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ORIGINAL ARTICLE

Comparison of methods for measuring zero shear viscosity in asphalts

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Abstract Permanent deformation or “rutting” is a common mode of failure in asphalt pavements. In order to better determine why rutting occurs, current research is focussed on the rheological properties of the asphalt binder. Zero shear viscosity (ZSV) seems to adequately explain how the asphalt binder contributes to the rutting behaviour of the pavement. Still, the measurement of ZSV in a reliable and reproducible way is an open field of discussion. This work looks into the repeatability, benefits and duration of two test methods to measure ZSV: the creep test and frequency sweep test. To account for the influence of the asphalt type, six different conventional and modified asphalts were tested. A statistical analysis was performed to study the variability of each test method and a comparison between both was made.

Keywords Rutting · Zero shear viscosity · Frequency sweep test and creep test

1 Introduction

Rutting is a common mode of distress of asphalt pavements. It is defined as the progressive accumulation of permanent deformations produced by traffic. The main factors contributing to this process are increased traffic densities, heavy loads, slow traffic and high temperatures. The primary causes that induce excessive permanent deformations in asphalt concretes are: poor quality of materials, faulty mixture design, poor construction practice and/or incorrect selection of the appropriate asphalt type.

The asphalt contribution to permanent deformation process has traditionally been handled by looking at the asphalt binder’s consistency based on penetration and softening point tests (ASTM D 5-86 and D 36-89, respectively). Nowadays, with the addition of polymer modified asphalts (PMAs), the asphalt rutting characterizations attained through these empirical tests is insufficient. Determining the fundamental rheological properties is the proper manner to characterize the asphalt binder’s rutting behaviour.

In the 1990’s, The Strategic Highway Research Program (SHRP) developed a new specification to classify asphalts based on rheological properties and their relation to the asphalt mixture performance. The SHRP specification presents the parameter $G^*/\sin\delta$ associated with pavement rutting behaviour. It is well known that rutting performance is efficiently classified by $G^*/\sin\delta$ in conventional and multigrade asphalts, but this parameter underestimates a PMAs

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behaviour [1–6], especially those with large amounts of delayed viscoelasticity, like PMAs with SBS polymers [7, 8].

The response of conventional asphalts when resisting small shear stresses is linear and their viscosity is independent of shear rate. In contrast, PMAs behave like a pseudo plastic fluid in which the rheological response depends on the shear rate. However, for very low shear rates, this behaviour turns less complex and becomes similar to conventional asphalts. In this case the energy is dissipated until the flow resistance that the asphalt structure offers reaches a constant value. At this point the viscosity becomes independent of shear rates. This viscosity is called Zero Shear Viscosity (ZSV) and is a physical property of the asphalt.

Sybilski [9, 10] suggests the use of the ZSV concept related to rutting characterization. He founds a good correlation between ZSV and rutting performance in mixtures prepared with different asphalts, including PMAs. Phillips and Robertus [11] concluded that ZSV is the key quantity determining the binder contribution to permanent deformation in asphalt pavement rutting. Current research [12, 13] shows that ZSV is a reasonable parameter for controlling the effect of the binder with respect to permanent deformation.

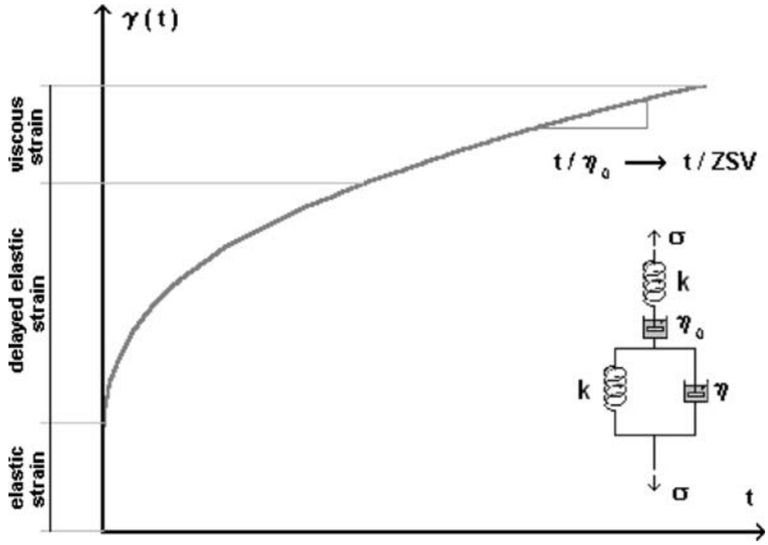
This paper compares the repeatability and advantages of two test methods for measuring ZSV: the creep test and frequency sweep test. Six different conventional and modified asphalts were studied. A statistical analysis was performed to study the variability of each method and a comparison between both was made.

