Quantification of polymer content in binder by modified MSCR test

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ABSTRACT

The Multiple Stress Creep and Recovery Test (MSCRT) is a testing method using the dynamic shear rheometer (DSR) to determine viscoelastic and viscous binder properties in order to assess the influence on the asphalt deformation behavior. The applicability of the test conditions and the corresponding simplified evaluation method formulated in the standards are reviewed in this paper. Here, rheological analyses are performed of the creep recovery curves corresponding to the Burgers model. The results of MSCRT prove that the viscous behavior of the binder is sufficiently described under the defined test conditions. However, the viscoelastic behavior may be determined more precisely at equi-viscose temperature. Further on the modification of the test enables to assess the effectiveness of the polymers contained in the bitumen on the one hand or the polymer content on the other.

Keywords: Creep, Modified Binders, Polymers, Viscosity

1. INTRODUCTION

The MSCR test is a repeated creep recovery test, which was developed in the United States for a better characterization of bitumen. The aim of this new procedure was to predict the influence of bitumen on the deformation resistance of asphalt pavements. Therefore correlations between MSCRT results on bitumen and deformation tests on asphalt were investigated.
The MSCR test is carried out at 50 ° C and at 2 stress levels (0.1 kPa and 3.2 kPa) according to AASHTO [1, 2]. In DSR the bitumen sample is loaded for a period of 1 s and then unloaded for a period of 9 seconds. This combination occurs 10 times for each stress level. Therefore the MSCR test requires 200 s. The determination of the stress levels are carried out so that the sample is loaded within the LVE-region at 0.1 kPa and beyond the LVE-region at 3.2 kPa. Higher stress levels result in higher deformation rates. Here the polymers are stretched and thus become more effective.
Bitumen is a visco-elastic material whose rheological properties are strongly temperature dependent. With increasing temperature, the elastic components decrease while the viscous components increase. Hard unmodified bitumen may exhibit the same rheological properties like a soft one but at a significantly higher temperature (about + 10 K). The knowledge of the temperature at which the rheological properties are the same, are helpful to make decisions about the applicability of bitumen. This procedure is applied for many decades by determination of the Softening Point Ring and Ball E_{RaB} for unmodified bitumen. The modification of bitumen with polymers strongly changes the viscoelastic behavior of the bitumen, especially in the high temperature range. A temperature, at which the exact rheological properties can be adjusted, like with regular unmodified bitumen, cannot be determined. With increasing polymer content the rheological properties are changing. The material properties of the base bitumen overlap with those of the polymer network, so that the properties of the base bitumen are difficult to isolate testing polymer modified bitumen by means of DSR. Retardation tests are suitable in principle in order to address the rheological behavior of bitumen and asphalt. Rheological properties can be mathematically derived by combining Burger's elements spring and damper. This is usually performed in regions of low temperatures [3, 4, 5] for axial tension. At constant tension and stressless loading condition, the model can be described by equation 1.

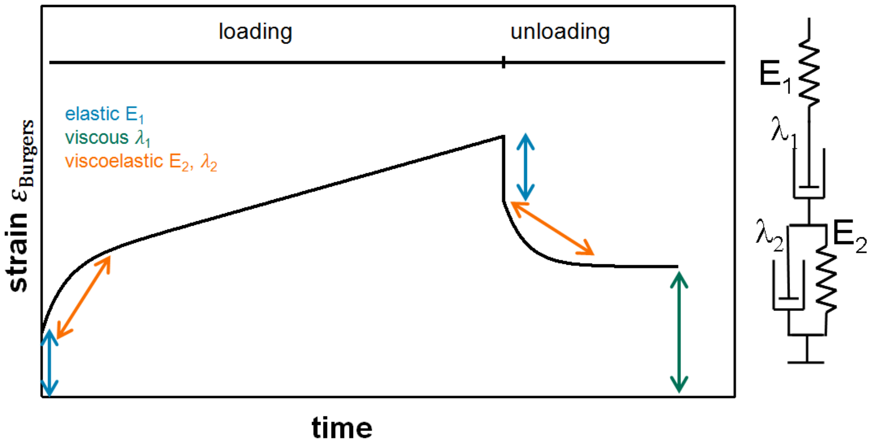

Figure 1. Strain history during retardation test and corresponding rheologial model.

