

INFLUENCE OF MIXING TIME AND TEMPERATURE IN HOT MIX ASPHALT STIFFNESS DURING ASPHALT MODIFICATION

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ABSTRACT

When an asphalt binder is modified through a wet process, high temperatures and long mixing times are used. In most research carried out on this subject, it has been concluded that, when modifying asphalts through a wet process, the base asphalt's stiffness increases. However, the contribution the used modifier makes to this mechanical parameter is unclear, as well as what is the effect of elevated mix temperatures (T) and times (t). In order to evaluate the influence of T and t , this study designed an experimental phase that attempts to simulate within a laboratory, the wet modification process of an AC 60-70 asphalt binder. AC 60-70 was exposed to similar T and t to those found in several asphalt modification studies, in order to then manufacture with said asphalts, Hot Mix Asphalts (HMA). HMAs were subjected to Marshall Tests, Indirect Tensile Strength Tests (ITS), Cantabro Test, Resilient Modulus and Permanent Deformation Tests. Each of the test results were subjected to an Analysis of Variance (ANOVA) tests. As a general conclusion, we obtained that T and t have a significant influence in the asphalt binder response as well as in the response of HMAs, mainly in terms of stiffness and adherence.

Keywords: mix temperature, mix time, modified asphalt, hot mix asphalt, stiffness.

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1. INTRODUCTION

Asphalt binder modification has been a widely studied and used technique all around the world. With said technique, mechanical, chemical and rheological properties of asphalt binders are modified with the purpose of attempting to improve their behavior in terms of stiffness, cohesion and adherence with mineral aggregates, as well as durability, a decrease of thermal susceptibility, aging resistance, moisture damage resistance, fatigue resistance and rutting resistance, among others. The materials that are mostly used in order to modify asphalt binders are thermoplastic polymers such as elastomer or plastomer type (e.g., plastic waste, low and high density polyethylene, crumb rubber, styrene-butadiene styrene – SBS, styrene-butadiene rubber – SBR, ethylene vinyl acetates – EVA, polyethylene), fibers (mineral fibers, Polyester, Polyacrylonitrile, glass and steel fibers) and nanomaterials (e.g., nanoclay, carbon nanotubes, nano silica, nano-TiO₂, nanoplatelets, nanofibers, graphene oxide), most of them which must be mixed in molten asphalt under high shear, at high temperatures, and on some occasions, using high mix times with the purpose of the modifier having a significant influence in the binder's behavior [1]. This type of modification is known as wet process. A summary of temperatures (T), and mix times (t), as well as shear velocity (SV) used by different researchers in order to modify asphalt binders in their studies is presented in Table 1. It is observed that for the most part, they use T between 150 and 180°C, t between 15 and 120 minutes and SV between 120 and 6900 rpm.

Table 1 T , t and SV used by different researchers

Reference	T (°C)	t (minutos)	SV (rpm)
[2]	180-185	30, 60, 90, 120, 150	4100, 5800
[3]	180-185	-	1000-1200
[4]	150	120	4400
[5]	190	60	3600
[6]	180	60	1000
[7]	160	40	1550
[8]	155	15	4000
[9]	160	70	2800-4000
[10]	160-170	30 - 60	120
[11]	160	120	4000
[12]	180	30	4500
[13]	170	50	4000
[14]	150	100	5000
[15]	170	100	500-5000
[16]	165	60	4000
[17]	160	120	4000
[18]	180	60	5500-6500
[19]	180-185	30	6900
[20]	160	45	1500
[21]	160	120	2000

Based on the reviewed literature, when an asphalt binder is modified through wet process, the result is generally a notable increase in its stiffness, expressed through the decrease of penetration and ductility, as well as an increase in softening point, viscosity, shear modulus (G^*) and grade performance (PG) at high service temperatures [22-27]. Likewise, in hot mix asphalt (HMA) that use these asphalt binders, there is also a general reporting of a notable increase in stiffness, expressed in the increase of resilient or dynamic modulus and in rutting resistance [16, 21]. However, none of the studies reviewed evaluated the effects of time and temperature to which asphalt binder is subjected when modified through a wet process. In other words, none of the studies discriminate what is the real contribution of the modifier in the asphalt binder rigidizing process, and what part of it is due to the processes of exposing the binder to elevated temperatures during prolonged time periods in the process of mixing.

