Effect of Thermal Freeze/Thaw Cycles on the Rheological Behaviour of Limestone-Modified Bituminous Concrete

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Abstract

The purpose of this article is to expand knowledge on the rheology of asphalt concrete with different percentages of limestone fines that have been affected by freeze-thaw thermal cycles. Freeze-thaw cycles cause swelling and settling in pavements, resulting in degradation of the pavement structure. Deformations start with small cracks and then develop parallel or perpendicular to the road axis. The sequence and frequency of freeze-thaw cycles lead to pavement deterioration, which can be transmitted to other layers of the road in the absence of prompt intervention measures. Degradation can take various forms, including deformations, cracks, detachment, and potholes. To simulate the freeze-thaw cycle phenomenon of asphalt concrete in the laboratory, a freezer and a temperature-controlled chamber were used to generate the real freeze-thaw cycle conditions in winter. The obtained results demonstrate that the addition of limestone filler proved to be highly significant as it ensured an increase in the dissipated energy based on the percentage of filler added to the mixture. After the application of freeze-thaw cycles, higher values of dissipated energy were obtained compared to the control sample. Freeze-thaw cycles have an effect on increasing the dissipated energy, which leads to improved resistance to cracking.

Keywords: limestone fines, rheology, behavior, freeze-thaw, Fénix test, dissipated energy, displacement.

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1-Introduction

Asphalt mixtures are commonly used throughout the country's territory. Their various formulations are tailored to the specific locations and intended uses of the areas involved. Their utilization provides a high level of comfort. The surfaces can be opened to traffic immediately after their implementation. The application of asphalt mixtures always involves compaction, which should be carried out under suitable weather conditions *[SMAEG, 2011].*

In bituminous mixtures, the binder provides cohesion by wetting the mineral grains and enveloping them with a continuous film. The granular skeleton represents the rigid framework of the mixture. The size distribution of different aggregates determines the particle size composition of the granular skeleton, which can be continuous or discontinuous depending on the intended use of the mixture and the desired properties *[Nguyen, 2006].*

The objective of blending different granular classes is to create a granular skeleton with a void content that is neither too low nor too high. A very low void content hinders the introduction of an adequate amount of binder to coat all the grains without saturating the mixture, while a high void content promotes the development of permanent deformations through post-compaction, as mentioned in *[Grimaux and Hiernaux, 1977]* and *[Cross and Brown, 1992].*

Bituminous materials are highly heterogeneous polyphasic materials. Their mechanical characteristics and properties are determined by the mechanical and physical properties of their components, as well as the volumetric composition of the mixture *[Nguyen, 2006].*

A bituminous mastic is composed of bitumen and filler (also known as "fines"), which are defined by standards as the elements passing through an 80 μm sieve. These fines are fine products obtained through fragmentation and may also contain pollutants. Therefore, the mastic, formed by suspending the fines in the bitumen, becomes the actual binder. It is observed that the agent responsible for agglomerating the granular skeleton of an asphalt mixture is, in fact, the mastic, which is the bitumen-filler mixture.

The fact that bituminous mastic can be considered as a suspension, while bitumen itself is a suspension, is a very interesting finding. This can be explained by the difference in size between the asphaltene particles (a few nm) and the mineral fines (a few μm), allowing the bitumen to be treated as a homogeneous medium with respect to the fines *[Lesueur and Little, 1999].* The filler absorbs a portion of the introduced binder and forms the mastic, which is stiffer and less thermally and kinetically susceptible than pure binder.

The main objective of this study is to investigate the effects of the addition of limestone fines to bituminous mixtures subjected to freeze-thaw cycles on the thermomechanical properties of the asphalt mixtures.

2-Effect of Freeze-Thaw Cycles

In general, the freeze-thaw action is a phenomenon that occurs during winter and early spring in cold climates. Virtually all surface soils are subjected to freeze-thaw action, the extent of which depends on the prevailing local climate and precipitation.

The freeze-thaw action can be divided into two phases: freezing and thawing of the soil water.

For road pavements, the freeze-thaw action becomes critical when the freezing phase is accompanied by significant uplift of the pavement surface (Fig.1), or when the thawing phase is accompanied by significant softening of the pavement structure (Fig.2).

