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ENGLISH VERSION…..………...

Ecuadorian asphalt aging in laboratory and field. Relationships and analysis

El envejecimiento del asfalto ecuatoriano en laboratorio y obra. Relaciones y análisis

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Abstract
The aging of asphalt binders has a great impact on the behavior of pavement. The specifications for performance grade include tests on short-term (RTFOT) and long-term (PAV) aged residues. However, aging is greater at the top of wearing course than deeper zones, long-term tests do not correlate accurately with field aging and in the latter, the incidence of local temperature is not considered accurately. The objective is to relate the aging of Ecuadorian asphalt at the laboratory level, applying different conditioning times in the PAV, with the field aging from asphalts recovered from the top of pavements surface in selected roads of the Andean and Coastal regions, with different construction times and cumulated degree-days (CDD), using certain rheological parameters. As a result, in the Ecuadorian asphalt, the RTFOT residue is equivalent to field aging of 1.1 years in the Coastal region and 1.7 years in the Andean region. On the other hand, the usual PAV of 20 hours corresponds to field aging of 5.8 years for the Coastal region and 9.3 years for the Andean region. The progressive evolution of damage to our roads, without considering traffic, corresponds to conditioning times in the PAV between 13.0 and 26.6 hours.

Keywords: *Asphalt; aging; cumulated degree-days; rheological parameters; damage.*

Resumen
El envejecimiento del asfalto impacta el comportamiento de los pavimentos. Las especificaciones por grado de desempeño incluyen ensayos a residuos envejecidos a corto plazo (RTFOT) y a largo plazo (PAV). Sin embargo, el envejecimiento es mayor en la parte superficial de la capa de rodadura, las pruebas a largo plazo no se correlacionan con precisión con el envejecimiento en el campo y la incidencia de la temperatura local tampoco se considera adecuadamente. El objetivo es relacionar el envejecimiento del asfalto ecuatoriano a largo plazo a nivel de laboratorio, aplicando en el PAV diferentes tiempos de prueba, con el envejecimiento en obra a partir de asfaltos recuperados de capas asfálticas en las regiones andina y costera del Ecuador, con diferentes tiempos de colocación y grados-día acumulados, empleando ciertos parámetros reológicos. Como resultados, el residuo del RTFOT equivale a un envejecimiento en obra de 1.1 años en la región costera y de 1.7 años en la andina. Por otra parte, al PAV de 20 horas le corresponden tiempos de envejecimiento de 5.8 años en la costera y 9.3 años en la andina. La evolución de los deterioros en nuestras vías, corresponden a tiempos en el PAV entre 13,0 y 26,6 horas respectivamente.

Palabras clave: Asfalto; envejecimiento; grados-día acumulados; parámetros reológicos; deterioros.

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1. Introducción

The aging of asphalt binders is one of the determining factors in the performance of pavements and in their useful life, producing changes in the chemical and physical properties of the binder that make it harder and more brittle. This situation affects the durability of the asphalt mix, causing loss of flexibility at medium and low temperatures. Raveling and surface-initiated cracking are the primary distresses. Aging can be classified in two stages: the first stage or short-term aging occurs in the presence of high temperatures, during mixing, storage, transport, placement, and compaction; the second stage or long-term aging takes place at ambient temperature, during the pavement service life.

The reactions that are generated in bitumen due to aging are dependent on its chemical composition, and an increase in the amount of oxygenated functional groups, such as carbonyls and sulfoxides, carbon-carbon double bonds, and aromaticity, is expected to occur (Mouillet et al., 2008); (Yang et al., 2015). For this reason, different asphalts may have different increases in their viscosity with the same aging time and temperature (Petersen, et al., 1993).

Since the aging of the asphalt binder significantly influences the performance of asphalt mixtures, the performance grading (PG) system, AASHTO M 320, includes criteria for tests on residues obtained by procedures that simulate short- and long-term aging. For the short term, the Rolling Thin Film Oven (RTFO) procedure, AASHTO T 240, is used, and for the long term, the Pressure Aging Vessel (PAV) method, AASHTO R 28. Both technologies consider the fact that thermal oxidation is the main factor in the field aging.

Although in asphalt samples aged in RTFOT and then exposed in the PAV an equivalent aging between 5 and 10 years is expected in the pavement in service (Bahia and Anderson, 1994); (Brown et al., 2009), several works have tried to deepen into these criteria. In Costa Rica, a greater increase in oxygenated compounds that provide rigidity to the bitumen molecules (such as sulfoxides and carbonyls) was observed for outdoor asphalt samples compared to those processed in the PAV. In other words, the PAV is not capable of completely simulating the oxidation of bitumen (Villegas et al., 2018).