Said studies have not clearly considered that on some occasions, the modification process could induce aging and degradative phenomena in both the asphalt and modifier [2]. During the mixing process, asphalt binder could undergo oxidation and volatilization processes associated with aging [28-29], which could be possible causes for the most part of the increased stiffness reported in reviewed studies. Yang et al. [30] for example, mention that the high temperatures used in mixing and compaction processes of modified asphalt mixes inevitably generate asphalt binder aging. Gamarra and Ossa [31] proposed a thermo-oxidative aging process for asphalt binders based merely on the exposure to high temperatures and mechanical agitation during several periods of time ($T=163^{\circ}\text{C}$, $SV=1800$ rpm, $t=2.5, 5.0, 7.5$ y 10 h). When subjecting asphalt binder to said conditions, they reported changes in its physical-chemical and micro-structural properties, which were associated to aging.

Given what was mentioned above, this study intends to generate a contribution by attempting to simulate and evaluate the influence of exposure to time and temperature to which asphalt binders are subjected when modified through a wet process. ANOVA variance analysis with F -test was conducted on the obtained results, attaining a reliability level of 95% ($F_{0.05}$). This had the purpose of evaluating if said results were statistically different or not. In said analysis, if $F > F_{0.05}$ means that changes in evaluated parameters are significant respect to control sample.

2. MATERIALS AND METHODS

2.1. Materials

The aggregate and asphalt cement AC 60-70 (penetration test ASTM D-5 in 0.1 mm; PG 58-22) were characterized following the [32] standard. The results of lab tests are presented in Tables 2 and 3, respectively. Both materials meet the requirement established by [32] for the manufacturing of HMAs.

Table 2 Aggregate test results

Test	Method	Recommended	Result
Specific gravity/fine aggregate absorption	AASHTO T 84-00	-	2.65/1.8%
Specific gravity/gross aggregate absorption	AASHTO T 85-91	-	2.54/1.5%
Sand equivalent test	AASHTO T 176-02	50 minimum	80%
Fractured particles (1 side)	ASTM D 5821-01	85 minimum	92%
Soundness of aggregate using magnesium sulphate	AASHTO T 104-99	18 maximum	9%
Plasticity Index	ASTM D4318-00	Not plastic	Not plastic
10% of fines (dry resistance)	DNER-ME 096-98	110 kN minimum	140 kN
Micro-Deval	AASHTO T327-05	25 maximum	17.6%
Abrasion in Los Angeles machine	AASHTO T 96-02	25 maximum	22.8%

Table 3 Test results performed on AC 60-70

Test	Method	Unit	Recommended	Result
Tests on the original asphalt binder				
Penetration (25°C, 100 g, 5 s)	ASTM D-5	0.1 mm	60-70	63.7
Penetration Index	NLT 181/88	-	-1.2/+0.6	-0.94
Specific gravity	ASTM D-70	-	-	1.012
Viscosity (60°C)	ASTM D-4402	Poises	1500 minimum	1550
Softening point	ASTM D36-95	°C	48-54	48.2
Ductility (25°C, 5cm/min)	ASTM D-113	cm	100 minimum	>105
Flash and fire points	ASTM D-92	°C	230 minimum	295
Tests on the residue after RTFOT (Rolling Thin Film Oven Test)				
Mass loss	ASTM D-2872	%	0.8 maximum	0.69
Penetration (25°C, 100 g, 5 s)	ASTM D-5	%	50 minimum	64.4

2.2. Simulation of Asphalt Binder Modification through Wet Process

Considering the information displayed in Table 1, AC 60-70 was subjected to a shear mixing with $T=160, 170, 180^{\circ}\text{C}$ and $t=40, 60, 90$ minutes, at 3000 rpm. This had the purpose of simulating the asphalt binder modification process (wet process). In regards to asphalt binders subjected to these temperatures and time exposures (9 in total= $3T \times 3t$) penetration tests were conducted - P (ASTM D-5), softening point - SP (ASTM D36-95), specific gravity - SG (ASTM D-70) and viscosity - V (ASTM D-4402).

