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IMPACT OF MECHANICAL CHEMICALLY ACTIVATED RUBBER ON STRAIN AND FLOW BEHAVIOUR OF CRMB BINDERS

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ABSTRACT

One of the progressive trends which have been developed intensively in the last few years in road structures engineering are technical possibilities for optimal utilization of recycled rubber coming from old tires in asphalt mixtures and bituminous binders. In this connection there exist already several techniques, but at the same time, the knowledge about alterations in strain behavior of bitumen if modified by crumb rubber is still insufficient. Furthermore, the problem of heterogeneity of final composite binder is well known and not qualified completely. Presently known and used milling techniques lead only to a partial solution. Experimentally, the focus is therefore oriented on possible use of so called mechanical chemically activated micro-milled rubber gained by the disintegration process. Application of this treated rubber material should allow sufficient stability of resulting bituminous binder. The paper summarizes experimentally made modifications with use of milled and micro-milled waste rubber together with application of further chemical additives, like polyphosphoric acid, which improve the stability of binder composite structure. Alteration in strain behavior and in dynamic viscosity values are assessed by means of rheological testing of bituminous binders which have not been aged o degraded. In this study, oscillatory measurements of strain characteristics (complex shear modulus in terms of temperature or frequency sweeps) have been done in temperature range 20-70°C and in a wider frequency interval, simulating various traffic loading effects. Measurements are executed strictly in linear area of viscoelastic behavior of this material. Simultaneously, multi-creep stress recovery test with determination of characteristics describing compliance were performed. Last but not least, flow curves describing the level of workability of designed modified bituminous binders are compared and analyzed by application of rotational viscosimetry. Recommendations for further use of bitumen modification by pulverized rubber are given including potentials in increased strain resistance.

Keywords: Activated Rubber, Crumb Rubber Modified Bitumen, Dynamic Shear Modulus, MSCR Test, Dynamic Viscosity

I. INTRODUCTION

As stated in different studies elaborated worldwide, it is assumed that the yearly waste production of old tires reaches more than 110-115 mil. tires of different types and composition. This might represent more than 7,000 kT of recyclable rubber most of it coming from EU countries and North America [1]. In many countries, different regulations and legal standards have been set for waste management of old tires. In the developed countries, it has for several years forbidden putting of the old tires on landfills and other solutions are forced. If following the European waste management strategies, the most preferred solution is recycling and reuse, in the second step then is energy use. The areas which are for several decades understood as potential fields for crumb rubber utilization are asphalt pavements and suitable bitumen modifications.

Generally, two methods are known for crumb-rubber use in asphalt mixes: (1) dry process during which crumb rubber is added directly to the asphalt mixture as a flexible modifier and to substitute part of the finer aggregates and (2) wet process where the bitumen is modified. Materials described in this paper follow the second process. Nevertheless one of the crucial issues related to this product is the homogeneity of the final crumb rubber modified bitumen (CRmB). Of course it is possible to produce CRmB directly at an asphalt mixing plant and there are various solutions of continuous blending. The question always prevails if this is the most suitable solution guaranteeing high-performance ready-to-use binder. If CRmB is produced industrially in a refinery, quality control is better and final products properties are declared. In case of EU, the producer is further responsible for necessary steps related to European directive on Registration, Evaluation, Authorization and Restriction of Chemicals (REACH). The target is to get a binder which can be transported for longer distances and can be stored for several days. Homogeneous binder is required, which is usually not easy to achieve because of very strong chemical sulfur bonds in the rubber. Several approaches can be found worldwide, e.g. based on polyphosphoric acid or macrocyclic polymers, to improve the stability between bitumen and dissolved rubber particles. Usually the additive itself is not the overall solution and the composite material crumb rubber-bitumen-additive works only for limited rubber content.

Based on this knowledge, this paper focuses on two issues. (1) using a special type of disintegration technique for producing pulverized rubber with particles <0.8 mm including impact assessment of such rubber on bitumen performance and (2) analyzing several types of additives which might help to produce a storage stable product.

