Impact of selected WMA additives on normal PMB used for a standardized SMA

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Digital Object Identifier (DOI): dx.doi.org/10.14311/EE.2016.351

ABSTRACT

WMA techniques are well known for many years, even if still not increasingly popular in Europe. In the Czech Republic so far these techniques – mainly different types of wax based additives and surfactants – were used for asphalt concrete and usually in combination with a normal pen grade (70/100 or 50/70). As part of an ongoing research the focus was laid on SMA mixes which are as in any other country used mainly as a top wearing course for pavements with highest loadings and life-time expectations. Even if it is still allowed to use normal distilled bitumen, the best effect is achieved if PMBs are applied. For this reason a typical mix design for SMA11 mix was selected and more than 15 different binders have been used divided in three groups – two where effect of different WMA additives was analysed on PMB 25/55-55 of two selected origins and last group containing a more modified binder (not for WMA) and a 50/70 binder modified by PPA. As WMA additives different waxes have been used, selected surfactants and a nano-based additive.

Firstly all bituminous binders were analysed by empirical tests, dynamic viscosity, MSCR and frequency sweep test. Then the mixes were prepared with a temperature reduced by 20-25°C in comparison to reference mix, For all mixes voids content, stiffness, ITSR, rutting and crack propagation was analysed and will be summarized in the paper.

Keywords: Additives, Asphalt, Durability, Stiffness, Warm Asphalt Mixture

1. INTRODUCTION

The use of polymer-modified bituminous binders in SMA mixes in motorways and roads with high level of heavy loaded vehicles generates a number of advantages. The mixes are characterised by higher resistance to permanent deformation, good skid resistance and they also suppress the noise generated by traffic. On the other hand, SMA mixes with PMB require higher temperatures during mixing, paving and compaction in order to ensure sufficient coating of the aggregate with the bitumen.

In the Czech Republic and in other European countries, traditional compacted asphalt mixes constitute approximately 80-90 % of all pavements. Usual HMAs are designed, produced and paved under temperatures ranging from 150°C to 180°C (for mastic asphalts the temperature even exceeds 230°C). Such high temperatures ensure the necessary viscosity of the bituminous binder which facilitates good workability of the entire asphalt mix during paving and subsequent compaction to guarantee durability of the mix in the pavement structure during its life. The traditional technological methods are usually energy-intense and represent sources of greenhouse gas and other hydrocarbon compound and vapour emissions. Even though, according to the latest IARC study, the vapours have no carcinogenic effects they are still widely discussed and monitored. Lowering of the necessary working temperature reduces both the concentrations and the qualitative compositions of such vapours. Therefore, the production of WMA needs suitable additives (synthetic waxes, fatty acid amides, surfactants etc.) which will provide the necessary viscosity and sufficient adhesion between the aggregate and the binder despite a lower production and paving temperature. The WMA types of asphalt mixes are suitable particularly from the environmental perspectives (lower power consumption due to reduced temperature during both production and paving, reduced CO_2 and odour production in mixing plants, easier recycling of reclaimed asphalt etc.); they provide slower aging of the bitumen and the paving itself can be completed even at of lower temperatures.

2. OBJECTIVES

The primary objective of this paper is comparing the impact of several additives for WMA on two different PMB binders labelled 25/55-55. The comparison involves both empirical and functional testing of bituminous binders, as well as a comparison of the impacts of individual bituminous binders on the selected standardised mix SMA 11+ with mixing temperature reduced by 20-25°C in relation to the reference (control) mix.

Semi-warm or warm asphalt mixes represent an established standard in road building practice nowadays. Unfortunately, the Czech Republic along with other European countries still fails to use, or even fully appreciate, the potential of such mixes. The causes can be found in many areas, starting with insufficient technical expertise, lack of trust in novel technical solutions, evaluation of any and all construction activities solely through the lowest price prism to the lack of importance such solutions bear to some public investors. In the latter case, we can repeatedly encounter the opinion that working temperature reductions are not important for public investors at all. If we disregard social responsibility that every entity in the industry has to a certain degree, such an opinion can be justifiable, particularly if the cold mixes deliver no other added value – which might be more event quality of paving or compaction, if the working temperature reduction is not exploited to the full, or the possibility of processing reclaimed material in the asphalt mix without the need for excessive heating (when the required workability has been reached). Of course, other problems might be encountered in this area; however, these concern the production limits of mixing plants rather than road building, particularly due to the existence of the so-called “dew point” and the risk of condensation and clogging especially in the mixing plant filter areas. However, these cannot act as arguments that should result in a lack of support for semi-warm or warm asphalt mix development. Ultimately, energy always has to be generated. The generation has used primarily non-renewable sources, which are however not endless, not to mention the charged issue of greenhouse gas emissions.

3. SCOPE OF EXPERIEMNTAL ASSESSMENT OF PMB BINDERS

Based on the findings obtained within the framework of research centre CESTI activities in 2013 when 50/70 and 70/100 bituminous binders were assessed in combination with various additives to reduce viscosity or improve surface activity, the attention shifted to PMB binders in 2014. The assessment of the effects of various additives and applied solutions appropriate for the production of warm asphalt mixes involved a comparison of different types of polymer-modified binders, i.e. besides the different types characterised by different classification based on penetration and softening point, this included a comparison of identical types of PMBs by two different manufacturers. A set of alternatives with various types of additives (either synthetic waxes – FTP, RH, or nanochemical additives based on silanes or surfactants based on amides or various tenside chemical groups) were selected for use in the binders. For the sake of a clearer presentation of the results, the individual options and PMBs assessed are divided into two logical sets.

Table 1: Assessed bituminous binders of two analyzed Gross of variants

Bitumen	ID	Bitumen	ID
PMB 25/55-55	PMB 25/55-55	**PMB 45E**	PMB 45E
PMB 25/55 + FTP	3% FT paraffin	**PMB 45E + FTP**	3% FT paraffin
PMB 25/55 + RH	2% RH	**PMB 45E + RH**	2% RH
PMB 25/55 + Zycotherm	0.1% Zycotherm	**PMB 45E + Zycotherm**	0.1% Zycotherm
PMB 25/55 + Rediset	0.5% Rediset	**PMB 45E + Rediset**	0.5% Rediset
PMB 25/55 + CECA	0.5% CECA	**PMB 45E + CECA**	0.5% CECA
PMB 25/55 + Evotherm	0.5% Evotherm	**PMB 45E + Evotherm**	0.5% Evotherm

For the first set, a standard polymer-modified bitumen by a foreign manufacturer was selected (PMB 25/55-55) and further combined with six different additives. The second set is an identical type of binder supplied by a different manufacturer and the same additives are tested. The comparison from the point of view of standard requirements is based on the specifications stipulated by the standard ČSN EN 14023 (Bitumen and bituminous binders – Specification framework for polymer modified bitumens) for individual types of PMBs. The aforementioned list only includes the characteristics required by the standard which were measured for the bituminous binders in question. Oscillatory DSR test and MSCR test were performed to verify the functional characteristics. The standard is not being harmonised at the moment and, therefore, it stipulates no minimum requirements.

The third set consisted of several types of PMBs according to EN 14023. The set also included a 50/70 binder with PPA. Nevertheless these results are not included in this paper.

