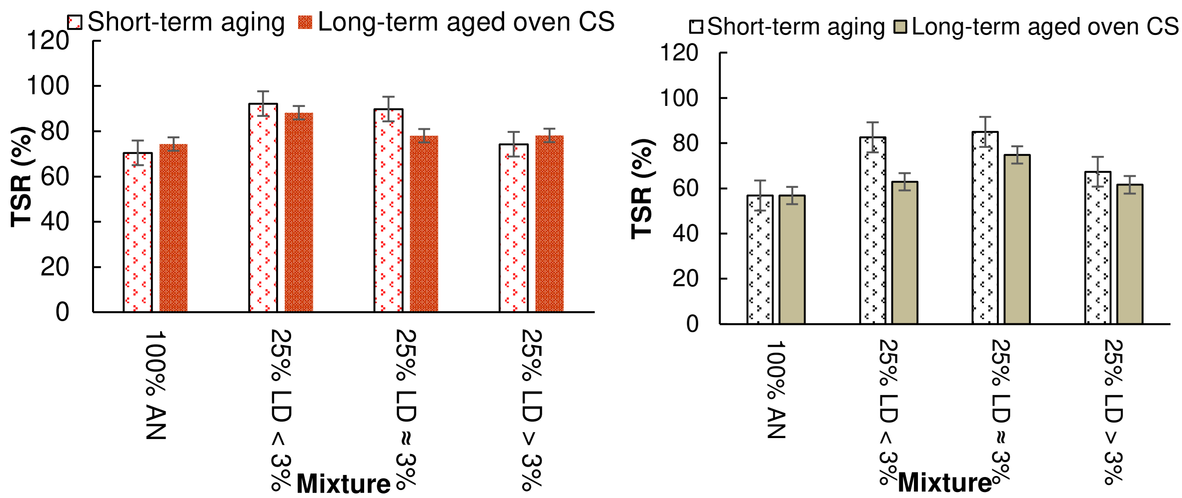


Figure 5. TSR result for CSs submitted to a) 1 MID cycle and b) 5 MID cycles aged outdoors

For outdoor aging samples, it is possible to verify that all of their TSR values were greater than 100% when subjected to 1 MID cycle, as shown in Figure 5a, even for the lowest standard deviation value, meaning that the values of tensile strength with conditioning were higher than those not conditioned, a result that may be related to the lower air voids of these in relation to samples aged in the laboratory. It can be inferred that, due to the lower air voids, the level of severity to which they were subjected in conditioning was minimized by the smaller volume filled with water during saturation and, thus, less volumetric expansion caused by water during the freezing cycle. Analyzing the samples with 25% LD at different levels of expansion, it was not possible to make any statement of their behavior, however it is possible to observe that the sample of 25% LD ≅ 3 obtained lower TSR values than mixtures with different expansion values.

For the CSs submitted to 5 cycles of MID (Figure 5b), the greatest damage is noticeable in the samples that were aged for 12 months outdoors, however, it was not possible to observe a correlation with the samples that underwent 1 cycle for the other mixtures and time aging. Unlike the expansion of the steel aggregate used, it did not show any tendency for the moisture damage to improve or worsen for these analyzed samples.

For samples aged in the laboratory, the results are shown in Figure 6. It is possible to verify that the TSR values were less than 100%, even for the highest standard deviation value. However, all CSs were above the minimum of 70% required by DNIT 031 (2006).



(a) (b)

Figure 6. TSR result for CSs submitted to: a) 1 MID cycle and b) 5 cycles of MID aged in an oven

It is observed that the samples with substitution of the natural aggregate for LD co-product, presented higher values than the samples with 100% of natural aggregate, indicating that there was an improvement in resistance to MID with the substitution of AN for LD. This result may be related to the presence of calcite minerals, as shown in the mineralogical analysis, which increases the ligand-aggregate adhesion. In addition, the presence of iron oxide (Fe_2O_3), may also have contributed to the improvement in the adhesiveness of asphalt mixtures, as described in Lucas Júnior (2018) and Cala et al. (2019a; 2019b). For the mixture with LD <3%, the reduction in IDT values was even smaller, resulting in a greater TSR. For the highest level of expansion, a greater reduction (but similar to that of AN) was observed, showing that only for the mixture with 25% LD> 3%, the expansion may have contributed to decrease the TSR of this mixture, although the values are still close.

