

# Comparison of influence of ageing on low-temperature characteristics of asphalt mixtures

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**Abstract.** Ability of relaxation of asphalt mixtures and thus its resilience to climate change and traffic load is decreasing by influence of aging – in this case aging of bituminous binder. Binder exposed to climate and UV ages and becomes more fragile and susceptible to damage. The results of the research presented in this paper are aimed to finding a correlation between low-temperature properties of referential and aged asphalt mixture specimens and characteristics (not low-temperature) of bituminous binders. In this research there were used conventional road binders, commonly used modified binders and binders additionally modified in the laboratory. The low-temperature characteristics were determined by strength flexural test, commonly used in the Czech Republic for High Modulus Asphalt Mixtures (TP 151), and semi-cylindrical bending test (EN 12697-44). Both of the tests were extended by specimens exposed to artificial long-term aging (EN 12697-52) – storing at 85 ° C for 5 days. The results were compared with characteristics of binders for finding a suitable correlation between characteristics of binders and asphalt mixtures.

## 1. Introduction

Asphalt mixture is a complex material used in asphalt pavement layers. The pavement is exposed to traffic load, weather conditions, UV radiation, climate changes etc. All these factors can cause ageing of asphalt mixtures which can lead to micro cracks and surface damages [1], which is significant source of premature degradation especially at low temperatures.

The asphalt mixtures are composed of aggregate, filler, bituminous binder and air voids. Also some additive can be provided for artificial improvement of asphalt mixture's properties. Asphalt mixture behaviour depends not only on properties of particular components, but also on air voids content, therefore compaction rate on the pavement.

Ageing in asphalt mixtures occurs mainly because of ageing (oxidation) of bituminous binder. The influence of ageing of aggregate was not proven yet, all the researches are focused solely on binder ageing. The binder oxidises due to its exposing to air especially at high temperature. The binder becomes harder (its penetration lowers), but more brittle – it can crack more easily, especially at low temperatures, and low temperature cracking can occur. The influence of binder ageing has been found to vary due to crude oil source, used additives, characteristics of asphalt mixture and characteristics of climate [2].

Resistance to fatigue is one the most significant characteristics of asphalt mixture. Unfortunately the fatigue test is very time and cost consuming. Further very important characteristics in countries like the Czech Republic is the material behaviour at low temperature range. Low temperature causes



shrinkage of asphalt layers and therefore the tensile stresses are growing. The tensile stress increases with decrease of the temperature. When the stress reaches the asphalt mixtures tensile strength, the crack arise. The low temperature cracks occurs from the bottom of the layer and expands to the top. The climate changes and traffic load repetitions causes the propagation to the pavement surface. When the cracks occur on the surface, the water can infiltrate the pavement and the bearing capacity of pavement is significantly lowered [3].

Low temperature characteristics of asphalt mixtures can be determined by a lot of different laboratory tests. The tests differ in shape of test specimens, testing temperatures, testing devices etc. The method are for example [3] - [8]:

- Single-Edge Notched Beam (SENB) under Three or Four-Point Bending;
- Unnotched Beam Three-Point Bending;
- Disc-Shaped Compact Tension test (DCT);
- Indirect Tensile Test (IDT);
- Semi-Circular Bending Test (SCB);
- etc.

For this research two testing methods were chosen – Semi-Circular Bending Test (SCB) and Three-Point Bending of unnotched beams (3PB). The SCB test is worldwide popular, so many researchers use it in their work and therefore there are many comparable data available. The test is easy for required equipment and the specimens can be also easily prepared from core sample. For appropriate results it is very important to pay an increase attention to specimen preparation. The preparation is easy on its self, but it has to be done with precise. Compacted Marshall's specimens or core samples are cut to height of 50 mm, then halved and notched in the middle of bottom side. All the cutting has to be done exactly parallelly and centrally. The improper cutting can negatively influence the test results.

The three point bending test of unnotched specimens was chosen because of its common use in the Czech Republic for High Modulus Asphalt Mixtures (HMAC). The test is also easy for required equipment. The beam specimens are cut from the slabs used for rutting test. As in previous test the precise cutting work is required for adequate data.