II. HIGH SPEED MILLING (GRINDING)

Different industrial field may presently exist without mechanical crushing or pulverization of materials, starting from the exploitation of natural resources, power generation and metallurgy to the paper industry or the production of building materials. The majority of industrial products as known today could not exist without grinding [2], which is understood as a process of refining the grain size and increasing the specific surface of material, but also as a process of opening particle grains [3].

One of the trends which has been going through intensive development in the recent time is high energy milling (HEM). High speed milling (HSM) is understood as a sub-type of HEM which is characterized by large amounts of energy transferred per material unit treated in the pulverizing process. The term of HEM or HSM still lacks precise definition in the literature. It shares all its basic aforementioned characteristics, i.e. refinement of the grain size, increase in the specific surface, opening of grains, etc., with milling in its traditional sense. But, unlike classical milling, certain phenomena occur in HEM/HSM. It is these effects into which some part of consumed energy is transformed and used, mainly: (1) mechanical-chemical activation; (2) production of higher rates of micro- and/or nano-particles; (3) higher efficiency of using consumed energy for the creation of new surfaces.

Mechanical-chemical activation is studied by a branch called mechanical chemistry, which may be considered as an interdisciplinary field of science. With some simplification, it deals with the initiation and enhancement of the efficiency of chemical and physical processes through mechanical effects [4].

III. ASSESSED CRUMB RUBBER MODIFIED BINDER VARIANTS

The assessment of the different experimental bituminous binders modified by crumb rubber (CRmB) involved an application of pulverized rubber of three grading (granularity) levels. At the same time, the effects of several catalysts were checked; these meant special organic solutions on an anhydrous basis, the varied chemical composition and pH levels. Catalyst K2 is neutral, i.e. with pH=7, catalysts K3 and K4 have a slightly acidic nature with pH=5. All three catalysts are based on a combination of methane $(CH2)_n$ and SO_2 compounds bond in a complex hydrocarbon chain. Simultaneously, an original Czech additive, Polyol was applied; it is a by-product of an innovative chemical recycling method for polyurethane. Last but not least, an additive commercially known as Vestenamer was used which is sufficiently well established from the ROAD+ technology. This additive is a mix of linear and macro-cyclical polymers, chemically termed trans-polyoctenamer (TOR). This was applied together with crumb rubber at a content not exceeding 5% and then mixed in the bitumen. Variations of experimentally tested crumb rubber modified binders further discussed in this paper are summarized in Table 1. The choice for the basic bituminous binder was the regular neat bitumen 50/70 with a defined interval for the softening point as 46-54°C and the penetration interval of 50-70 dmm.

Table 1: Summary of assessed crumb rubber modified bituminous binders

Bitumen variant	Additives	Pulverized rubber	Bitumen composition
CR-L7_2	-	15 %; 0,8 - 1,0mm	50/70 + 15% CR
CR-L7_2_K2 @150	Catalyst K2	15 %; 0,8 - 1,0mm	50/70 + 15%CR + 5%K2@150°C
CR-L7_2_K2 @170	Catalyst K2 @170°C	15 %; 0,8 - 1,0mm	50/70 + 15%CR + 5% K2 @170°C
CR-L7_2_K3 @150	Catalyst K3	15 %; 0,8 - 1,0mm	50/70 + 15%CR + 5%K3@150°C
CR-L7_2_K3_P	Catalyst K3 + Polyol	15 %; 0,8 - 1,0mm	50/70 + 15%CR + 5%K3 + 1%Polyol
CR-L7_2_K4 @150	Catalyst K4	15 %; 0,8 - 1,0mm	50/70 + 15%CR + 5%K4@150°C
CR-L7_2_K4 @170	Catalyst K4	15 %; 0,8 - 1,0mm	50/70 + 15%CR + 5%K4@170°C
CR-L7_2_K4_P	Catalyst K4 + Polyol	15 %; 0,8 - 1,0mm	50/70 + 15%CR + 5%K4 + 1%Polyol
CR-L7_2_V	Vestenamer	15 %; 0,8 - 1,0mm	50/70 + 15%CR + Vestenamer
CR-L8_2_K4 @150	Catalyst K4	15 %; 0,5 - 0,8mm	50/70 + 15%CR + 5%K4@150°C
CR-L9_2_K4 @150	Catalyst K4	15 %; 0,1 - 0,3mm	50/70 + 15%CR + 5%K4@150°C
CRmB_1	Catalyst K4 + Polyol	15 %; 0,1 - 0,3mm	50/70 + 15%CR + 5%K4 + 1%Polyol
CRmB_2	Catalyst K4	15 %; 0,1 - 0,3mm	50/70 + 15%CR + 5%K4
CRmB_3	Catalyst K3	15 %; 0,1 - 0,3mm	50/70 + 15%CR + 5%K3
CRmB_4	PPA	15 %; 0,1 - 0,3mm	50/70 + 15%CR + 1%PPA

