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Influence of hydrated lime on linear viscoelastic properties of bituminous mastics

Cong Viet Phan¹, Hervé Di Benedetto¹, Cédric Sauzéat¹,
Didier Lesueur², Simon Pouget³

Abstract The influence of hydrated lime (HL) on linear viscoelastic (LVE) behavior of mastic is investigated. One bitumen and five mastics with different HL content are investigated. The same 35/50 penetration grade bitumen was tested and used for mastics preparation. The LVE properties were obtained by means of complex shear modulus tests using a dynamic shear rheometer (DSR) on a wide range of temperatures and frequencies.

The time–temperature superposition principle in the LVE domain was applicable. Experimental results were modeled using the 2S2P1D model previously developed at the University of Lyon/ENTPE. The reinforcement effect of HL on LVE behavior of mastics was observed and quantified. Results show that the volume of filler increase when limestone is replaced by HL at constant mass, may explain a large part of the observed stiffening effect.

Keywords Linear viscoelastic behavior · Hydrated lime · Mastic · Bitumen · Complex shear modulus · Rheological modeling

1 Introduction

Many studies were carried out on mastics (i.e. blends of bitumen and only finest aggregates part, called filler) (Palade et al. 2000; Delaporte et al. 2007, 2009; Liao and Chen 2011; Van Rompu et al. 2012; Wang et al. 2013; Mannan et al. 2015; Riccardi et al. 2018; Santagata et al. 2016; Jiang et al. 2018). It is generally admitted that the mastic and not the bitumen is the real glue that bonds the larger aggregates inside the bituminous mixtures. Then a better understanding of thermomechanical behavior of mastic should help to establish a rational link between the behavior of bitumen and bituminous mixtures. Furthermore, the effect of filler can be more easily studied in mastic than in bituminous mixtures. The important role

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of filler on the compaction and performance of bituminous mixtures is underlined by some authors (Anderson and Goetz 1973; Shashidhar and Romero 1998; Lesueur et al. 2018). The filler has a reinforcement effect which depends on several parameters such as size, type, grading and concentration, as shown by studies on mastics (Delaporte et al. 2007, 2009; Anderson and Goetz 1973; Durand et al. 1997; Lesueur and Little 1999; Kuity and Das 2016; Rieksts et al. 2018; Roberto et al. 2018).

HL is often added, as an efficient and interactive filler, in order to improve the moisture resistance of bituminous mixtures (Lesueur and Little 1999). Several studies stated that linear viscoelastic properties are improved when HL replaces “classical” mineral filler (Tunnicliff 1967; Kim et al. 2003; Kim and Little 2004). Kim et al. (2003) characterized the stiffening effect of fillers in bituminous binder through micromechanical and/or rheological methods. They measured LVE properties, using dynamic shear rheometer (DSR), for several mastics produced with two binders and different fractions of limestone or HL. They demonstrated that HL filled binders behavior was significantly different from the limestone filled binders behavior. The physico-chemical interaction between HL and bitumen might be the explanation for such a difference.

However, it must be underlined that these test results were obtained at high temperatures. Other studies (Lackner et al. 2005; Johansson and Isacson 1998) performed at low temperatures exhibited different results. The stiffening effect observed on material containing HL was similar to other “classical” mineral fillers.

The results presented in this paper are part of a wider research on thermomechanical behavior of bituminous mixtures containing HL, involving three partners, the companies LHOIST and EIFFAGE Infrastructures and University of Lyon /ENTPE.

The LVE behavior of five mastics with different HL content was investigated. They were produced with the same 35/50 penetration grade bitumen. DSR was used to perform complex shear modulus tests (G^*), which allowed one to determine the LVE properties on a wide range of temperatures (T) and frequencies (f). Experimental results were fitted using the 2S2P1D (2 Springs, 2 Parabolic creep elements and 1 Dashpot) Linear viscoelastic model. This powerful model for bituminous materials, including bitumens, mastics and mixtures, was developed at ENTPE (Olard and Di Benedetto 2003; Di Benedetto et al. 2007; Tapsoba et al. 2014; Pham et al. 2015; Perraton et al. 2016; Di Benedetto et al. 2016; Gayte et al. 2016). A specific analysis is conducted to investigate and identify the influence of HL on the LVE behavior of mastics.

2 Experimental procedures and materials

2.1 Dynamic shear rheometer (DSR)

Tests were performed using a dynamic shear rheometer (DSR) (Physica MCR 501), which is shown in Fig. 1. The DSR tests were performed on cylindrical specimens of mastic or bitumen sandwiched between two plates, classically called “plate-plate” DSR geometry. The applied torque and the rotation angle were continuously measured. During tests, the temperature of the sample was ensured with a thermal chamber.