Despite great advances in understanding the evolution of asphalt aging and simulating field aging under laboratory conditions, there are still several issues that affect and are not well understood, such as the selective absorption of asphalt by the mineral aggregates, the variation of aging through the thickness of the asphalt layer on site, and certain characteristics of the mixture such as the percentage of voids and the thickness of the binder film. In studies on aged asphalt, empirical physical properties or rheological parameters are usually evaluated, obtained by means of viscometers, dynamic shear rheometers (DSR) or bending beam rheometers (BBR).

(Mallick and Brown, 2004) evaluated the use of the RTFOT and the PAV as aging tests. They compared the behavior of asphalts aged in the laboratory and similar ones used in mixtures placed in 6 test sections in 5 North American states. They used comparison measurements with DSR and BBR equipment, reaching the conclusion that the RTFOT and PAV tests allow characterizing aging in the short and long term, respectively. However, since the sections were evaluated between 0 to 3 years after construction, the conclusions regarding the PAV were drawn using models of variation of the parameters evaluated over time.

In the North American state of Minnesota, (Li et al., 2006) compared three types of binders (one of them unmodified), aged in the laboratory (RTFOT and PAV), with those recovered from cores proceeding from 5 years old pavements where those binders were used. Each core had previously been cut into 3 parts of 25 mm thickness. The binders were tested with DSR, BBR, DT (direct tension) and other tests. The results indicated differences in the aging effects within different locations inside the asphalt layer and significant differences between the recovered and the laboratory aged binders.

Another study in this direction was carried out by (Lu et al., 2008), analyzing unmodified and SBS-modified binders, aged in the laboratory using RTFOT, PAV and RCAT (Rotating Cylinder Aging Test) as well as field cores sampled from several roads in Sweden. It was found that after 10 to 30 years of road service, the extracted bitumens displayed a relatively low degree of aging, to which the equivalent laboratory aging durations were much shorter than that being standardized. The authors attributed this situation to the low percentage of air voids in the mix and to the sealing treatments but did not refer to the possible influence of the low local temperature.

Regarding the relationship of some characteristics of the binder and its effect on the durability of the mixture, some researchers have used certain innovative techniques such as the Glover-Rowe parameter and the ΔTc parameter, identifying ranges of values that allow considering damage from the beginning of cracking in a pavement until its critical state (Anderson et al., 2011); (King et al., 2012); (Anderson, 2014). Another parameter later included in this direction was the viscoelastic transition temperature (Widyatmoko et al., 2004); (García et al., 2019). Some of these techniques will be applied in this work.

In an investigation carried out in the state of Mississippi (Smith et al., 2018) binders were subjected to multiple PAV times and compared with properties observed after 2 or 4 years of field aging. The results indicate that a PAV rate of 11 h per year of simulation seemed adequate for binders aged on the pavement surface (binders extracted from the top 1.3 cm of the pavement surface), and a PAV rate of 3 h per year of simulation seemed most reasonable for binders aged at depth (binders extracted from 5.0 to 6.3 cm below the pavement surface). Considering these rates, the current 20 h protocol in R28 seems to simulate roughly 2 years of aging at the pavement surface and about 6.5 years at depth.

As can be seen from the studies consulted, the impact of aging is greater in the most superficial part of the pavement and the long-term asphalt aging in the laboratory does not correlate precisely with the placement time on site. In these cases, the incidence of weather has not been considered, especially temperature, a very important factor for the aging of asphalt on pavements in service. In the case of Ecuador, on the one hand, there are different well-defined climatic regions and on the other, the asphalt used comes from the Esmeraldas Refinery, which has had premature aging problems for many years (Vila and Mera, 2023).

The general objective of this work is to relate the aging of Ecuadorian asphalt at the laboratory level, applying different conditioning times in the PAV, with the field aging from asphalts recovered from the top of asphalt wearing layers in selected roads of the Andean and Coastal regions, with different construction times and cumulated degree-days (CDD), using certain rheological parameters, for the analysis of the implications of these tests in our conditions. (Figure 1) shows the work methodology used.

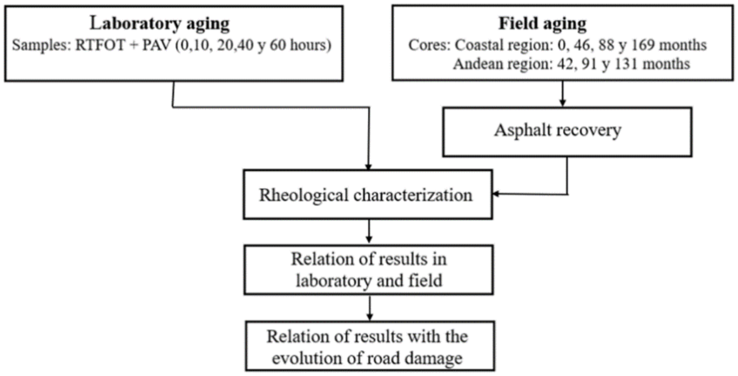

Figure 1. *Flow chart of the applied procedure.*

The CDD is the sum of the daily maximum temperature above freezing for all the days being considered from the time of construction to the time of core sampling (Newcomb et al., 2015).

