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RHEOLOGICAL PROPERTIES OF MASTIC IN RELATION TO THE ACTIVITY OF THE FILLER COMPONENT – A NEW CLASSIFICATION SYSTEM

REOLOGICKÉ VLASTNOSTI MASTIXU VE VZTAHU K AKTIVITĚ FILEROVÉ SLOŽKY – NOVÝ KLASIFIKAČNÍ SYSTÉM

ANNOTATION OF PH.D. THESIS
POJEDNÁNÍ K DISERTAČNÍ PRÁCI

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Abstract

The combination of bitumen and fine aggregate (filler) called "mastic" is a composite binder of asphalt mixtures. This paper proposes new general filler parameters that reflect the activity of the filler in mastics. The newly developed descriptive classification is based on the determination of different types of critical fractional volumes, which are an authentic reflection of the intrinsic properties of the filler. The fractional volumes have been derived from the projection of the rheological properties of each point of the concentration series of the mastic. This approach makes it possible to assess the nature and intensity of the direct interaction of the filler with a bituminous binder using some well-known procedures and test methods. A dynamic shear rheometer was used to investigate mastic materials over a wide concentration range in two service temperature ranges and two test configurations. The Multiple Stress Creep and Recovery test covers the medium temperature range; the high temperature range of the mastic is investigated by rotational viscosity. The paper clarifies the nature of the proposed parameters and their relationship to the mastic properties and the filler classification system. The methodology can be used to solve practical problems related to the rheological behaviour of mastics or asphalt mixes.

Keywords: Mastic, mineral filler, critical concentration volume, MSCRT, viscosity

Abstrakt

Kombinace asfaltu a frakce jemného kameniva (fileru) označovaná jako „mastix“ představuje kompozitní tmel reálných asfaltových směsí. Toto pojednání navrhuje nový systém obecných materiálových parametrů fileru, které odrážejí jeho aktivitu v mastixu. Nová deskriptivní klasifikace minerálních filerů je založena na stanovení různých typů kritických frakčních objemů, jež jsou autentickým odrazem vnitřní kvality plniva. Frakční objemy byly odvozeny ze souhrnné projekce reologických vlastností jednotlivých bodů koncentrační řady mastixů. Tento přístup využívá některé známé postupy a zkušební metody, které umožňují posoudit typ a intenzitu přímé interakce plniva s asfaltovým pojivem. Ke studiu mastixových materiálů v širokém rozpětí koncentrací fileru byl použit dynamický smykový reometr ve dvou aplikačních teplotních oblastech s odpovídajícími testovacími konfiguracemi. Zkouška MSCR (zkouška opakovaného napěťového namáhání a relaxace) pokrývá střední aplikační teploty mastixových materiálů (vozovka); oblast vysokých teplot (výroba a pokládka) je zkoumána pomocí měření rotační viskozity. Předkládané pojednání objasňuje podstatu navrhovaných parametrů a jejich vztah k vlastnostem mastixů a systému klasifikace filerů. Tuto metodologii lze použít při řešení praktických problémů spojených s reologickým chováním mastixových tmelů, potažmo asfaltových směsí.

Klíčová slova: Mastix, minerální filer, kritická objemová koncentrace, MSCRT, viskozita

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1. INTRODUCTION

Bituminous mastic consists of a bituminous binder and an aggregate fraction called "filler". Functional testing of mastic is one of the development trends in asphalt pavement research. This strategy offers a completely new perspective for assessing the performance of asphalt mixes. It allows a comprehensive analysis of the characteristic properties of the actual cohesive element, rather than focusing only on its components. The resulting rheological properties of the mastic are determined not only by the chosen bituminous binder, but also by the nature of the filler component and its concentration.

Filler activity is mainly related to factors such as particle size distribution, specific surface area values, petrographic origin, affinity of binder components to individual rock-forming minerals, particle shape and texture. One of the key factors appears to be the overall concentration of the mineral component, which determines the thickness of the bituminous film surrounding individual filler grains. The resulting complex interaction in mastic is thus a direct reflection of the intrinsic filler properties mentioned above, the filler concentration and the overall affinity of the bituminous binder to the filler particles. There are at least two types of filler in real asphalt mixes – imported alkaline filler (represented mainly by finely ground limestone flour/powder) and filler derived from the parent rock (baghouse filler). In some cases, source rock produces filler particles with very problematic effects on the rheology and other properties of asphalt mixes. A better understanding of the effects of specific fillers on bituminous binders would be of great importance, particularly with regard to the availability of mineral fractions as raw materials.

2. THEORETICAL BACKGROUND

The basic theoretical principles required to understand the rheological behaviour of mastics are based on the concept of fractional volumes. These are procedures known from the rheology of suspensions. In this concept, the actual control parameter of the resulting quality is not just a simple volume fraction of the filler in the mastic, but the ratio of the fractional content of the filler to the critical/limiting volume fraction of the filler (this ratio is derived by extrapolating the values measured over the whole concentration range). A very efficient way of evaluating fillers is to determine the Rigden voids (RV) – the voids in a dry compacted filler – according to EN 1097-4. The essence of the test is based on compacting a dried filler sample in a defined manner and determining the voids within the sample volume. The ability to achieve limit compaction proves to be a unique and recurring material property associated with the intrinsic properties of fillers. This filler property is related to the degree of stiffening of the mastic mortar. The value of RV also correlates with other measured quantities of the mastic, such as the softening point of the mastic or its penetration at 25°C. It correlates less with the viscosity and significantly less with the stiffness of the mastic (Grabowski, & Wilanowicz, 2008).

The property proportional to the Rigden voids can be expressed by a parameter called the maximum packing volume of the filler fraction Φ_{max} (when related to the amount of filler). This quantity, or rather its ratio to the volume of filler used, appears in various rheological descriptions of the behaviour of suspensions as a fundamental control parameter of the viscosity value. The most used rheological model of suspensions today is the Krieger-Dougherty equation (Krieger, & Dougherty, 1959).

$$\frac{\eta}{\eta_{med}} = \left(1 - \frac{\emptyset}{\emptyset_{max}}\right)^{-[\eta]\cdot\emptyset_{max}} \qquad \text{R-1}$$

η.............. Vicosity of the suspension
η_{med}.......... Viscosity of the medium
$\emptyset$............. Volume fraction of solids in the suspension
$\emptyset_{max}$......... Maximum volume fraction of solids in the suspension
$[\eta]$........... Intrinsic viscosity of the medium (2.5 for spheres)

The discovery of such a material characteristic of the filler led P. J. Rigden to create the "fractional voids concept", now referred to as the "free bitumen concept" (Rigden, 1947). When the filler occupies a fixed, non-compactible structure ("Rigden fraction/volume"), a certain amount of bitumen is required to fill the free voids in such a compacted system ("fixed bitumen"). Further addition of bitumen after the voids have been completely filled is called "free bitumen". The thickness of the bitumen film begins to increase as the particles are pushed further apart. This proportion of additional binder will affect the resulting properties of the system (Heukelom, 1965) towards lower stiffness and lower viscosity.

The discovery of such a material characteristic of the filler led P. J. Rigden to create the „fractional voids concept", now referred to as the „free bitumen concept" (Rigden, 1947). If the filler occupies a fixed, no longer compactible structure ("Rigden fraction/volume"), it is necessary to add a certain amount of bitumen to fill the free cavities in such a compacted system („fixed bitumen"). Further addition of bitumen after the voids have been entirely filled is called „free bitumen". The thickness of the bituminous film begins to increase because the particles are pushed further apart. This portion of additional binder will affect the resulting properties of the system (Heukelom, 1965) towards lower stiffness and lower viscosity. Having evaluated different types of rheological models to describe the composite mastic system, Faheem and Bahia came up with a more detailed "conceptual model" (Faheem, & Bahia, 2009; Faheem, & Bahia, 2010), which introduces the term "Influenced Bitumen Volume" (replacing Rigden's "fixed bitumen"). Rigden' approach was based on the volume of air voids in the dry compacted filler and on decreasing stiffness when exceeding a corresponding limit of binder volume in the mastic. The authors of the „conceptual model" followed up on Tunnicliff's idea of surface-interacting binder (Tunnicliff, 1962)., but realized its importance for high volumetric filler concentrations where most of the bituminous binder is bound in a bitumen film on the surface of the filler particles.

Faheem and Bahia introduced two concentration zones – concentrated and diluted. Only a minor part of the binder in the diluted zone is affected by interfacial interactions with the filler. Therefore, the stiffening effect is determined by the volumetric concentration of the filler, the Rigden voids and the nominal particle size of the filler (Faheem, & Bahia, 2009; Faheem, & Bahia, 2010). In the concentrated zone, the stiffening depends on the ratio of this part of the "surface-consumed" bituminous binder to the total volume of the bituminous film – "Interacting volume of bitumen" (Faheem, & Bahia, 2009; Faheem, & Bahia, 2010). The resistance of the mastic to the applied stress is controlled by its weakest part, i.e., the volume fraction of the bituminous binder, which is not adsorbed or chemically bound on the surface of the filler particles. As the volume concentration of the filler begins to increase, more and more of the intact binder is consumed. The transition from the diluted to the concentrated zone can be examined from the dependence of a suitable rheological quantity on the filler volume concentration. At this critical point, the measured value begins to deviate from the trend valid for the diluted zone.

The conceptual model includes the following considerations. At a certain volume concentration limit, the entire volume of the binder is already in the "affected state" resulting from the surface properties of the solid filler component. The bituminous film surrounding the filler particles is then formed only by the affected binder. Any further increase in the filler content will lead directly to a reduction in the thickness of the bituminous film and thus to a greater influence of the surface interaction. By projecting the trend, the so-called critical filler concentration (Figure 1) can be estimated. This concentration point is constructed as the intersection of the trend lines in the diluted zone and the trend of material property change in the concentrated filler area, Figure 1 (left), Cross B. The critical filler concentration can be interpreted in several ways.

3. OBJECTIVES

It is known that some fractions of fine aggregate from certain sources can induce unexpected rheological behaviour in asphalt mixes. The critical component of these fractions can be considered as fine particles (filler) below 0,063 mm in size. When studying the activity and interaction of the fine particles in the asphalt binder, the rheology of the mastic must be addressed. The need to differentiate mastics containing filler materials from different mining areas was the stimulus to find a simple and reliable method with sufficient descriptive power to capture the variability of the filler properties.

4. MATERIALS AND METHODS

The test set consisted of 12 petrographically different fillers (the majority of which contain mica) or their combinations. The sample set was supplemented with mica standards represented by materials with high mica content or containing highly reactive mica fillers. The filler samples were prepared by grinding a 0/4 fraction of the aggregate in a vibratory grinding mill. This was followed by forced sieving under constant material loading (using a flat brush with long strokes). Only the 0,000–0,063 mm fraction was used to prepare the mastic samples. The laboratory preparation of the mastic was carried out with the binder Azalt 70/100 (total) in accordance with the procedures prescribed by the EN 13179-1 standard (delta ring and ball test). Three of the mastics contain the 50/70 binder from the Litvínov refinery. The basic sequence of filler concentrations included the points 6,25–12,5–25,0–37,5–50,0% by volume. For low activity fillers it is necessary to add the concentration point of 62,5 %.vol. The fractional parameters for the compared materials can be calculated from two concentration zones that are clearly separated on the concentration curve of the rheological variable (region A and B). Each concentration region defines a different constitutive state of the filler. A Malvern Kinexus Pro+ Dynamic Shear Rheometer (DSR) was used to measure the rheological properties of the mastic. The materials were tested using two different types of instrument configuration.

The Multiple Stress Creep and Recovery Test (MSCRT) was performed in accordance with EN 16659. Three loading sequences were applied at shear stress levels of 0.1kPa, 3.2kPa and 6.4kPa and a temperature of 60 °C. The resulting values of the deformation after the cumulative pulsed creep loading reflect the resistance to deformation of the mastic at medium temperatures.

The viscosity at the temperature of 135 °C was determined according to the specification in Annex B of the ČSN 65 7222-3 standard (ČSN 65 7222-3, 2018). The temperature ramp with a gradient of 0.6 °C/min and 25 mm plate-to-plate geometry at shear rate D = 2 s^{-1} was used. As the target value of 135°C was always approached from the higher temperatures of the top of the temperature ramp, the sample was considered to be flow consolidated. As the conditions for uniform shear rate throughout the cross section of the geometry are not met, the values obtained should be considered only as the apparent viscosity determined under the conditions tested.

When measuring the rotational viscosity of suspensions, the unfulfilled condition of constant laminar flow must always be taken into account. The influence of particle morphology and filler content is obvious. At higher concentrations there is also a risk of shear failure of the sample due to lack of cohesion. Nevertheless, both methods have sufficient accuracy and repeatability to detect differences between specific mastics. A definite advantage is that the tests can be run consecutively without the need to re-prepare the sample. Unless otherwise stated, the gap was set at 1.5 mm in both test configurations.

