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Fernández Gómez, Wilmar Darío; Rondón Quintana, Hugo Alexander; Reyes Lizcano, Fredy Alberto

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Efectos del envejecimiento por radiación UV en las propiedades químicas y reológicas de los cementos asfálticos extraídos de dos mezclas asfálticas

Efeitos do envelhecimento por radiação UV nas propriedades químicas e reológicas dos cimentos asfálticos extraídos de duas misturas asfáticas

Wilmar Darío Fernández Gómez[1*]; Hugo Alexander Rondón Quintana[1]; Fredy Alberto Reyes Lizcano[2].

[1] Faculty of Environment and Natural Resources, Center of Pavements and Sustainable Material, Universidad Distrital Francisco Jose de Caldas, Bogota, Colombia.
[2] Department of Civil Engineering, CECATA Research Center, Pontificia Universidad Javeriana, Bogota, Colombia.

*wfernandez@udistrital.edu.co

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Abstract
Asphalt aging is a critical issue for pavement engineering because aging reduces asphalt pavement durability. We studied the incidence on asphalts chemical and rheological properties not only by UV radiation but also by pressure and temperature exposition. Two asphalt cement AC 60-70 and AC 80-100 were used to manufacture two types of samples. All samples were binders obtained from neat asphalts as control samples and extracted from the top of asphalt mixtures briquettes. Neat binders were aged following Peformance Grade SUPERPAVE® method. Also, SUPERPAVE® dense graded methodology was used to manufacture the MD-12 asphalt mixtures. The top of the briquettes was subjected to periods of ultraviolet radiation and condensation of 2 hours each, during 1000 hours. After aging treatments, the aged binder's complex moduli increase and phase angles reduce but showed similar results in both asphalts. Nevertheless, the aging ratio measured through the colloidal instability index was two times from the AC 60-70, while in the AC 80-100 was 1.5 times after 50 hours PAV, and was 1.9 times after 1000 hours UV treatment. The aforementioned could be explain due to binder film thickness, which is different in asphalt mixture respect to PAV test. SARA fractionation showed increases in asphalthene moieties in all asphalts after all treatments and it should explain ductility loss and rigidity increase on asphalt mixtures after aging.

Keywords: *Asphalt Oxidation, SARA fractionation, Asphalt Aging, UV radiation.*

Resumen
El envejecimiento del asfalto es un problema crítico en la ingeniería de pavimentos porque el envejecimiento reduce la durabilidad de los pavimentos asfálticos. Este artículo establece los efectos en las propiedades químicas y reológicas no solo por tratamientos de radiación ultravioleta sino también por exposición a temperatura y presión. Se utilizaron dos cementos asfálticos AC 60-70 y AC 80-100 para evaluar dos tipos de muestras. Las muestras de control son los asfaltos mencionados sin envejecer y otros extraídos de mezclas asfálticas. Los asfaltos originales se envejecieron siguiendo el protocolo del grado de desempeño de SUPERPAVE®. También el método SUPERPAVE® se utilizó para realizar el diseño

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de la mezcla asfáltica tipo MD-12. La superficie de las briquetas se sometió a periodos de radiación UV y de condensación de dos horas cada uno, hasta alcanzar 1000 horas. Después de los tratamientos de envejecimiento, el módulo complejo aumentó y el ángulo de fase decreció de manera similar en los dos asfaltos. Sin embargo, para el caso del tratamiento en PAV, la relación de envejecimiento medida a través del índice de inestabilidad coloidal fue de dos veces para el AC 60-70, mientras que en el AC 80-100 fue de 1,5 veces. Para el caso del tratamiento UV esta relación AR fue de 1,9 veces para ambos asfaltos. Lo anterior es debido al espesor del asfalto, que es diferente en la mezcla asfáltica comparado con el espesor del asfalto en la prueba PAV. El fraccionamiento SARA mostró incrementos en los asfaltenos lo que puede explicar la pérdida de ductilidad y el aumento de la rigidez después del envejecimiento.