## 2. Test methods of asphalt mixtures

### 2.1. Resistance to crack propagation

The resistance to crack propagation (semi-cylindrical bending test) was determined in compliance with the requirements of EN 12697-44 [14]. The essence of the test is a three-point bending using semi-cylindrical specimens with a defined notch in the middle of bottom specimen face. The test procedure was performed at a test temperature of 0 °C applying a standardized loading rate of 5 mm/min.

The test specimens were manufactured according to EN 12697-30 (Specimen preparation by impact compactor) using 2x50 blows of the Marshall hammer (50 blows from each side of the specimen) and then cut on laboratory saw.

The test specimens were divided into two sets. First set ("virgin") was left at laboratory conditions and the second set ("aged") was exposed to artificially ageing procedure specified in prEN 12697-52 [13]. The simulation of ageing was realized by conditioning the specimens in laboratory oven at temperature of 85 °C for 5 days. After the ageing procedure the Marshall's specimens were cut into the test specimens.

The significant resulting parameter is a fracture toughness [ $\text{N}\cdot\text{mm}^{-3/2}$ ]. The fracture toughness is defined as force multiplied by correction factor comprising the dimensions of the specimen. For fracture toughness and maximum stress the ageing indexes were calculated as a ratio of resulting value of aged specimen to virgin (unaged) specimen's value. The ageing index shows the thermo-oxidation degradation potential of the bituminous binder in an asphalt mixture.

## 2.2. Three-point bending (Tensile bending strength)

Tensile bending strength (simple destructive 3-point beam test) was tested in observance to the method given by the Technical specifications of the Ministry of Transport of the Czech Republic - TP 151 [9]. It is necessary to point out, that this is a specific test used mainly in the Czech Republic and required presently only for HMAC mixtures. The test was used in this research for its long history and sufficient results with HMAC and its practicability and repeatability which can be provided by a regular road laboratory.

Tensile bending strength is tested on beams, which can be cut from slabs used for wheel tracking test. The slab can be used for cutting the test specimens only in the case, when it is not too deformed from wheel tracking test. If the track would be too deep or the slab would be anyhow deformed from the test, it is necessary to prepare new slab specially for this bending beam test. The part of the slab under the wheel track is thrown away and only unloaded part are used as test specimens.

TP 151 [9] specifies testing temperature in a range from -20 °C to 0 °C. For sake of this research, temperature of 0 °C was chosen. TP 151 [9] specifies two testing velocities of loading (loading rates): 50 mm/min and 1.25 mm/min. The slower speed was chosen to allow certain comparability with the results of the SCB test.

The increase of the force and deformation during the test was recorded and data points were used for drawing a stress-strain diagram. The diagram was used additionally to determine the fracture energy, which can be defined as energy expended for beam to break. Fracture energy is calculated as the area under the curve of the force-deformation diagram. The data recorder used in the research records 50 data points per second.

The significant resulting parameter of three point beam bending test is flexural strength and flexural modulus of elasticity, both in [MPa]. Again the ageing indexes were calculated.

## 3. Test methods of bituminous binders

The results of test performed on asphalt mixtures were compared with results of bituminous binder tests for possible identification of any correlation between these parameters. On the bituminous binders the following test set was applied:

- Ring and ball softening point test (EN 1427);
- Needle penetration test at 25 °C (EN 1426);
- Complex shear modulus  $G^*$  and phase angle  $\delta$  determination at 40 °C for selected frequencies within the 0.1-10 Hz range and under shear stress of 2000 Pa (controlled stress mode) using dynamic shear rheometer HAAKE MARS (ČSN EN 14770);
- Multiple stress creep recovery test (MSCR) according to US methodology, AASHTO 70-09 and in compliance with prEN 16659 at 60 °C.

The bituminous binders were additionally artificially aged by 2 different methods in the laboratory. These methods were chosen because of the available laboratory equipment. The ageing methods were:

- TFOT (Thin-Film Oven Test) – where the binder is exposed to temperature of 163 °C for 5 hours (EN 12607-2);
- PAV (Pressurized Ageing Vessel) – where the binder is under pressure exposed to the temperature of 100 °C for 20 hours (EN 14769). Ageing was performed on a bituminous binder sample which had been subjected to short-term ageing first.