$$\varepsilon_{Burgers}(t) = \frac{\sigma_{const.}}{E_1} + t \cdot \frac{\sigma_{const.}}{\lambda_1} + \frac{\sigma_{const.}}{E_2} \cdot \left(1 - e^{-\frac{E_2}{\lambda_2} \cdot t}\right) \quad \text{(Equation 1)}$$

with
$\varepsilon_{Burgers}(t)$ = time-dependent strain [-],
$\sigma_{const.}$ = constant stess [Pa],
E_1 = modulus of elasticity of Maxwell-Model [Pa],
λ_1 = viscosity of Maxwell-Model [Pa·s],
E_2 = modulus of elasticity of Voigt-Kelvin-Model [Pa],
λ_2 = viscosity of Voigt-Kelvin-Model [Pa·s].

The evaluation of the MSCR test is greatly simplified. The remaining creep and its percentage recovery at the end of the recovery period are determined. For each loading step only the depicted points in Figure 2 per cycle (initial load, end of loading, end of decompression) are included in the calculation of the results. Other data are not considered when evaluating the experimental data.

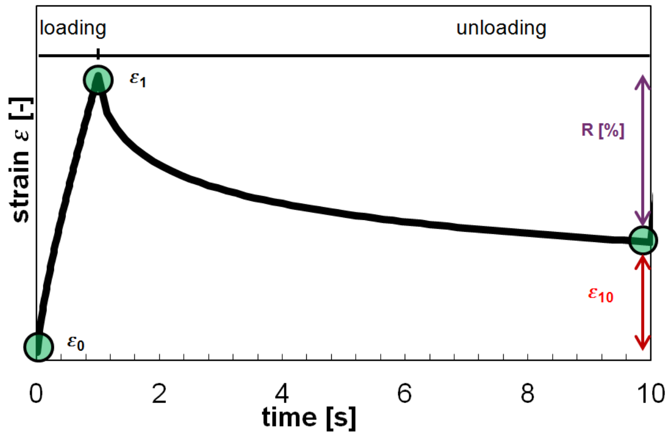

Figure 2. Strain history during MSCR-test and considered data points for evaluation (green).

$$R = \left(\frac{\varepsilon_1 - \varepsilon_{10}}{\varepsilon_1}\right) \cdot 100\ [\%] \text{ (Equation 2)}$$

with
R = recovery [-],
ε_0 = strain at beginning of loading [-],
ε_1 = strain at the end of loading [-],
ε_{10} = strain at the end of recovery [-].

$$J_{nr} = \frac{\varepsilon_{10}}{\sigma}\ [kPa^{-1}] \text{ (Equation 3)}$$

with
J_{nr} = non recoverable creep compliance [kPa^{-1}],
ε_{10} = strain at the end of recovery [-],
σ = applied stress [kPa].

The percentage recovery R describes the viscoelastic; the non recoverable creep compliance J_{nr} the viscous behavior of the binder. However, the results are no rheological characteristics but ratios.
In Germany the MSCRT is applied for a few years. In 2012 a national technical guideline was released to execute the MSCRT [6]. In Germany the test temperature at 60 °C and an additional stress level at 1.6 kPa are defined in contrast to the original form of MSCRT. Currently, nationwide test data are collected in order to derive consolidated findings for the characterization of polymer-modified bitumen and therefore, possibly provide an alternative test for the elastic recovery according to DIN EN 13398.