A critical factor in the repeatability of the test appears to be the placement of the sample in the rheometer. The mastic must be of a sufficiently high stiffness and must maintain a semi-solid consistency during the compression process. The sample must not melt completely as this is likely to lead to segregation and a reduction in the observed viscosity. The rheological results should be expressed as the ratio between the values of the measured rheological property of the mastic and the value of the same property of the original binder. However, the results of the MSCRT test were presented as strain for ε to better represent the nominal deformation at different stress levels. The relative dimensionless unit allows a better comparison of the results. ($\varepsilon = \Theta \cdot r/h$; Θ – deflection angle in rad, r – radius of geometry in mm, h – specimen height in mm).

5. RESULTS AND DISCUSSION

5.1. Concept of proposed methodology

This thesis presents a concept using intrinsic descriptive material constants to evaluate the interaction of an asphalt binder with a mineral filler. The methodology is intended to provide an unambiguous description of the materials, allowing to differentiate between different types of filler and to identify the phenomena that determine the resulting rheological behaviour of the mastic. The proposed approach is based on deriving the critical and rectified fractional parameters of the solid phase in mastics. These parameters are calculated from the mastic concentration curve. Thus, the evaluation of the mastic (different fillers in a constant binder) is related to the rheological behaviour throughout the concentration series. The rheological curve is divided into two constitutive parts - region A of high filter dilution (mutually isolated particles), in which increasing the filter concentration changes the value of the rheological variable only slightly. The second part of the curve after the parametric break - region B of low filler dilution (interacting solid phase particles), in which small filler additions substantially change the value of the rheological variable. Each part of the curve is described by its own fractional quantities. The concept is consistent with the "conceptual theory" (Faheem, & Bahia, 2009; 2010).

The use of a DSR (Dynamic Shear Rheometer) instrument for the determination of rheological parameters of concentration series of mastics seems to be a very suitable and promising approach. The variability of the instrument makes it possible to measure the properties in two temperature ranges corresponding to the production and paving of hot mix asphalt and to the conditions prevailing on the pavement during its service life. Two test protocols have been proposed, representing viscosity screening (high temperatures) and dynamic creep screening (medium service temperatures).

This summary only outlines the basic principles and relationships of the new descriptive system for evaluating filler activity in mastic. More fundamental analyses of the relationships and the use of fractional parameters to classify and analyse the interactions between the filler an d the asphalt binder are discussed in detail in Chapter E.8 of the full thesis. An overall general summary of the basic points of the proposed system, its contribution and the genesis of the concept is given in the final chapter of the thesis (Chapter E.10).

5.1.1. Description of mastic behaviour in high-temperature range

This temperature range is clearly related to production, but especially to the process of compacting hot mix asphalt (HMA). Greater insight into the exact rheology of mastic mortar, preferably at high concentrations, is highly desirable not only for mastic asphalt producers but also for solving problems associated with other technological processes. Intuitively, viscosity measurement is inherently best at capturing changes in the internal structure and general rheological classification of the material. Therefore, the parametric "edge" (abrupt change in mastic behaviour with concentration) is well expressed in the concentration-viscosity record, see Figure 1). Region A on this curve represents an area of low concentration where the isolated particles act more or less independently on the bituminous binder.

Region B is the area of high concentrations where the probability of contact between individual grains surrounded by the bituminous film gradually increases. However, the test results must be converted into material constants. These material constants (fractional parameters) should provide unambiguous characteristics of a given filler. Ideally, they should also quantify the influence of the dominant interaction in both regions. Such conversion can be achieved by appropriate scaling of the results. By using the correct type of axis scaling for the selected variables, it is possible to draw/calculate a regression line. This line represents a change in values in the region. This straight line will be controlled by the concentration of the filler and by the dominant type of interaction of the fine particles in the mastic. The descriptive material parameter of these interactions is the value of the critical volume fraction (filler concentration), which can be constructed as the intersection of the extrapolated linear dependence with the x-axis (Fig. 1). Reciprocal scaling ($1/Y = f(X)$, $Y = \eta_{rel}$, $X = c_{\%.vol}$) for region A and linear scaling ($Y = f(X)$, $Y = \eta_{rel}$, $X = c_{\%.vol}$) for region B were chosen to screen the viscosity of the mastic as a function of filler concentration. This setting converts the results into linear dependencies for the concentration ranges analysed. Reciprocal scaling, which provides a linear dependence for region A (a low concentration), acts as a magnifying glass and allows the subtle processes of region A to be visualised in detail.

Table 1. Viscosity screening – description of the designed critical fraction parameters.

Designation	Description	Details
FrX_A	Intersection of A_1/η_{rel} line and X-axis	Critical fractional volume of the mineral phase derived from region A in reciprocal scaling
FrX_B	Intersection of B_η_{rel} line and X-axis	Critical fractional volume of the mineral phase derived from region B in linear scaling
$SFrX_A$	$SFrX_A = FrX_A/S \cdot C$ *)	FrX_A rectified by the slope quotient S derived from the regression line equation intercepting the X-axis
$SFrX_B$	$SFrX_B = FrX_B * S \cdot C$ *)	FrX_A rectified by the slope quotient S derived from the regression line equation intercepting the X-axis
FrX_BS	$FrX_BS = S/FrX_B$	Rectified slope quotient S divided by FrX_B
Cross A	Intersection of linear branches A∩B_1/η_{rel} with weight in region A	Critical fractional volume constructed from both regional branches on reciprocal scale with weight in region A
Cross B	Intersection of linear branches A∩B_η_{rel} with weight in region B	Critical fractional volume constructed from both regional branches on linear scale with weight in region B

*) $Yvis_{rel} = S*X_{konc} + P$, *C – constant, mathematically adjusted to values expressed in positive integers*

By intersecting the lines at the chosen scaling, different types of fractional volumes (concentrations by volume) can be obtained. These critical fractional volumes can be further corrected for the slope value of the regression line, i.e., the angle that the line makes with the x-axis and which expresses the rate of change of the parameter as a function of increasing concentration. The resulting critical fractional volumes are shown in Table 1 and graphically represented by three real fillers in Figure 2.

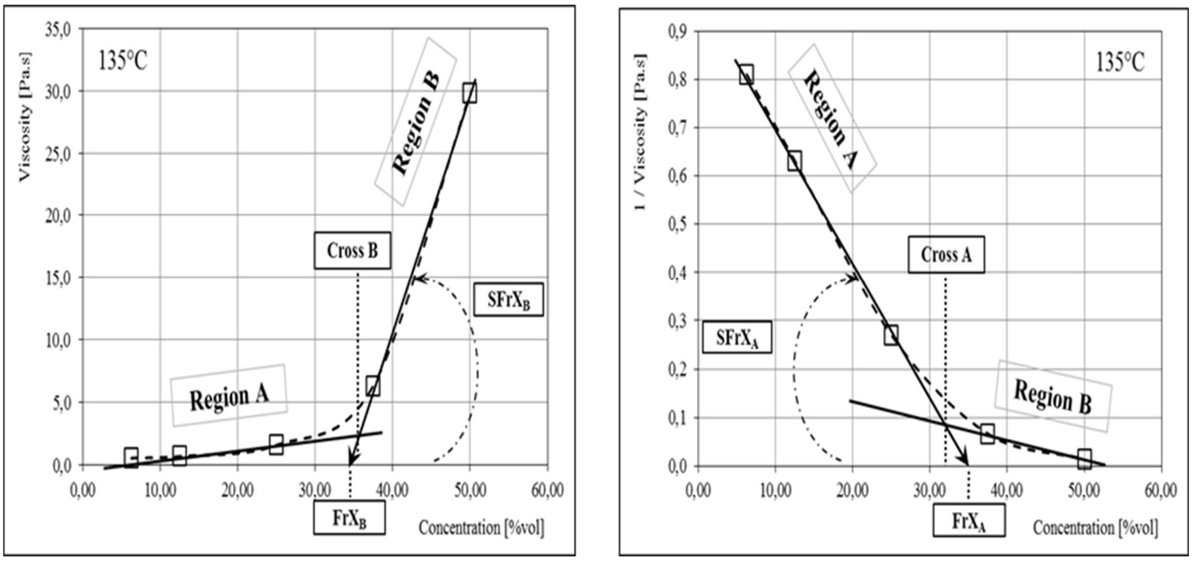

Figure 01. Viscosity screening of mastic at 135 °C – results scaling of concentration series.

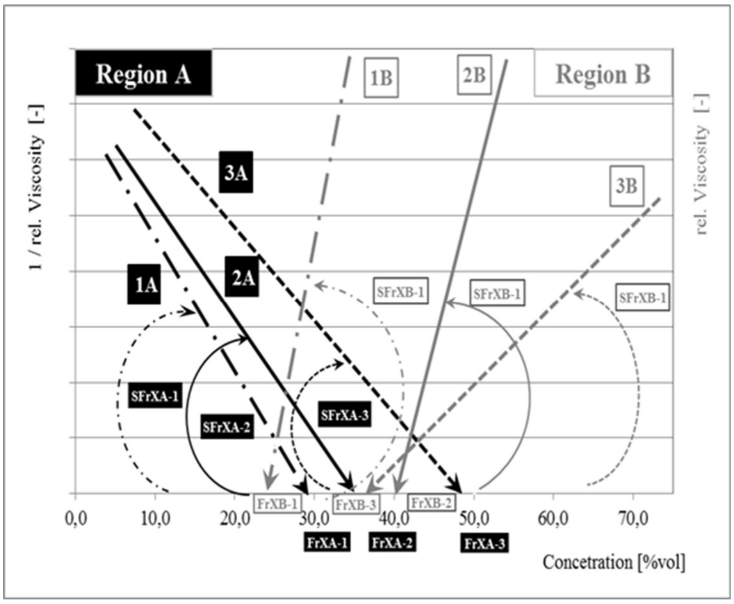

Figure 02. Viscosity screening of mastic – critical fractional parameters of three real fillers based on 70/100 bituminous binder.

5.1.2. Description of mastics behaviour in medium-temperature range

The determination of the viscoelastic properties of the mastic could be considered as an excellent way of describing the rheological behaviour of mastic mortar at medium application temperatures. However, the use of the oscillatory mode (stiffness shear modulus) does not adequately express the parametric "edge" of the viscosity screening. The individual measured stiffness values in the concentration series of a given type of mastic often produce very flat convex curves with a high polynomial coefficient of determination ($R^2 \approx 1$), indicating a lower sensitivity of the method. The transition region, where the mechanism of interaction changes, is wide and very flat. As a result, the resulting curve is completely unsuitable for calculating critical fractional volumes, especially for low stiffening fillers, Fig. 3.

Table 2. Creep screening of mastics - description of the designed critical fraction parameters.

Designation	Description	Details
R-Y	Intersection of B_1/ε line and X-axis; 1/ε → 0	Critical fractional volume of the mineral phase derived from region B in reciprocal scaling of the Y-axis
SR-Y	SR-Y = R-Y/S·C *)	Critical fractional volume R-Y rectified by the slope quotient S_A of the straight line through the X-axis
IG-Y	Intersection of B_log ε line with parallel line ε = –0,079 and X-axis **)	Critical fractional volume of the mineral phase derived from region B in logarithmic scaling of the Y-axis
SIG-Y	SIG-Y = IG-Y/S·C*)	Critical fractional volume IG-Y rectified by the slope quotient S from the straight line intercepting the parallel line to the X-axis at Y = – 0,079**)
I-X	Intersection of A_ε line and X-axis	Critical fractional volume of the mineral phase derived from region A in linear scaling the of Y-axis (including the first three points)
SI-X	SIX = I-X/S·C*)	Critical fractional volume I-X rectified by the slope quotient S from the straight line intercepting the X-axis**)
IG-X	Intersection of A_ε line and X-axis in logarithmic scaling	Critical fractional volume of the mineral phase from region A in logarithmic scaling of the X-axis (including the first three points)
SIG-X	SIG-X = IG-X/S·C*)	Critical fractional volume IG-X rectified by the slope quotient S from the straight line intercepting the X-axis**)

*) *$Y_{\varepsilon} = S \cdot X_{konc} + P$, C – constant, mathematically adjusted to a positive integer.*

**) *The value corresponds to an angular deformation of 0.1 rad with a gap of 1.5 mm and geometry of 25 mm.*

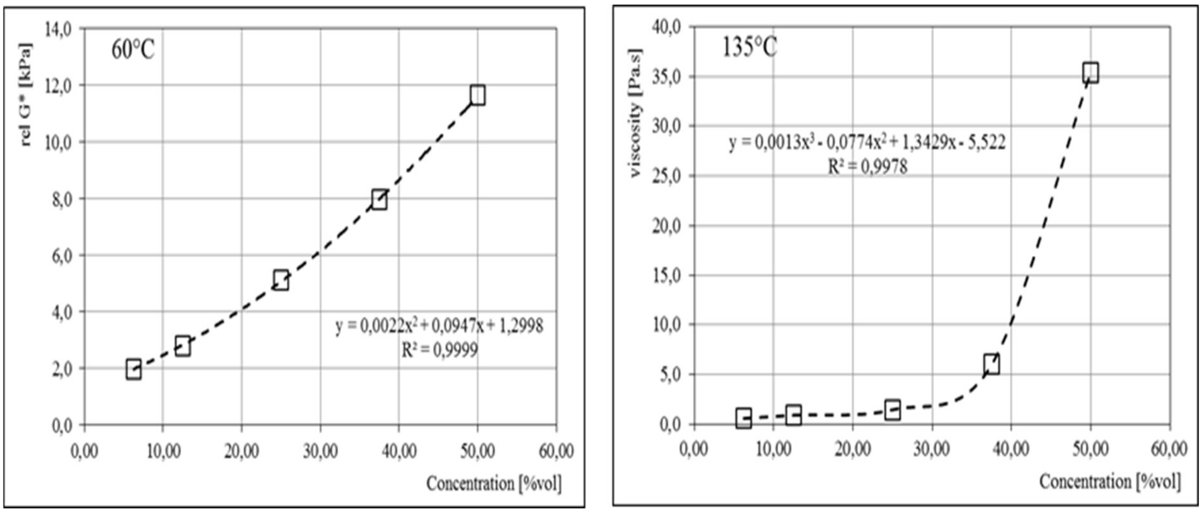

Figure 03. Comparison of oscillation and viscosity curves for concentration series of the selected filler.