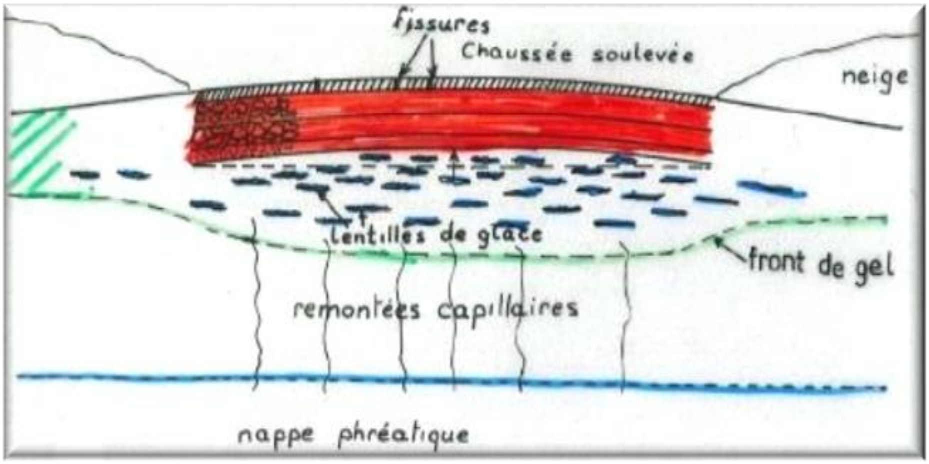

Fig. 1. The action of freezing *[Mauduit, 2010]*

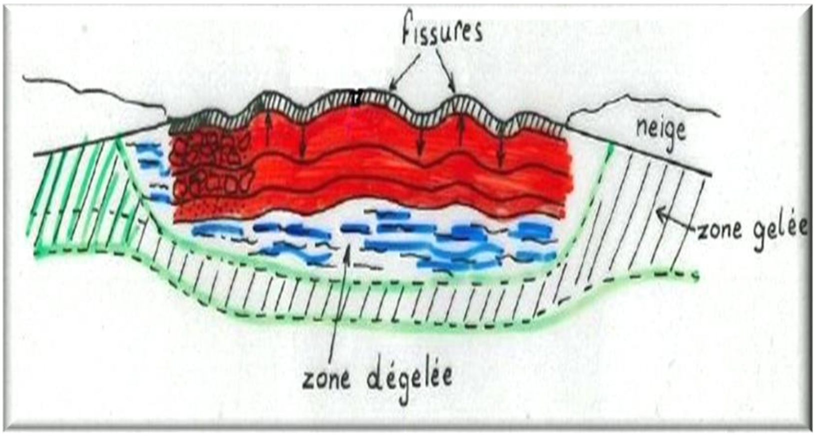

Fig. 2. The action of thawing *[Mauduit, 2010]*

3-Materials and Methods

3.1. Materials Used

The asphalt concrete was produced at the LECTO laboratory (Laboratory for Studies and Control of Structures).

3.1.1. Selection of Fresh Aggregates

This identification aims to verify that the proposed aggregates comply with the recommendations of the standards regarding their gradation, hardness, and cleanliness. The test results are compiled in Table 1.

Table 1: Physical Characteristics of Healthy Aggregates

Aggregate Testing	0/3	3/8	8/15	Specifications
Micro-Deval	-	-	13,74	≤ 20
Los Angeles	-	-	19,02	≤ 22
Flattening coefficient	-	10,39	10,21	≤ 25
Cleanliness Test	-	0,67	0,42	≤ 02
Bleu De METHYLENE	0,75	-	-	≤ 01
Equivalent de Sable	82,52	-	-	60 ≤
Coefficient of Friability of Sands	39,94	-	-	≤ 35

3.1.2. The New Binder

The bituminous binder used in this study is a class **40/50** binder obtained from a public works company in Bechar, Algeria. It is commonly used in airfield pavements and roads located in hot regions. The characteristics obtained, as shown in Table 2, demonstrate that the analyzed bitumen meets the requirements of the class 40/50 standard.

Table 2 : Characteristics of the Class 40/50 bituminous binder.

Quality	**Unit**	**40/50**
Softening point	°C	52 to 57
Penetrability at 25 °C	1/10 mm	40 to 50
Relative density at 25 °C	Gr/ml	1 to 1.10
Mass Loss to Heat	%	<1%
Flash Point	°C	> 250 °C
Ductility at 25°C	Cm	>60
Difference in TBA after RTFOT	D TBA °C	≤ 9

3.1.3. Proportion Calculation (Formulation)

a. Binder Content Calculation

The binder content is determined based on the specific surface area of the aggregates used and a selected binder content factor, which depends on the traffic volume.

T (binder content) = $\boldsymbol{\alpha}$ **. K .** $\sqrt[5]{\sum}$ **(en %)** (Table 3)

$\alpha = 2.65/\gamma G$ (γG is the density of the aggregates) = 0.98.

k = modulus of richness between 3.0 and 3.9.

