Development and verification of a suitable methodology for stability assessment of bitumen adhesion promoters

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ABSTRACT

Durability of pavement structures represents one of the most important aspects increasingly followed during last years. There are many reasons for this focus, most important are related to public budget constraints and the necessity to develop or rehabilitate road infrastructure. Since many years there has been a stress on using locally available resources which is for asphalt related mainly to the aggregates. On the other hand bitumen comes from a nonrenewable resource which is used to cover many demands of the world's society. To keep it simple, remaining byproduct is then classified as bitumen what has of course impact on its qualities as well.

In case of asphalt pavements durability is related to water and frost resistance of the mix. The water can cause problems if adsorbed by the aggregates. This happens if hydrophilic aggregates are used and the coating by bitumen is not good. For these reasons different additives are used to improve coating and turn the hydrophilic state to a hydrophobic one. Plenty of solutions are available on the market sometimes making the decision for not easy to make a right choice.

Since decades test methods have been developed to study durability aspects, adhesion or water susceptibility. In many countries like the Czech Republic simple methods are still used, often based only on observer's visual opinion and especially often qualifying only the status of a doped bitumen/mix when being produced. This is valid for all test methods defined in EN 12697-11 and similarly also in the national test method used in the Czech Republic. Based on this fact a research project was launched to ideally define a suitable test procedure for evaluating thermal stability and long-term activity of adhesion promoters if used in the bitumen and applied in a mix. This paper is describing how the test method was selected and evaluated, what are the principles the method is based on, if applicable only to the bitumen-aggregate coating quality or analyzing the water/water-frost impacts more. Results are given for the tests done and for assessment of different adhesion promoters. Test protocol is proposed at the end defining necessary conditions.

Keywords: Additives, Adhesion, Asphalt, Durability, Indirect tension

1. INTRODUCTION

The phenomena of bituminous binder – aggregate adhesion and asphalt mix moisture susceptibility have been the topic of extensive research since many years. Bitumen adhesion is one of the most important application properties of the binder which determines its ability to form bonds with the aggregate based on polar attraction and mechanical effects. It has a fundamental impact on the resulting parameters of the asphalt pavement quality and durability. The ability of the bitumen to adhere perfectly to the aggregate particle surface is crucial for the ability of the asphalt mix as such to resist weather conditions (e.g. rain, high temperatures, and frost), traffic impact and other unpredictable circumstances that occur in the pavement. Therefore, all of the associated factors must be taken into consideration. A faster procedure for assessing pavement durability based on the existing and reliable test methods is essential for reasonable application of innovative pavement concepts. Particularly in the mix design stage, optimal materials and combinations thereof can be selected to improve overall durability which will reduce the needs for resources and a longer pavement lifecycle can be achieved. Especially for new materials, like nano-technological adhesion promoters, viscosity improvers etc., there are no empirical data that could assist in the selection for more durable asphalt mix design.
Reduction of financial cost and increased effectiveness of production is today's greatest trend not only in the field of road construction. The main objective of the presented study was to develop a suitable procedure for laboratory assessment of asphalt pavement durability and long-term behaviour. The aim was to select appropriate laboratory method for ageing simulation, negative impact of water and frost which would, ultimately, reflect the conditions of layers within the pavement structure. Due to an enormous quantity of different variables, an exact prediction of behaviour of an aged pavement is practically impossible. Comparison of selected suitable laboratory methods for simulation of the asphalt mix ageing was therefore done. The study was based on the methods verifying the effects of adhesion promoters (methods to ascertain moisture susceptibility and adhesion). These are classified as qualitative and quantitative based on bitumen-aggregate tests or asphalt moisture susceptibility assessments. Qualitative tests provide subjective assessment - the evaluation is based on observations of the coated aggregate and comparison with some pre-defined scaling. Quantitative tests provide values for certain parameters like, for instance, the difference in strengths measured prior to and after specific conditioning. These tests are usually conducted on compacted test specimens and include: the Lottman test, conditioning test according to Tunnicliff and Root, modified Lottman test applied in USA (AASHTO T 283), test procedures according to European standard EN 12697-12 and many others.
This study chose one of the least suitable aggregates used for asphalt mix preparation in the Czech Republic. With respect to the results obtained, we focused closely on the properties of the aggregate as such in relation to its adhesion to bituminous binder. The physical and chemical surface properties of both the binders and the aggregate can be expressed by means of SFE (Surface Free Energy). SFE is defined as a relation between the energy and work needed to create a new material surface unit in a vacuum. SFE is generally considered as a useful tool to select materials with good water resistance. The Grenfell study also conducted the Rolling Bottle Test (RBT) and Saturated Ageing Tensile Stiffness (SATS) test to find out that aggregates with higher proportions of quarts and feldspar are more susceptible to moisture damage which also corresponds with the SFE value. Another option of determining the connection between adhesion and chemical composition of the aggregate is the peel test method developed to measure the adhesion force between the binder and the aggregate (Horgnies). The method consisted of leaving the coated aggregate in a water bath of 90 °C for 30 minutes, which caused a failure of the bond between the binder and the aggregate. X-ray Photoelectron Spectroscopy (XPS) was employed to highlight the spots on the aggregate surface where bituminous binders remained. In the granite sample, a binder layer was detected on mica and quartz minerals. Contrastingly, no binder residues were detected on alkaline feldspars which thus seem to be the cause of weak binder adhesion.

