EFFECT OF CALCAREOUS FILLERS ON BITUMINOUS MIX AGING

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Abstract

The aim of this paper is to explain phenomena associated with bitumen aging and establish design patterns that contribute to optimizing the aging resistance of bituminous mixes based on the rational addition of calcareous fillers (hydrated lime and limestone filler).

The potential benefits of using fillers to improve aging resistance are well-known. However, concepts related to the biphasic filler-bitumen system affecting some of the main characteristics of mixes are not always considered.

In this paper, a new study procedure, “Universal de Caracterización de Ligantes” (UCL®), a method for characterizing both conventional and polymer-modified binders developed at the Technical University of Catalonia, has been applied. In addition, rheological tests have been performed with the dynamic shear rheometer and non-routine procedures such as macromolecular analysis (e.g. gel-permeation chromatography and infrared spectroscopy). These techniques have provided greater insight into the causes and effects of aging based on the changes undergone by the bituminous binder.

The obtained results show the advantages of incorporating calcareous fillers into bituminous mixes provided that filler content does not exceed a “critical concentration” determined by the type of filler and binder to be used. Moreover, some variations on the concept of critical concentration of filler in bituminous mixes have been found, notably those resulting from the degree of mix aging and use of polymer-modified bitumens.

INTRODUCTION

Aging is one of the reasons for the failure of bituminous wearing courses. The consequences of this process are a decrease in the level of comfort and safety and an increase in pavement maintenance and rehabilitation activities, which results in higher costs.

Most of the research to date has focused on binders in isolation. The "aging" factor has been assessed by accelerated simulation of some of the mechanisms affecting this deterioration process.

Petersen (*1*), Khandal and Chakraborty (*2*) and other researchers observed that the primary causes of aging are light fraction volatilization and oxidation resulting from a "dual mechanism" by which oxygen is combined with highly reactive elements of the hydrocarbon. This gives rise to the appearance of cyclic-aromatic compounds. A slow oxidation process of benzylic carbons, whose main final product is ketones (*3*), is then initiated.

Classical characterization methods do not allow material behavior to be accurately predicted during service time since void content, aggregate-binder interaction and the effect of mineral filler and other additives dramatically change the characteristics of bitumen in the final mix (*4*). The irruption of polymer-modified binders into the road industry brought into question the validity of these processes.

The past few years have witnessed the emergence of new techniques for simulating transformation processes undergone by bitumen in bituminous mixes as it ages and the way it acts. Such methodologies consider not only the intrinsic characteristics of binders but also their properties as part of a mix with aggregates and filler (*5, 6*). For this reason, these procedures can be described as "functional".

It is well-known that filler addition to mixes improves their physical properties: bitumen can be thickened, thus modifying its viscous flow, adherence can be enhanced and the layer covering the aggregates thickened in order to delay the aging process (*7, 8*). Chemical evaluation studies on bitumen functional groups (*9*) stated that calcareous fillers cause both catalysts in bitumens favoring oxidation and polar molecules to be captured. These molecules, if free, would interact with products resulting from oxidation, thus giving rise to the appearance of ketones, anhydrids, etc. As a result, viscosity would increase. Gubler et al. (*10*) observed a physical effect, namely that small filler particles prevent oxygen diffusion through bitumen.

Despite the advantages, filler addition must be rationally controlled because filler type and content should be determined according to the desired volumetric and physical-mechanical properties (*11*). In previous studies, Ridgen (*12*) and Ruiz (*13*) suggest limiting filler addition to avoid exceeding a certain degree of volume concentration of the filler-bitumen system since excessive filler leads to high stiffness.

Filler "critical concentration" is reached when the mastic starts to stiffen. The system becomes more fragile and certain desired characteristics, such as flexibility, cohesion and durability, degrade. This process is intensified at low temperatures. The critical concentration corresponds to a dispersion of filler particles in the bitumen moving as freely as possible but in contact with each other, In other words, the critical concentration is attained when the applied stress is consumed in the viscous deformation of the continuous bituminous medium and frictional resistance between particles is at a minimum. Such a packing is expected in the sediment obtained by simple settling of filler dispersion in a fluid medium chemically related to bitumens, like kerosene. Ruiz (*13*) proposes a simple sedimentation test to find the "critical" value which guarantees mastic viscous behavior. This test is known as "Sediment Concentration", or most commonly, "Critical concentration" (Argentinean standard IRAM

1542):

$$C_s = \frac{P}{V.G_f} \tag{1}$$

where C_s is the critical concentration, P is the filler dry weight, V is the volume of filler settled in anhydro kerosene for 24 hours and G_f is the filler specific weight.

When filler is added to mixes, bituminous mastic viscosity increases gradually with increasing the volumetric concentration:

$$C_v = \frac{Filler\_Volume}{Filler\_Volume + Bitumen\_Volume} = \frac{\frac{P_f}{G_f}}{\frac{P_f}{G_f} + \frac{P_b}{G_b}} \tag{2}$$

where P_f is the filler weight percentage, P_b is the bitumen weight percentage, G_f is the filler specific weight and G_b is the bitumen specific weight.

In the case of distillation bitumens, when $C_v > C_s$, the biphasic system stops being viscous and an internal structure determining a net non-Newtonian flow appears, which renders the mix stiff.