3.1 Test methods applied and characteristics observed for the PMB variants tested

From the point of view of laboratory testing performed, both standard empirical characteristics and a wholesome set of functional tests were chosen. The empirical test framework determined:

- The softening point by the ring-and-ball method (EN 1427);
- Needle penetration at 25°C (EN 1426);
- Tensile properties of bituminous binders by the force ductility method (EN 13589);
- Elastic recovery at 25°C (EN 13398).

The functional tests represent the following set of measurements:

- Determination of complex shear modulus G* and phase angle δ under 60°C and 40°C (EN 14770) – dynamic shear rheometer (DSR);
- frequency sweep for G* and δ with a subsequent plotting of the master curve for the chosen reference temperature 20°C and temperature interval of 20-60°C;
- Multiple-stress creep-recovery test (MSCR) according to the American methodology AASHTO 70-09;
- Determination of dynamic viscosity (EN 13302).

4. RESULTS OF THE PMB VARIANTS ASSESSED

4.1 Empirical tests

Similarly to the chosen test sets, the results are divided into empirical and functional tests for the sake of clarity. In individual cases, the aforementioned grouping of samples of bituminous binders is assessed. The basic empirical characteristics, including elastic recovery, are summarised in table 2 and in figures 1-2.

A joint comparison of the penetration and softening points of the first and second sets of bituminous binders under assessment, we can stipulate the following conclusions. From the point of view of penetration, both reference binders are identical; in the case of the softening point, PMB 45E demonstrated a higher value – according to the standard specifications, it would basically meet the requirements for a PMB 25/55-55, if such a category were established in the standard. For this binder, the plasticity range has been extended; it can be expected that the bitumen has improved resistance to deformation under higher temperatures. This is also reflected by the penetration index value which suggests a lower thermal susceptibility.

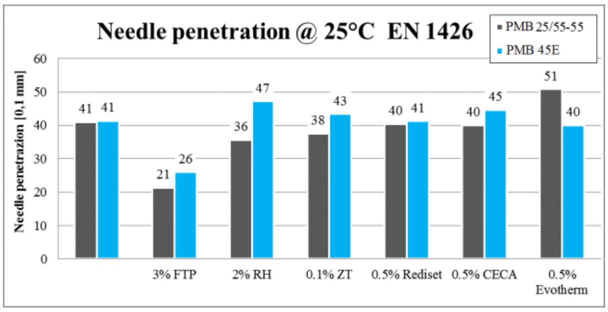

Figure 1: Needle penetration of PMBs from set 1 and set 2

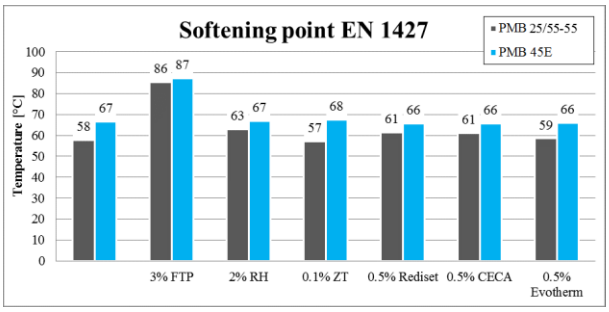

Figure 2: Softening point "ring&ball" of PMBs from set 1 and 2

From the perspective of additives applied to reduce viscosity or improve surface activity, we can conclude that the additives Rediset LQ, Zycotherm and CECA have no impact on penetration. This basically applies also from the point of view of the softening point where a slight increase is demonstrated solely by PMBs 25/55-55 with additives Rediset and CECA. Synthetic waxes have a different behavioural pattern. While FTP traditionally reduces penetration (in the case of the variants assessed almost by one half), RH causes just a slight decrease in penetration in one case; however, in the other case, it quite illogically increases penetration with a difference of 6 penetration units. With respect to the softening point, the finding for FTP reconfirms the standard expectations with a significant increase in the property which results in a substantial increase in the penetration index itself (the binder is expected to have improved resistance to permanent deformation and lower thermal susceptibility). For RH additive, the softening point results are as baffling as the penetration results. In the case of PMB 25/55-55, the increased softening point when compared to the reference binder is logical and expected since the penetration value was slightly lower; however, the softening point remains virtually unchanged for PMB 45E. The result of both the characteristics for the variants with Evotherm is similarly ambiguous. The overall higher values of penetration index for the individual binder variants with PMB 45E correspond with a higher penetration index of the binder itself.

Table 2: Results of basic empirical tests for assessed PMBs

Bitmunious Binder	Penetration [0.1 mm]	Softening point R&B [°C]	Penetration index [-]	Elastic recovery [%]
PMB 25/55-55	41	57,8	0,1	74,9
PMB 25/55 + FTP	21	85,4	3,0	55,2
PMB 25/55 + RH	36	62,8	0,8	72,3
PMB 25/55 + Zycotherm	38	57,0	-0,2	74,1
PMB 25/55 + Rediset	40	61,2	0,8	73,6
PMB 25/55 + CECA	40	61,2	0,7	74,3
PMB 25/55 + Evotherm	51	58,4	0,8	74,6
PMB 45E	41	66,6	1,8	81,8
PMB 45E + FTP	26	87,2	3,7	70,0
PMB 45E + RH	47	66,8	2,2	79,3
PMB 45E + Zycotherm	43	67,4	2,1	83,1
PMB 45E + Rediset	41	65,8	1,6	81,8
PMB 45E + CECA	45	65,6	1,8	82,3
PMB 45E + Evotherm	40	66,0	1,6	81,3

Elastic recovery represents a quick, simple test where the result can be used as an indicator of elasticity level of the modified bituminous binder. The values obtained for the individual PMB versions assessed are plotted in a separate chart (Fig. 3). From the perspective of assessing both PMB sets, it is obvious that elastic recovery is better in all cases for variants with PMB 45E, where values > 80 % were achieved. The values of the variants with PMB 25/55-55 are 9 % lower on average. With the exception of additive FTP, the remaining additives have no impact on the resulting elastic recovery. When FT paraffin is applied, elastic recovery decreases by 15-25 % while a higher reduction was recorded in combination with PMB 25/55-55.