2. WATER SUSCEPTIBILITY

Moisture in general is capable causing loss of adhesion which results in damage to the asphalt pavement. Therefore, it is closely associated with moisture susceptibility. A typical mechanism of water damage is loss of adhesion due to moisture penetration in the bitumen-aggregate interface and stripping the bitumen film down to the gradual extraction of the aggregate from the pavement surface. As soon as the process starts the speed of pavement degrading increases until potholes are formed. The pace of such degrading is strongly affected by the ageing of the asphalt mix. Therefore, it is important to consider the effect of ageing when moisture susceptibility is evaluated. To create water-resistant mixes with limited loss of adhesion, suitable surfactants or lime hydrated are used. Nonetheless, the exact long-term effects of the chemical additives are not always known. Besides, the additives do not deliver a general solution to the problems caused by moisture in all materials when the necessary construction methods are used.

3. AGEING

Over 30 methods of ageing for either bulk asphalt mix or compacted asphalt test specimens have been developed in the recent decades. For those, the material is usually stored under higher temperature (30 °C to 100 °C) for a stipulated number of hours, days or weeks which is intended to accelerate the oxidative processes caused by atmospheric oxygen. In some cases, pure oxygen or ozone is used as oxidising agent, while elsewhere additional overpressure is applied to speed up the reaction. The ageing methods allow simulating a condition of long-term ageing in several days, thus presenting a situation closely resembling the effect on the layer within the pavement structure after several years.

3.1 Asphalt mix ageing vs. ITS characteristic assessment

Chemical ageing of the binder is an important aspect; however, asphalt ageing can be expressed more simply based on the connection with the mechanical properties of the mix, like indirect tensile strength. The test is very simple and cheap from the perspective of test specimen preparation and in-situ sample collection. The test can be used to determine tensile strength of the mix which correlates with the properties concerning cracking of the pavement. The higher the indirect tensile strength can be measured the higher resistance to cracks can be expected.

According to the study (Islam et al.) focused on resistance of aged specimens by ITS following findings were gained. Indirect tensile strength (ITS) of asphalt test specimens with voids content of 5.4 % increases during the ageing process. After the first day of ageing, strength increased by 18 %. The strength measured increased by 28 % after 5 days of ageing. The research also suggests that the highest increase in ITS occurs during the first few days from the start of ageing, i.e. roughly within 5 days; after that, the strength increase is minimal and further ageing does not have so much impact on the strength increase any more. The authors explain such steep increase of strength by the asphalt film hardening. When aged laboratory specimens were compared to bore-samples extracted in-situ (Interstate 40, New Mexico) – asphalt mix exposed to real weather conditions and traffic loads for 1 year. The specimens demonstrated ITS value increase by 19 % against the control specimens which is comparable to samples subjected to 1-day laboratory ageing. The study thus suggests that from the point of view of ITS comparison, 1 year in the field corresponds with 1 day of ageing in the drying room.