## 4. Test variants of asphalt mixtures

For this research 2 different asphalt mixtures were chosen. The first one was a superior stone mastic asphalt mixture with maximum particle size of 11 mm (SMA 11S) with 5.8 M% of bituminous binders. And the second one was high-stiffness modulus asphalt mixture with maximum particle size

of 22 mm (HMAC 22S) with 4.8 M% of bituminous binders. Both mixtures were optimized within research studies which are presented in [11, 12].

The variants cover common unmodified binder with gradation 50/70, polymer modified binder PMB 40/80-85 and four binders modified in laboratory by “VP” additive. The “VP” additive is alpha-polyolefin based synthetic wax. The additive should improve adhesion of bitumen to aggregate and increase the strength characteristics. The additive was dosed to the bitumen in 1 M%, 5 M% and 6 M%. The modified binder was subsequently used for mixture manufacture. In one of the variants “VP” was combined with 3 M% of SBS modifier. Detailed data about bitumen characteristics and advanced functional tests can be found e.g. in [10]. The test variants with used binders with air void contents of mixtures (determined in compliance with EN 12697-8) are summarized in Table 1.

**Table 1.** Summary of test variants of SMA 11S and HMAC 22S mixtures and their air voids content.

Mix		Binder	Binder content (%)	Air voids content (%)
SMA	<b>50/70</b>	50/70		3.4%
	<b>5V</b>	50/70 + 5 % VP	5.8%	3.7%
	<b>6V</b>	50/70 + 6 % VP		4.1%
	<b>SBS+VP</b>	50/70 + 3 % SBS a 1% VP		3.0%
HMAC	<b>5V</b>	50/70 + 5 % VP	4.8%	2.6%
	<b>6V</b>	50/70 + 6 % VP		3.3%
	<b>PMB</b>	PMB 40/80-85		2.6%
	<b>PMB+VP</b>	PMB 40/80-85 + 1% VP		4.2%

## 5. Results of the test methods

Due to the exposure of the asphalt specimens to high temperatures and air flow (simulation by artificial laboratory ageing at temperature of 85 °C for 5 days), the bituminous binder in asphalt mixture oxidized and changed the properties of the mixture. The binder penetration lower and therefore the strength properties of asphalt mixture grows to a certain level. If the temperature of mixture decreases due to weather conditions the binder becomes more brittle. At low temperatures and under repeating traffic load the freeze cracks can occur and it causes the degradation of the pavement. This risk of freeze crack occur increases with ageing of the binder.

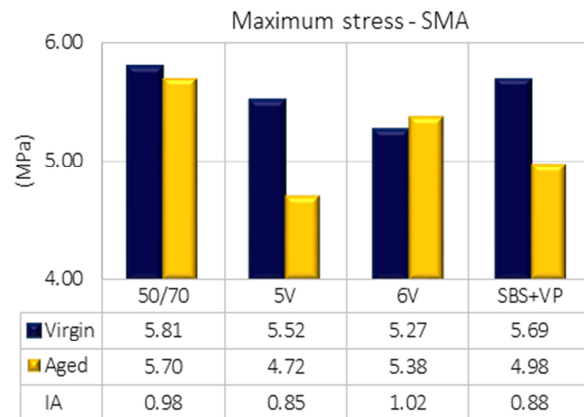
### 5.1. Resistance to crack propagation

As written above the resistance to crack propagation was determined according to EN 12697-44 [14] on notched semi-cylindrical specimens. In the results of this test there is visible a little unusual phenomenon. The expected trend of low temperature test parameter measured at 0 °C is that aged specimens will reach higher value. The aged binder increases to certain point the strength characteristics of the mixture. Nevertheless the fracture toughness of aged specimen of SMA mixtures in most cases decreases in comparison to virgin specimen. This phenomenon can be followed for surface layer mixtures through many recurrences taken in CTU Road laboratory during last 2 years. The fracture toughness of surface layers mixtures reaches usually lower values for aged specimens. These unusual results are described for example in [13].

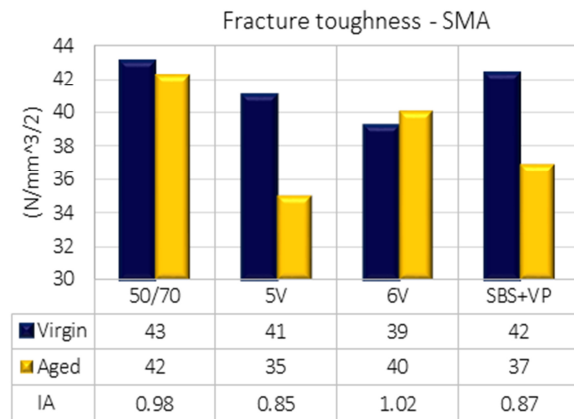
The HMAC mixtures evince usual predictable trend, where fracture toughness is increased for aged specimens.