$\sum$(Conventional Specific Area) =0.25G+2.3S+12S+135F (m²/kg).

G: proportion of elements greater than 6.3mm.

S: proportion of elements between 6.3mm and 0.315mm.

S: proportion of elements between 0.315mm and 0.08mm.

f : proportion of elements smaller than 0.08mm.

Table 3: Calculation of Binder Content

% Fine limestone	α	towards	$\sum$	$\sqrt[5]{\sum}$	T
4 %	0,98	3,45	11,56	1,63	5,50
5 %	0,98	3,45	12,76	1,66	5,61
6 %	0,98	3,45	13,96	1,69	5,72
7 %	0,98	3,45	15,16	1,72	5,81

b. Aggregate content calculations

Table 4: Percentage of each aggregate

% of each aggregate	4%	5%	6%	7%
Gravel 8/15	37%	37%	37%	37%
Gravel 3/8	21%	21%	21%	21%
Sand 0/3	38%	37%	36%	35%
fine limestone	4%	5%	6%	7%

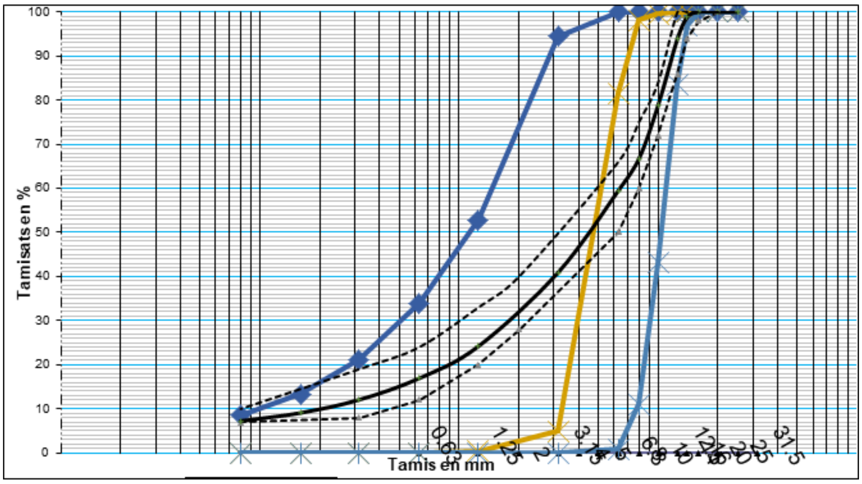

Fig. 3. The particle size of the aggregates

Table 5: Asphalt Characteristics

% Fine limestone	% Bitumen	Marshall Stability	% Environmental	Creep	Compactness
4 %	5,51	10,42	4,00	2,70	96,00
5 %	5,62	10,31	3,63	3,73	96,37
6 %	5,72	10,21	3,21	3,67	96,79
7 %	5,81	10,10	2,86	3,00	97,14

3.1.4. Limestone Fines

Limestone fines were used as a modifying agent in our asphalt concrete formulation in varying proportions, with a limestone content of 95.90%.

3.2. Test Method

This study is based on formulations with varying percentages of limestone fines, including:
- 4% of limestone fines.
- 5% of limestone fines.
- 6% of limestone fines.
- 7% of limestone fines.

The experimental method for thermal cycling in this study is as follows:
1. Begin by using a freezer to regulate and control the desired low temperature.
2. Place the Marshall specimens inside the freezer to initiate the freezing cycle.

3. Afterward, remove the Marshall specimens from the freezer and transfer them to a temperature-controlled chamber to induce thawing (thawing cycle).
4. Repeat the thermal cycles on the bituminous concrete specimens to generate freeze-thaw cycles.

The asphalt concrete was subjected to thermal cycles involving freezing and thawing. The test temperatures used were -5°C for freezing and 25°C for thawing. The samples were exposed to 100 cycles of thermal loading. Figure 4 illustrates a temperature cycle over a 24-hour period. As depicted in the figure, the duration of freezing and thawing was the same, lasting 12 hours each. For each applied thermal load, a constant duration of 3 hours was ensured during the test.