3.2 Simulation of ageing in laboratory conditions

We strive to grasp the entire process of asphalt mix ageing and identify the causes thereof. Bituminous binders react with oxygen under higher temperatures during production in a mixer (short-term ageing). Once the asphalt layer has been laid, the binder continues reacting with atmospheric oxygen (long-term ageing). Laboratory simulations of both short- and long-term ageing are described e.g. by AASHTO R 30-02. The bulk asphalt mix is put in a dryer set to 154 °C for 2 hours or to 135 °C for 4 hours to simulate short-term ageing. Long-term simulation consists of leaving the compacted test specimens in the dryer for 120 hours (5 days) under 85 °C. European countries are currently working on an analogous standard, prEN 12697-52, where the procedure for short-term ageing does not differ from the US standard (bulk asphalt mix at 135 °C for 4 hours). For the purposes of long-term ageing, the bulk asphalt mix is tempered at 60 °C for 14 days or at 85 °C for 9 days. To accelerate ageing, bulk asphalt mix may be put in a Pressure Ageing Vessel (PAV) for 20 hours at 90 °C. The European standard allows also the ageing method where compacted specimens are tempered for 5 days at 85 °C. For the sake of comparison, in this study two types of long-term laboratory ageing are considered: (a) bulk asphalt mix at 85 °C for 9 days and (b) tempering test specimens at 85 °C for 5 days.

One of the main aspects tested within the study focused on verification of the long-term effects of selected adhesion promoters in the asphalt mix and the ways they are affected by the selected method of long-term laboratory ageing. To verify the impact of adhesion promoters, unaged asphalt mixes were prepared to allow a comparison.

For the sake of a simple comparison, one set of test specimens was put in a heating chamber while another set had a protective collar created by a PVC case and tightening straps which prevented deformation caused by temperature. In the case of the last set of test specimens, the PVC case was later replaced by a sturdy metal mesh intended to better simulate the real conditions, i.e. hot air access not only from the bottom and top part of the specimen but also from the sides to ensure even ageing of the test specimens.

4. INPUT MATERIAL ANALYSIS AND MIX DESIGN

4.1 Input materials

Within this study straight-run bitumen 50/70 fulfilling requirements of CSN EN 12591 was used. For particular tests – mainly the bitumen-aggregate adhesion test according to CSN 73 6161 – several aggregate types have been selected different in their mineralogy. These aggregates are normally used in the Czech Republic for asphalt mix production. Their specification is summarized in Table 1. Based on more detailed analysis one aggregate type was later experimentally selected and used for laboratory mix design and moisture susceptibility tests.

Table 1. Basic characterization of used aggregates

Quarry	Mineralogy	Short description
Markovice	Amphibolite	Fine-grained metamorphic mineral, grey color.
Chlum	Clinkstone	Extrusive volcanic mineral of phorphyric origin, grey color.
Libodřice	Amphibolite	Medium grained mineral partially honeycomb formed with shiny particles, grey color
Litice	Spilite	Extrusive, metamorphic mineral of green-grey color.
Kobylí Hora	Granulite	Granulite of grey-greenish color with appearance of tourmaline a biotite.
Měrunice	Basalt	Grey to black eruptive rock.
Zbraslav	Mixed rock (ash rock, metatuf, spilite)	Slightly metamorphic, mainly pelitic and psamitic rocks, minerals of acidic and alkali minerals.
Zbečno	Spilite	Sedimentary rock with higher content of shale, silt and greywacke.

