Complex shear modulus and phase angle of crumb rubber modified binders containing organic warm mix asphalt additives

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ABSTRACT

Warm Mix Asphalt (WMA) is a new technology that has been developed to reduce the production and compaction temperature of asphalt mixtures and, consequently, greenhouse gas emissions and energy consumption. The main objective of this technology is to improve the mixture´s workability without significantly affecting the mechanical properties of the pavement materials. However, since WMA technology is still under study, crumb rubber modified (CRM) binders remain under investigation and the influence of these additives must be clearly identified.

This research presents the elastic, viscoelastic and viscous properties at intermediate and high temperatures of 15% and 20% CRM binders that incorporate different types and quantities of WMA additives. The study reports the results of the tests performed over a wide range of temperatures and frequencies using a dynamic shear rheometer (DSR).

The results of this research suggest that CRM binders with WMA additives are appropriate for countries with hot climates because the additives increase the complex modulus (stiffness) at the high temperatures and as the phase angle curve was shifted on lower values over a wide range of frequencies thus avoiding permanent deformation (resistance to rutting). The study also shows that an increased content of additive shifted the complex modulus in high frequency regions and as a result the stiffness at low temperatures was slightly increased.

Keywords: crumb rubber; warm mix asphalt; additive; binder properties, dynamic shear rheometer (DSR), complex modulus, phase angle, master curves, wax

1. Introduction

Warm mix asphalt (WMA) technology may be the solution to the main drawback of mixtures containing crumb-rubber modified (CRM) binders: larger amounts of greenhouse gas (GHG) emissions are produced (compared to conventional mixtures) due to higher production temperatures. The rubber lends greater viscosity to the binder but rubberized binders used in the construction of flexible pavements result in an improvement in resistance to rutting, fatigue and thermal cracking. They lower traffic noise and maintenance costs as well as contributing to prolonged pavement life [1-4].

Reducing the manufacturing and compaction temperatures will lead to a lower energy consumption and GHG emissions. The benefits of the WMA technology combined with the effective reuse of a solid waste product would make asphalt rubber (AR) mixtures with WMA additives an excellent, environmentally-friendly material for road construction.

However, it is necessary to guarantee a lower viscosity of the CRM binders without affecting bitumen performance at pavement service temperatures. Although some research has been carried out regarding organic additives in bitumen: the determination of the wax content [5, 6], crystallization properties [7, 8], chemical structure [9, 10] or their influence on bitumen and asphalt performance [11-15]; the influence of organic additives on CRM binders has not been studied in great detail [16, 17]. For temperatures above the wax relative melting point, the change in rheology was different and mainly related to a reduction of viscosity with respect to the base bitumen used. Due to these reductions, a general drop in the theoretical mixing and paving temperatures was recorded for both paraffinic and polyamide-like waxes [18].

To understand pavement performance it is important to first understand the rheological properties of bitumen. Mixtures with stiff bitumen may be more susceptible to fatigue and cracking and those with binders that suffer deformation and flow easily are more prone to rutting. There are conventional tests (softening point and ductility) which predict the maximum temperature, the permanent deformation properties and the cohesive properties. Nevertheless, this basic characterization cannot completely describe the viscoelastic properties needed to relate fundamental physical binder properties, especially for modified binders. In fact, to represent the bitumen´s behaviour, the cyclic (oscillatory) and creep tests are considered to be the best techniques [19].

In order to evaluate the high and intermediate temperature properties of different CRM binders with WMA additives, a dynamic shear rheometer (DSR) was used to determine the elastic, viscoelastic and viscous properties of the binders over a wide range of temperatures and frequencies.

2. Materials and test program

The following sections describe the materials used throughout the whole investigation.

2.1. Virgin binder, crumb modifier and WMA additives.

A bitumen 50/70 was chosen for this research as virgin binder is widely used to produce asphalt mixtures at conventional temperatures. Only one batch of crumb rubber manufactured by mechanical grinding at ambient temperature (50% from truck tyres and 50% from car tyres) was used in this research to ensure consistency.

The basic specifications of this bitumen (penetration grade and softening point), the fractionation analysis as specified in the NLT 373/94 standard and the crumb rubber gradation and thermo-gravimetic analysis is detailed in previous published studies [16, 17].

For this study, only one of the existing WMA technologies was chosen (organic additives), and a comparative study between two different kinds of waxes (Sasobit® and Licomont BS 100®) was carried out. The first additive used is a Fischer–Tropsch (F-T) wax; a long-chain aliphatic hydrocarbon wax produced by treating hot coal with steam in the presence of a catalyst (melting range of pure wax between 95°C and 110°C). The other organic additive is a fatty acid amide manufactured synthetically by reacting amines with fatty acids and pure melts between 120°C and 155°C.

The rheological characterization at high and intermediate temperatures was carried out for 11 different CRM binders. Two of them were the control binders, B15 and B20, which are CRM binders with 15% and 20 % by weight of rubber added to a 50/70 neat bitumen (B). All the others are CRM binders with the selected WMA additives and the dosage rate of the WMA additives referred to the bitumen weight were 2% and 4%. In Table 1 there is a list of all the binders and their corresponding names that will be referred to hereafter.

Table 1
Binders tested

Binder name	Bitumen / Rubber (%)	Additive (%)	Additive name
B	100/0	0	-
B15	85/15	0	-
B15+2S	85/15	2	Sasobit
B15+4S	85/15	4	Sasobit
B15+2L	85/15	2	Licomont BS 100
B15+4L	85/15	4	Licomont BS 100
B20	80/20	0	-
B20+2S	80/20	2	Sasobit
B20+4S	80/20	4	Sasobit
B20+2L	80/20	2	Licomont BS 100
B20+4L	80/20	4	Licomont BS 100

2.2. Preparation of CRM binders containing WMA additives

An oil bath with a maximum temperature of 225ºC, a mixer with a maximum velocity of 15,000 rpm, fitted with a propeller agitator and a one-litre metal container for mixing was used for the preparation of the binders. The oil bath has a temperature probe which can be introduced into the mixing receptacle, allowing the temperature of the binder to be controlled with a precision of ± 1 °C. A bitumen sample of 750 g was heated at 140 °C and then placed in the oil bath. WMA additives were carefully added to the bitumen and the blends were subsequently mixed for 15 minutes at 4,000 rpm, ensuring that the additive was properly incorporated. The blend was then heated to 185ºC and the crumb rubber was added. The mixture was blended for 30 minutes at 2,000 rpm then for another 30 minutes at 900 rpm at a constant temperature of 185ºC. Reheating and homogenization were carefully carried out at a controlled temperature in order to obtain reproducible results [20]. Special attention was then paid to the thermal history and storage conditions of the test samples before testing (1 h at 25 °C ± 0.5 °C), because of their influence on rheological measurements [21].

2.3. Rheological tests

To provide information about the trend of the complex modulus and the phase angle in function of frequency sweeps, the tests were performed at different temperatures. A fixed shear stress at a constant temperature was applied during the dynamic tests and the angular frequency changed between 1 and 100 rad/s.

