

Optimal hydrated lime concentration in asphalt binder to improve photo degradation resistance

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Abstract

The effect of short-term oxidation, evaluated by using a Rolling Thin Film Oven Test, and the effect of weathering aging on the oxidation process of hydrated lime modified asphalts were studied. Hydrated lime (HL) has been known as an additive for asphalt mixtures and is now considered as a binder additive that increases asphalt mixture durability. HL was added to the binder in contents of 5, 10, 20, and 30 wt.%. The HL modified binders were aged by using the Rolling Thin Film Oven Test and weathering tests with xenon radiation. A storage stability test by measuring the softening points of HL modified binders was performed. The Thin Layer Chromatography with Flame Ionization Detection (TLC-FID) analysis was used to determine the fractions of resins, aromatics, asphaltenes and saturates of the binders. The aging of HL modified binders was evaluated by using Fourier transform infrared spectroscopy. According to the stability test, a hydrated lime concentration value close to 20 wt.% is the critical point at which there is a risk of segregation of the asphalt binder and hydrated lime during transportation and storage. For the sample weathering aged for 250 hours, a decrease of saturated and aromatic fractions, furthermore an increase in asphaltenes were observed for all samples. Results have shown that additions of 10 wt.% of hydrated lime in asphalt binders resulted in a higher photodegradation resistance among the other asphalt mixtures considering the carbonyl index, stability test and colloidal instability test results.

Keywords: hydrated lime; asphalt binder; weathering aging; infrared spectroscopy; colloidal instability.

1. Introduction

Oxidation is one aspect of asphalt aging that results in volatile loss and

consistency increase of the asphalt binder. Moisture is another variable in

the asphalt aging process and promotes a cohesion loss between the aggregate

and binder (stripping) (Araújo *et al.*, 2013). Temperature and solar radiation also play a key role in asphalt aging studies (Araújo *et al.*, 2013).

Durrieu *et al.* (2007) has reported that the influence of ultraviolet radiation on the aging of the upper layers of the asphalt pavement cannot be ignored since the degradation is rapid: after 10 hours exposure to ultraviolet radiation, the asphalt oxidation levels are similar to those achieved after being subjected to the Pressure Ageing Vessel (PAV) and Rolling Thin Film Oven Test (RTFOT), or after one year of service.

The aging process affects the chemical constitution of asphalt binders, increasing the content of the asphalt phase and decreasing the concentration of the maltene phase of the asphalt binder (Corbett and Schweyer, 1981, Petersen, 2009, Petersen and Glaser, 2011). In recent years, asphalt photo-degradation has been considered as a relevant parameter in the study of asphalt pavement performance (Lins *et al.*, 2008). The Rolling Thin Film Oven Test (RTFOT, ASTM D2872) is used to replicate the short-term aging that occurs during the mixing operation, while the Pressure Aging Vessel (PAV, ASTM D6521) is adopted to reproduce the long-term aging. The RTFOT and PAV tests mainly simulate the thermal-oxidative aging that is caused by heat and oxygen, but the photo-oxidative aging which is caused by the ultraviolet (UV) radiation and oxygen is often ignored, and there is no standardized test that allows quantitative evaluation of the photo-oxidative degradation of bitumen (Mouillet *et al.*, 2008, Xiao *et al.*, 2015, Kuang *et al.*, 2014, Xu *et al.*, 2015, Lopes *et al.*, 2014, Araújo *et al.*, 2013). Mouillet *et al.* (2014) emphasize the relevant effect of UV radiation on asphalt aging and cited that the UV exposure may affect the bitumen's properties of pavement upper layers more strongly than the PAV simulation in laboratory (without UV action) does. Wang *et al.* (2016) also reported the strong dependence of the service life of polymer modified asphalt on its weather resistance.