To guarantee durability of the mix, some types of aggregate are more or less suitable from the point of view of adhesion between the bitumen and the aggregate. Therefore, selected adhesion promoters to ensure good quality bond to the aggregate particle – binder interface – were chosen experimentally. In this case, the adhesion is active. The aggregate and bituminous binders are two different materials which often have opposite surface properties; therefore, adhesion depends on a close bond between the two materials. To meet the condition of active adhesion, the aggregate must be free of any water to achieve perfect adhesion to the bituminous binder. In contrast to the bitumen, aggregate is often hydrophilic; this means that it adsorbs water easily. The following adhesion promoters were used on the basis of the aforementioned assumptions: IP chemical additive dosed to 0.30 %-wt. of binder, AH chemical additive dosed to 0.30 %-wt., ZT nano-chemical additive dosed to 0.10 %-wt., WF chemical additive dosed to 0.3 %-wt. of binder. Last but not least mechanical-chemically activated micro-filler originating from the Palestine limestone sludge was chosen to act as an intelligent replacement for traditional fillers. It is a dehydrated sludge from limestone marble cutting, further modified by pulverizing in a special type of high-speed grinding machine (disintegrator).

4.2 Mix design

For the purposes of assessing the effect of ageing on adhesion promoters, from the perspective of adhesion and moisture susceptibility, mix AC_{bin} 16 intended for the binder course was designed. Aggregate fractions 8/16, 4/8 a 0/5 originated from the Chlum quarry, sand 0/4 was obtained from Sojovice. The individual fraction representations are indicated in Table 2 while the requirements for individual sieve fractions according to CSN EN 13108-1 were met. Marshall test specimens were prepared according to standard CSN EN 12697-30+A1.

Table 2. Mix design for asphalt concrete AC_{bin} 16 (according to EN 13108-1)

Mix components	Content (%)	Content in the asphalt mix (%)
Crushed aggregate Chlum 8/16	37.0	35.08
Crushed aggregate Chlum 4/8	16.0	15.17
Crushed aggregate Chlum 0/5	30.0	28.44
Sand Sojovice 0/4	10.0	9.48
Limestone filler Velké Hydčice	7.0	6.64
Straight-run bitumen 50/70	-	**5.20**

5. TEST DEFINITION

5.1 Determining adhesion between the binder and aggregate

In the Czech Republic, non-harmonised test according to CSN 73 6161 has been used to describe the quality of bitumen to aggregate adhesion; it uses a sample of pre-heated aggregate, 8/16 mm or 8/11 mm fraction, weight of 300±3 g and binder to the quantity of 12±0.3 g at a temperature depending on the bitumen gradation (in this study the aggregate temperature was 160±5°C and the bitumen temperature amounted to 170±5°C). After manual mixing of both components the mix was put in a glass vessel and equally leveled. Subsequently, after cooling the sample down for one day, the sample was conditioned in water at 60±3°C for 60 minutes. When the sample has been removed from the water, the coating of aggregate particles by the binder is assessed visually. Under ČSN 73 6161, bitumen to aggregate adhesion is evaluated using a similar system as in the case of method B given in EN 12697-11:

- excellent – if >75% particles of the test sample have the A characteristic of bitumen film bond with aggregate showing minimum stripping; the characteristic may not be lower than B for the remaining particles;
- good – if >75 % coated aggregate particles of the test sample have the B (or better) characteristic of bitumen film bond with aggregate; the characteristic may not be lower than C in the remaining grains;
- satisfactory – if >75 % particles of the test sample have the C (or better) characteristic; the characteristic may not be lower than C in the remaining particles;
- unsatisfactory – if <75 % particles have the C characteristic, [3].

5.2 Determining the test specimen moisture susceptibility

The determining moisture susceptibility test was conducted in compliance with standard EN 12697-12. The essence of this test is dividing test specimens prepared for each assessed asphalt mix into two groups of identical size. One group is kept at laboratory temperature without any further conditioning. The other group is saturated by water and put in a water bath of a higher temperature. In the case of this study nine test specimens were made for each mix and they were divided in the following manner: the first and second groups contained 3 specimens each; the remaining three test specimens were used to determine the moisture susceptibility of the asphalt mix with a single frost cycle as described in paragraph 5.3. After tempering, indirect tensile strength was determined for each of the three groups according to EN 12697-23 under the stipulated test temperature. The ratio between indirect tensile strength of the water-saturated test specimen group and test specimens kept at laboratory environment is known as ITS ratio.