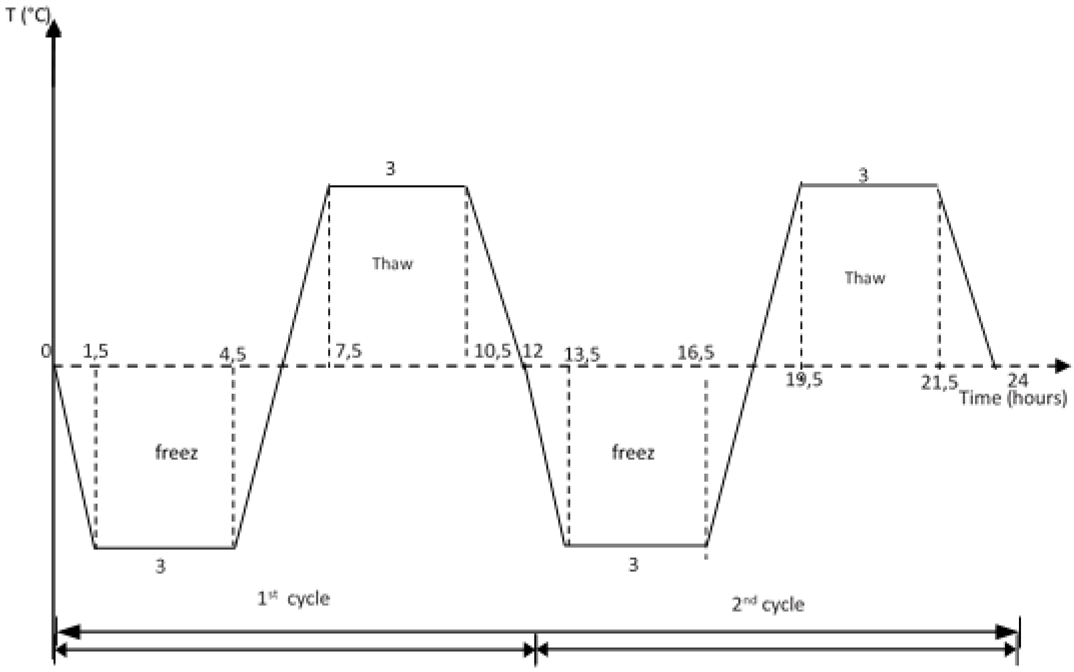

Fig. 4. Two Thermal Charging Cycles

3.3. Fénix Test

Various tests have been developed to assess the ability of bituminous materials to withstand this type of degradation. After undergoing different conditioning procedures, the materials are tested using methods such as the Marshall test, Lottman test, Duriez test, etc., which involve indirect tensile or simple compression testing. These tests primarily focus on failure under monotonic loading, while the viscoelastic behavior and fatigue performance are less frequently evaluated to assess their evolution under this type of degradation. This is why the changes in different types of behavior of bituminous materials under the influence of water, freeze-thaw cycles, and aging are still not well understood *[Tran D.T, 2020].*

A new experimental test has been developed by the Road Research Laboratory at the Technical University of Catalonia, Barcelona, Spain, to evaluate the crack resistance of asphalt concrete mixes by calculating the dissipated energy during the cracking process.

The Fénix test is used to calculate the dissipated energy during the cracking process, which is a combination of all the energies released during the deformation and cracking of the material. Evaluating this energy is an effective way to measure the crack resistance of asphalt concrete mixes *[Pérez-Jiménez et al., 2010] [Clopotel et al., 2012] [Valdés et al., 2011].*

The Fénix test, much like the Marshall test, is an experimental procedure that, similar to Marshall, is used to assess and quantify certain properties of the mixture. Its primary purpose is to determine the force required to crack a semi-cylindrical test specimen by applying tensile stress along its diametrical plane (Fig. 5).

The test specimen is attached to plates in its diametrical plane, which are then secured to the anchors of the testing machine. The test is conducted at a constant piston displacement speed of 1 mm/min. During the test, the applied stress is recorded as a function of displacement. The test specimen has a small slit between the two plates to induce the initiation of specimen cracking.

During the analysis of the load-displacement curve resulting from the test, various related parameters can be defined that characterize the mechanical properties and strength of the mixture *[Pérez-Jiménez et al., 2016].* Since its development, the Fénix test has been widely used in numerous studies to determine parameters such as effectiveness, sensitivity, and validity *[Puigrefagut Pla, 2018].*

The study of the rheological behavior of bituminous mixtures was conducted at the FIMAS laboratory. The Fénix test was performed on half-Marshall specimens at a temperature of 20°C (Fig. 5).

Cutting and gluing samples onto plates

Application of the test

Reading the results

Fig 5.Fenix Trial

4- Results and Discussion

4.1. Samples without cycles

The primary objective of this study was to investigate the potential for enhancing the performance of bituminous concrete by incorporating limestone fines into the formulation.