2.3. Asphalt Control Mix Design

The control HMA-19 (19 mm is the maximum nominal size of particles) was designed based on Marshall Test (AASHTO T-245). The samples were compacted at 75 blows per face. The granular size distribution of the HMA-19 is presented in Table 4 in accordance to [32]. In order to obtain the optimum asphalt content (OAC), three samples were manufactured and tested for each percentage of asphalt binder in mass of 4.5%, 5.0%, 5.5%, and 6.0%. The mixing and compaction temperatures were 150°C (asphalt viscosity of 170 cP) and 140°C (asphalt viscosity of 280 cP), respectively. Additionally, the volumetric composition (air voids in volume- V_a , voids in mineral aggregate- VMA , and voids filled with asphalt- VFA) and the resistance under monotonic load (stability- S , flow- F , and S/F ratio) were obtained.

Table 4 Particle size distribution of HMA-19

Sieve	Sieve [mm]	Percent passing (%)	Percent retained (%)
3/4"	19.00	100.0	0.0
1/2"	12.50	87.5	12.5
3/8"	9.50	79.0	8.5
4	4.75	57.0	22.0
10	2.00	37.0	20.0
40	0.43	19.5	17.5
80	0.18	12.5	7.0
200	0.075	6.0	6.5
Bottom	-	-	6.0

The results of Marshall tests on the HMA-19 control samples are shown in Table 5. The OAC was 5.3%, based on the requirements established by [32] for HMA-19 mixtures.

Table 5 Marshall test results of control HMA-19

AC (%)	S (kN)	F (mm)	S/F (kN/mm)	V_a (%)	VMA (%)	VFA (%)
4.5	11.3	4.0	2.8	6.8	16.7	59.6
5.0	12.1	3.8	3.2	5.1	16.3	68.6
5.5	12.4	3.7	3.3	3.5	16.0	77.9
6.0	11.3	4.2	2.7	2.4	16.0	85.3

2.4. Marshall and Indirect Tensile Strength (ITS) Tests

Using the OAC of HMA-19 control mix, new Marshall samples were manufactured, but employing the 9 asphalt binders obtained in the wet modification simulation process. Three HMA-19 samples per each asphalt binder were manufactured to perform the Marshall test. The manufacture procedure was the same to control HMA-19. However, mixing and compaction temperature were 15°C upper (165°C and 155°C , respectively). These temperatures were chosen considering that in practice, mixes that used asphalt binders modified through wet process used higher mixing and compaction temperatures due to the increased viscosity of asphalt binder [33-35]. Neither the traditional criterion of viscosity was followed in order to determine said temperatures nor superior temperatures were used, given

that several authors advise so, in order not to notoriously degrade and age the asphalt binder and/or the modifier [36-38].

For the case of ITS test, initially six Marshall-type samples were manufactured for the control HMA-19 following ASTM D4867 standard and using the OAC, AC 60-70, and employing mixing and compaction temperatures of 150 °C and 140 °C, respectively. Three samples were tested under “unconditioned” method (dry condition - *ITS-D*) and other three under “conditioned” (vacuum saturated or wet - *ITS-W*). Unlike the ASTM D4867 standard process, samples were not manufactured to reach a void content of $7\pm1\%$ because researchers wanted to evaluate the resistance of the samples without changing the air void content achieved at OAC. Additionally, other six samples (three *ITS-D* and three *ITS-W*) were manufactured and tested using the OAC and employing the 9 asphalts obtained from the simulation of the wet modification process (54 samples). These samples were tested following the same procedure described above, in a similar way to control HMA-19. Just as for the Marshall test, in the case of this mixes, mixing and compaction temperature was 15°C upper (165°C and 155°C, respectively) compared to control HMA-19.