Shear stress defined in literature for similar tests were assumed (The shear stress applied depends on the test temperature and must belong to the linear region for the analysed binder). Table 2 contains a list of the chosen couple test temperature – shear stress.

Table 2
Temperature shear stress couple

Test Tª (ºC)	**Shear Stress (Pa)**
-10	40,000
0	20,000
10	10,000
20	5,000
30	2,000
40	1,000
50	500
60	200
70	100
80	50

The stress sweeps consist in the application of an increasing shear stress on the specimen at a fixed frequency and temperature: at 10°C the shear stress changes between 10^3–10^6 Pa while at 40°C the range becomes 40 – 40000 Pa. The frequency sweeps were done from 1 to 10 Hz in the temperature range between 10 and 80ºC at 10ºC intervals. The plate–plate configuration, 25 mm diameter and 2 mm gap sample geometry was used.

The frequency sweeps were carried out inside the linear viscoelastic region of the studied asphalts. The temperature during the plate's geometry change, from 8 mm diameter to 25 mm diameter, was between 20°C and 30°C, as recommended in the literature, in order to obtain results which best fit the real trend [30].

The superposition principle (TTSP) gives equivalence between temperature and frequency and allows the construction of a representative curve, designated master curve, for the material for a wider frequency range. The TTSP implies that the rheological data obtained at higher and lower temperatures can be equated simply and graphically with lower and higher frequency respectively. With the data obtained, frequency sweeps can be used to plot a single curve at a reference temperature for an extended frequency range through shift factors. Materials which verify the TTSP principle are called "thermo-rheologically simple" [22].

According to TTSP, master curves are generated by selecting a reference temperature and using shift factors to fit an overall continuous curve at a reduced frequency or time scaled: the shift factors are applied to the frequency values and they assume different values for each temperature data series until the curves converge into a single smooth function.

The amount of the shifting required at each temperature to form the master curve is of particular importance and it is performed by defining the shift factors. Master curves are realized for both phase angle and complex modulus versus the reduced frequency.

3. Results and discussion

3.1. Frequency dependence comparative for control binders

The binders were tested with a Dynamic Shear Rheometer (DSR). The standard used for this test is EN 14770:2012 (Determination of complex shear modulus and phase angle - Dynamic Shear Rheometer (DSR)) [23].

Fig. 1 shows that the addition of rubber to the neat bitumen increases complex modulus G*, consequently, the stiffness of the binders is increased. The phase angle values decreased, as the addition of rubber increases the elasticity of the binders. The reference temperature was 60°C.

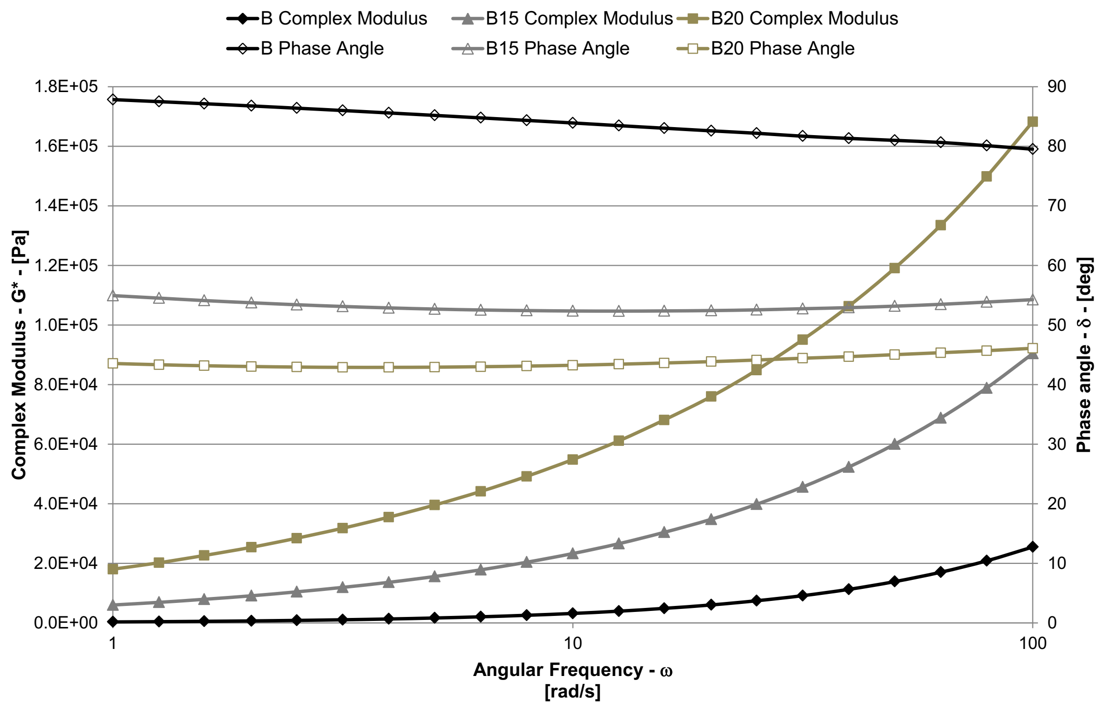

Fig. 1.Frequency dependence comparative for control binders (reference temperature 60°C).

3.2. Frequency dependence for 15% and 20% CRM binders with WMA additives

The influence on frequency dependence of B15 of each WMA additive with increasing contents can be seen in **Fig. 2** and **Fig. 3** and for 20% CRM binders can be observed in **Fig. 4** and **Fig. 5**. In each case both complex modulus G* and phase angle δ are plotted at the reference temperature 30°C. As may be observed, for the 15% and the 20% CRM binders both additives increased the complex modulus, and as a result, both waxes increased the stiffness of their respective control binder. The organic additives also lowered the phase angle, increasing elasticity significantly. As a result the mixtures containing additives will have superior stiffness and elasticity, and this is due to the presence of waxes in crystalline form to realize a new molecular organization of the bitumen.

