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Road and Rail Infrastructure III

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INFLUENCE OF CHEMICAL CATALYSTS AND SELECTED ADDITIVES ON BEHAVIOR OF CRUMB RUBBER MODIFIED BITUMEN

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Abstract

Since several years research in the field of crumb rubber modified bitumen (CRMB) is again an important area of interest. This fact is driven by restrictions for land-filling of old tires and their energetic use as a solid fuel e.g. in cement production. At the same time some experts expect that the use of higher content of crumb rubber in bitumen can further decrease the noise reducing potential if used in suitable acoustic asphalt mixes. Last but not least using crumb rubber as a modifier creates on the market binder which would fill the application gap between distilled bitumen and PMBs. Within the research done at Czech Technical University in Prague different types of micro-pulverized rubber has been used together with chemical catalysts suitable for temporary devulcanization of the rubber. Further for improving the homogeneity and storage stability of the CRMB additional additives have been tested and their effects compared. For the bitumen samples traditional as well as performance-based tests have been done. Within the traditional test softening point, penetration and elastic recovery test should be stressed out. For performance-based testing dynamic shear rheometer has been used for running frequency sweeps for temperatures between 20 and 60°C. At the same time multiple stress creep recovery test has been done for test temperature of 60°C. Dynamic viscosity has been assessed as well. Paper summarizes gained findings and recommendations made for most suitable combinations of bitumen – CR content – catalyst and additive.

Keywords: crumb rubber modified bitumen, pulverized rubber, chemical catalysts, penetration, complex shear modulus, storage stability, elastic recovery

1 Introduction

As stated in many reports and studies it is assumed that the yearly waste production of old tires worldwide reaches more than 110 mil. tires of different type and composition. This might represent more than 7,000 kT of used rubber most of it coming from EU countries and North America. In many regions and countries different regulations or legal standards have been set how to deal with the waste of old tires. In the developed countries it is since several years forbidden put the tires on landfills (in some parts of USA slashed tires can be land filled) and other solutions are preferred. If following the European waste management strategy the most preferred solution is recycling and reuse. One area, which is for several decades understood as a potential field of crumb rubber utilization, are asphalt pavements and especially modification of bituminous binders.

Generally two technical directions are know – dry process during which crumb rubber is added directly to the asphalt mixture as a modifier and substitute to part of the finer aggregates and wet process where the bitumen is modified. The focus of this paper follows the second process which might fill the gap between distilled bitumen and polymer modified bitumen

(in performance and price). Nevertheless one of the crucial issues related to this type of bitumen modification is the homogeneity of the final crumb rubber modified binder (CRmB). Of course it is possible to produce this type of bitumen directly on an asphalt mixing plant and there are various solutions of bitumen blenders which are applied for such production. The question always prevails if this is the most suitable solution. If CRmB is produced industrially in refinery or a bitumen manufacturing plant quality control should be better and producer has to declare the properties of final product. In case of EU producer is responsible also for all necessary steps related to REACH directive. The target then is to get a binder which can be transported for longer distances and ideally can be stored on a mixing plant for a few days. I.e. homogeneous storable bitumen is required, which is usually not easy to reach because of very strong sulphur bridges in the rubber. Several approaches can be found worldwide based e.g. on polyphosporic acid or macrocyclic polymers. Usually the additive itself is not the solutions and the composite material crumb rubber-bitumen-additive works only for limited rubber content. At the same time successful techniques applied in this area practically usually are not able to be used as binders for bituminous emulsion production because of undiluted rubber particles in the bitumen composite which would most probably clog the nozzles on a spraying bar.
Based on this knowledge this paper focuses on two issues. Using special type of disintegration technique for producing grinded rubber with particles ‹1.0 mm in different grading including the assessment of effect of such rubber on bitumen performance and analyzing selected types of catalysts and additives which might help to produce a storage stable product. The process of high speed grinding (disintegration) is described e.g. in [1].