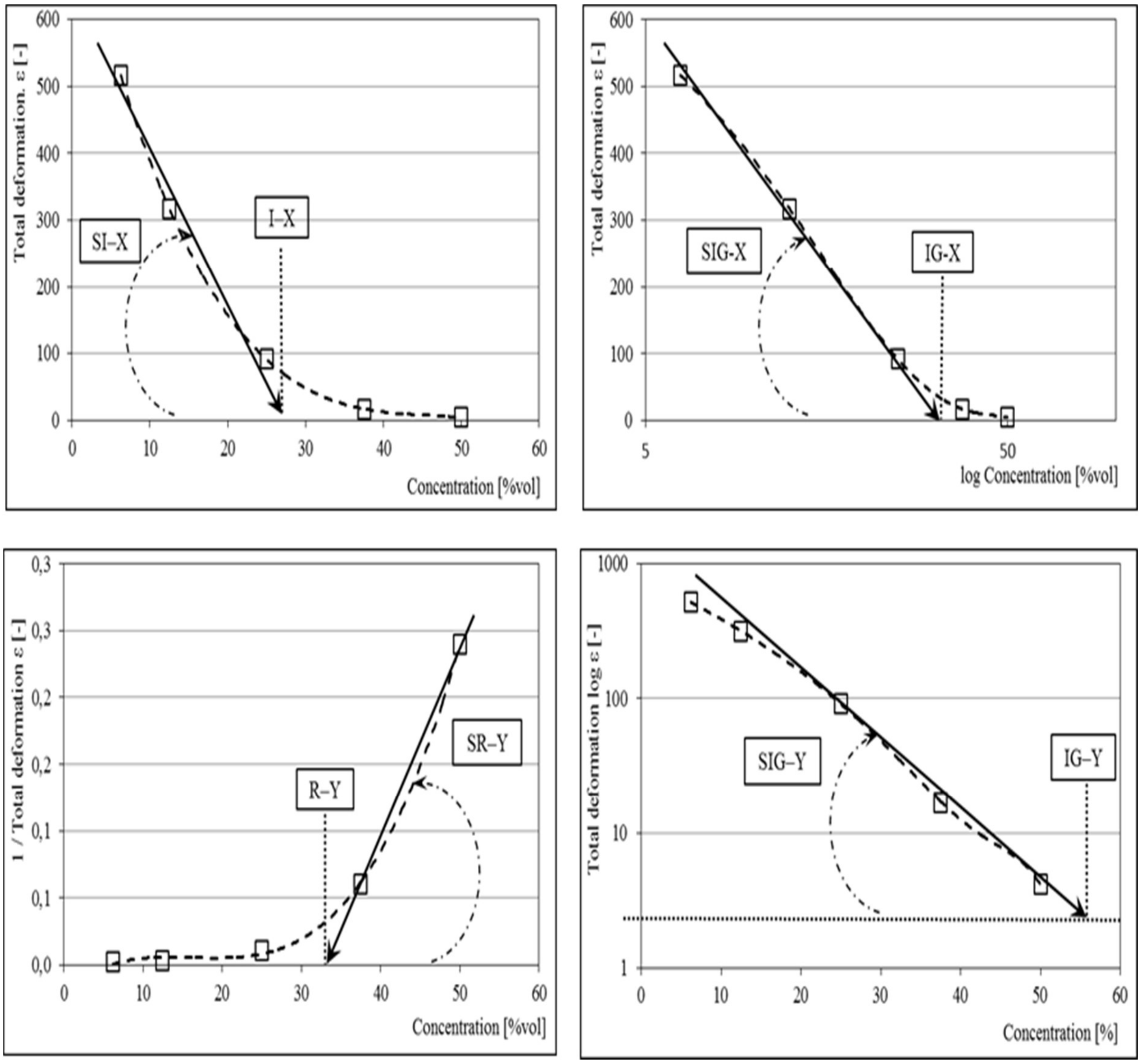

Figure 04. Cumulative creep screening of mastic (MSCRT) – results scaling of concentration series.

The application of the creep test seems to be a very suitable experimental configuration. A change in trend in region B is very well expressed in comparison with the oscillation mode. The ten-load pulse test more closely reflects the natural distribution and orientation of the filler particles in the mastic. This is because the non-spherical particles of the material are gradually oriented along the flow direction. This phenomenon is mainly associated with high concentration phyllosilicate minerals (an initial decrease in viscosity). For some materials, deagglomeration can also be observed (an initial increase in viscosity). The stress controlled MSCRT was performed in accordance with EN 16659 (EN 16659, 2016) and was originally designed for testing modified bituminous binders. The prescribed shear stress of 0,1 kPa and 3,2 kPa may be too low to clearly differentiate the binders. The test therefore included a third loading sequence at a shear stress level of 6,4 kPa. The results are deliberately expressed as nominal strain, which is directly proportional to the absolute angular deviation (in radians), so that the difference in deformation at different levels of shear stress can be seen.

For the construction of meaningful straight lines provided by the MSCRT method, it is necessary to choose different scaling of the results. For region A (low concentration area), it is appropriate to use linear or logarithmic scaling X depending on how many points are included in the line construction. For region B (high concentration of the filler), logarithmic or reciprocal scaling of the Y-axis must be used (in the case of logarithmic scaling Y, the X-axis intersects the Y-axis arbitrarily in the value corresponding to an angular deformation of 0 1 rad at 1,5 mm gap and 25 mm geometry diameter). The parameters are shown in Figure 4 and Table 2.

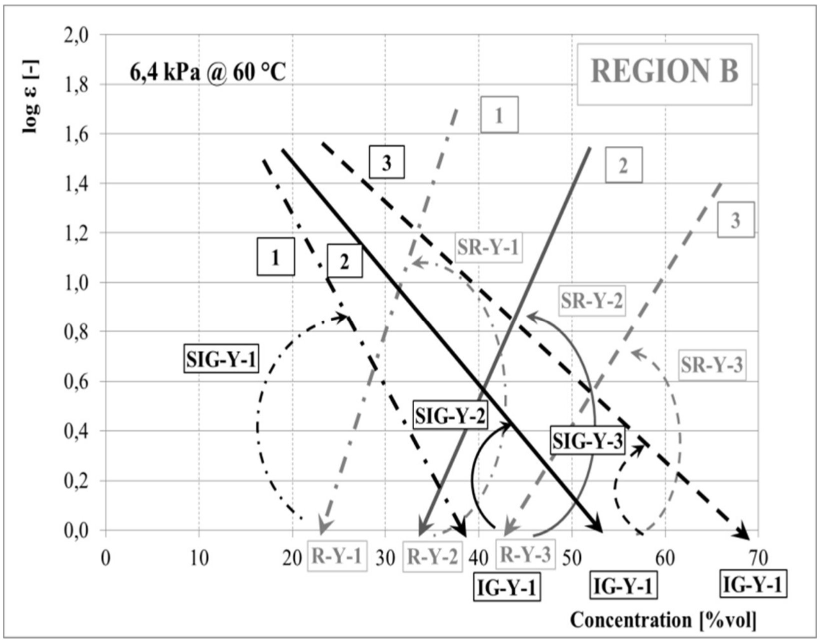

Figure 05. Creep screening of mastic – basic critical fraction parameters in region B of three different types of mastic based on 70/100 bituminous binder.

5.2. Discussion – viscosity screening

In general, concentration series were used to evaluate the reinforcing properties of fillers in mastic mortar over the whole concentration range. The empirical material parameters derived from the viscosity screening could provide some valid information on the filler-bitumen interaction.

Plotting the resulting viscosity curve of region A on a reciprocal scale (1/Y) is a common rheological technique. In the flow arrangement, this trick shows very well how "mutually non-interacting" particles affect the surrounding bituminous layers. In particular, the first two points (6,25 % and 12,5 % by volume) are crucial in this region. They quantify the interaction that occurs under shear loading of the diluted material and show how rapidly the total amount of the affected bituminous binder increases (assuming there is no mutual interaction of particles). It would be logical to expect that the straight line would pass through point 1 on the Y-axis of the reciprocal relative values. However, deviations can occur in real samples (measurement inaccuracy, three-point trend line calculation). Fillers with a significantly higher effect on the surrounding bituminous binder (higher stiffening gradient) produce a trend line that is at a sharper angle to the Y-axis and intersects the X-axis at a lower concentration in region A (Fig. 2). This intersection defines a critical fractional volume FrX_A. A possible physical interpretation of FrX_A postulates that all of the bituminous binder has already been affected by the filler under prevailing mechanisms of independent interaction.

This effect is not necessarily based solely on the adsorption of the bituminous binder. It may also be partially influenced by the shape, porosity and microtexture of the surface, etc. (similar properties determine the value of the voids in the dry compacted filler). In real conditions for materials with a higher stiffening gradient, the third point of the trend line may deviate slightly from this line (which is based on only two concentration points), as this point may be in the transition region where the influence of other particles begins to manifest itself.

Figure 2 shows the basic critical parameters obtained from the viscosity screening of three real fillers. If the intersection of the regression line of the apparent viscosity of region A in linear scaling with the x-axis lies beyond the critical volume fraction FrX_B ($FrX_A > FrX_B$), region B already represents the conditions for a fully affected binder. In this case, the increase in viscosity is not likely to be driven by the disappearance of free unaffected asphalt, but rather by processes such as a reduction in the thickness of the bituminous film and an increase in the statistical frequency of direct contact of the filler particles (covered by the absorbed binder). Cohesive behaviour at small distances between two particles could also play a role. In this case, the regression line for calculating the critical fractional volumes FrX_B reliably reflects the properties of region B.

However, estimating the viscosity at the 50,0 %.vol concentration point from its measurement at the 37,5 %.vol concentration point could be misleading. In this concentration region, the primary controlling variable appears to be the gradient describing an increase in the intensity of the interaction of individual filler particles. This interaction occurs under conditions where the bituminous binder is fully influenced by the physico-chemical effects of the surface of the filler particles. In other words, there is almost no free/unaffected asphalt. The position of the FrX_B fractional volume locates the onset of this dependence by determining the critical concentration on the X-axis.

In the region of high filler concentrations, the differences between the mastic viscosities of the individual fillers are best expressed by the rectified parameter $SFrX_B$. or FrX_BS. Such rectified fraction parameters take into account the rate of change of the measured viscosity with increasing concentration (correction by slope). Figure 2 clearly shows the disparity of the fillers – which would otherwise appear to be the same based on the critical fractional volume of FrX_B.

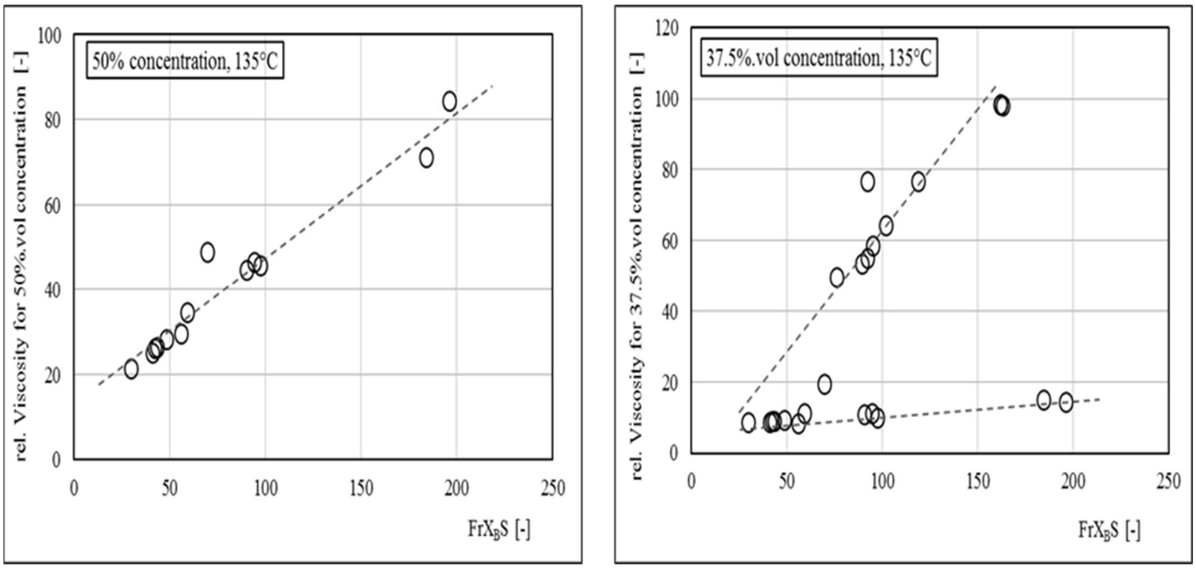

Figure 06. Relationship between the critical fraction value FrX_BS (left), the slope S_A (right) and viscosity values of mastic at the concentration points of 50,0 %.vol and 37,5 %.vol.