Dry (unsaturated) test specimens were stored on a straight surface under laboratory temperature of (20±5) °C. The second group was saturated in a vacuum vessel under the pressure of (6.7±0.3) kPa for (30±5) minutes. These specimens were then put to a water bath of (40±1) °C for (70±2) hours. When the time was up, both groups of specimens were stored in a climatic chamber at (15±1) °C for at least two hours; indirect tensile strength was determined immediately afterwards. The ITS ratio for the group of wet and dry test specimens [4] was determined according to the formula:

$$ITSR = 100 \times \frac{ITS_w}{ITS_d}$$

ITSR – indirect tensile strength ratio [%];
ITS_w – average indirect tensile strength of water saturated test specimens [kPa];
ITS_d – average indirect tensile strength of dry test specimens [kPa].

5.3 Determining the moisture susceptibility of test specimens with one freezing cycle

The moisture susceptibility test of specimens with one freeze cycle was conducted in compliance with the US technical standard AASHTO T 283-03. Again, the essence of the test is comparing the properties of two groups of compacted asphalt mix specimens where the first group is left dry and the other group is water saturated and subsequently one freezing cycle is applied. The American method is based on the principle of test specimen compaction described e.g. in the California methodology by a roller compaction (AASHTO T 312), or in the Superpave methodology using a gyratory compactor (AASHTO T 247), to achieve the required void content of (7.0±0.5) %. When this test method is applied for asphalt mixes in the Czech Republic, the test specimens were compacted by 2x25 impacts by the Marshall compactor which constitutes a partial modification of the US test method.
The dry test specimens (group one) were taken out of the moulds and put on an even surface under laboratory temperature (20±5) °C for (24±3) hours. Subsequently, the test specimens were placed in a climatic chamber with a temperature of (15±1) °C for at least two hours. Afterwards, the specimens were tested. The other group (water saturated test specimens with one freezing cycle) were placed on a perforated mat in a vacuum vessel. Each specimen was then put in a plastic bag with (10±0.5) ml water; the bag was then sealed tightly and placed in the freezer at (-18±3) °C for at least 16 hours. After that time, the specimens were taken out of the freezer and the plastic bags were taken off. The test specimens as such were then put in a bath containing distilled water at (60±1) °C for (24±1) hours. After this conditioning the specimens were transferred to a water bath with regulated temperature of (15±1) °C. Immediately after the aforementioned period, the indirect tensile strength test could be conducted. [5]
Indirect tensile strength and the ITS ratio were determined in a same way like described in 5.2.

6. RESULTS DELIVERED BY THE RESEARCH

6.1 Evaluation of the adhesion test

The test of adhesion between bitumen and aggregates was conducted and evaluated according to paragraph 5.1. The test involved 8 different types of aggregate and bitumen 50/70 improved subsequently by chemical adhesion additives: AH, ZT, IP or WF. To verify the effects of adhesion promoters and the effect of storage time, the variants of binders were subjected to short-term ageing (TFOT) according to CSN EN 12607-2. The binder was exposed to ageing effects for three times 5 hours at 163 °C. Table 3 summarises the results of unaged binder which indicates the results of one version with reference binder only and the results of a combination with a selected adhesion promoter for each aggregate type. The results were assessed as unsatisfactory for the binder with no additives and aggregate from Chlum (clinkstone) and Zbraslav (mixed rock: ash rock, metatuf and spilite). The worst results were recorded for aggregate from the Kobylí hora quarry (granulite), where only 35 % of the aggregate remained coated by the binder after the adhesion test. The aggregates from Markovice, Libodřice, Měrunice and Zbečno scored satisfactory evaluation according to the results; the percentage of aggregate particle coating amounted to roughly 80 %. The best result was scored when aggregate from the Litice quarry (spilite) was used; in this case the surface of the aggregate was stripped in approx. 10 %, this means good affinity between the binder and the aggregate. The positive effect of the additives could be proven for all types of aggregate. On average, the percentage of coated surface of the aggregate increased by 10 % when each individual additive was applied. The additives had the greatest effect on aggregates from Kobylí Hora, Chlum and Zbraslav, which failed the adhesion test without an additive. In these cases the adhesion improved by 20-30 %. With the remaining specimens, adhesion improved as well with the only exception of the aggregate from Litice, which scored excellent results even without the additive. A higher dose of additives resulted in a slight improvement of adhesion of the individual aggregate variants; or at least the adhesion did not deteriorate. The only exception was the higher dose of adhesion additive AH, where adhesion deteriorated; however, a repeat measurement was recommended for this case.
Focusing on the comparison of the aged binder, the results are arranged logically (Table 4) as in the preceding case. As is obvious from the measurements and can be expected, ageing has a positive effect on the adhesion of bituminous binders to aggregate. Based on the majority of results, it can be noted that the level of aggregate particle coating by bitumen improves, or the remaining specimens do not demonstrate a deterioration of adhesion. In the case of the