2 Assessed crumb rubber modified binder variants

The assessment of the group of experimental bituminous binders modified by crumb rubber (CRmB) involved an application of finely pulverised 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 chemical composition and pH of which vary and are know-how protected. Catalyst K2 is neutral, i.e. with pH=7, catalyst K3 has a slightly acidic nature with pH=5. Both catalysts are based on a combination of methane, $(CH_2)_n$ and SO_2 compounds bond in a complex hydrocarbon chain. Simultaneously, an original Czech additive, Polyol was applied; according to the information available, this is a by-product of an innovative chemical recycling method for polyurethane. Last but not least, 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 is applied together with crumb rubber by 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 standard distilled bitumen, 50/70, for which standard ČSN EN 12591 defines the interval for the softening point as 46-54°C and the penetration interval of 50-70 dmm. Such basic criteria allow an approximate assessment of the effect of the combination of the crumb rubber and individual chemical additives applied on the individual characteristics. Simultaneously, the requirements stipulated by German regulations for standardised binders of the GmB (CRmB) type can be used where GmB 25/55-50 or 25/55-55 appears to be the most appropriate for further assessment. The threshold parameters are summarized in Table 2.

Table 1 Assessed experimentally designed CRmB variants

Bitumen variant	Additives	Pulverized rubber	Bitumen composition
CR-L7_2	–	15 %; 0,8 – 1,0 mm	50/70 + 15% CR
CR-L7_2_K2 @150	Catalyst K2	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + 5%K2@150°C
CR-L7_2_K2 @170	Catalyst K2 @170°C	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + 5% K2 @170°C
CR-L7_2_K3 @150	Catalyst K3	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + 5%K3@150°C
CR-L7_2_K3_P	Catalyst K3 + Polyol	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + 5%K3 + 1%Polyol
CR-L7_2_K4 @150	Catalyst K4	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + 5%K4@150°C
CR-L7_2_K4 @170	Catalyst K4	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + 5%K4@170°C
CR-L7_2_K4_P	Catalyst K4 + Polyol	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + 5%K4 + 1%Polyol
CR-L7_2_V	Vestenamer	15 %; 0,8 – 1,0 mm	50/70 + 15%CR + Vestenamer (4:100)
CR-L8_2_K4 @150	Catalyst K4	15 %; 0,5 – 0,8 mm	50/70 + 15%CR + 5%K4@150°C
CR-L9_2_K4 @150	Catalyst K4	15 %; 0,1 – 0,3 mm	50/70 + 15%CR + 5%K4@150°C

Table 2 Target values of German specifications for terminal blended CRmBs

Characteristic	Unit	GmB 25/55-50	GmB 25/55-55
Penetration	dmm	25-55	25-55
Softening point	°C	›50	›55
Elastic recovery	%	›50	›50
Complex shear modulus G* @ 60°C, 1,59 Hz, 2 mm gap	Pa	≥ 6000	≥ 8000
Phase angle δ @ 60°C, 1,59 Hz, 2 mm gap	°	‹65	‹65

3 Selected test methods

Standard empirical testing and test methods were selected to assess selected functional characteristics within the evaluation of the impact of the micro-pulverised rubber. Empirical characteristics:
- softening point determination by means of the 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).

Functional characteristics:
- determination of the complex shear modulus G* and phase angle δ under 60°C and under 40°C;
- frequency sweep for G* and δ with subsequent plotting of the master curve for the reference temperature of 20°C;
- dynamic viscosity determination (EN 13302).

The elastic recovery determination followed the EN 13397 technical standard. The principle of the test is extending the sample in a water bath under 25°C to the bitumen fibre length of 20.0 cm and subsequent observation of the sample shrinking for 30 minutes while the fibre is cut off at the beginning. The test provides the recovery value expressed as a percentage.
The measurement of dynamic viscosity is based on the degree of the sample's resistance to the stress caused under the selected angular velocity. A defined torsion stress is applied to the sample to obtain the relative resistance to spindle revolution. The measurement is taken under various test temperature. The condition is important primarily with modified bitumi-

nous 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. The standard stipulates a rotational spindle viscometer as the measuring apparatus and ranges for the shear rate ($1 – 10^4\ s^{-1}$) and dynamic viscosity ($10^{-2} – 10^3$ 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 determination 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 viscose and elastic behaviour of the binder can also be examined through the determination of the complex shear modulus and phase angles 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 behaviour 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 case, we used the previous findings of the CTU Faculty of Civil Engineering, for bituminous binders 50/70 and 70/100. The shear stress of τ=2000 Pa is considered safe and appropriate.
Additionally using the time-temperature superposition principle – TTS, the values obtained from measurements under various temperatures and load frequencies may be summarised (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.
Measurements taken under invariable shear stress within the interval of the selected stress frequency called the “frequency sweep“(FS) form a common test. The test is intended for verification of the bituminous binder behaviour under varying temperatures and frequencies, or stress durations. The test frequencies were chosen from a range of 0.1 – 10 Hz for the binders examined. According to the previous findings and conclusions of the U.S. SHRP, the value of 1.59 Hz is very important since, according to the Van der Poel’s nomograph, it corresponds with the shear effect of traffic load at 90 km/h, or load duration of 0.13 s.

