Creep Flow Behavior of Asphalt Rubber Binders. The Zero-Shear Viscosity Analysis

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ABSTRACT. *The development of the European technical specification aims at characterizing the asphalt binder behavior by means of rheological measurements so as to evaluate performance-related properties of binders. In this context an experimental investigation was carried out at the University of Parma in order to characterize asphalt rubber binders by the Zero-Shear Viscosity (ZSV) analysis. This specific test highlights the binder performance in the operating conditions typical of hot climate, when pavement damages can occur in consequence of viscous flow phenomena.*

The experience reported in this paper focuses on the determination of Zero-Shear Viscosity (ZSV) by creep mode. In order to evaluate the creep mode approach at crumb rubber modified binders, some theoretical and experimental critical aspects are highlighted.

The results obtained pinpoint the different aspects and dynamics linked to the mechanical behavior of crumb rubber modified binders. The results also show how addition of crumb rubber (CRM) to bitumen can produce a viscosity and compliance deviation towards typical polymer-modified bitumen behavior. In accordance with experimental methodology and data analysis, test execution highlights how rheological analyses are applicable to binders showing different behavior, thus allowing mechanical characterization and performance comparison.

KEYWORDS: *Creep Flow, Zero-Shear Viscosity, Bitumen Rutting, Damage Characterization*

1. Introduction

Zero-shear viscosity is a rheological concept introduced for the evaluation of mixing and compaction temperatures of modified binders. A further development of the zero-shear viscosity approach occurred in order to introduce new performance-related properties in the European standardization of a new technical specifications system. At this point in time the rheological approach seems to be unavoidable in order to develop a correct system of classification that may include both traditional and special binders (Stawiarski A. *et al.*, 2004). Rheometry applied to bituminous binders allows the validation of physical and mathematical models that describe the behavior of these materials by quantifying - with an increasing accuracy - their reaction to stress (Christensen *et al.,* 1992, Bahia *et al.,* 1995). On the basis of this concept many European countries have established research programs for the definition and development of a new rheometric test method, able to predict how binders will behave in operating conditions (Stawiarski *et al.,* 2004). Zero-Shear Viscosity is the rheological parameter identified and proposed to evaluate the anti-rutting performance of binders (Sybiliski, 1996).

This paper presents the application of the Zero-Shear Viscosity concept to asphalt rubber binders. In this context the paper aims at analyzing the behavior of bitumen modified by crumb rubber on the basis of the creep test. This test was developed for traditional and polymer-modified binders, but has not been applied to asphalt rubber binders yet. So, the experience reported in this paper is intended for evaluating if the zero-shear viscosity approach is a correct approach for the characterization of crumb rubber modified binders.

Zero-shear viscosity must be independent of the test conditions in order to be considered an intrinsic property of materials. Therefore the experimental program here proposed is based on the quantitative evaluation of time and stress parameter influence on the measurements. The tests conducted can also provide different useful results for the development of the rheometric technique and the assessment of the creep test as a tool for ranking crumb rubber modified binders on the basis of their performance.

2. Test equipment and materials

2.1 *Materials and sample preparation*

Materials selected for the experimental tests are three asphalt binders:

- CRM0: traditional bitumen (pen 76 dmm, T_{RB} = 49.5°C)
- CRM16: traditional bitumen CRM0 + with 16% CRM
- CRM20: traditional bitumen CRM0 + 20% CRM

Binders CRM16 and CRM20 were modified in laboratory by crumb rubber 16% and 20% rubber content referred to the weight of the bitumen.

The crumb rubber used for binder modification is obtained by cryogenic process and the particles' diameter is 0.6 mm (figure 1). Digestion time and methodology are referred to as ASTM D-6114.