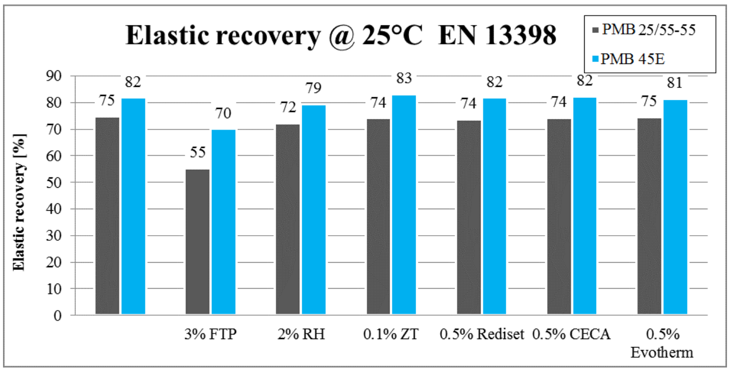

Figure 3: Elastic recovery of PMB binders of set 1 and 2

The long-established test for PMBs is the force ductility method where a bitumen fibre is stressed to elongate to 400 mm under the stipulated temperature. In this regard, we should emphasise that the temperature under which the test took place was not identical for all binders tested. The tests have been started with the temperature of 5°C; however, if two test samples failed before reaching 400 mm, the water bath temperature was increased by 5°C. The resulting values (indicated in Table 3) represent the median of at least two valid measurements; the temperature under which the condition was met is stated for each binder variant assessed. The individual columns present the values of deformation energy (E_s = deformation energy when the maximum force F_s is reached; $E_{0,2}$ = deformation energy upon elongation to 200 mm; $E_{0,4}$ = deformation energy upon elongation to 400 mm and $E_{2\text{-}4}$ = deformation energy attributed to the elongation interval between 200 mm and 400 mm). The deformation energy E_s can also be defined as the bituminous binder energy that affects its resistance to cracking. The higher the energy the better the probability that the bituminous binder will improve resistance to deformation. In this regard, the highest values were achieved by PMB 25/55-55, both variants with additive CECA and, basically, also both variants with additive Evotherm MA3. This is also reflected in the highest force value, F_s. Moreover, the binders were the only ones that allowed testing under 5°C, i.e. they are characterised by very good behaviour under increased temperatures of 10°C where most of the remaining binders were tested. The only case where the measurement had to be taken under 15°C was the option PMB 25/55-55 + FTP. From the perspective of $E_{2\text{-}4}$, the deformation values correspond with the above; it is obvious that for example Zycotherm causes a slight decrease in the resulting deformation energy.

Table 3: Results of force ductility test for assessed binders

Bituminous binder	**Force**	**Deformation energy**				**Temp.**
	F_s	**E_s**	**$E_{0,2}$**	**$E_{0,4}$**	**$E_{2\text{-}4}$**	T
	[N]	**[J]**	**[J]**	**[J]**	**[J]**	**[°C]**
PMB 25/55-55	108,70	0,42	9,76	17,65	7,88	5
PMB 25/55-55 + FTP	78,00	0,30	6,22	8,67	2,45	15
PMB 25/55-55 + RH	56,10	0,22	5,10	9,17	4,07	10
PMB 25/55-55 + Zycotherm	51,00	0,20	5,21	8,89	3,67	10
PMB 25/55-55 + Rediset	57,73	0,22	5,65	10,03	4,38	10
PMB 25/55-55 + CECA	113,87	0,45	9,99	18,15	8,15	5
PMB 25/55-55 + Evotherm	113,67	0,44	9,96	18,05	8,09	5
PMB 45E	46,90	0,18	5,42	10,85	5,43	10
PMB 45E + FTP	82,53	0,33	7,37	13,08	5,71	10
PMB 45E + RH	49,00	0,20	5,23	10,58	5,35	10
PMB 45E + Zycotherm	50,57	0,20	5,58	11,14	5,56	10
PMB 45E + Rediset	52,53	0,21	5,63	11,09	5,47	10
PMB 45E + CECA	102,57	0,42	10,22	20,09	9,87	5
PMB 45E + Evotherm	93,80	0,38	9,73	19,29	9,56	5

The course of the force ductility diagram indicated in Figures 4 a 5 clearly shows that there are more substantial force decreases with increasing elongation in binders with FTP. Significant differences in the curves plotted occur due to the different temperatures under which the measurements were taken. For a sample with FTP in the first set, a high tensile force was measured at the beginning of elongation although this test was the only one measured at 15°C. With lower temperatures, the sample with FTP failed before reaching the stipulated elongation; this indicates poor ductile properties which emphasise the need for proper verification of the binder, and subsequently the asphalt mix behaviour, within the low temperature range. Similarly high tensile force values are indicated for the FTP sample in set 2, too.

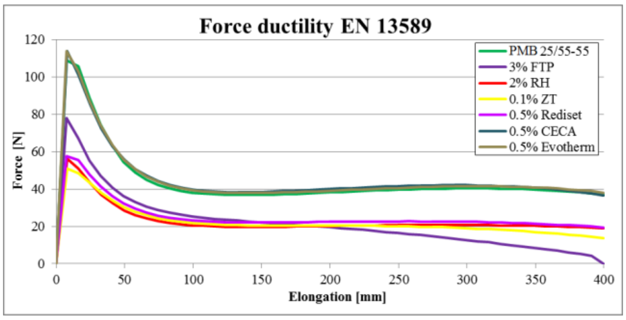

Figure 4: Diagram of force ductility for PMB binders – set 1

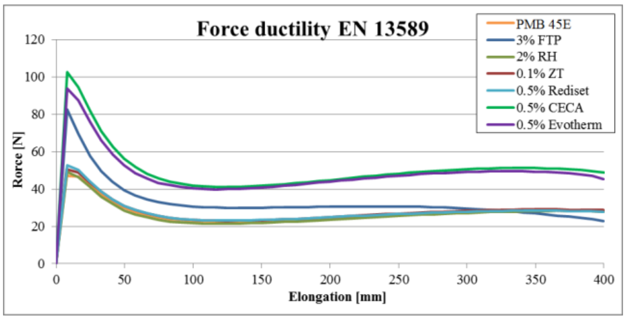

Figure 5: Diagram of force ductility for PMB binders – set 2

4.2 Dynamic viscosity

Dynamic viscosity is an important value for the purposes of describing the bitumen workability. The characteristic is also important from the perspective of bitumen transport and handling e.g. at the mixing plant. Based on this reason, bitumen samples are compared under at least 135°C and 150°C to the measurement or conversion completed for shear rate of 6.8 s^{-1}. Measurements are also taken for other shear rates and for the thermal range of 100-150°C which permits displaying the viscosity flow curves. The results of dynamic viscosity evaluation are presented in the following table and figures. The dynamic viscosity test defined for shear rate of 6.8 s^{-1} (standardized shear rate considered according to the American standards, as a reference parameter to determine bitumen workability in the mix) was as expected (see Table 4), the lowest value being demonstrated by the variants of binders with FTP (in the case of PMB 25/55-55, a reduction in viscosity by min. 35 %, in the case of PMB 45E, by at least 22 %). Again, this demonstrated the impact of this WMA additive which has been used for a number of years in the field of warm mix asphalts due to this very reason. The effects of the remaining additives are not too significant. Interest dynamic viscosity values were recorded by PMBs with additive RH which should work similarly to FT paraffin. In the case of PMB 25/55-55, viscosity increased slightly in comparison to the reference binder; this can be considered a not-quite-credible result. Contrastingly, the application with PMB 45E recorded viscosity reduced to a level comparable to additive FTP.

Table 4: Dynamic viscosity values at 6.8 s^{-1} (20 rpm) shear rate

Bitumen	**Dynamic viscosity @ T**	
	135°C	**150°C**
	[Pa.s]	[Pa.s]
PMB 25/55-55	1,20	0,87
PMB 25/55-55 + FTP	0,80	0,51
PMB 25/55-55 + RH	1,23	1,10
PMB 25/55-55 + Zycotherm	1,16	0,78
PMB 25/55-55 + Rediset	1,19	0,79
PMB 25/55-55 + CECA	1,19	0,74
PMB 25/55-55 + Evotherm	1,14	0,82
PMB 45E	1,33	0,93
PMB 45E + FTP	1,04	0,71
PMB 45E + RH	1,01	0,68
PMB 45E + Zycotherm	1,29	0,79
PMB 45E + Rediset	1,25	0,78
PMB 45E + CECA	1,20	0,83
PMB 45E + Evotherm	1,18	0,78

4.3 Oscillation test and complex shear modulus determination

Functional tests constitute a general trend that allows better and more complex predictions of bitumen or asphalt mix behaviour. In the field of bituminous binders, rheological properties under medium and high operation temperatures can be determined with the help of a broad range of test methods that determine the complex shear modulus G*, phase angle δ or dynamic viscosity and shear compliance of the material using the dynamic shear rheometer (DSR) for thermal ranges reaching from -30°C to 200°C. Narrower intervals, like 20-60°C, are usually selected in relation to medium or higher temperatures occurring on a road during operation.