Several anti-aging additives have

been studied to produce modified asphalt binders to mitigate the effects of detrimental agents such as solar radiation, temperature and oxygen (Araújo *et al.*, 2013). Carbon black could block the UV rays, but the addition of carbon black has a negative effect on the low-temperature performance of bitumen (Yamaguchi *et al.*, 2005, Cong *et al.*, 2014). Other anti-aging agents used in asphalt are styrene-butadiene rubber (Zhang and Hu, 2013), styrene-butadiene-styrene (Cong *et al.*, 2014, Cortizo *et al.*, 2004), polyphosphoric acid and sulfur (Zhang and Hu, 2013), lignin (Pan, 2012), and graphite (Pan *et al.*, 2014).

Hydrated lime is beneficial to asphalt mixtures used in pavements (EuLA, 2010; Aragão *et al.*, 2010). The use of HL as an additive was intensified during the 1970s in the USA, preventing moisture damage (Lesueur *et al.*, 2013; Little and Epps, 2001). Other beneficial effects of HL modified asphalts are the reduction of chemical aging of the bitumen, increasing rutting resistance, improvement of fatigue resistance and improvement of toughness and resistance to fracture growth at low temperatures (Lesueur *et al.*, 2013; Little and Apps, 2001).

Little and Petersen (2005) confirm that the use of HL as a filler impacts the micro-crack induced damage, micro-damage healing, and plastic and viscoelastic flow in both mastics and mixtures. Zou *et al.* (2013) also report that hydrated lime mitigates the deleterious effect of oxidation and moisture.

The chemical effect of HL on bitumen was studied by Plancher *et al.* (1976). Hopman *et al.* (1999) showed that hydrated lime was more effective than limestone filler in asphalt mixtures. Hydrated lime reacts with acids, anhydrides and 2-quinolones of the bitumen (Petersen *et al.*, 1987). Hydrated lime reduces the content of ketones, anhydrides and carboxylic acid which are products of asphalt aging and are neutralized by the hydrated lime that remains adsorbed on the hydrated lime particle surface (Sebaaly *et al.*, 2006). This fact prevents the particles from

further reaction. Since the polar molecules are reactive, their removal generates an overall slower aging kinetics. The diffusion of the polar molecules to the bitumen-aggregate interface is also inhibited, and only the remaining non-acidic surfactants of the bitumen can move to the bitumen-aggregate interface (Stuart, 1990). The surfactants are amine-based (Durrieu, 1977) and they are not easily displaced by water, unlike anionic surfactants (Curtis *et al.*, 1993). Thus, the increase of moisture resistance by the addition of hydrated lime in asphalt binders is due to the neutralization of polar molecules by HL. Carboxylic acids and 2-quinolones are irreversibly adsorbed on the hydrated lime particle surface. The adsorption prevents their subsequent adsorption by siliceous aggregates to produce a moisture sensitive mixture (Sengul *et al.*, 2012).

The intensity of hydrated lime and asphalt binder interactions depends on bitumen chemistry and on bitumen crude sources (Verhasselt and Puiatti, 2004). Hydrated lime modifies the surface of aggregates, generating a surface composition (with calcium ions) and roughness (with precipitates) more favorable to bitumen adhesion. Hydrated lime can then treat the clay particles adhered to the aggregate surface, inhibiting their deleterious effect on the mixture. Finally, the high porosity of HL explains its stiffening effect above room temperature (EuLA, 2010).

Huang *et al.* (2005) reported that HL is more effective when added to the bitumen rather than its use as aggregate. In this work, hydrated lime was added to asphalt binder as a modifier, as antioxidant agent and not as antistripping agent, in different concentrations corresponding to 1.5, 1, 0.5, and 0.25 wt.% (percentage by total weight of aggregate) as it was used as filler in hot mix asphalts. Comparing the different concentrations of hydrated lime added to binder of 5%, 10%, 20% and 30% (percentage by total weight of binder), the optimum hydrated lime concentration to be added to asphalt binder, which represents a gap in literature, was determined.

2. Experimental

This research was developed by using asphalt cement – CAP 50/70 supplied by Petrobras, with additions of 0% (S0 sample),

5% (percentage by total weight of binder) of hydrated lime (S1 sample), 10% (S2 sample), 20% (S3 sample) and 30% (S4 sample).