In this investigation, the tested cylindrical sample (diameter 4 mm and thickness 1.5 mm) was placed in the DSR at 40 °C and cooled for 2 h before testing. Three or four repetition tests were performed for each material.

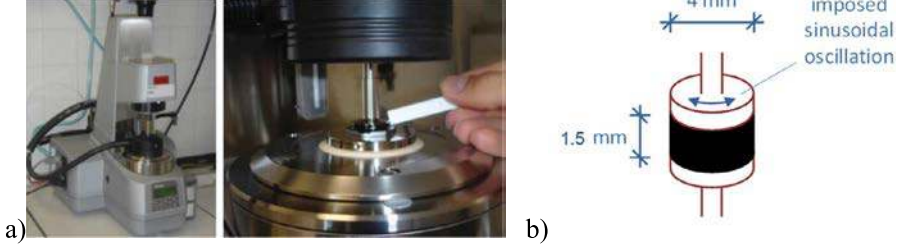


Fig. 1 (a, left) DSR apparatus used for tests; (a, right) close-up on a sample; (b) scheme giving the size of the samples

Table 1 T , f and strain amplitude used for DSR tests

T (°C)	60 50 40 30 15	10 0 -10 -12.9 -16.5
γ (%)	3 1 1 0.30.3	0.10.10.030.01 0.01
f (Hz)	0.03; 0.119; 0.475; 1.89; 7.54; 30	0.01; 0.038; 0.144; 0.548; 2.08; 7.9; 30

A sinusoidal rotation was applied and the corresponding torque was measured. Shear strain $\gamma(t)$ and shear stress $\sigma(t)$ could then be obtained. They are written in complex notation in Eq. (1):

$$\sigma(t) = \sigma_0 e^{i\omega t} \quad \text{and} \quad \gamma(t) = \gamma_0 e^{i(\omega t - \varphi)} \quad (1)$$

where σ_0 and γ_0 are the amplitude of shear stress and shear strain, respectively. φ is the phase lag.

The complex shear modulus G^* can be calculated as follows:

$$G^*(\omega) = \frac{\sigma_0 e^{i\omega t}}{\gamma_0 e^{i(\omega t - \varphi)}} = |G^*| e^{i\varphi} = G_1 + iG_2 = |G^*| \cos \varphi + i |G^*| \sin \varphi \quad (2)$$

where $|G^*|$, G_1 , and G_2 represent, respectively, the norm, the real part and the imaginary part of G^* .

Samples were loaded at different frequencies (f) and temperatures (T) as indicated in Table 1. The selected maximum strain amplitude that was selected for each T is also indicated in Table 1. This amplitude was selected to remain within the linear domain.

2.2 Tested materials

One bitumen and five mastics were studied. A 35/50 penetration grade bitumen (called B3550) was tested and the same bitumen was used to prepare the mastics that have different fractions of HL and limestone. The filler and HL content were chosen within the range used in bituminous mixture containing 5.1% bitumen and 8% filler in total mass (either containing only limestone filler, or 6.75% limestone filler and 1.25% HL, or 5.5% limestone filler and 2.5% HL). The composition in mass and the volume of the tested materials and their name are given in Table 2. A reference name is assigned M3550-5.1 (indicating the penetration grade bitumen and bitumen content in corresponding mixture) followed by information on the HL content: 0, 1.25, 2.5, vol(1.25%) or vol(2.5%). As indicated previously, presented study is part of a wider research on thermomechanical behavior of bituminous mixtures containing HL.

Table 2 Tested materials: name and composition

Material	% in mass				% in volume			
	Bitumen	Limestone filler	HL	Total of filler	Bitumen	Limestone filler	HL	Total of filler
B3550	100	0	0	0	100	0	0	0
M3550-5.1-0	40.2	59.8	0	59.8	65.3	34.7	0	34.7
M3550-5.1-1.25	40.2	50	9.8	59.8	64.4	28.6	7	35.6
M3550-5.1-2.5	40.2	40.1	19.7	59.8	63.5	22.6	13.9	36.5
M3550-vol(1.25%)	39.2	60.8	0	60.8	64.4	35.6	0	35.6
M3550-vol(2.5%)	38.3	61.7	0	61.7	63.5	36.5	0	36.5

Considering the lower density of HL (2.24 g/cm^3) than the one of limestone filler (2.8 g/cm^3), the substitution of limestone filler with the same mass leads to an increase of the global volume of filler. In order to differentiate effects of mass and volume, two mastics with the same volume of fine particles were produced with only limestone filler. The mastic named M3550-vol(1.25%) has the same filler volume fraction as M3550-5.1-1.25 (which is 35.6%) and M3550-vol(2.5%) has the same filler volume fraction as M3550-5.1-2.5 (which is 36.5%).