Palabras clave: *Oxidación del Asfalto, fraccionamiento SARA, Envejecimiento Asfalto, Radiación UV.*

Resumo

O envelhecimento do asfalto é um problema crítico na engenharia de pavimentos porque reduz a durabilidade dos pavimentos asfálticos. Este artigo apresenta a incidência do envelhecimento sobre a oxidação e as mudanças nas propriedades químicas e reológicas de dois asfaltos extraídos de duas misturas asfálticas. O envelhecimento foi feito tendo em conta os processos de temperatura, pressão e radiação UV. Para fabricar as misturas asfálticas tipo MD-12 de concreto asfáltico foram utilizados dois cimentos asfálticos com graus de penetração (ASTM D-5, mm/10) de 60-70 e 80-100. A mistura foi elaborada empelando os lineamentos da metodologia SUPERPAVE® para misturas densas. Os corpos de prova foram expostos a tratamentos de radiação UV e de condensação em períodos alternados de duas horas, até completar 1000 horas de envelhecimento numa câmara projetada e construída para esta pesquisa. Depois de someter as mostras a tratamento UV, foram extraídos os asfaltos da superfície dos corpos de prova e comparou-se com o asfalto exposto ao método de envelhecimento acelerado PAV. Depois dos tratamentos de envelhecimento, o módulo complexo aumentou e o ângulo de fase desceu nos asfaltos. No entanto, para o caso do tratamento PAV, a relação de envelhecimento AR (medida usando o Índice de Estabilidade Coloidal) foi de 2 vezes para o CA 60-70 em quanto para CA 80-100 foi 1,5 vezes. Para o caso do envelhecimento usando UV, a relação AR foi 1,9 vezes para ambos asfaltos. Isto é devido principalmente, a que as espessuras dos asfaltos são diferentes, tanto na mistura asfáltica quanto no ensaio PAV. O fraccionamento SARA mostrou aumentos nos asfaltenos o que pode explicar a diminuição da ductilidade e o aumento da rigidez depois do envelhecimento.

Palavras-chave: *oxidação de asfalto, SARA, Envelhecimento do asfalto, Radiação UV.*

Introduction

Aged asphalt shows decreased adhesion between the aggregate and the binder, especially when the binder thickness is thin [1]. Moreover, the binder's ductility changes, causing brittleness, which is associated with increased stiffness and viscosity. Occasionally, slight asphalt aging proves to be desirable, as the asphalt mixture stiffens, reducing ductility [2]. However, further aging is not recommended as it becomes brittle under loads [3,4]. According to Kim *et al.* [5], premature failure or poor performance of asphalt pavements is often the result of a weak adhesion between the binder and the aggregate particles. Aging changes asphalt characteristics and it is usually accompanied by hardening [6]. One of the most important factors of asphalt aging is oxidation, as it leads to asphalt hardening and subsequent weakening. Excessive hardening often leads to cracking of the asphalt layer of the pavement at low operating temperatures [7].

Asphalt is composed by different chemical composites classified as saturates (S), aromatics (A), resins (R) or asphaltenes (A), in such a proportion that a viscous material is formed, and it is commonly used in cement mixtures of asphalt concrete for road construction. Oxidation and volatilization leads to alterations in these groups since molecules are rearranged and carbonyl and sulphoxide groups are formed. [8]. Thus, the oxidation of asphalt involves irreversible chemical reactions due to the asphalt's components and atmospheric oxygen, which can be vastly accelerated by the presence of ultraviolet light [9].

UV radiation on asphalts has rapid and important effects on service pavements [10,11]. This is because the high energy contributes to the fractionation of asphalt molecules, which, together with oxidation induced by temperature, moisture and air, leads to asphaltene formation [12]. The rise of solid fractions in the asphalt, directly increases rigidity (reduced ductility); thus, the asphalt embrittels, and, over time, cracking occurs and the asphalt mixture asphalt fails [13,14]. This effect is more pronounced in hot mixed asphalts. Asphalt aging is classified into two types: short- and long-term [15–18]. Short-term aging mainly results from the oxidation and volatilization of the asphalt mixture's binder during manufacture in the factory (including storage) and construction work (laying and compaction). Long-term aging is also due to oxidation, but hardening occurs *in situ* during the pavement's service life [11].