reference sample with bitumen 50/70, using the aggregate from Kobylí Hora and Zbraslav, the coating level improved due to the influence of ageing time. In contrast to unaged binder, it is visible that the level of coating is satisfactory or, excellent, in the case of aggregate from the Chlum quarry.

Table 3. Results of adhesion test according to CSN 73 6161 for unaged bitumen

Aggregates	50/70 (reference)			50/70 + 0.3% AdHere 65-00			50/70 + 0.6% AdHere 65-00			50/70 + 0.3% Impact 8000		
Markovice	B-C	85%	Satisfactory	B	90%	Very good	B-	87%	Satisfactory	A-B	95%	Very good
Litice	B	90%	Very good	B-C	85%	Satisfactory	C-D	75%	Satisfactory	B-	87%	Satisfactory
Libodřice	C	80%	Satisfactory	A -	97%	Excellent	C+	83%	Satisfactory	A-	97%	Excellent
Chlum	D +	73%	Unsatisfactory	C	80%	Satisfactory	A-B	95%	Very good	C-	77%	Satisfactory
Kobylí Hora	E	50%	Unsatisfactory	C	80%	Satisfactory	C-D	75%	Satisfactory	C	80%	Satisfactory
Měrunice	C -	77%	Satisfactory	B -	87%	Satisfactory	A-B	95%	Very good	A-B	95%	Very good
Zbraslav	D	70%	Unsatisfactory	B -	87%	Satisfactory	C+	83%	Satisfactory	B-C	85%	Satisfactory
Zbečno	C	80%	Satisfactory	C +	83%	Satisfactory	B-C	85%	Satisfactory	B-C	85%	Satisfactory

Aggregates	50/70 + 0.6% Impact 8000			50/70 + 0.1% Zycotherm			50/70 + 0.3% Wetfix BE		
Markovice	B-	87%	Satisfactory	B +	93%	Very good	B-	87%	Satisfactory
Litice	B-	87%	Satisfactory	B -C	85%	Satisfactory	B-C	85%	Satisfactory
Libodřice	C+	83%	Satisfactory	A-B	95%	Very good	B	90%	Very good
Chlum	C	80%	Satisfactory	C -	77%	Satisfactory	C	80%	Satisfactory
Kobylí Hora	B-C	85%	Satisfactory	C	80%	Satisfactory	C	80%	Satisfactory
Měrunice	B	90%	Very good	B +	93%	Very good	B-C	85%	Satisfactory
Zbraslav	B-C	85%	Satisfactory	C +	83%	Satisfactory	B-C	85%	Satisfactory
Zbečno	B-C	85%	Satisfactory	B -C	85%	Satisfactory	B-C	85%	Satisfactory

