Influence of hydrated lime on linear viscoelastic properties of mastics

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ABSTRACT

An ongoing study on influence of hydrated lime on mechanical properties of bituminous mixtures is carried out in the framework of collaboration between the LHOIST Group and University of Lyon /ENTPE. In this paper we focus on linear viscoelastic properties of mastic. Different mastics were produced with the same bitumen penetration grade, same bitumen content and different hydrated lime content. Hydrated lime replaces a part of limestone filler (the same mass of removed filler for the same mass of added hydrated lime). Linear Viscoelastic properties of both produced mastics and bitumen were determined using a dynamic shear rheometer (DSR). First results and analyses are presented in this paper.
The Time-Temperature Superposition Principle (TTSP) was verified with good approximation. The 2S2P1D model developed by ENTPE team was used for modelling linear viscoelastic properties of bitumen and mastics. Effect of hydrated lime is discussed.

Keywords: Asphalt, Complex Modulus, Lime, Mastic Asphalt, Rheology

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ABSTRACT.

An ongoing study on influence of hydrated lime on mechanical properties of bituminous mixtures is carried out in the framework of collaboration between the LHOIST Group and University of Lyon /ENTPE. In this paper we focus on linear viscoelastic properties of mastic. Different mastics were produced with the same bitumen penetration grade, same bitumen content and different hydrated lime content. Hydrated lime replaces a part of limestone filler (the same mass of removed filler for the same mass of added hydrated lime). Linear Viscoelastic properties of both produced mastics and bitumen were determined using a dynamic shear rheometer (DSR). First results and analyses are presented in this paper.

The Time-Temperature Superposition Principle (TTSP) was verified with good approximation. The 2S2P1D model developed by ENTPE team was used for modelling linear viscoelastic properties of bitumen and mastics. Effect of hydrated lime is discussed.

Keywords: Linear viscoelastic properties, hydrated lime, mastic, complex shear modulus, rheological modeling

1. INTRODUCTION

In order to better understand the properties of bituminous mixtures, many researchers have studied mastics, i.e. blends of only bitumen and filler. The idea is that the bitumen is not considered as the material bonding together the aggregates inside the mixture, but it is the blend between bitumen and the finest elements of the mineral skeleton, i.e. the mastic. Characterizing mechanical properties of mastic may contribute to correlate the behaviour of bitumen with the one of the bituminous mixture. In addition, filler effect could be determined more directly in mastic than in asphalt. Mineral fillers are known to affect the properties of bituminous mixtures and play an important role in their compaction and their performance [1; 2]. Studies on mastic have shown it that the reinforcement brought by filler in bitumen may depend on different parameters such as type, size, grading or concentration [1; 3; 4; 5; 6; 7; 8; 9]. Different kinds of filler may indeed be used and hydrated lime is one of them.

Several studies showed that properties such as viscosity are similarly increased when hydrated lime is used instead of "classical" mineral filler [10; 11; 12]. However, it should be mentioned that such results were obtained from tests performed at high temperatures. On the contrary, the studies carried out at low temperature showed that hydrated lime is similar to other mineral fillers in terms of stiffening effect at low temperature [13; 14].

In this paper, presented results are part of an ongoing study on the influence of hydrated lime on properties of bituminous mixtures. This study is carried out in the framework of a collaboration between the LHOIST Group and University of Lyon /ENTPE.

In this paper, the Linear Viscoelastic (LVE) behaviour of three different mastics with and without lime and the corresponding bitumen is presented. The LVE properties of the materials are obtained by means of complex shear modulus tests (using dynamic shear rheometer) at several temperatures and frequencies. A modelisation of complex modulus is made with the 2S2P1D (2 Springs, 2 Parabolic creep elements and 1 Dashpot) model, developed at ENTPE laboratory [15; 16; 4; 17; 18; 19]. Effect of hydrated lime on LVE properties of mastics is specifically studied.

The testing procedure and the tested materials are presented in section 2. The results of complex shear modulus tests for the four different tested materials are exposed in section 3. Section 4 introduces the 2S2P1D model and the fitted parameters. Section 5 focuses on the analysis of results.

2. EXPERIMENTAL PROCEDURES AND MATERIALS

2.1. Description of the dynamic shear rheometer (DSR)

Tests were performed using a Dynamic Shear Rheometer (Physica MCR 501). This device is shown in Figure 1. A cylindrical sample of bitumen or mastic is attached to two plates. One of the plates is fixed while the other one rotates, thus imposing a shear loading to the sample. Loading can be performed in stress control or strain control mode. The rotation angle of the moving plate and the applied torque are continuously measured. A thermal chamber was used for thermal conditioning of the sample during the test.

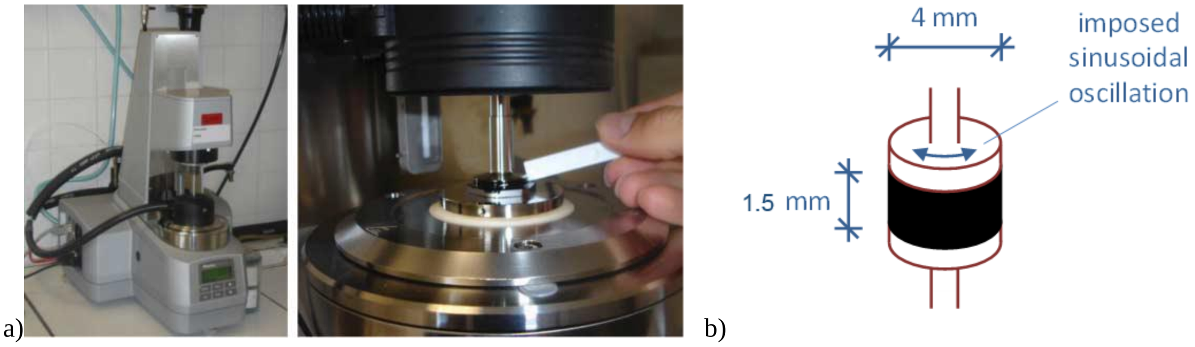


Figure 1: (a, left) DSR apparatus (Physica MCR 501) used for bitumen tests; (a, right) close-up on a test sample; (b) scheme of a DSR test and sample size

In the present study, a "plate-plate" configuration was used. The tested cylindrical sample had a diameter equal to 4 mm and was 1.5 mm thick. The specimens were cored in a molded plate of approximately 1.5 mm thick. They were placed on the DSR plates at 40°C and cooled for 2 hours. For each material, three repetition tests were carried out. The compliance of the DSR spindle, which affects significantly the results for high torque values, was corrected.