Figure 1. *Crumb rubber used for binder modification*

2.2 *Equipment*

The tests were carried out by means of a Dynamic Shear Rheometer (DSR). The temperature during the tests was controlled by means of a Peltier conditioning cell. An air-operated suspension system guaranteed an important reduction in the friction between the moving parts of the rheometer (figure 2). This way allowed a high level of precision of the measurements even for a very low shear rate. The selected measurement system is represented by the double plate configuration with a 25 mm diameter and a 2.0 mm gap. The test temperature was set at 60°C with a maximum admitted deviation of ±0.01°C from the selected temperature during the whole experiment. Before each test the samples were subjected to a thermal conditioning period of 30 minutes.

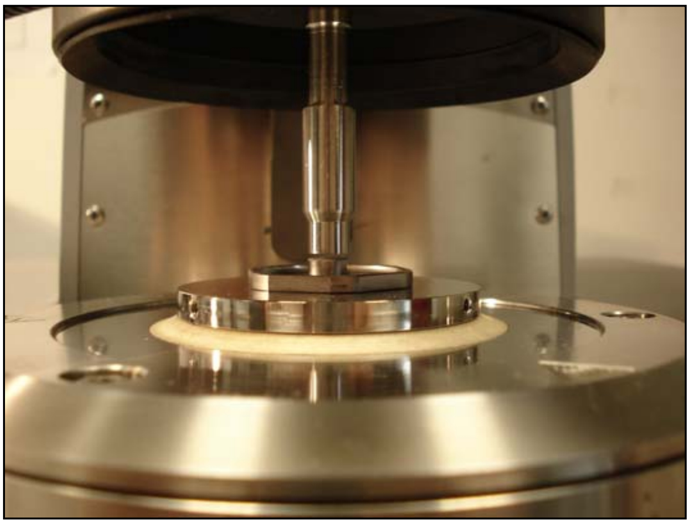

Figure 2. *Dynamic Shear Rheometer*

3. Experimental program

3.1 *Static creep test and zero-shear viscosity (ZSV) concept*

Zero-shear viscosity is the viscosity of a material measured under particular conditions that allow it to become an intrinsic property of the material itself.

As it was found that the SHRP parameter for high service temperature G*/sinδ underestimates the performance of binders with high delayed elasticity (Phillips *et al.,* 1996, Desmazes *et al.,* 2000, De Visscher *et al.,* 2004, Delgadillo *et al.*, 2006, Bahia *et al*. 2001), the concept of zero-shear viscosity (ZSV) was introduced in order to evaluate the contribution of binders to rutting resistance of asphalt pavements.

The literature available describes many test methods for the experimental determination of zero-shear viscosity of asphalt binders based on the use of the dynamic shear rheometer under different experimental conditions, both oscillatory and creep mode (Marasteanu *et al.,* 2004, Binard *et al.,* 2004). The creep mode methodology analyzed in this investigation was selected for CEN standardization. Zero-shear viscosity in creep mode can be obtained by realizing a steady state obtained for very low shear rate. So, the experimental approach here proposed is based on the verification of two different theoretical conditions:

1. Creep time condition: $t \rightarrow \infty$.
   The creep time must be elevated in order to allow the exhaustion of delayed elastic phenomena, thus allowing to reach a steady-state flow ($d\gamma/dt \rightarrow$ cost).

2. Stress condition: $\tau_0 \rightarrow 0$.
   Rheological measurement has to be carried out within the Newtonian region of flow in order to verify that the steady-state reached is independent of shear rate ($d\gamma/dt \rightarrow 0$).

$$\tau = \tau_0 \rightarrow 0, \quad t \rightarrow \infty \quad \Rightarrow \frac{d\gamma}{dt} \rightarrow 0 \ , \qquad (1)$$

In the domain defined by conditions 1 and 2, the viscoelastic behavior of materials tested is well described by Burger's equation. So, when the measurement verifies the conditions presented, zero-shear viscosity can be extrapolated by means of the measurement of creep compliance J(t), according to the theoretical equation of Burger model:

$$J(t) = J_e + J_{de} + J_v = \frac{1}{G_0} + \frac{1}{G_1}\left(1 - e^{-t\frac{G_1}{\eta_1}}\right) + \frac{1}{\eta_0}t \qquad (2)$$

where J_e is the instantaneous elastic compliance, J_{de} is the delayed elastic compliance, and J_v is the viscous compliance. According to the equation above, if the steady state is reached, only the viscous compliance ($J_v = t/\eta_0$) increases and the parameter η_0 of Burger's model can well represent the zero-shear viscosity of material.