This finding means that the entire viscosity behaviour at higher concentrations can be described by only one fractional parameter, which characterises the material very well and reflects the predominant mechanism and intensity of interaction in mastics. The difference between the $SFrX_B$ and FrX_BS parameters lies in the nature of the functional control variable (slope S_A). For the FrX_BS variable, the functional dependence is directly proportional to the slope A ($FrX_BS = f(S_A)$). On the other hand, the variable $SFrX_B$ is determined by a power-law relationship ($SFrX_B = f(1/S_A)$), which therefore corresponds to a different type of scaling with a different absolute position of the fillers in the graphical representation, see Figures 6 and 7. Based on observations from the evaluation of other sets of fillers, the reciprocal scaling appears to be slightly preferable as it better distinguishes the filler material. It also appears necessary to increase the number of concentration points used to define the control line in the higher concentration range. As a result, the $SFrX_B$ values could also be informative about the filler behaviour at lower concentrations. The point is that the extraction region for calculating the fractional parameters is shifted from the regression line region B to the transition region (left shift) without affecting the predictive ability for estimating the rheological response to load (viscosity) in the entire concentration region B.

The $SFrX_B$ or FrX_BS parameters can be an essential tool for mastic control in solving material workability problems. The ability of the rectified FrX_BS parameters to sort fillers according to their activity in the mastic is demonstrated in Figures 6 and 7. Unlike the point of 37.5 %.vol, the set of values at the concentration point of 50 %.vol does not include phyllosilicate standards (mica) or rocks with highly reactive mica. Mastics with these fillers are not measurable in this concentration range (low SFrX$_B$ range, Figures 6 and 7 left). Logically, in the case of the fractional parameter SFrX$_B$, its value decreases as the critical fractional parameter FrX$_B$ shifts to lower values (the phenomenon occurs earlier) and the steepness of the regression line (more intense interaction). Therefore, this parameter is more suitable as a tool for sorting a group of fillers.

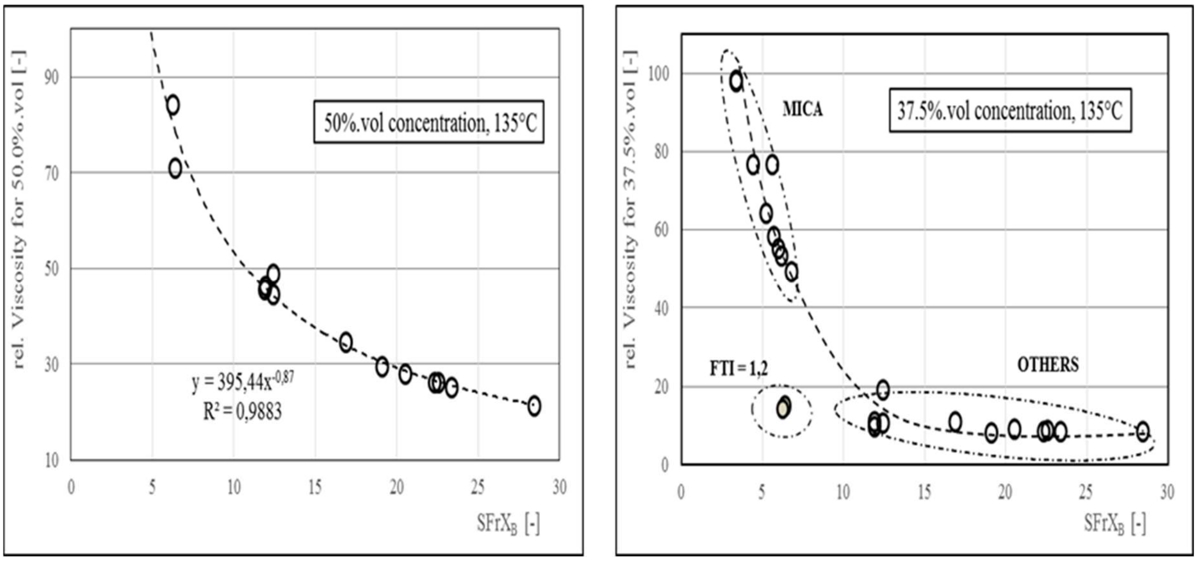

Figure 07. Relationship between the critical fraction value SFrX$_B$ (left), FrX$_B$ (right) and the viscosity values of mastic at the concentration point of 50,0 %.vol.

When examining the relationship between the rectified fraction SFrX$_B$ and the critical volume fraction FrX$_B$ (sorting properties by FrX$_B$), it is possible to identify several subgroups that reflect the different reactivity of their members under conditions of interaction of individual particles with adjacent asphalt layers on their surface. However, the results may be subject to experimental error, which can be reduced by increasing the number of measurement points in a given concentration range.

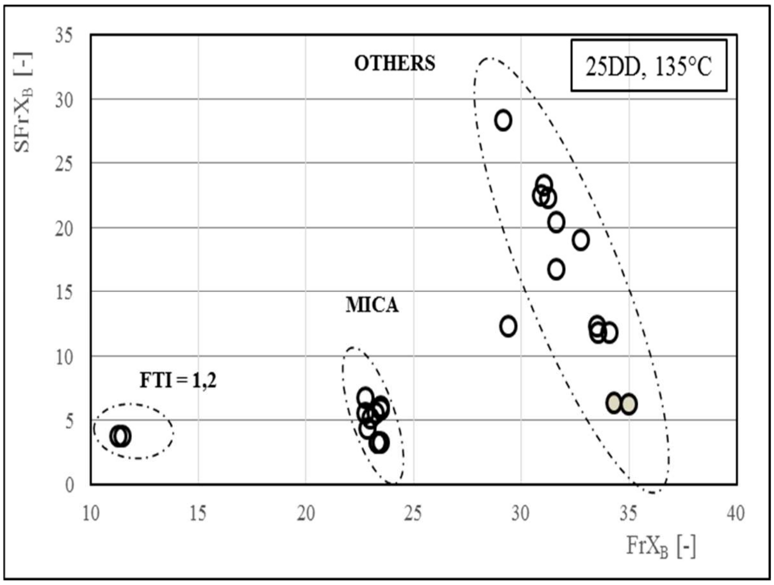

Figure 8. Relationship between the parameter FrX$_B$ versus SFrX$_B$ based on viscosity screening.

This split within the group is even more apparent when the viscosity results are sorted by the value of the uncorrected critical fractional volume FrX_A, FrX_B, Cross B or the rectified parameter $SFrX_A$ (low concentration range). If only FrX_B is used, the results are divided into two dependencies corresponding to a weak and a strong interaction between filler and binder. In the case of strong filler reactivity, the "edge" on the viscosity-concentration curve appears much earlier, so that the critical fractional volume FrX_B is significantly smaller.

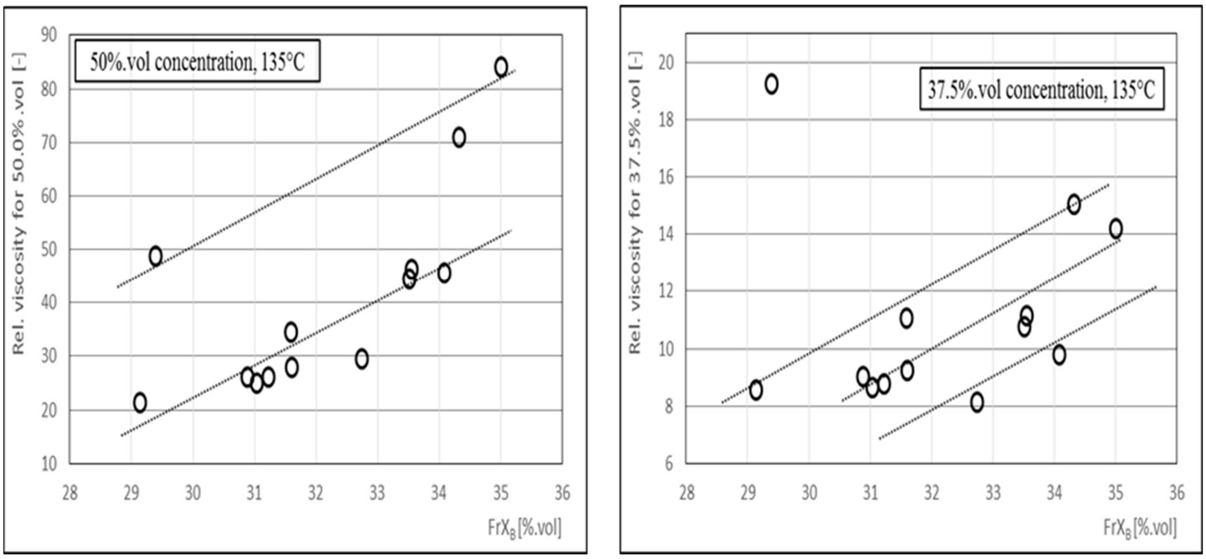

Figure 09. Sorting parameter FrX_B for viscosity values at the concentration point of 50,0 %.vol (left) and 37,5 %.vol (right).

The parameters of region A reflect the behaviour of the isolated filler particles. While being partially valid for higher concentrations, they can also provide information about the nature of the filler activity in relation to bituminous binder. The low $SFrX_A$ and FrX_A identify mica materials with a strong relationship to the bituminous binder.

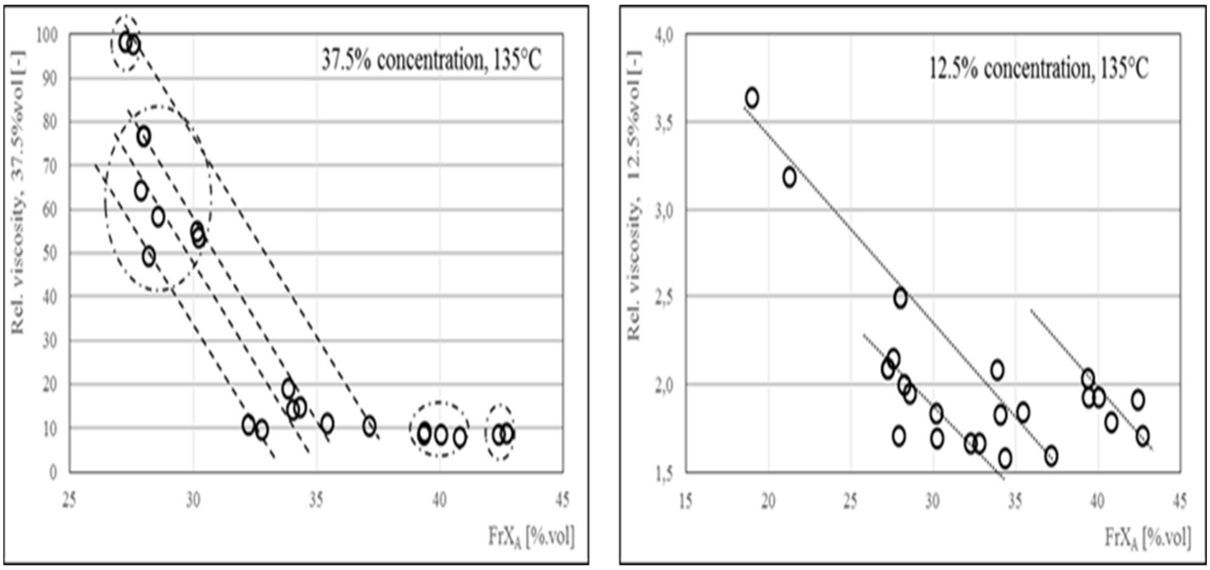

Figure 10. Viscosity controlled by critical fractional parameter FrX_A for high (left) and low (right) concentration.

This clustering also appears when the filler samples are examined based on other critical fractional parameters and at various zooming. Analogous phenomena can also be observed in creep screening. Typically, the linear dependences are shifted translationally shifted along the coordinate axis.