4 Experimental results and discussion

The results are further divided into several logical groups which assess either the effect of the catalyst or the additive applied, or the influence of the pulverised rubber or the temperature chosen for the production of the CRmB binder.

4.1 Influence of chemical catalyst applied in pulverized rubber

Samples of experimentally prepared CRmB binders with the application of different catalysts were compared, namely versions K2, K3 and K4 to the quantity of 5 %-wt. together with pulverised rubber to the quantity of 15 %-wt. in the bitumen. The individual binder variants were prepared under processing temperature of 150°C. Bituminous binder 50/70 modified by 15 %-wt. of crumb rubber is used as the reference sample.
Out of the three catalysts tested, catalyst K4 affects the tested parameters best. Catalysts K2 and K3 cause slightly less significant decrease of the bituminous binder penetration where they fail to meet the requirements for 50/70; however, they meet the requirements for GmB binders. Analogously, from the perspective of the softening point, catalysts K2 and K3 result in higher values while the use of catalyst K4 in combination with 15 % rubber basically remains unchanged. All three binders meet the requirement for GmB 25/55-50. The option with catalyst

K2 achieved the best results in the storage stability test where the difference between the lower and upper softening point is 4.5°C. The elastic recovery results demonstrate a poorer effect of catalyst K4 where the value only amounts to about 12 %. In general, none of the binders assessed meets the requirement stipulated in the German regulations for GmB binders.
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 primarily 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.

Table 3 Empirical characteristics of assessed CRmB binders

Bitumen variant	Penetration [0.1 mm]	Softening point [°C]	PI [-]	Storage stability [°C]	Elastic recovery [%]
CR-L7_2	79	49.7	-1.0	9.9	8.8
CR-L7_2_K2 @150	49	57.4	-0.1	4.5	30.3
CR-L7_2_K3 @150	47	58.8	0.4	7.9	34.2
CR-L7_2_K4 @150	54	51.4	0.1	8.2	11.9

Table 4 Dyamic viscosity values of assessed CRmB binders

Bitumen variant	Dynamic viscosity @ 6.8 s^{-1} (20 rpm); [Pa·s]	
	135°C	150°C
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

The complex shear modulus values under the two temperatures chosen and frequency of 1.59 Hz are greatly affected primarily when catalyst K3 was applied, where CRmB binder reach noticeably higher values of G* than the bituminous binders versions with the remaining two catalysts or CRmB with no catalyst applied. In this context, we can note that the three versions achieve almost identical results.

Table 5 Complex shear modulus values for selected CRmB binders

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

From the perspective of the flow curves (see Figure 1), 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 – no effect whatsoever.