The figure (1, 2, 3 or 4) following the material name indicates the replicated test number.

3 Experimental results for G^*

Figure 2 presents data and 2S2PID model simulations (presented further) for the three or four replicates on the six materials in the Cole–Cole axes (a) and in the Black diagram (b). For each material, it can be considered, as a first approximation, that data are situated on a unique curve when changing f and T . Then the time–temperature superposition principle (TTSP) can be applied to the materials.

The master curves at a reference temperature T_{ref} equal to $15 \text{ }^\circ\text{C}$, are plotted in Fig. 3 for $|G^*|$ (upper) and φ (lower). It presents $|G^*|$ (in log scale) and φ , respectively, as a function of the equivalent frequency ($a_T f$, where a_T is the shift factor presented in next section).

3.1 Shift factors

The construction of master curves requires shift factor (a_T) values. These values are presented in Fig. 4 as a function of T . This figure shows a good repeatability of obtained a_T values between the three or four replicates of each material. Moreover, a_T values are very close for the six tested materials. This result confirms previous results from our team showing this parameter is fixed by the bitumen whatever granular skeleton (Delaporte et al. 2007; Olard and Di Benedetto 2003; Pouget et al. 2012; Mangiafico et al. 2014). The two constants C_1 and C_2 of the WLF (Williams, Landel and Ferry) (Williams et al. 1955) equation (Eq. (3)), used to fit a_T data, are indicated in Table 3. We have

$$\log(a_T) = -\frac{C_1(T - T_{ref})}{(C_2 + T - T_{ref})} \quad (3)$$

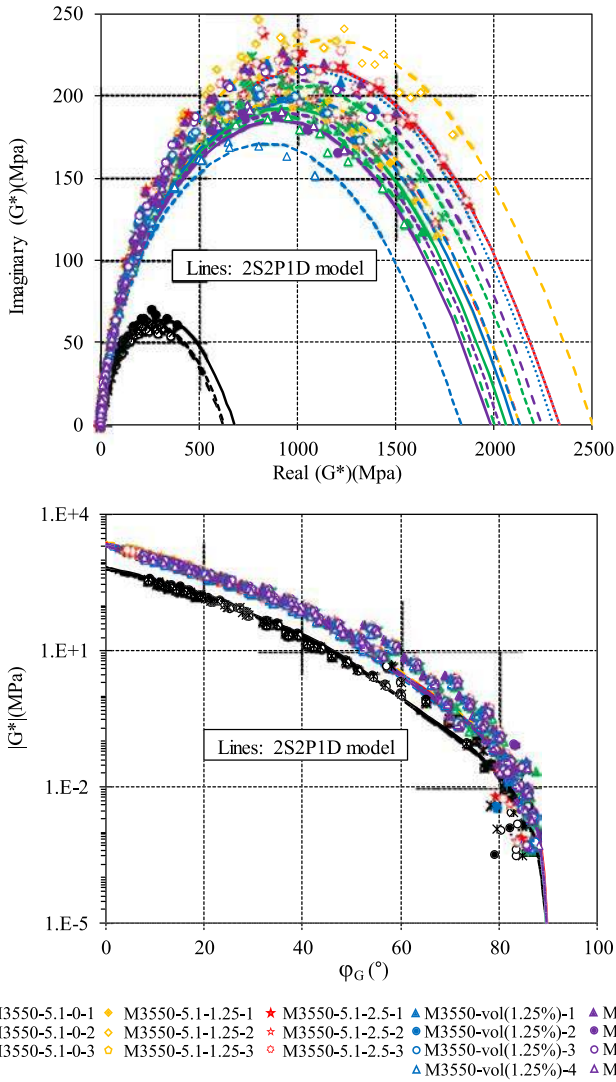


Fig. 2 Experimental data and 2S2P1D simulation curves (constants given in Table 3) for tested materials: Cole–Cole axes (upper); Black diagram (lower).

4 Modeling

Experimental results are modeled using the LVE model, 2S2P1D, which was developed at the ENTPE. This model is a generalization of the Huet–Sayegh model consisting of two spring elements, two parabolic creep and one dashpot. An analogical representation is given in Fig. 5. It is widely used to model the LVE behavior in 1D and 3D of any bituminous material: bitumens, mastics and mixtures.