Tests were performed in strain control mode, by applying a sinusoidal shear strain signal $\gamma(t)$ with a frequency f and measuring the corresponding shear stress $\sigma(t)$. Using complex notation, the sinusoidal shear stress of amplitude σ_0 and the sinusoidal shear strain of amplitude γ_0 may be expressed as:

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

where ω=2πf and t is the time.

Complex shear modulus G* is obtained as

$$G^*(\omega) = \frac{\sigma_0 e^{i\omega t}}{\gamma_0 e^{i\omega t-\varphi}} = |G^*|e^{i\varphi} = G_1 + i\,G_2 = |G^*|\cos\varphi + i\,|G^*|\sin\varphi \tag{2}$$

where |G*| is the norm of the complex shear modulus, G_1 is the real part and G_2 is the imaginary part of complex shear modulus. Angle φ is the phase lag.

Complex modulus tests were performed at several temperatures, from -16.5°C to 70°C, and several frequencies, from 0.010 Hz to 30 Hz, as shown in Table 1.

Table 1: Temperatures and frequencies used for DSR complex modulus tests

Temperature (°C)	-16.5 -12.9 -10 0 10	15 30 40 50 60 70
Frequency (Hz)	0.01 0.038 0.144 0.548 2.08 7.9 30	0.03 0.119 0.475 1.89 7.54 30

2.2. Materials

Four materials including bitumen and mastics are considered. The same 35/50 penetration grade bitumen was used for testing (called B3550) and mastics preparation. The three tested mastics were prepared with the same bitumen content: 40.2% in mass. Two types of fillers, hydrated lime and limestone, were used. One mastic contained only limestone filler, while for the two others, hydrated lime replaces a part of limestone filler in order to keep the same total mass of mineral component. Materials name and composition are indicated in Table 2. The volume content was calculated using volumetric mass of 1.02 g/cm^3 for bitumen, 2.8 g/cm^3 for limestone filler and 2.24 g/cm^3 for hydrated lime. These two last values were measured at Lhoist laboratory following European standard EN 1097-7.

To differentiate the mastics, a reference name is attributed B3550-5.1 (indicating the bitumen name) followed by a number 0, 1.25 or 2.5 (indicating the hydrated lime content). As indicated before, presented results are a part of an ongoing wider study. Such mastics are considered to be the mastics found in tested mixtures of the study, which are composed of 5.1% in total mass of the same 35/50 bitumen and 8% in mass of filler (either composed only with limestone filler, or 6.75% limestone filler plus 1.25% hydrated lime, or 5.5% limestone and 2.5% hydrated lime).

A number (1, 2 or 3) is added to the material name to distinguish the 3 repetitions tests performed for each material.

Table 2: Tested materials name and composition

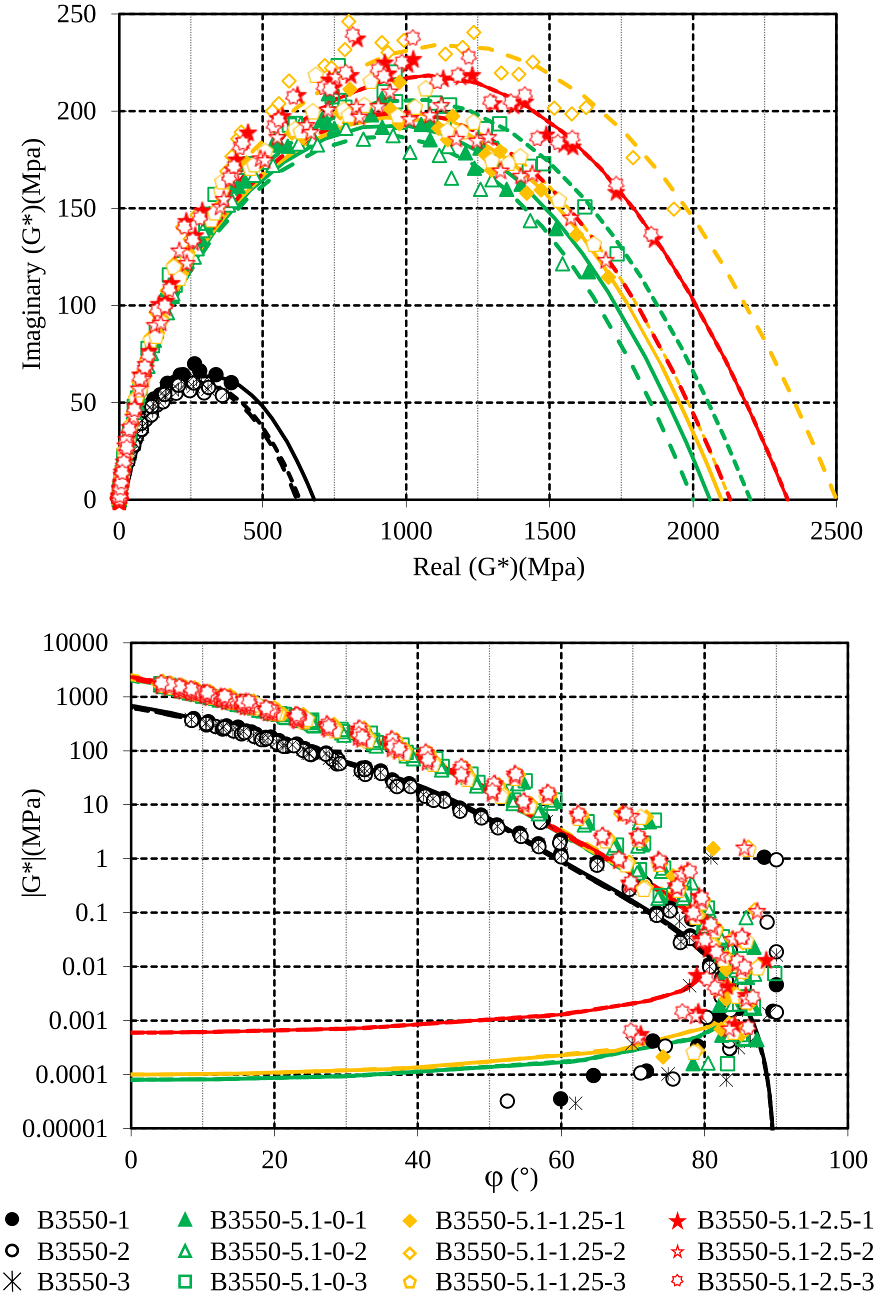
Material name	**Limestone filler content (% in mass)**	**Hydrated lime content (% in mass)**	**Bitumen content (% in mass)**	**Limestone filler content (% in volume)**	**Hydrated lime content (% in volume)**	**Filler volume concentration (c_f=V_{filler}/ V_{mastic}) (%)**
B3550	0	0	100	0	0	0
B3550-5.1-0	59.8	0	40.2	34.7	0	34.7
B3550-5.1-1.25	50	9.8	40.2	28.6	7	35.6
B3550-5.1-2.5	40.1	19.7	40.2	22.6	13.9	36.5