For all mixtures the ageing index was calculated. The closer the ageing index (labelled as “IA” in figures) to 1.0 is, the less sensitive the mixture is to bitumen ageing effects. The most stable results were achieved by SMA mixture with 50/70 and 6% of VP and HMAC mixture with 5 % of VP. Like a

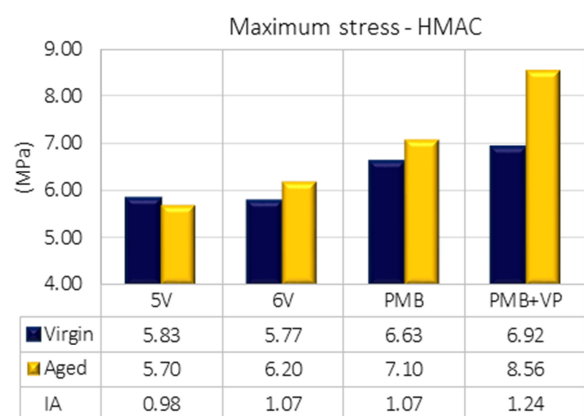
bad variant seems to be combination of 1 M% of VP with polymer modified binder. That causes high increase in fracture toughness and a high ageing index.



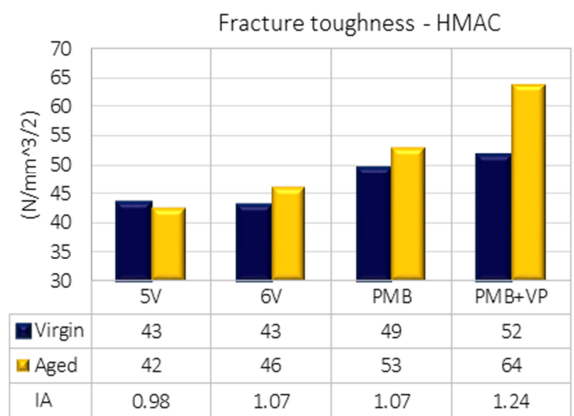
**Figure 1.** Maximum stress – SMA mixtures.



**Figure 2.** Fracture toughness – SMA mixtures.



**Figure 3.** Maximum stress – HMAC mixtures.



**Figure 4.** Fracture toughness – HMAC mixtures.

### 5.2. Three-point bending (Tensile bending strength)

The tensile bending strength was determined in compliance with the Czech Technical specifications TP 151 [9] on unnotched beam specimens.

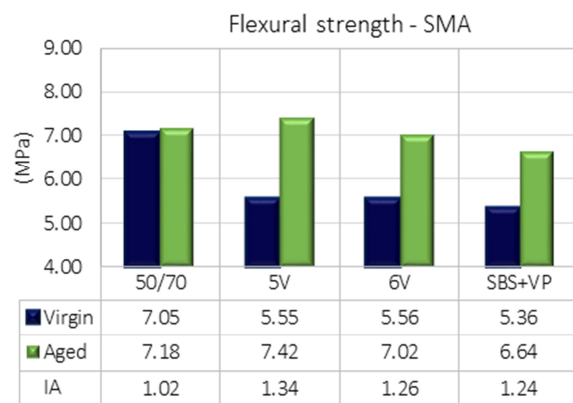
The common trend of flexural strength is that flexural strength of aged specimen is higher than flexural strength of virgin specimen. Again it applies the closer the ageing index (IA) is to 1.0, the less sensitive is the mixture to ageing.

The most stable values were reached by the SMA with 50/70 and HMAC with PMB. It appears as the VP additive enables the binder to age (oxidize) more easily and so the mixture becomes volatile over the life time. The same trend was visible even in the previous test, but the influence was not that noticeable.