A common oscillatory test is taken under constant shear rate (selected value τ=2000 Pa) in the interval of selected frequencies that are called "frequency sweep" (FS). The test is intended to verify the behaviour of bituminous binders under various temperatures and frequencies, or load duration, which might simulate varying compositions and speeds of the multiple stresses in action as subsequently encountered in the pavement. For the binders concerned, the test frequencies were chosen within the range of 0.1 – 10 Hz. Based on earlier findings and conclusions of the U.S. SHRP, the value of 1.59 Hz bears high significance (according to the Van der Poel nomogram, it roughly corresponds with the shear effect of traffic load at 90 km/h, or load duration of 0.13 s).

The resulting main characteristic of the complex shear modulus describes, in particular, resistance to permanent deformation, and defines the fatigue behaviour of the material. The measurement of dynamic shear establishes, besides the complex value, also the viscous and elastic behaviours of the bitumen based on the phase angle under varying temperatures and frequencies; this covers a broad range of possible conditions to which the bitumen is exposed. In the case of a subsequent data transformation, for instance for a specific temperature as selected, the time and temperature superposition principle allows obtaining a master curve.

The following table summarises the values of complex shear modulus G*, phase angle δ and characteristics G*/sin(δ) or G*xsin(δ) which have been defined in the past as the functional qualitative indicators within the framework of the SHRP research program. The values are indicated for test temperatures 40°C and 60°C. The measurements were taken in the plate to plate geometry (PP25) with a 1 mm gap.

Table 5: Complex shear modulus values for 40°C and 60°C

Bitumen	**1.59 Hz @ 40°C**				**1.59 Hz @ 60°C**			
	G*	**δ**	**G*/sin(δ)**	**G*x sin(δ)**	**G***	**δ**	**G*/sin(δ)**	**G*x sin(δ)**
	[kPa]	[°]	[kPa]	[kPa]	[kPa]	[°]	[kPa]	[kPa]
PMB 25/55-55	93,8	66,6	102,2	86,1	9,5	71,1	10,0	9,0
PMB 25/55-55 + FTP	351,0	54,4	431,7	285,4	29,8	64,8	32,9	27,0
PMB 25/55-55 + RH	104,0	66,8	113,1	95,6	10,2	71,4	10,8	9,7
PMB 25/55-55 + Zycotherm	86,2	66,3	94,1	78,9	8,6	70,7	9,1	8,1
PMB 25/55-55 + Rediset	95,5	65,5	104,9	86,9	8,9	70,6	9,4	8,4
PMB 25/55-55 + CECA	101,2	65,1	111,5	91,8	9,9	70,0	10,5	9,3
PMB 25/55-55 + Evotherm	95,8	66,0	104,8	87,5	8,8	71,2	9,3	8,3
PMB 45E	131,0	62,7	147,4	116,4	13,4	66,6	14,6	12,2
PMB 45E + FTP	160,0	60,9	183,1	139,8	14,8	65,8	16,2	13,5
PMB 45E + RH	67,6	66,0	74,0	61,7	7,3	68,6	7,9	6,8
PMB 45E + Zycotherm	83,6	65,3	92,0	75,9	8,4	68,1	9,1	7,8
PMB 45E + Rediset	90,5	65,0	99,9	82,0	9,1	68,1	9,8	8,4
PMB 45E + CECA	104,9	65,0	115,7	95,0	10,4	68,9	11,1	9,7
PMB 45E + Evotherm	99,0	64,8	109,4	89,5	8,9	68,3	9,5	8,2

From the point of view of evaluation, oscillation dynamic measurements can be divided into two approaches of the analysis performed. In the first case, the shear modulus and phase angle values were assessed for the frequency of 1.59 Hz and for two selected temperatures. The results obtained are summarised in Table 5. Complex shear modulus G* indicates the overall material resistance to deformation. The highest values of G* and lowest values of δ are achieved by binders with additive FTP; therefore, they appear to be more elastic and better resistant to permanent deformation under higher temperatures. In the case of PMB 25/55-55 with 3% FTP, the result can be considered a bit extreme, particularly when compared to the proportion established in the other case where binder PMB 45E is applied. What is baffling – see some of the results presented before – is the effect of additive RH which, in the case of set 1, results in a slight increase of G* while the phase angle is maintained; contrastingly, for set 2, the variant with RH scores the worst and basically records a 50 per cent decrease in G*. Similarly, the impact of the remaining additives in both sets is quite surprising; while in set 1, the remaining additives neither improve nor deteriorate the values of G* and the value of angle δ remains basically unchanged, in set 2 all of the remaining additives result in a lower G* and a slightly higher δ value – as if the application of the additive had a negative impact on the stiffness while the binder loses a small proportion of its elasticity at the same time. Theoretically, such different behaviours of the additives are certainly possible; however, common knowledge of rheology or mechanics fails to explain it for this type of material; moreover, it is likely not to be related to these aspects and an explanation must be searched within the chemical composition and, possibly, different effect and co-working of the binder additives which, despite being of the same classification, are based on different input materials and use elastomers of different origins.

The second approach selected assumes oscillation frequency sweeps to be completed within the thermal range of 20-60°C with frequencies from the interval of 0.1-10 Hz. Subsequently, the aforementioned principle of temperature and time superposition is applied and all values measured are related to the selected temperature; in the case of the results presented, this was 20°C (Fig. 6-9). This fact allows a good assessment of the deformation effects of individual binders within a broad frequency range which gives us an opportunity to interpret various impacts of traffic loads and intensities affecting the materials in the pavement structure. The graphic representation used in this case is called the master curve. Alternatively, the data can be represented using the Black diagram which applies a semi-logarithmic scale to plot the dependence of the phase angle and complex shear modulus.