IV. SELECTED TEST METHODS

Standard empirical procedures and performance based tests were selected to evaluate the impact of the pulverized rubber and used additives or catalysts. Assessed empirical characteristics are:

- softening point determination, ring and ball method (EN 1427);
- determination of needle penetration under 25°C (EN 1426);
- determination of elastic recovery under 25°C (EN 13397);
- storage stability test; 72±1 h and temperature of 180°C (EN 13399).

Performance based (functional) characteristics:

- determination of the complex shear modulus G* at 60°C and 40°C;
- frequency sweep for G* and δ with determination of the master curve for the reference temperature of 20°C;
- dynamic viscosity determination (EN 13302);
- multiple stress creep recovery test [1; 2].

This paper focuses on the analysis of performance based characteristics. The dynamic viscosity assessment is based on the degree of resistance to the stress caused under the selected angular velocity. Dynamic viscosity is a value of significance for the description of bitumen workability. From this perspective, samples of bituminous binders were compared as a standard under the temperatures of 135°C and 150°C with a measurement or conversion for shear rate 6.8 s^{-1}. Flow diagrams were assessed as well. A defined torsion stress was applied to the sample to obtain the relative resistance to spindle revolution. The measurement was taken under various test temperatures. The condition is important primarily with modified bituminous binders or in cases where bituminous binders are improved or modified by various additives. In accordance with the findings and recommendations of the U.S. SHRP program, measurements for distilled binders should be taken for the temperature of 135°C which is considered a suitable representative for the determination of workability level of the sample in question [5 ; 6]. The standard stipulates a rotational spindle viscometer as the measuring apparatus and ranges for the shear rate (1-104 s-1) and dynamic viscosity (10-2 – 103 Pa.s) under temperatures ranging from 40°C to 200°C. The temperature regulation equipment must be capable of regulation with the precision of ± 0.5°C.

The assessment of complex shear modulus G* and phase angle δ of bituminous binders using the dynamic shear rheometer (DSR) is governed by technical standard EN 14770. Simultaneously with the measurement of dynamic shear, the viscosity and elastic behavior of the binder can be examined under varying temperatures and frequencies which, together, cover a broad spectrum of possible conditions to which the bituminous binder might be exposed. The determination of G* and δ in the oscillation test is usually carried out for a temperature range of 20-100°C. A specific stress frequency or a pre-defined frequency spectrum is selected. To obtain relevant results, the linear area of visco-elastic behavior must be defined, i.e. in the regime where the test is conducted with controlled stress; the constant shear stress for the test must be specified. In this study, the previous findings of the CTU in Prague were used. The shear stress of τ=2,000 Pa is considered as safe and appropriate.

Additionally using the time-temperature superposition principle, values obtained from measurements under various temperatures and frequencies may be summarized (transposed) in a single characteristic known as master curve for the selected reference temperatures which, in the case of the results presented below, amounted to 20°C.

V. RESULTS AND DISCUSSION

5.1 Dynamic Viscosity

The dynamic viscosity test with a focus on 20 rpm (the reference velocity considered by the American standards) shows a positive impact of catalyst K4 under both 150°C and 135°C. It is obvious that particularly catalyst K3 significantly increases the dynamic viscosity value mainly under 135°C. The overall course of the viscosity (flow curve) in the thermal range of 100°C to 150°C demonstrates the best results for the option with catalyst K4 within the framework of CRmB binder comparison. Comparing the effect of rubber particle size, binders with rubber of 0.1-0.3 mm and 0.8-1.0 mm grading scored better. Even in this case, slightly illogically, the medium grading applied stands out. The overall course of viscosity in the range of 100°C to 150°C demonstrates poorer results for the option with 0.5-0.8 mm fraction applied.