For samples aged in an oven and conditioned with 5 freezing and thawing cycles, it is possible to verify that there was a decrease in TSR in relation to the results obtained with only 1 cycle, as expected. In Figure 6b, when comparing samples with and without substitution of natural aggregate for steel aggregate, it is noticeable that samples with LD had a higher TSR value. With regard to the difference in expansion of the steel aggregate, it can be seen once again that the level of expansion of the slag did not affect the performance of the mixtures differently.

3.3. Evaluation of the expansion of the composition of aggregates after aging and conditioning of MID

Initially, the expandability test was performed, according to the DNIT 113/2009 - ME standard, on the three steel aggregate samples, pure LD as shown in Figure 7a, thus characterizing the expansion of the aggregate. As the asphalt mixtures studied are composed of 25% of LD slag and 75% AN, it was necessary to perform the mixture expansion test, shown in Figure 7b. This result, corroborated with the expected, for each mixture we had a new expansion value lower than that of the pure steel aggregate and the expansion values of the mixtures, 1.01%, 2.10% and 2.14%, increased as the increase in expansion of the steel aggregate used. It was also verified if the solvent used for the extraction of the bituminous material could influence the expansion results. For this verification, an asphalt mixture was prepared, and after the extraction of the binder, the expansion test was performed showing the values of 0.98%, 2.09% and 2.16% for mixtures 25% LD <3% , 25% LD ≅ 3% and 25% LD> 3%, respectively. As shown in Figure 7c, it can be seen that the solvent used hardly changed the expansion value of the mixture.