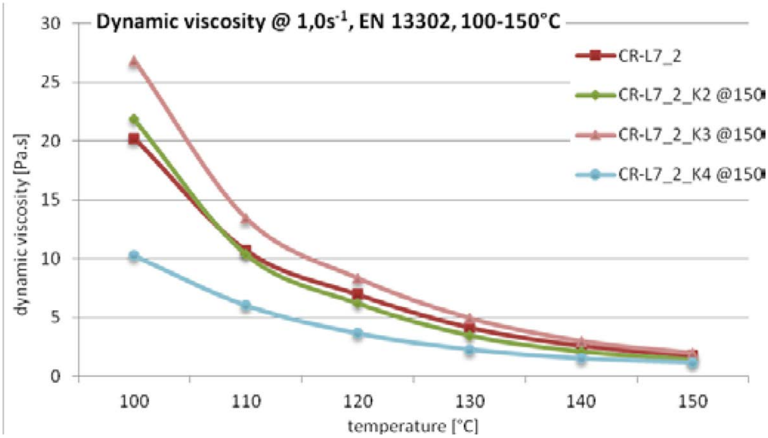

Figure 1 Flow (viscosity) curves for CRmBs with different catalysts

The aforementioned assessment of the complex shear modulus for selected temperatures and frequencies is also obvious in the case of the master curves prepared. The CRmB option with catalyst K3 records higher stiffness within the entire frequency interval – even if the conversion using the TTS principle is applied. 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, we could infer that the CRmB 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. This is supported by the phase angle course where the values fluctuate slightly. It should also be pointed out in relation to the course of the phase angle master curve that the only version of CRmB binder with catalyst K3 follows a non-standard course and reaches low values which indicate prevailing elastic behaviour in the interval of 10^{-5} – 10^{-1} Hz. The reason for such course is currently difficult to explain despite repeated measurement sessions. Such binder would nevertheless have excellent fatigue resistance.

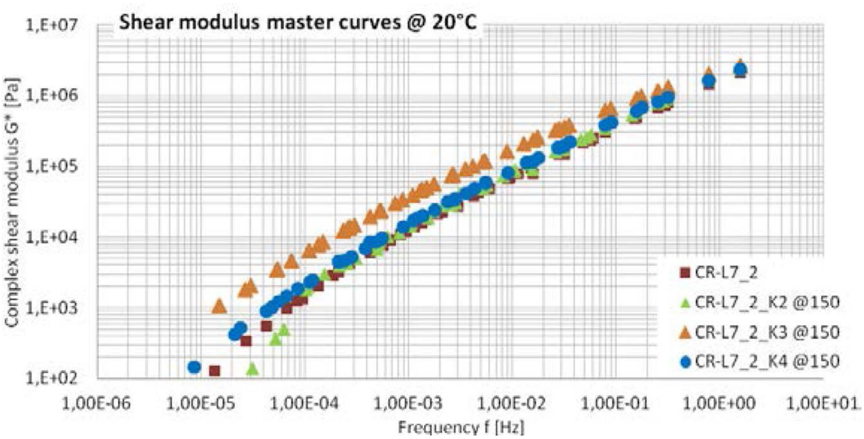

Figure 2 Complex shear modulus master curve for CRmBs with different catalysts

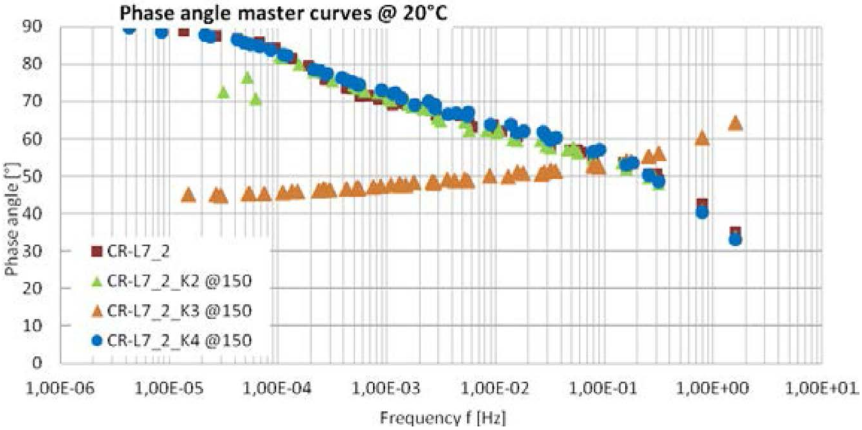

Figure 3 Phase angle master curve for CRmBs with different catalysts

4.2 Influence of pulverized rubber grading (granularity)

In this group of assessed binders, three levels of pulverised rubber grading with the same catalyst (K4) are compared; the catalyst is always applied to the same quantity of 5 %-wt. of pulverised rubber. The same distilled bituminous binder is used for all three CRmB variants; the quantities of pulverised rubber in the binder are identical, i.e. 15 % by mass. Again, CRmB with bitumen 50/70 with 15 %-wt. pulverised rubber of 0.8-1.0 mm grading is chosen as the reference binder.

Out of the three fractions of crumbed (pulverised) rubber applied, the binder with rubber of the 0.8-1.0 mm a 0.1-0.3 mm grading achieves the least effect on empirical properties. The 0.5-0.8 mm grading caused a significant decrease of CRmB binder penetration; in this case the option fails to meet the requirements for GmB 25/55-50(55). In the remaining two cases, the requirements from the perspective of both penetration and the softening point are met. Contrastingly, the noticeable increase of the elastic recovery value is interesting in the case of CRmB with 0.5-0.8 mm pulverised rubber. When the three gradings are compared no clear trend can be detected. From the perspective of the storage stability test, the option with the least grading (finest rubber) where the difference between the upper and lower softening points is 4.6°C (acceptable from the point of view of the requirements for polymer-modified bitumen) records the best results. The elastic recovery results point to an inferior effect of the 0.8-1.0 mm fraction.