Standard laboratory test results for the 50/70 asphalt binder are shown in Table 1.

Physical Properties	Test Method	Value	Specification
Solubility in trichlorethylene (wt.%)	D 2042	99.9	99.5 (min.)
Softening point (°C)	D 36	49.8	46 (min.)
Penetration (0.1 mm)	D 5	54	50-70
Flash point (°C)	D 92	328	235 (min.)
Brookfield viscosity 135°C (cP)	D 4402	337.5	274 (min)
Ductility (25°C) (cm)	D 113	> 150	60 (min)
Brookfield viscosity 177°C (cP)	D 4402	62.6	57-285
Relative density at 20/4°C	D 70	1004	No value
Brookfield viscosity 150°C (cP)	D 4402	168.6	112 (min)

Table 1
Physical properties of asphalt cement.

The properties of the HL used, which was provided by the National Miika Company (Brazil), are shown in Table 2.

The commercial classification of the hydrated lime studied is CHI.

Individual mixtures were prepared with the same blend of asphalt

binder to avoid variability. Pure bitumen and dry hydrated lime were mixed for 10 minutes at a temperature of 120 °C.

Chemical Properties	Value	Unity
Total oxides (nonvolatile basis)	≥ 90	%
Insoluble Materials	≤ 8	%
Total oxides not hydrated	≤ 10	%
CaO associated with CaCO ₃	≤ 6.4	%
CO ₂	≤ 5	%
Sieve Size #30	≤ 0.5	%
Sieve Size #200	≤ 10	%

Table 2
Properties of used hydrated lime.

2.1 Storage stability test

The binder samples were poured into an aluminum tube (32 mm in diameter and 160 mm in height). The tube was sealed and stored vertically in an oven at 163°C for 5 days, then removed, cooled to room temperature, and cut horizontally into three equal sections. The samples taken from the top and

bottom sections of the tube were used to evaluate the storage stability of the hydrated lime modified asphalts by measuring their softening points. Samples are named as S₀, S₁, S₂, S₃, and S₄ that contain the HL concentrations of 0, 5, 10, 20, and 30 wt.%, respectively. If the difference of the softening points

between the top and bottom sections was less than 4°C, the sample was considered to have good high-temperature stability. If the softening points differed by more than 4°C, the hydrated lime modified asphalt was considered to be unstable (Zhang *et al.*, 2010). The analyses were performed in triplicate.

2.2 Standard aging procedures

Following the determination of the properties of the materials used in this study and the preparation of the samples, two conventional tests were used to simulate oxidative aging: Rolling Thin Film Oven Test (RTFOT) and weathering tests.

Short-term aging was addressed by the RTFOT. Binders were aged as described in the ASTM D2872 Standard. In this test, a thin asphalt film of 35 g is continuously rotated in a glass vessel at 163 °C for 85 minutes with an air injection every 3 to 4 seconds. The hardening of the asphalt during the test causes a decrease in penetration and increase in the softening point, according to data reported in literature, it correlated well with the hardening of the binder that occurs during the machining

of an asphalt mixture.

Long-term aging was addressed by the weathering test to simulate the ultraviolet aging. A photo degradation test (ultraviolet ASTM D-4798) was used to address the long-term aging instead of the standard long-term aging test (PAV, ASTM D 6521, AASHTO R28) adopted by the Strategic Highway Research Program (SHRP) of asphalt binder performance testing because the objective is the use of hydrated lime as antioxidant agent to improve the photodegradation resistance of the asphalt binder.