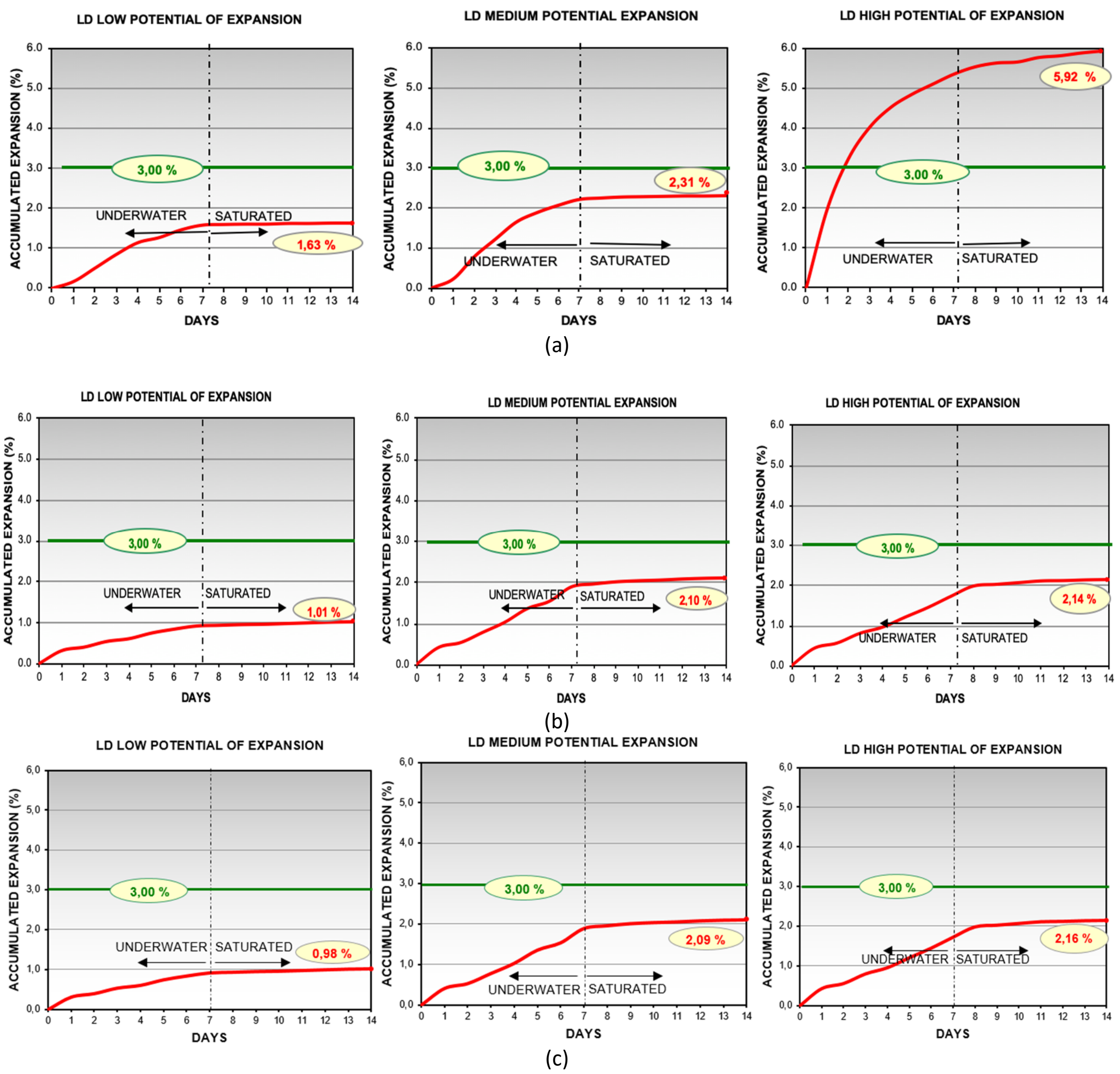


Figure 7. Results of the expansion potential test: a) pure slag, b) mixture 25% LD and 75% AN and c) mixture after solvent extraction

After performing the MID test, the tested CSs were separated for the extraction of the asphalt binder and for the PTM test. Figure 8, presents graphically the behavior of the expansions of the mixtures according to the aging methods used.