According to the obtained results, it can be observed that the dissipated energy increases with the percentage of added limestone fines in the mixture, ranging from **4% to 7%** (Fig. 7). This increase in energy dissipation has a positive effect on improving the performance of the mixture, as confirmed by *[Riahi, 2015].* The limestone fines have a favorable impact on the mechanical properties of the concrete. In addition to their granular role leading to increased compactness, they also contribute to higher resistance to cracking.

The asphalt mixtures obtained with limestone fines exhibit improved stability and better compactness, making them more resistant to deformation, as stated by *[Yaici, 2005]. [Diagne,* *2017]* also demonstrates that the addition of limestone fines in bituminous concrete enhances the stability of the mixture.

Based on the results, it can be concluded that the maximum displacement varies within the range of **[0.55 to 0.75 mm]** depending on the tension, which ranges from **[0.45 to 0.55 MPa]** (Fig. 6). However, when discussing the changes in dissipated energy, it is evident that there is a significant variation of approximately **420 J/m²** between the **[4%-5%]** and **[6%-7%]** limestone fines content. This explains the effect of adding limestone fines in increasing the dissipated energy, leading to an improvement in crack resistance (Fig. 7).

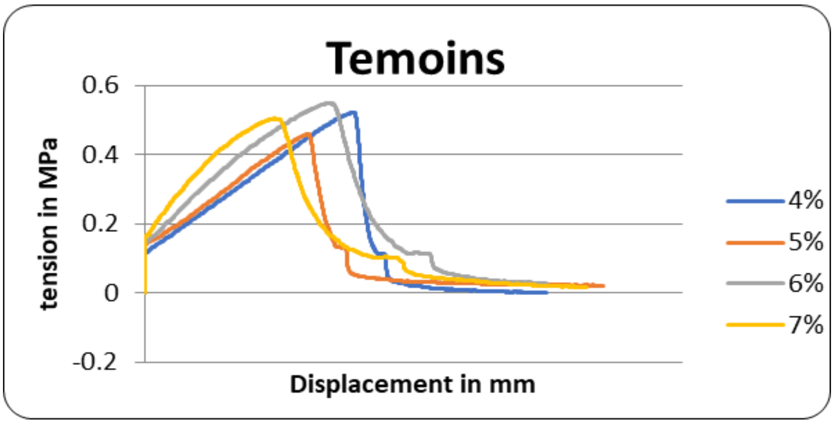

Fig. 6. Tension in MPa as a function of displacement in mm for each percentage of fine limestone

(Control samples)

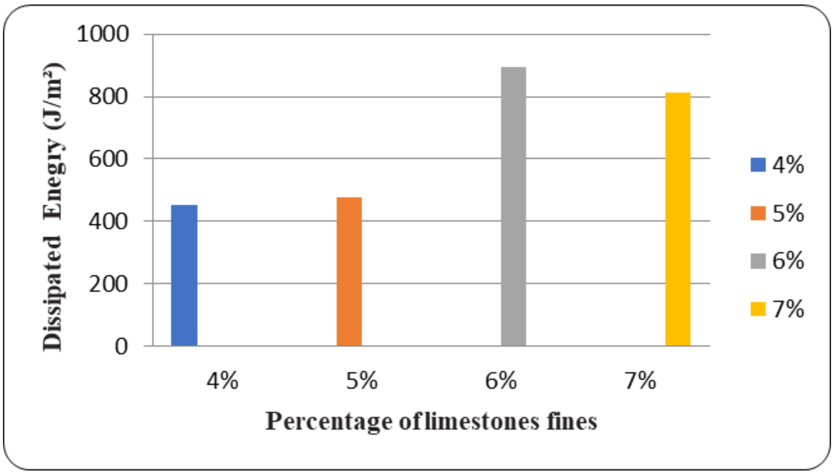

Fig. 7 .Energy dissipated (J/m²) as a function of the percentage of fine limestone

(Control samples)

4.2. Samples after Freeze-Thaw cycles

Thermal effects remain a broad and extensively discussed topic in bituminous materials. This is partly due to the fact that temperature variations affect different properties or characteristics. Mechanical properties are temperature-dependent, and crack resistance varies with temperature *[Laveissiere, 2002].*

After applying freeze-thaw cycles, it was observed that the maximum displacement values varied within a range of **[0.63 to 0.89 mm]** depending on the varied tensions ranging from [**0.47 to 0.54 MPa**] (Fig. 8). The maximum displacement increased after the application of freeze-thaw cycles.