$$t \rightarrow \infty \quad \Rightarrow \quad \frac{dJ(t)}{dt} \rightarrow \frac{dJv}{dt} = \frac{1}{\eta_0} \qquad (3)$$

According to the theoretical assumptions of this method, the ZSV of a material can be calculated by fitting experimental data with Burger's model or by the inverse of the average slope assumed by curve J(t) during the steady-state flow, according to the following equation:

$$ZSV = \Delta t / \Delta J \quad [Pa \cdot s] \qquad (3)$$

where Δt represents a time interval expressed in seconds (s) and ΔJ is the difference between the final compliance modulus and the compliance modulus at the start of interval Δt. In equation 3 the compliance modulus is expressed in Pa^{-1}.

Here below we will examine the approach suggested and highlight the theoretical assumptions and some experimental evidence that could be observed during the tests on bitumen modified by crumb rubber.

3.2 *Organization and aim of the experimental investigation*

The viscosity of asphalt binders depends on shear rate when the stress applied is far form the zero-shear conditions (Garrick, 1992). So, zero-shear viscosity must be independent of the test conditions in order to be considered an intrinsic property of materials.

This research aims at evaluating the validity of this assumption with a view to providing useful results for the development of the method and assessing the validity of the creep test as a tool to rank the performance of asphalt rubber.

During the investigation the creep test was repeated several times by varying the parameters that mainly define it, i.e. time (t) and tension (τ).

The range of variation was determined by keeping in mind that the creep time has to be long enough to guarantee that a steady flow state is achieved and that the tension applied will respect the zero-shear condition.

In accordance with the above, the range of parameters was defined (table 1).

Table 1 *Reference parameters for the execution of the creep test*

Type of binder	**Range of stress, τ** [Pa]	**Range of time, t** [hours]	**Temperature, T** [°C]
Traditional Binder	10÷100	1	60
CRM Binder	10÷100	1÷8	60

4. Test results and analysis

4.1 *Creep test and assessment of Zero-Shear Viscosity*

The tests were initially carried out by imposing mean values to the parameters (τ = 50 Pa, t = 1 hour for traditional binder and τ = 50, t = 4 hours for asphalt rubber binder). The influence on the final result of the variation of such parameters was determined on the basis of the results of this first phase.

At this stage, measurements were taken to check the variation, as a function of time, of the most important variables that describe the behavior of materials when it is subjected to actions in steady state: strain $\gamma(t)$, shear rate $d\gamma/dt$, compliance modulus $J(t)$, and viscosity $\eta(t)$.

Different results were obtained in relation to the type of bitumen. For what concerns binder with 16% rubber content (Figure 3) the parameters' trend was similar to what can be typically measured for traditional bitumen (as for bitumen with 0% CRM). In this case one can say that the effect of the rubber is mostly evident in terms of viscosity increase, whereas there is only a slight deviation in the flow behavior.

As for traditional binders, also in this case the steady flow conditions rise, as is proved by the fact that the slope of function J(t) shows a constant value and the curves referring to the three independent measurements overlap fairly well.