In order to assess resistance to short-term and long-term aging in asphalt cements (hereafter AC), RTFOT (Rolling Thin Film Oven Test) and PAV (Pressure Asphalt Vessel), respectively, have been the most common tests. However, other tests for short-term aging include the Rolling Microfilm Oven Test (RMFOT), Tilt Film Accelerated Aging Test (TFAAT) and the Oven Durability Test [18]. For long-term aging, Rotating Cylinder Aging Test (RCA), Iowa Durability Test (RTD), Pressure Oxidation Bomb (POB), SHRP-PAV, High Pressure Aging Test (HiPAT), microwave aging and ultraviolet/infrared light treatments (UV) have been employed. [18]

In that way, to study the aging phenomenon, asphalt mixtures are subjected to different treatments such as heat, oxidation, UV or infrared treatments [19]. Throughout these treatments, asphalt mixtures are exposed to high temperatures for certain periods of time. Oxidation tests also combine high temperature and pressure. Even more, Airey [18], Rondon and Reyes [20] and Fernández- Gómez *et al.* [19] have suggested that ultraviolet radiation is an important tool for the analysis of the durability of asphalt and asphalt mixtures. Although research involving exposure to UV radiation has increased (e.g. [9,10,16,21–32]), standard treatments simulating the disturbance caused by solar radiation on asphalt binders as well as studies on the chemical properties of asphalt binders in service under sunlight are still scarce.

Therefore, in order to evaluate the effects the aging by UV radiation on hot mixed asphalts, we evaluated the changes in the chemical and rheological properties of two types of asphalt cements (AC 60-70 and AC 80-100) extracted from asphalt concrete mixtures artificially aged by UV radiation and aged by accelerated temperature and pressure procedures. The former was done using a UV chamber that simulated field conditions with day/night radiation and condensation cycles while the latter was done by Pressure Aging Vessel test. As both AC 80-100 and AC 60-70 asphalts are widely used for pavement construction in Colombia, the results of this research provide information respect to long term ultraviolet effects on asphalts within the asphalt mixtures in terms of chemical and rheological properties.

Materials

Two different types of samples were used to evaluate the aging effects. On the one hand, neat AC 60-70 and AC 80-100 were chemically and rheologically tested. In addition, AC's were subjected to Superpave® (AASHTO MP-1) aging treatments: RTFOT according to ASTM 2878-04 for short term aging and PAV (ASTM 6521 -08) during 20 and 50 hours for long term aging. On the other hand, two sets of asphalt mixtures were manufactured in the laboratory and were exposed to UV aging during 1000 hours. Each set was mixed with AC 60-70 and AC 80-100, respectively, with different air void content. After 1000 hours of UV treatment, asphalt cements were extracted from the surface of asphalt mixtures samples and were evaluated through SARA fractionation test (ASTM-D4124-09) and Infrared Spectroscopy.

As all asphalts for pavements used in Colombia are provided by Ecopetrol S.A., the state-owned oil company, the samples of this study were not the exception. Hence, both AC 80-100 and AC 60-70, asphalts are widely used for pavement construction in the country. Table 1 shows the results of the physical properties of both neat asphalt cements used in this study.

In order to manufacture asphalts mixtures in laboratory, the physical characteristics of aggregates were also evaluated (Table 2).

Table 1. General Characteristics of AC 80-100 and AC 60-70. Three samples were used for each test.

Test	Method	Units	AC 80-100	Stand. Dev.	AC 60-70	Stand. Dev.
Original Asphalt						
Penetration (25°C, 100 g, 5 s)	ASTM D-5	0.1mm	82.7	0.67	61.0	1.5
Specific Gravity	INV. E-707	-	1.007	-	1.012	-
Penetration Index	NLT 181/88	-	0.22	-	-0.13	-
DSR Viscosity(60°C)	ASTM D-4402	Pa-s	136	0.08	145.6	0.08
Ductility (25°C, 5cm/min)	ASTM D-113	Cm	>105	7.6	>105	11.0
Softening Point	ASTM D-36-95	°C	50.5	0.51	52.4	2.9
Solubility in Trichlorethylene	ASTM D-2042	%	>99		>99	
Water Content	ASTM D-95	%	<0.2		<0.2	
Flash Point	ASTM D-92	°C	358		323	
Residue after RTFOT						
Mass Loss	ASTM D-2872	%	0.5		0.3	
Penetration (25°C, 100 g, 5s)	ASTM D-5		49.5	4.0	39.7	1.3
PAV (20 hours)	ASTM 6521 -08		34.8	1.1	36.8	2.5

Table 2. Physical characteristics of aggregates. Fluvial materials came from Coello and Guayuriba Rivers in Colombia.