2.5. Cantabro Test

Cantabro test is a useful tool for providing durability indexes (generally including non-load-associated cracking and raveling or weathering) of dense graded asphalt mixes [39]. Three Marshall-type samples of control HMA-19 and mixtures using the 9 asphalts obtained from the simulation of the wet modification process were tested following NLT 352-86. The mixing and compaction temperature of Marshall and ITS tests were used. Each sample was tested at 20 °C in an Angeles abrasion drum for 500 revolutions without the charge of steel spheres. The sample mass loss *ML* was expressed as a percentage of the original sample mass and the final mass taken after the test.

2.6. Resilient modulus and permanent deformation tests

Resilient modulus tests (BS EN 12697-26) under temperatures of 10, 20 and 30°C and three loading frequencies (2.5, 5.0 and 10.0 Hz) were performed on the control HMA-19 and mixtures using 4 asphalts obtained from the simulation of the wet modification process ($T=160, 180^{\circ}\text{C}$, and $t=40, 90$ minutes).

Mixes with $T=170^{\circ}\text{C}$, $t=60$ minutes were discarded, given that the intent was to evaluate the lower and higher values of T and t in response to being under cyclic load. In addition, based on the results obtained with Marshall, ITS and Cantabro tests, it was evidenced that there is a proportional correlation of parameters evaluated with T and t . Each resilient modulus test was carried out on three samples. Permanent deformation tests (BS EN 12697-25) under repeated square-wave load with a stress of 100 kPa, test temperature of 40°C and 3600 load cycles (N) were performed. Since the resilient modulus test is not destructive, the same samples employed in resilient modulus tests were used for permanent deformation tests. Marshall samples were preloaded under 10 kPa during 10 min. After preloading time, a stress of 100 kPa was applied in 3600 pulses (N) with 1 s load, followed by a rest period of 1 s (frequency of 0.5 Hz). Both tests were performed using a Nottingham Asphalt Tester Machine.

3. RESULTS

3.1. Properties of Asphalt Binders

The results of P , SP , SG and V of asphalt binders that were subjected to the wet process modification simulation are displayed in Figures 1-4. The V of asphalts that were subjected to the simulation was normalized with regard to the value obtained for control AC 60-70 (VC). The observed result is that asphalt binder increases its stiffness (decrease of penetration and increase of softening point and viscosity) and specific gravity when elevating temperature and exposure time. Said increases were statistically significant according to ANOVA analysis ($F > F_{0.05}$). These changes are coherent to those reported by diverse researchers when asphalt binder ages due to oxidation [40-48].