The objective of this paper is to show the role of calcareous fillers against bitumen aging in bituminous mixtures of in-service pavements. Thus, design criteria for the optimization the aging resistance of mixtures are proposed.

In this work, the results of the latest research on advantages of volumetrically incorporating fillers into bitumens to increase aging resistance are presented. The main tool employed to study the "aging factor" is "Universal de Caracterización de Ligantes (UCL$^{®}$)". This method allows assessing the functional response of several different types of mastics to mixes with preset grading characteristics by quantifying disgregation loss resulting from reduced cohesive properties (caused by aging and extreme thermal conditions). The analyzed variables are binder and filler type and nature, and relative proportions between these two materials. Additionally, aged binder behavior has been assessed by rheological testing and macromolecular analysis. Observation of changes has provided greater insight into the causes and effects of aging.

METHODOLOGY

UCL$^{®}$ is based on the Cántabro test. This method is used to assess filler functional properties; that is, the degree of cohesion of the resulting standard mix is determined and variations of this degree produced by temperature, the action of water and aging are observed (*14*).

The standard mix was prepared with an aggregate grading ranging from sieve size No.4 to No.30, thus obtaining 28% of void content. Marshall compaction was used to prepare test specimens. This process consisted of 50 blows each side of the test specimen, with a binder content of 4.5% by mass of aggregate. An equiviscosity criterion was applied (3 Poise) to select the mix compaction temperature. A high quality crushed granite aggregate and two polymer-modified bitumens (SBS and EVA) and a 70/100 conventional bitumen were chosen. Hydrated lime and limestone dust were employed as filler materials, the proportions varying according to their corresponding critical concentrations ($C_s = 0.17$ and $C_s = 0.37$, respectively). Table 1.

Filler type	Specific gravity (g/cm^3)	Critical concentration	Cv/Cs Ratio	Filler content (g)	Filler/bitumen Ratio (in weight)
Without filler			0	0	0
Hydrated Lime	2.351	0.165	0.5	9.5	0.21
			1.0	21.2	0.47
			1.3	28.1	0.62
			1.5	35.1	0.78
Calcium carbonate	2.771	0.370	0.5	27.5	0.61
			1.0	71.1	1.58
			1.3	111.6	2.48
			1.5	151.0	3.36

Table 1 Characteristics and contents of filler used in the study

The experimental process was developed at different test temperatures and aging times, and Performance Curves were obtained to assess the effect of these variables on mixes.

Test temperatures ranged from -10 to 70°C and aging times were 0, 2, 4 and 7 days. The test specimens were placed in an oven and heated at 80°C with forced ventilation. Subsequently, abrasion loss was measured with the Cántabro test (NLT-352), which consists of subjecting test specimens to abrasion at 300 drum revolutions in a Los Angeles drum with no abrasive charge to determine percentage particle loss of the specimen.

After specimen testing, binders were recovered by controlled distillation with dichloromethane, a solvent which does not require high temperatures to "wash" the mix, and they were then subjected to several tests, namely viscosity, dynamic shear rheometer and macromolecular analysis.

Bitumens were characterized in three different states: "virgin"; from tested specimens without laboratory aging to simulate the short-term "moderate" aging which occurs during specimen preparation; and after extraction from tested specimens subjected to laboratory aging, as specified by the method. Binder behavior was then compared to assess changes in bitumens and the protective role of filler against aging.

Viscosities were determined with a rotational Brookfield RVD III viscometer. Binders were tested at 100, 135 and 150°C whereas conventional bitumen was tested at 60°C.

For complex shear modulus analysis, a Dynamic Shear Rheometer (DSR) was used at an oscillation speed of 10 radians/second, which corresponds to a 1.6 Hz frequency.

Molecular weights and distributions were determined by gel-permeation chromatography using LKB-2249 equipment, with µ-Styragel columns (10^5 and 10^2 Å) and a Shimadzu UV detector at 254nm. Tetrahydrofuran was used as a solvent.

Infrared spectroscopy was performed with a Shimadzu IR-435 spectrometer.

ANALYSIS OF RESULTS

UCL Method

The "Performance Curves" reflect thermal susceptibility of the binder as assessed from its cohesion properties in the standard mix at a range of potential service temperatures. To verify how this property is affected by binder aging, several tests were conducted after subjecting specimens to different laboratory aging periods. The so-called "Set of Performance Curves" where each curve represents loss changes with temperature for each degree of aging was then elaborated.

Figure 1 shows the Set of Performance Curves for the standard mix with SBS polymer-modified bitumen and increasing proportions of hydrated lime.