Test	Method	Result
Coarse Aggregate Specific Gravity	ASTM D 854–00	2.65
Sand Equivalent	ASTM D 2419-95	58%
Fractured Faces	ASTM D 5821-01	88%
Elongation Ratio	INV. E-230	2.8%
Blue Methylene	INV. E-235	4.8%
Soundness of Aggregates	ASTM C 88-99a	1.0%
Microdeval	ASTM D6928-03	10.5%
Wear Resistance Los Angeles Machine	ASTM C 131-01	28.6%

Mixtures Design

Cylindrical asphalt mixture samples (briquettes) in the MD-12 standard (following [33]) of 10cm diameter and 20cm high were manufactured in laboratory also following the SUPERPAVE® methodology (Table 3). Briquettes were compacted in a gyratory compactor (GPC) to ensure that air voids content was 4% and 10% per AC type. Three samples were made for each type of AC, thus, 12 briquettes were analyzed in total. The asphalts used were not previously subjected to oven aging with RTFO because AC underwent short term aging in the mixing and compaction process.

Table 3. Granular distribution of Asphalt Mixture MD-12.

Normal	19.0mm	12.5mm	9.5mm	4.75mm	2.00mm	425mm	180mm	75mm
Alternate	3/4”	1/2”	3/8”	No.4	No.10	No. 40	No. 80	No.200
% Passing	100	80-95	70-88	49-65	29-45	14-25	8-17	4-8

Exposure to UV Rays

Briquettes were subjected to UV aging treatment after the mixing and compacting process. An Atlantec brand chamber with eight lamps emitting radiation at a wavelength of 340nm in the UVA range equivalent to 0.77W/m^2/nm was used. Periods of two hours of radiation at 60°C and condensation at 50°C were operated. The UV chamber had a free area inside of 40cm width by 110cm length and 25cm height to support samples; thus, the MD-12 briquettes were organized in order to ensure that the upper face of each one was irradiated (Figure 1). Also, to avoid radiation over the body of the samples, each one was protected with a polymer foil (Figure 2). Rotation between the two periods was made, aimed at simulating current material exposure to environmental conditions, where daytime exposure is followed by a rest period (night). During the latter, physical and chemical changes of the material are supposed to take place [34].

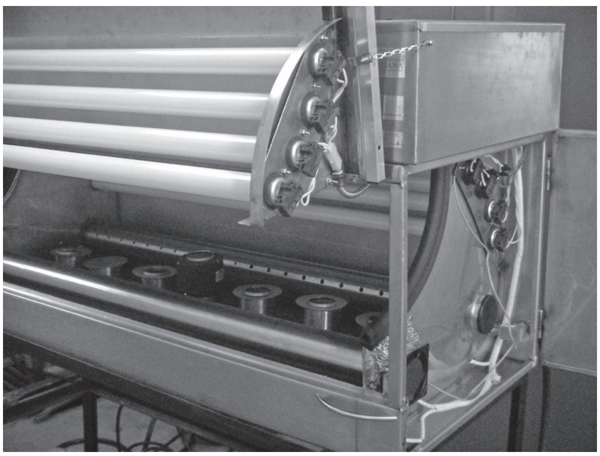

Figure 1. Ultraviolet chamber and samples disposition.

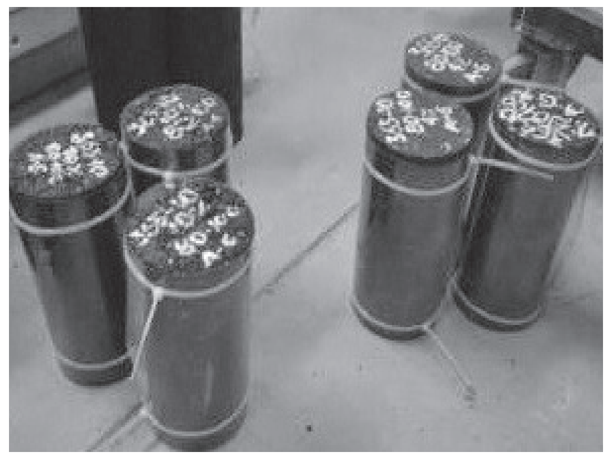

Figure 2. Protection around samples to ensure top face exposition.

Samples exposure time was 1000 hours to simulate long term aging shifting periods of radiation and condensation. The 1000-hour test was chosen because the value of the radiation wavelength of 340nm corresponds, on average, to 4.04kWh/m^2/day in Bogota (168.3kW/m^2), Colombia [34] UVA-340 lamps typically have little or no UV output bellow 300nm and they allow good correlations with actual outdoor weathering (ASTM G 154-06). As the aging chamber used has an irradiance value of 5.5W/m^2 (ASTM G 154-06) for each cycle, the 1000 hours UVA radiation simulated three years of solar exposure in Bogota, Colombia (Table 4).