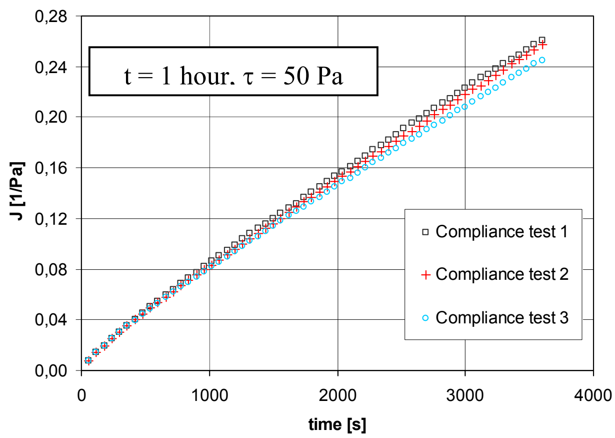

Figure 3. *Results of the creep test on binder with 16% CRM: evolution of compliance as a function of time (three independent measurements)*

Under these conditions, the viscosity measured in the lapse of time considered (i.e. the last 900 seconds of the test) tends to become constant: this value represents the ZSV of the material.

Like other properties of this kind of binders (Antunes *et al.* 2004, Loh *et al.*, 2000, Bahia *et al.* 1994), the situation described above will change if the rubber content rises from 16% to 20%. By observing the evolution in time of the variables recorded, indeed, one can notice a change in the state of the flow (figures 4, 5) and, although the test was continued for up to 4 hours, both viscosity and shear rate do not seem to tend to a constant value (figure 4). The steady-state flow is not reached.

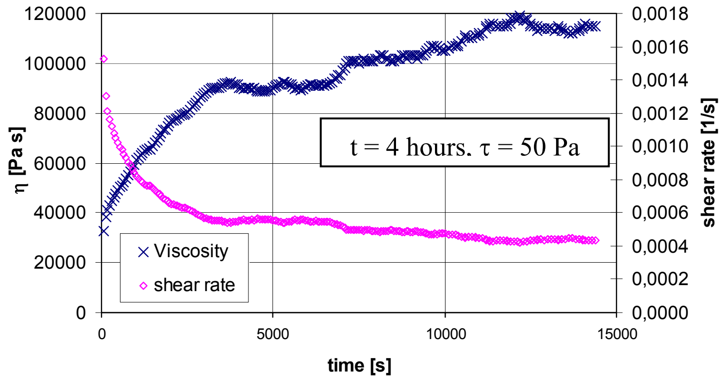

Figure 4. *Results of the creep test on binder with 20% CRM: evolution of viscosity and shear rate as a function of time*

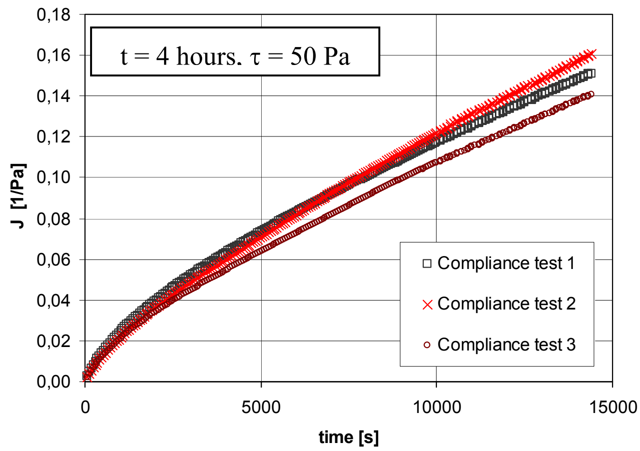

Figure 5. *Results of the creep test on binder with 20% CRM: evolution of compliance as a function of time (three independent measurements)*

The recorded trend, however, which can be ascribed to the very nature of the binder, did produce coherent and comparable results. In fact, the determination of the ZSV is based on the assumption that the first derivative of function J(t) calculated against time is a constant and that J(t) can be consequently approximated, within the calculated lapse of time, to a linear function such as

$$J(t) = at + b \qquad (3)$$

This is what actually happens with bitumen having 20% CRM content too, therefore the following equivalence can be considered valid:

$$\Delta t / \Delta J = 1/(dJ/dt) = \tau/(d\gamma/dt) = \eta_0 \quad (4)$$

In the case of bitumen with remarkably high crumb rubber content (20%) one can see that, during the last 900 seconds, the assumption of linearity for function J(t) can be considered correct, although the viscosity will not really reach a steady value (Montepara *et al.,* 2006).