The dynamic viscosity test focusing on 20 rpm shows that 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 thermal range of 100°C to 150°C demonstrates poorer results for the option with 0.5-0.8 mm fraction applied.

In the case of comparing the pulverised rubber grading effect on the complex shear modulus, the difference is obvious and the crumb rubber grading 0.5-0.8 mm achieves significantly better results. It should also be noted that for the options with rubber grading of 0.8-1.0 mm the application of catalyst K2 results in complex modulus value decreasing by 30-50 %. Contrastingly, the CRmB version with catalyst K4 and rubber grading of 0.1-0.3 mm achieves values that are basically comparable to the reference CR-L7_2 option.

Table 6 Empirical characteristics of assessed CRmB binders

Bitumen variant	Penetration [0.1 mm]	Softening point [°C]	PI [-]	Storage stability [°C]	Elastic recovery [%]
CR-L7_2	79	49.7	-0.1	9.9	8.8
CR-L7_2_K4 @150	54	51.4	-0.5	8.2	11.9
CR-L8_2_K4 @150	20	74.5	1.5	10.5	61.8
CR-L9_2_K4 @150	50	55.7	0.1	4.6	23.6

Table 7 Dyamic viscosity values of assessed CRmB binders

Bitumen variant	Dynamic viscosity @ 6.8 s^{-1} (20 rpm); [Pa·s]	
	135°C	150°C
CR-L7_2	4.09	1.54
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

Table 8 Complex shear modulus values for selected CRmB binders

Bitumen variant	60°C	40°C
	Complex shear modulus G* [Pa]	
CR-L7_2	3 309	66 742
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

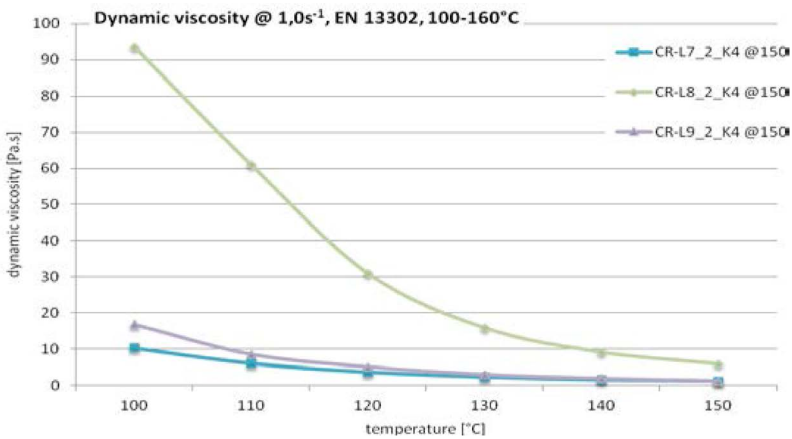

Figure 4 Flow (viscosity) curves for CRmBs with pulverized rubber of different grading

The assessment of the dynamic viscosity flow curve courses for the selected shear rates shows a significant difference between the binder with rubber of 0.5-0.8 mm grading and the remaining two versions. At 100°C the viscosity increases basically six-fold; nevertheless, even at 140°C the difference in viscosity is almost quadruple. Analogously to the complex shear modulus, the reason for the change in CRmB binder behaviour if this specific pulverised rubber fraction is applied and if all other conditions of bituminous binder sample preparation and heating remain the same is interesting and difficult to explain.