2. GOALS AND OBJECTIVES

The change of the material behavior of bitumen is evidenced by a decrease in viscoelasticity with increasing temperature, the viscous percentage however increases. It is assumed that the bitumen within polymer modified bitumen assesses the same material behavior as regular bitumen and that characteristic changes are attributed to the effect of the polymers.
The viscoelastic properties of the polymer structure can be assigned when using a test temperature where the viscoelasticity of the bitumen is considerably lower than that of the polymer structure. Higher viscoelasticities may be achieved when the applied polymers exhibit a high efficiency or when increasing the polymer content. Figure 3 (left) shows the first loading and unloading curve of a regular bitumen 50/70 in MSCRT at a temperature of 60 ° C. The bitumen exhibits a very low viscoelastic material behavior. As a result of the unloading process only a very slight reshaping of the sample is detectable. If the bitumen is modified with elastomers its viscoelasticity increases significantly; the resistance to applied loads increases (see Figure 3, right).

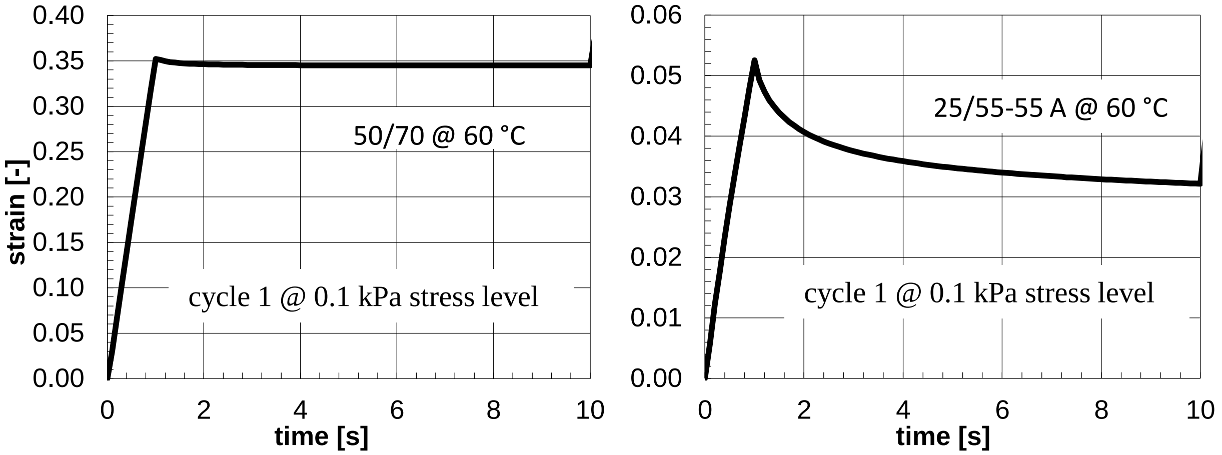

Figure 3: First loading- unloading cycle executing MSCRT at 60 °C

In order to describe the quality of the polymer modification, retardation tests in the dynamic shear rheometer are (DSR) carried out; on the one hand at a constant test temperature of 60 ° C and on the other hand at a test temperature at which the binders exhibit the same viscosity. By adding polymers, the viscosity of the resulting binder is being increased, which results in a higher equi-viscose temperature compared to pure bitumen. The aim of the study is to state the effectiveness of the polymer used as well as to detect the polymer content in the polymer-modified bitumen by the modification of MSCRT.

3. MATERIALS AND TESTS

The investigations are carried out for 7 bitumen; 3 unmodified and 4 polymer modified bitumen. Type 40/100-65 A of the polymer-modified bitumen PMB is highly modified; the level polymer modification of the other PMB is moderate (s. Table 1).