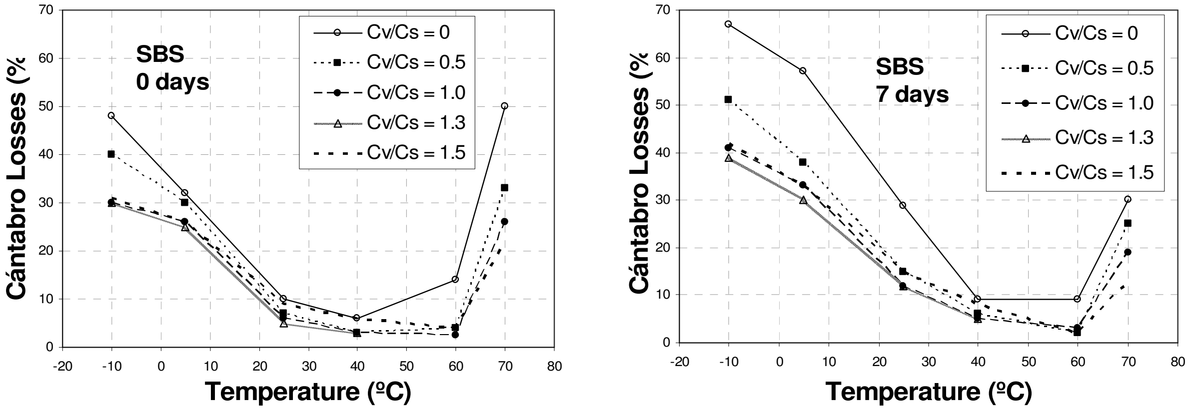

FIGURE 1 Performance Curves. Mixtures with SBS Modified Bitumen and Hydrated Lime. Effect of Filler Content and (a) 0 days of Aging and (b) 7 days of Aging.

Observation of the mix without added filler reveals that losses were greater with low and moderate temperature aging. It is also observed that the slope of curves increased gradually, particularly at 5 and 25°C. For temperatures above 50°C, losses decreased with aging as a result of binder hardening, which enables it to withstand high temperatures before softening.

Losses decreased with higher proportions of hydrated lime to a C_V/C_S = 1.3 relation for all test temperatures and aging stages. However, the effect was greater at low temperatures, where filler protection implied lower binder hardening and greater mix cohesion. Note that in Figure 2, for example, the curve for a 7-day aging period of the mix with a lime concentration of C_V/C_S = 0.5 is similar to that of the non-aged specimen without added filler. This means that hydrated lime addition, even when in moderate proportions, inhibited long-term aging effects almost completely.

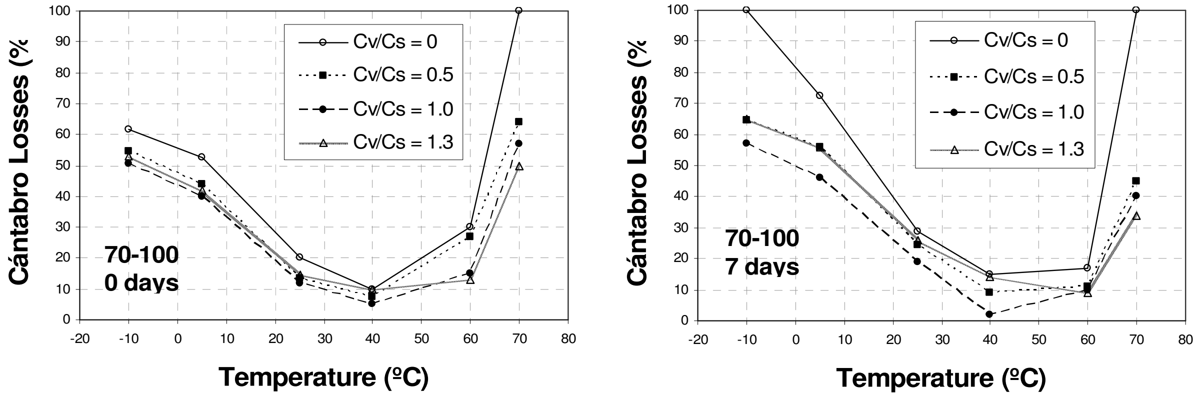

FIGURE 2 Performance Curves. Mixtures with Conventional Bitumen and Hydrated Lime. Effect of Filler Content and (a) 0 days of Aging and (b) 7 days of Aging.

This trend is slightly inverted for a concentration of C_V/C_S = 1.5. In this case, a greater influence of mastic stiffness than bitumen softening resulting from filler protection can be noticed.

No significant changes are observed at moderate and high temperatures. The reasons for this can be that losses are low; therefore, small percentage variations are negligible. Second, the mastic stiffens, although filler protection against aging implies lower binder hardening.

In mixes with EVA-modified bitumen, losses were generally greater than with SBS-modified bitumen under equal filler addition and aging conditions, the reason being the better binding and elastic properties of the latter.

Much higher losses were observed when mixes were prepared with conventional binder than when modified binders were used, particularly at extreme test temperatures, see Figure 2.

Thus, hydrated lime addition can be advantageous provided the critical concentration is not exceeded (i.e. $C_v/C_s = 1.0$) since too much filler resulted in greater losses at low and moderate temperatures, especially in aged specimens.

In mixes where limestone dust was used as filler, results were in reasonable agreement with those obtained for mixes with hydrated lime content at equal *Cv/Cs* relations. However, for equal concentrations, a higher weight proportion of limestone filler than of hydrated lime was necessary. For instance, for $C_v/C_s = 1$, a filler/bitumen relation of 0.47 was needed when using lime whereas the proportion rose to 1.58 when limestone dust was added to the mix.

If equal weight proportions were compared, the performance of hydrated lime would be much better.

The obtained results made it possible to estimate the optimum *Cv/Cs* relations for each mix type according to its aging resistance using the UCL method.

