ENGLISH VERSION..........

Evaluation of a rejuvenator as an additive in asphalt

Valoración de un rejuvenecedor como aditivo en el asfalto

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Fecha de Recepción: 15/09/2020
Fecha de Aceptación: 05/01/2021
PAG 222-232

Abstract

Ecuadorian asphalt has the particularity of being prone to premature aging; therefore, the asphalt pavements have insufficient durability. The objective of this experimental work is to assess the effect that a modification with the rejuvenating agent Sylvaroad RP-1000 produces on the properties of AC 20 asphalt from the Esmeraldas Refinery, as well as evaluating its impact on the quality of a typical asphalt mix, without reclaimed asphalt pavement, prepared with modified and unmodified asphalt. The asphalt used is also classified as PG 64-22 and when mixed with 2.5% by weight of the rejuvenator, the grade changed to PG 58-28. Additional studies were carried out using the ΔTc, Glover-Rowe parameters and the corresponding transition temperatures, showing improvements in the results. At that point, a dense mix with 5.5% asphalt was designed. Stiffness modulus, cyclic compression and fatigue modulus tests were applied with the NAT equipment to the specimens. When using modified asphalt, the fatigue behavior improved remarkably. Additionally, an aging process in oven at 85 ° C was applied to both mixtures, measuring the stiffness modulus and fatigue at 8 days, which demonstrated an adequate behavior only in the mixture made with rejuvenator.

Keywords: *Asphalt, asphalt mix, rejuvenating, aging, NAT*

Resumen

El asfalto ecuatoriano tiene la particularidad de ser proclive al envejecimiento prematuro incidiendo en que los pavimentos asfálticos presenten una insuficiente durabilidad. El objetivo de este trabajo experimental es valorar el efecto que produce en las propiedades del asfalto AC 20, procedente de la Refinería de Esmeraldas, su modificación con el agente rejuvenecedor Sylvaroad RP-1000; así como evaluar su incidencia en la calidad de una mezcla asfáltica típica, sin material fresado, preparada con asfalto sin modificar y modificado con rejuvenecedor. El asfalto utilizado clasifica como PG 64-22 y al ser mezclado con 2.5% en peso del rejuvenecedor, su grado cambió a PG 58-28. Se realizaron estudios adicionales empleando los parámetros ΔTc, Glover-Rowe y las temperaturas de transición, evidenciándose mejoras en los resultados. Luego se diseñó una mezcla densa con 5.5% de asfalto; a las briquetas confeccionadas se le aplicaron pruebas de módulo de rigidez, compresión cíclica y fatiga con el equipo NAT. Al emplear asfalto modificado el comportamiento a la fatiga mejoró notablemente. Adicionalmente, se aplicó un proceso de envejecimiento en horno a 85°C a ambas mezclas, valorándose el módulo y la fatiga a los 8 días, que demostraron un buen comportamiento solo en la mezcla con rejuvenecedor.

Palabras clave: Asfalto, mezcla asfáltica, rejuvenecedores, envejecimiento, NAT

1. Introduction

For economic and environmental reasons, the use of Reclaimed Asphalt Pavement (RAP) has been increasing in many countries; however, the use of high percentages of RAP in a recycled mix will make it less workable, more difficult to compact, and more prone to cracking and weathering (Li et al., 2008), (Mogawer et al., 2012). One solution to these problems is the use of softer base asphalts or the use of recycling or rejuvenating agents. These agents can restore the rheological characteristics of the recycled binders and their mixture, with the base binder to the desired performance requirements (Arámbula et al., 2018).

It is known that flexible pavements in Ecuador present some types of deterioration within a few years of being constructed or rehabilitated. Fatigue or block cracking are the most common types of damage. This may be attributed, in part, to the fact that most of the asphalt used comes from the Esmeraldas Refinery, which has the particularity of being prone to premature aging and therefore to low durability. This can be verified by viscosity classification, where non-compliance with quality requirements in the residue of the rotary thin film test or RTFOT is frequent (Vila et al., 2017). Therefore, in works developed at the Road Laboratory of the Catholic University of Santiago de Guayaquil (UCSG), rejuvenating additives have been directly used with Ecuadorian asphalt cement to try to decrease the intensity of its aging, also to assess its incidence on the behavior of the mixtures (Icaza and Mera, 2018).