3. EXPERIMENTAL RESULTS

3.1. Complex shear modulus G*

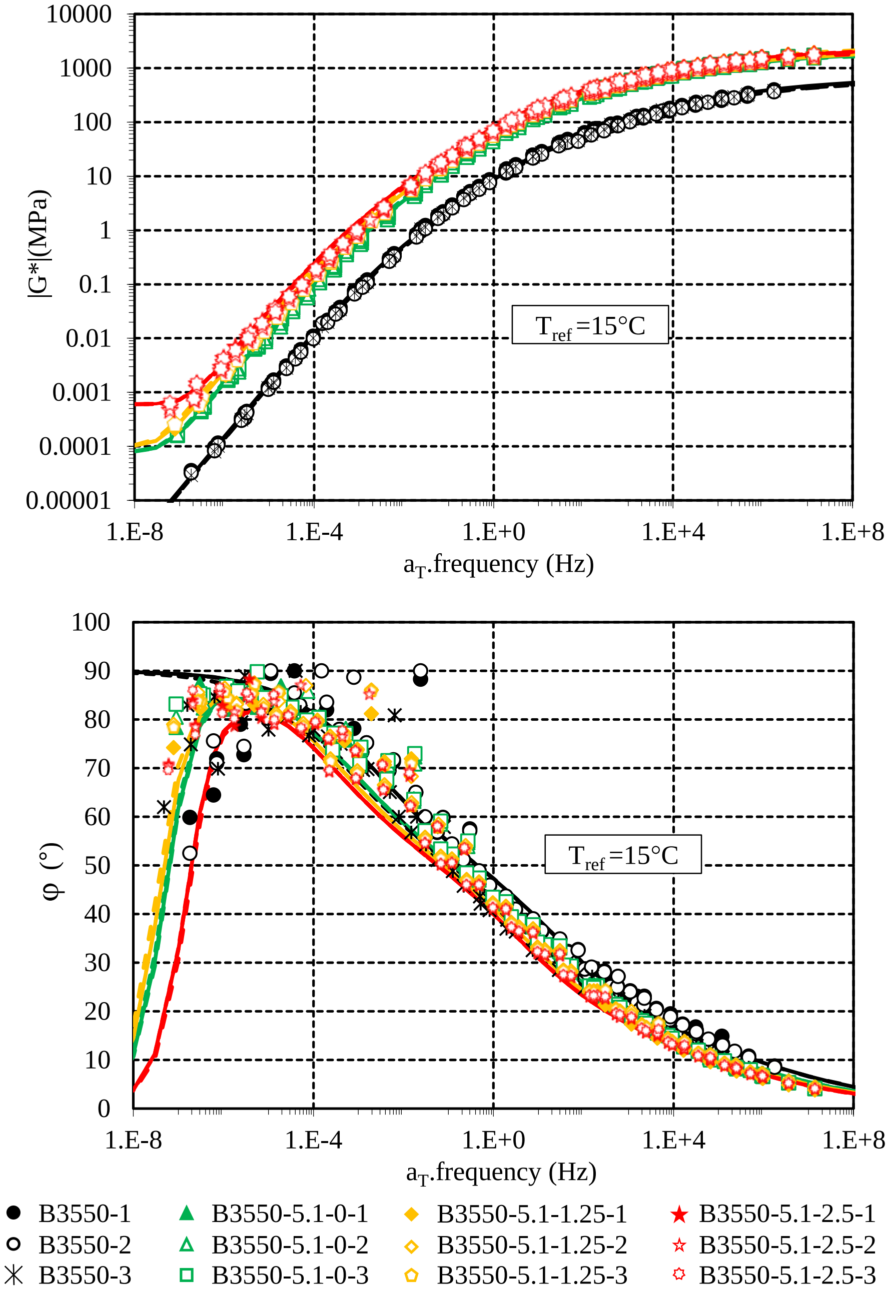
Figure 2 shows the curves in the Cole-Cole and in the Black's spaces for the 3 replicates tests on the 4 materials. The Cole-Cole diagram plots the imaginary part of complex shear modulus (G*) as a function of its real part. The curve in Black's space plots the norm of G* as a function of its phase angle. The observed unique curve for each sample validates the Time –Temperature Superposition Principle (TTSP) in the linear viscoelastic (LVE) domain for all the materials.