Figure 3 plots "Cántabro Losses vs. Filler Content" for SBS-modified bitumens and hydrated lime at different test temperatures and aging times. For the worst conditions (-10°C), the optimum value was 1.3 approximately, although mixes behaved similarly between 1.0 and 1.5. Higher values indicated an excess of filler. At other low and moderate temperatures, both types of mixes behaved similarly. This is generally in agreement with results from other studies following the same research line where the mechanical response of standard mixes was assessed with the Barcelona Tracción Directa test (BTD$^{\circledR}$) (*15*). At high temperatures (70°C), losses diminished as filler content was increased. No optimum value was established.

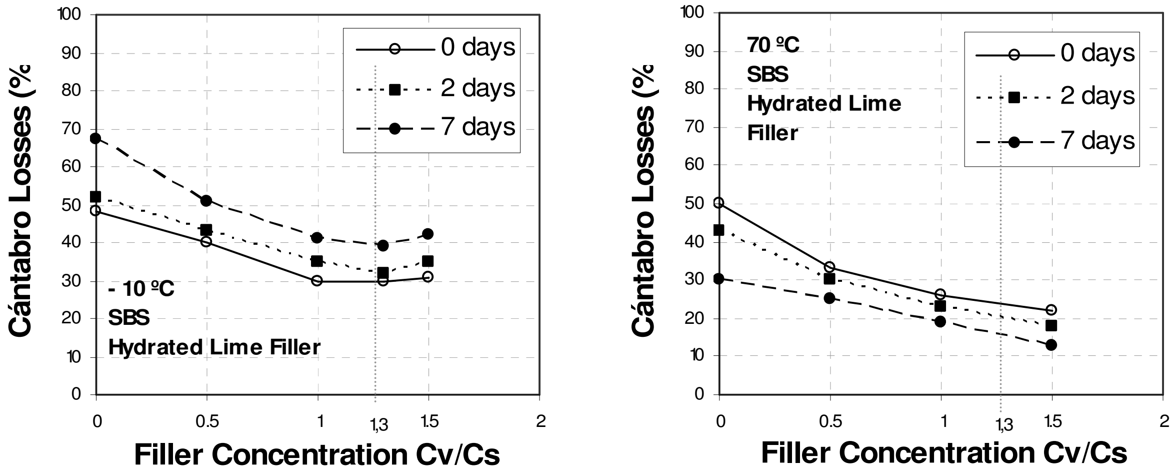

FIGURE 3 Cántabro Losses Versus Filler Concentration. Mixtures with SBS Modified Bitumen and Hydrated Lime tested at (a) -10ºC and (b) 70ºC for Different Aging Periods.

For conventional bituminous mixes, results suggest that the optimum filler concentration corresponds to a *Cv/Cs* = 1.0 relation, which is in good agreement with Ruiz's studies and subsequent experience, see Figure 4. Losses increased for *Cv/Cs* = 1.3, particularly as the mix aged. This situation, which is common for all assessed low and moderate temperatures and for both fillers, would be indicative of a shift in the optimum filler content towards slightly lower values than those of the critical concentration (*Cv/Cs* = 0.8 or 0.9) as aging progresses.

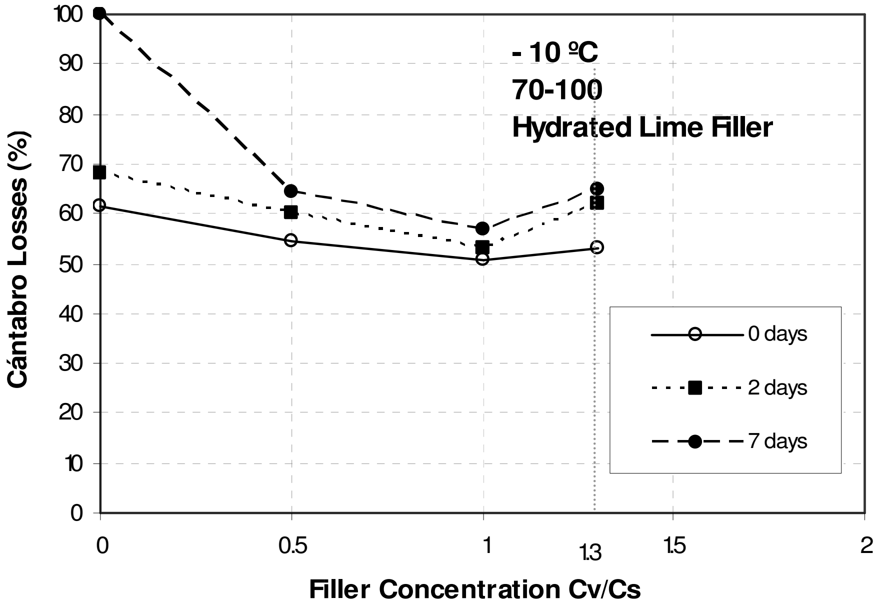

FIGURE 4 Cántabro Losses Versus Filler Concentration. Mixtures with Conventional Bitumen and Hydrated Lime tested at -10ºC for Different Aging Periods.