Table 4. UV Exposure and Equivalent Field Months.

Exposure Periods UV	Units	Equivalence				
Chamber	Hours	50	100	200	500	1000
Field	Months	2.3	4.6	9.2	18	36

Methods and Results

Fourier Transform Infrared Spectroscopy

In order to determine the spectroscopy of asphalt mixtures with 4% and 10% air voids, after 1000 hours in UV chamber, 2g samples were extracted from the briquette's surface. A solution with chloroform and each sample was prepared, placed on two bromide plates and installed in a Shimadzu FT-IR 8300 spectrophotometer. The infrared spectrum was measured at room temperature without the sample, and then with the sample inside. As chloroform evaporated naturally with air contact, it did not affect the measurements. An AC 60-70 and AC 80-100 sample, not exposed to UV radiation was used as a control (Neat asphalt).

The peaks at 1050 and 1650cm^{-1} wavelength (sulphoxide and carbonyl, respectively), of neat asphalts in the infrared spectra indicate that they oxidate even before entering the manufacture process (Figures 3 and 4). The spectra of mixtures with 4% and 10% air void content (shadow area), showed a wider peak at the same wavelength, indicating that UV radiation generates oxidation.

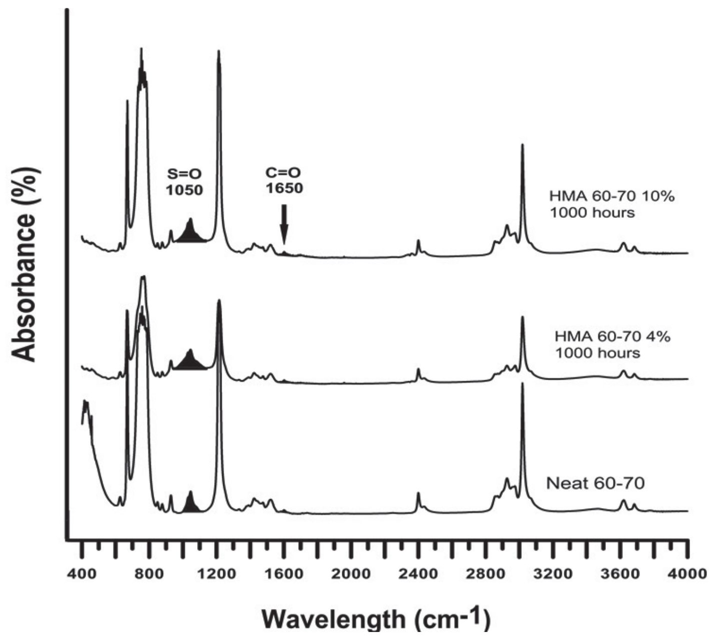

Figure 3. Spectra of Asphalts AC 60-70 extracted from HMA with 4% and 10% air voids subjected to 1000 hours in aging in UV chamber, in contrast to neat asphalt.

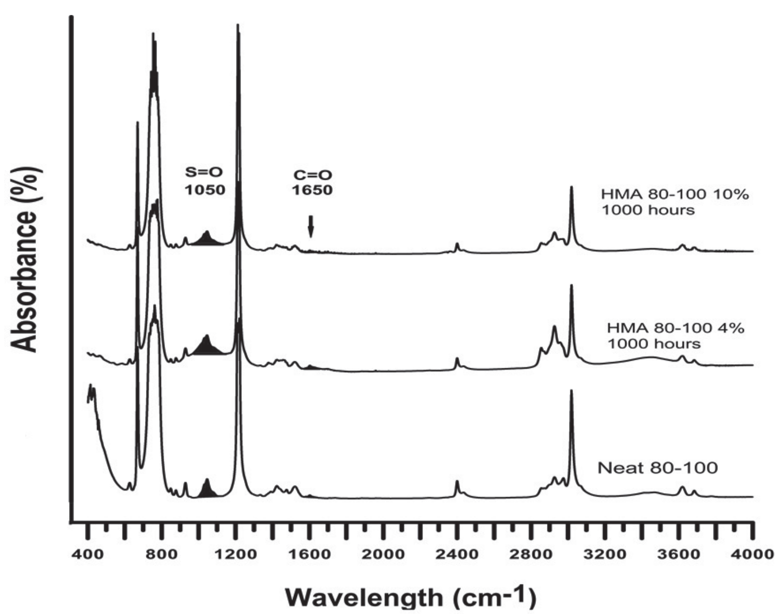

Figure 4. Spectra of Asphalts AC 80-100 extracted from HMA with 4% and 10% air voids subjected to 1000 hours in aging in UV chamber.