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The objective of the present experimental work is to evaluate the effect on the properties of AC 20 asphalt, coming from the Esmeraldas Refinery, its modification with the rejuvenating agent Sylvaroad RP-1000; as well as to evaluate its incidence on the quality of a typical asphalt mix, without milled material, prepared with unmodified asphalt and modified with rejuvenator.

2. Binders used

2.1 Classification by viscosity

The base binder used was asphalt cement from Esmeraldas, which was modified with the addition of the indicated rejuvenating agent. For the dosing of the rejuvenating agent, the first step was to ensure compliance with the RTFOT residue requirements. Subsequently, for the mixtures, it was verified that this dosage was not excessive, because if it were, it would produce a very soft binder that would have a negative impact on the rutting resistance of the mixture. Conversely, a very low dosage could help reduce the binder's brittleness but without a pronounced effect on the improvement of fatigue resistance.

The modification employed involved mixing 97.5% by weight of AC 20 base asphalt with 2.5% by weight of rejuvenator. (Table 1) shows the test results for viscosity classification, according to the Ecuadorian standard (INEN 2515, 2010).

Table 1. *Test results according to viscosity classification*

Test	AC -20 MIN	AC -20 MAX	Original Asphalt	Asphalt + Rejuvenator
Viscosity, 60°C (Pa.s)	160	240	235	100
Viscosity, 135°C (mm 2/s)	300	-	345	277
Flash point (°C)	232		290	305
Specific gravity 25ºC/25ºC	-	-	1013	1011
Softening Point ºC			49.5	47.0
Penetrarion 25°, 100g, 5s (0.1 mm)			68	100
Penetration index	-1.5	+1.0	-0.6	-0.2
Tests on residue from rolling thin-film oven test:				
Viscosity, 60°C (Pa.s)	-	800	1315	716
Mass Change (%w/w)	-	1.0	0,049	0,063
Ductility , 25°C, 5 cm/min (cm)	50	-	25	55,5

In the base asphalt, although the viscosity test on the original bitumen at 60°C, would allow the sample to be identified as AC 20. The viscosity and ductility requirements are not met in the RTFO residue tests, which is a fairly widespread problem in this asphalt and which is precisely the reason for this research. When modified with rejuvenator, the residue requirements can be met, but the viscosity of the original asphalt is then not met. This behavior should be considered when adding rejuvenators in the manner proposed in this study.

2.2 Classification by performance grades

(Table 2) shows the values obtained in the Superpave tests according to INEN 3030-2017. Theoretically a PG 64 -22 asphalt could satisfy the temperature ranges of all our geographical regions, obtained according to the Superpave criteria and shown in (Table 3) (Vila et al., 2017). However, in our roads there is a poor performance in the face of intermediate temperatures, identified with fatigue cracking. It is widely known that the control parameter for these temperatures employed by Superpave, i.e., "G*.Senδ", correlates poorly with fatigue.

On the other hand, as pavements age they begin to exhibit cracking and aggregate spalling. Although the stresses produced by traffic increase destructions, the evolution of rheological properties of aged asphalt can be damaging enough by itself to cause block cracking due to stresses caused by thermal gradients (King et al., 2012).

This criteria is considered valid for the ranges of fluctuating daily temperatures throughout the year in Ecuador, with more significant gradients in the Sierra region, where precisely these types of damage: block cracking and spalling or "peeling", are more frequent and intense. With the addition of a rejuvenator, as shown in (Table 2), a PG 58-28 is obtained, which can undoubtedly help to reduce these negative effects, with greater possibilities in the Sierra region due to the high temperatures reached.