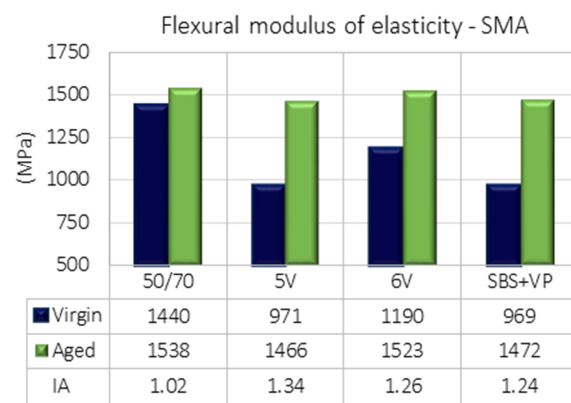
Quite surprisingly the SMA mixture with 50/70 reached again very high values in comparison to modified mixtures. The SMA mixture with 50/70 reached the highest values in both of the low temperature tests. It would be expectable, that modified binder would achieve better low temperature properties.

Among the HMAC mixture the highest results were achieved by the asphalt mixture with application of 6 % VP. For this mixture the influence of aged binder is the least visible (the ageing index is only 1.08).

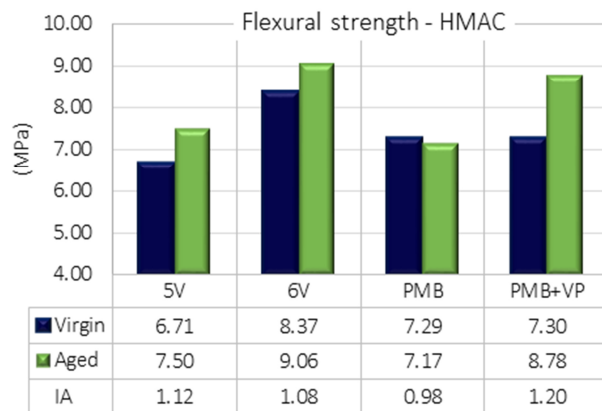
Additionally to common test parameter the fracture energy was calculated. The fracture energy was determined as an area under the stress-strain diagram. The fracture energy can be defined as the energy needed for the beam to expend its bearing capacity and collapse. This is another parameter, which can and should be determined during the test. Focusing only on maximum values of force and deformation can be misleading. For example in the case of HMAC mixture with PMB, the flexural strength and the corresponding modulus of elasticity are very similar, but the virgin specimens needed almost 30 % more of fracture energy to collapse. So with certain point of vigilance it is possible to say, it would take more repetitions of loading for this virgin mixtures to collapse.



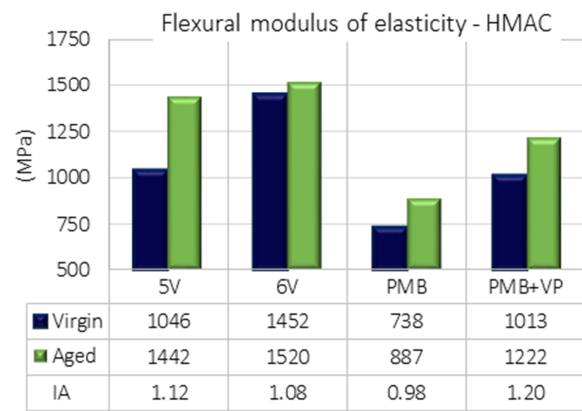
**Figure 5.** Flexural strength – SMA mixtures.



**Figure 6.** Flexural modulus of elasticity.

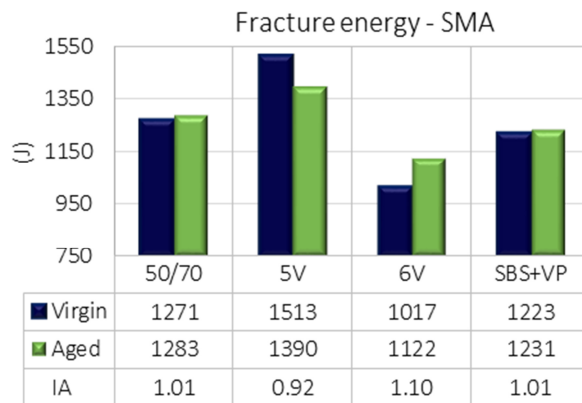
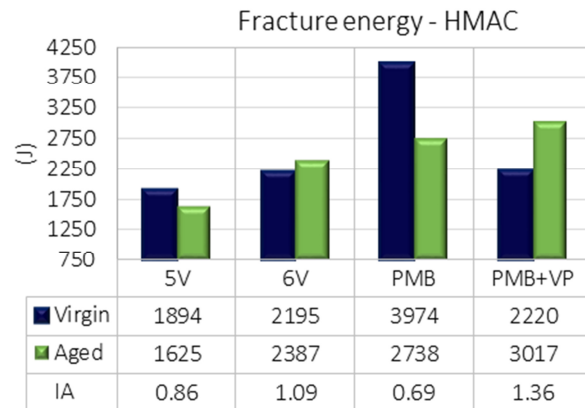