Table 2: Dynamic Viscosity of Assessed CRmB Binders for Two Temperature Levels

Bitumen variant	Dynamic viscosity @ 6.8 s^{-1} (20 rpm) [Pa.s]	
	135°C	150°C
50/70 REF	0.59	0.30
CRmB_1	4.09	1.31
CRmB_2	5.54	1.15
CRmB_3	3.58	1.24
CRmB_4	3.58	2.42
CR-L7_2	4.09	1.54
CR-L7_2_K2 @150	5.54	1.28
CR-L7_2_K3 @150	7.10	1.74
CR-L7_2_K4 @150	3.05	1.00
CR-L8_2_K4 @150	13.75	4.50
CR-L9_2_K4 @150	2.06	1.03
CR-L7_2_K3_P	6.86	1.84
CR-L7_2_K4_P	2.90	1.21
CR-L7_2_V	5.30	3.40

If dynamic viscosity is assessed from the viewpoint effect of selected additives, the combination of catalyst K4 with Polyol scores better and also has good results in the entire thermal range. In contrast to the reference binder modified solely by pulverized rubber, the version with catalyst K3 and Polyol recorded the worst course of dynamic viscosity out of all of the versions compared. Nevertheless, TOR also has a negative effect on dynamic viscosity as has been proven within the experimental measurements at CTU also for other options of application of pulverized rubber and this additive.

From the data in Fig. 1 comparing binders CRmB_1 to CRmB_4, it is obvious that the CRmB_4 sample demonstrates higher dynamic viscosity values primarily under the higher temperature. With 135°C, the highest viscosity was detected for the binder with catalyst K4. This does not quite correspond with the flow curves indicated below where, in case of selected shear rates, the dynamic viscosity value of binder CRmB_4 is always higher. Contrastingly, the sample marked CRmB_3 seems to be the most suitable and catalyst K3 has the most positive effect on dynamic viscosity.

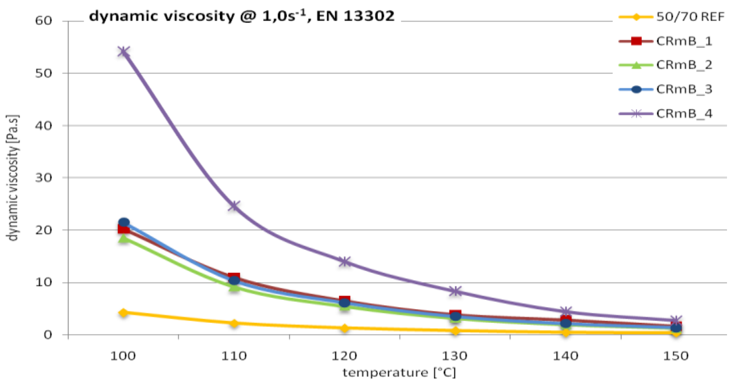

Figure 1: Viscosity curves for CRmB with pulverized rubber of 0.1-0.3 mm size; shear rate 1.0 s^{-1}

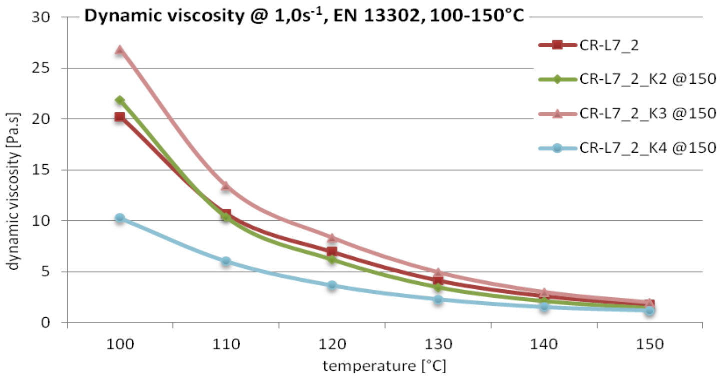

Figure 2: Viscosity curves for CRmB binders with different catalysts; shear rate 1.0 s^{-1}