Table 3 2S2P1D and WLF constants for the tested samples (Reference temperature 15 °C)

Material	Sample	G^*							WLF		
		G_{00} (MPa)	G_0 (MPa)	k	h	δ	τ_0 (s)	β	C_1	C_2	
B3550	1	0 ^a	680				0.00016				
	2	0 ^a	620								
	3	0 ^a	630								
M3550-5.1-0	1	0 ^a	2060				0.00053				
	2	0 ^a	2000								
	3	0 ^a	2200								
M3550-5.1-1.25	1	0 ^a	2100				0.00086				
	2	0 ^a	2500								
	3	0 ^a	2130								
M3550-5.1-2.5	1	0 ^a	2330				0.0012				
	2	0 ^a	2130								
	3	0 ^a	2330								
M3550-vol(1.25%)	1	0 ^a	2300	0.225	0.59	3.8	0.0008	230	30	213	
	2	0 ^a	2100								
	3	0 ^a	2130								
	4	0 ^a	1830								
M3550-vol(2.5%)	1	0 ^a	2330				0.0009				
	2	0 ^a	1980								
	3	0 ^a	2240								
	4	0 ^a	2030								

Note: ^aChosen arbitrarily due to lack of accuracy of the experimental device

Equation (4) gives G^* at a fixed T :

$$G_{2S2P1D}^*(\omega) = G_{00} + \frac{G_0 - G_{00}}{1 + \delta(j\omega\tau)^{-k} + (j\omega\tau)^{-h} + (j\omega\beta\tau)^{-1}} \quad (4)$$

where

$$j^2 = -1$$

ω : radian frequency, $\omega = 2\pi f$

k, h : exponents such that $0 < k < h < 1$;

δ : constant

G_{00} : static modulus when $\omega \rightarrow 0$; G_0 : glassy modulus when $\omega \rightarrow \infty$

β : dimensionless constant, related to Newtonian viscosity $\eta = (G_0 - G_{00})\beta\tau$

τ : characteristic time, function only of T .

To obtain G^* at any T , it is possible to use the TTSP. In this case, only one parameter τ_0 and the shift factor value are required to calculate τ values (Eq. (5)); one uses

$$\tau(T) = a_T(T)\tau_0 \quad (5)$$

where a_T is the shift factor at T . τ_0 is equal to the τ value at the chosen T_{ref} . It has to be highlighted that only seven constants (G_{00} , G_0 , δ , h , k , β , τ_0) are required to entirely characterize the LVE properties at a given T . In this paper, a_T was approximated by the

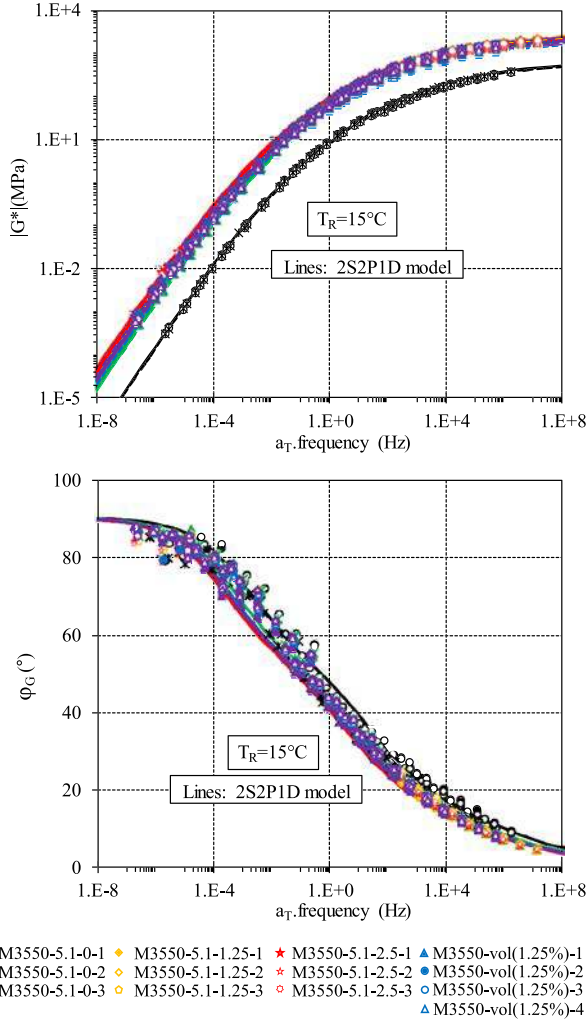


Fig. 3 Experimental data and 2S2P1D simulation curves (constants given in Table 3) for tested materials: master curves of the complex modulus, norm (upper) and phase angle (lower).

WLF equation (Eq. (3)). Finally, the number of constants amounts to nine, including C_1 and C_2 fitted at $T_{ref} = 15^\circ\text{C}$.

Figures 2 and 3 show that the 2S2P1D model fits rather well experimental results of all tested materials on the whole range of f and T . 2S2P1D constants for all samples are given in Table 3.