2. Aging of samples at laboratory level

Asphalt cement from the Esmeraldas Refinery was used without modification; its characteristics are shown in (Table 1). Although the original binder complies with the characteristics of an AC-20, according to NTE INEN 2515, it does not comply with the viscosity and ductility of the RTFOT residue.

Samples were prepared and conditioned according to an experimental design without replicates consisting of tests in RTFO at 163ºC with an aging time of 85 minutes, plus tests in PAV at 100ºC and 300 psi, applying 5 different aging times: 0, 10, 20, 40 and 60 hours. In all the samples certain rheological parameters were evaluated, first, the viscosity. Since aged asphalt has a very high viscosity, it was decided to evaluate it at 135ºC using a Brookfield rotational viscometer.

Table 1. *Asphalt cement tests*

Test	Unit	Requirements AC-20	Result
Absolute viscosity, 60°C	Pa. s	200 ± 40	238
Kinematic viscosity, 135ºC	mm²/s	300 min	375
Flash point Cleveland open cup	°C	232 min	292
Solubility in trichlorethylene	%	99,0 min	xxx
Specific gravity 25°C/25°C		Report	1,012
Penetration, 25°C, 100 g, 5 s	0,1 mm		66
Softening point	°C		51,0
Penetration Index		-1.5 a +1.0	-0,3
RTFOT Residue (163°C, 85 min)			
Viscosity, 60°C	Pa. s	800 max	1360
Mass change	% w/w	1.0 max	-0,06
Ductility 25°C, 5 cm/min	cm	50 min	27

Studies were also carried out with DSR equipment to determine the master curves of aged asphalt, according to the Christensen-Anderson model (Christensen et al., 2017), using frequency sweeps between 0.1 Hz and 37.5 Hz, varying temperatures between 10°C and 70°C with 10°C increments. (Figure 2) shows a visual representation of the main parameters of these curves.

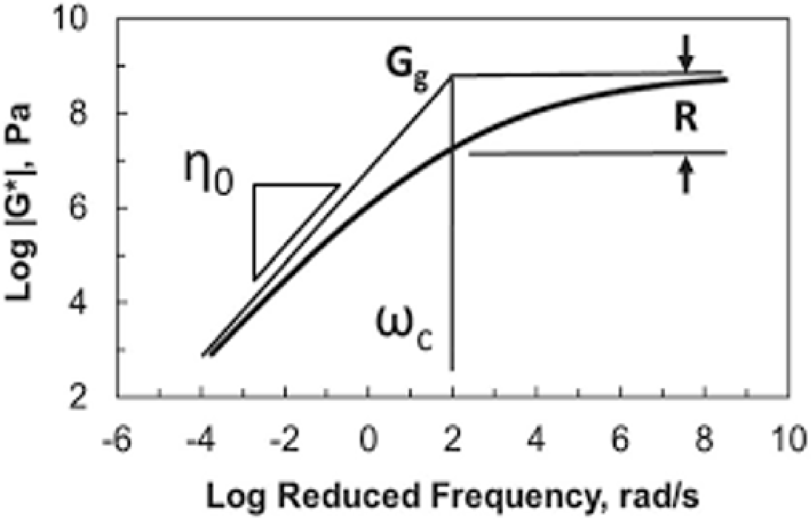

Figure 2. *Typical master curve parameters (Christensen et al., 2017).*

The glassy modulus (Gg) is the largest complex modulus |G| reached at extremely high frequencies or very low temperatures. In general, it is assumed as a constant value of 109 Pa for all binders. Low shear rate-limited viscosity, ηo, is another characteristic often used to describe the high temperature-low frequency asymptote of the master curve. The crossover frequency (ωc) corresponds to the frequency where the phase angle (δ) is 45°, that is, where the elastic component of the complex modulus (G') is equal to the viscous component (G''). For most binders, ωc is almost equal to the intersection of Gg (purely elastic) and ηo (Newtonian or purely viscous flow) at a particular reference temperature (To). The crossover modulus (Gc) corresponds to the value of |G*| in ωc, and the rheological index (R-Value) is the difference between the logarithmic values of Gg and Gc. With aging, for a To, the R-Value increases, ωc decreases, and Gc decreases (Anderson et al., 1994).*

The viscoelastic transition temperature (Tt) , also called the crossing temperature or melting point, is another of the parameters considered to assess aging. It corresponds to the temperature of the asphalt in which the elastic and viscous components of |G| are equal, that is, where its phase angle (δ) is 45° using a constant test frequency. It can be assumed that the transition point from solid to fluid shifts as molecular motion is hindered with aging, so more energy (increased temperature) will be required to generate the flow. The performance of binders that remain below the warning threshold of 32°C after 20 h PAV aging, and below the limit of 45°C after 40 h PAV aging is considered satisfactory, using a frequency of 10 rad/s (García Cucalón et al., 2019). The criteria correspond to the temperature range of the US state of Virginia.*

The mathematical determination of the values ωc, Gc and Tt is carried out from the equation of the master curve obtained according to the applied model, processing the DSR data, which must include temperature, frequency, phase shift angle, complex shear modulus, storage (elastic) modulus and loss (viscous) modulus. In our case, each of the samples was tested with seven different temperatures applying ten frequencies per temperature. The construction of the master curve has been detailed by some researchers (Booshehrian et al., 2013) and can be facilitated with the use of an Excel spreadsheet with the Solver add-on.