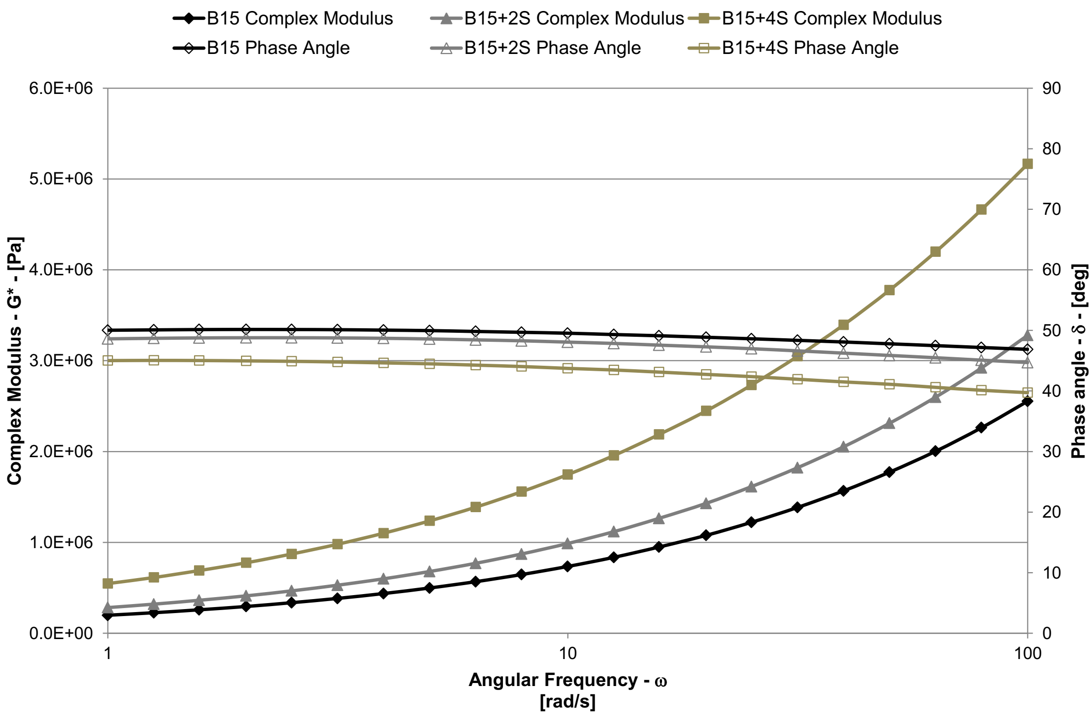

Fig. 2 Influence of Sasobit® on frequency dependence of B15 (reference temperature 30°C).

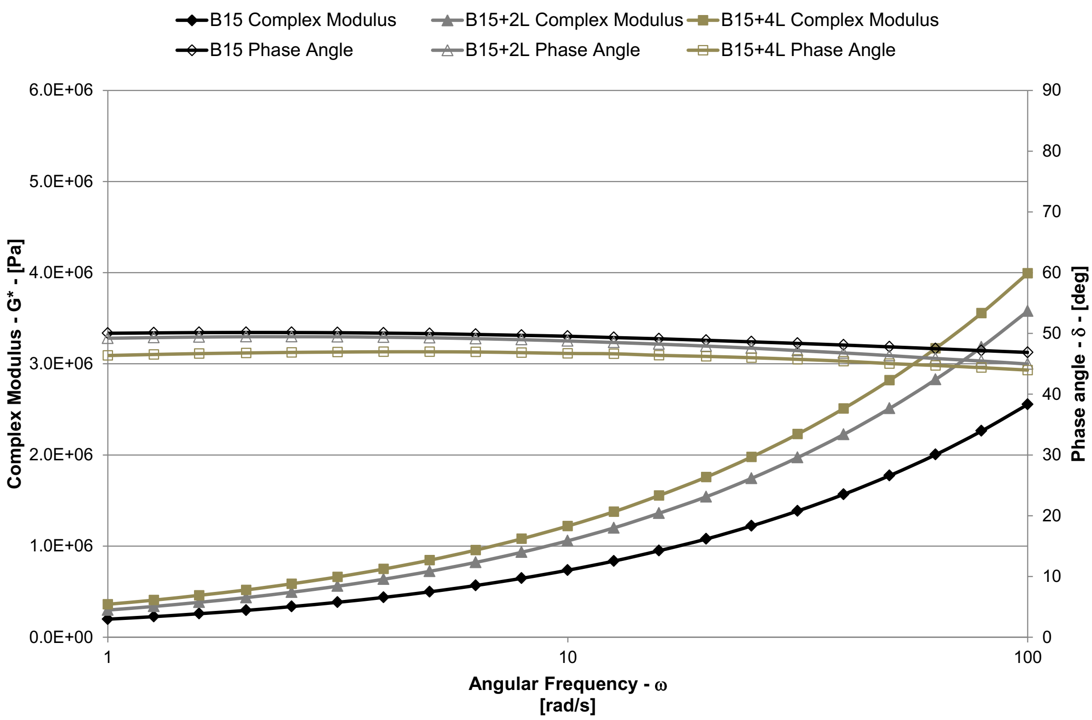

Fig. 3 Influence of Licomont BS 100® on frequency dependence of B15 (reference temperature 30°C).

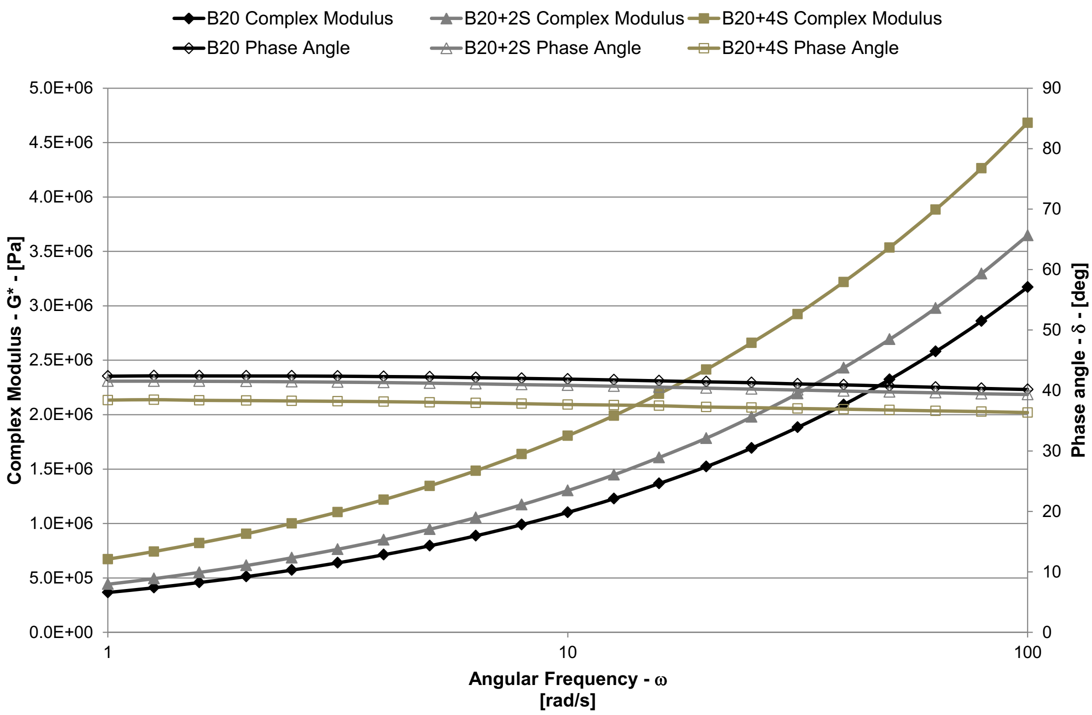

Fig. 4 Influence of Sasobit® on frequency dependence of B20 (reference temperature 30°C).