In Table 3, the constants k , h , δ and β are identical for the six tested materials. This result confirms conclusion obtained by previous authors (Delaporte et al. 2007, 2009; Olard and Di Benedetto 2003; Yusoff et al. 2013; Pouget et al. 2012) that these constants are only dependent on the crude oil origin of bitumen, regardless of the penetration. It is recalled that the same 35/50 bitumen was used to produce all the tested materials. τ_0 , which is also bitumen dependent, and the constants G_0 and G_{00} depend on filler content and characteristics. Considering the accuracy of the experimental device, $|G^*|$ values below 300 Pa could not

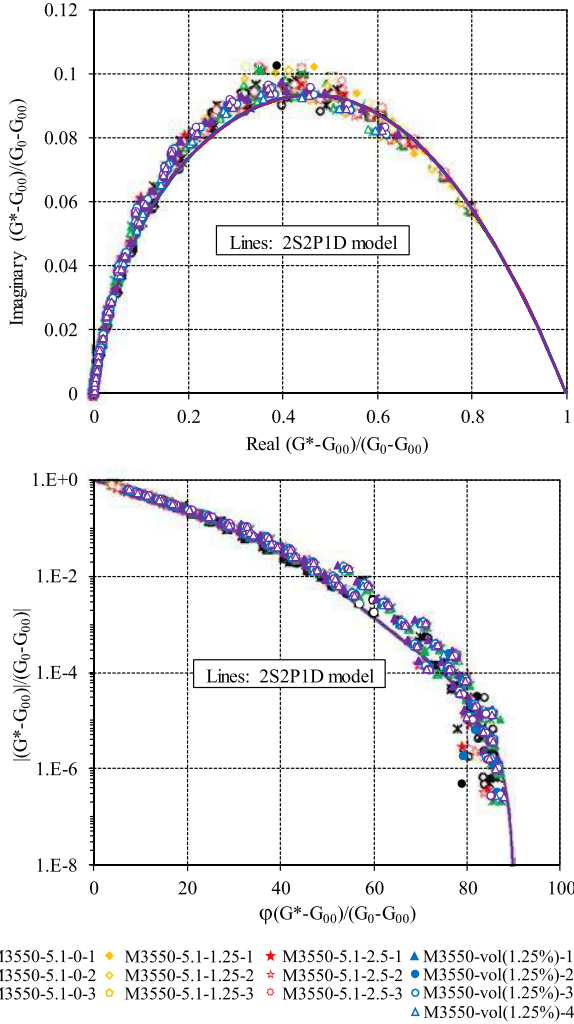


Fig. 6 Experimental data for normalized complex modulus and 2S2P1D simulation curve (constants given in Table 3) for tested materials: Cole–Cole axes (upper); Black diagram (lower).

G_{nor}^* is plotted in the Black diagram and the Cole–Cole axes in Fig. 6. The master curves are given in Fig. 7.

Figure 6 shows that all curves are superimposed. In order to obtain the same value of $\tau_0 = 0.00053$ in Fig. 7, different T_{ref} are chosen for each material ($T_{ref} = 10.99$ °C for B3550, $T_{ref} = 15$ °C for M3550-5.1-0, $T_{ref} = 16.52$ °C for M3550-5.1-1.25, $T_{ref} = 17.57$ °C for M3550-5.1-2.5, $T_{ref} = 16.28$ °C for M3550-vol (1.25%), $T_{ref} = 17.57$ °C for M3550-vol (2.5%)). Figure 7 shows that all data are superimposed, which is an important output. It should be highlighted that all master curves are identical when reference temperature is adjusted to obtain the same value of τ_0 . This result confirms that filler (and more generally aggregates) only influences G_0 , G_{00} and τ_0 values. All other constants are inherited from the bitumen.