The master curves for the norm of the complex modulus (|G*|) and for its phase angle (φ) are plotted in Figure 3 at a reference temperature T_{ref} equal to 15°C. The horizontal axis is the equivalent frequencies (which is equal to a_T multiplied by the frequency (f)) and the vertical axis are the norm of the complex modulus and the phase angle, respectively. Some discrepancy may appear in Cole-Cole and Black's diagram, but the master curves show a good repeatability. The limit of accuracy for measurement of phase angle at high temperature (70°C) seems reached for the bitumen (whose phase angle is expected to tend towards 90° at high temperature).



Lines: 2S2P1D modelling

Figure 2 : Experimental results for tested materials: complex modulus in Cole-Cole axes; complex modulus in Black diagram. 2S2P1D model was used to simulate data (Table 3)



Lines: 2S2P1D modelling

Figure 3: Experimental results for tested materials: master curves of the complex modulus (norm and phase angle). 2S2P1D model was used to simulate data (Table 3)

3.2. Shift factors

The shift factor values (a_T) used for the construction of the master curves are presented in Figure 4 as a function of temperature. A good repeatability is found for a_T values between the 3 replicates for all materials. Furthermore, values of a_T are close for all tested materials. The constants C_1 and C_2 of the WLF (Williams, Landel and Ferry 1955) law (equation 3) used to fit a_T data, are given in Table 3.

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

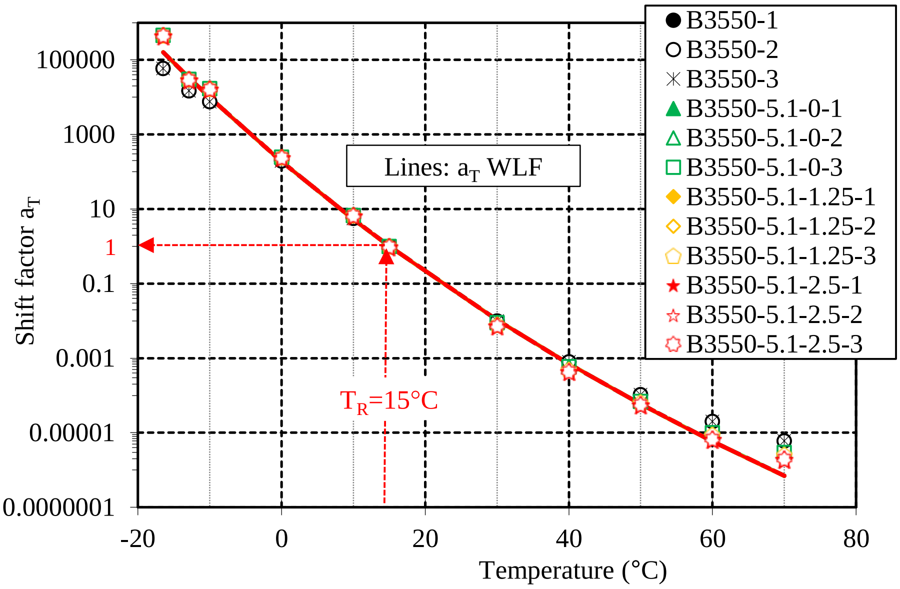


Figure 4: Shift factors a_T identical for G* of the tested materials and WLF fitting law (equation 4) (constants given in Table 3)

4. MODELLING

The 2S2P1D (2 Springs, 2 Parabolic creep elements and 1 Dashpot) model, developed at the University of Lyon/ENTPE, is a generalization of the Huet-Sayegh model [15]. This model is based on a simple combination of physical elements (spring, dashpot and parabolic creep element). The graphical representation of the 2S2P1D model is given in Figure 5. This model is widely used to model the linear viscoelastic unidimensional and tridimensional behavior of bituminous materials (including bitumens, mastics and asphalts) [4; 15; 16; 17; 18; 19].

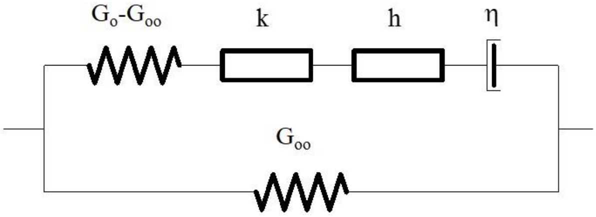


Figure 5: Analogical representation of 2S2P1D model (Olard and Di Benedetto 2003; Di Benedetto et al. 2007)

The complex modulus at a given temperature, is given by equations 4:

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

where: j is the complex number defined by $j^2 = -1$

ω is the pulsation, $\omega = 2\pi f$, (f is the frequency)

k, h: constant such as $0 < k < h < 1$; δ: constant

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

β: parameter linked with η, the Newtonian viscosity of the dashpot, $\eta = (G_0 - G_{00})\beta\tau$

τ: characteristic time values, which is the only parameter depending on temperature:

$$\tau(T) = a_T(T).\tau_0 \qquad (5)$$

where a_T is the shift factor at temperature T. At reference temperature T_{ref}, $\tau = \tau_0$. Seven constants (G_{00}, G_0, δ, k, h, β, τ_0) are therefore required to completely characterize the LVE properties of the tested materials at a given temperature. The evolutions of τ was approximated by WLF law (equation 3). τ_0 was determined at the chosen reference temperature T_{ref} = 15°C. When the temperature effect is considered, the number of constants becomes nine, including the two WLF constants (C_1 and C_2 calculated at the reference temperature) (equation 3).