Table 2. *Results of the tests according to PG classification*

Test		**Range**	**Original Asphalt**	**Asphalt + Rejuvenator**
ORIGINAL BINDDER				
Flash Point Temp, °C		230 mín	290	305
Viscosidad, Pa.s	135°C	3 max	0.32	0.26
Dynamic Shear (G*/sinδ, 10 rad/s), kPa	46°C	1,0 mín	14.50	8.81
	52°C		6.25	3.49
	58°C		2.68	1.42
	64°C		1.19	0.63
	70°C		0.55	xx
RTFOT RESIDUE (163°C, 85 min)				
Mass loss, %		1,0 max	-0.049	-0.063
Dynamic Shear (G*/sinδ, 10 rad/s), kPa	46°C	2,2 mín	47.70	22.90
	52°C		21.30	9.74
	58°C		9.70	4.37
	64°C		4.59	2.01
	70°C		2.21	xx
	76°C		1.10	xx
PAV RESIDUE (100°C, 20 hr)				
Dynamic Shear (G*/sinδ, 10 rad/s), kPa	31°C	5000 max	989	xx
	28°C		1390	xx
	25°C		1950	1150
	22°C		2750	1620
	19°C		3800	2270
	16°C		5140	3120
	13°C		xx	4230
	10°C		xx	5720
Stiffness, MPa (60 s)	−24°C	300 max	381	452
m value		0,300 mín	0.268	0.263
Stiffness, MPa (60 s)	−18°C	300 max	197	127
m value		0,300 mín	0.294	0.327
Stiffness, MPa (60 s)	−12°C	300 max	87	63
m value		0,300 mín	0.345	0.355
PERFORMANCE GRADE		**--**	**64 -22**	**58 -28**

Table 3. *Asphalt pavement temperature ranges according to Superpave*

REGIONS	COAST	SIERRA	AMAZONIA
Maximum temperatures, °C	54 a 58	42 a 52	52 a 57
Minimum temperatures, °C	8 a 16	-3 a 8	7 a 15
Intermediate temperatures, °C	35 a 41	24 a 34	34 40

3. Additional studies on binders

A few years ago, an expert from the Asphalt Institute expressed the following opinion:

The next step in the evolution of asphalt technology is to set a parameter for the control of binders at intermediate temperatures, i.e., related to deteriorations associated with fatigue cracking and durability. A group of new tests and parameters have been proposed: Linear Amplitude Sweep (LAS), R-value, Glover-Rowe, ΔTc and Double Edge Notched Tension (DENT) to name a few, but asphalt scholars have yet to agree on the one they believe best relates to cracking at intermediate temperatures (Anderson, 2016a).

Therefore, it was deemed appropriate to perform some additional tests within our scope.

3.1 Parameter Δ Tc

This parameter is determined by performing additional calculations with the data obtained from the beam flexural beam rheometer (BBR) test. "ΔTc" is calculated according to (Equation 1):

$$\Delta Tc = Tc,m - Tc,S \qquad (1)$$

Donde Tc,m es la temperatura crítica correspondiente al valor de la pendiente "m" (pendiente de relajamiento de Where Tc,m is the critical temperature corresponding to the value of slope "m" (stiffness relaxation slope) equal to 0.300 with loading time of 60 seconds and Tc,S is the critical temperature corresponding to the value of stiffness equal to 300 MPa obtained with loading time of 60 seconds.

Although the "ΔTc" is obtained at low temperatures, this parameter is an indicator of binder quality and durability that can correlate very well with other parameters linked to intermediate temperatures (Anderson et al., 2011). As the asphalt ages, the value of "ΔTc" increases, indicating what is considered a loss in relaxation properties. The values related to the onset of cracking and the presence of cracks are 2.5 and 5.0°C respectively. The calculated values are presented in (Table 4).

It is highlighted that in some works (Anderson, 2016b) the parameter "ΔTc" is designated as the difference between "Tc,S" and "Tc,m", which could create some confusion in the interpretation of results due to the change of sign it produces.

3.2 Glover - Rowe criterion

This criterion, which can be represented in a Black's diagram, responds to (Equation 2):

$$G'/(\eta'/G') = G^*. ((\cos \delta)^2 / \sin \delta). \omega \qquad (2)$$

Provided that the test frequency (ω) the variables: complex modulus (G) and phase angle (δ) are known, a damage curve can be created in Black's Diagram. The Glover - Rowe parameter is obtained from frequency swept DSR tests at 15°C and 0.005 rad/s (King et al, 2012). Values below 180 kPa suggest that no block cracking will exist, between 180 and 600 kPa that cracks are developing and above 600 kPa that cracking already exists. The results are summarized in (Table 4).*

3.3 Viscoelastic transition temperature

It is known that in the master curves of an asphalt the crossover frequency represents a balance between the elastic component (G') of the complex modulus and the viscous component (G''), and a transition from a more *solid-like state to a more fluid-like state given the viscoelastic behavior. In recent years, research has been conducted (Garcia et al., 2018) on the crossover temperature at a constant test frequency, also referred to as crossover temperature, viscoelastic transition temperature (T_{VET}), melting temperature or simply $T\delta=45°$.*

In the previously referred work and from correlations with the Glover - Rowe parameter, the behavior of binders remaining below the warning threshold (32°C), after 20 h of aging in PAV equipment, and below the cracking limit (45°C) after 40 h of aging in PAV, tested at 10 rad/s, is considered satisfactory. (Table 4) shows the transition temperatures obtained according to these criteria.