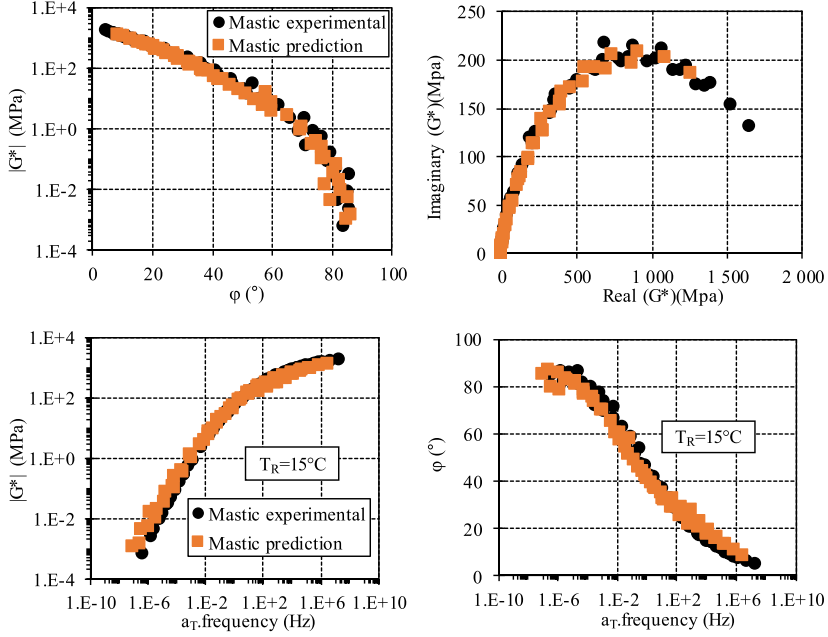


Fig. 8 Prediction of mastic M3550-5.1-1.25 LVE behavior from DSR tests on corresponding bitumen B3550 using a SHStS transformation.

Table 4 Values of α used for predicting the mastic results from the binder.

Material	M3550-5.1-0	M3550-5.1-1.25	M3550-5.1-2.5	M3550-vol(1.25%)	M3550-vol(2.5%)
α	0.52	0.73	0.88	0.70	0.75

G_{00_mastic} , G_{0_mastic} and α (this constant depends on the formulation). The α value can be determined from the parameter τ of 2S2P1D model. If the TTSP is verified, Eqs. (8) and (9) can be considered:

$$G^*(f, T) = G^*[f a_T(T), T_{ref}] \quad (8)$$

$$\tau_{mastic} = 10^\alpha \tau_{bitumen} \quad (9)$$

The values of α are given in Table 4 for each of the tested materials. As an example, the comparison between experimental results and values of prediction using the SHStS transformation (Eq. (7)) from B3550 to M3550-5.1-1.25 is showed in Fig. 8. This figure reveals that the transformation gives a successful prediction of mastic LVE behavior from bitumen behavior on the whole T and f range. The same conclusion is valid for the other tested materials.

5.2 Effect of HL and volume of fine particles content

As indicated before, the five mastics were prepared with the same bitumen. For two mastics M3550-5.1-1.25 and M3550-5.1-2.5, HL replaces a part of limestone filler keeping the

same total mass of filler than mastic M3550-5.1-0. As the volumetric mass of HL is lower than the one of limestone filler, the volume of fine particles increases with HL content. Then the results obtained for these three mastics does not allow differentiating the effects of the two parameters: the introduction of HL and the volume increase. In order to quantify the influence of these two effects, the results obtained for mastics containing the same volume of fine particles (only limestone filler or limestone filler and HL) were also analyzed. M3550-vol(1.25%) was compared with M3550-5.1-1.25 (volume fraction of filler 35.6%) and M3550-vol(2.5%) was compared with M3550-5.1-2.5 (volume fraction of filler 36.5%). The average values of the different duplicated tests for each material were used in the analysis.

The complex reinforcement coefficient R^* introduced by Delaporte (Delaporte et al. 2008, 2009) is used to compare the G^* two mastics on the whole range of considered T and f . R^* is defined as the ratio between the G^* of two mastics (X and Y) at the equivalent frequency ($f_e = a_T f$) when the TTSP is validated:

$$R^* = \frac{G_{\text{mastic}X}^*(f_e)}{G_{\text{mastic}Y}^*(f_e)} \quad (10)$$

R^* is a complex number. Its norm and its phase angle can be considered thus:

$$|R^*| = \frac{|G_{\text{mastic}X}^*(f_e)|}{|G_{\text{mastic}Y}^*(f_e)|} \quad \varphi_R = \varphi_{\text{mastic}X} - \varphi_{\text{mastic}Y} \quad (11)$$

As a_T values of the bitumen and the five mastics are very close and can be considered as equal, the same values of f_e are obtained for the same value of f and T for the six tested materials. Then G_{bitumen}^* and G_{mastic}^* are taken at the same $T-f$ couples to calculate R^* .

Three groups of comparison are proposed:

- Mastics having the same mass of filler but different contents of HL, which gives information on the effect of HL at constant mass of filler. The two concerned mastics are M3550-5.1-1.25 and M3550-5.1-2.5 with reference mastic M3550-5.1-0.

- Mastics with the same volume of fine particles but different contents of HL. The two concerned mastics are M3550-5.1-1.25 (with HL), with reference M3550-vol(1.25%) (without HL) for a volume of filler: $V_{\text{filler}} = 35.6\% V_{\text{tot}}$, and M3550-5.1-2.5 (with HL), with reference M3550-vol(2.5%) (without HL) for a volume of filler: $V_{\text{filler}} = 36.5\% V_{\text{tot}}$.