2S2P1D simulations of the G* for the tested materials are presented in Figure 2, 3. As can be seen in these figures, the model fits rather well the data on the whole range of temperatures and frequencies. Used 2S2P1D constants are reported in Table 3 for all samples.

Table 3: Constants of the 2S2P1D model and WLF fitting curves for the tested samples (reference temperature: 15°C).

Material	**Sample**	**G***							**WLF**	
		G_{00}	**G_0**	**k**	**h**	**δ**	**τ_0**	**β**	**C_1**	**C_2**
B3550	1	0	680	0.225	0.59	3.8	0.00016	230	30	213
	2	0	620							
	3	0	630							
B3550-5.1-0	1	0.00008	2060				0.00053			
	2	0.00008	2000							
	3	0.00008	2200							
B3550-5.1-1.25	1	0.0001	2100				0.00086			
	2	0.0001	2500							
	3	0.0001	2130							
B3550-5.1-2.5	1	0.0006	2330				0.0012			
	2	0.0006	2130							
	3	0.0006	2330							

The constants exposed in Table 3 show an interesting property of the 2S2P1D model. It has already been shown by previous authors [3; 4; 18; 19] that k, h, δ and β constants are only dependent on the bitumen origin, regardless of its penetration. τ_0 which is also bitumen dependent and other parameters (G_0, G_{00}) depend on the granular skeleton characteristics. All studied materials were designed with the same 35/50 penetration grade bitumen, k, h, δ and β constants are also found independent of the materials.

5. ANALYSIS OF RESULTS

5.1. Normalised curve

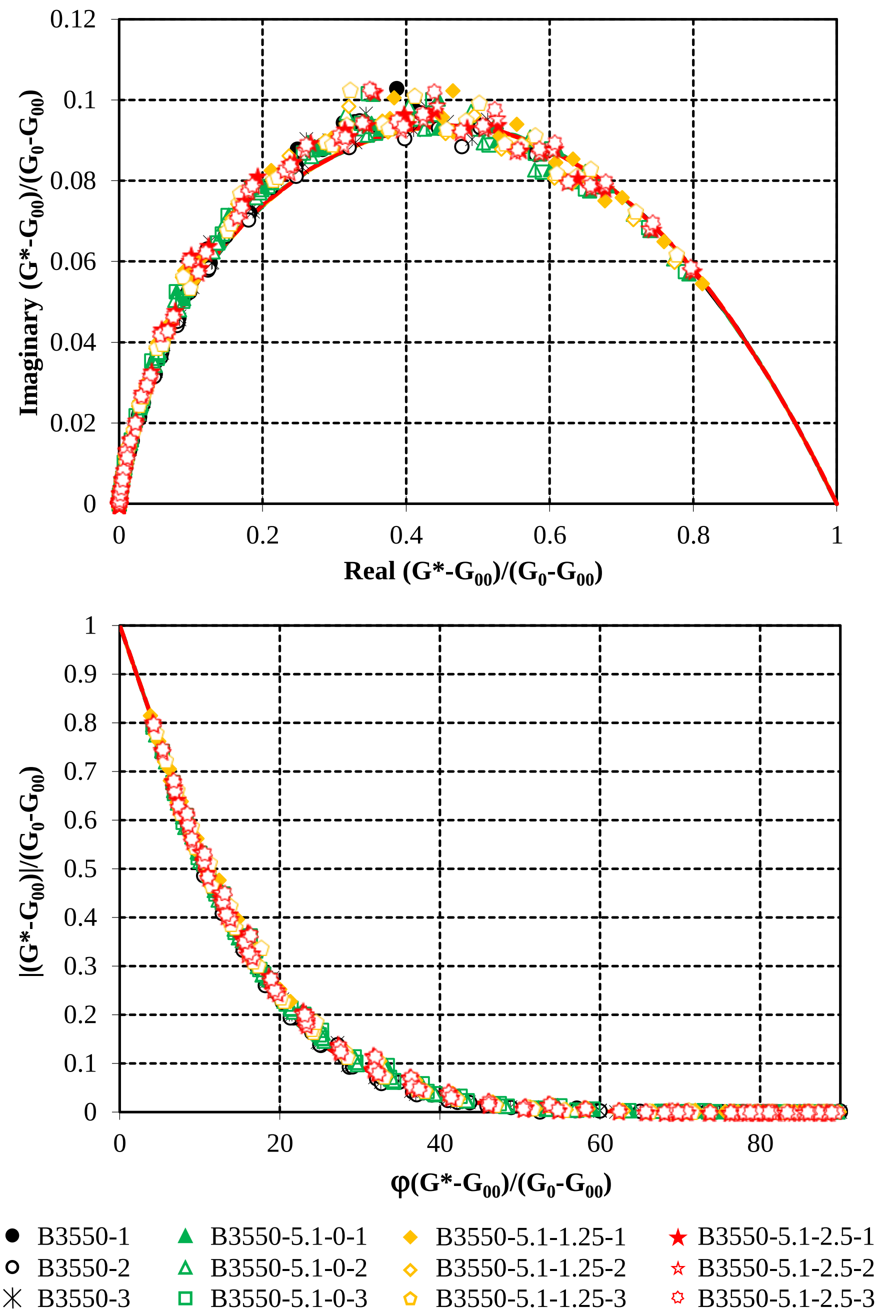
In this section, normalised curves are considered in order to compare the materials.

Normalised complex modulus G^*_{nor} is calculated as follows:

$$G^*_{nor} = \frac{G^* - G_0}{G_0 - G_{00}} \qquad (6)$$

The viscoelastic behaviour of each material may be observed, without the influence of asymptotic values G_0 and G_{00}, which are expected to depend on filler characteristics.