Table 1: used materials

	Level of polymer modification		
	-	**moderate**	**high**
Binder	70/100	45/80-50 A	40/100-65 A
	50/70	25/55-55 A	-
	30/45	10/40-65 A	-

Viscosity measurements are carried out in a temperature range from 20 to 90 °C using DSR operating in rotation mode. Here the specific equi-viscose temperatures are identified. A regular bitumen 50/70 serves as reference at a temperature of 60 °C. The temperature to be determined is the one at which a viscosity of about 300 Pa·s is present. The equi-viscose temperatures are given in Table 2.

Table 2: equi-viscose temperatures of investigated bitumen

Binder	Level of polymer modification	equi-viscose temperature [°C]
70/100	-	57.1
50/70		60.0
30/45		68.3
45/80-50 A	**moderate**	63.1
25/55-55 A		74.1
10/40-65 A		75.9
40/100-65 A	**high**	87.4

Since the measurements are to be executed within the LVE-region, the creep-recovery tests are carried out at a stress level of 0.1 kPa. The LVE-region was reviewed for all binders and temperatures. The loading time of 1 s is maintained. Additional measurements were performed in order to check the viscoelastic reshaping of the binder at an unloading duration of 9 s [6, 7].

It has been proofed that the viscoelastic recovery is finished after 9 s for regular bitumen. Using polymer-modified bitumen, it could be stated that after 9 s the recovery is not completely finished jet but after 21 s or at equi-viscose temperature after 33 s. Since the viscoelastic recovery after 9 s unloading is very low, the results are not affected decisively. Therefore it will be continued to work with this unloading period of 9 s.

The evaluation of the creep-recovery test is carried out in two ways. On the one hand the characteristics J_{nr} and $R_{0,1}$ are deducted as defined by the MSCRT, on the other hand, the regression parameters of the Burgers model (E_1, E_2, λ_1, λ_2)

are determined. In this study, the approach of the Burgers model is applied on the results from the MSCRT despite the presence of shear stress. From the Burgers-parameters the viscoelastic deformation component is determined using equation 4.

$$vel(Burgers) = \frac{\sigma}{E_2} \cdot \left(1 - e^{-\frac{E_2}{\lambda_2} \cdot t)}\right) \text{ (Equation 4)}$$

with
vel (Burgers) = viscoelastic recovery amount of the Voight-Kelvin-Model [-],
E_2 = modulus of elasticity of the Voigt-Kelvin-Model [Pa],
λ_2 = viscosity of the Voigt-Kelvin-Model [Pa·s]
t = loading period [s].

4. RESULTS

The test results at 60 °C are shown in Table 3; the results of the equi-viscose temperatures are given in Table 4.

Table 3: test results at 60 °C

Binder	MSCRT		Burgers		
	$R_{0.1}$ [-]	J_{nr} [kPa^{-1}]	E_2 [Pa]	λ_1 [Pas]	λ_2 [Pas]
50/70	0.441	3.549	119.5	3.0	78.8
25/55-55 A	42.521	0.307	27.5	30.6	50.3
70/100	1.394	4.326	143.0	2.3	119.8
40/100-65 A	98.898	0.007	10.7	160.9	18.0
45/80-50 A	33.143	1.832	8.2	5.2	9.9
30/45	11.380	0.838	69.3	11.8	85.8
10/40-65 A	47.049	0.187	40.9	45.7	64.6

Table 4: results at equi-viscose temperatures

Binder	MSCRT		Burgers		
	$R_{0.1}$ [-]	J_{nr} [kPa^{-1}]	E_2 [Pa]	λ_1 [Pas]	λ_2 [Pas]
50/70	1.464	3.392	126.2	3.0	79.8
25/55-55 A	14.799	3.775	13.8	2.6	14.8
70/100	1.445	3.254	165.1	3.1	105.8
40/100-65 A	78.500	0.963	2.1	6.3	2.5
45/80-50 A	31.579	2.656	6.4	3.4	7.7
30/45	4.003	2.964	69.6	3.4	52.9
10/40-65 A	35.049	1.730	7.8	4.9	13.2

In the following, correlations of MSCRT with rheological parameters are to be considered. A function of temperature is detected from the correlation of the characteristic value $R_{0.1}$, representing the approximate viscoelastic material behavior and the viscoelasticity of the Burgers model (Figure 4). A linear relationship at a constant test temperature of 60 °C cannot be assumed but at equi-viscose temperatures.