From the point of view of comparing the dynamic viscosity determination, the course over the entire thermal range is important. For the selected shear rate of 1.0 s-1 the difference in dynamic viscosity values of the reference binder, CRmB_4 and the remaining versions assessed is quite obvious. This confirms the effect of the phosphoric acid as repetitively detected in the past and it results in higher dynamic viscosity values [7]. CRmB_4 achieves significantly higher viscosity; in contrast to that, the remaining samples have very similar courses as well as individual values. This is not quite supported by the course of the flow curve of binder CRmB_1 under lower shear rate 0.1 s-1. On the differences in dynamic viscosity in the interval of 100-120°C, the binder CRmB_4 remains within the range of 2-10 fold increase of the characteristic in relation to the reference asphalt 50/70 in case of the higher shear rate.

Comparing assessed catalysts used for improved CRmB stability, particularly a significant difference between the influence of catalysts K3 and K4 is obvious. At 130°C, the viscosity difference is double-fold, under 100°C the flow curve values differ even more. From the point of view of the above stated, the influence of catalyst K4 is either similar to low-viscosity additives or helps improve dissolution of the rubber particles in the CRmB binder composite. From the perspective of the course of dynamic viscosity, catalyst K2 appears neutral when compared to the no-catalyst CRmB version which showed no effect whatsoever.

The flow curves, if comparing chemical additives show basically identical courses for CRmB binder with catalyst 3 + Polyol and the binder with Vestenamer in relation for the shear slope selected. The two versions have approx. 1.5 times higher values, particularly in the interval of 100-120°C. The course of the curve for the binder containing catalyst 4 and Polyol is interesting; even under lower temperatures included in the assessment, the dynamic viscosity achieved is quite low when compared to the remaining samples. This can probably be attributed to the influence of the chemical additives applied; in comparison with the CRmB versions from the preceding group, Polyol probably has no influence.

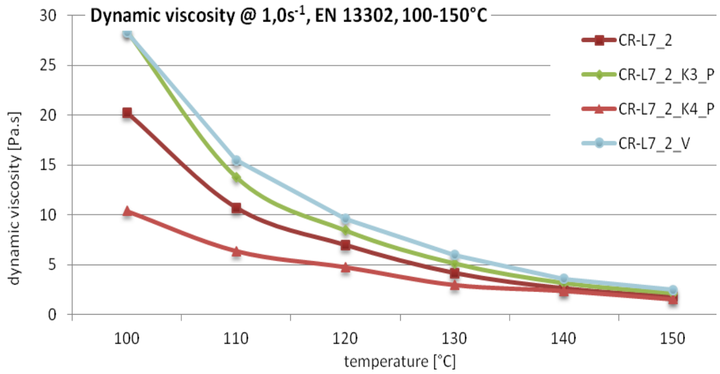

Figure 3: Viscosity curves for CRmB binders with selected additives; shear rate 1.0 s^{-1}

5.2 Dynamic Shear Modulus

Test methods which determine the complex shear modulus G* and phase angle δ using dynamic shear rheometer (DSR) allow performance assessment under medium and higher operation temperatures. The essence of the basic test is a parallel oscillating plate device allowing heating or cooling of the bitumen sample between the plates. Constant gap is always utilized (plates of Ø25.0 mm and gap of 1.0 mm or plates of Ø8.0 mm and gap of 2.0 mm). In the case of the results described below and the transformation of the data measured into a master curve, the measurements were taken for the temperature range of 60-20°C and frequency range of 0.1-10 Hz for each temperature measured, using the test procedure called the frequency sweep. The data obtained was subsequently used, applying the superposition of time and temperature principle, to generate the master curve for the complex shear modulus and phase angle depending on the frequency as an independent variable. The reference temperature to which the data for all temperatures was related and converted was set to 20°C.