SARA Evaluation

The separation of SARA fractions was made using Corbett´s liquid chromatography column (1979), following the ASTM D-4124 specification. The procedure was performed for two replicates per sample. To confirm alterations of the asphalt in terms of the SARA fractions, an evaluation was performed before and after 1000 hours of aging in the UV chamber, as well as after 20 and 50 PAV treatment.

Results of the SARA fractionation, showed that almost all cases exhibited more asphaltenes and less saturates, aromatic and resins (Table 5). These changes in SARA fractions are explained by the oxidation that occurs during the aging process, as evidenced by infrared spectra. In that way, UV radiation possesses sufficient energy to ionize the atoms or form free radicals, which are reactive and hasten molecular breakdown (cracking). When this happens, the small molecules reconfigure the fractions, affecting the SARA separation and new fractions are formed. Besides, the aging ratio (AR) for 1000 hours of exposure in the UV chamber is greater than the AR for 20 hours of PAV treatment for these two AC's (Table 5), which means that the long term UV treatment (1000 hours) is more oxidative than PAV and both AC 60-70 is slightly susceptible to long-term aging than AC 80-100. In contrast, PAV 50 hours was more aggressive for AC 60-70 than AC 80-100 due to AR increased 1.54 times in the former and 1.25 times in the latter respect to PAV 20 hours. Finally, AR obtained from 1000 UV hours in contrast to 50 hours PAV was more aggressive for AC 80-100 and similar to PAV 50 hours which confirm more susceptibility of AC 60-70 to aging in long term periods of exposition.

Table 5. SARA Fractionation of Aged Asphalts.

Material	Treatment	Saturates	Aromatics	Resins	Asphaltenes	Losses	Colloidal Instability Index	Stand. Dev.	AR*
80-100	Neat	18.3	30.5	40.4	9.6	1.1	0.39	0.00	1.0
	1000 Hours UV	22.0	19.7	33.6	16.8	7.3	0.73	0.01	1.9
	PAV 20 H	12.8	28.3	37.8	19.0	2.1	0.48		1.2
	PAV 50 H	19.3	21.4	38.1	15.1	6.9	0.58		1.5
60-70	Neat	15.9	23.2	37.5	8.9	14.5	0.41	0.00	1.0
	1000 Hours UV	22.4	20.5	33.2	18.7	4.7	0.77	0.02	1.9
	PAV 20 H	13.8	25.8	40.0	20.4	0	0.52		1.3
	PAV 50H	18.6	21.0	29.4	21.7	8.4	0.80		2.0

* Aging Ratio

Even though the colloidal instability index consistently increases along the aging process, this index depends on the type of asphalt and on the aging treatment. Although several studies have established that the PAV treatment can represent several years of asphalt oxidation [36-38], the UV treatment have produced a more aggressive aging, as the colloidal instability index has shown similar relations to different periods of PAV while higher in the UV chamber. Probably, this is because the chamber simulates in a similar fashion the day/night periods occurring on the road, producing similar results for weathered asphalts, as reported by Rondon *et al.* [39].

Rheological Properties

Asphalt PAV residues were tested in a Dynamic Shear Rheometer (DSR) TA 2000 ex with parallel plates 8 mm in diameter at a frequency of 10 rad/s and 1% strain (ASTM-D6373-07). AC 60-70 and AC 80-100 exhibited a 58(16) Performance grade PG. 58 is the high temperature and 16 is the medium temperature, low temperature was not evaluated because it is not useful for tropical countries. Figures 5 and 6 display the results of rheological tests performed with the DSR on AC 80-100 and AC 60-70 subjected to PAV oven aging (long-term). The results correspond to the mean of ten measurements of each of three samples. All AC's underwent changes in its complex modulus G*. However, a higher G* was obtained when exposure time was 50 hours. The complex modulus was even higher in the case of AC 60-70 (between 150% and 192% approximately) than the experienced by AC 80-100 (between 61% and 93%, approximately). Conversely, the phase angle (δ) was smaller after 50 hours of exposure. This difference ranges from 13% to 20% for both AC.