- Mastics without HL having different volumes of filler. The two concerned mastics are M3550-vol(1.25%) having a volume of filler of 35.6% and M3550-vol(2.5%) having a volume of filler of 36.5%, with reference M3550-5.1-0 (volume of filler of 34.7%).

$|R^*|$ and φ_R of mastics containing the same mass of filler are presented as a function of equivalent frequency (f_e) in Fig. 9. This figure shows that the mastic made with HL (but also with higher volume of fine particles) exhibits a higher $|G^*|$ than the one produced without HL ($|R^*|$ is always greater than 1 on the whole range of considered T and f). At (15°C; 10 Hz), which is used in the French pavement design method, the reinforcement is approximately 22% ($|R^*| = 1.22$) with M3550-5.1-1.25 and approximately 36% ($|R^*| = 1.36$) with M3550-5.1-2.5. The parameter φ_R is always negative. This indicates that the phase angle is reduced for mastics with HL filler (having the same total mass of filler). Meanwhile this reduction is rather small (less than 3° for most of the data). It may be seen that stiffening effects are $T-f$ dependent as was already observed (Delaporte et al. 2007; Lesueur et al. 2013; Wortelboer et al. 1996; Phan et al. 2016, 2017). It is more pronounced at low f and/or high T . Almost no effect exists at high f /low T , as $|R^*| = 1$ and $\varphi_R = 0$.

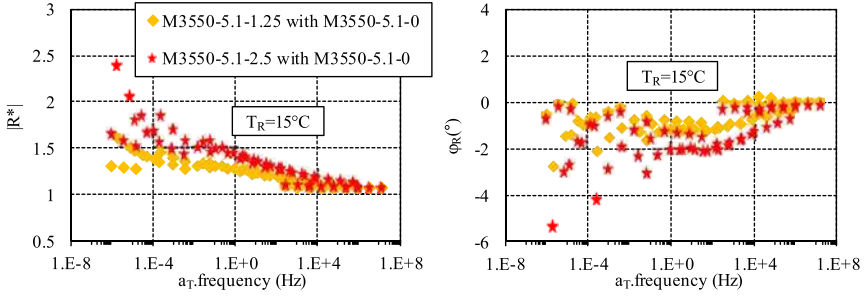


Fig. 9 Complex reinforcement coefficient R^* at $T_{ref} = 15\text{ }^\circ\text{C}$ for mastics containing the same mass of filler but different HL contents: Norm (left) and phase angle (right).

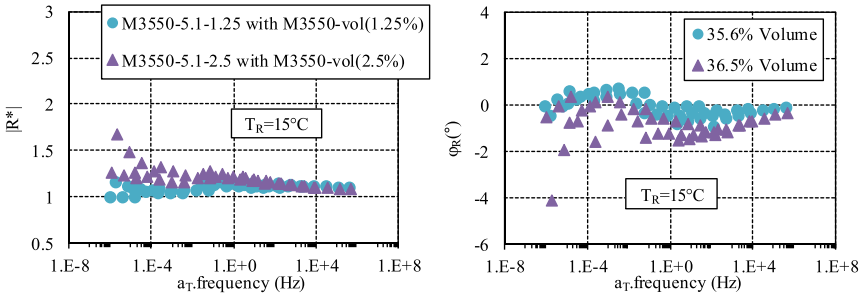


Fig. 10 Complex reinforcement coefficient R^* at $T_{ref} = 15\text{ }^\circ\text{C}$ obtained with a reference mastic having the same volume of filler but different HL contents: Norm (left) and phase angle (right).

The influence of the HL with constant volume of filler is presented in Fig. 10. Again, the $|R^*|$ values are always greater than 1 and increase when the equivalent frequency f_e decreases (high T /low f). The same conclusion than the one proposed for mastics at constant mass of filler (Fig. 9) can be drawn. Meanwhile, the stiffening effect is much less pronounced when volume of filler is maintained constant. For 1.25% of HL (M3550-5.1–1.25) the $|R^*|$ value is very close to 1 (and φ_R close to 0) on the whole T – f range, indicating no effect of HL. For 2.5% of HL (M3550-5.1–2.5) the $|R^*|$ values are greater than 1.25 for f_e lower than 1, which confirm HL effect when increasing HL content. Then the change in volume induced by the lower volumetric mass of HL can only partly explain stiffening for higher percentage of HL. At ($15\text{ }^\circ\text{C}$; 10 Hz) the reinforcement is close to 8% ($|R^*| = 1.08$) with 1.25% of HL and 15% ($|R^*| = 1.15$) with 2.5% of HL. φ_R values, which are mainly negative, are smaller than 2° for most of the data, indicating a small effect on this parameter.