The Glover – Rowe (G-R) parameter, which is obtained from DSR tests with frequency sweep at 15°C and 0.005 rad/s, according to the (Equation 1), based on |G| and δ, was also included in the analyses.*

$$G - R = |G^{\times}|.\,(\cos\delta)^2/\sin\delta \qquad \textbf{(1)}$$

With aging, the G-R value will increase. The damage zone obtained from the correlations between the results of the DSR tests and ductility tests carried out in previous studies (Kandhal, 1977), must originate from a G-R value of 180 kPa, finding the pavement cracked due to poor durability when exceeding 600 kPa (Anderson et al., 2011).

3. Obtaining samples of aged binders from different road sections

Cores with a diameter of 150 mm were extracted from different sections of roads in the Coastal and Andean regions. This work was part of a research project developed at the Faculty of Engineering of the Catholic University of Santiago de Guayaquil. The impact of temperature in the two regions and the construction times of the sections were unified using the criterion of cumulated degree-days, CDD (Vila et al., 2022). See (Table 2).

An important selection criterion was that all the mixes were made with crushed alluvial materials of good quality, meeting the same granulometric requirement of a dense mix. The asphalt cement in all cases was AC-20 from the Esmeraldas Refinery, without modification, dosed between 5.8 and 6.0% by weight. In general, it can be considered that the quality deficiencies of Ecuadorian asphalt, once aged in RTFOT, have remained uniform throughout the last 20 years.

***Table 2.** Summary of field sites*

Road	Location	Time (months)	CDD, ºC-days
Coastal region			
Km 26- Puerto Inca- Naranjal	31+000	0	0
Milagro- Naranjito- Bucay	19+240	46	42.079
Milagro- Naranjito- Bucay	33+240	88	80.498
Ampliación del paso por Durán	1+400	169	154.593
Andean region			
Alóag- Santo Domingo	15+520	42	23.761
Alóag- Santo Domingo	5+100	91	51.482
Alóag- Santo Domingo	24+650	131	74.112

In the cores, the aged asphalt of the most superficial layer was recovered in a thickness of 12 mm, using the procedures indicated in the ASTM D2172/D2172M-11 and ASTM D5404/D5404M-12 standards. See (Figure 3). Dichloromethane was used as a solvent. The properties evaluated were the same as those of the asphalts aged in the laboratory.

***Figure 3.** Rotary evaporator equipment used.*

4. Results obtained in the rheological characterization of aged asphalts

(Table 3) and (Table 4) show the results obtained in the different rheological parameters after the aging processes.

Table 3. *Results in asphalts with laboratory aging*

Parameter	Hours in PAV after 85 min in RFTOT				
	0	10	20	40	60
Viscosity, 135 °C (Pa. s)	0,75	1,22	2,24	3,82	10,15
R-value	2,51	2,87	3,23	3,52	4,17
ωc, 20 °C (rad/s)	19,2457	1,3719	0,0744	0,0055	0,000025
Tt, 10 rad/s (°C)	17,8	25,8	33,2	39,6	53,7
G-R, 15°C (kPa)	12,6	61,6	234,1	526,3	1640,7

Table 4. *Results in recovered asphalts with field aging*

Parameter	CDD (ºC-days)						
	0	23.761	42.079	51.482	74.112	80.498	154.593
Viscosity, 135°C (Pa. s)	0,66	0,83	1,11	1,67	3,97	4,08	6,1
R-Value	2,5	2,68	2,74	3,13	2,78	3,15	3,56
ωc, 20 °C (rad/s)	11,3132	8,202	3,2125	0,1111	0,0646	0,0313	0,0002
Tt, 10 rad/s (°C)	19,6	20,7	23,9	33,3	35	37,9	48
G-R, 15°C (kPa)	21,4	15,4	30,9	198,1	606,7	417,2	2301,5

(Figure 4), (Figure 5), (Figure 6), (Figure 7) and (Figire 8) show the relationships between the results of each rheological parameter, taken from (Table 3) and (Table 4).