***Table 4.** Temperature ranges*

Criterion	Original Asphalt		Asphalt + Rejuvenator	
	RTFOT	PAV	RTFOT	PAV
Δ Tc, ºC	XXX	4.06	XXX	0.66
Glover - Rowe, Kpa	19	121	8	63
Crossover Temperature, ºC	31.2	36.9	14.1	32.0

(Table 4) also shows that for the PAV residue in the base asphalt, both the ΔTc value and the transition temperature are in the cracking zone according to the criteria used; this situation is not reflected by the Glover-Rowe parameter. On the other hand, the addition of the rejuvenator significantly improved the behavior evaluated with these criteria.

4. Asphalt mix characteristics

For this research, a dense-grained mix with a maximum nominal size of 12.5 mm was used as a reference, using good quality mineral aggregates of basaltic origin. (Table 5) shows the characteristics of the aggregates and (Figure 1) shows the combined granulometry used.

A Marshall design was carried out to determine an optimum asphalt content of 5.5% (by weight). With this percentage, briquettes were made for each type of asphalt (without and with rejuvenator). Before the compaction process, the mixes were placed in an oven at 135°C for 2 hours.

***Table 5.** Characteristics of mineral aggregates*

Parameter	Norm	Result	Requirement
Specific gravity	INEN 857	2.871	-
Absorption	INEN 857	0.97	-
Abrasion	INEN 860	9.9	<40%
Soundness Of Aggregates by Sodium Sulfate	INEN 863	0.81	<12%
Sand Equivalent value	ASTM D 2419	67	>50%
Flat and extended particles	ASTM D 4791	5	<10%

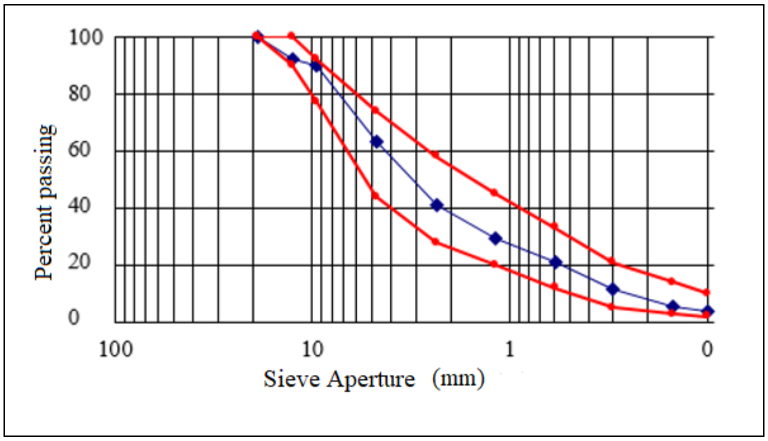

Figure 1. *Combined grain size of the stone skeleton.*

The comparative tests were performed with a Nottingham Asphalt Mix Tester (NAT) equipment: stiffness modulus (EN 12697-26:2012), permanent deformation test under uniaxial cyclic compression (creep) and fatigue test. The last two tests were performed under the equipment manufacturer's methodologies (Cooper, 2002), which partially coincide with the corresponding European standards.

It should be noted that for the tests to the performance tests with the NAT equipment, the quality requirements developed by the UCSG Road Laboratory were considered, and are currently being tested by the Ministry of Transport and Public Works (MTOP) of Ecuador (Vila, 2017).

5. Results obtained

5.1 Rigidity Module

Its determination was performed at the temperature of 20°C, using a controlled deformation level of 5 microns and sinusoidal waves with time interval between the beginning of the load pulse and the point at which the load is maximum, of 0.12 seconds. The results of these tests are presented in (Table 6).