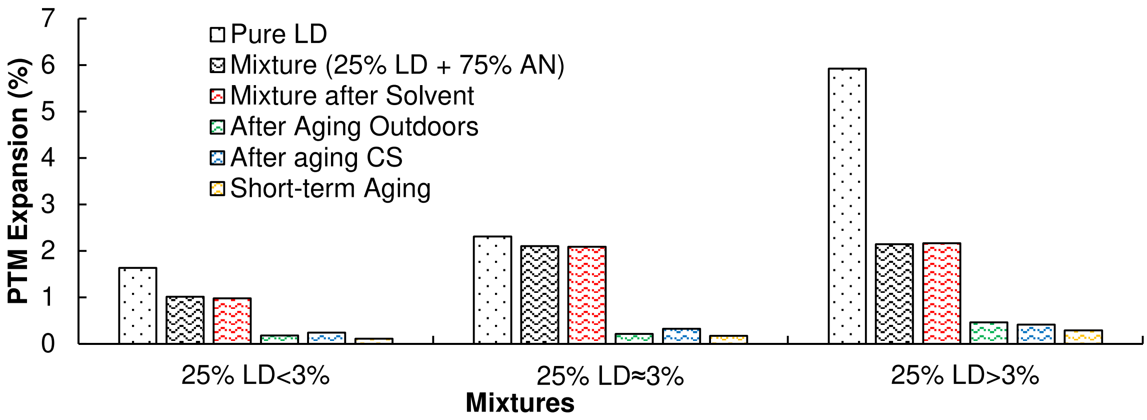


Figure 8. Analysis of Aggregate's Expansion after Aging and MID

Analyzing the expansion results, it is possible to verify that there was a decrease in the expansion values after the aggregate samples went through the aging and conditioning of MID. As the expansion values decreased, it is believed that during the aging and conditioning process, there may have been chemical reactions where they made the steel aggregate stabilize. This stabilization may have occurred due to two reasons individually or in combination: i) hydration of free CaO and MgO, causing the steel aggregates present to expand during the aging and MID procedures; ii) reaction of free CaO and MgO, present in the steel aggregate, with binder, and thus would no longer be free to form calcium hydroxide - $Ca(OH)_2$-, nor magnesium hydroxide - $Mg(OH)_2$-, resulting in a stable mixture in terms of volumetric expansion in addition to slowing the aging of binder due to interactions between the chemical compounds of the two materials. It should be noted that this occurred for the mixtures under study, where the level of substitution of AN by LD was 25%, but it should be noted that for substitution levels greater than 25%, it is necessary to assess these effects in the performance of asphalt mixtures.

3.4. Statistical analysis

Based on the results obtained experimentally in the laboratory, analysis of variance (ANOVA) was performed for a 95% degree of reliability and multiple mean comparison. In order to ratify the conclusions about the tensile strength, the study of the influence of the type of mixture (100% AN, 25% LD <3%, 25% LD≅3% and 25% LD> 3%) and the aging method (natural aging for 12 months, short-term and long-term of loose and compacted mixtures). Table 5 presents the results obtained for the statistical analysis of the influence of the type of mixture and aging on the IDT results.

In general, it is possible to conclude that in all cases both the materials used in the mixtures and the aging directly influence the IDT values. It is also possible to state that the higher the level of expansion, the greater the degree of significance in the second order effects between the type of mixture and the aging procedure. Parallel to this analysis, it becomes necessary to assess whether the results found for IDT in each case can be considered statistically the same or different. Thus, using the multiple mean comparison, a comparison was made between mixtures only with AN and the mixture with LD at the highest level of expansion (most severe case), as shown in Table 6.

Table 5 – ANOVA - influence of the type of mixture and aging on the tensile strength of the mixtures

Variation source	SS	Degree of freedom	MS	F	p	%
			25%LD<3%			
Effect	79.24300	1	79.243	7927.6	0.0000	100.00%
Mixture	0.12760	1	0.1276	12.766	0.00254	99.75%
Aging	1.31125	3	0.4370	43.726	0.0000	100.00%
mixture * aging	0.11131	3	0.037	3.712	0.0335	96.64%
Error	0.15993	16	0.0100			
			25%LD≅3%			
Effect	81.622	1	81.622	7883.0	0.0000	100.00%
Mixture	0.05042	1	0.05042	4.869	0.0422	95.77%
Aging	1.15068	3	0.38356	37.044	0.0000	100.00%
mixture * aging	0.20342	3	0.0678	6.549	0.0042	99.57%
Error	0.16567	16	0.0103			
			25%LD>3%			
Effect	81.585	1	81.58	10398.6	0.000	100%
Mixture	0.0513	1	0.051	6.54	0.021	97.89%
Aging	1.1428	3	0.380	48.55	0.000	100%
mixture * aging	0.2080	3	0.069	8.84	0.001	99.89%
Error	0.1255	16	0.007			

NOTE: SS = sum of squares; MS = medium square; F = F-statistic (Fisher-Snedecor F distribution with k-1 and n-k); p = significance probability

Table 6 – Multiple comparison of tensile strength means for LD> 3%

Multiple average comparison						
Mixture	**Aging**	**IDTtaverage**	**1**	**2**	**3**	**4**
25%LD >3	Short-term	1.570	****			
100% AN	Natural	1.580	****			
100% AN	Short term	1.623	****			
25%LD >3	Natural	1.750	****	****		
25%LD >3	Long-term CS.	1.930		****	****	
25%LD >3	Long-term loose mixture	1.940		****	****	
100% AN	Long-term CS.	2.067			****	****
100% AN	Long-term loose mixture	2.290				****