Granite plates of 150mm x 100 mm were coated with 0.6 mm binder film containing the four different concentrations of hydrated lime aiming to simulate the thickness of the asphalt binder coating on

the stone in the hot mix asphalt (Araújo *et al.*, 2013). The granite stones coated with S₄ to S₁ samples containing 5 wt.%, 10 wt.%, 20 wt.% and 30 wt.% of hydrated lime modified binder already aged using the RTFOT testing were then exposed in the Q-Sun Xe-1 weathering chamber, according to the ASTM D-4798 (2008) standard, in cycles of 80 minutes (64 minutes of xenon radiation, and 16 minutes of xenon radiation and water). The specimen temperature was 60°C and irradiation was at 340 nm = 0.35 W/m². Variable power from 3500 to 6500 W maintained the intensity of irradiation (Araújo *et al.*, 2013). The samples were exposed in the weathering chamber for 10, 20, 30, 40, 50, 100, 150, 200 and 250 hours.

2.3 Thin layer chromatography with flame ionization detection (TLC-FID)

The TLC, 2% (w/v) solutions of

the unaged and aged base asphalts

contained dichloromethane, and 1μL

sample solution was spotted on chromanols using a spotter. The separation of bitumen into four generic fractions (saturates, aromatics, resins and as-

phalthenes) was performed in three-stage developments using n-heptane, toluene and dichloromethane/methanol (95/5 by volume), respectively. The

fractions were determined by means of Iatroscan MK-5 analyzer (Zhang *et al.*, 2010). The analyses were done in triplicate.

2.4 Fourier transform infrared (FTIR) spectroscopy

After the weathering tests, the binder samples were submitted to the FTIR – Fourier Transform Infrared Spectrometer analysis using Attenuated Total Reflection (ATR). The ATR technique is suitable for tackling samples that offer serious problems in the normal transmission mode, as the sticky films, which is the case of the binder. With ATR, the layer thickness is not critical, as the small penetration depth

is related to the ATR phenomenon itself (Willis *et al.*, 2010). Fourier Transform Infrared Spectroscopy (FTIR) was performed using a Thermo-Nicolet Nexus 470 spectrometer, and spectra were obtained in absorbance units in the range of 4000 to 400 cm^{-1} recorded with 32 accumulations reading and resolution of 4 cm^{-1} , by attenuated total reflection with a 300 μm ZnSe crystal in reflection mode,

under a nitrogen atmosphere. The FTIR spectra were analyzed using software OMNIC. The spectra, after application of baseline correction (nonlinear), were normalized and the Carbonyl Index (I_c) was used to assess aging. Carbonyl Index is the ratio of peak area of νCH_2 (1454 cm^{-1}) and νCH_3 (1375 cm^{-1}) and the peak area of $\nu\text{C}=\text{O}$ (1700 cm^{-1}). The analyses were performed in triplicate.

3. Results and discussion

3.1 Storage stability

Compatibility between the hydrated lime and the asphalt binder is necessary to avoid separation during storage, pumping, and asphalt binder application and to achieve the expected properties of the pavement (Brandrup and Immergut, 1989). A softening point

variation has been accepted to evaluate the asphalt binder stability (Zhang *et al.*, 2011). The high temperature stabilities of the modified binders are shown in Figure 1.

Only sample S4 (30 wt.% of hydrated lime) was considered unstable.

As the difference in the softening point before and after the storage for mixture S3 was close to 4°C , the sample was considered a stable mixture. The critical concentration of lime in the binder regarding the stability condition is 20 wt.% of hydrated lime.

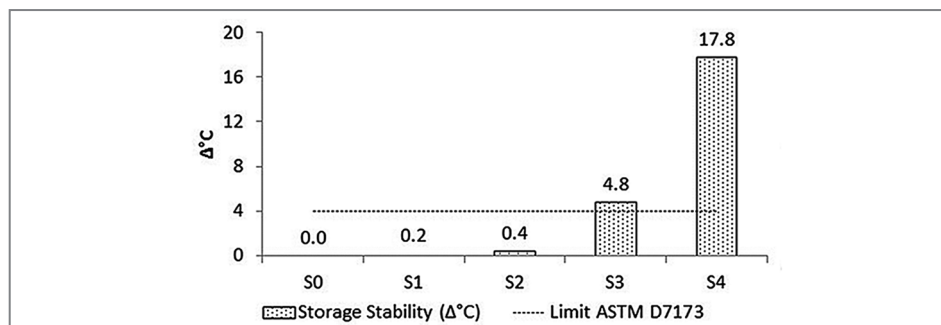


Figure 1 Storage stability of hydrated lime modified binders.