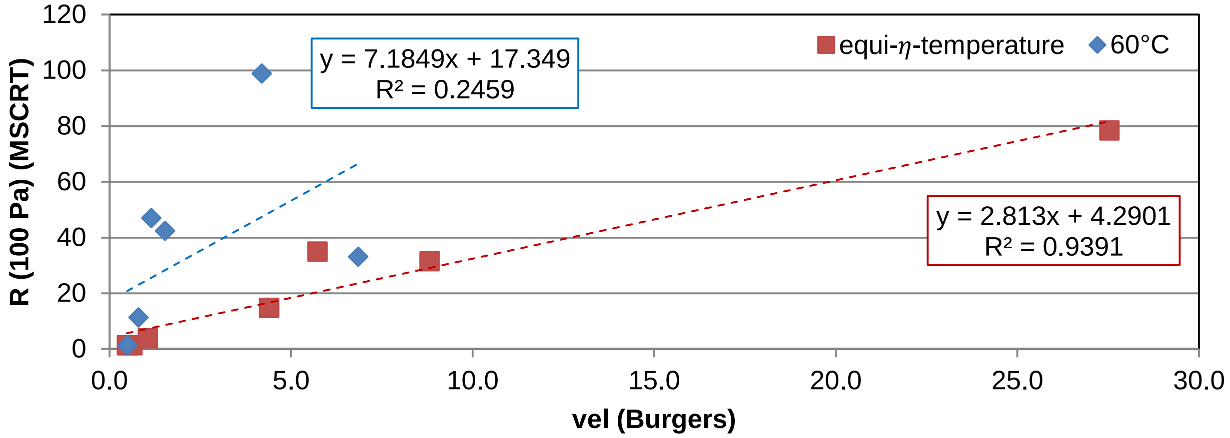

Figure 4: correlation of viscoelastic components R(100 Pa) und vel (Burgers)

With respect to the viscous behavior the characteristic parameter J_{nr} (from MSCRT) bears relation to the viscous component of the Burgers model vel. Figure 5 shows a linear correlation with high coefficient of determination for the measured values at equi-viscosity temperature. At constant test temperature of 60 °C, this dependency can be described by a power function.