In Table 5, only the influence of the replacement of 25% of natural aggregates by LD slag and the effect of this replacement in the IDT values after aging were evaluated to see if there is statistical difference between them. In Table 6, a multiple comparison analysis (Tukey Test) is then made using the mean values of mixtures with 100% AN and 25%LD>3% and different aging conditions, to see if statistically the mean value in each situation is the same or different. The software Statistica automatically separates into groups after performing the Tukey Test, which makes a comparison of all possible pairs of means, allowing the creation of confidence intervals (CI) for all differences. A CI configures itself in a range of probable estimates, which the true value of the parameter can assume. In Tukey's test, for the level of significance level of 5%, there is a 95% probability of the CI containing the true mean value. A CI for the difference between the paired means that contains the value zero indicates that the difference is not significant, that is, that there does not exist a difference between the means. The test is necessary when there is a difference, as it completes the analysis by comparing and then showing the result of all combinations of pairs of means. In this study it was identified four groups, named 1,2,3,4 in Table 6. The symbol "****" is used to identify mixtures that have mean IDT values statistically similar (CI with zero value).

It is observed that the result points to the existence of 4 groups. From group 1, it is possible to observe that the IDT value of CSs with 100% natural aggregate aged outdoors (natural aging) for 12 months is statistically equal to the IDT of mixtures with 25% LD> 3%, under the same conditions. This shows the possibility of replacing the natural aggregate with LD slag> 3%. From group 2, the most relevant result is that the IDT of the mixture 25% LD> 3% aged outdoors is equal to that of the mixture 25% LD> 3% subjected to long-term aging, which indicates that the degree of aging found in 12 months in the field would be similar to that found simulating under laboratory conditions. From group 3, it is possible to extract two different results: the first is that there is no statistical difference between the IDT values if the mixture 25% LD> 3% was aged in a loose or compacted state; the second result is that the IDT of the mixture 25% LD> 3% long-term aged is statistically similar to that obtained in the mixture with 100% AN. Finally, from group 4, it shows that long-term aging methods (loose mixture or compacted specimen) resulted in statistically similar results.

4. CONCLUSIONS

In this work, the mechanical behavior of asphalt mixtures with and without partial replacement of the natural aggregate by steel slag aggregates was evaluated considering different steel slag aggregate in terms of expansion potential. The mixtures were subjected to different aging procedures and damage induced by moisture in order to verify how the asphalt mixtures would be affected by possible expansions of the steel aggregates during short- and long-term aging. Based on the results, it is possible to conclude that:

- Regarding the evaluated mechanical performance, no significant difference was found in the tensile strength on the mixtures containing steel slag aggregates at different levels of expansion when the mixtures were aged in both the short- and long- term by different aging methods.
- Regarding the resistance to moisture induce damage of mixtures aged in the laboratory, it was found that the TSR values of the samples subjected to MID test were above of 70%, and it can be observed that the samples with substitution of the natural aggregate by LD slags showed higher values than the sample with 100% natural aggregate. This result can indicate the better adhesion of the steel aggregate to the asphalt binder in comparison to the natural aggregate. The better adhesion can be justified by the mineralogy of the steel aggregate, with the presence of iron and calcium oxides that may be acting positively in a better aggregate-binder adhesion.
- For samples aged in an oven and MID conditioned for 5 cycles, some samples did not meet 70% on TSR, as it was observed in the results of the mixture with the 100% natural aggregate sample and the LD sample with expansion greater than 3%. However, this conditioning is quite severe and only indicated that even with different expansion levels, the behavior of mixtures containing LD steel slags was similar, indicating that the slag cure process may not be necessary since this variable (level of expansion) did not show considerable negative impact on results.
- Regarding the variation in the PTM expansion results of the composition of aggregates extracted from the asphalt mixtures after the conditioning procedures, a reduction in the

expansive effects was observed. It is considered here that there may have been hydration of the free calcium and magnesium oxides or interaction of these oxides with the binder compounds making the mixture stable. It is emphasized that these hypotheses need to be verified using other methodologies and scales of analysis.