Dynamic Viscosity

Figure 5 summarizes the consequences of conventional bitumen aging in rotational viscosities at 60°C and the benefits of calcareous (hydrated lime, in the present case) filler addition to mixes. These results fit with findings obtained by Huang and Robertson (cited by Sebaaly (*16*)).

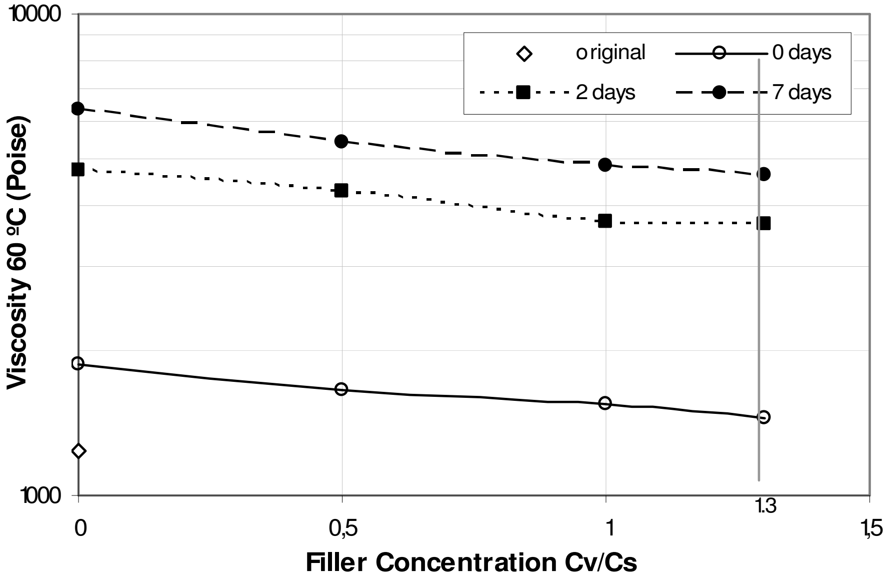

FIGURE 5 Viscosity at 60°C of 70-100 Binder Extracted from Samples with Hydrated Lime Versus Filler Concentration.

For example, it can be deduced that an accelerated 7-day laboratory aging process, which is equivalent to long-term in-service aging of mixes with *Cv/Cs* = 1.0, is similar to an accelerated 2-day laboratory aging process (medium-term in-service) for mixes without filler content (*17*). Therefore, filler addition prolongs the useful life of mixes.

For equal filler contents and aging periods, hydrated lime performed better than limestone dust in dynamic viscosity tests.

The base bitumen of the modified binders subjected to aging hardened and their polymers degraded. Such degradation might have been caused by swelling and dissolution of the polymers after being heated at high temperatures as well as by bond breaking, cross linking, radiations and chemical reactions. The combined action of hardened bitumen and a degraded elastomeric phase brings about changes in the original characteristics of polymer-modified bitumens. For this reason, performance differs from that of conventional bitumens.

Whatever the sense and magnitude of the observed rheological variations, only the most significant comparative changes resulting from the use of fillers and the employed concentrations have been considered for the present work, which aims to estimate the range of influence of mineral filler addition to mixes.

Figure 6 illustrates the case of EVA-modified bitumen. The response of SBS-modified bitumen was similar.

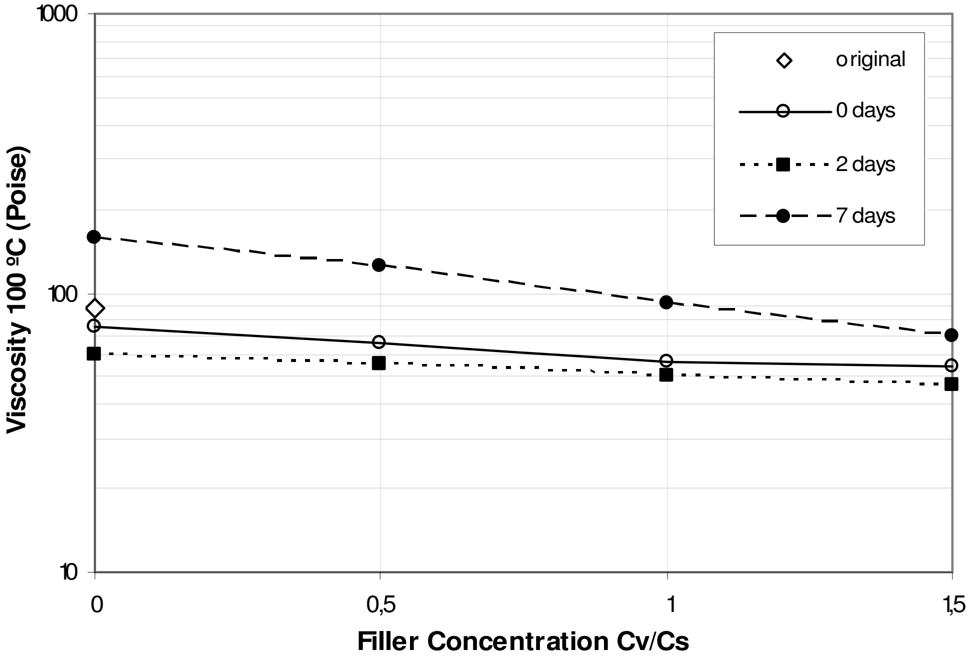

FIGURE 6 Viscosity at 100ºC of EVA Modified Binder Extracted from Samples with Hydrated Lime Versus Filler Concentration.