**Figure 7.** Flexural strength – HMAC mixtures.



**Figure 8.** Flexural modulus of elasticity – HMAC mixtures.



**Figure 9.** Fracture energy – SMA mixtures.**Figure 10.** Fracture energy – HMAC mixtures.

### 5.3. Properties of bituminous binder

The bituminous binder used for manufacturing of the asphalt mixtures were tested virgin, then were exposed to laboratory ageing by TFOT method and then the TFOT sample were additionally subjected to PAV ageing method. The results of performed tests are summarized in Table 2.

**Table 2.** Selected performance characteristics of used bituminous binders.

	Penetra- tion (x 0.1 mm)	Pen. index (-)	Softening point (°C)			Complex shear modulus at 40 °C, G*/sin(δ) (kPa)			Complex shear modulus at 40 °C, G* <sub>x</sub> sin(δ) (kPa)			Elastic recovery 3.2 kPa (%)
	Virgin	Virgin	Virgin	TFOT	TFOT +PAV	Virgin	TFOT	TFOT +PAV	Virgin	TFOT	TFOT +PAV	Virgin
50/70	64	-1	49	56	65	50.4	156.6	721.0	48.6	125.2	389.6	0.4
PMB	54	5.2	85	90	92	68.7	114.7	312.4	53.9	86.7	207.3	88.5
5V	42	1.7	66	73	83	175.9	531.2	1708.2	140.2	310.3	645.4	3.3
6V	39	2.5	72	75	83	190.7	735.1	1308.5	152.4	391.6	507.6	2.9
SBS+VP	32	1	74	75	82	226.6	486.7	632.2	163.5	279.8	331.8	28.8
PMB+VP	46	5.1	88	92	92	88.2	158.6	358.6	68.6	114.9	229.7	90.4

## 6. Correlation between low temperature properties of asphalt mixtures and the bitumen characteristics

The correlation indexes were calculated always as R-squared value for linear trend line. For preserving the paper extent the data were shown in tables not in more comprehensible figure. The Figure 11 and 12 (shown below) are used for demonstration of calculations and easier understanding the results.

The first correlation data were the comparison of the properties of virgin mixtures and virgin binders. The fracture toughness and flexural strength were compared with penetration, softening point and penetration index, complex shear modulus measured at 40 °C and elastic recovery measured by MSCR at 60 °C and 3.2 kPa.

Very good correlation appears between fracture toughness and penetration index. It applies for both types of asphalt mixtures. The higher the penetration index is, the higher the fracture toughness is.

The correlation index  $R^2$  for HMAC mixtures seems to be higher, which can be most likely caused by usage of only modified binders. In case of SMA mixtures there was one non modified binder (50/70) which behaves differently from the others. Very good correlation index can be also found for elastic recovery measured by MSCR. If the binder 50/70 would be eliminated, the correlation index  $R^2$  would reach 0.671 (instead of 0.085). Also the correlation index for  $G^*\sin(\delta)$  would increase to 0.374.