- In general, there was no statistically significant difference between the resistance values found, showing the feasibility of replacing natural aggregate with 25% LD slag for the production of asphalt mixtures. It was also found that for this percentage of substitution, the expansive effects are insignificant. However, it is necessary to evaluate these effects for higher substitution percentages.

ACKNOWLEDGEMENTS

This study was financed in part by ArcelorMittal Tubarão, and by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001. The second author also thanks the CNPQ for the productivity scholarship.

REFERENCES

Araújo, M. F. A. (2007) *Efeito da Radiação Solar no Envelhecimento do Concreto Betuminoso Usinado a Quente*. MSc thesis. Universidade Federal de Ouro Preto, Ouro Preto. Available in: <http://www.repositorio.ufop.br/handle/123456789/2845> (Access in: 08/24/2021).

ABNT (2021) *NM 51: Agregado graúdo – Ensaios de abrasão "Los Angeles".* Brazilian Technical Standards Association, Rio de Janeiro.

ABNT (1984) *NBR 6458: Grãos de pedregulho retidos na peneira de 4,8 mm determinação da massa específica, da massa específica aparente e da absorção de água.* Brazilian Technical Standards Association, Rio de Janeiro.

ABNT (2016) *NBR 7181: Análise granulométrica.* Brazilian Technical Standards Association, Rio de Janeiro.

ABNT (1990) *NBR 11303: Análise química de materiais refratários aluminosos por espectrometria de fluorescência de Raios X.* Brazilian Technical Standards Association, Rio de Janeiro.

Beshears, S., and A. Tutumluer (2013) *Reclaimed Asphalt Pavement with Steel Slag Aggregate: Successful Use in Illinois Pavements.* TR News, n. 288, p. 46-47. Available in: <http://citeseerx.ist.psu.edu/viewdoc/download?doi=10.1.1.698.3443&rep=rep1&type=pdf> (Access in: 08/25/2021).

Cala, A.; Caro, S.; Lleras, M., and Rojas-Agramonte, Y.(2019a) Impact of the chemical composition of aggregates on the adhesion quality and durability of asphalt-aggregate systems. *Construction and Building Materials*, v. 216, p. 661–672. DOI: 10.1016/j.conbuildmat.2019.05.030.

Cala, A.; Caro, S.; Lleras, M., and Rojas-Agramonte, Y. (2019) Understanding the role of the chemical composition of aggregates on the moisture susceptibility of asphalt mixtures. *Transportation Research Meeting*, Report/Paper Numbers: 19-04533, p. 1–7.

DNER (1995) *ME 043: Misturas Betuminosas a Quente – Ensaio Marshall*. Nacional Department of Roads, Rio de Janeiro.

DNER (1997) *ME 054: Equivalente de areia.* Nacional Department of Roads, Rio de Janeiro.

DNER (1994) *ME 078: Agregado graúdo - Adesividade a ligante betuminoso*. Nacional Department of Roads, Rio de Janeiro.

DNER (1994) *ME 089: Agregado – Avaliação da durabilidade pelo emprego de solução de sulfato de sódio ou de magnésio*. Nacional Department of Roads, Rio de Janeiro.

DNER(1995) *ME 152: Agregado em estado solto – determinação da massa unitária*. Nacional Department of Roads, Rio de Janeiro.

DNER 91994) *ME 262: Escória de aciaria para pavimentos rodoviários*. Nacional Department of Roads, Rio de Janeiro.