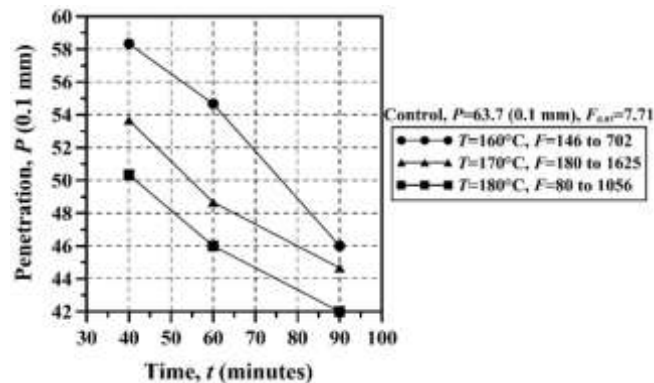


Figure 1. Evolution of penetration with temperature and exposure time

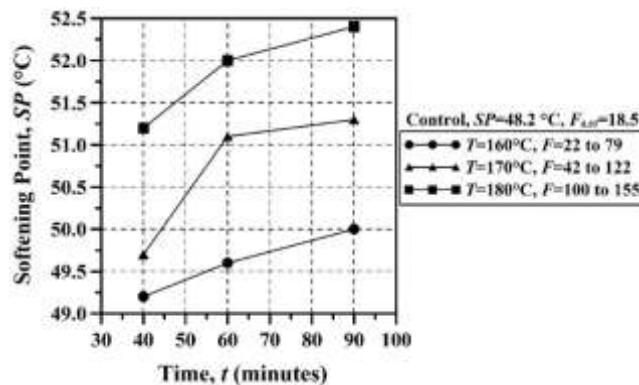


Figure 2. Evolution of softening point with temperature and exposure time

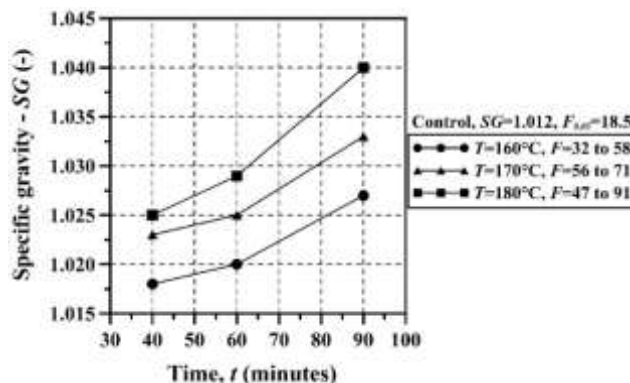


Figure 3. Evolution of specific gravity with temperature and exposure time

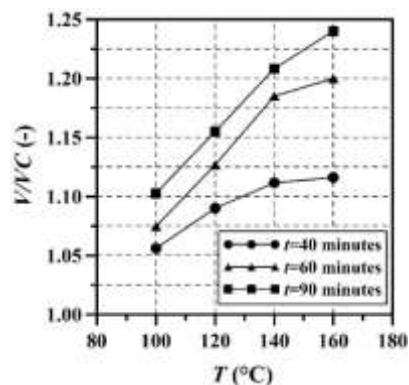


Figure 4. Evolution of V/VC ratio with temperature and exposure time

3.2. Marshall and ITS Test

Marshall Test results are presented in Figures 5 and 6. When comparing with the control mix and considering the ANOVA analysis, these report a statistically significant increase ($F > F_{0.05}$) in resistance under monotonic load (S/F ratio) when there is an increase in T and t (Figure 5). This happens despite that air voids increase slightly (Figure 6). This is mainly because of the increase of stiffness in asphalt binder as a process of oxidation it undergoes when subjected to elevated temperatures and exposure times. S/F increases between 21.7% y 29.5% when subjected to 160°C during 40 to 90 minutes. For the case of $T=170^{\circ}\text{C}$ and 180°C , said increases were of 30.6% to 40.3%, and 38.7% to 48.8%, respectively.