If we compare the values of the dissipated energy, there is a significant variation between the **[4%-5%]** and **[6%-7%]** limestone fines content, with a difference of approximately **430 J/m²** (Fig. 9). Indeed, the samples subjected to freeze-thaw cycles exhibit higher values of dissipated energy compared to the control samples, and this increases with the percentage of limestone fines. This indicates that there is an aging effect due to freeze-thaw cycles, which leads to softer asphalt mixtures. This characteristic provides the modified mixture with increased ductility, resulting in improved crack resistance.

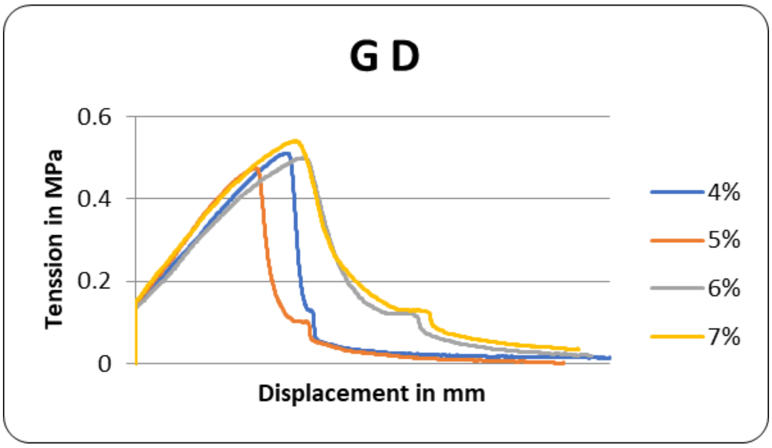

Fig. 8. Tension in MPa as a function of displacement in mm for each percentage of fine limestone (Samples after freeze-thaw cycles)

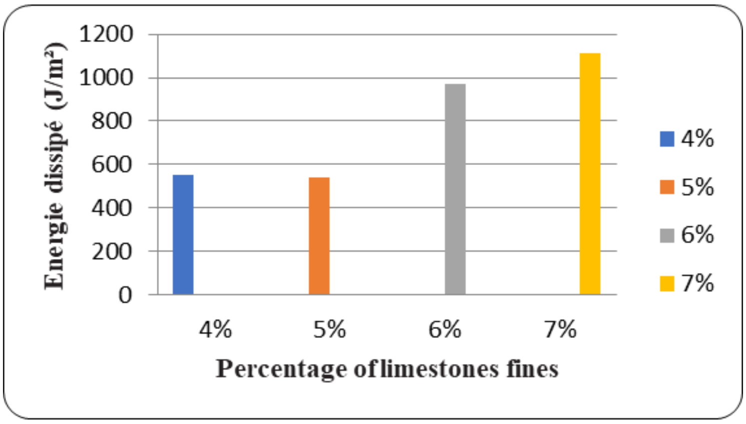

Fig. 9 .Energy dissipated (J/m²) as a function of the percentage of fine limestone (Samples after freeze-thaw cycles)

4.3. Comparison of the Freeze-Thaw Cycle Effects

4.3.1. 4% limestone fines content

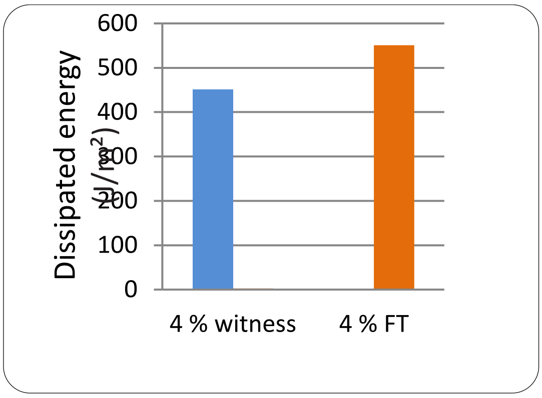

Fig.10 . Energy dissipated (J/m²) (Control sample and sample after freeze-thaw cycles with 4% limestone fines)

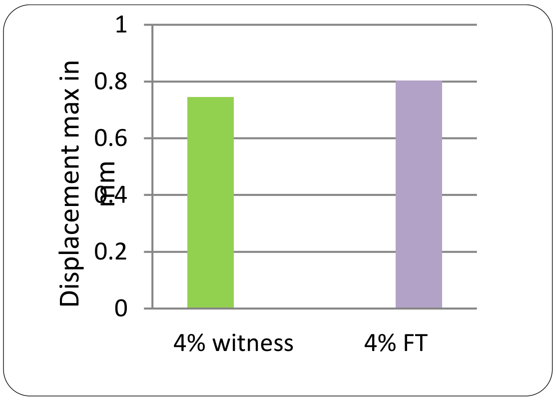

Fig.11 . Max displacement (mm)(control sample and sample after freeze thaw cycles with 4%Limestones Fines)