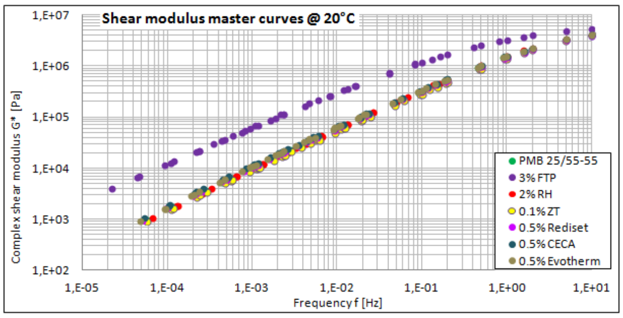

Figure 6: Master curves of complex shear modulus for PMBs of set 1

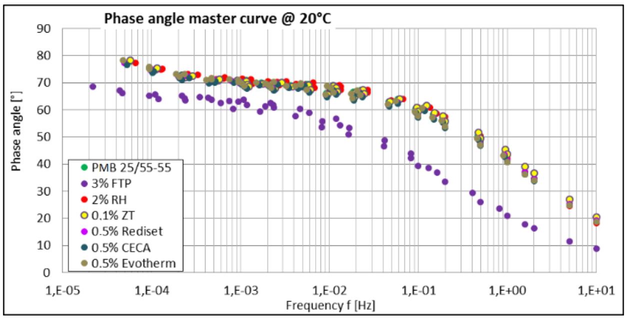

Figure 7: Master curves of phase angle for PMBs of set 1

The master curves also provide values for the frequencies outside the functional scope of the device. However, the behaviour of the binder can be expressed in a single diagram for a broad range of possible loadings of the bitumen in the pavement. In the case of set 1 of the binders assessed, Fig. 6 clearly suggests that the binder with the FTP additive achieves a significantly higher value of G*; it indicates a higher stiffness of the binder within the entire frequency range. This potential of the binder has been visible in the previous result already; the master curve confirms its validity. If the curves were accompanied by tangents and the slope thereof used as a thermal susceptibility indicator, the PMB variant with FTP additive still demonstrates slightly superior behaviour. The aforementioned results, particularly concerning the improved resistance to deformation, also correspond with the results obtained from the empirical tests. The remaining binders in set 1 have no fundamental differences from the basic binder PMB 25/55-55. This is reflected in the basic characteristic, SHRP G*/sin δ (under 60°C), which is highest for the binder with FTP and lies around 10 kPa in the other cases which meets the minimum condition of 1 kPa.

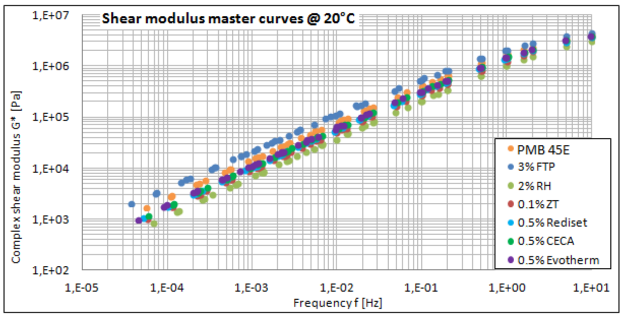

Figure 8: Master curves of complex shear modulus for PMBs of set 2

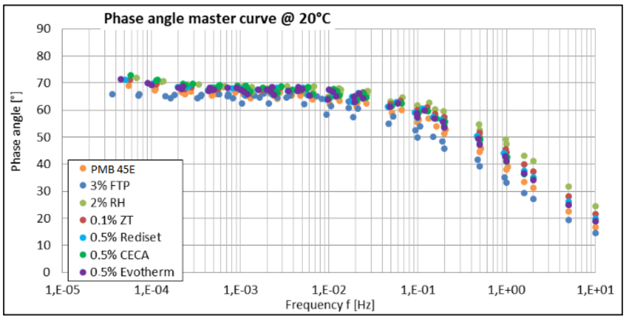

Figure 9: Master curves of phase angle for PMBs of set 2

In the case of bituminous binders in set 2, the individual options show slightly different behaviour from the master curve perspective. A higher resistance to deformation is demonstrated by the variant with FTP with a smaller difference compared to PMB 45E; the difference is no longer visible under the frequency of approx. > 5 Hz. Due to that, the lower thermal sensitivity of binders with this additive, which was discovered in the previous set, is confirmed. The remaining additive result in a slight deterioration of the master curves; the binder with RH is characterised by the curve with the lowest values; compared to our knowledge of this additive, the result is questionable.

4.4 Multi-stress creep recovery test (MSCR)

The last test presented is the assessment of multiple stress and recovery of the bitumen sample from the perspective of resistance to plastic deformation, the so-called MSCR test. The test method determines the percentage of elastic recovery and the value of irreversible shear compliance. The elastic recovery serves as an indicator of the sample's return to the original shape when stressed and left to recover several times. The percentage is used to determine the

degree of elastic response and strength dependencies for both modified and non-modified bituminous binders; it is a very good indicator of the elasticity levels of both types of bituminous binders.
The method is suitable for assessment of bitumen behaviour under high temperatures. The advantage of the test in contrast to previously described elastic recovery of ductility test (developed especially for PMBs) is the speed of the test and the possibility of choosing different temperatures and various loading levels for the test. High elastic recovery of the modified binders results in much smaller permanent deformations. Therefore, from the quality assessment perspective, binders with higher elastic recovery values are assumed to be more suitable and better-performing. At the same time, such binders are expected to achieve the lowest shear compliance.

Table 6: MSCR test results for assessed binders

Bitumen	0,1 kPa			3,2 kPa		
	Strain ε [-]	**El. recovery [%]**	**J_{nr} [1/kPa]**	**Strain ε [-]**	**El. recovery [%]**	**J_{nr} [1/kPa]**
PMB 25/55-55	2,39	50,16	0,34	33,21	**27,87**	**0,59**
PMB 25/55-55 + FTP	0,54	103,58	0,00	4,27	**84,28**	**0,02**
PMB 25/55-55 + RH	1,63	62,10	0,19	21,45	**42,70**	**0,33**
PMB 25/55-55 + Zycotherm	1,80	47,83	0,28	26,55	**27,39**	**0,47**
PMB 25/55-55 + Rediset	1,52	56,86	0,21	23,52	**35,09**	**0,39**
PMB 25/55-55 + CECA	1,89	58,29	0,21	24,48	**37,41**	**0,39**
PMB 25/55-55 + Evotherm	1,70	55,00	0,23	24,69	**32,60**	**0,42**
PMB 45E	1,03	103,50	-0,01	7,64	**104,95**	**-0,01**
PMB 45E + FTP	1,06	136,23	-0,07	4,86	**138,71**	**-0,09**
PMB 45E + RH	1,92	107,27	-0,03	11,20	**107,30**	**-0,03**
PMB 45E + Zycotherm	2,05	61,70	0,27	29,87	**37,13**	**0,51**
PMB 45E + Rediset	1,26	104,22	-0,01	10,83	**100,57**	**0,00**
PMB 45E + CECA	1,50	90,77	0,04	9,67	**100,26**	**0,00**
PMB 45E + Evotherm	1,36	117,10	-0,06	8,59	**107,51**	**-0,03**

The test describes the behaviour of the bitumen under the selected temperature of 60°C with the concurrent shear stress of 0.1 kPa and 3.2 kPa, always in ten loading cycles for each stress level. The measurement yields the value of bitumen elasticity, expressed as the value of irreversible shear compliance J_{nr} at the same time. The lower this value, the higher proportion of stress the binder can absorb without being too susceptible to plastic deformation. Based on the current findings, loaded pavements are required to have ultimate $J_{nr} < 1$ kPa^{-1} for the stress of 3.2 kPa and temperature of 60°C. The test was completed for all options; the results are the medians obtained of at least two samples (see Table 6).
In set 1, by far the best results are achieved by the PMB variant with FTP again (see Fig. 10). Irreversible shear compliance is virtually zero and the binder demonstrates a high elastic recovery value. The remaining binders show similar values; the finding of a greater improvement compared to the reference binder for both characteristics observed is surprising. In all cases, the value of J_{nr} under the stress of 3.2 kPa was significantly lower than the required 1 kPa^{-1}.
With the exception of bitumen with Zycotherm, the results achieved by set 2 (see Fig. 11) can be considered quite even; the variant with FTP demonstrated a slight improvement again. In the overall proportion, this correlates well with the master curve comparison. Disregarding the aforementioned exception, the binders demonstrate virtually zero shear compliance and high elastic recovery. The reason for the distinctively poorer result of the variant with Zycotherm is not clear as the additive generally has no impact on resistance to permanent deformation. For this binder further measurements are envisaged. The negative measurements were repeatedly confirmed by multiple re-measurements of some samples. The question is whether the compliances are so small due to high elastic recovery that the necessary precision of measurement is impossible to achieve or it is a system error of the test performance.