**Table 3.** Correlation between fracture toughness of virgin asphalt mixtures and virgin binders.

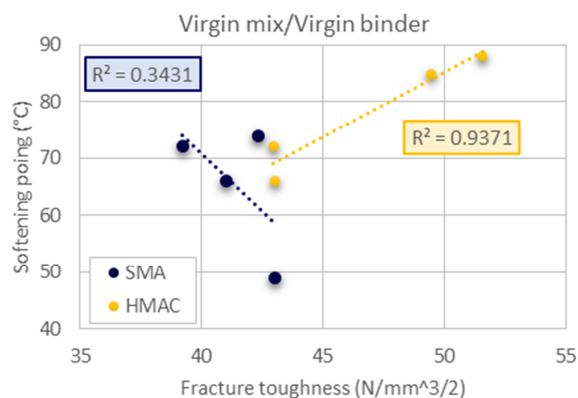
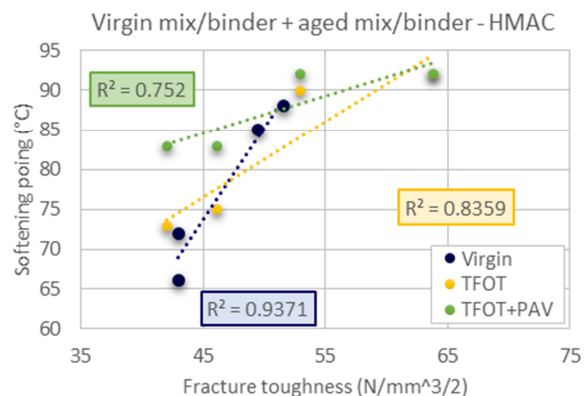
Mix	Binder	Fracture toughness (N/mm <sup>3/2</sup> )	Penetration (x 0,1 mm)	Softening point (°C)	Penetration index (-)	Complex shear modulus at 40 °C		Elastic recovery
		<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>
SMA	50/70	43	64	50	-1	50.4	48.6	0.36
	5V	41	42	66	1.7	175.9	140.2	3.33
	6V	39	39	72	2.5	190.7	152.4	2.89
	SBS+VP	42	32	74	1	226.6	163.5	28.82
	Correlation index R <sup>2</sup> =		<b>0.235</b>	<b>0.343<sup>*)</sup></b>	<b>0.814</b>	<b>0.243</b>	<b>0.341</b>	<b>0.085</b>
HMAC	5V	43	42	66	1.7	175.9	140.2	3.33
	6V	43	39	72	2.5	190.7	152.4	2.89
	PMB	49	54	86	5.2	68.7	53.9	88.46
	PMB+VP	52	46	88	5.1	88.2	68.6	90.44
	Correlation index R <sup>2</sup> =		<b>0.496</b>	<b>0.937<sup>*)</sup></b>	<b>0.918</b>	<b>0.888</b>	<b>0.893</b>	<b>0.967</b>

\*) shown in Figure 11

The correlation between flexural strength and the bitumen characteristics is not that noticeable as for the previous test. There are good correlations for SMA mixtures, but they are not proofed in any possibility with HMAC mixtures. For SMA mixtures the comparison with complex shear modulus works very well, however there is a zero correlation for HMAC mixtures. In case of fracture toughness the correlation index  $R^2$  with complex shear modulus reached almost 0.9 for HMAC and 0.24, resp. 0.34 for SMA mixtures.

**Table 4.** Correlation between flexural strength of virgin asphalt mixtures and virgin binders.

Mix	Binder	Flexural strength (MPa)	Penetration (x 0,1 mm)	Softening point (°C)	Penetration index (-)	Complex shear modulus at 40 °C		Elastic recovery
		<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>	<i>Virgin</i>
SMA	50/70	7.05	64	49	-1	50.4	48.6	0.36
	5V	5.55	42	66	1.7	175.9	140.2	3.33
	6V	5.56	38.8	72	2.5	190.7	152.4	2.89
	SBS+VP	5.36	32.1	74	1	226.6	163.5	28.82
	Correlation index R <sup>2</sup> =		<b>0.961</b>	<b>0.942</b>	<b>0.748</b>	<b>0.969</b>	<b>0.988</b>	<b>0.276</b>
HMAC	5V	6.71	42	66	1.7	175.9	140.2	3.33
	6V	8.37	38.8	72	2.5	190.7	152.4	2.89
	PMB	7.29	54.3	85	5.2	68.7	53.9	88.46
	PMB+VP	7.30	45.6	88	5.1	88.2	68.6	90.44
	Correlation index R <sup>2</sup> =		<b>0.133</b>	<b>0.001</b>	<b>0.001</b>	<b>0.091</b>	<b>0.092</b>	<b>0.044</b>

**Figure 11.** Comparison of properties of virgin mixtures and virgin binders.**Figure 12.** Comparison of properties of virgin mixtures and virgin binders and aged mixtures and aged binders.