Table 6. Stiffness modulus results

Specimen	Stiffness Modulus at 25º, MPa	
	Original Asphalt	Asphalt + Rejuvenator
1	3218	**2704**
2	3062	2487
3	2959	2530
Average	3080	2574
Standard Deviation	130	115
Coefficient of Variation	4.2	4.5

According to the proposed requirements mentioned above, the qualification of asphalt mixtures for wearing courses according to the modulus of rigidity is:

Satisfactory mixes: 3000 to 4000 MPa,

Tolerable mixes: 2500 to 3000 Mpa and 4000 to 5000 Mpa, and

Inadequate mixtures: less than 2500 Mpa or greater than 5000 Mpa.

Analyzing the average values, it can be observed that the mix with an asphalt base is the one with the highest value (3080 Mpa), which qualifies as satisfactory. It is observed that the inclusion of the rejuvenator decreases the value of the stiffness module (2574 Mpa), although it still qualifies as a tolerable mix.

5.2 Permanent deformation under cyclic compression

In this test a loading cycle consists of applying a stress for 1 second followed by 1 second of rest, with quadratic waves. The test was performed at a temperature of 40°C, with a load magnitude of 100 Kpa, measuring the vertical deformations caused by 3600 repetitions of this load. The results are shown in (Table 7).

Table 7. *Results of the uniaxial cyclic compression tests*

Specimen	Uniaxial cyclic compression test at 40º C, %	
	Original Asphalt	Asphalt + Rejuvenator
1	0.739	**0.989**
2	0.916	0.970
3	1.027	0.759
Average	0.894	0.906
Standard Deviation	0.145	0.128
Coefficient of Variation	16.2	14.1

According to the proposed requirements, the qualification of asphalt mixtures, based on the percentage of deformation in the uniaxial cyclic compression test, is:

Satisfactory mixes: deformations less than or equal to 1%, and

Inadequate mixes: deformations greater than 1%.

Analyzing the average values of the briquettes, it was observed that the two mixtures have values below 1% deformation, which indicates that satisfactory behavior is expected in the mixtures with respect to plastic deformation. However, the addition of a rejuvenator always tends to increase the deformations, in this case in a very small range.

5.3 Indirect tensile fatigue under controlled strain

In order to perform the fatigue test, it is necessary to previously determine the module of rigidity (Sm), using the same stress (σ) with which the aforementioned test will be performed. With the fatigue test, the number of load applications required to reach breakage, or a maximum deformation of 5 mm is obtained. The loading time is 120 milliseconds and the temperature for the study is 20°C. Considering the Poisson's coefficient (μ) with a value of 0.35, it is possible to calculate the initial tensile unit strain (ε) according to (Equation 3):

$$\varepsilon(\boldsymbol{mm/mm}) = \frac{\boldsymbol{\sigma(kPa)*(1+3\mu)}}{\boldsymbol{Sm\ (kPa)}} \qquad \textit{(3)}$$

(Figure 2) represents the obtained adjustment lines. It is important to mention that according to the requirements referred to for the fatigue case, it is noticed that if the points for the analyzed mixture fall below the line corresponding to the percentile (dotted black line), the estimated behavior will be inadequate. If the points fall above the average line (dashed black line), the estimated performance will be satisfactory. Between the two lines the behavior can be considered as tolerable.

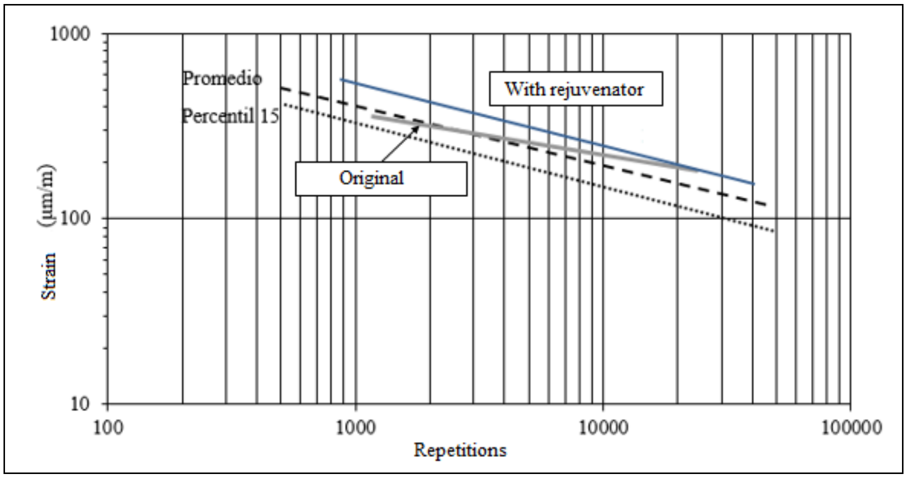

Figure 2. *Fatigue tests results.*

As shown in (Figure 2), the trend line of the asphalt with rejuvenator is located above the average line, which indicates an adequate fatigue behavior according to the proposed criteria. For the base asphalt (without rejuvenator) the behavior is between tolerable and adequate.