The figures clearly show the “competition” between the hardened bitumen and the degradation of the polymeric net. When the polymer-modified bitumens used for the standard mixes with appropriate filler content were subjected to moderate aging, viscosity decreased with respect to that of the original filler. This demonstrates the pre-eminence of polymer chain scission. However, when the base bitumen was subjected to more intense aging and hydrated lime content was low, the bitumen appeared unprotected. As a result, the base bitumen had eventual greater consistency, and viscosity increased and approached or even exceeded the original viscosity because of hardening of the base bitumen and the additional effect of polymeric cross-linking.

Macromolecular analysis techniques

Physical tests are often insufficient to interpret the mechanisms responsible for bitumen aging. In the case of conventional binders, the effect of oxidation and volatilization of the lightest bitumen compounds may account for the aging process. Based on this information, it is possible to know how to slow the process. In polymer-modified bitumens, the combined effects of base bitumen aging and polymer degradation often mask structural changes in the binder which are not detected by laboratory tests. In view of this problem, chromatography and infrared spectroscopy techniques are proposed to understand aging-associated processes.

In this work, tests on conventional bitumens recovered from mixes with and without filler content and subjected to different laboratory aging periods in UCL test specimens are presented.

Gel-Permeation Chromatography (GPC)

The chromatographic profiles of conventional bitumen exhibit a unimodal distribution because of the column system used in this analysis.

As an example, Figure 7 plots the profiles of the virgin bitumen and that obtained from two 7-day aged specimens, one without filler and one with a hydrated lime proportion of C_v/C_s = 1.0.

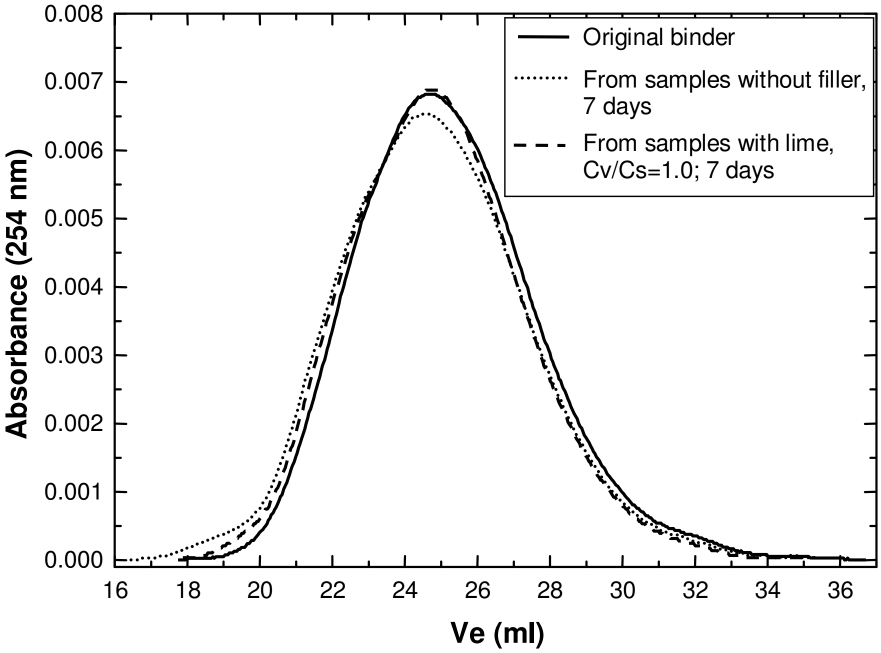

FIGURE 7 Elugrams of Aged Bitumen at 7 days. Mixture without Filler and with Hydrated Lime Filler (Cv/Cs 0 1.0) and with Virgin Bitumen.

In comparison with the virgin bitumen elugram, the distribution curve of the aged sample without filler widens and shifts slightly to the left. As a result, the number of low molecular size products decreases proportionally and that of the most polar and associated ones increases noticeably, thus appearing a tail at low V_e. In contrast, the aged binder of the sample containing an optimum proportion of hydrated lime clearly reflects the protective effect of the filler. In this case, the distribution curve becomes narrower and the size of the tail is reduced at low elusion volumes.

These chromatographic tests provide clear evidence of the advantages of using limestone as mineral filler to improve the aging resistance of conventional bitumens.

Infrared Spectroscopy (IR Analysis)

The analyzed samples are the same as above. Each sample was dissolved in Cl_4C at a 20 mg/ml concentration. IR analysis was performed by casting the solution onto a NaCl window and then evaporating it in an oven.

Figure 8 shows the obtained spectra. The abscissa values correspond to the wavenumbers (cm^{-1}). On the 1790/1690 cm^{-1} band, no singularities are observed in the virgin bitumen curve. However, a change in the spectrum of the 7-day aged sample without added filler can be seen. The change is not so noticeable in the Cv/Cs = 1.0 sample, suggesting a lower degree of oxidation due to the beneficial effect of hydrated lime against aging. These bands correspond to the C=O bond stretching.