3.2 SARA analysis

To clarify the effect of hydrated lime addition on the chemical composition of asphalt binder, Thin-Layer Chromatography with Flame Ioniza-

tion Detection (TLC-FID) was used before and after aging to analyze the four generic fractions, namely saturates, aromatics, resins and asphaltenes

(Zhang *et al.*, 2011). The results of samples before and after 250 hours of weathering testing are shown in Figure 2.

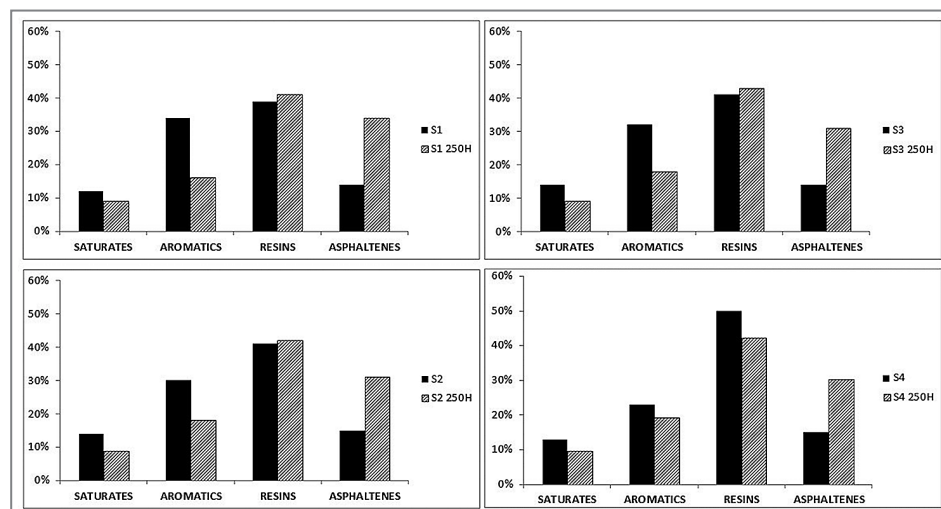


Figure 2 SARA results of hydrated lime asphalt binder before and after 250 hours of weathering testing.

In asphalt and saturated fractions, significant changes caused by the addition of hydrated lime were not observed. In relation to the asphalt binder, the addition of 30 wt.% of hydrated lime produced a decrease in the aromatic content of material and an increase of resin concentration for mixture S4. The reduction of the concentration of aromatics in the mixture with the highest lime content can be explained by adsorption of the aromatic fraction by

the excess of hydrated lime.

For the samples aged for 250 hours, reductions in aromatic concentrations were observed. Aromatics first become resins and then asphaltenes (Farcas, 1998). There was no significant loss of saturate fractions due to the oxidation process. A significant increase of asphaltene fraction was observed between 100 and 143% (Figure 2). An increase of 5% of resin fraction for S1, S2 and S3 samples was identified after ag-

ing. The S4 sample presented a reduction in the resin fraction after aging. This can be explained by the adsorption of aromatic fraction by the excess of hydrated lime which resulted in a lower concentration of aromatics available for oxidation.

Using the values obtained by SARA analysis before and after aging, the Colloidal Instability Index (ICI) was calculated. The index used to characterize the colloidal equilibrium among the phases is calculated:

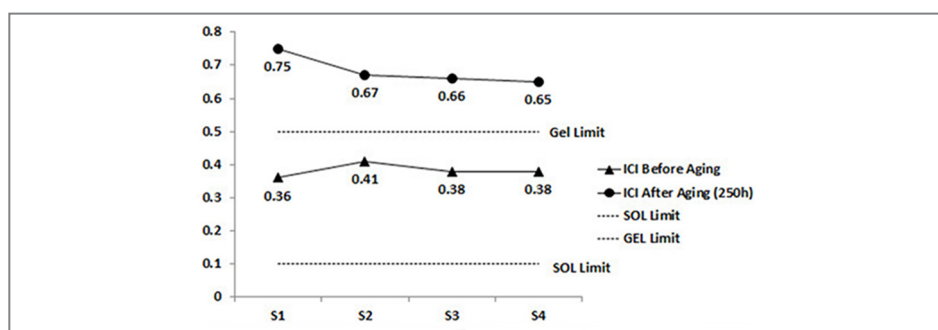
$$I_{ci} = \frac{\text{Saturated} + \text{Asphaltenes}}{\text{Resins} + \text{Aromatics}} \quad (1)$$

The ICI index values less than 0.1 are characteristic of unstructured binders, whose behavior can be called SOL. The ICI values greater than 0.5 represent a well-

structured binder with GEL behavior. Intermediate values between 0.1 and 0.5 represent an asphalt binder called sol-gel (Gaestel *et al.*, 1971). It is considered that

the larger the ICI, the greater the asphalt colloidal instability. All samples had a sol-gel behavior before aging and a gel behavior after aging (Figure 3).

Figure 3
Colloidal Instability Index results.



3.3 Infrared spectroscopy analysis

The monitoring of the aging was performed by studying the spectra regarding the characteristic band centered at 1700 cm^{-1} of the carbonyl function ($\text{C}=\text{O}$), the peak at 1032 cm^{-1} of the sulfoxide

function, the peak at 1600 cm^{-1} of the aromatic function ($\text{C}=\text{C}$), the peak at 1454 cm^{-1} for (CH_2), and the band centered at 1375 cm^{-1} (CH_3).

To eliminate discrepancies regard-

ing thickness of the film, the carbonyl index, sulfoxide index and the aromatic index were calculated in relation to the CH_3 and CH_2 peak areas (Araújo *et al.*, 2013):

$$I_c = \frac{\text{Area of the carbonyl band centered around } 1700 \text{ cm}^{-1}}{\text{Area } \text{CH}_2 \text{ centered around } 1454 \text{ cm}^{-1} + \text{Area } \text{CH}_3 \text{ centered around } 1375 \text{ cm}^{-1}} \quad (2)$$

$$I_s = \frac{\text{Area of the Sulfoxide band centered around } 1032 \text{ cm}^{-1}}{\text{Area } \text{CH}_2 \text{ centered around } 1454 \text{ cm}^{-1} + \text{Area } \text{CH}_3 \text{ centered around } 1375 \text{ cm}^{-1}} \quad (3)$$

$$I_{\text{Aro}} = \frac{\text{Area of the Aromatic band centered around } 1600 \text{ cm}^{-1}}{\text{Area } \text{CH}_2 \text{ centered around } 1454 \text{ cm}^{-1} + \text{Area } \text{CH}_3 \text{ centered around } 1375 \text{ cm}^{-1}} \quad (4)$$

For quantitative analysis, the peak area and not the peak height was taken into consideration. Peak height and band area in a FTIR spectrum are widely used to indicate the concentration of a chemical bond. For asphalt oxidation quantitative analysis, the band area rather than the peak height is used for two reasons: (1) band areas provide less variation (Yut and Zofka, 2011) and (2) several vibrations may occur in the same band so that it is

difficult to obtain the single peak height (Lamontagne *et al.*, 2001).

Sample S4 (30 wt.% of lime) presented the highest carbonyl index after 30 h of weathering tests. The carbonyl index of the S1 sample presented a high value at 10 h of aging, after the RTFOT testing. The S1 sample, with the lowest content of hydrated lime, showed the lowest photodegradation resistance and the highest value of carbonyl index. The

carbonyl index of S3 and S2 samples showed the maximum peaks of carbonyl index after 50 h and 200 h of weathering tests, respectively. After 250 h of the weathering test, S3 (20 wt.% of hydrated lime) and S2 (10 wt.% of hydrated lime) samples showed higher photodegradation resistance values compared to the other samples, considering the carbonyl index. The carbonyl index values of all binders are shown in Figure 4.