4.3.2. Percentage 5% of fine limestone

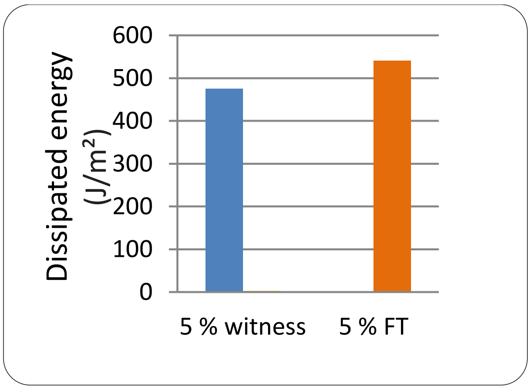

Fig.12 . Energy dissipated (J/m²) displacement (mm)(Control sample and sample after freeze-thaw cycles with 5% limestone fines)

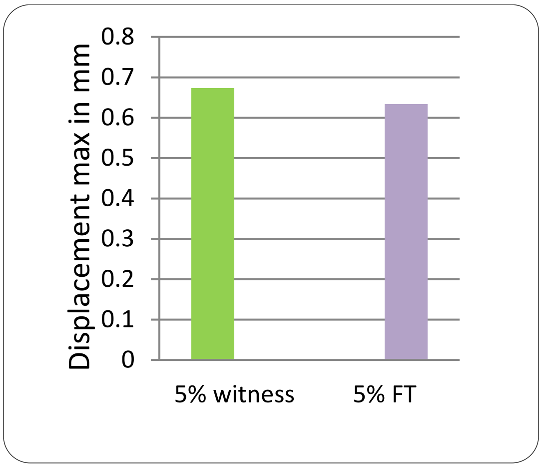

Fig. 13 . Max displacement (mm)(Control sample and sample after freeze-thaw cycles with 5% limestone fines)

4.3.3. Percentage 6% of limestone fines

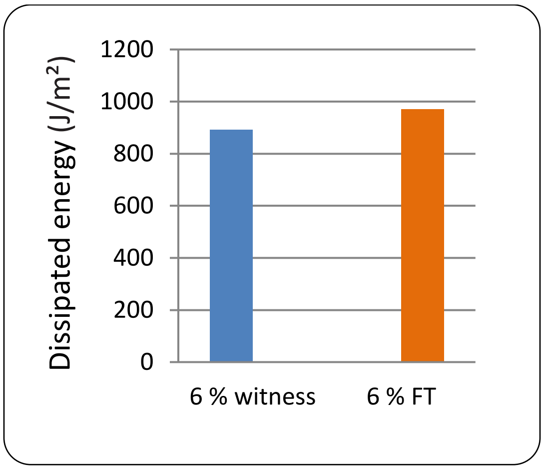

Fig.14 . Energy dissipated (J/m²)(Control sample and sample after freeze-thaw cycles with cycles with 6% limestone fines)

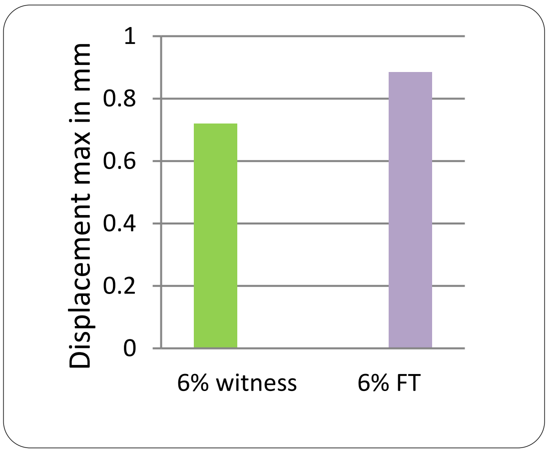

Fig.15 . Max displacement (mm)(Control sample and sample after freeze-thaw with 6% limestone fines)

4.3.4. Percentage 7% of limestone fines

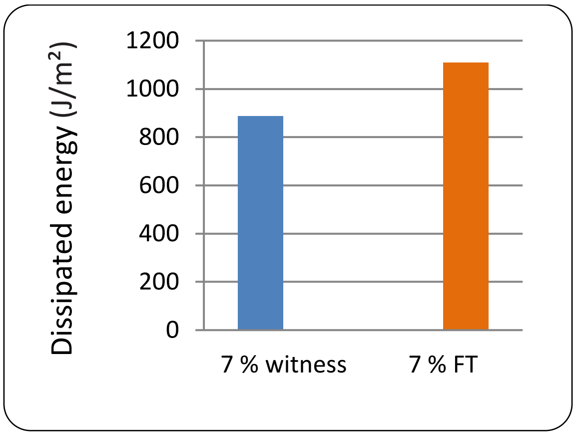

Fig. 16. Energy dissipated (J/m²)(Control sample and sample with 7% of limestone fines)