This is also demonstrated by the limited variation in the results of the three independent measures (table 2).

Table 2. *Results of the tests accomplished (three independent measures) in accordance with the method considered: T = 60°C, τ = 50 Pa*

Binder	***0% CRM***	***16% CRM***	***20% CRM***
ZSV test 1 [Pa s]	205	15517	134168
ZSV test 2 [Pa s]	214	15000	113982
ZSV test 3 [Pa s]	188	16364	132685
average [Pa s]	**202**	**15627**	**126945**
dev. [Pa s]	14	688	11251
dev. [%]	6,7	4,4	8,9

4.2 *Evaluation of the influence of the test parameters on the results*

In order to proceed with the investigation and ascertain the reliability of the results obtained by the creep test with binder at high CRM content (20% CRM), the tests were repeated by changing the essential parameters of time and stress.

4.2.1. *Influence of stress - τ*

Theoretical assumptions impose that rheological measurement has to be carried out within the Newtonian region of flow. In order to verify the validity of this assumption the measurements has been repeated varying the shear stress applied from 10 Pa to 100 Pa. This suggests how crucial it is to select the correct stress in order to obtain reliable results.

What was found for crumb rubber modified, indeed, is that by changing the stress, an increasing variability is recorded, in both the evolution of the values and the final results, by increasing the percentage of modifying agent (figures 6, 7).

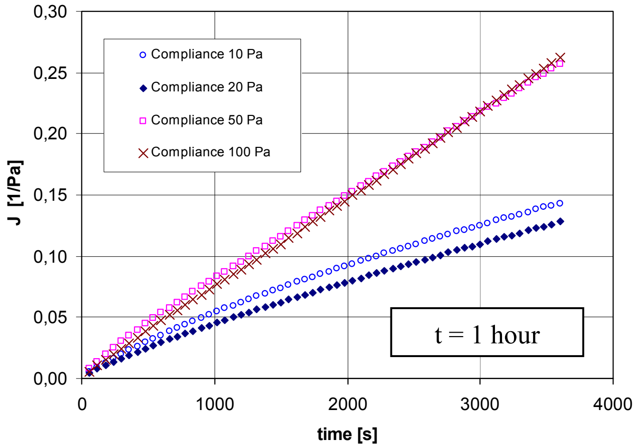

Figure 6. *Evolution of compliance at 60°C for bitumen with 16% CRM content at different stress values*

In the case of bitumen with 16% CRM content the level of stress will influence the test by causing a change in curve J(t) which can be recorded as a variation in both shape and final slope. At the lower levels of stress (10 Pa and 20 Pa) the curves will show a shape similar to that of highly modified bitumen.

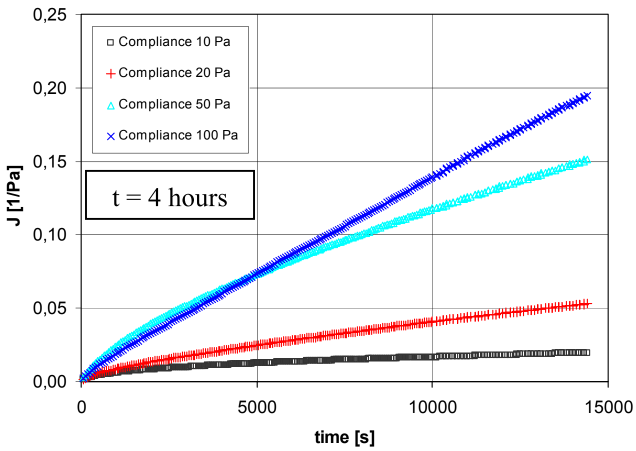

Figure 7. *Evolution of compliance at 60°C for bitumen with 20% CRM content at different stress values*