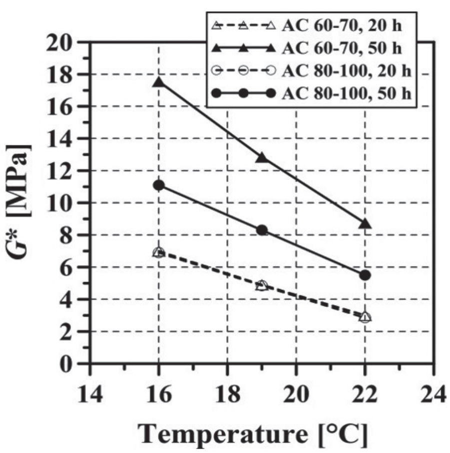

Figure 5. Comparison of Complex Modulus vs. Temperature for Asphalts AC 60-70 and AC 80-100 at 20 and 50 Hours of PAV Exposure The G* for unaged AC, could not be included, because the values are less than 10Pa at this temperatures.

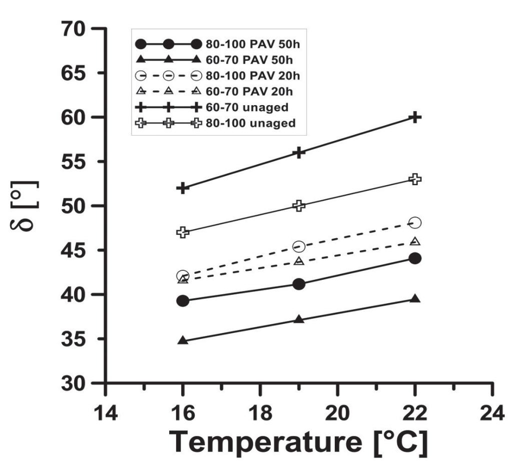

Figure 6. Comparison of the Phase Angle vs. Temperature for Asphalts AC 60-70 and AC 80-100 at 20 and 50 Hours of PAV Exposure.

Discussion and Conclusions

The UV aging process affected the mechanical and chemical properties of extracted asphalt mixtures. As expected, mechanical properties in the studied asphalts exhibited significant increases in the complex modulus and decreases in phase angle. In the case of the two asphalts studied, after 20 hours of PAV aging, the complex modulus and aging ratio showed similar results. Nevertheless, after 50 hours of PAV the AC 60-70 aging ratio measured through the colloidal instability index was two times of the original (neat), while the AC 80-100 increased by 1.5 times. If 20 hours PAV means 8 years in the field [37], both asphalts will have the same aging susceptibility. However, after this time AC 60-70 seemed to be more susceptible to aging, losing ductility and hence, they become more fragile and exhibit more cracking.

Although the infrared spectra was an important tool for determining the presence or absence of oxidation in the neat and aged asphalts, as the increase in the areas under the curve between aged and neat asphalts was slight. Despite Carbonile and Sulfoxide peaks in Figures 3 and 4, did not evidence measurable oxidation, SARA fractionation presented in Table 5 showed important increases in asphaltenes moieties, which is an indication of aging process. Hence, the above implies that small changes in the chemical properties generates important changes in mechanical behavior, as increases in |G*| (Figure 4).

This study demonstrate different chemical and mechanical effects on binders due to different aging treatments. Hence, the tratments do not accurately replicate field conditions despite some variables such as temperature, pressure, humidity or maximum radiation were controlled in laboratory. However, aging ratio from colloidal instability index shows more aggressive aging by UV treatment than PAV 20 hours because the film of binder is quite different. While, binder thickness in PAV sample is near two milimeters, binder thickness over aggregates in asphalt mixtures is between six to ten microns [40]. Therefore, in order to successfully reproduce the conditions that asphalt experiences throughout its service life, future studies should simultaneously simulate all the conditions above mentioned and take into account the binder film thickness.

The neat Colombian asphalts studied should not be used in all regions of Colombia, particularly in areas were temperature exceeds 58°C or is below

16°C according to PG classification. Considering that countries located in the tropical region are exposed to higher UV radiation levels, this study provides useful information on UV, as an important that should be considered when designing asphalt mixtures. Further studies about UV alteration on service pavements must be addressed.

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