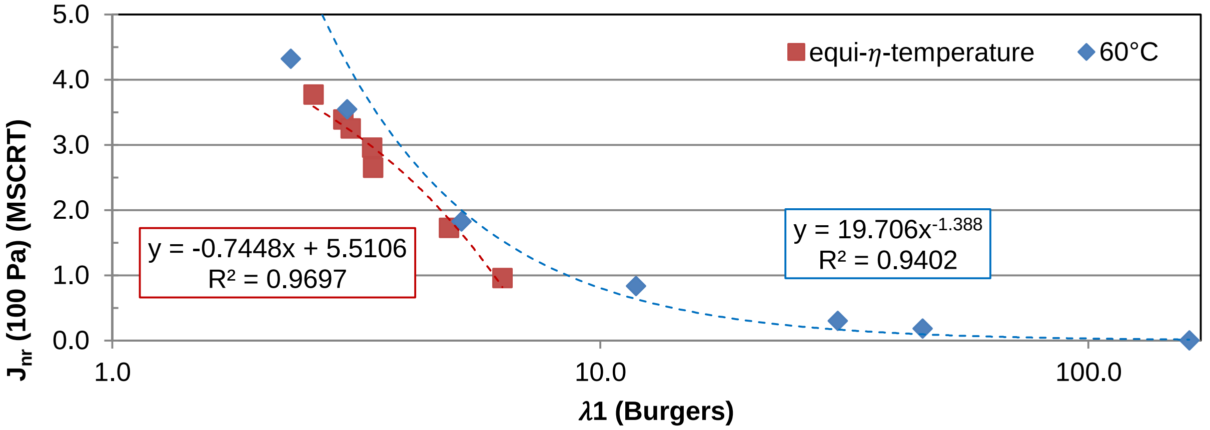

Figure 5: correlation of the viscous parameters J_{nr} (100 Pa) and λ_1

The viscous ratio of bitumen increases with increasing temperature, the viscoelastic ratio of bitumen decreases. The ratio of viscoelasticity using PmB at equi-viscosity temperature is to be attributed to the effect of the contained polymers. The quotient of the viscoelastic ratio (vel) and the viscous ratio (v) is calculated from the obtained data in order to assess the effectiveness of the applied polymer, or to quantify the polymer content in the polymer-modified bitumen. For MSCRT the approximate ratio vel/v can be derived:

$$ratio\ \frac{vel}{v}\ (MSCRT) = \frac{R_{(100\ Pa)}}{J_{nr}(100\ Pa)\cdot\sigma}[-] \quad \text{(Equation 5)}$$

Figure 6 reveals the ratio vel/v (MSCRT) as a function of the level of polymer modification.

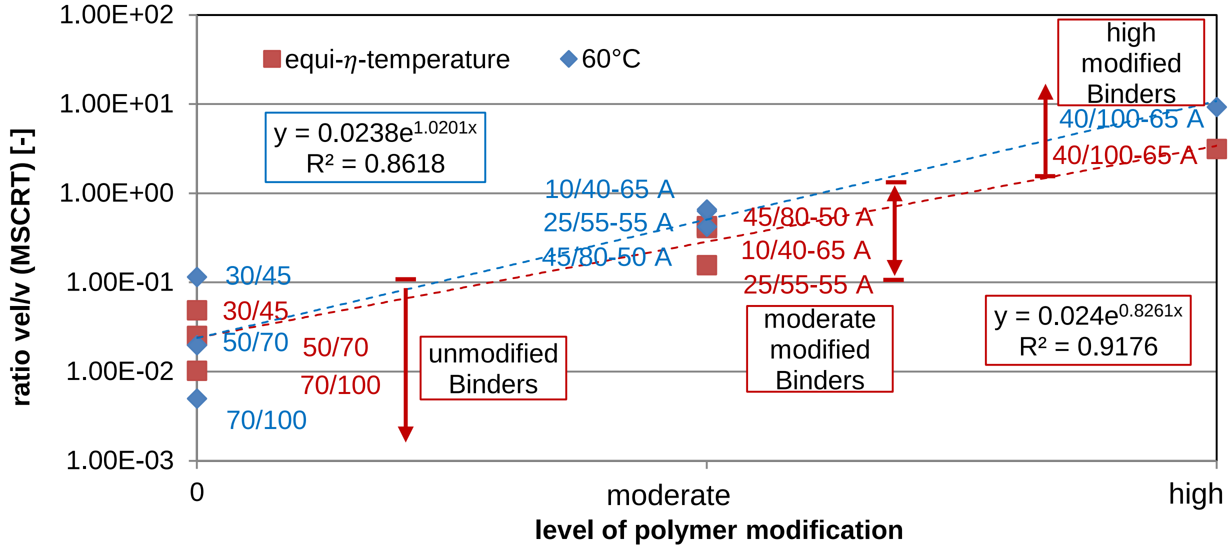

Figure 6: Ratio vel/v (MSCRT) as a function of polymer modification level

The quotient vel/v increases with increasing polymer content. This can be observed both at constant test temperature of 60 °C as well as at equi-viscosity temperature. At 60 °C this behavior is more pronounced.
Analyzing the rheological creep recovery curve according to Burgers, the ratio vel/v is defined:

$$ratio\ \frac{vel}{v}\ (Burgers) = \frac{\frac{\sigma_{const.}}{E_2}\cdot\left(1-e^{-\frac{E_2}{\lambda_2}\cdot t}\right)}{t\cdot\frac{\sigma_{const.}}{\lambda_1}}\ [-] \quad \text{(Equation 6)}$$