2 Measurement of ZSV

There are various experimental methods to measure ZSV: creep test at a constant stress until the asphalt reaches a steady state flow, frequency sweep test, shear rate sweep test and multi creep test.

During a creep test, a constant stress is applied on a sample, and then the deformation is measured as a function of loading time. The asphalt first shows an instantaneous elastic strain followed by a delayed elastic strain, and finally, if the test duration is long enough, a pure viscous strain. This behaviour can be explained by the Burger model (Fig. 1 and Eq. 1). Usually this model is written in terms of creep compliance instead of strain. Creep compliance is defined as the ratio of measured strain to assigned stress and is proportional to deformation and independent of imposed stress provided it is sufficiently small (within linear viscoelastic domains). In this model the pure viscous strain is represented by the term t/η_o (where η_o represents the asphalt ZSV). During the test it is necessary to reach a pure viscous strain stage that is called steady state flow. When this stage is reached, the strain rate tends to be constant and the η_o obtained by applying the Burger model is the ZSV of the asphalt. The time required to achieve

Fig. 1 Creep curve and Burger's model



the steady state flow depends on the binder type and can take a few minutes for conventional asphalts and up to hours for PMAs. In some cases steady state cannot be achieved.

$$J(t) = \frac{\gamma(t)}{\tau_0} = J_0 + J_{\mathrm{d}}\psi(t) + \frac{t}{\eta_0} \quad (1)$$

where $\gamma(t)$: strain; τ_0: constant stress applied; J_0: elastic compliance (elastic strain); $J_{\mathrm{d}}\Psi(t)$: delayed elastic compliance (delayed elastic strain); t/η_0: pure viscous compliance (viscous strain); $\Psi(t)$: memory function; η_0: ZSV.

The shear stress applied to the sample must be low enough to keep the measurement inside the linear viscoelastic regime. In this regime the response to a sum of stresses is equal to the sum of responses to an individual stress [14] and, most importantly; viscosity becomes independent of the shear stress applied. In conventional binders a stress below 5,000 Pa is within this range [15], but for PMAs, a stress between 20 and 50 Pa is necessary [16].

In a frequency sweep test an asphalt sample is tested at different oscillation frequencies in a dynamic shear rheometer (DSR) at a specific test temperature. For each frequency, complex viscosity is evaluated.

Asphalt behaviour is generally characterised by decreasing viscosity with increasing frequency between two well defined values: Zero Shear Viscosity (η_0) at zero frequency and limiting viscosity (η_∞) at an infinitely high frequency (Fig. 2). For low frequencies viscosity data tend to a plateau value; a trend that is clearly visible for conventional asphalts but not for PMAs (Fig. 3). For some asphalts this plateau value is impossible to obtain [17].