Due to the higher correlation indexes for further comparison presentation only the fracture toughness was chosen. The fracture toughness of virgin specimens was compared with virgin binder properties and the aged specimens were compared with binder performed to two different ageing methods.

The correlation indexes  $R^2$  for HMAC mixtures demonstrates quite interesting trend, the indexes are in all cases higher than 0.6. More than half of the indexes almost reaches 0.9. The highest indexes are achieved for parameter  $G^*/\sin(\delta)$  of complex shear modulus and combination of TFOT + PAV ageing methods. From point of view of correlation between these tests, the combination of TFOT + PAV seems to evince results which correlate more with asphalt mixture specimens' ageing (5 day at 85 °C).

For SMA mixtures TFOT + PAV combination also evinces higher correlation, but the correlation indexes on its own are too low to be meaningful.

**Table 5.** Correlation between fracture toughness of virgin asphalt mixtures and virgin binders and aged asphalt mixtures and aged binders.

Mix	Binder	Fracture toughness		Softening point (°C)			Com. shear modulus at 40°C, $G^*/\sin(\delta)$ (kPa)			Com. shear modulus at 40 °C, $G^* \times \sin(\delta)$ (kPa)		
		Virgin	5d@85 °C	Virgin	TFOT	TFOT+PAV	Virgin	TFOT	TFOT+PAV	Virgin	TFOT	TFOT+PAV
SMA	50/70	43	42	49	56	65	50	157	721	49	125	390
	5V	41	35	66	73	83	176	531	1708	140	310	645
	6V	39	40	72	75	83	191	735	1309	152	392	508
	SBS+VP	42	37	74	75	82	227	487	632	164	280	332
	Correlation index $R^2 =$			0.343	0.480	0.554	0.243	0.172	0.243	0.172	0.250	0.341
HMAC	5V	43	42	66	73	83	176	531	1708	140	310	645
	6V	43	46	72	75	83	191	735	1309	152	392	508
	PMB	49	53	85	90	92	69	115	312	54	87	207
	PMB+VP	52	64	88	92	92	88	159	359	69	115	230
	Correlation index $R^2 =$			0.937 <sup>*)</sup>	0.836 <sup>*)</sup>	0.752 <sup>*)</sup>	0.888	0.569	0.888	0.569	0.759	0.893

<sup>\*)</sup> shown in Figure 12

## 7. Conclusion

Within the scope of the presented research study, two different asphalt mixtures were tested in range of low temperature. The low temperature properties were tested by two different methods – semi-cylindrical bending test and unnotched beam three point bending. The parameters were compared with properties determined on bituminous binder. The assignment on binder was softening point, penetration, complex shear modulus at 40 °C and elastic recovery at 60 °C and after stressing by 0.1 kPa and 3.2 kPa (MSCR test).

The set of test specimens was enlarged by the set of specimens aged exposed to artificially ageing procedure (5 days in 85 °C). The set of binders was enlarged by the set of binders exposed to artificially ageing by the TFOT method (5 hours at 163 °C) and by the combination of TFOT and PAV method (20 hours under the pressure at 100 °C).

The parameters were compared in intension to found some correlation between the properties of asphalt mixtures and input bituminous binders.

Even though the Czech Republic belongs to countries with cold winters and snow season the low temperature properties of asphalt mixtures are not ordinarily required. The only exception are HMAC mixtures, where the Czech Technical specifications TP 151 [9] demands the minimum value determined by unnotched beam three point bending test.

The correlation indexes determinate for HMAC shows certain connection between the properties of asphalt mixtures and binders. The higher the softening point is, the better low temperature characteristics are achieved. The smaller the complex shear modulus is, the better low temperature characteristics can be achieved. There is also a correlation between the aged binder by combination of TFTO and PAV method.

For point of view of time consumption the determination of binder properties is easier and faster than that for asphalt mixtures. The comprehensive binder properties can show a lot of final mixtures properties without time and monetary consuming tests. The asphalt mixture tests are always taken on larger amount of the tested composite material. The binder can be tested in amount of a few grams. It is very important to include more extensive sets of binder testing (functional based) into the standard and standardized test methods.

It is necessary to point out that the compassion was taken only for a narrow set of asphalt mixtures and only with 4 different types of binders for each mixture. It would be important to enlarge the set of different kind of mixtures (ACs) and bigger variety of assessed binders.

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