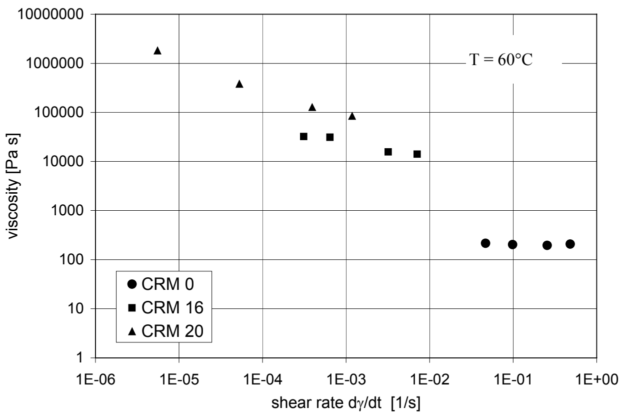

Figure 8. *Influence of stress level on results*

The slope of the asymptotic line is influenced by the entity of the stress in the case of bitumen with 20% CRM content too (figures 7 and 8). Consequently, as shown in table 3, the influence of the stress will be reflected in the final test results.

Table 3 – *Measurements of ZSV according to the different stress levels applied (average of three independent measures)*

Binder	*ZSV at different stress levesl - τ*			
	10 Pa	**20 Pa**	**50 Pa**	**100 Pa**
CRM 0%	214	195	202	207
CRM 16%	32184	31667	15627	14066
CRM 20%	1809160	379010	126945	84583

For bitumen without CRM the variability of the test lies within the range of normal uncertainty which is typical for DSR tests (Santagata*.,* 1996). On the contrary, in the two other cases (asphalt rubber binders) an increase in ZSV is recorded by reducing the stress. That becomes quite relevant when, as for bitumen with 20% CRM content, the test is accomplished at 10 Pa. In this case the recorded

ZSV goes from approx. $1.8 \cdot 10^6$ Pa·s to approx. $1.3 \cdot 10^5$ Pa·s, so becoming bigger by one order of magnitude compared to the value measured at $\tau = 50$ Pa. In the case of 20% CRM it can be observed that there is no linear relationship between shear stress and shear rate; therefore, at this temperature (T = 60 C°), a univocal value of ZSV probably does not exist since the material does not include a Newtonian region in its flow behavior.

4.2.2. *Influence of the creep time – t*

Another difficult issue when taking measurements on binders with high CRM percentage is how to determine the duration of the test. As seen in paragraph 4.2.1, even after 4 hours the shear rate of bitumen with high rubber content may still be on the decrease. In such cases, therefore, ZSV could be underestimated.

In this phase of the investigation, several tests were executed on bitumen with 20% CRM with different creep times, i.e. 1, 2, 4 and 8 hours.

Figure 9 shows that after 8 hours (28800 s) a steady state seems to be partially achieved and the slope of function J(t) seems to tend to a constant value. Consequently, the fitting by Burger's equation can provide a value of parameter η_0 connected to the zero-shear viscosity of materials but not verifying the condition about the stress independency. So, the parameter η_0 determined in this case can represent a *low shear viscosity* value.

In the case of bitumen with 20% CRM concentration, a steady-state flow (which is essential to get measures in line with the theoretical prescriptions) can only be achieved on condition that the stress is applied for a sufficiently long time: the duration of the test, therefore, appears to be, once again, a fundamental parameter for crumb rubber modified binders too.

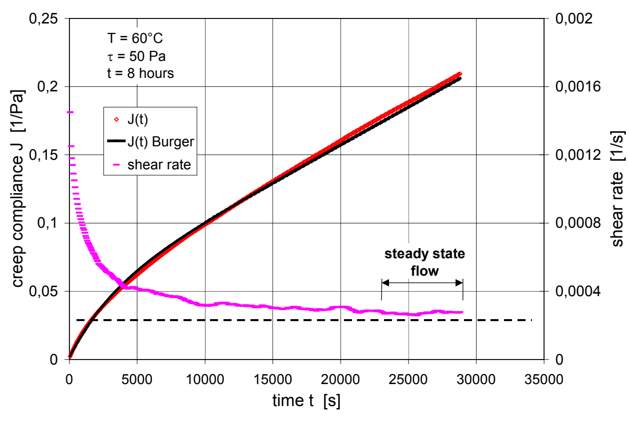

Figure 9. *Curves J(t) and η(t) obtained after 8 hours.*

The comparison shown in figure 10 defines the variability of the result based on the different creep times (τ = 50 Pa).