6. Study of laboratory aged mixtures

From the previous analyses, it can be stated that both mixtures present acceptable behaviors, which is a very interesting result. However, it must be considered that in all the tests carried out, the mixtures have not undergone any long-term aging process. For this reason and taking into account that fatigue behavior, is one of the most important in asphalt pavements in Ecuador, it was decided to make sets of briquettes with the mixtures studied to evaluate their performance after an aging process in the laboratory.

This evaluation consisted of keeping the briquettes for 8 days in an oven at 85°C to develop long-term aging, which can be estimated at approximately more than 9 years or more than 18 years, depending on whether the zone is dry - with freeze or wet - without freeze (Bell et al., 1994). These estimates were obtained for some North American asphalt cements placed in different regions of that country, so they should be considered only as references. To consider the effect of aging with days in the kiln, stiffness modules were measured every two days on briquettes of both combinations during a 10-day period. The results obtained can be seen in (Table 8) and (Figure 3). Fatigue tests were also performed at 4 and 8 days.

Table 8. Modules of stiffness in briquettes with different aging times.

Mix	Specimen	Stiffness modulus (MPa)				
		2 days	4 days	6 days	8 days	10 days
Original Asphalt	1	6071	6392	6485	7053	7203
	2	5571	5828	6537	7021	6736
	3	5126	5628	5997	6627	6856
	Avarage	5589	5949	6340	6900	6932
	Standard dev.	473	396	298	237	243
	Coef. of Variation	8.5	6.7	4.7	3.4	3.5
Asphalt + Rejuvenator	1	4470	5508	5615	5530	5648
	2	4250	4673	4789	4873	5064
	3	4385	4551	4612	4660	4682
	Avarage	4368	4911	5005	5021	5131
	Standard dev.	111	521	535	453	487
	Coef. of Variation	2.5	10.6	10.7	9.0	9.5

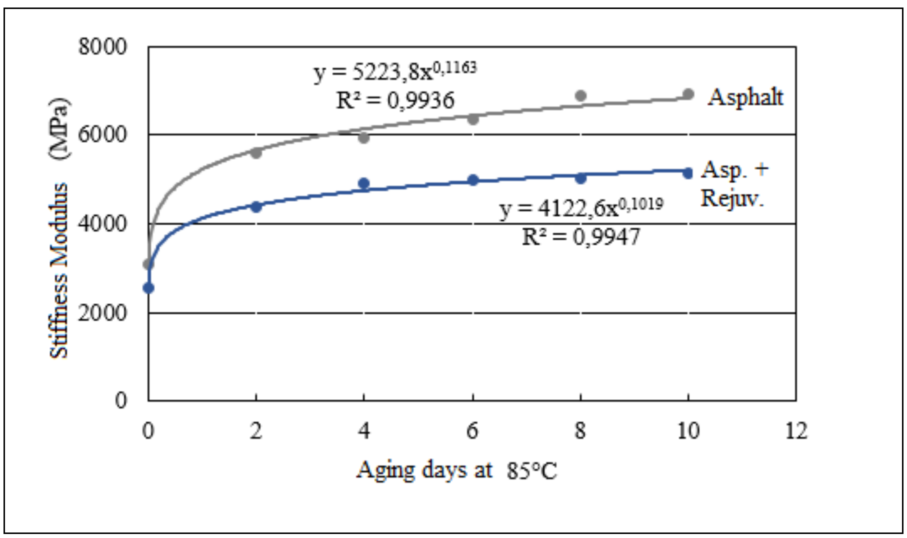

Figure 3. *Variation of stiffness modulus with the number of days in the oven.*

As shown in (Figure 3), the average stiffness modulus of the mixes corresponding to the base asphalt tends to stabilize at a value close to 7000 MPa after 8 days, while the average modulus of the mix with rejuvenator stabilizes at a value of approximately 5000 MPa after 4 days. In other words, when approaching a maximum stiffness in the mix due to aging, the use of the rejuvenator at a dosage of 2.5% by weight allows a decrease in the modulus of 2000 MPa, which represents 28.6%, which should have a positive impact on fatigue behavior.