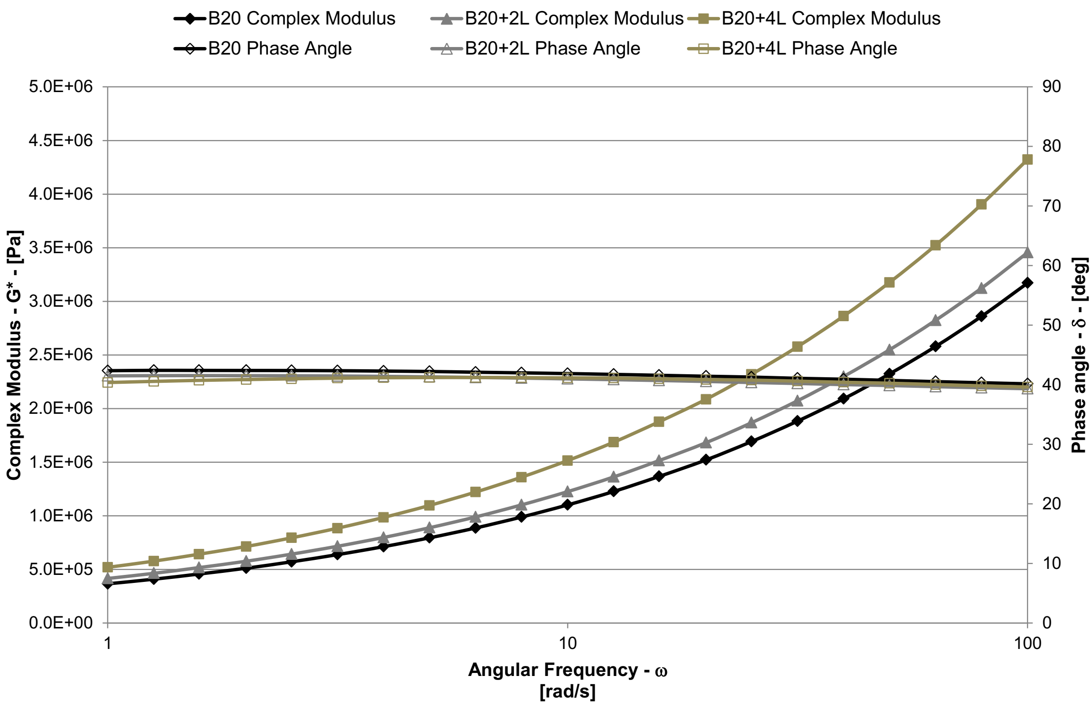

Fig. 5 Influence of Licomont BS 100® on frequency dependence of B20 (reference temperature 30°C).

In **Fig. 6** and Fig. 7 the complex modulus and the phase angle of the binders containing rubber and 4% the waxes studied are plotted together for comparison. In both cases, the additive that leads to the highest complex modulus and to the lowest phase angle is Sasobit®, therefore, this wax increases stiffness and elasticity more than Licomont BS 100®.

On the other hand, as the amount of rubber increased, the stiffness and the elasticity of the binders also increased. Hence, the pavements containing binders with rubber and any of the additives studied offer much greater stiffness and elasticity, which also result in resistance to permanent deformation.

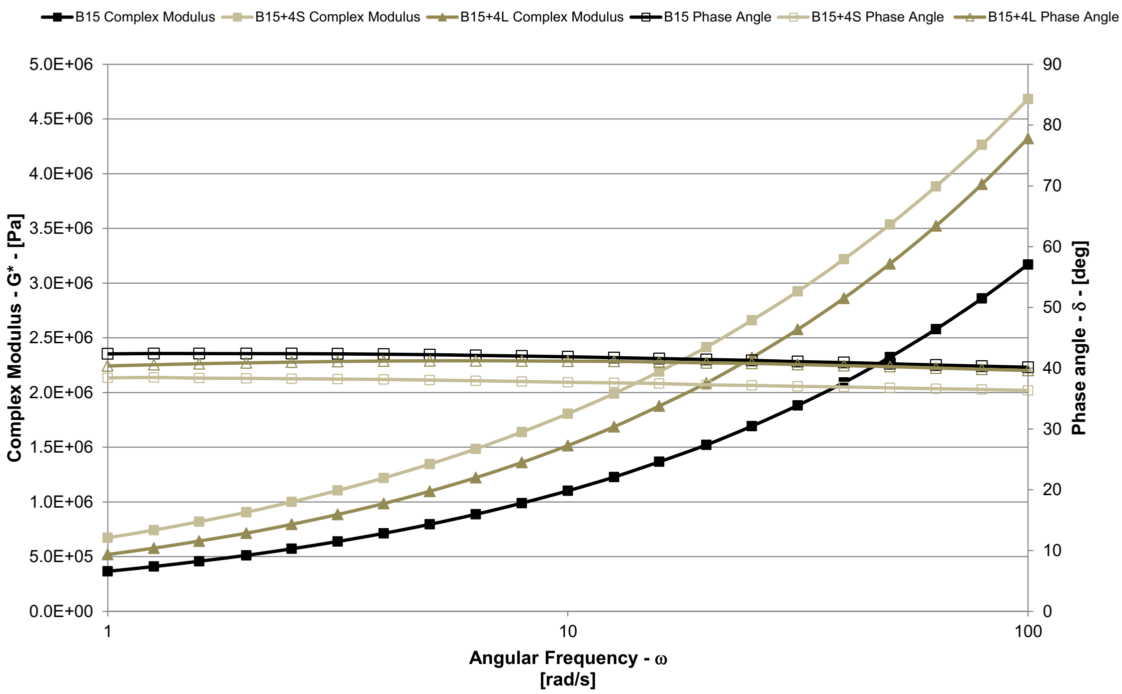

Fig. 6 Frequency dependence comparative for 15% CRM binders (reference temperature 30°C).

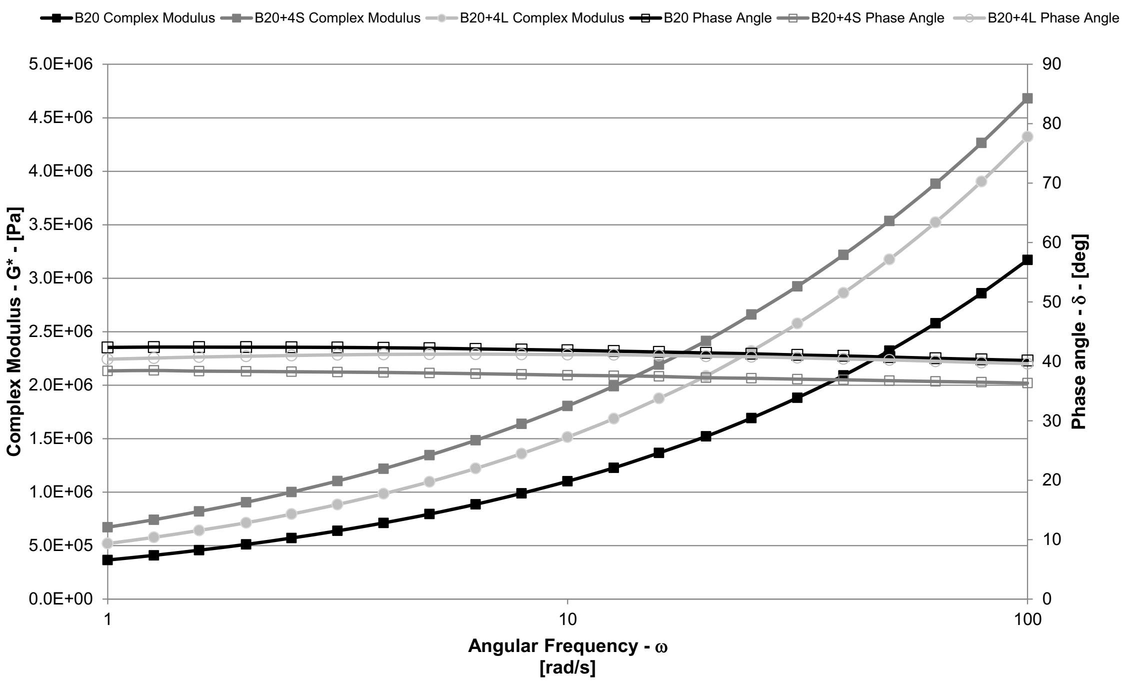

Fig. 7 Frequency dependence comparative for 20% CRM binders (reference temperature 30°C).