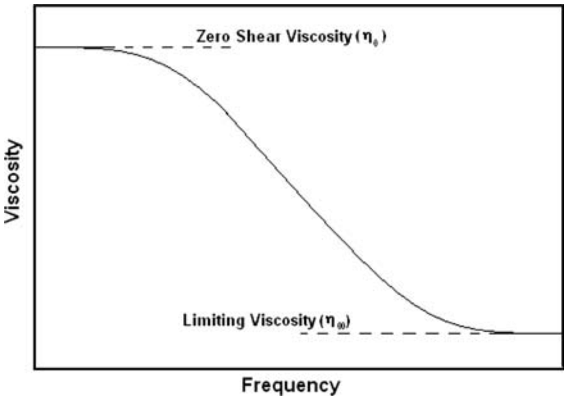


Fig. 2 Viscosity–frequency curve

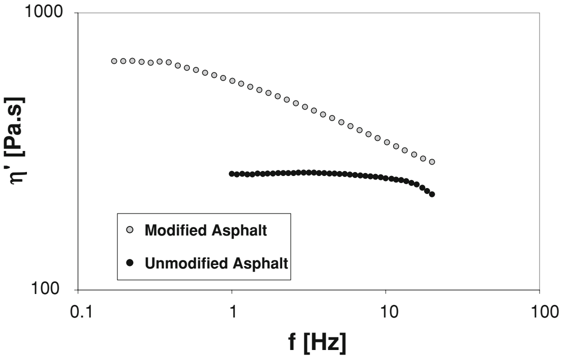


Fig. 3 Complex viscosity versus frequency for unmodified and modified asphalts

The Cross model is used to fit the complex viscosity data [18] (Eq. 2), obtaining ZSV as a result. However, in PMAs at low frequencies, the viscosity rate becomes very high so that the fit with the Cross model gives an unrealistically high value of ZSV. To resolve this issue, it is convenient to calculate the viscosity at a very low frequency, for example 0.001 Hz [19], which is known as "low shear viscosity" (LSV). The LSV can be measured directly with DSR in oscillation mode at low frequencies in combination with low strain amplitudes [20]. The smaller the frequency at which LSV is taken, the closer the value will approximate the ZSV.

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + (k \cdot f)^n} \quad (2)$$

where η: viscosity data; η_0: ZSV; η_∞: limiting viscosity; K and n: model constants; f: frequency in Hz.

The frequency sweep test must be done inside the linear viscoelastic region of the studied asphalt. With this aim, the strain sweep test defines the maximum strain limits to use on the asphalt binder. During the test, increasing strains at a constant frequency are applied to the sample and the complex modulus (G*) is measured as a function of strain. In a plot of G* versus strain (Fig. 4) it can be seen how G* remains constant at low deformations until it starts to drop at higher strain values. Airey arbitrarily defines the linear viscoelastic limit as the strain at which the G* value drops to 95% of the maximum value measured [21].

Regarding other tests for obtaining ZSV, the shear rate sweep test is similar to the frequency sweep test, but the asphalt sample is tested at different shear

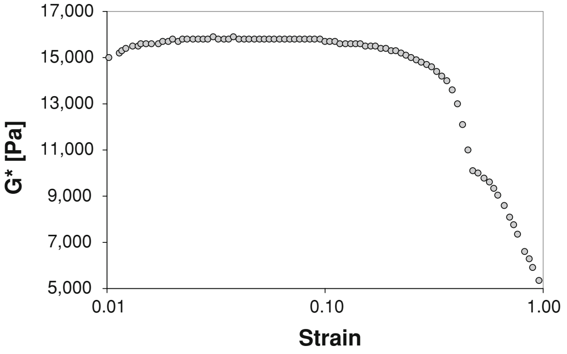


Fig. 4 Complex modulus (G*) versus strain

rates. Again the Cross model is used to fit the viscosity data and to obtain the asphalt's ZSV as in the frequency sweep test. Finally, in a multi creep test the sample is subjected to a repeated sequence of shear loading and unloading at a constant shear stress (100 cycles), during which the strain response is measured as a function of time. Then Burger's model is applied to obtain ZSV just as in creep test [22].

Many of the difficulties associated with the measurement of ZSV are related to obtaining values in the first Newtonian region, where extremely low shear rates must be applied. Sometimes this approach becomes impossible due to equipment limitations or when the Newtonian region does not exist for the asphalt being tested. Moreover, the Cross model application involves the extrapolation of data, which can result hazardous when the data does not have a clear plateau value.

3 Experimental

3.1 Asphalts

Six asphalts currently used in Argentina were selected to compare the advantages of each method for measuring ZSV, including: three unmodified asphalts (C1, C2 and C3) with different viscosity at 60°C (from 147 to 316 Pa.s), one multigrade asphalt (M) and two of the most common PMAs (P1 and P2, prepared with EVA and SBS polymer, respectively). Table 1 presents the following characteristics for each asphalt: penetration, softening point, Brookfield viscosity at 60°C, torsional recovery test results, and Performance Grades according to the SHRP specification. The properties of the original and aged asphalts obtained in the rolling thin film oven test (RTFOT) are included as well.