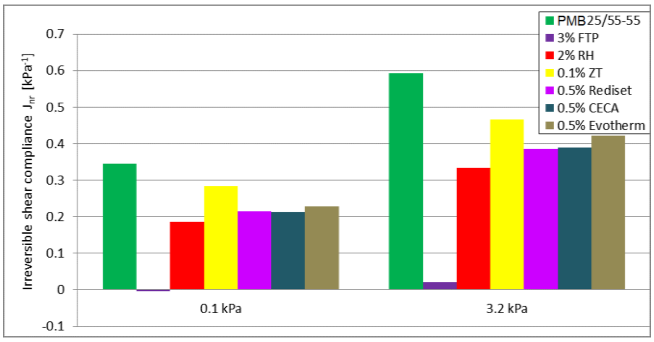

Figure 10: Irreversible shear compliance for PMBs; set 1

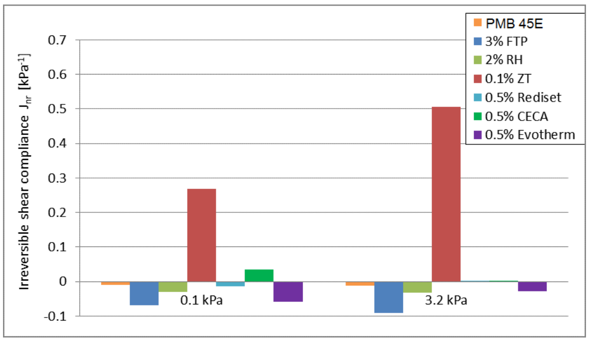

Figure 11: Irreversible shear compliance for PMBs; set 2

5. EXPERIMENTAL ASSESSMENT OF STONE MASTIC WARM ASPHALT

From the point of view of asphalt mixes, stone mastic asphalt SMA 11 was selected. The mix design was not optimised at all; a design prepared in compliance with the test protocol by Eurovia CS was used. The aggregate was spilite from Litice. The bitumen content amounted to 5.8 % by mass of the mix. At the same time, fiber additive S-Cell was added to each variant of the SMA 11 asphalt mix (0.3 % by mass of the mix). The basic asphalt mix options were manufactured and compacted under 160°C. The options that could be classified as “warm mix” ones had not standard manufacturing temperature optimisation according to the method recommended in technical conditions TP238. In compliance with the findings obtained in 2013 for AC surf 11 mixes with similar binder combinations (the differences were particularly the base consisting of 50/70 bitumen), the variants with one of the additives used had the manufacturing and compaction temperatures reduced to the 140°C level. The framework of searching for innovative solutions for WMA involved the assessment of two sets of bituminous binders.

5.1 Selected test results of the SMA mix variants assessed

The following tests were performed for the individual variants of SMA 11+ asphalt mix:

- Determination of asphalt mix bulk density, maximum bulk density and void content in the mix (EN 12697-6+A1, EN 12697-5+A1, EN 12697-8);
- Determination of water susceptibility by means of indirect tensile strength test (EN 12697-23);
- Determination of water susceptibility with one frost cycle by indirect tensile strength test (ITSR according to EN 12697-12), modified method according to AASHTO T283;
- Determination of the stiffness modulus by cyclic indirect tensile stress test (IT-CY according to EN 12697-26);
- Determination of bending tensile strength and relaxation of asphalt mix under 0°C (TP 151);
- Determination of resistance to crack propagation under 0°C and -10°C in accordance with EN 12697-44.

6. RESULTS OF THE SMA MIX ASSESSED

6.1 Water susceptibility

Fig. 12 and Fig. 13 indicate the resulting ITSR values determined in accordance with the method stipulated in the harmonised EN 12697-12. Besides the results, the experimental assessment also involved the indirect tensile stress ratios determined by the modified methodology according to AASHTO T283, which are illustrated in Fig. 14 and Fig. 15. In general, this test is not required for SMA 11 type of mix; however, it was employed within the assessment to allow evaluating the durability of the asphalt mixes. As is obvious from the results presented, the individual options behave with minor differences in the two bitumen sets assessed; in the case of the set with binder PMB 45 E, the mixes on average score better, i.e. with lower water susceptibility. While, in the case of set 2, the results are quite even (with the exception of the mix with Evotherm), in set 1 the ITSR indicator increases with the application of CECA and with the use of Evotherm. Particularly the latter behaves differently when PMBs of different origins are used.