3.1. Master curves comparative for control binders

Fig. 8 illustrates a comparative study between the control binders of this study. Considering the complex modulus master curves, it can be seen that the addition of increasing percentages of rubber shifts the curves on higher values in the low-frequency region (high temperatures) and on lower values in the high-frequency region (low temperatures). Compared to the base bitumen, this implies a double effect on the resistance of the pavement regarding both permanent deformation resistance at high temperatures and thermal cracking at low temperatures. At high frequencies (rapid load conditions), B20 has a lower value compared to B15. This proves that the addition of rubber improves the behaviour of the binder at low temperatures. At low frequencies (slow load conditions), the higher rubber content shows a relevant increase in the binder stiffness and as a result the resistance to permanent deformation is improved.

The phase angle curves also show evident differences; a marked inflection Starting over the wide range of frequencies closely due to the presence of rubber is observed. The data series recorded at different temperatures appear quite discontinuous. It was also noted that as the addition of rubber to the base bitumen increases, the phase angle decreases over a wide range of frequencies.

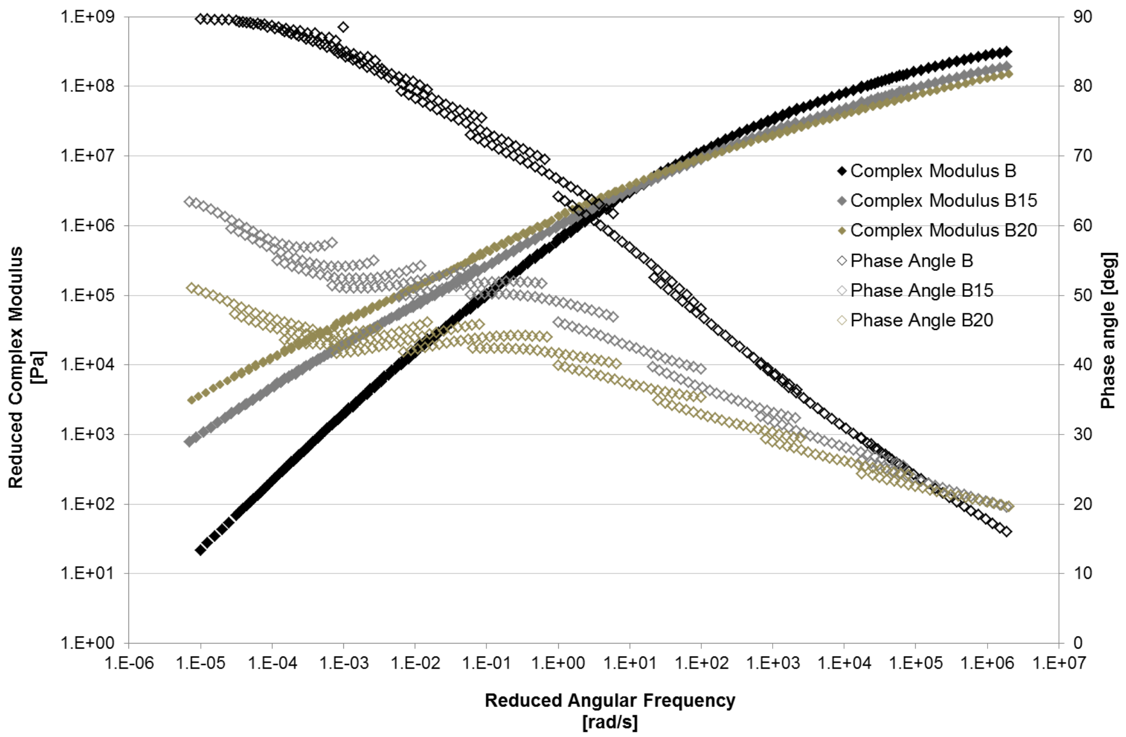

Fig. 8 Master curves comparative for control binders

3.2. Master curves for 15% and 20% CRM binders with WMA additives

Fig. 9 and Fig. 10, illustrate the effects of the WMA additives on a 15% CRM binder and the effects of the WMA additives on a 20% CRM binder are presented **in Fig. 11 and Fig. 12.** It can be observed that in all the cases the addition of increasing percentages of additive shifts the curves on higher values in the low-frequency region (high temperatures) and in the high-frequency region (low temperatures).

It is noteworthy to mention that in all the cases over the wide range of frequencies, the phase angle decreases as the amount of additive increases, thus, all the waxes studied improve the elasticity of their respective control binder. The phase angle curves show a marked inflection due to the presence of rubber and additive and the data series recorded at different temperatures appear quite discontinues.

The contribution of the waxes in the crumb rubber modified bitumen gives final values of very high modulus, also higher than what is required by more traditional applications in road pavements. The result is so emblematic of a bituminous binders very extremes that have also a higher cost of production. However, the exasperation of modifier content allows better highlight and better understand the rheological responses of binders.

Master curves of B describe the canonical behavior of the unmodified bitumen. All rheological functions have monotonic and continuous curves. At higher reduced frequencies (typically referable to lower test temperatures), the storage modulus tends to a constant value of about 1 GPa.

At lower reduced frequencies (typically referable to temperatures higher than 60°C), the loss modulus approaches the viscous asymptote with slope equals to 45° in the bi-logarithmic plot. In the same region, the phase angle tends to 90°.

The situation changes with the addition of crumb rubber where the rubber digestion process maintains the presence in the sample of the particles that make less than perfect continuity of the master curves of the phase angle. The master curves of phase angle shows a landing in the slope of the curve at the intermediate temperatures and a reduction of crossover frequencies.

The changes in the shape of the master curves are much more detectable with the addition of organic additives. The first macroscopic difference is that for B and B15 or B20 /wax blends the Time Temperature Superposition Principle (TTSP) does not apply, or at least not in the whole investigated temperature (frequency) range.

The simple explanation is that the physical meaning of TTSP for a viscoelastic material implies that the temperature does not affect its structure. A material is thermo-rheologically simple if temperature only alters the Brownian dynamics of molecules and molecular segments, hence changing only the absolute value of the relaxation times and not the overall relaxation function [24]. All binders containing organic WMA additives being their structure strongly dependent on temperature as temperatures approach those of the interval start melting of the waxes.