3.2 Test procedures

The ZSV was obtained for each binder through the frequency sweep and creep test methods. The measurements were made with a DSR Phaar Physica SM-KP controlled by a Rheolab MC-100. A test temperature of 60°C was chosen to represent a critical condition for rutting, considering the pavement temperatures that a typical road can reach. Plate–plate geometry of a 25 mm diameter and a 1 mm gap was used in all tests. Samples of the different asphalts were prepared and then both test methods were

Table 1 Asphalt binder properties

Asphalt Modification Argentina standard	C1 – CA-10	C2 – CA-20	C3 – CA-30	M Multigrade –	P1 EVA AM2	P2 SBS AM3-C
Original						
Penetration to 25°C	89	60	58	60	64	71
Softening point [°C]	47.4	54.2	51.8	58.3	69.2	88.5
Brookfield viscosity at 60°C [Pa.s]	147.2	256.0	316.0	1224.0	270.4	7472.0
Torsional recovery [%]	–	–	–	–	67.2	76.7
RTFOT aged						
Penetration to 25°C	59	44	37	42	45	53
Softening point [°C]	51.8	58.2	56.8	67.8	69.4	74.2
Brookfield viscosity at 60°C [Pa.s]	262.4	480.0	724.0	6760.0	1880.0	–
PG	*58–22*	*64–22*	*64–16*	*70–22*	*70–28*	*70–22*

carried out seven times for each asphalt type to obtain the repeatability of each method and to compare between both.

3.2.1 Creep test

Creep tests were done through DSR in stress control mode following the CENprEN 15325 standard protocol [23]. ZSV was calculated from data collected during the last 15 min of the creep test (Eq. 3). Creep tests of 1 and 4 h, for conventional and modified asphalts, respectively, were carried out according to the standard's specifications. In the particular case of modified asphalts, when the steady state was not achieved after 4 h of creep, the test was continued 4 h more and ZSV was calculated regardless of whether the steady state was achieved or not.

$$\eta_0 = \frac{900\text{s}}{J_{\text{end}} - J_{900\text{s before end}}} \ [\text{Pa.s}] \tag{3}$$

where J_{end}: final compliance of retardation creep test; $J_{900\text{s before end}}$: 900s compliance before the end of retardation creep test.

The shear stress applied to the sample must be low enough to keep the measurement inside the linear viscoelastic regime of the asphalt. Due to the DSR resolution, it was not possible to apply a stress value lower than 100 Pa in this work. However, in all studied cases the linear viscoelastic regime was ensured.

3.2.2 Frequency sweep test

Previous to the frequency sweep tests, strain sweep tests for frequencies of 1 and 10 Hz at 60°C were carried out for all studied binders (Fig. 5) to define the linear viscoelastic strain limits. Table 2 shows the maximum strain limits obtained.

Frequency sweep tests were done in the range from 1 to 20 Hz at 60°C for unmodified asphalts. In the cases of modified asphalts, two frequency sweep in the range from 1 to 20 Hz at 60 and 80°C were done (in the linear viscoelastic asphalt domain) to build a master curve at a reference temperature of 60°C using the time–temperature superposition principle valid for a viscoelastic material [24]. Then viscosity data for lower frequencies were obtained as Anderson recommends [25].

As recommended by De Visscher, the Cross model was used to fit complex viscosity data and extrapolate complex viscosity values at a frequency of 0.001 Hz [19]. The LSV values obtained by this method were assumed close to the real ZSV value and compared with ZSV values obtained from the creep test for the same binder.