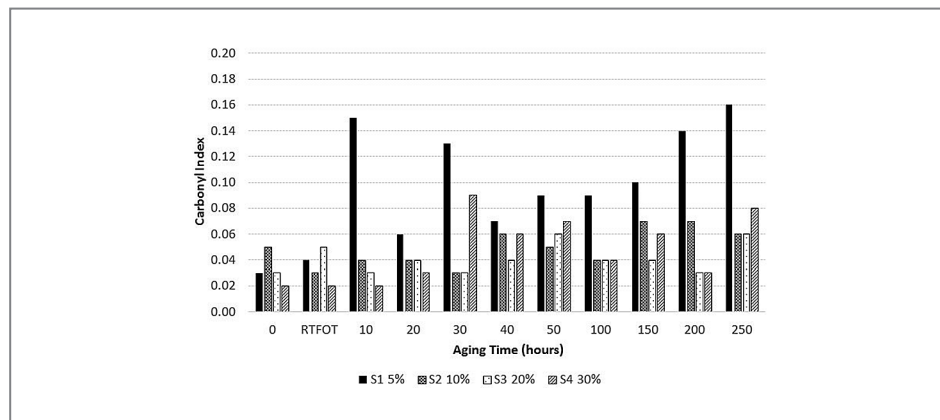


Figure 4 Carbonyl Index (I_C) of the S1, S2, S3 and S4 samples for different aging times.

According to Figure 5, samples S3 and S4 reached the highest sulfoxide index after 10 h of aging. For S4 (30 wt.% of hydrated lime) and S3 (20 wt.% of hydrated lime) samples, a sharp increase in sulfoxide index after the RTFOT test was observed. This fact can be explained by the high temperature (163°C) used in the

RTFOT test (Petersen and Glaser, 2011). For sample S2 (10 wt.% of hydrated lime), a reduction in the sulfoxide index after the RTFOT test was identified; this fact can be explained by the high value of the sulfoxide index before aging. When the sample was heated, the thermal decomposition of sulfoxides was favored over

the sulfoxide formation (Ouyang *et al.*, 2006). The values of sulfoxide index were similar for all asphalt binder samples after 10 hours of aging. This can be explained by the low concentration of sulfur of the asphalt binder used, since it is not modified with polymeric additives such as styrene-butadiene-styrene.

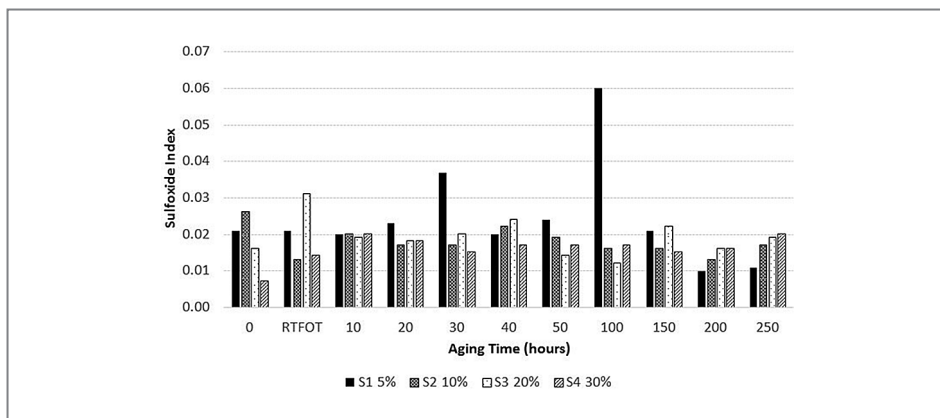


Figure 5 Sulfoxide Index of the S1, S2, S3 and S4 samples for different aging times.

For sample S4 (30 wt.% of hydrated lime), an aromatic index reduction was identified after 20 hours in the weathering chamber as shown in Figure 6. Peaks of maximum values of aromatic index were observed at 30 h, 150 h and 250 h of aging.