The rheological changes and the TTSP failure are not only due to the wax melting. The structural changes are again ascribable to re-arrangements of the network formed by the solid particles of binders. Keeping in mind the partial unreliability of data generated by the TTSP, we can still compare the master curves of the base bitumen and the blends (**Fig. 13** and **Fig. 14**).

Starting from high reduced frequencies, low temperatures, we can observe that the moduli partially overlap. This corresponds to a modest stiffening effect exerted by the wax, but confirms that at low temperatures the rheology of the blends is mainly controlled by the base bitumen. At low frequencies, especially in presence of amidic additive, the phase angle does not describe the conventional monotonic trend. In this region, the amidic wax introduces fundamental modifications in bitumen viscoelasticity with a reduction of the phase angle of phase up to values of 30°.

As both types of WMA additives increase the complex modulus, the different rheological behavior can be explained by the different chemical nature of the same.

A long-chain aliphatic hydrocarbon wax due to its apolar characteristic, probably cannot establish any specific interactions with bitumen molecules or, whether interactions occur, they have to be limited to weak affinity with maltenic oils. On the other side, thanks to their polar nature amidic waxes are probably prone to bond themselves with polar fractions of bitumen: asphaltenes and resins. This affinity and polar attraction can form a sort of gel structure where the solid-like units are made by alternation and association of asphaltene molecules and wax crystals [25] while the rubber powder has an preferencial interaction with the maltenic oils of the bitumen.