The transformation of the data measured into the master curve practically provides an illustration of the relationship between stress and deformation by means of the complex shear modulus curve, given for a very broad frequency interval. It primarily describes the resistance to permanent deformation and defines the fatigue behavior of the material. In the first step four experimental CRmB binders were compared to the reference binder and, at the same time, the impact of chemical additives were assessed. The data obtained out the difference between the reference sample, CRmB_4 binder and the remaining three versions of rubber-modified bitumen which have practically identical courses of the master curves. It can be noted that from the point of view of the complex shear modulus, it is not determining whether catalyst 3 or 4 will be preferred; it is even obvious that the effect of Polyol equals zero from the perspective of this characteristic. In the case of the phase shift, it is quite obvious that the application of rubber in the bituminous binder improves the elastic properties within the entire frequency interval, i.e. it increases the elastic component of the bituminous binder. Options CRmB_4 seems the most resistant against fatigue characteristics and permanent deformation; it clearly demonstrates the additional benefit of phosphoric acid which probably causes a change of the chemical structures not only in the bituminous binder but also in the used rubber granulate. It achieves higher values of the complex shear modulus (primarily in the interval of 10-6 to 10-1 Hz) but, in the frequency range of 10-6 to 10-3 Hz, it also demonstrates a higher proportion of the elastic component than the other samples tested. Moreover, in the case of the complex shear modulus, it is obvious that the combination of ground rubber and organic acid reduces the thermal sensitivity of bituminous binders. In the case of the remaining three CRmB binders, this is slightly worse. However, it is obvious that this characteristic will be by far the worst for the reference binder.

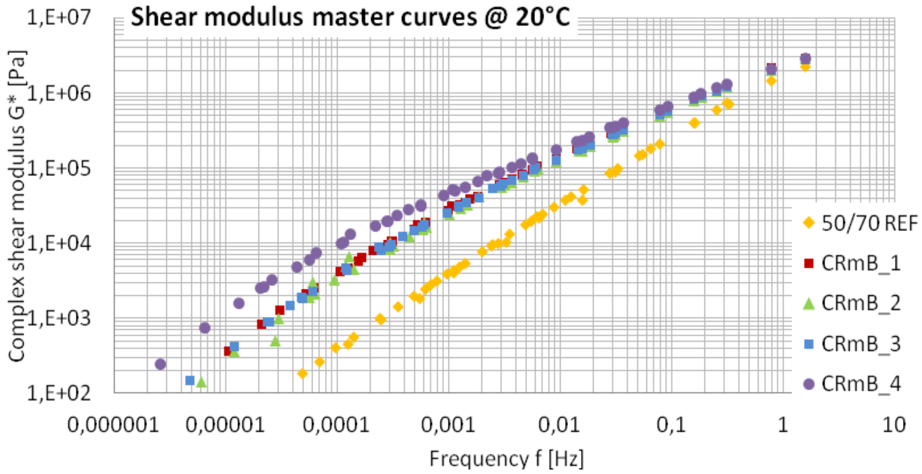

Figure 4: Master curves for CRmB binders with pulverized rubber of 0.1-0.3 mm size; ref. temperature 20°C

The G* values under the key temperatures (40°C and 60°C) and frequency of 1.59 Hz are greatly affected primarily when catalyst K3 was applied, where CRmB binder reached noticeably higher values of G* than the bituminous binders versions containing the remaining two catalysts or CRmB with no catalyst applied. In this context, it has to be noted that the three assessed catalysts achieve almost identical results.

The assessment of the master curves for the CRmB binders examined with the determination of the influence of the pulverised rubber grading shows that the best values of the complex shear modulus by far are achieved by CRmB with rubber of 0.5-0.8 mm grading; for small frequencies, the differences are adequate to 1-2 decimal orders. At the same time, it is evident that this version of CRmB scores lowest on thermal susceptibility. Within the framework of the assessment of the remaining two versions, the binders are similar with minimum impact of the grading of the pulverised rubber applied. With respect to the finest and coarsest granulate, it must be assessed why the medium-granularity achieved such different results. In this context, the question is whether this result could have been caused by the effects of the relevant catalyst on the rubber granulate.