At 10 h and 200 h, the index reached its lowest value and the aromatic concentration decreased by 54% compared to the initial value. The S3 sample reached the lowest content of aromatics after 30 h of aging, and the aromatic fraction increased at the end

of the weathering test. The same behavior was observed for the S2 sample. The S1 sample showed high aromatic index values at 10 and 30 h, after which the aromatic index decreased and this sample showed the highest aromatic index after 250 h of aging.

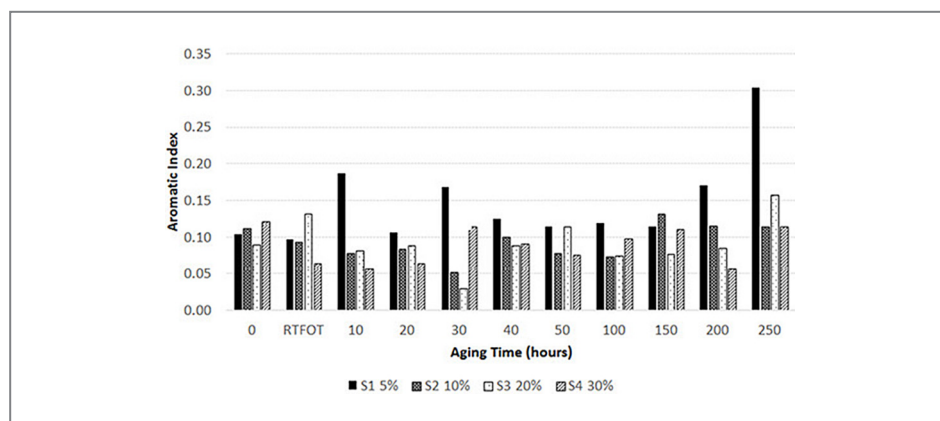


Figure 6 Aromatic Index of the S1, S2, S3 and S4 samples for different aging times.

The reduction of the aromatic index may have occurred because of oxidative reactions that led to the opening of aromatic

rings and formation of quinones. The increase in aromatic compounds can be explained by the naphthenic cluster

dehydrogenation process, cleavage with short aliphatic side chains, and increased condensation (Siddiqui and Ali, 1999).

4. Conclusions

This study was conducted to determine the optimal hydrated lime concentration to improve photodegradation resistance of the asphalt binder. A summary of findings is presented as follows:

- According to the stability test, the hydrated lime concentration value close to 20 wt.% is the critical point at which there is a risk of segregation of the asphalt binder and hydrated lime during transportation and storage.

- For the samples aged for 250 hours, a decrease of saturated and aromatic fractions was observed for all samples. A significant increase of the asphaltene fraction occurred for the lime modified asphalt binders. An increase of 5% of resin

was obtained for S1, S2, and S3 samples after 250 hours of aging. The S4 sample presented a reduction in the amount of resin after aging.

- The FTIR spectroscopy showed an increase of carbonyl groups after the weathering test, a result of oxidation of the asphalt binder. The binders modified with hydrated lime showed a different aging index (IC=O) according to the concentration used. Considering the carbonyl index, after 250 hours of the weathering test, the S2 sample, with 10 wt.% of hydrated lime in asphalt binder showed a higher photodegradation resistance than samples with 30 wt.%, 20 wt.% and 5 wt.% of hydrated lime.

- The sulfoxide index (IS=O) was

not considered as a criterion for the selection of the optimized content of hydrated lime in the asphalt binder due to the low concentration of sulfur in the asphalt binder and due to a lack of significant index change after 10 hours of aging.

- For the aromatic index (IC=C), a reduction in the aromatic fraction in the initial stages of aging was observed. The evaluation on the IC=C was not conclusive regarding the hydrated lime concentrations.

- As the results of the storage stability tests, colloidal instability index, and carbonyl index, the optimum concentration of hydrated lime in the asphalt binder was 10 wt.%.

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