The results of the mix with asphalt without additives, shown in (Figure 3), are lower than those obtained in a previous study (Cedeño, 2015), where for the mix used, modules close to 9000 MPa were obtained at 8 days. This difference in values should consider, among others, the quality of the mineral aggregates used.

The results of the fatigue tests performed on the two mixes under non-aging conditions and after 8 days of aging are shown in (Figure 4). As can be seen, the mix with rejuvenator has a much better fatigue behavior in both conditions, moving from a "satisfactory" to a "tolerable" position, while the mix without rejuvenator ends the 8 days in an "inadequate" state.

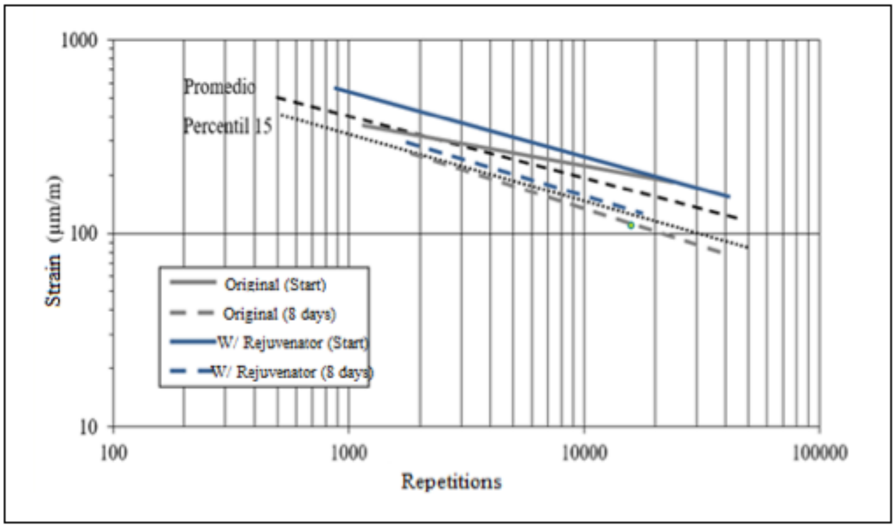

Figure 4. *Fatigue test results with aged mixes*

7. Conclusions

The rejuvenator used as an additive for Ecuadorian asphalt AC 20 allows meeting the corresponding requirements to the residue of the RTFO test, which is technically very convenient to counteract the usual failures that occur in our asphalt pavements. On the other hand, additional studies using the parameters ΔTc, Glover - Rowe and the transition temperature, all associated to the behavior before intermediate temperatures, show a great potential for its application. For the PAV residue in the base asphalt, both the ΔTc value and the transition temperature are in the cracking zone according to the criteria used, which allows us to identify this problem in our asphalt. The addition of the rejuvenator significantly improved the behavior evaluated with these criteria.

The results of the tests on the asphalt mixes, without aging, show that the mixes made with binder without and with rejuvenator meet the performance requirements performed with the NAT equipment. However, in the tests on specimens with long-term aging (8 days in an oven at 85°C), it was observed that the effect of such aging had a stronger impact on the mix with the base asphalt without rejuvenator, in which the stiffness modulus stabilized at approximately 7000 MPa, while the modulus obtained with rejuvenator stabilized at a value of 5000 MPa. This denotes that the addition of such a product would allow obtaining a less stiff mixture over time.

When analyzing the fatigue laws for the two mixtures, at the end of the indicated aging process, it can be concluded that the rejuvenator improves the performance of the mixture because the corresponding adjustment line is above the 15th percentile line, qualifying it as a "tolerable" mixture. On the other hand, the adjustment line for the mix with asphalt without rejuvenator is below the aforementioned limit, reaching "inadequate" levels.

In general, although the direct application of rejuvenators to asphalt cement produces a decrease in its original viscosity, it is possible to produce asphalt mixtures that perform satisfactorily in our climatic conditions. Especially the fatigue behavior, which is our major problem, is significantly improved.

8. Recomendations

Promote a particular technical specification to facilitate the use of the rejuvenator studied, as well as to investigate its behavior in asphalt mixtures produced with aggregates of different mineralogical compositions.

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