Table 3. Complex shear modulus values for two temperature levels; f = 1.59 Hz, tau=2,000 Pa

Bitumen variant	60°C	40°C
	Complex shear modulus G* [Pa]	
CR-L7_2	3 309	66 742
CR-L7_2_K2@150	N/A	73 377
CR-L7_2_K3@150	22 926	131 374
CR-L7_2_K4@150	1 480	48 300
CR-L8_2_K4@150	81 200	555 000
CR-L9_2_K4@150	3 400	75 000
CR-L7_2_K3_P	12 292	99 943
CR-L7_2_K4_P	N/A	21 600
CR-L7_2_V	10 512	146 283

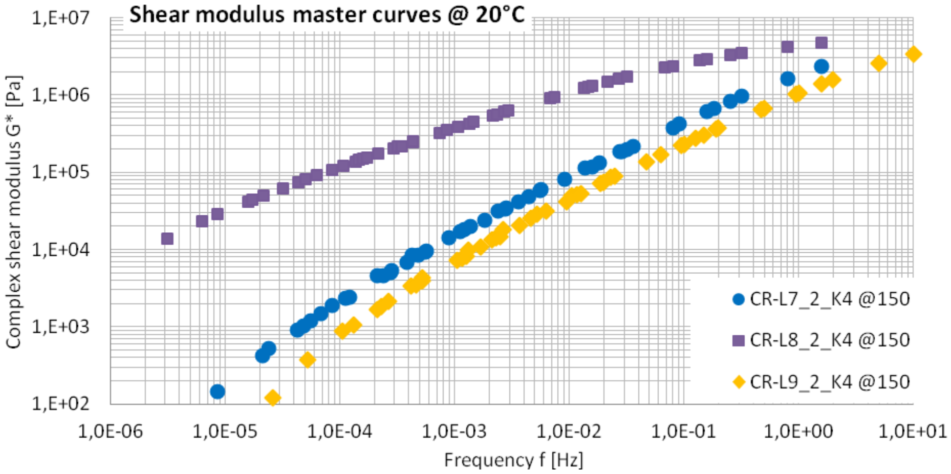

Figure 5: Master curves for CRmB binders with selected pulverized rubber variants; ref. temperature 20°C

The findings of G* assessment for selected temperatures and frequencies are also obvious in the case of the master curves prepared. The CRmB option with catalyst K3 records higher stiffness within the entire frequency interval. The values of all binders assessed subsequently even out in the highest frequency interval, i.e. under 20°C and the 1-10 Hz spectrum. Based on this CRmB, the binder with catalyst K3 will have the lowest thermal susceptibility value. In contrast to that, the course of the CRmB binder with catalyst K2 values in the smallest frequency interval (from 10-4 Hz) is interesting; the G* values decrease significantly.

If selected chemical additives are compared, the G* master curve has an analogous course for CRmB binders with catalyst K3 + Polyol and with Vestenamer alone. In both cases, higher modulus values are achieved; the binders should demonstrate improved resistance against deformation. This is reflected also in the courses of the phase angle master curve according to which the elastic component prevails in binders within the 10-6 to 10-1 interval. This is a quite important finding for the area of higher temperatures in particular. In the case of the last two binders, it is obvious that CRmB with catalyst K4 + Polyol has the least favorable course from the point of view of both G* and δ; it is likely to demonstrate the highest thermal susceptibility figures.