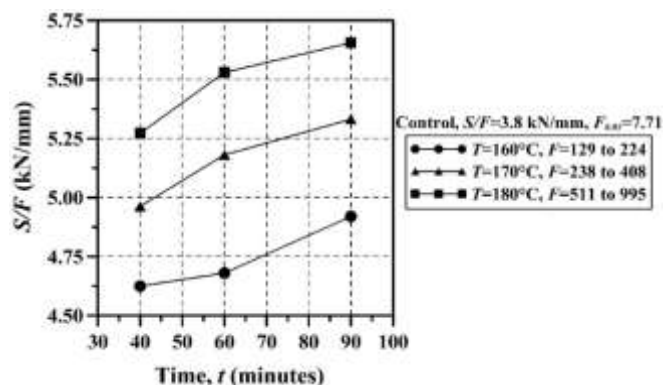


Figure 5. Evolution of S/F ratio with temperature and exposure time

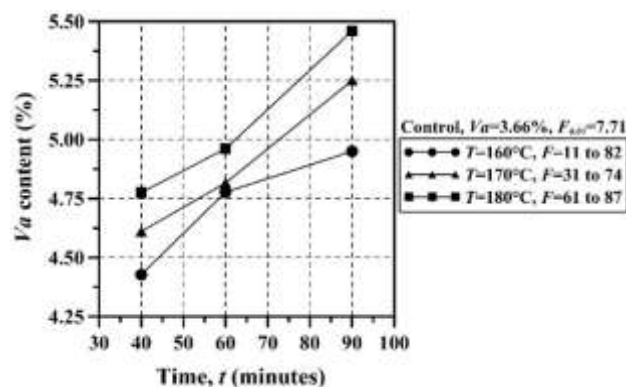


Figure 6. Evolution of V_a with temperature and exposure time

ITS test results are presented in Figure 7. These show that both $ITS-D$ as well as $ITS-W$ decrease with the increase of T and t . This is because of the loss of adherence between the

asphalt binder and aggregate as a product of when the latter undergoes oxidation when subjected to elevated temperatures and exposure times [21, 37, 49-50]. Compared to the control mix, only mixes manufactured with asphalt binder exposed to $T=160^{\circ}\text{C}$ and $t=40$ and 60 minutes presented a greater $ITS-D$, however this change was not statistically significant based on ANOVA analysis ($F < F_{0.05}$). For the case of $ITS-W$, there was only a slight increase observed (not statistically significant, $F < F_{0.05}$) when using asphalt binder subjected to $T=160^{\circ}\text{C}$ and $t=40$ minutes. For the rest of asphalt binders, HMA mixes displayed a lower $ITS-D$ and $ITS-W$ with relation to the control mix, and said changes were statistically significant for $T=170^{\circ}\text{C}$, 180°C and $t=60, 90$ minutes.

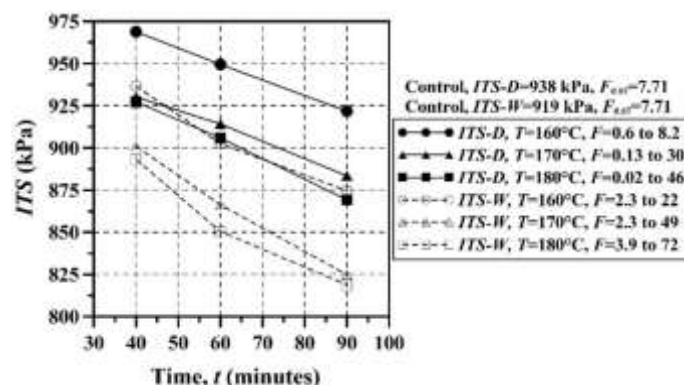


Figure 7. Evolution of ITS with temperature and exposure time

From a chemistry standpoint, a possible explanation of the change observed in asphalt binder characterization tests such as Marshall and ITS tests are described below. Asphalt binder is a colloidal dispersion of asphaltenes (A) in an oily matrix comprised of saturated (S), naphthenes, aromatics (Ar) and resins (R). S+Ar+R comprise the fraction named maltene [37, 51]. (A) increases the viscosity of asphalt binder and provide it with stiffness, (R) provides cementing characteristics and adherence and oils (S and Ar) provide manageability and aging protection. During the asphalt binder oxidation process, the reaction with atmospheric oxygen produces a molecular restructuring which decreases the fraction of maltenes and increases A. In other words, during the aging process, the proportion of maltenes/asphaltenes decreases, which finally results in a more rigid binder, that is brittle and that has less capacity of adhering to the aggregate [21, 37, 40, 50, 52]. During asphalt binder oxidation, the less polar fractions become polar fractions, increasing the latter [31, 37, 51, 53]. According to [54], adhesion is directly related to non-polar fractions of asphalt binder, while stiffness is related to polar fractions. On the other hand, oxidation increases LMS (large molecular size) parameter in asphalt binder molecules, and decreases the content of small molecules, increasing molecular weight [44, 51, 55-56], which could additionally explain the increase of SG and V.