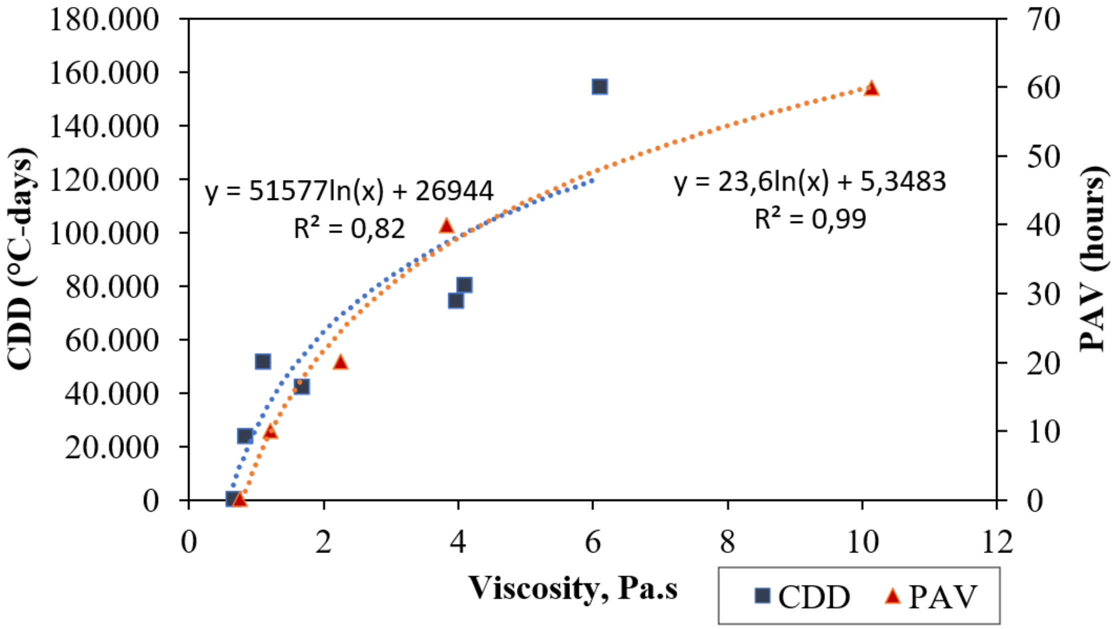

Figura 4. *Viscosity variations for laboratory and field aged asphalts*

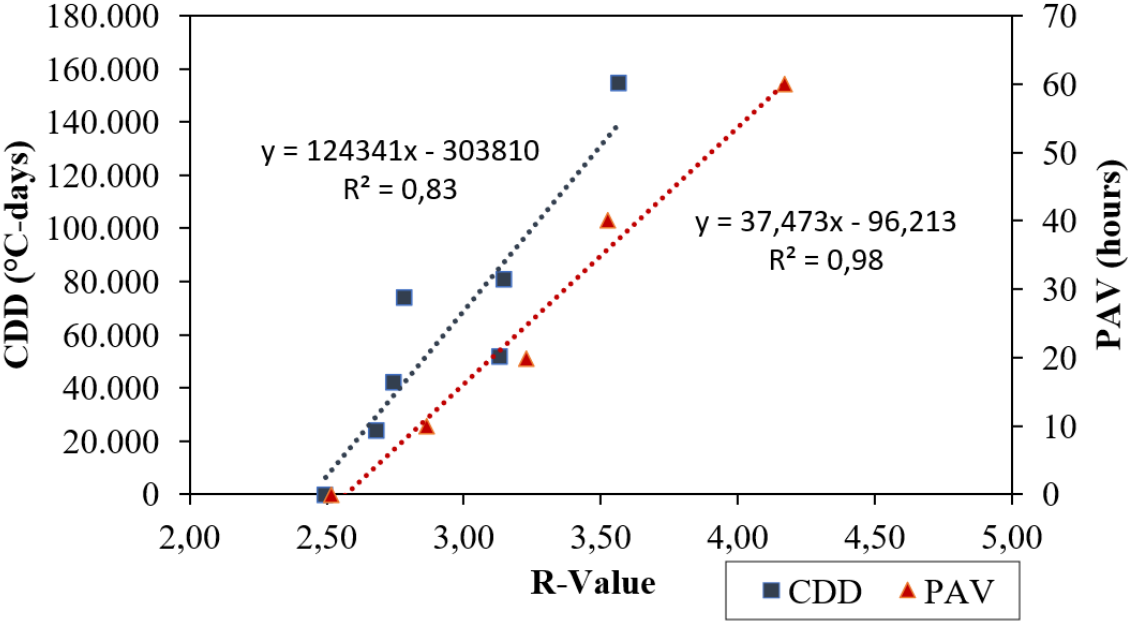

Figure 5. *R-Value variations for laboratory and field aged asphalts*