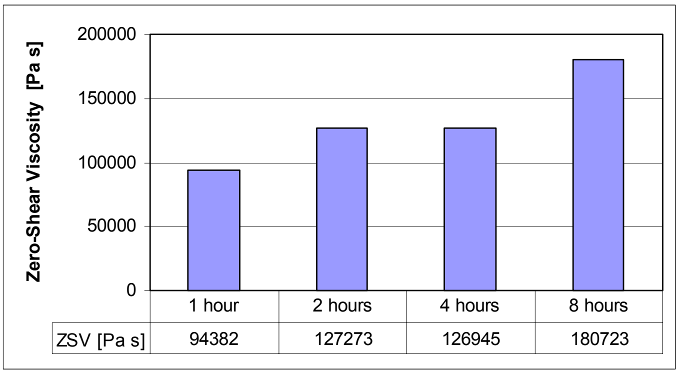

Figure 10. *Influence of the creep time on ZSV @ 60°C for bitumen with 20% CRM concentration (τ =50 Pa).*

5. Conclusions

The findings of the investigations presented in this paper highlight several aspects that relate to the understanding of the mechanical behavior of asphalt rubber binders, and also a few aspects to the development of static creep method which is typical of the rheological analysis applied to road construction. The definition of new technical specifications for asphalt binders at both European and US level is presently the centre of continuous and fruitful discussions and is therefore quite dynamic. So, the original contribution of this article consists in having pointed out, by experimental measures, some special and critical features which are essential for a correct application of the test on asphalt rubber binders.

Actually, through the analysis of the curves and the comparison of the final results, it has been pointed out that the selection of the test parameters may often lead to remarkable variations in the interpretation of the mechanical behavior of material. Similarly to what is observed by PmB, the asphalt rubber binder with 20% CRM concentration will not reach steady flow conditions - at a low shear rate - in a well-definable time. Whilst a traditional binder achieves a steady flow state in one hour, binders with high CRM content will almost always need a longer time, not easy to define. Experimental investigation also shows that the definition of the applied shear stress is fundamental for the reliability of the result, as it has been demonstrated that the high rubber content causes a non-linear relationship between shear stress and shear rate near the zero-shear conditions too. So test results showed that parameter η_0 determined in this case can represent a *low shear viscosity* value but don't represent the zero-shear viscosity of material because it is not an intrinsic property independent by the stress conditions.

On the other hand, although it was conceived for traditional or polymer-modified bitumen only, the rheological approach can be applied with good results to bitumen modified by crumb rubber. In fact it has been demonstrated that the viscous compliance value (J_v) is extremely sensitive to variations in the content of CRM within the binder, and that the flow conditions of the latter vary according to the rubber percentage.

Furthermore, it has been emphasized that the analysis of the curves is able to show the effect caused by the addition of recycled tire rubber on the behavior of binders by shifting it towards the domain of polymer-modified binders. In this sense one can see that the binder with 20% CRM concentration shows a variation compared to the basic bitumen, and that the flow at very low shear rate changes from Newtonian to non-Newtonian, similarly to what is often recorded by binders modified by SBS polymers.

In any case, our investigation clearly shows that the ZSV numeric value creates a distinction between traditional binders and binders modified by CRM rubber. This suggests that the Zero-Shear-Viscosity, despite the necessary approximation described above (particularly in the case of high rubber content), may represent a useful parameter to classify this kind of binders and forecast their behavior under some operating conditions.

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