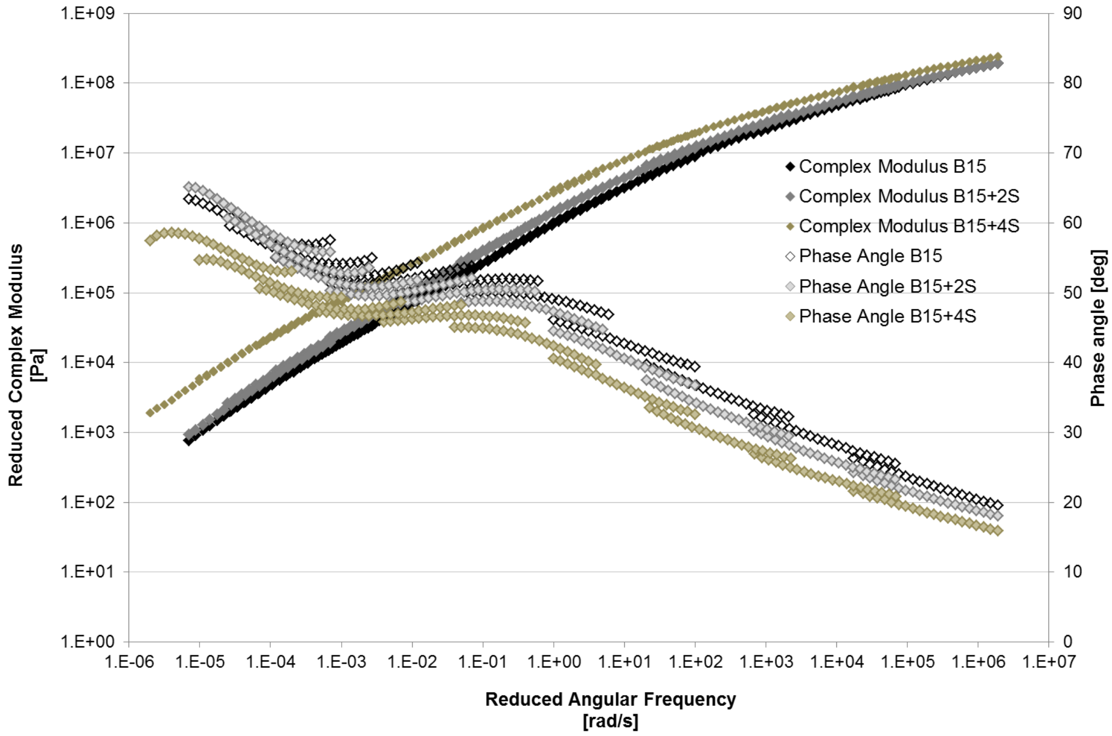

Fig. 9 Master curves for B15 with Sasobit®

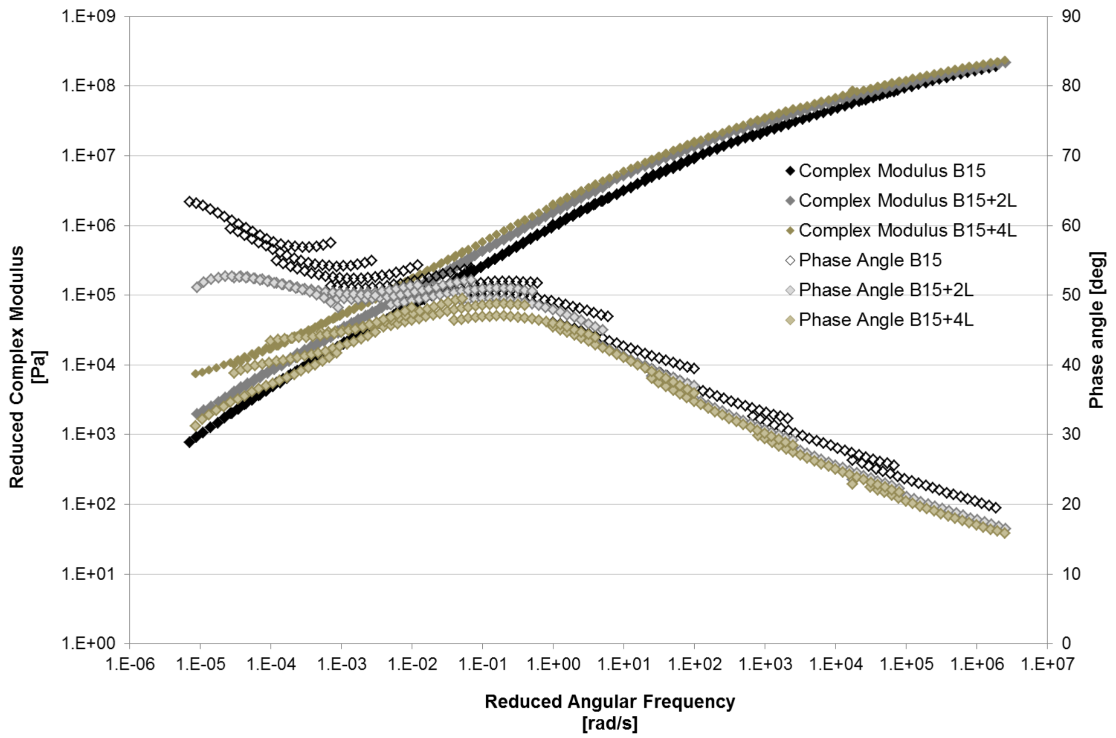

Fig. 10 Master curves for B15 with Licomont BS 100®

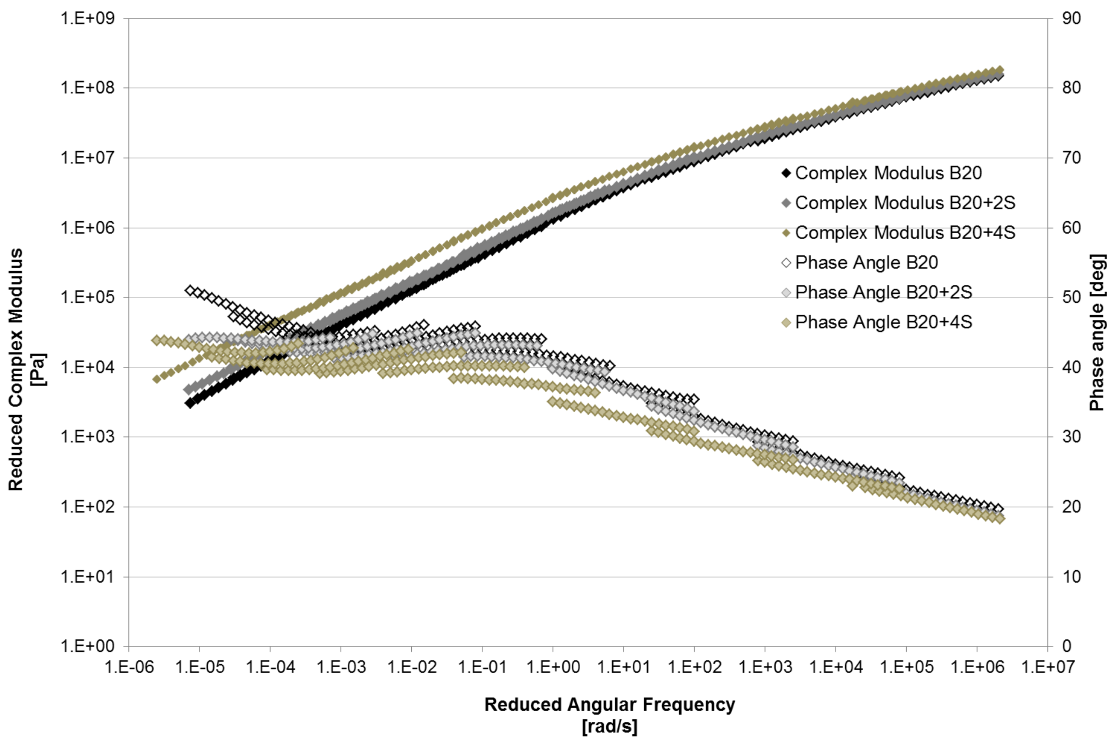

Fig. 11 Master curves for B20 with Sasobit®

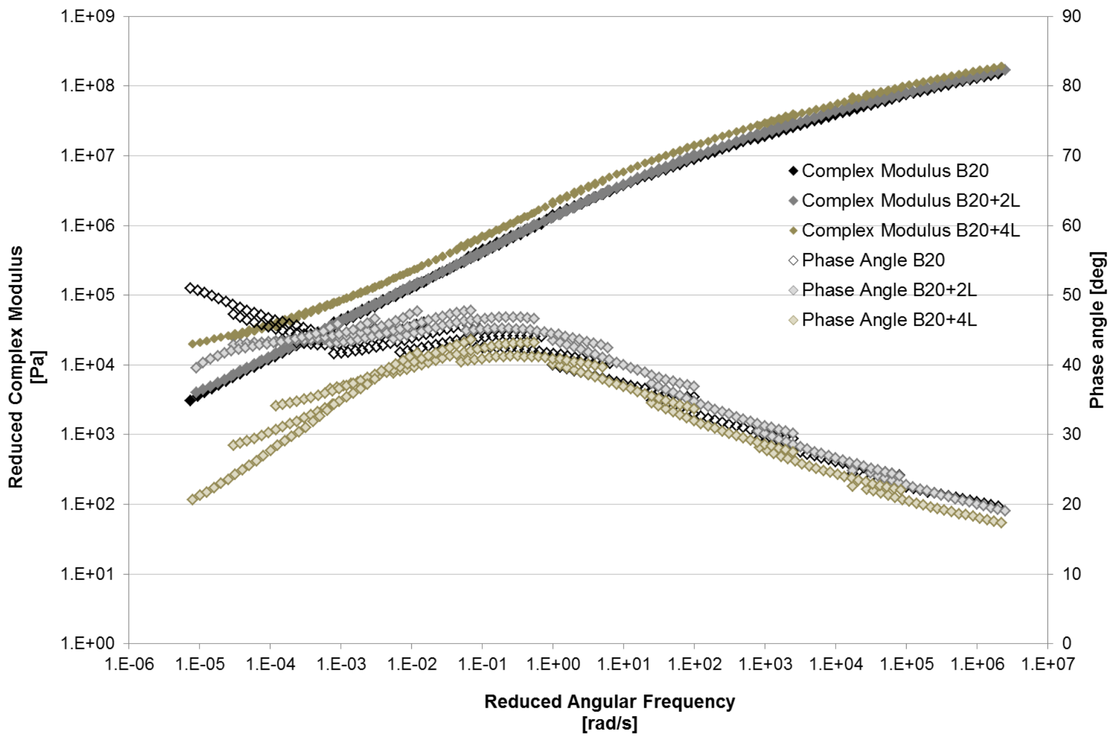

Fig. 12 Master curves for B20 with Licomont BS 100®

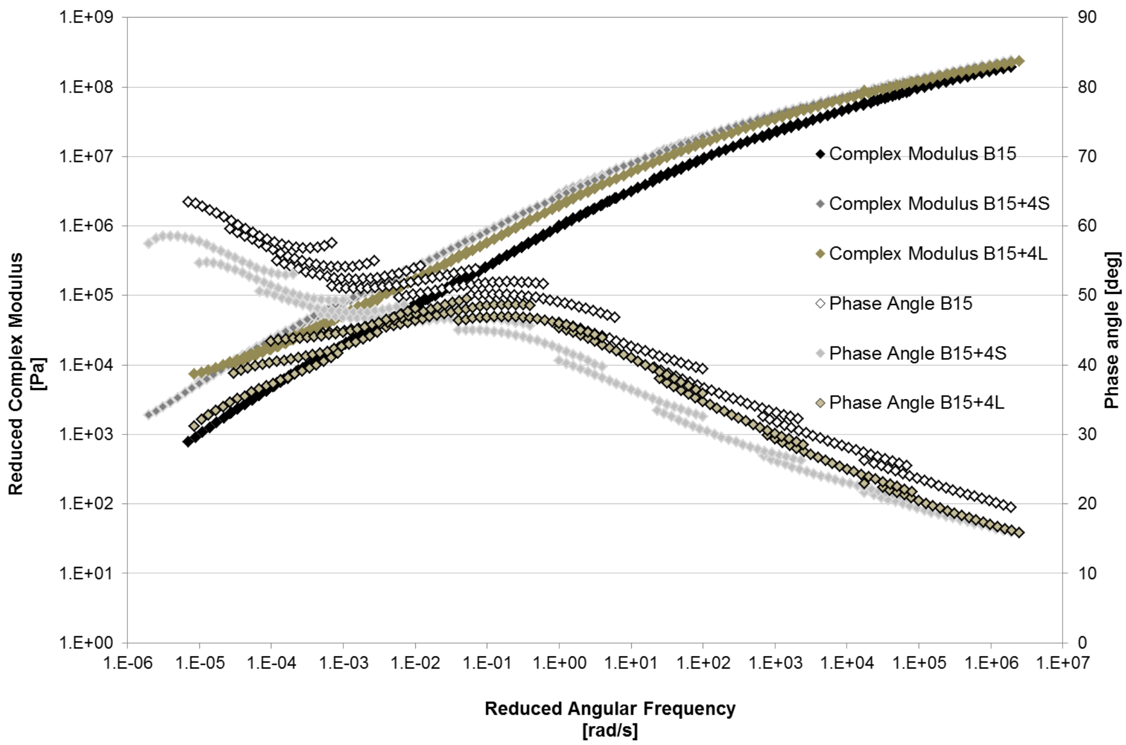

Fig. 13 Master curves comparative for 15% CRM binders

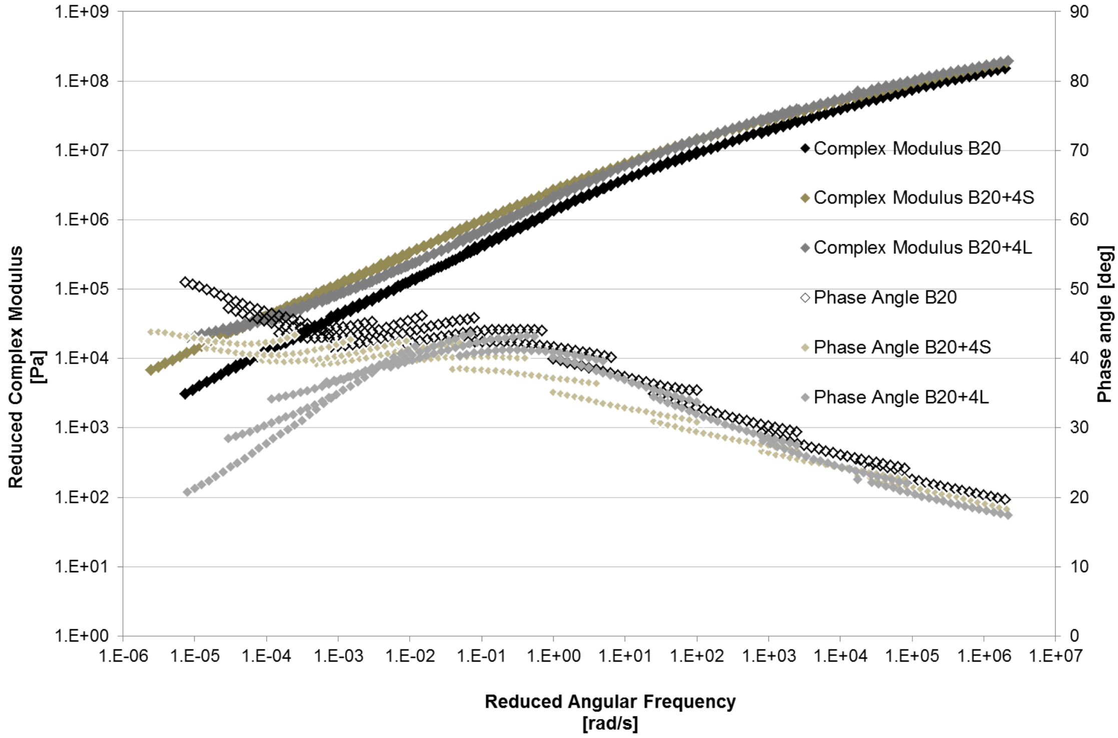

Fig. 14 Master curves comparative for 20% CRM binders

4. Conclusions

In this study, the synergistic use of two bitumen modifiers, rubber and Warm Mix Asphalt (WMA), is designed to improve the workability of the blends while paving roads by combining the elastic properties of rubber with the fluidizing effect due to the melting of the wax at certain temperatures. In order to evaluate the high and intermediate temperature properties of crumb rubber modified (CRM) binders with WMA additives, they were manufactured using different percentages of CRM (15% and 20% by modified binder weight) and two WMA additives (Sasobit® and Licomont BS 100®) at 2% and 4% content. Each binder was tested using a dynamic shear rheometer (DSR) which defines the elastic, viscoelastic and viscous properties of bitumen over a wide range of temperatures and frequencies. The following conclusions were drawn for the binders created:

- For all the binders studied, the effect of the WMA additives was the same. Compared to their respective control binders, at high frequencies (low temperatures/rapid load conditions), the stiffness was slightly increased, meaning that thermal cracking at low temperatures will be more likely to appear. On the other hand, it must be noted that in the high frequency region the addition of rubber improves the binder behaviour at low temperatures when the 15% and 20% CRM binder were compared to the base bitumen. At low frequencies (slow load conditions/high temperatures), the higher content of any of the additives studied shows a relevant increase in the binder stiffness and therefore resistance to permanent deformation is improved at high temperatures avoiding rutting. At low frequencies the addition of rubber improves the blends behaviour.

- It was also observed that as the amount of additive increases, the phase angle decreases over a wide range of frequencies, demonstrating that the addition of the studied waxes makes the binders more elastic, especially at lower frequencies (high temperatures), improving the binder behaviour. Also, the increasing content of rubber shows a relevant increase in the binder stiffness and thus, the resistance to permanent deformation is improved due to the presence of the two bitumen modifiers used in this study. This implies a double effect on the improvement of the resistance of the pavement regarding permanent deformation.