3.3. Cantabro

Control mix displays a greater Cantabro wear resistance compared to mixes manufactured with asphalt binders exposed to different values of T and t . As T and t increase, Cantabro wear resistance in mixes decreases. This can be because of the fact that asphalt binders subjected to different T and t values are more rigid and brittle than control mix AC 60-70. Additionally, mixes with these asphalt binders display a greater V_a content than control mix, which allows the aggregate to separate more easily during the test. Based on ANOVA analysis, when comparing the results of both mixes, changes reported are statistically significant ($F > F_{0.05}$).

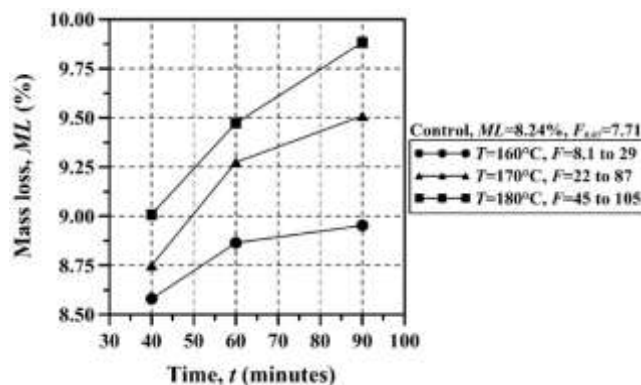


Figure 8. Evolution of ML with temperature and time exposure

3.4. Resilient Modulus and Permanent Deformation

Resilient modulus and permanent deformation test results are showed in Figures 9 and 10, respectively. These results were coherent to those obtained in the Marshall test. The increase of asphalt binder stiffness when subjected to different T and t , generated an increase in resilient modulus (Figure 9) and permanent deformation resistance (Figure 10). Said increases were not statistically significant $F > F_{0.05}$ merely for the case of $T=160^\circ\text{C}$ and $t=40$ minutes. ANOVA analysis for permanent deformation test was calculated at $N=3600$ (final load cycle).