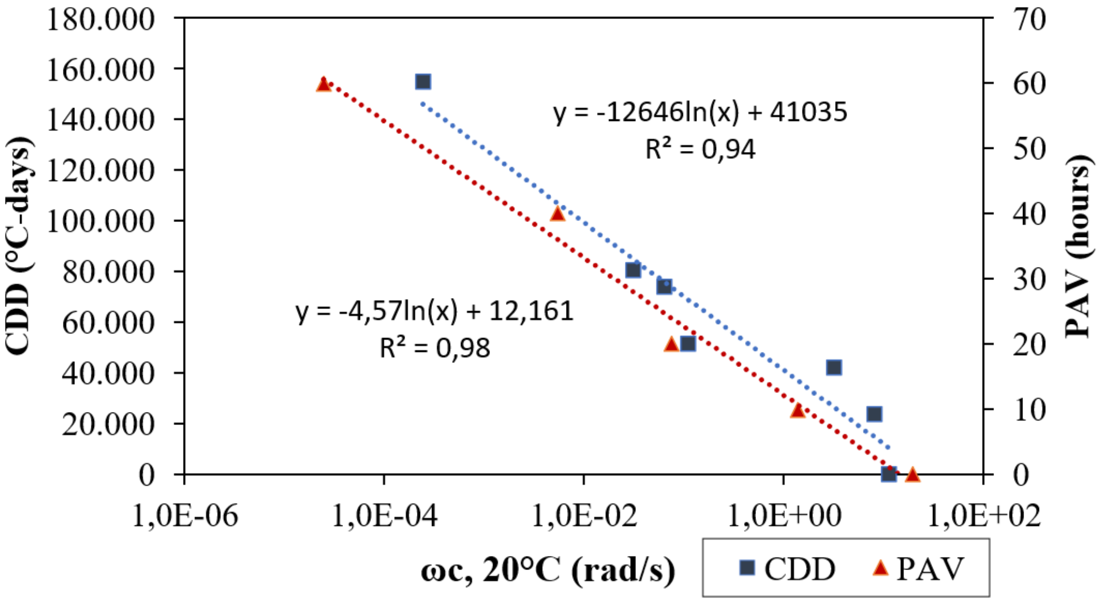

Figure 6. *Crossover frequency variations for laboratory and field aged asphalts*

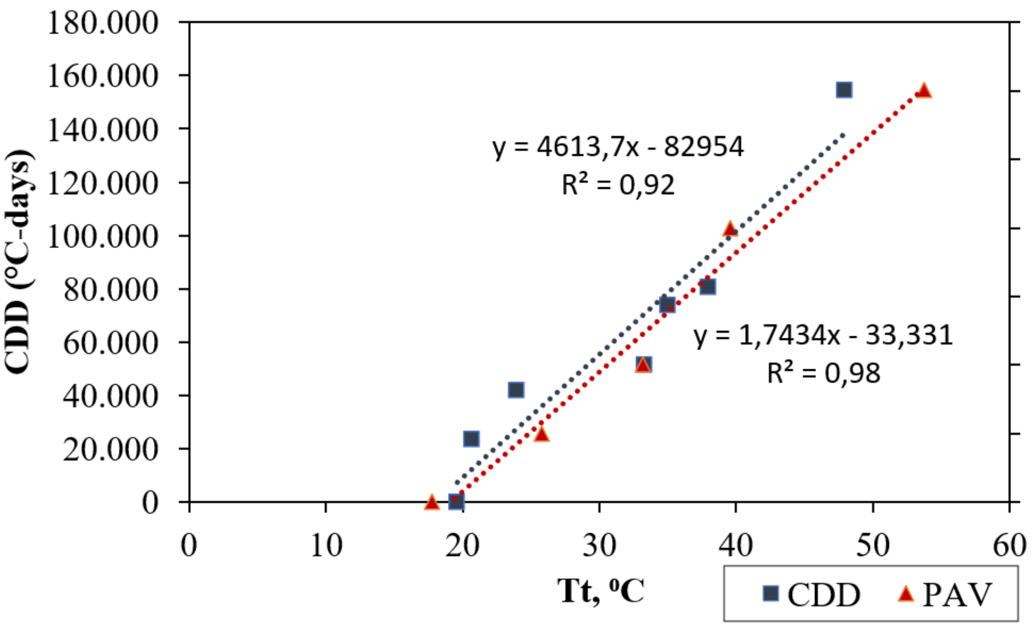

Figure 7. *Viscoelastic transition temperature variations for laboratory and field aged asphalts*