- It was found that CRM binders with WMA additives have a peculiar and quite complex mechanical response, connected also with the melting/crystallizing properties of the wax and the degree of residual crystallinity into the blend. The effects of wax on the binder rheology were found to be mainly controlled by the chemical nature of the organic additive.
- The complex modulus (stiffness) is increased at the high temperatures avoiding permanent deformation and the phase angle curve is shifted on lower values over a wide range of frequencies. These increases represent a positive contribution to road pavements, although implying a higher cost.

To summarize, despite the non-applicability of the time-temperature superposition principle, at least not for all investigated frequencies, the master curves of the CRM binders have highlighted the significant change produced by the additives regarding their rheological behaviour. As engineering implications emerge as increased contents of rubber and wax shifted the curves on higher values in the low-frequency region (high temperatures). And on the other hand, increased contents of rubber shifted the curves on higher values in the high-frequency region (low temperatures). This demonstrates that compared to a conventional mixture which contains the neat bitumen, the mixtures containing rubber and WMA additive in the binder will improve the resistance of the pavement to rutting at high temperatures and that, at low temperatures, although the WMA additive deteriorates the blend´s behaviour regarding thermal cracking, this is compensated by the presence of rubber. In this study it was also observed that a higher content of WMA additive increased notably the elasticity of the respective control binder as the phase angle decreased over a wide range of frequencies (consequently a wide range of temperatures). Finally, the results of this research indicate that CRM binders with WMA additives are appropriate for countries with a hot climate although further tests should be made to evaluate the behaviour of the mixtures that contains these binders at low temperatures.

Compliance with Ethical Standards:

The authors declare that they have no conflict of interest.

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