DNIT (2009) *113- ME: Pavimentação rodoviária – Agregado artificial – Avaliação do potencial de expansão de escória de aciaria*. Instituto de Pesquisas Rodoviárias - IPR, Rio de Janeiro.

DNIT (2006) *031- ES: Pavimentos flexíveis - Concreto asfáltico*. Instituto de Pesquisas Rodoviárias - IPR, Rio de Janeiro.

DNIT (2017) *136- ME: Pavimentação asfáltica – Misturas asfálticas – Determinação da resistência a tração por compressão diametral*. Instituto de Pesquisas Rodoviárias - IPR, Rio de Janeiro.

DNIT (2018) *180 - ME: Pavimentação – Misturas asfálticas – Determinação do dano por umidade induzida*. Instituto de Pesquisas Rodoviárias - IPR, Rio de Janeiro.

Groenniger, J.; Cannone, F. A.; Isailovic, I.; Wang, D., and Wistuba, M. P. (2017) Experimental investigation of asphalt mixture containing Linz-Donawitz steel slag. *Journal of Traffic and Transportation Engineering* (English Edition), v. 4: p. 372-379. DOI: 10.1016/j.jtte.2017.05.009.

IABR (2021) *Relatórios De Sustentabilidade - Setor do Aço no Brasil - Produtos e Mercado*. Instituto Aço Brasil. Available in: <http://www.acobrasil.org.br/sustentabilidade/assets/pdfs/Aco-Brasil-Completo.pdf> (Access in: 08/24/2021).

Lucas Júnior, O. L. (2018) *Influencia da adesividade agregado-ligante no trincamento por fadiga de misturas asfálticas*. MSc thesis. Universidade Federal do Ceará. Fortaleza. Available in: <http://www.repositorio.ufc.br/bitstream/riufc/34068/1/2018_dis_jlolucasjunior.pdf> (Access in: 08/24/2021).

Kim, K.; Haeng Jo, S.; Kim, N., and Kim, H. (2018) Characteristics of hot mix asphalt containing steel slag aggregate according to temperature and void percentage. *Construction and Building Materials*, v. 188, p. 1128-1136. DOI: 10.1016/j.conbuildmat.2018.08.172.

Moura, B. L. R. (2020) *Avaliação da adesividade ligante-escória sob aspectos físicos, químicos e termodinâmicos e correlação com desempenho mecânico de misturas asfálticas contendo diferentes fíleres melhoradores de adesividade*. MSc thesis. Universidade Federal do Espírito Santo. Vitória. Available in: <http://portais4.ufes.br/posgrad/teses/tese_14448_Disserta%E7%E3o_Barbara_VF_Corrigido.pdf> (Access in: 08/24/2021).

National Academies of Sciences, Engineering, and Medicine (2017). *Long-Term Aging of Asphalt Mixtures for Performance Testing and Prediction*. Washington, DC: The National Academies Press. DOI: 10.17226/24959.

PTM (2016) *130: Evaluation of Potential Expansion of Steel Slags*. Pennsylvania Department of Transportation, PA, p. 1–8.

Shen, D., C. M. WU, and J. C. DU. (2009) Laboratory Investigation of Basic Oxygen Furnace Slag for Substitution of Aggregate in Porous Asphalt Mixture. *Construction and Building Materials*, v. 23, p. 453–461. DOI: 10.1016/j.conbuildmat.2007.11.001.

Tavares, D. S. (2012). *Avaliação Laboratorial de Mistura Asfáltica do Tipo SMA com Agregado Siderúrgico*. MSc. thesis. Universidade Federal do Rio de Janeiro, Rio de Janeiro.

Teixeira, J. E. S. L.; A. G. Schumacher; P. M. Pires; V. T. F. C. Branco, and H. B. Martins (2019) Expansion Level of Steel Slag Aggregate Effects on Both Material Properties and Asphalt Mixture Performance. *Transportation Research Record*, v. 2673, p. 506-515.DOI: 10.1177/0361198119835513.