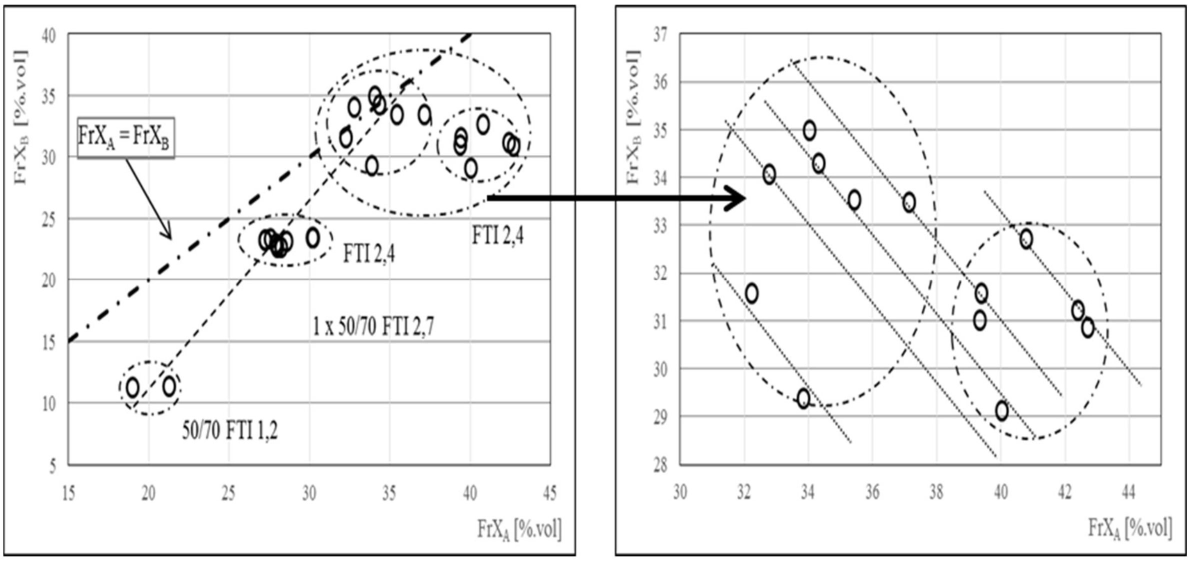

Figure 11. Relationship between critical fractional volumes FrX$_A$ and FrX$_B$ (left); the same in detail (right).

Other types of groupings can be obtained by comparing different critical parameters. These group relations open up a different perspective from which the nature and intensity of the interactions can be observed. The simplest example is a relation of the critical fractional parameter of FrX$_A$ and FrX$_B$ obtained for each concentration zone (Figure 11).

The central straight line shows a relationship between the filler particles in the isolated and highly concentrated states (see parity line FrX$_A$ = FrX$_B$). One of the filler groups in the upper part of the diagram deviates from the group line, indicating a change in the nature of the interaction. When the critical fractional volume FrX$_B$ is higher than the critical fractional volume FrX$_A$ (FrX$_A$ < FrX$_B$), the interactions of the filler particles behind the "parametric edge" take place under the condition of a partially unaffected binder (area above the parity line, Figure 11 left). Fillers (particles) below the parity line interact under the fully affected binder (FrX$_A$ > FrX$_B$). It follows that increasing the concentration of the filler leads to a reduction in the spacing between the filler particles and therefore a reduction in the thickness of the asphalt layer, which will have a strong effect on the viscosity of the mastic system. The detailed view of this plot area (Figure 11, right) also allows the results to be interpreted as a translational shift of the results along the Y-axis.

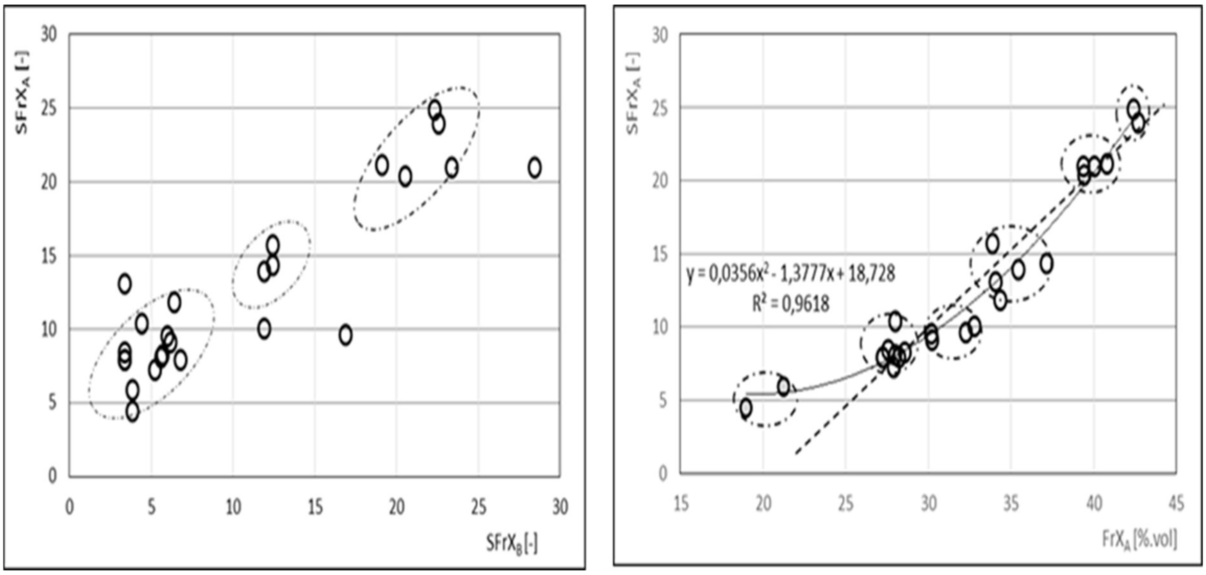

Figure 12. Relationship between the critical fractional volumes $SFrX_B$ – $SFrX_A$ (left) and FrX_A – $SFrX_B$ (right).

Fillers can also be sorted according to the critical fraction parameter $SFrX_B$ versus $SFrX_A$ and FrX_B versus $SFrX_B$. With respect to the critical fractional size $SFrX_B$, the fillers can again be divided into subgroups, taking into account weak and strong interactions. The FrX_A – $SFrX_A$ correlation should be a linear regression due to the principle of plotting critical fraction parameters.

The above combinations can be used to investigate and quantify the effect of specific fillers on the resulting properties of the mastic. Further research is required for a more detailed understanding of the correlations and the reorganisation of the fillers into different groups.

5.3. Discussion – creep screening

Creep screening using the MSCRT method at intermediate service temperatures has been used mainly to characterise the true deformation resistance of mastic. The whole concept is based on the evaluation of the accumulated creep deformation. The developed parameters I-X, SI-X, R-Y, SR-Y (linear scaling of the X and Y axes), IG-Y, SIG-Y (logarithmic scaling of the Y axis) and IG-X, SIG-X (logarithmic scaling of the X axis) are described above in Figure 4. The prefix S indicates a correction of the resulting fractional volumes by the slope of the regression straight line. As shown below, the empirical parameters IG-Y, SR-Y and IG-X (I-X) can be used as material parameters to describe the influence of the filler on this mastic property.

The sample set for creep screening is similar to the set for viscosity screening. The mica standards, which have a specific and robust interaction with the bituminous binder, are again included in the sample set. There is also a shift of the results relative to each other along the Y-axis. This phenomenon is probably related to the petrographic characteristics and granulometry of the filler. The strength of the interaction increases linearly with the increasing filler content. Therefore, the slope of the shifted curves is maintained as in the case of viscosity screening. The most significant achievement is that the stiffening effect of the filler at higher concentrations can be characterised by a single general parameter.

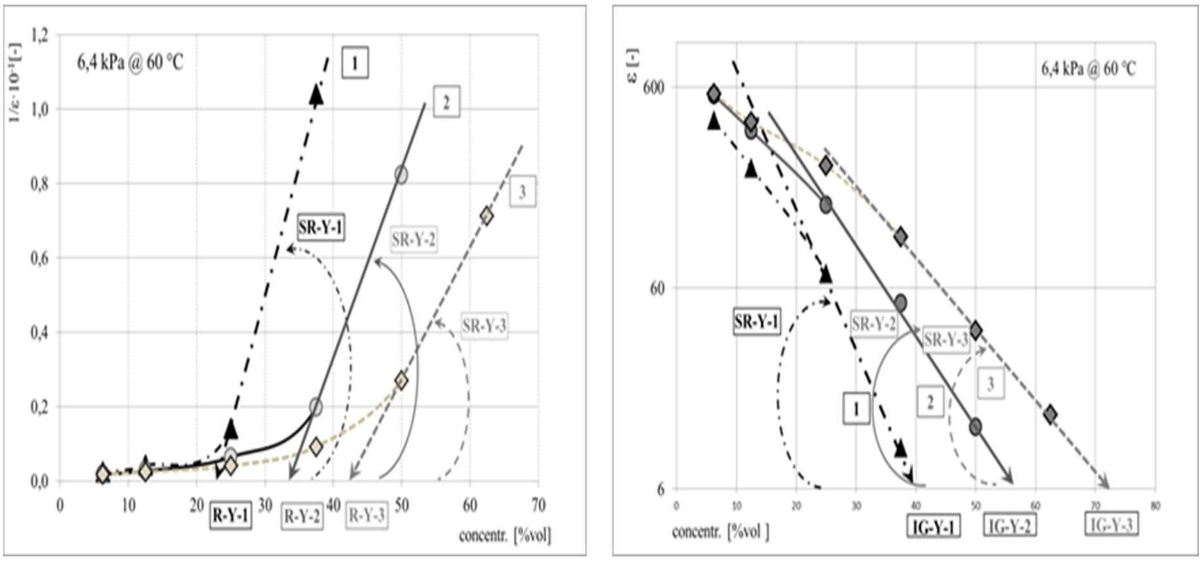

Figure 13. Demonstration of the variability of the parameters R-Y, SR-Y, IG-Y, SIG-Y by describing three real types of fillers in the concentration range B.

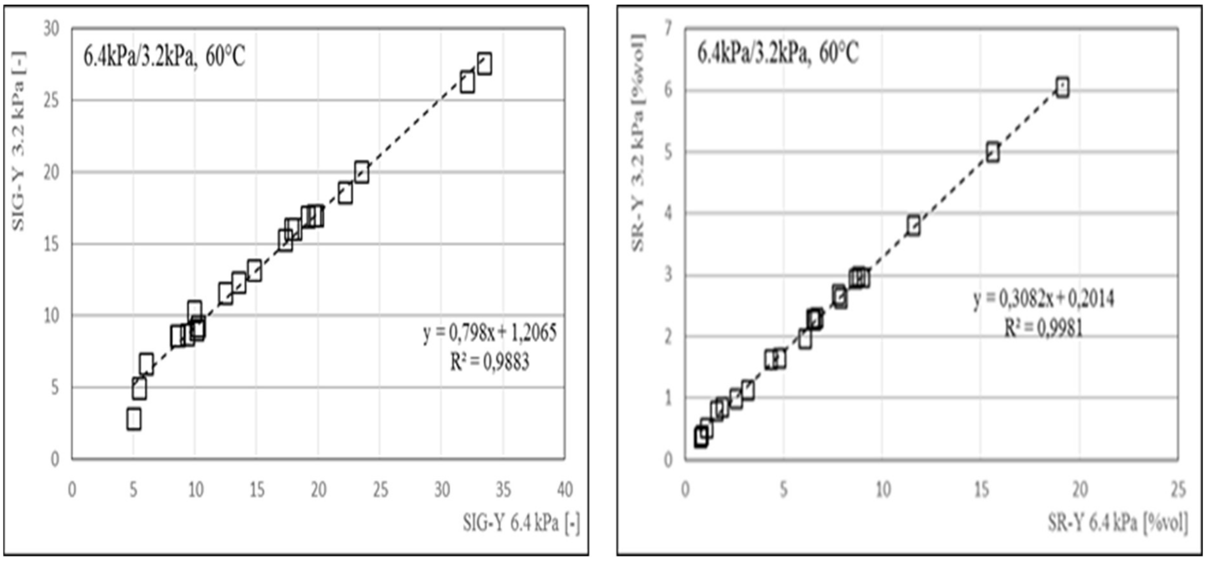

Figure 14. Correlation of SIG-Y_4P quantities (left) and SR-Y_4P quantities (right) between two shear stress levels (6,4 kPa and 3,2 kPa).

In order to better differentiate between the different types of mastic, it was necessary to introduce an additional shear stress level of 6,4 kPa in addition to the standard shear stress level of 3,2 kPa. The amount of absolute cumulative deformation (strain) at a constant gap of 1,5 mm in the test geometry corresponds to the deformation resistance of the specimen at the applied stress level. Except for two outlying values, there is a robust correlation between the absolute cumulative deformation in strain for the shear stress of 6,4 kPa and 3,2 kPa ($R^2 = 0{,}99$ for the concentration point of 37,5 %.vol and $R^2 = 0{,}99$ for the concentration point of 50,0 %.vol). The correlation between the concentration points of 50,0 % and 37,5 % confirms the reliability of the sample preparation ($R^2 = 0{,}94$ for the shear stress of 6,4 kPa, $R^2 = 0{,}95$ for the shear stress of 3,2 kPa).

The IG-Y parameters prove to be one of the best correlating indicators of resistance to deformation (strain at the 50,0 %vol concentration point, second degree polynomial dependence, logarithmic scaling of the x-axis, R^2 = 0,98; 6,4 kPa). Correction by the slope of the straight line of the regression gives the indicator SIG-Y and reduces the correlation to R^2 = 0,91.