The same representations may be used for G^*_{nor} as for G*, namely Cole–Cole axes, the Black diagram (Figure 6) and master curves (Figure 7).



Lines: 2S2P1D modelling

Figure 6 : Normalised complex modulus in Cole–Cole axes and in the Black diagram.

Figure 6 shows that there is no observable difference between the tested materials. Master curves are also identical if plotted for the same $\tau_{0,}$ which corresponds to different reference temperatures, as indicated in figure 7. In these figures 6 and 7, the master curve obtained with the 2S2P1D model is also plotted considering $\tau_0 = 0.00053$ (T_{ref}= 10.99°C for B3550, T_{ref}= 15°C for B3550-5.1-0, T_{ref}= 16.52°C for B3550-5.1-1.25, T_{ref}= 17.57°C for B3550-5.1-2.5) and k, h, δ and β given in Table 3. The important output is that all curves superimpose with pure bitumen results. This confirms that properties of mastics are inherited from the properties of bitumen in these axes. The filler may only influence the G* asymptotic values (G_0 and G_{00}) and τ_0 value.

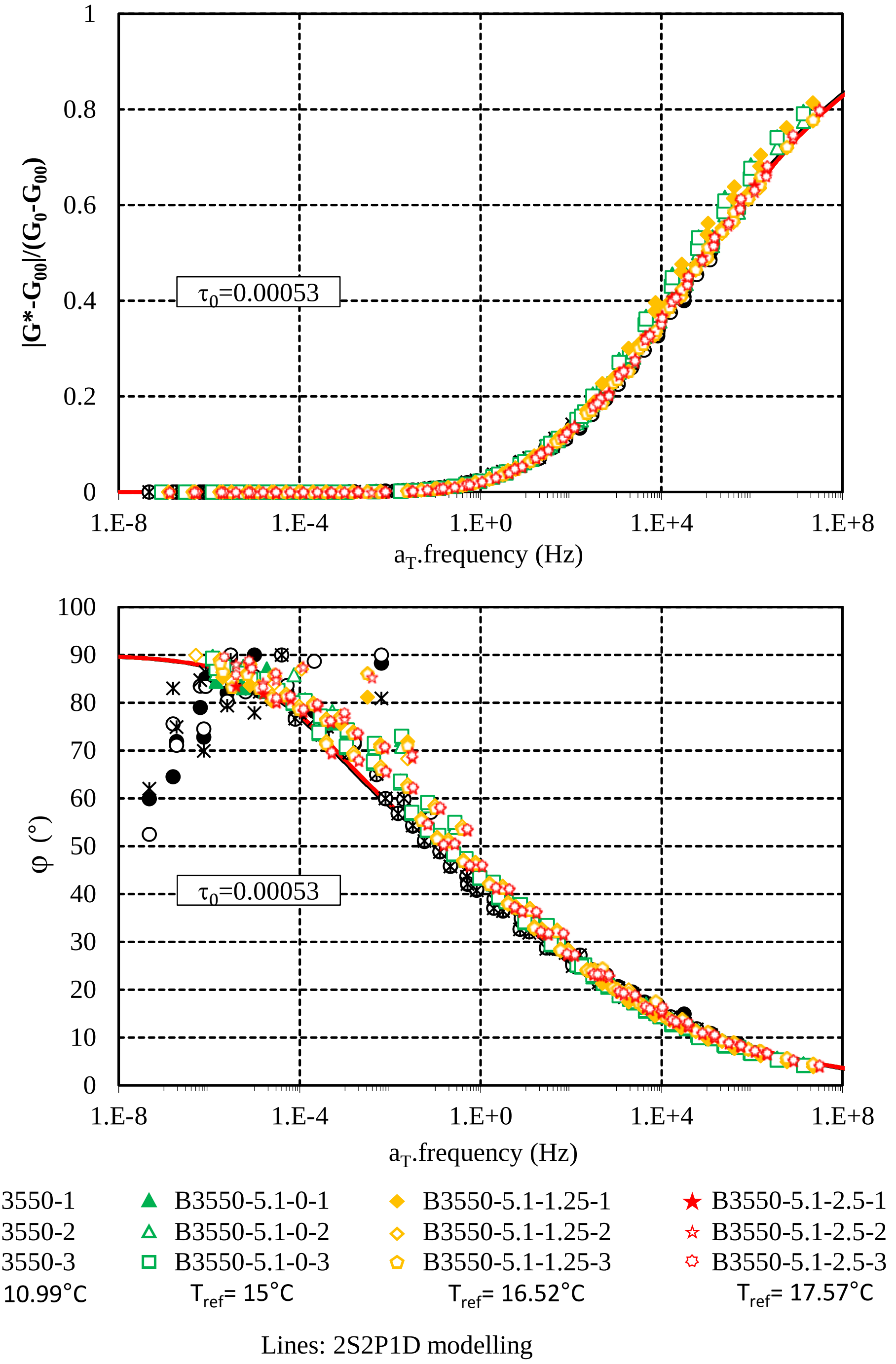


Figure 7 : Master curves of the normalized complex modulus (norm and phase angle) with $\tau_0 = 0.00053$

5.2. Effect of hydrated lime

As explained before, the three different mastics were produced using the same bitumen, the same bitumen content (in mass) and the same filler content (in mass). For two mastics, hydrated lime replaces a part of limestone filler keeping the filler total mass constant. As the density of hydrated lime is lower than the one of limestone filler, the filler volumetric concentration increases with hydrated lime content. The comparison between the results obtained for the three mastics, with and without hydrated lime, allows estimating coupling effect of hydrated lime and volume concentration of filler on linear viscoelastic properties of mastic. For each material, the values used for the comparison are average values of the 3 replicates tests.