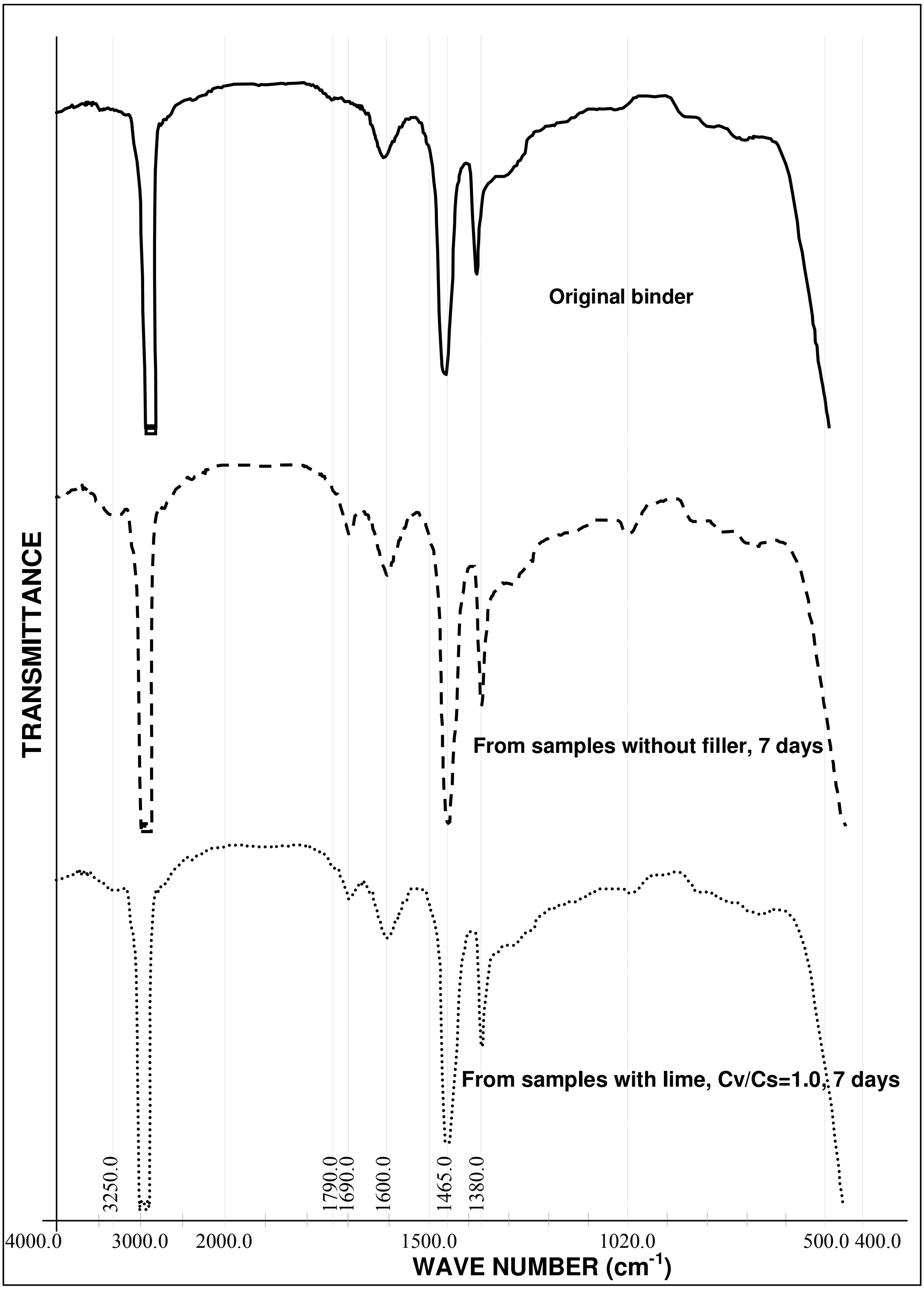

FIGURE 8 IR Analysis. Conventional Bitumen for Different Aging Periods.

A small band can be observed in the aged binder without filler at 3250 cm^{-1}, which corresponds to the O-H bond stretchings that result from the bitumen oxidative degradation. This band is not observable in the virgin bitumen spectrogram and hardly noticeable in that of the aged bitumen of the sample with filler.

The band appearing at 1020 cm^{-1}, assigned to the S=O stretching of the sulphoxide formed by oxidation of the S atom in the bitumen, is also indicative of the effects of aging since it is hardly noticeable in the virgin bitumen, very noticeable in the aged bitumen of the mix without filler and attenuated in the aged bitumen of the mix prepared with a filler content of Cv/Cs = 1.0.

Testing was performed by transmittance. As a consequence, only a qualitative analysis could be conducted.

Dynamic Shear Rheometer (DSR)

The intrinsic rheological characteristics of binders have also been studied by analysis of the complex shear modulus with a Dynamic Shear Rheometer (DSR).

The test temperature was 60°C, which is representative of bitumen behavior under in-service conditions approximately (in summer, Mediterranean climate). The linear viscoelastic behavior of the analyzed binder was tested by controlled deformation measuring the effort required to move the plate at a 1.6 Hz frequency.