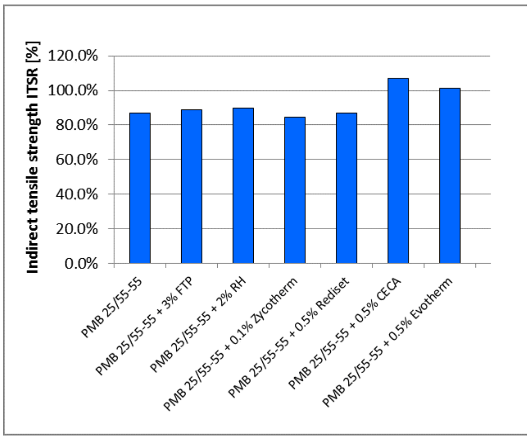

Figure 12: Water susceptibility according to EN 12697-12; set 1

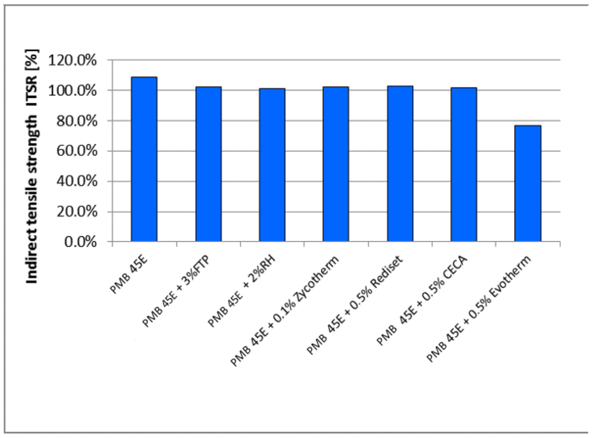

Figure 13: Water susceptibility according to EN 12697-12; set 2

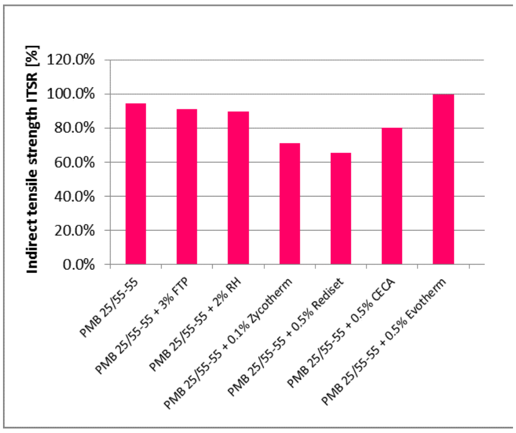

Figure 14: Water susceptibility according to AASHTO T283; set 1

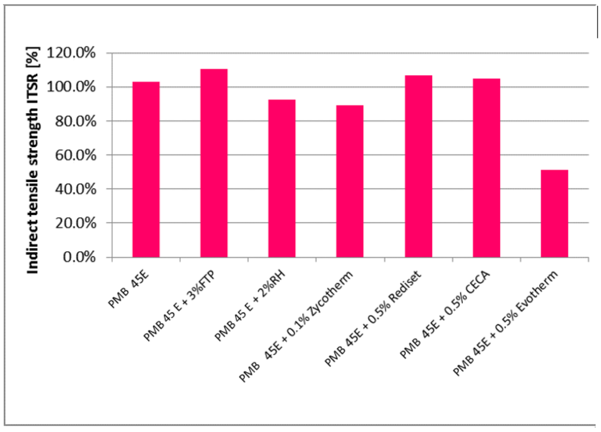

Figure 15: Water susceptibility according to AASHTO T283; set 2

6.2 Stiffness results

From the point of view of deformation characteristics in relation to the behaviour in moderate temperature range, the stiffness of individual asphalt mixes was assessed. To a certain degree (not completely), the stiffness could substitute for the values of asphalt mix resistance to permanent deformation. The stiffness by IT-CY method was determined for 5°C, 15°C and 27°C. For the sake of clarity, the results are given in Table 7 and no figure is presented (it would provide a better illustration of the tangent of stiffness decrease depending on the temperature which suggests conclusions on the thermal susceptibility of the asphalt mix).

If the result evaluation is restricted to 15°C which is considered crucial from the point of view of pavement structure design according to technical conditions TP 170, it is obvious that the application of additives to reduce the asphalt mix manufacturing temperature does not increase the stiffness values. In contrast to that, all cases (with the exception of option PMB 25/55-55 + FTP) demonstrate a fall which is more pronounced in the variant with Rediset and Evotherm within the first set. However, this fact cannot be explained by changes in the void content; this would apply in the case of the option with Rediset, not in the other case. In the case of set 2, the decrease of all mixes with additives is generally smaller; the most distinctive decreases were shown by mixes with Zycotherm and CECA. The change cannot be explained as a consequence of a change in the void content in this case, either. Also the comparison of the two basic PMBs is interesting where the second binder tested shows a higher value; however, if we focus on thermal susceptibility, the thermal dependency could suggest a slightly increased thermal susceptibility of this binder.

If the stiffness of the asphalt mix at 15°C were compared to the complex shear modulus of bitumen at both temperatures observed (40°C and 60°C), it would be obvious that certain interdependence only applies to the basic PMBs. In cases where the binders are combined with individual additives, any comparability under the selected temperature disappears completely. Therefore, the binder complex shear modulus values give no indication of any improvement or deterioration of the stiffness modulus of the asphalt mix.

Table 7: Stiffness comparison for assessed mixes

Mix	**Used bitumen**	**Stiffness (MPa) @ T**		
		5°C	**15°C**	**27°C**
SMA_1	PMB 25/55-55	16 900	7 000	3 050
SMA_2	PMB 25/55-55 + FTP	16 100	7 000	3 650
SMA_3	PMB 25/55-55 + RH	15 350	6 800	2 550
SMA_4	PMB 25/55-55 + Zycotherm	16 500	6 050	1 700
SMA_5	PMB 25/55-55 + Rediset	13 250	5 750	2 400
SMA_6	PMB 25/55-55 + CECA	15 050	6 600	2 050
SMA_7	PMB 25/55-55 + Evotherm	13 800	5 200	1 750
SMA_10	PMB 45	17 600	8 350	2 950
SMA_11	PMB 45 + FTP	10 900	6 100	2 700
SMA_12	PMB 45 + RH	13 700	6 700	2 750
SMA_13	PMB 45 + Zycotherm	10 850	5 600	1 750
SMA_14	PMB 45 + Rediset	13 450	5 950	1 900
SMA_15	PMB 45 + CECA	13 400	5 600	2 600
SMA_16	PMB 45 + Evotherm	14 300	6 600	1 800

6.3 Crack propagation test

The assessment of asphalt mix behaviour under low temperatures in this paper presents the results of the test of mix resistance to crack propagation carried out on semi-cylindrical test specimens according to EN 12697-44. This is a rather fresh test method which can be performed on test specimens with 100 mm or 150 mm diameter. The modified specimens with weaker cross-sections (with the groove) are loaded by a constant deformation speed of 5 mm/min. The test yields the derived maximum stress and, primarily, the fracture toughness. Within the framework of several years' activities at CTU Prague, the test has been performed under 0°C and -10°C; lately, assessments have even been made under -5°C or -15°C to obtain the thermal dependency and assess the tangent of the fracture toughness development.

The results as presented show that the fracture toughness values for lower temperatures are usually slightly higher. It is also obvious that in case of the binder set 1 assessed, the individual mixes can be divided into two sets. In the first case, the application of an additive to reduce the manufacturing temperature does not have a negative effect on the fracture toughness characteristic (RH and Zycotherm); in the other case, a slight deterioration in relation to the initial binder can be noticed. The difference is more obvious under the lower test temperature. Generally, the phenomenon should only be noticed in relation to mixes where synthetic waxes are applied to the bituminous binder, thus giving rise to a concern about the impact of the wax on deteriorating asphalt mix behaviour within the cold temperature range. However, this assumption has not been verified when set 1 of bituminous binders was assessed. Similarly, the effect of void content which could be related to the lower manufacturing temperature was not confirmed either. In the case of set 2 of the bituminous binders assessed, the aforementioned is confirmed by additives RH and Zycotherm, although just for the temperature of 0°C. At the same time, the mix with Evotherm demonstrated better behaviour in this case. Under the lower test temperature, the results differ and the binder with FTP and Rediset scored better in comparison to set 1 findings. From the perspective of void content, the variants have a slightly higher value than the reference mix.