Test results focusing on volume of filler effects without HL are presented in Fig. 11. The reinforcement effect by increasing the volume of filler is observed on the whole T – f range: $|R^*|$ is always greater than 1 and φ_R is always negative (except for some data at high T). At 15°C and 10 Hz, this effect is close to 14% ($|R^*| = 1.14$) when volume of filler increased from 34.7% to 35.6% and 18% ($|R^*| = 1.18$) when volume of filler increased from 34.7% to 36.5%.

As shown before (Fig. 6), the master curves of the G_{nor}^* of all tested materials are superimposed. This result indicates than only three constants of 2S2PID model are different for the materials, namely τ_0 , G_0 and G_{00} . The accuracy of the experimental device would

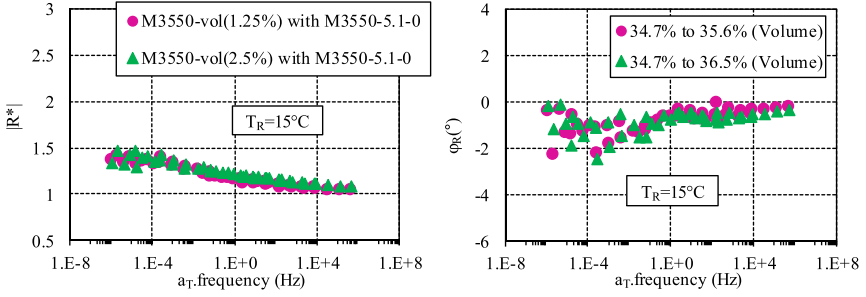


Fig. 11 Complex reinforcement coefficient R^* : Norm (left) and phase angle (right) for mastics without hydrated lime (effect of the increase of volume of fine particles) at $T_{ref} = 15^\circ\text{C}$.

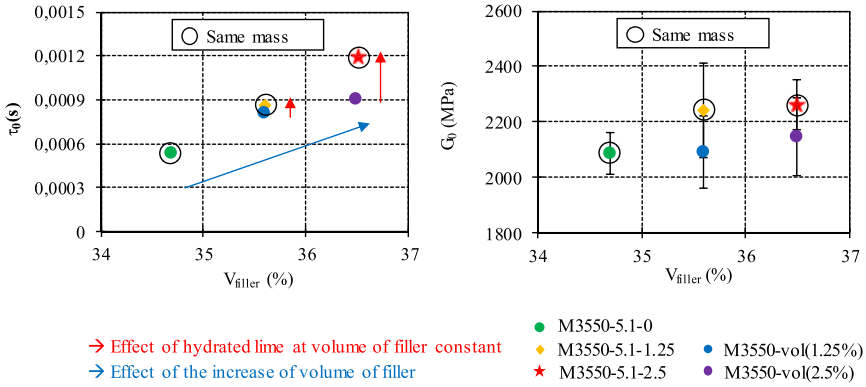


Fig. 12 τ_0 and G_0 as a function of volume of filler (%)

not allow one to obtain G_{00} . The obtained values for τ_0 (at 15°C) and G_0 are plotted as a function of the volume of filler in Fig. 12, for the five mastics. This figure reveals that both parameters G_0 and τ_0 increase with HL and volume of filler content. The influence of each effect can be clearly identified.

6 Conclusions

The study presented in this paper aimed at evaluating the influence of HL on the LVE behavior of mastic. Five mastics produced with the same mass of filler or the same volume but with and without HL and the used bitumen were tested with a DSR. The influence of HL on LVE properties is quantified on a wide range of T and f by means of R^* . From the obtained results and performed analyses, the following remarks can be made:

- The TTSP was verified as a first approximation for the six tested materials. The a_T values are very close for the six tested materials.
- The LVE behavior is correctly simulated with 2S2PID model on the whole range of tested T and f .

– G_{nor}^* presents superimposed curves for the six tested materials in the Cole–Cole and the Black axes. This result confirms that normalized Cole–Cole and Black curves are fixed by the bitumen.

– The transformation SHSTS proposed by University of Lyon/ENTPE yielded successful predictions of mastic LVE behavior from bitumen LVE behavior.

– The reinforcement effect of HL on LVE behavior of mastics was observed and quantified. This reinforcement effect is T – f dependent, it is much more pronounced at high T and/or low f . It should be underlined that, if mass is kept constant, volume of filler increases when limestone is replaced by HL. This volume increase can explain a large part of the observed stiffening effect when introducing HL at constant mass of filler. The introduction of HL at constant volume of filler has nearly no effect for HL content of 1.25%, but starts to be slightly visible for a HL content of 2.5%.

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