4 Test results and discussion

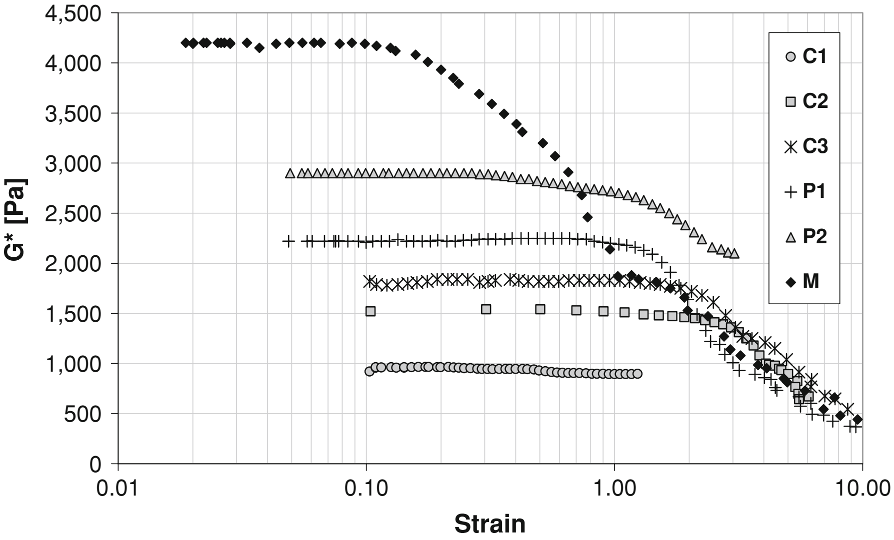
The primary goal of this work was to study the repeatability, test time consumption and benefits of the frequency sweep and the creep test methods for measuring ZSV. A statistical analysis was done to study the variability of each method and a comparison between both was made.

Table 3 shows the mean values of ZSV and LSV obtained by creep and frequency sweep test method, respectively, as well as the respective coefficient of variation (CV) for the different asphalts. The creep tests for P2 asphalt (SBS modified polymer) were dismissed because reliable measurements of ZSV were not possible to obtain. Over the three tests performed on the P2 asphalt, different and unrealistically high values were obtained. The equipment resolution was the main cause for not obtaining a reliable measurement.

Both test methods gave similar results for the other binders, as the box graphics show (Fig. 6). It is important to note that the box graphics do not show atypical extreme values.

Excluding P2 creep test results, the test repeatability was good. The CVs for the unmodified asphalts were lower than 10% and between 15 and 20% in the case of M and P1 modified asphalts for both methods. The CV for P2 frequency sweep test results was comparable to those obtained for the unmodified asphalts.

The measurements for unmodified asphalts show smaller CVs than in M and P1 modified asphalts. This fact can be explained because the modified asphalts have a more complex behaviour. The measurement is very sensitive to the polymer concentration and distribution in the sample, affecting the obtained value. The polymer network arrangement plays an important role in the variability of test results. However, the presence of SBS polymer did not have as large an effect on the repeatability of P2 asphalt.



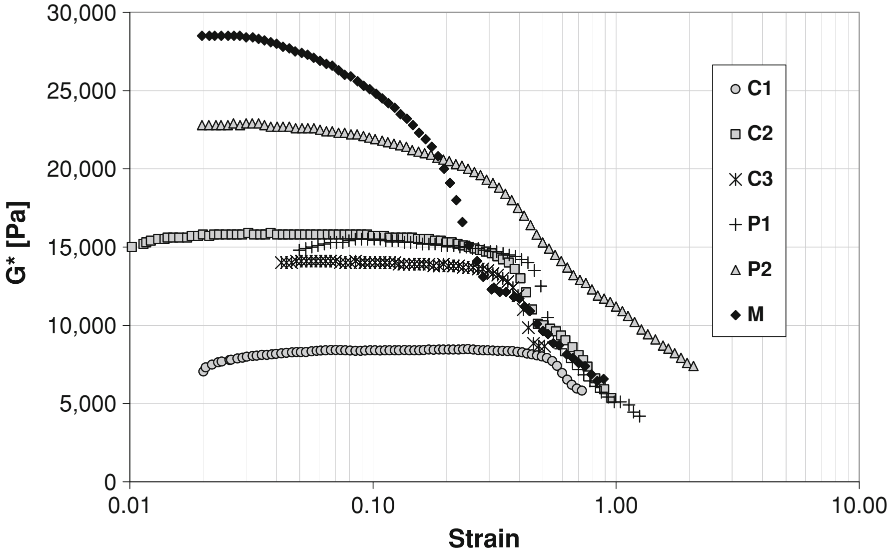


Fig. 5 Complex modulus (G*) versus strain. $f = 1$ Hz (*above*); $f = 10$ Hz (*below*)

Table 2 Maximum strain limits

Asphalt	Strain limits [%]					
	C1	C2	C3	M	P1	P2
$f = 1$ Hz	55.7	191.0	185.0	20.0	112.0	56.7
$f = 10$ Hz	47.3	23.5	17.7	5.7	32.3	11.5

The CVs obtained in this work are similar to those reported by other authors [16–26], including those reported in CENprEN 15235 round-robin [23].