Figure 9 presents a brief analysis of the obtained results. It can be seen that aging leads to an increase of the complex shear modulus (G*) and a decrease of the phase angle (δ). Such changes are explained by the effects of light fraction oxidation and volatilization: proportional rise of asphaltene content, decrease in the number of polar compounds, intermolecular associations and changes in the colloidal structure. This causes the binder to be more complex, and therefore its behavior progressively deviates from the Newtonian flow.

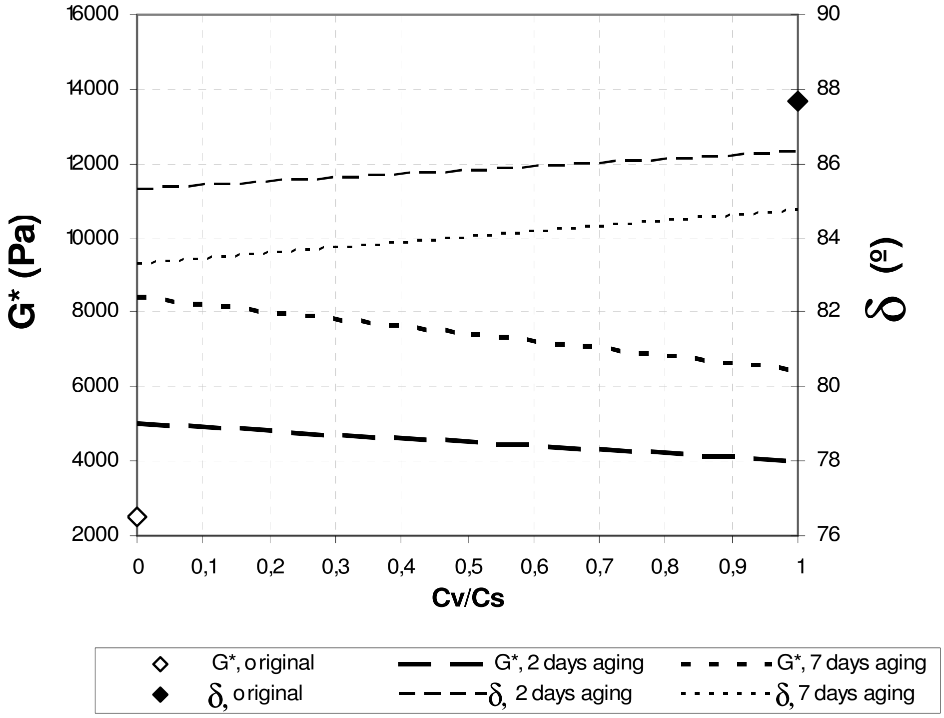

FIGURE 9 DSR tests. Variation of the Complex Shear Modulus and the Phase Angle with Filler Concentration for Different Aging Conditions.

The modulus value increases as aging proceeds but at a lower rate when the bitumen is "protected" by the filler. Observation of the decrease in the phase angle produced by aging reveals the same phenomenon: as in the case of G*, in the aged sample, the value of δ progressively approaches that of the virgin bitumen as the C_v/C_s relation is augmented by the effect of the filler. This is indicative of better aging resistance.

CONCLUSIONS

The results of this work agree in general with those of previous research on the benefits of adding calcareous fillers to bituminous mixes to improve aging resistance, and consequently prolong the useful life of pavements. Moreover, some conclusions about quantitative aspects of such advantages and certain limitations have been reached.

The UCL® method has proved to be a very useful tool for this study, and the Set of Performance Curves has allowed changes in cohesion associated with mix aging and test temperatures to be observed.

For equal volumetric C_v/C_s relations, the fillers used in this study, i.e. hydrated lime and limestone dust, exhibit similar responses. Nonetheless, considering their weight proportions, a mush greater limestone dust content is required to obtain the same results because of the different values of critical concentration of both fillers.

Conventional bitumen mixes begin to stiffen from critical concentrations of filler (C_v/C_s = 1), in agreement with many Argentinean studies on this topic. However, the tests in this work lead to an additional conclusion: as the mix ages, the optimum C_v/C_s relation values tend to be less than one.

Also important is that the relation could be as high as C_v/C_s =1.3 for polymer-modified bitumen mixes since, in this case, the undesirable effects of excessive filler content are not observed.

Results have also qualitatively and quantitatively ratified other previously known aspects of bitumen behavior: a better response of SBS-modified bitumens than EVA-modified bitumens and the theoretical important advantages of polymer-modified binders over conventional binders.

Determining binder intrinsic characteristics by DSR, before and after mix aging, with and without filler addition, has been extremely helpful in understanding the evolution of aging factors and comparatively assessing the improvements achieved by filler addition. It has been found that the complex shear modulus (G*) clearly increases more slowly and the phase angle (δ) decreases more slowly because of aging.

Despite limitations, the protective effect of filler against conventional bitumen aging has been corroborated by viscosity tests since bitumens do not become so consistent. In polymer-modified bitumens, a "competition" between factors favoring consistency (formation of cross-linking products in the polymeric net and base bitumen hardening) and those with the opposite effect, such as polymer chain scission, has been observed.

Macromolecular analysis techniques (GPC and IR spectrography) have explained some of the above phenomena. Thus aging results in the generation of bigger composites by oxidation and polar associations in the base bitumen, whereas filler addition reduces these effects.

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