The complex reinforcement coefficient R_L* is introduced to quantify the reinforcement effect due to the presence of hydrated lime on the complex modulus of the mastic. R_L* is defined as the ratio between the complex modulus of the mastic with hydrated lime at the equivalent frequency f_e and the one of the mastic without hydrated lime at the same equivalent frequency f_e, follows:

$$R_L^* = \frac{G^*_{mastic\ with\ hydrated\ lime}(f_e)}{G^*_{mastic\ without\ hydrated\ lime}(f_e)} \qquad (7)$$

R_L^* is a complex number. Its norm and its phase angle could be calculated in the Equation 8:

$$|R_L^*| = \left|\frac{G^*_{mastic\ with\ hydrated\ lime}(f_e)}{G^*_{mastic\ without\ hydrated\ lime}(f_e)}\right| \qquad \varphi_L = \varphi_{mastic\ with\ hydrated\ lime} - \varphi_{mastic\ without\ hydrated\ lime} \qquad (8)$$

It has to be underlined that, as the bitumen and the 3 mastics have the same shift factors, complex modulus of bitumen and mastics are considered at the same temperature – frequency couples.

In Figure 8, $|R_L^*|$ and φ_L were plotted as a function of equivalent frequency. This figure reveals that the mastic containing hydrated lime (but also with higher volume concentration of filler) exhibits a stiffer modulus $|G^*|$ than the one of the mastic without hydrated lime. At 15°C and 10 Hz, which accounts for the French pavement design method, this effect is close to 22% ($|R_L^*|=1.22$) with B3550-5.1-1.25 and 36% ($|R_L^*|=1.36$) with B3550-5.1-2.5. These results may suggest that the stiffening effect of hydrated lime is temperature-dependent, as was already observed on mastics [4; 20; 21]. Regarding φ_L, this parameter is always negative, showing that the use of hydrated lime filler (keeping filler total mass constant) causes a reduction of the phase angle of the mastic. It can be concluded again that theses mastics containing hydrated lime exhibits less viscous properties.

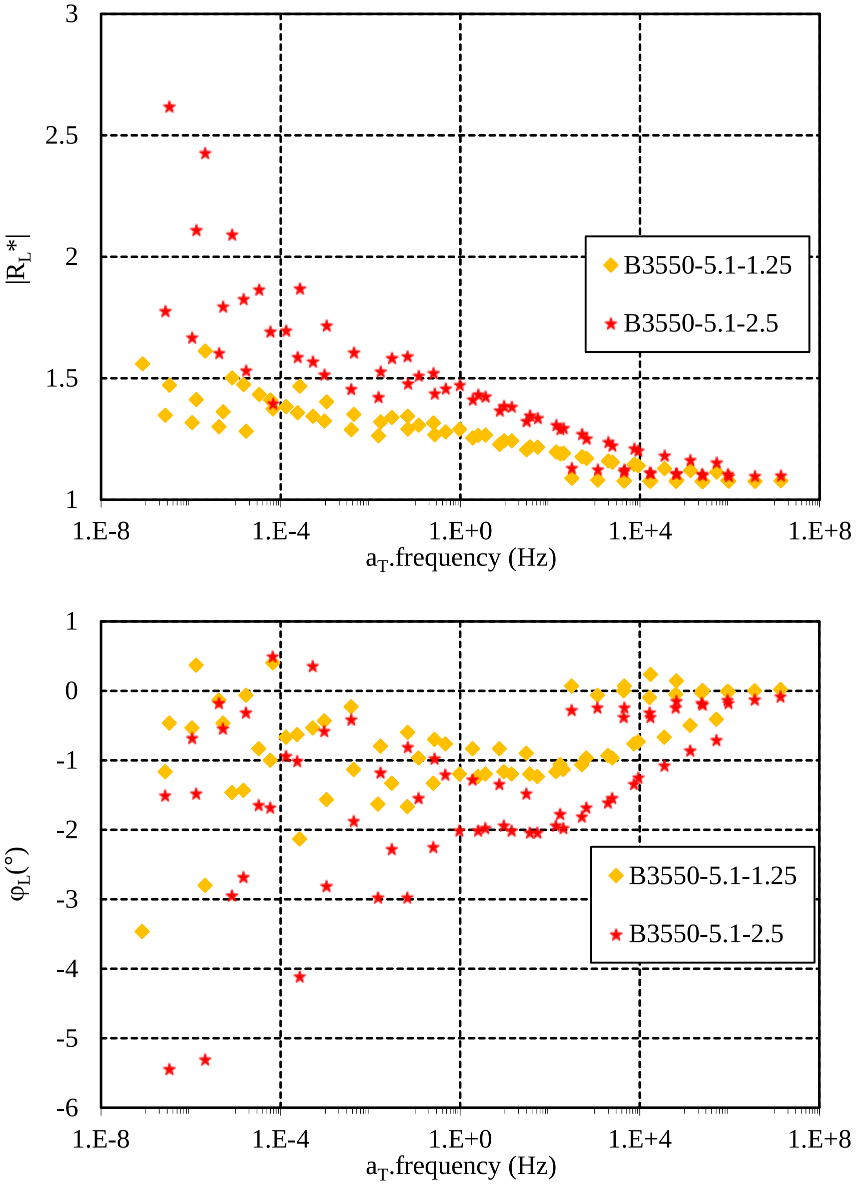


Figure 8 : Complex reinforcement coefficient R_L^* of 2 mastics B3550-5.1-1.25 and B3550-5.1-2.5. Norm (top) and phase angle (bottom). T_{ref}=15°C

As explained before, it is possible to superimpose the master curves of normalised complex modulus by choosing different reference temperatures for each material (see Figure 7). Considering the 2S2P1D model, the shift on the equivalent frequency axis could also be obtained by changing the τ_0 value obtained at a reference temperature of 15°C. The variation of this parameter τ_0 [or in an equivalent way the corresponding variation of chosen reference temperature (for $\tau_0 = 0.00053$)] can be studied. It can be interpreted as a temperature (or frequency) shift and gives an indication of the stiffening (or softening) between the tested materials.