The frequency sweep tests show smaller CVs than the creep tests in cases where both methods were done.

The frequency sweep test results for P2 asphalt show good repeatability. However, because no plateau region was reached during the test procedure, it would not be appropriate to assume that the LSV value obtained in the test is comparable to the asphalt's ZSV. As seen in Fig. 7, the data from two of the seven tests performed on P2 asphalt are superimposed, but far from falling within the plateau region. These data conditions create a situation where the Cross Model can generate erroneous ZSV values.

An ANOVA analysis was done to confirm that mean values of both methods were representative of the same population (Table 3). The ANOVA analysis shows that the differences in the mean values among

Table 3 Test results and one-way analysis of variance (ANOVA)

Asphalt	Test method	N° test	Mean [Pa.s]	SD [Pa.s]	CV [%]	NT	EVT	*P*
C1	Creep	7	140.3	12.59	8.9	Passed ($P = 0.195$)	Passed ($P = 0.480$)	*0.717*
	F. Sweep	7	142.5	9.66	6.7			
C2	Creep	7	226.0	19.04	8.4	Passed ($P = 0.468$)	Passed ($P = 0.130$)	*0.740*
	F. Sweep	7	229.0	11.60	5.1			
C3	Creep	7	290.0	26.92	9.3	Passed ($P = 0.837$)	Passed ($P = 0.232$)	*0.389*
	F. Sweep	7	279.5	15.95	5.7			
M	Creep	7	1463.0	280.75	16.5	Passed ($P = 0.453$)	Passed ($P = 0.353$)	*0.993*
	F. Sweep	7	1461.7	216.94	14.7			
P1	Creep	7	570.2	93.95	19.1	Passed ($P = 0.326$)	Passed ($P = 0.642$)	*0.426*
	F. Sweep	7	610.7	89.81	14.8			
P2	Creep[a]	–	–	–	–	–	–	–
	F. Sweep	7	1485.3	114.37	7.7			

SD Standard deviation, *CV* coefficient of variation, *NT* normality test, *EVT* equal variance test, *P* Probability of being wrong in concluding that there is a true difference between the groups (*P* value > 0.05)