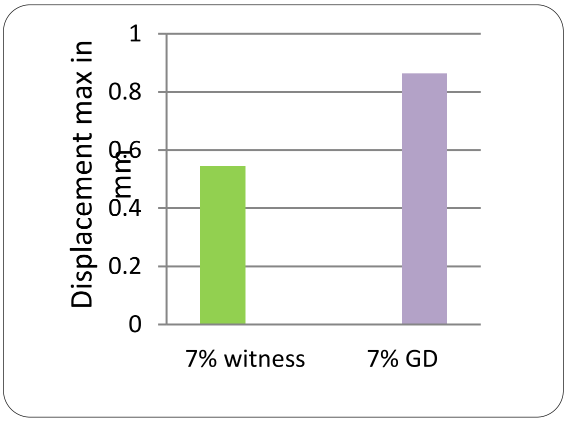

Fig. 17 . Max displacement (mm) (Control sample and sample with nearly freeze-thaw cycles with7% limestone fines)

It is observed that there is an increase in the maximum displacement and dissipated energy between the control sample and the sample after the application of freeze-thaw cycles. This finding is consistent with several studies, such as *[Tran D.T, 2020]*, which aligns with our study.

4.4.Rate of change of energy dissipated

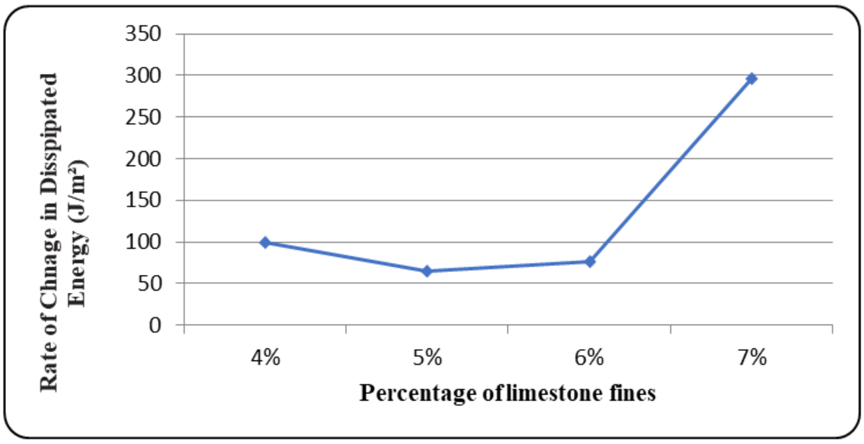

Fig. 18 . Rate of change of energy dissipated in (J/m²) as a function of Percentage of limestone fines (control sample and after freeze-thaw cycles)

It is worth noting that the rate of variation in dissipated energy is optimal at **the 7% limestone fines** content compared to other percentages, with a difference of **200 J/m².**

5- Conclusion

Bituminous mixtures appear to be relatively insensitive to the effects of freeze-thaw cycles. Therefore, winter degradation cannot be simply explained by material damage alone. The observed phenomena on-site, such as potholes, delamination, or peeling of the pavement layer, are likely related to other failure mechanisms associated with the structures. It would be important to further investigate the role of interfaces between pavement layers. Studying the damage to interfaces subjected to freeze-thaw cycles could be a promising avenue for future research *[Tran D.T, 2020].*

In our study, we can conclude the following:

- The addition of limestone fines is highly beneficial as it increases the dissipated energy in relation to the percentage of fines added to the mixture. The incorporation of limestone fines results in increased stiffness of the asphalt mixture, which explains its improved crack resistance that increases with the addition of limestone fines.

- After subjecting the samples to freeze-thaw cycles, higher values of dissipated energy were obtained compared to the control samples, which is in line with the findings of *[Tran D.T, 2020].* This suggests that there is aging due to freeze-thaw cycles, leading to softer asphalt mixtures. This characteristic imparts increased ductility to the modified mixture, resulting in improved crack resistance.

- For selection purposes, we opted for the sample with a **7% limestone fines content**, as it exhibited the highest rate of increase in dissipated energy and also had a higher maximum displacement compared to other percentages. This indicates improved crack resistance.

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