In order to evaluate the effect of hydrated lime, both parameters τ_0 and G_0 are plotted as a function of hydrated lime content in Figure 9. These parameters are the only one, with G_{00}, likely influenced by filler, as shown with normalised complex modulus (as already shown by Delaporte et al., 2007). Figure 9 reveals that G_0 and τ_0 increase with lime content. Even if the presence of hydrated lime has a clear impact on these parameters, it is not possible to determine the origin of this effect, chemical or physico-chemical (due to hydrated lime, which would be considered as "active" filler), or physical (due to the increase of volumetric concentration of filler).

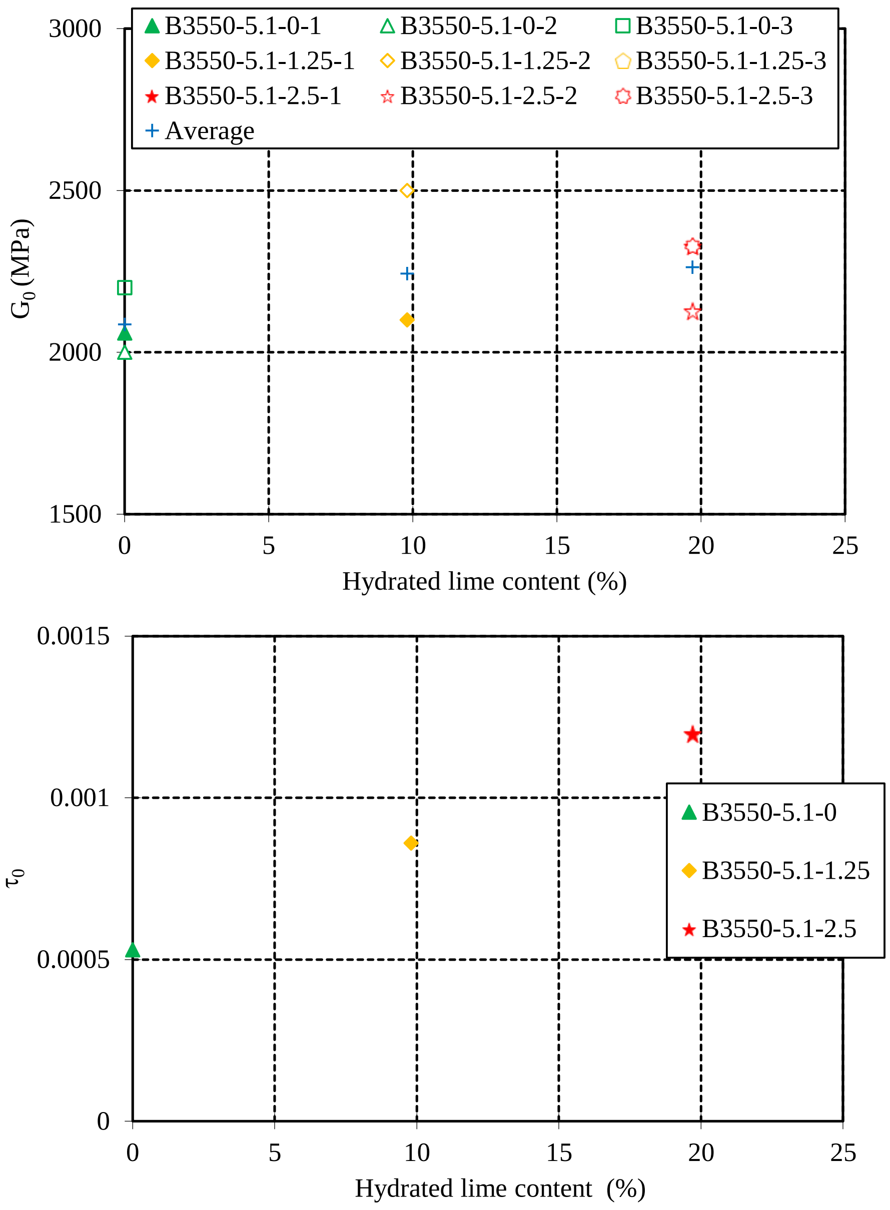


Figure 9 : G_0 and τ_0 as a function of hydrated lime content (in mass) in the mastic

6. CONCLUSIONS

The objective of the research work presented in this paper was to study the linear viscoelastic behaviour of mastic containing hydrated lime. A bitumen and three mastics having same content in mass of filler but with and without hydrated lime were tested using the dynamic shear rheometer (DSR). The potential for reinforcement when replacing a part of limestone by same mass of hydrated lime is quantified on the whole range of temperatures and frequencies by the complex reinforcement coefficient R_L*. It is a new coefficient introduced in this study. The following conclusions can be drawn from the obtained results:

- The Time-Temperature Superposition Principle was verified for the four tested materials. The shift factors used for the construction of the master curves can be considered as identical for the bitumen and three mastic with and without hydrated lime.
- The 2S2P1D model simulates correctly the complex modulus data on the whole range of considered temperatures and frequencies.
- Normalised parameter G^*_{nor} shows no difference between the 4 materials in the Cole–Cole and the Black axes. This result confirms that Cole–Cole and the Black curves are only function of the bitumen.
- The reinforcement effect of hydrated lime on linear viscoelastic properties of mastics was observed. It should be underlined that, as mass is kept constant, volume of filler increases when limestone is replaced by hydrated lime. It is then not possible to discriminate between an "active" influence of the hydrated lime (chemical for instance) and the volumetric concentration increase due to the presence of hydrated lime in the analysis.

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