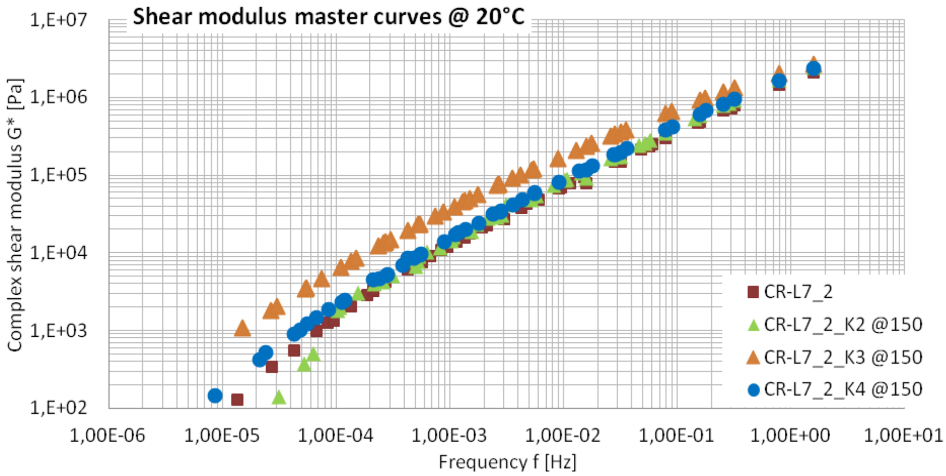

Figure 6: Master Curves for CRmB Binders with Different Catalysts; Ref. Temperature 20°C

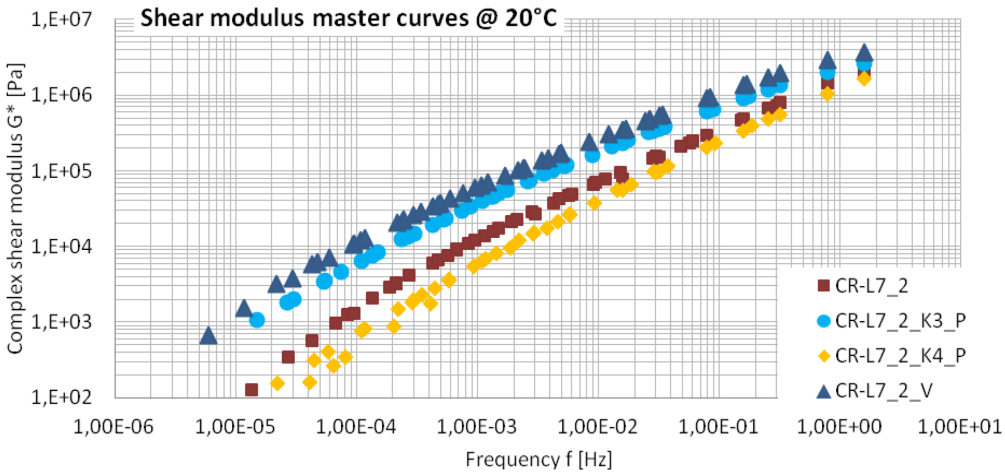

Figure 7: Master curves for CRmB binders with different chemical additives; ref. temperature 20°C

VI. CONCLUSION

The comparisons conducted and divided on the basis of the possible influences of multiple CRmB binder options reveal certain tendencies in some cases. This means particularly the effect of crumb rubber granularity, where it was rather surprisingly demonstrated that particularly 0.8-1.0 mm is suitable for use with respect to dynamic viscosity. Contrastingly, rheological properties are best affected by the 0.5-0.8 mm granularity, to a lesser degree even by the finer grading of 0.3-0.5 mm. However, in this case the results might be affected by the proportion of the particle size and the gap between oscillation plates in the test apparatus. Due to that, for instance the German technical regulations concerning CRmB binders stipulate a 2 mm gap even for the geometry of PP25 to eliminate any possible influence of not dissolved rubber particles.

The choice of a suitable catalyst is not absolutely clear. Catalyst K4 has a positive effect on viscosity and basic properties of bituminous binders; in contrast to that, catalyst K3 modifies elastic recovery and complex shear modulus. Catalyst K2 appears to be the most appropriate alternative to reduce the difference in softening points after the storage stability test. The effects of additives on selected properties demonstrate that the empirical properties and the course of viscosity are improved primarily by a combination of catalyst K4 with Polyol. The storage stability and the complex shear modulus master curve are positively affected particularly by Vestenamer which, nevertheless, results in increased dynamic viscosity in the CRmB versions assessed according to the measurements taken and thus it has negative impact on workability. Again, the storage stability has been most distinctively affected by catalyst K3, this time in combination with Polyol.

VII. ACKNOWLEDGEMENT

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