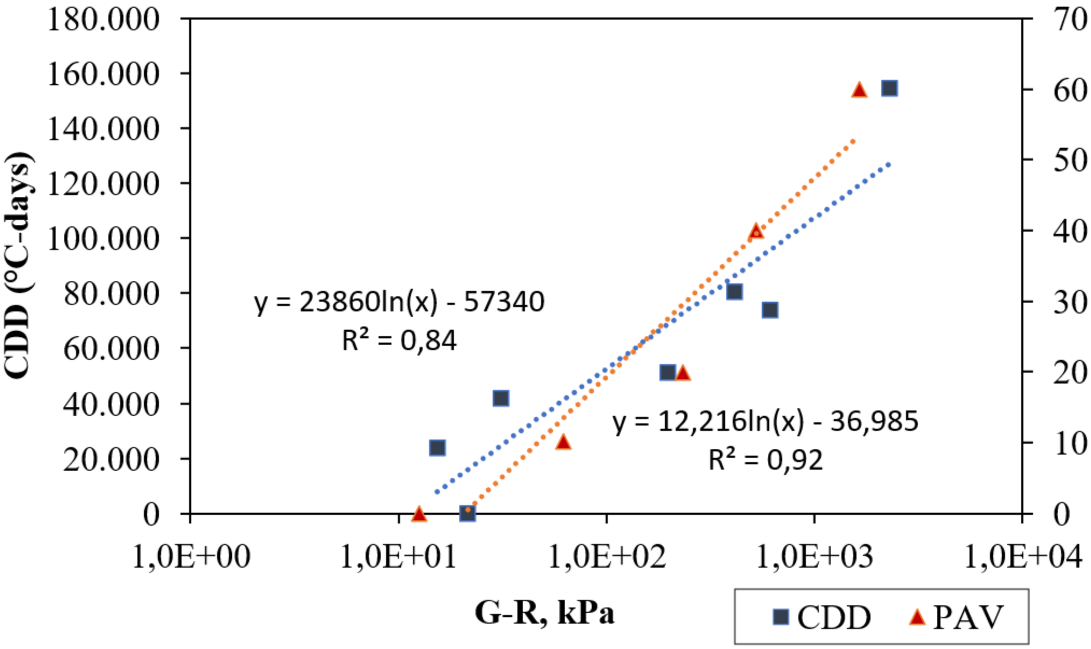

Figure 8. *Glover-Rowe parameter variations for laboratory and field aged asphalts*

In general, the results show a significant increase in the rheological parameters evaluated with aging, except for frequency, where it decreases. These results confirm the expected behavior. In figures 4 to 8, the R2 determination coefficients showed better correlation between the tests carried out in the laboratory, than the recovered asphalt on site. These coefficients varied between 0.82 and 0.99 but were mostly above 0.90.

5. Relation of the results of aging in the laboratory and field

From the equations shown in (Figure 4), (Figure 5), (Figure 6), (Figure 7) y (Figure 8), the values of the rheological parameters were calculated for the PAV of 0, 10, 20, 30, 40, 50 and 60 hours, and with these values the corresponding CDD. The results obtained were graphed in (Figure 9), which shows the well-established linear relationship between PAV and CDD, with a high R2 value.

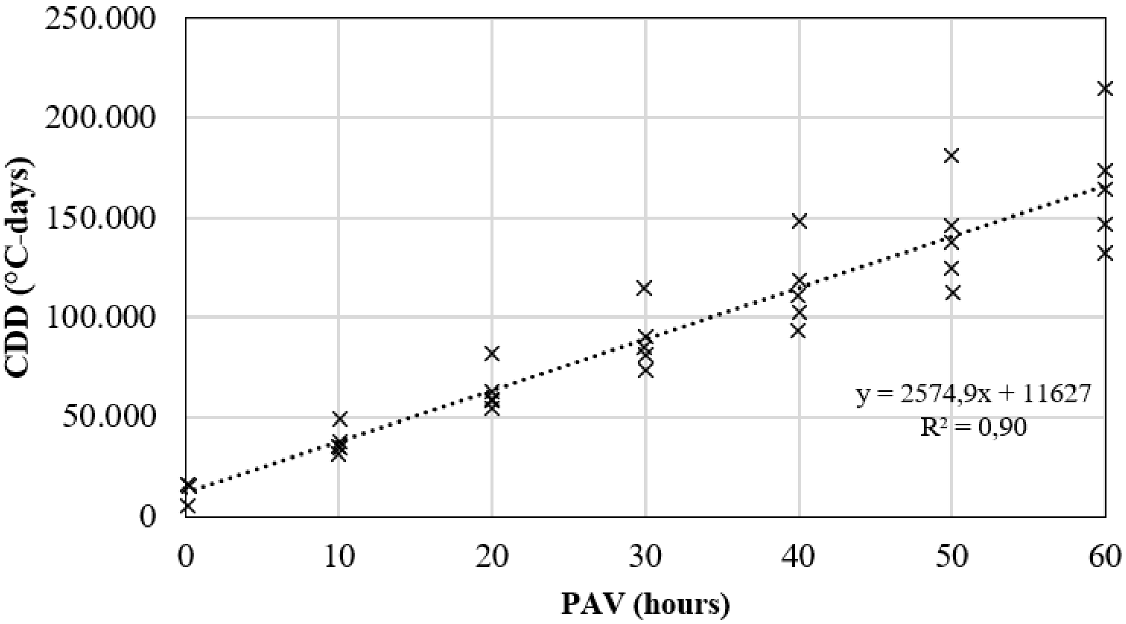

Figura 9. *Variation of CDD versus hours in PAV.*

With the relationship shown in (Figure 9), two significant aspects related to the quality of our asphalt were checked: the CDD corresponding to the PAV equal to 0 hours (only RTFOT) and to the PAV of 20 hours (usually used). With the CDD, the estimated service time in months for the regions was subsequently calculated, according to the (Equation 2) and (Equation 3) obtained in previous studies (Vila et al., 2022). (Table 5) shows the results obtained.