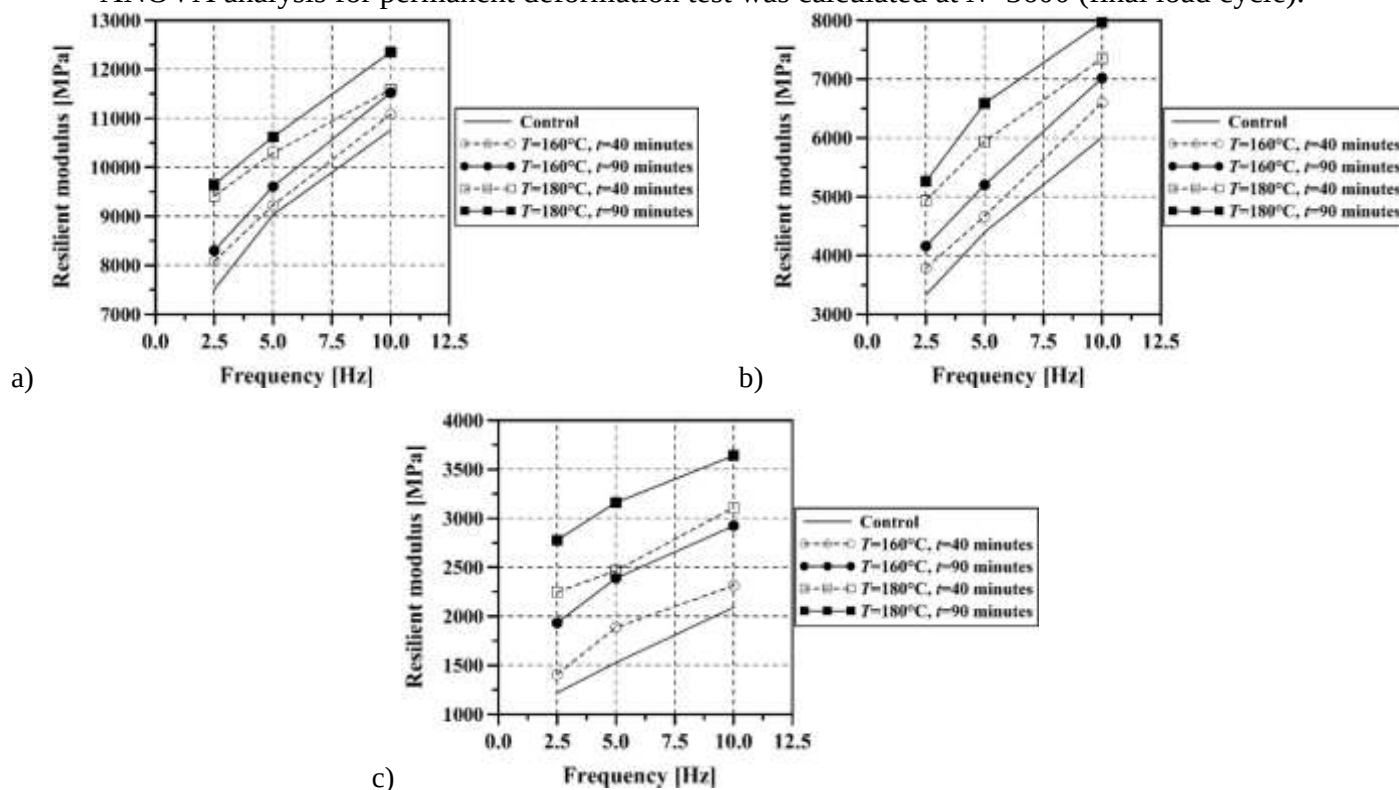


Figure 9. Resilient modulus at a) 10°C, b) 20°C and c) 30°C

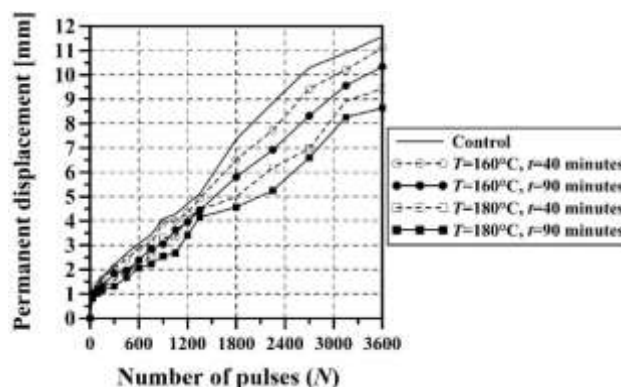


Figure 10. Permanent deformation results

4. CONCLUSIONS

This study simulated and evaluated within a laboratory, the influence of temperature and exposure time to which asphalt binders are subjected when modified through wet process. We want to make clear and highlight that the conclusions reported may only be considered for the AC 60-70, given that all asphalt binders display different resistances to different oxidation processes. Based on the results obtained, it is concluded that the asphalt binder underwent a notable and statistically significant increase in its stiffness (decrease of penetration, softening point and viscosity) when exposed for 40 to 90 minutes to temperatures between 160° and 180°C. This is mainly due to the asphalt's oxidation. This increase in stiffness and asphalt binder oxidation had a significant influence from a statistical standpoint (in most cases, and especially when it was exposed during 40 to 90 minutes to temperatures between 170° and 180°C), increasing resistance under monotonic load, stiffness under cyclic load and resistance to permanent deformation in mixes. Based on Cantabro and ITS tests, it also influenced in decreasing the adherence between the aggregate and asphalt binder. All of this leads to thinking that in future studies of asphalt modification through wet process, the increases of stiffness reported both for the asphalt binder and for hot mix asphalt must discriminate the influence of the modifier separately to the one of temperatures and exposure times during mixing (asphalt-modifier). Likewise, said studies must consider the influence of temperatures and exposure times on the possible loss of adherence as a product of asphalt binder oxidation.

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