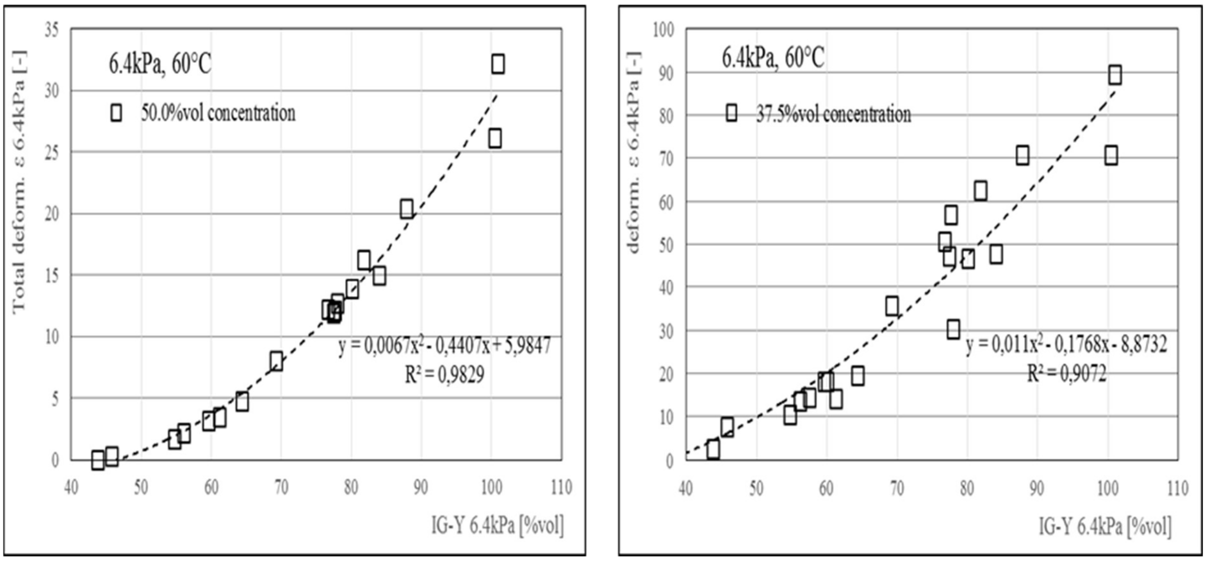

Figure 15. Absolute cumulative deformation as a function of IG-Y_5P parameter at 50,0 %.vol concentration (left) and IG-Y_5P at 37,5 %.vol concentration (right).

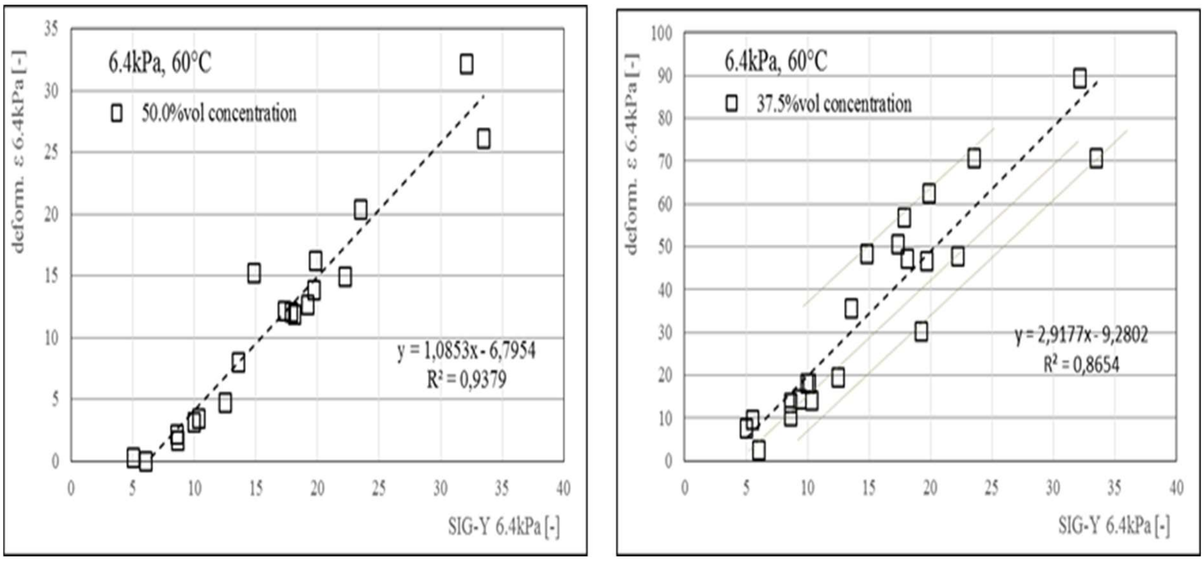

Figure 16. Relationship between absolute cumulative deformation and the SIG-Y_5P parameter at 50,0 %.vol. concentration (left) and at 37,5 %.vol. concentration (right).

The key fractional parameters can be constructed from different points on the concentration curve. The basic design of the concentration series covers five concentration points of the filler in the mastic (5P concentration range). However, already in the viscosity screening, the need to increase the number of detection points in the transition region and also in the high concentration region became apparent. Analysis of the results shows that the transition region, where the so-called parametric edge is located, provides crucial information about the behaviour of the mastic material even at higher filler concentrations. The high value of the extracted information from the transition region is due to the fact that the two control mechanisms of the filler interaction in the asphalt binder are intertwined in this region. The adsorption of bitumen binder onto the surface of the filler particles (part of the control mechanism in region A) affects the direct friction between the par ticles in region B (the main control mechanism in region B).

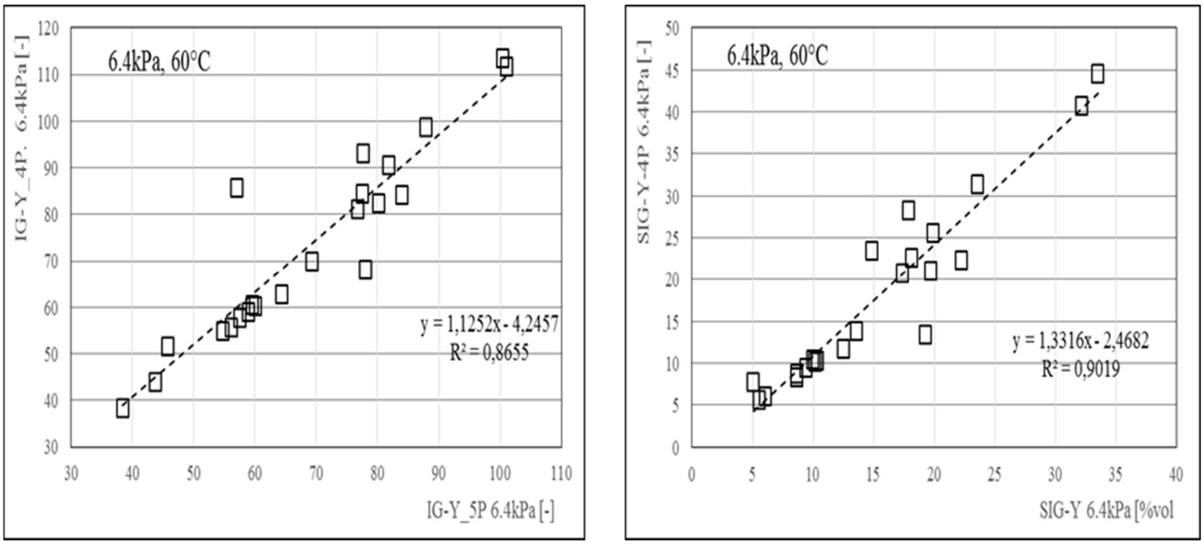

Figure 17. Relationships between IG-Y_5P and IG-Y_4P (left) or between SIG-Y_5P and SGI-Y_4P P1) (right).

This is evident from the values of the coefficients of determination of the correlation of these fractional material parameters with the strain resistance values at different concentration points. In the case of the IG-Y (SIG-Y) material variable, we can only construct these parameters from four points, which is a consequence of the fact that materials with a high mica content cannot be measured at 50 %vol concentration due to their high stiffness. For other materials, the calculated parameters constructed from a four-point concentration series (4P range) instead of five points (5P range) generally give better values of the coefficient of determination in relation to the resistance to deformation. For example, if the IG-Y parameter is derived from the 5P range instead of the 4P range, the quality of the correlation decreases – for the 37,5 %.vol concentration point from R^2 value of 0,95 to $R^2 = 0{,}84$ and for the 50,0 %.vol concentration point from R^2 value of 0,91 to $R^2 = 0{,}84$. The relationship between IG-Y and SIG-Y for the 4P and 5P range is shown in Figure 17. As will be shown below, the same conclusions can be applied to the material parameter SR-Y.

The SR-Y parameter is the other sensitive material characteristic – it takes into account the position of the critical fractional R-Y volume on the x-axis and, at the same time, the specific gradient of the change in mastic deformation resistance with increasing filler concentration (correction by slope). The SR-Y quantity can also define the differences in mastic behaviour by regrouping the sample set into at least two linear dependencies with a very close correlation (Figure 18).

P1) Fractional parameters derived from 5P – five concentration points or 4P – four concentration points.

The SR-Y parameter is the other sensitive material characteristic – it takes into account the position of the critical fractional R-Y volume on the x-axis and, at the same time, the specific gradient of the change in mastic deformation resistance with increasing filler concentration (correction by slope). The SR-Y quantity can also define the differences in mastic behaviour and identify the classified mica standards by regrouping the sample set into at least two linear dependencies with a very close correlation (Figure 18).

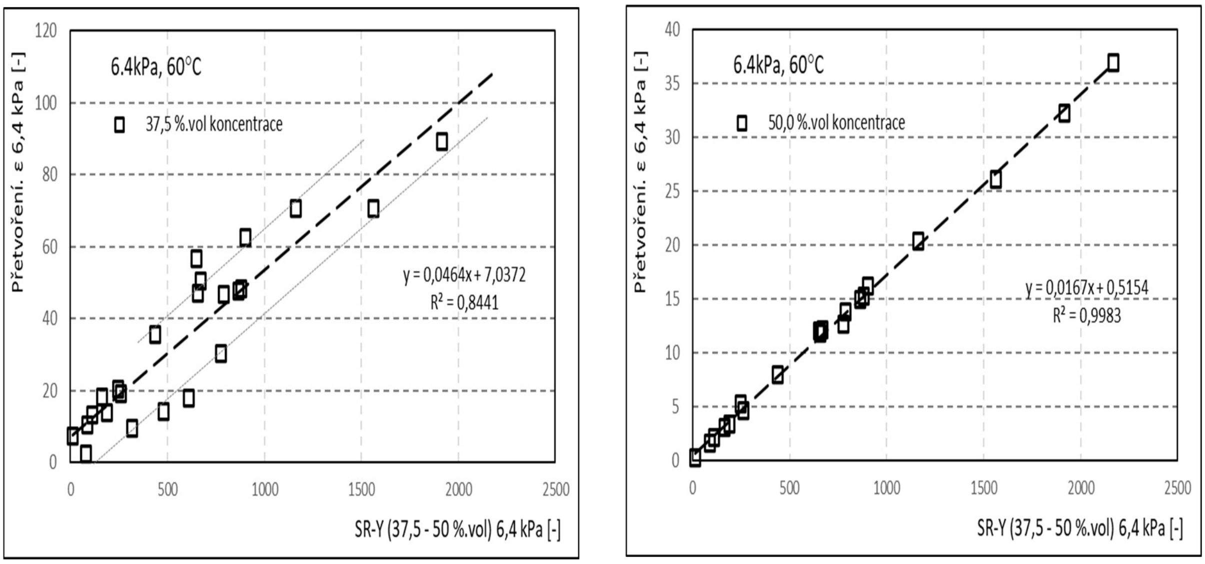

Figure 18. Relationship between SR-Y (37,5–50,0 %.vol, P5) and absolute deformation for 37,5 %vol (left) and 50,0 %vol (right) concentration points.