$$\text{Coastal: } CDD = 914{,}75 \times \text{Months} \quad (2)$$

$$\text{Andean: } CDD = 565{,}74 \times \text{Months} \quad (3)$$

Table 5. *Relations between the PAV of 0 and 20 hours and the service time*

PAV (hours)	CDD (°C-days)	Region	Service time	
			months	years
0	11.627	Coastal	12,7	1,1
		Andean	20,6	1,7
20	63.125	Coastal	69,0	5,8
		Andean	111,6	9,3

As can be seen, the results measured directly from the RTFOT residual (PAV of 0 hours) already imply certain aging times on site, which vary from approximately 1.1 to 1.7 years depending on the region. The PAV of 20 hours corresponds to 5.8 years for the Coastal region and 9.3 years for the Andean region. These last times are within the estimated range of long-term aging of the PAV, although *close to its extreme values. However, it should be noted that these results are related to the most superficial thickness (12 mm) of the asphalt layer, which is most affected by aging. In addition, all the cores were extracted from roads with rigorous construction controls and adequate drainage.*

6. Relation of the results with the evolution of the field damage

The observations made on roads in Ecuador have shown that the probable range of the damage evolution, from its beginning to its critical phase, is from 45,000 to 80,000 ºC-days, which imply for the coastal region times between 4.1 and 7.3 years and for the Andean region between 6.6 and 11.8 years (Vila and Mera, 2023). (Figure 9) can be used to correlate this range with the PAV hours in the laboratory, determining that the CDD of 45,000 ºC-days is reached with a PAV of 13.0 hours and that of 80,000 ºC-days with a PAV of 26, 6 hours.

These results allow to establish a relationship between the selected parameters with the durability of the mixture, which can help in the studies carried out in this regard in the country. The values of the rheological parameters determined for the range of CDDs associated with damage also contribute, which are shown in (Table 6).

***Table 6.** Relationship between the values of the rheological parameters and the damage range*

Parameter	Calculated value	
	CDD: 45.000 ºC-days	CDD: 80.000 ºC-days
Viscosity, 135°C (Pa. s)	1,38	2,46
R-Value	2,91	3,28
wc, 20 °C (rad/s)	0,84	0,042
Tt, 10 rad/s (°C)	26,6	34,4
G-R, 15°C (kPa)	60	182

The determined range of Tt to cover from the beginning of the deteriorations to the critical state in Ecuador includes from 26.6 to 34.4°C, much stricter than the reference of 32 to 45°C, obtained in the United States. That is, instead of needing to stay below the warning threshold of 32°C, with our asphalt, the threshold is 28°C. If it reached 32°C, the level of damage would already be considerable. Regarding the G-R parameter, the range obtained from 60 to 182 kPa is also below the reference range (180 to 600 kPa). Note that if the thresholds obtained are lower than the references, our poor-quality asphalt will reach them faster.

7. Conclusiones

In the work, good relationships have been obtained between the rheological parameters evaluated: viscosity, R value, frequency, viscoelastic transition temperature and the Glover-Rowe parameter, both with the aging times in the PAV, and with the field aging considering the CDD. They made it possible to achieve a linear relationship between PAV and CDD, with an R2 value of 0.90.

For the asphalt cement produced at the Esmeraldas Refinery, the RTFOT residual (PAV: 0 hours) implies a CDD of 11,627 ºC-days, which is equivalent to an on-site aging of 1.1 years in the coastal region and 1.7 years in the Andean. In other words, the aging of this residue does not correspond to that of the mixture recently produced, transported, laid out and compacted on the road. On the other hand, the usual PAV of 20 hours corresponds to a CDD of 63.125 ºC-days, with service times of 5.8 years for the coastal region and 9.3 years for the Andean region, which constitutes a considerable difference.

To assess the estimated range of evolution Ecuadorian road deterioration, that is, from its beginning to its critical phase, which include CDD from 45,000 to 80,000 ºC-days, the times in the PAV would be 13.0 and 26.6 hours respectively. For this CDD range, the viscosity (135°C) would be between 1.38 and 2.46 Pa. s, the R value between 2.91 and 3.28, the frequency (20°C) between 0.84 and 0.042 rad/s, the transition temperature (10 rad/s) between 26.6 and 34.4°C and the G-R parameter (15°C, 0.005 rad/s) between 60 and 182 kPa. When comparing the ranges shown for some of the parameters evaluated with other international criteria, such as those existing for theecommendatios G-R parameter and the transition temperature, the presence of a greater impact of aging in Ecuadorian asphalt is demonstrated.

8. Recommendations

Expand the study carried out considering other sections of roads with different CDD, as well as contrast their damage with the ranges determined for the different rheological parameters.

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