Similarly to the individual G* values determined for 40°C and 60°C, the master curve of CRmB with rubber of 0.5-0.8 mm grading reaches noticeably higher complex shear modulus values than the remaining fractions. In the field of very low frequencies (this corresponds with values of the complex shear modulus under higher temperatures), the difference even amounts to two orders. Only from the frequency of about 1.0 Hz the complex shear modulus values come closer together. At the same time, the curve for bituminous binders with rubber grading of 0.5 to 0.8 mm is flatter which means lower thermal susceptibility of the bituminous binder which can be expressed through the slope of the master curve. When the two remaining bituminous binders are compared it is obvious that from the perspective of the complex shear modulus and the expected resistance to deformation effects, the CRmB binder version with the finest pulverised rubber grading records the lowest values. Based on this comparison of the characteristic, very fine pulverisation unfortunately appears to be less appropriate. This is subsequently supported by the phase angle values where the master curve of the same binder reaches the highest δ values and, therefore, the viscose component of the complex shear modulus prevails over a great proportion of the frequency interval; in comparison to binder CR-L8_2 we can conclude on a lower fatigue life expectation. The aforementioned bituminous binder with pulverised rubber of 0.5-0.8 mm, in contrary, demonstrates a rather elastic character.

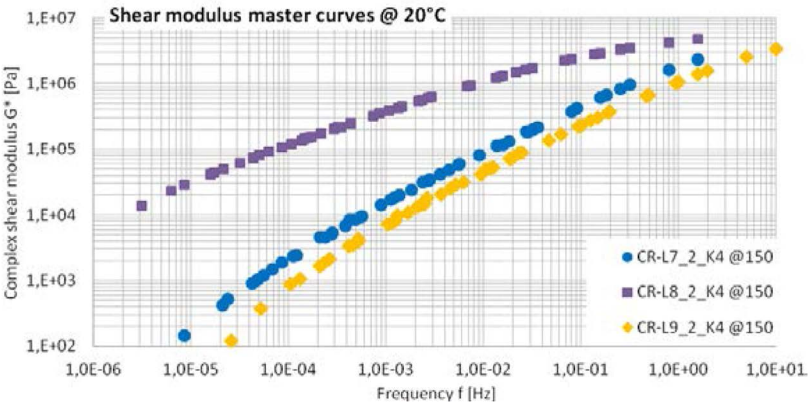

Figure 5 Complex shear modulus master curve for CRmBs with pulverized rubber of different grading

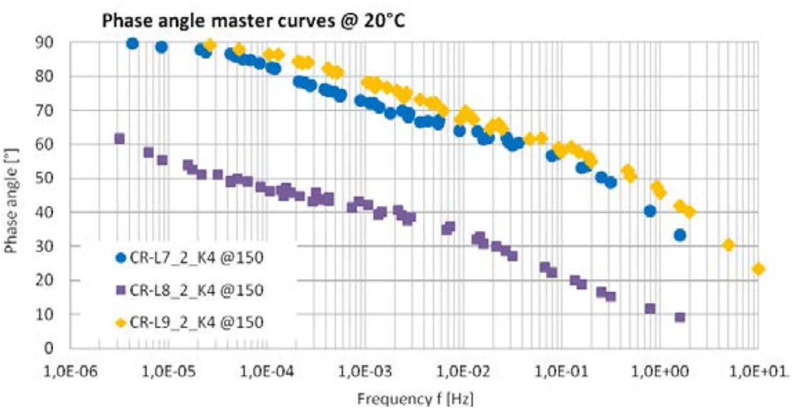

Figure 6 Phase angle master curve for CRmBs with pulverized rubber of different grading

The assessment of the course of master curves for the CRmB binders examined with the determination of the influence of the pulverised rubber grading, in accordance with the remaining results, it is obvious 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 binder 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.
In the case of phase angle analysis, the qualitative parameters of CRmB binders with rubber of 0.5-0.8 mm grading are confirmed. Within almost the whole frequency interval, the binder demonstrates prevailingly elastic behaviour which predetermines its very good resistance to fatigue and, simultaneously and in combination with higher G* values, high resistance to permanent deformations.

5 Conclusion

The comparisons conducted and divided on the basis of the possible influences and comparisons of multiple CRmB binder versions reveal certain tendencies in some cases. From the perspective of crumb rubber granularity, it was rather surprisingly demonstrated that particularly fraction 0.8-1.0 mm is suitable for use within the framework of determining dynamic viscosity and empirical properties. 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 it is possible that the results are affected by the proportion of the granulate grading and the gap between oscillation plates in the test apparatus utilised. Due to that, for instance the German technical regulations concerning GmB binders stipulate a 2 mm gap even for the geometry of PP25 to eliminate any possible influence of undissolved rubber granulate particles. Not even the choice of a suitable catalyst is 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.

Acknowledgments

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