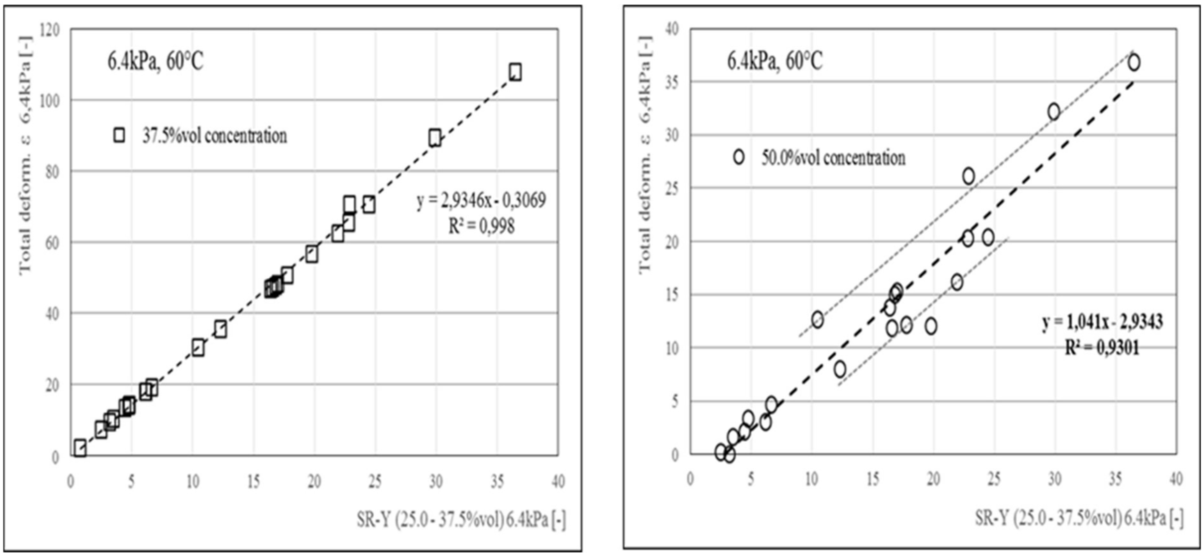

Figure 19. Relationship between SR-Y (25,0–37,5 %.vol, P4) and absolute deformation for 37,5 %vol (left) and 50,0 %vol (right) concentration points.

The critical fractional volumes extracted from the high concentration region (5P region) have a good predictive ability for higher concentration points. (SR-Y, deformation ε, R^2 = 0,98 at 50,0 %.vol; R^2 = 0,82 at 37,5 %.vol). If the rectified parameter SR-Y derived from the concentration points 25,0 % and 37,5 % (four-point concentration series) is used, the accuracy of the deformation results agreement increases at both concentration points; at

37,5 %.vol (R^2 = 1,0), at 50.0% (R^2 = 0,93). In this case the coefficients of determination R^2 for the SR-Y reach more balanced values and the original sample collection provides a much more compact view. This finding implies that the extraction region of 25,0–37,5 % carries more accurate information about the interaction mechanism in the high concentration region than the calculated fractional quantities directly from this region. The correlation between the two options for deriving the SR-Y parameters is shown in Figure 20.

The sensitivity of the MSCRT methods leads to further differentiation of initially homogeneous groups. The correlation between the fractional parameters SR-Y (R-Y) of the base material set derived from various extraction intervals (P3 – P4 versus P4 – P5) shows that there are at least three groups with slightly different behaviour. The same result is obtained from the relation of the SR-Y value to the deformation value at the concentration point 37,5%vol, extracted from the concentration range (37,5–50,0 %.vol). Mica standards are very well identified by comparing the extraction variants of R-Y quantities.

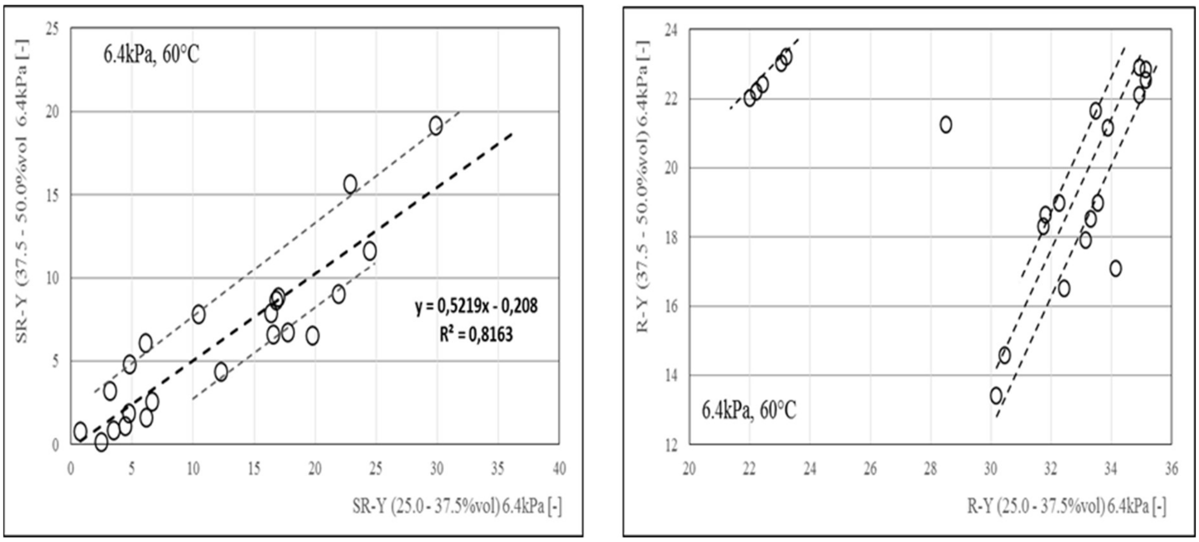

Figure 20. Relation between parameters SR-Y (37,5–50,0 %vol) and SR-Y (37,5–50,0 %vol) (left), the relation between parameters R-Y (25,0–37,5 %.vol) and R-Y (37,5–50,0 %.vol) (right).

In sorting the cumulative deformation resistance of mastic mortar assessed by the SR-Y rectified fractional volume value, the slope of the regression line acts as a controlling element. This fact is evident from Figure 21, as the correlation between slope A and cumulative deformations is inherently very high. In the construction of the SR-Y parameter, the initial fractional volume R-Y serves to adjust the position of the parametric edge of the deformation resistance on the X-axis. The increasing mutual interactions of the asphalt binder adhering closely to the surface of the filler particles (or, with increasing concentration, the mutual friction of the particles) begin to dominate beyond this critical filler fraction concentration (R-Y). Due to the application of reciprocal scaling, the resulting relationship between the SR-Y parameter and the deformation resistance is linear.

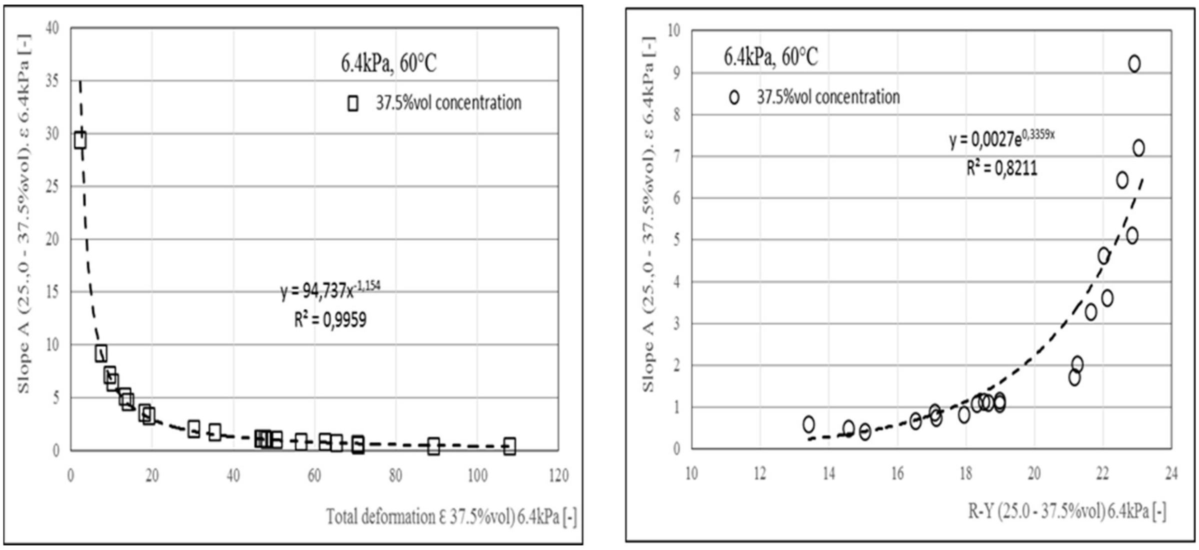

Figure 21. Relationships between the S_A slope of the regression line (25,0–37,5 %.vol) and the absolute deformation ε at the 37,5 %.vol concentration point (left); the relationship between R-Y (25,0–37,5%vol) and the S_A slope of the regression line (25,0–37,5 %.vol (right).

The setting of fixed concentration points seems to be a problem because some materials are not measurable at a concentration point of 50 %vol. Calculating their critical values only from four points affects these parameters which may carry slightly different information. This approach is sufficient for general material classification but is a limiting factor for full research purposes. If the critical parameters of SR-Y and IG-Y are obtained uniformly only from the range of 25,0–37,5 %.vol. concentration, these quantities are equivalent and undifferentiated. Simultaneously, they are sufficiently predictive of mastic deformation resistance at higher filler concentrations. Identification and more precise description of the relationships will require a larger set of samples and reconsideration of the number and positioning of the concentration points. The calibration of the fractional quantities concept to a system of 8 (8P) contracted concentration points is described in Chapter E.8 of the thesis.

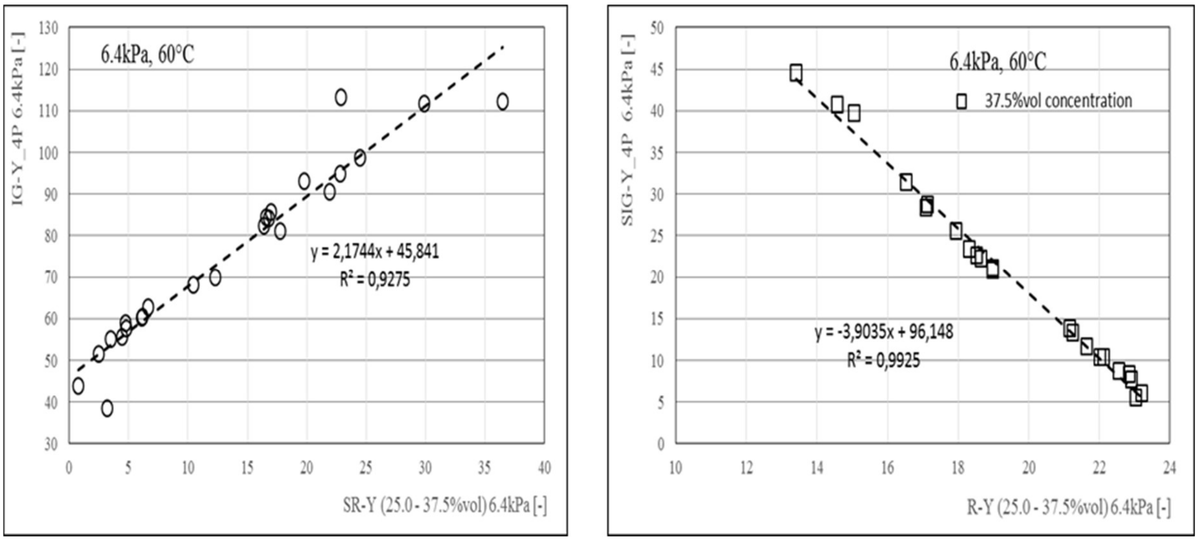

Figure 22. Relationship between SR-Y and IG-Y or R-Y and SIG-Y, all parameters extracted from the same concentration range.

The ability to construct a single material property for each filler that describes its general activity over the entire concentration range is of great benefit in the context of material differentiation. The material parameters SR-Y, IG-Y and SIG-Y correlate well with the deformation stability of mastics in region B. These parameters make it possible to compare the influence of the filler of a given granulometry on the deformation properties of the mastic over the whole range of higher concentrations. This solution could also facilitate the study of the effect of key filler variables on the rutting resistance of HMA or the tailoring of HMA design.

Many other relationships can be constructed in a similar way to describe other individual ways of classifying resins that reflect filler activity. A surprising fact was found when evaluating the I-X, IG-X parameters against the deformation values at high filler concentrations. In terms of information value, I-X, IG-X are equivalent. Although the I-X parameter represents the low concentration region A, this critical fractional volume I-X can very well estimate the general deformation resistance of the mastic at high filler concentrations (R^2 = 0,84; 37,5 %.vol). In contrast, the I-X fractional parameter fails precisely in the region of low concentrations.

It can be concluded that the interactions in the highly concentrated bitumen suspension are controlled by various factors, but the binder adhered to the particle surface appears to play a crucial role in the overall rheological behaviour of the mastic. A more plausible explanation is that the I-X parameter extracted from the first three concentration points already belongs to the transition region by its right extreme point. The influence of this point (25,0 %vol) is therefore significant. In addition, there may be a higher error in the determination of the cumulative strain at very low concentrations. Therefore, this factor cannot be used to describe the cumulative deformation properties in the low concentration range, where the scatter of the results is very large (in contrast to the viscosity screen), see Figure 23. This demonstrates the importance of the transition region.