[a] The equipment resolution did not allow obtaining a proper measure

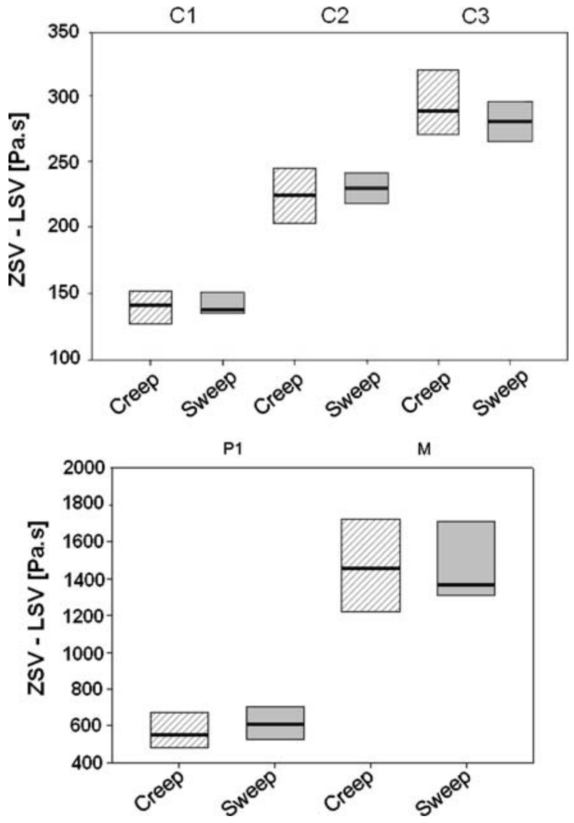


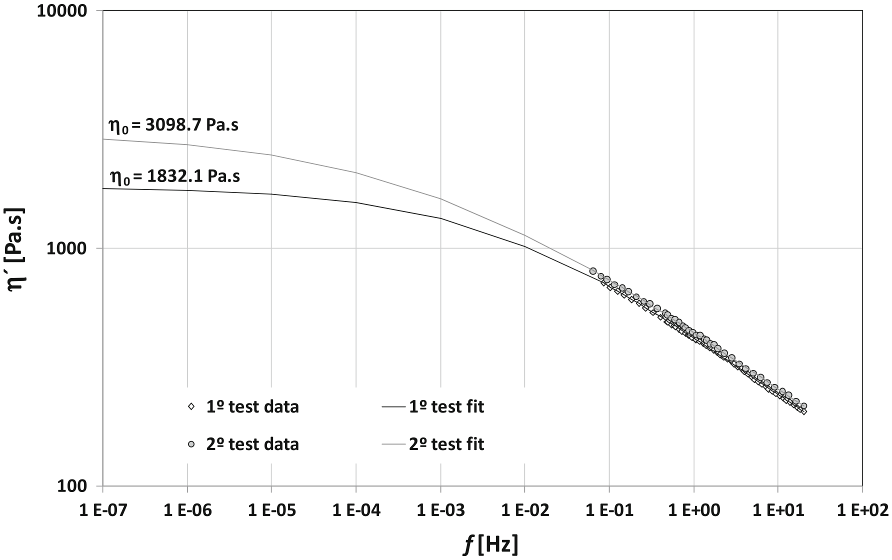
Fig. 6 Graphic comparison of ZSV results by creep and frequency sweep test. Unmodified asphalts (*above*); modified asphalts (*below*)

the treatment groups are not great enough to exclude the possibility that the difference is due to random sampling variability, and are therefore a statistically insignificant (in all cases *P* values are greater than 0.05). As a consequence, frequency sweep and creep test methods give equally reliable results.

In reference to the test protocols, the test time consumption was an important difference between methods. The Frequency sweep test required a maximum of 2 h for sample preparation and testing in all studied cases, whereas the test time for the creep test depended on the studied asphalt type. One hour was required for conventional asphalts, but in modified asphalts (M and P1) 4 h were needed to reach the steady state flow. In the particular case of P2, the steady state was not reached after 8 h, and even worse, the binder's structure was affected.

Nowadays in Europe, the ZSV is studied as a specification parameter to characterize the asphalt contribution to pavement rutting behaviour. As these specification tests are performed on a regular basis, swiftness and repeatability become desirable qualities. In this way, the frequency sweep test is faster and easier to perform and seems like a more practical method than the creep test for specification purposes. However, the results must be studied carefully in the cases of modified asphalts.

Fig. 7 Complex viscosity versus frequency for two samples of P2 asphalt (SBS modified)



5 Conclusions

The benefits of creep and frequency sweep test methods for measuring ZSV in asphalts were analyzed in this work. The main conclusions are indicated as follows.

From an ANOVA statistical analysis it appears that frequency sweep and creep tests give comparable LSV and ZSV results for unmodified (C1, C2, C3), multigrade (M) and EVA polymer modified (P1) asphalts. Both methods have acceptable repeatability of results, comparable to that reported by other authors.

Regarding the effect of binder characteristics on the variability of results, the measurements of M and P1 asphalts show more variability than those of conventional asphalts, which is in accordance with their more complex behaviour.

The frequency sweep test in P2 asphalt (SBS modified polymer) shows good repeatability, similar to unmodified ones. However, due to further examination of the test data, it would not be appropriate to assume that the LSV value obtained in the test is comparable to the asphalt's ZSV.

The frequency sweep test is an easy test method to perform, but its results must be studied carefully in the case of modified asphalts. Regarding the test time consumption, both methods are similar for unmodified asphalts, while duration significantly increases in the creep test for modified asphalts.

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