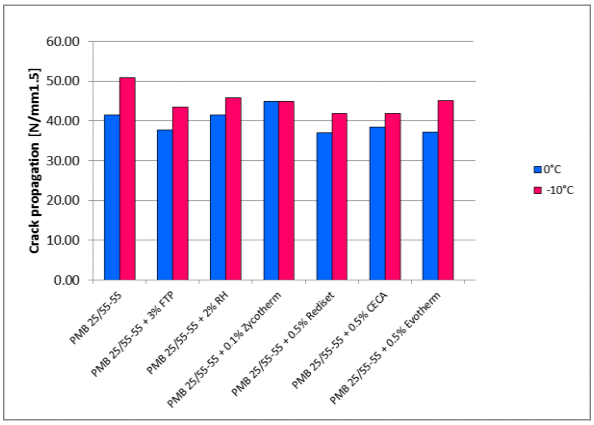

Figure 16: Results of fractural toughness of WMAs from set 1

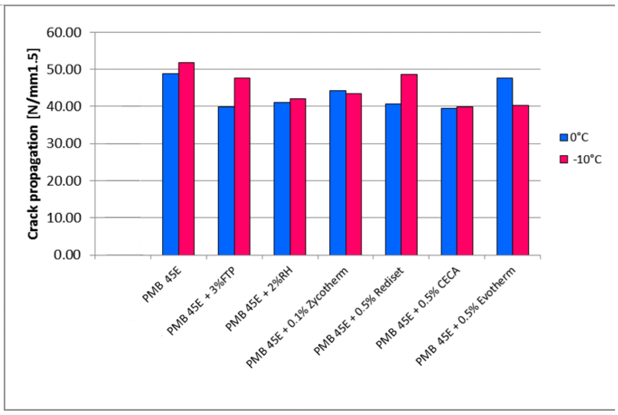

Figure 17: Results of fractural toughness of WMAs from set 2

6.4 Results of bending tensile strength and relaxation tests

Bending tensile strength was tested in compliance with the method stipulated in the Technical Conditions of the Ministry of Transport of the Czech Republic, ČR TP 151, under the temperature of -5 °C with the choice of one loading speed (50mm/min) due to the subsequent relaxation test. A summary of the results is given in Figure 18. A comparison of the bending tensile strengths of individual options of PMBs and WMA additives clearly shows that the reference mix with PMB has similar scores, as well as the option with FTP. The highest values were determined for the mix with Zycotherm. The remaining combinations of modified binders demonstrate significantly lower bending tensile stress values which is not quite in accordance with the findings from mix behaviour under low temperatures as confirmed by the crack propagation test. Focusing on the comparison of set 2 of the options with PMB 45E, the same trend can be noted (just the variant with PMB 45 only reaches the same values as the mix with FTP; in the first set, it was the option with Evotherm); this corresponds quite well with the results of fracture toughness for 0 °C as indicated in Figure 17. For the remaining options, we can also notice lower bending tensile stress values again.

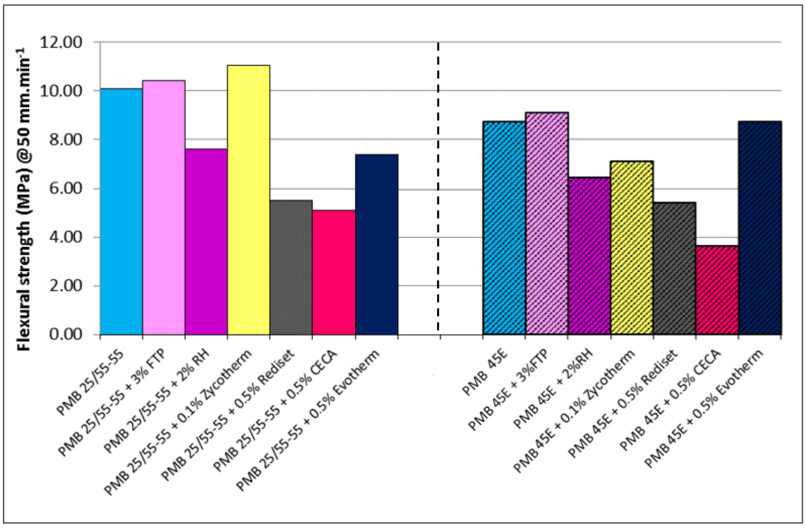

Figure 18: Bending tensile strength values of assessed mixes

The relaxation test was set to a water bath of 0°C according to the method stipulated by TP 151 for asphalt mix beams of 50x50x320mm dimension. For the sake of clarity, Figure 19 presents all mixes sorted similarly to the preceding tests. The application of PMB by a different manufacturer is represented by the shaded area. The results very well show that the mixes employing both types of modified binders in combination with Evotherm are the best to relax, i.e. the stress delivered is reduced to 50 % level the fastest. Combinations with RH and Zycotherm score double values. The highest values are demonstrated by the additive CECA and the value is higher yet in case of PMB 45E. The results show that mixes with lower bending tensile strengths, for example if Rediset or CECA is used, relax noticeably more slowly than mixes with high strength characteristics. In the case of application of synthetic waxes, Zycotherm and Evotherm, the relaxation time is lower than in the case of adding just PMB to the mix.

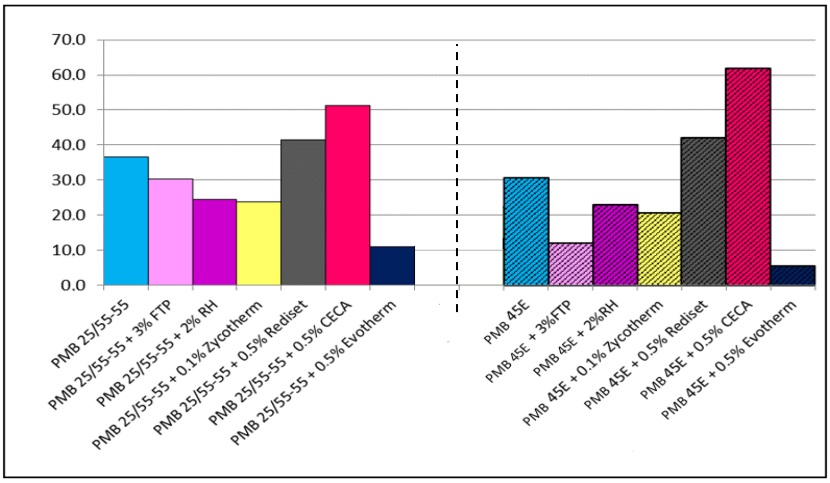

Figure 19: Results of relaxation for assessed mixes at 0 °C

7. CONCLUSION

The extensive experimental study assessed six different additives, which can be classified as warm mix asphalt additives. They were applied to two types of PMB of the same classification according to ČSN EN 14023. All of the binders assessed were subsequently used to test options of SMA 11 in order to verify the deformation behaviour and overall durability of the asphalt mix. Starting with the assessment of asphalt mix durability as expressed by the water susceptibility characteristics, it is obvious that the application of the additives does not affect this qualitative parameter fundamentally. In some cases, the water susceptibility was noticed to decrease even in the case of observing the modified method according to AASHTO T283, which applied an additional freezing cycle. In this case, an exception was the binder PMB 45E + Evotherm, where a more noticeable reduction in the ITSR value was observed.

In the case of deformation characteristics of the asphalt mixes, a slight reduction of the stiffness modulus values was observed. However, it can be generally noted that with respect to the SMA 11 mix utilisation, all assessed options provide sufficient stiffness values. The crucial finding is that SMA mixes in combination with PMBs were not proven to demonstrate higher stiffness values when WMA additives (even in the case of synthetic waxes) were added. The findings could not be supported by a comparison with the assessment of selected functional characteristics of bituminous binders where the results differed and, at least in the case of the FTP additive, always ended up with higher complex modulus scores. Similarly, no clear dependence can be seen for low operation temperatures, either. Although the force ductility test (e.g. in the case of PMB 25/55-55+FTP) indicates a higher susceptibility to earlier brittle fracture under lower temperatures (5-10°C), this fact was not proven for asphalt mixes when the crack propagation test or bending tensile strength test was carried out. Therefore, it is impossible to prove that a suitable correlation between used binders and asphalt mixes exists.

ACKNOWLEDGEMENTS

This paper was prepared under the funding scheme of Competence centre program by the Technology Agency of the Czech Republic within the project Centre for effective and sustainable transport infra-structure (CESTI), project no. TE01020168.

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