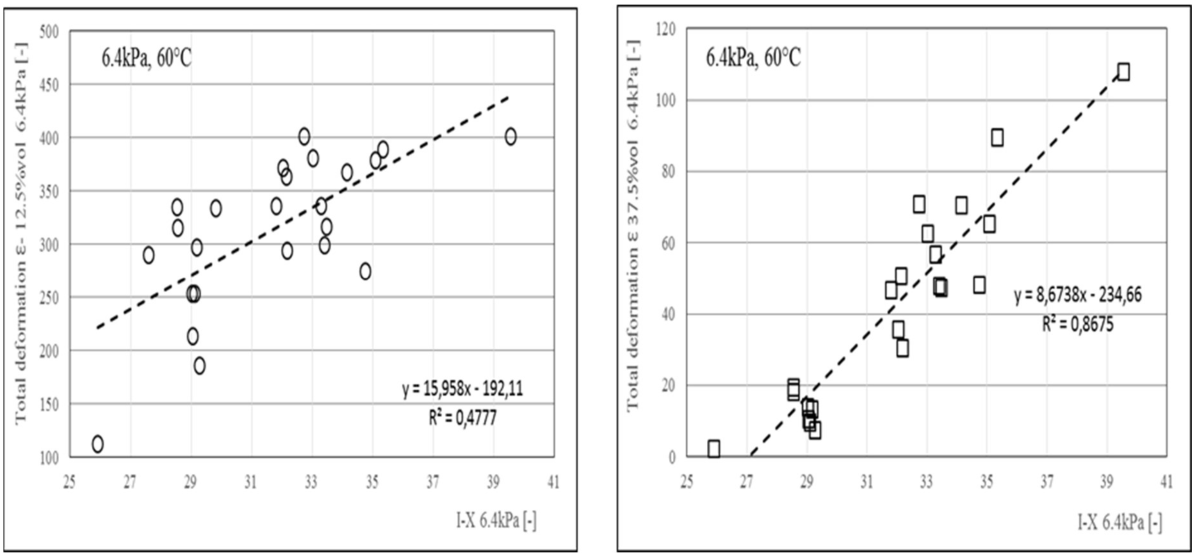

Figure 23. Relationship between parameters I-X and the cumulative deformation at the concentration points 12,5 %.vol (left) and 37,5 %.vol (right).

The intrinsic properties of the I-X parameter also correlate well with the parameter extracted from the high concentration region (SR-Y or IG-I_4P). On the other hand, no meaningful general correlation of SI-X with relatively significant differences in deformation resistance can be detected in this group of filler samples.

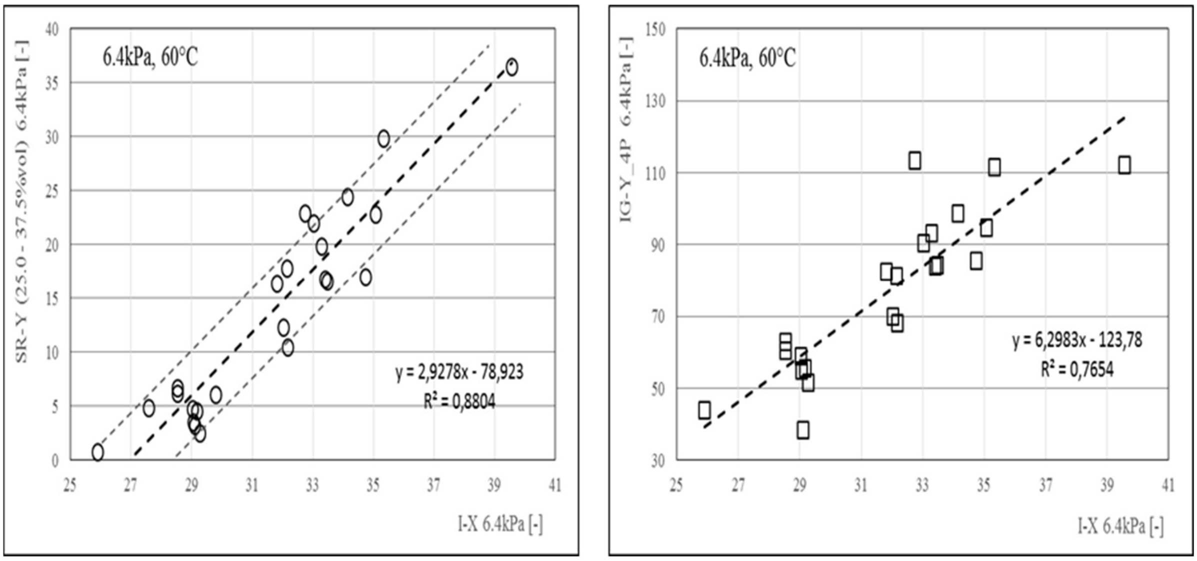

Figure 24. Relationship between parameters I-X and SR-Y (37,5–50,0 %.vol) (left) or IG-Y_4P (right)

The surface properties (adhesion) mentioned above are probably the reason for the geometric structures in Figure 24 (the correlation of deformation values between the concentration point of 6,25 %vol and 50,0 %vol at a stress level of 6,4 kPa). It is worth noting that the slopes of the straight lines for all of the parallel shifted lines in the diagram are very similar to each other. This may indicate a difference in the intensity of the control interaction between the bituminous material and the filler. The interpretation of the results is possible in several ways and remains open for further research. From the results of this thesis, it appears that these substructures (also visible for different relationships between fractional parameters) are controlled by some elements from the mineral composition of the filler.

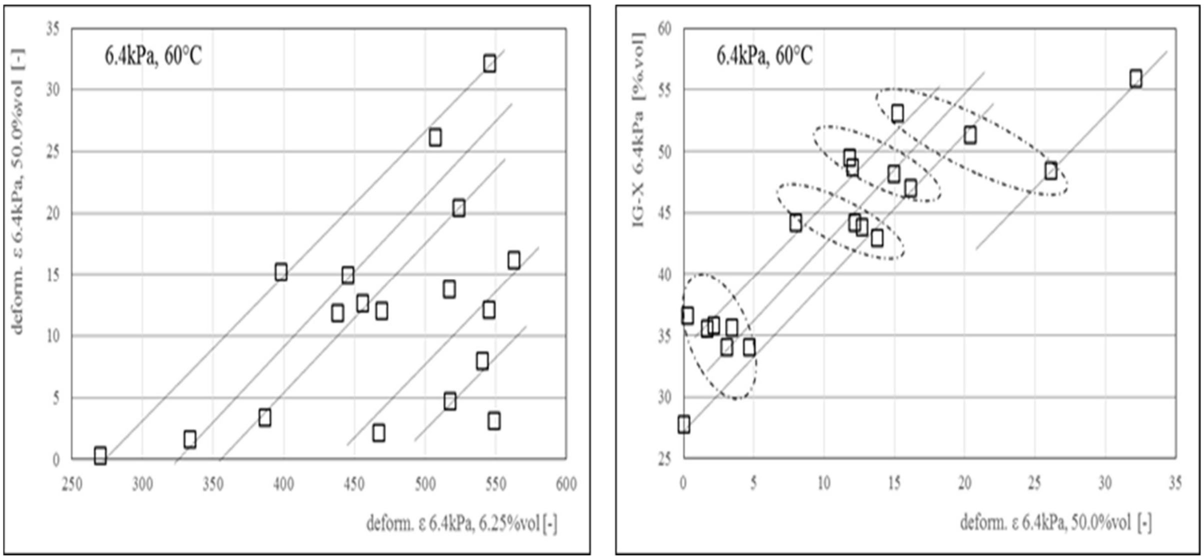

Figure 25. Relationship between deformation at 50,0 %.vol and critical parameter IG-X (left) and deformation at 6,25 %.vol (right), 6,4 kPa.

5.4. Relationships between parameters derived from the high and medium temperature regions

Although the rheological tests used are based on different loading modes, correlations can be found between some of the fractional parameters even though they are derived from different temperature ranges. This is particularly the case for the SR-Y or IG-Y parameters derived from the concentration range 25,0–37,5 %.vol or 4P. The quality of the correlations SR-Y or IG-Y with the parameters $SFrX_A$ (FrX_A) and $SFrX_B$ (FrX_B) is expressed by the coefficient of determination R^2, which varies around 0,75; see Figure 25.

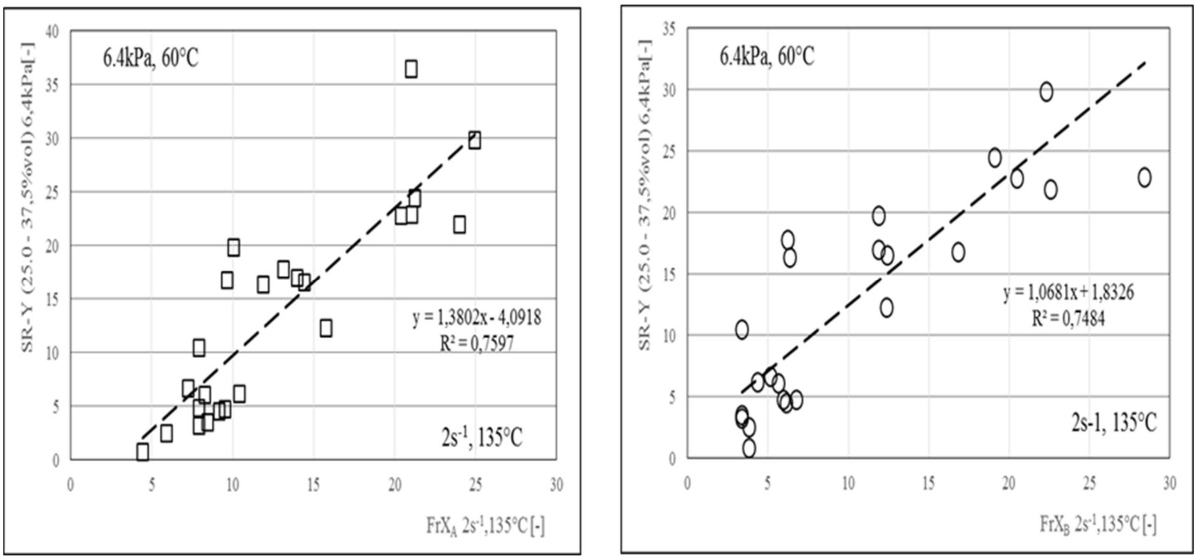

Figure 26. Relationship between parameter FrX_A (left) or FrX_B (right) and critical parameter SR-Y.

Similar relationships can be found for the parameters IG-Y versus FrX_A (FrX_A) (R^2 = 0,73) or FrX_B (R^2 = 0,79). This is a consequence of the mutual substitutability of the parameters SR-Y and IG-Y, as can be seen in Figure 21. (Note that $SFrX_A$ is also equivalent to FrX_A).

The other inter-parameter relationships are usually burdened with significant variance and the coefficient of determination is less than 0,55). Interestingly, the quantities that should characterise the behaviour in the high dilution region (region A), i.e., low filler concentration in the mastic (region A), are practically independent of each other (R^2 = 0,18).

6. CONCLUSIONS

- The proposed filler classification system was primarily designed to describe the behaviour and activity of petrographically different types of fillers in interaction with the asphalt binder, including the effect of their granulometry and specific surface area.
- The basis of the system is a description of filler activity based on newly developed single fractional parameters. These parameters allow fillers to be grouped (similar behaviour) and their performance to be sorted according to their material constants.
- The measurement of filler activity in mastic has been redefined. Filler performance was captured from rheological data over a wide concentration range reflecting two states of the filler - high dilution (isolated particles) and low dilution (interparticle friction). Each concentration region was defined by its own fractional variables. This approach allows each mechanism of interaction to be studied on an individual basis.
- Based on the results, the notion of a "parametric edge" and the importance of the region surrounding it were redefined. The rheological quantities measured in this region carry the most valuable information. This knowledge allowed the optimisation of the extraction variant for the calculation of fractional parameters.
- The system contains different parameters of the material fractions and by comparing them with each other and relating them to the rheological quantities, the mechanism of interaction between the filler and the asphalt binder can be studied. The system is dual because it allows the study of both the effect of the filler and the effect of the asphalt binder on the mastic.
- The thesis verified the concept of fractional quantities and calibrated it on a new set of fillers. As part of the calibration of the fractional quantities concept, the efficiency of each fractional quantity was evaluated and the extraction concentration intervals from which these quantities are calculated were optimised.
- As part of the study, new functional test protocols were optimised that can be used to study the rheological behaviour of mastics with minor modifications. The rheological approach to the evaluation of mastics using a dynamic shear rheometer is based on known rheologically reliable methods with acceptable repeatability. The selected rheological configurations provide mastic characteristics in the range of medium (MSCRT) and high (rotational viscosity) application temperatures.
- The use of creep or viscosity screening of concentration series provides a new comprehensive view of the evaluation possibilities of fillers and mastics derived from them. The results show that the presented methodology can be a powerful tool for further research as it allows the measurement and classification of interactions at the mastic components. Future research should focus on the influence of petrographic composition, granulometry, etc.
- The deformation or viscosity behaviour of the mastic in the high concertation zone can be expressed by a single composite parameter valid for any filler concentration. This parameter allows a better general comparison of the resins.

- The overall interpretation of the results, particularly for the relationships between the critical fractional parameters, which allows individual fillers to be rearranged into groups of similar activity in the mastic.
- Nevertheless, the new descriptive approach has great potential for the study of fillers. Some aspects of the methods have already been used to study the effect of some aggregate production processes on the properties of hot mix asphalt.

7. REFERENCES

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