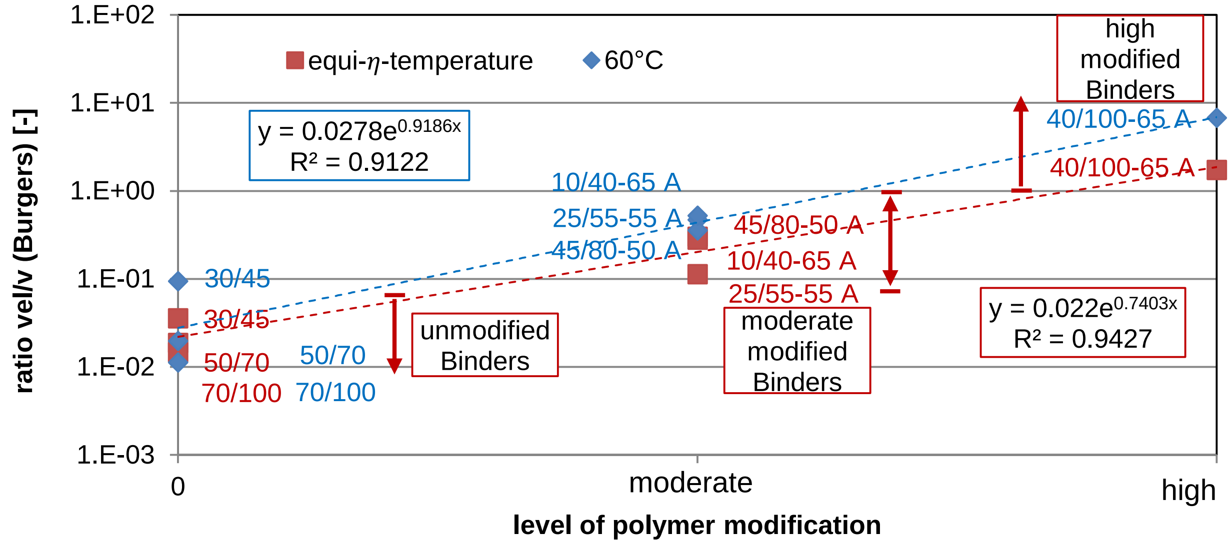

Figure 7: Ratio vel/v according to Burgers in dependence on polymer modification level

A rheological evaluation according to the Burgers model also causes a bracing of the ratio of viscoelastic and viscous deformation depending on the level of polymer modification (Figure 7). For the rheological evaluation the determined coefficients of determination are comparatively higher than the specified parameters by approximation due to MSCRT-evaluation. A particularly clear differentiation in the results can be carried out using equi-viscosity temperatures.

5. CONCLUSIONS

The MSCR test is a repeated creep recovery test in order to approximately predict the viscoelastic and viscous properties of bitumen. Although the overall deformation history is recorded during the test, the evaluation is currently limited on merely 3 data points. The evaluation of the entire deformation curve based on the Burgers model promises a higher benefit. Executing this procedure rheological parameters can be determined as a result, opposing the conventional evaluation. The description of the viscous deformation by MSCRT nearly equals those of the Burgers model. Nevertheless the viscoelastic deformation component cannot be described adequately for all binders using the MSCRT evaluation at a constant test temperature.

The test at equi-viscosity temperature offers significant advantages. When the creep-recovery test is carried out at equi-viscosity temperature at a stress level of 0.1 kPa, the ratio of visco-elastic to viscous strain component may be used to determine the impact of the applied polymers. If the type of modification is known, the results can be calibrated and the degree of polymer modification can be determined exactly.

6. REFERENCES

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