Table 4. Results of adhesion test according to CSN 73 6161 for aged bitumen

Aggregates	50/70 (reference)			50/70 + 0.3% AdHere 65-00			50/70 + 0.6% AdHere 65-00			50/70 + 0.3% Impact 8000		
Markovice	B	90%	Very good	A-B	95%	Very good	B+	93%	Very good	A-B	95%	Very good
Litice	C-	77%	Satisfactory	C	80%	Satisfactory	C	80%	Satisfactory	B	90%	Very good
Libodřice	B-C	85%	Satisfactory	B-	87%	Satisfactory	B-	87%	Satisfactory	B	90%	Very good
Chlum	A-	97%	Excellent	A-B	95%	Very good	B	90%	Very good	C	80%	Satisfactory
Kobylí Hora	C-	77%	Satisfactory	C+	83%	Satisfactory	C	80%	Satisfactory	C-D	75%	Satisfactory
Měrunice	B	90%	Very good	B-	87%	Satisfactory	B-C	85%	Satisfactory	B	90%	Very good
Zbraslav	B-C	85%	Satisfactory	B-C	85%	Satisfactory	C+	83%	Satisfactory	C+	83%	Satisfactory
Zbečno	C	80%	Satisfactory	B	90%	Very good	B-C	85%	Satisfactory	B-	87%	Satisfactory

Aggregates	50/70 + 0.6% Impact 8000			50/70 + 0.1% Zycotherm			50/70 + 0.3% Wetfix BE		
Markovice	B+	93%	Very good	B	90%	Very good			
Litice	A-	97%	Excellent	B	90%	Very good			
Libodřice	A-B	95%	Very good	C+	83%	Satisfactory			
Chlum	B-C	85%	Satisfactory	C	80%	Satisfactory			
Kobylí Hora	C+	83%	Satisfactory	B-C	85%	Satisfactory			
Měrunice	B	90%	Very good	B	90%	Very good			
Zbraslav	B	90%	Very good	B-C	85%	Satisfactory			
Zbečno	B-	87%	Satisfactory	B	90%	Very good			

6.2 Evaluation of moisture susceptibility tests

The study involved the preparation of one set of test specimens with the reference binder as well as 6 variants of asphalt mixes with additives to improve adhesion. Selected laboratory ageing methods were evaluated in all variants of mix AC_{bin} 16; a set of unaged specimens was prepared for the sake of comparison. A simple comparison of indirect tensile strengths compared the effect of individual variants of adhesion improvement both prior to ageing as such and after the selected simulations of ageing of test specimens or bulk asphalt mix.

6.2.1 Comparison of indirect tensile strengths

The following figure 1 compares the indirect tensile stresses of test specimens left in air, both for the unaged asphalt mix versions and for selected laboratory ageing variants.

Focusing on the first columns of individual variants of the mixes unaffected by ageing, the following conclusions are suggested by the results. The reference mix reached the indirect tensile strength of 2.39 MPa. The mix variant using a mechanical-chemically activated micro-filler scored almost the same result as the reference mix. The diagram also shows that the application of adhesion promoters decreased the indirect tensile strength in comparison to the reference mix. The greatest reduction by 0.48 MPa in comparison to the reference mix was observed for the variant with ZT additive and in the case of a higher dose of IP additive. However, the difference could have been caused by insufficient tempering of the test specimens. As suggested by the latest results of a partial research just completed, testing indirect tensile strength at different temperatures showed an increase of the strength on average by 17.5 % if the test temperature was by 2 °C above the prescribed temperature of 15 °C; on the other hand temperature by 2 °C below the prescribed value reduced the strengths by 3.8 %. Even the slightest change in temperature affects the results as such.

The second columns (black shading) represent the ITS of test specimens where the first method of laboratory ageing was applied. The method consisted of preparing Marshall test specimens which are then put in a climatic chamber with forced circulation at 85 °C for 5 days. As is obvious from the results, strength of the reference mix and of mix with a higher dose of AH additive scored lower strength than unaged mix; for the remaining mixes, the indirect tensile strengths increased which was one of the assumptions. Comparing the results of aged test specimens, the mix with ZT additive and micro-filler demonstrate higher strengths than the reference mix (by 0.28 MPa and 0.40 MPa, respectively). The remaining values are similar, or lower on average.

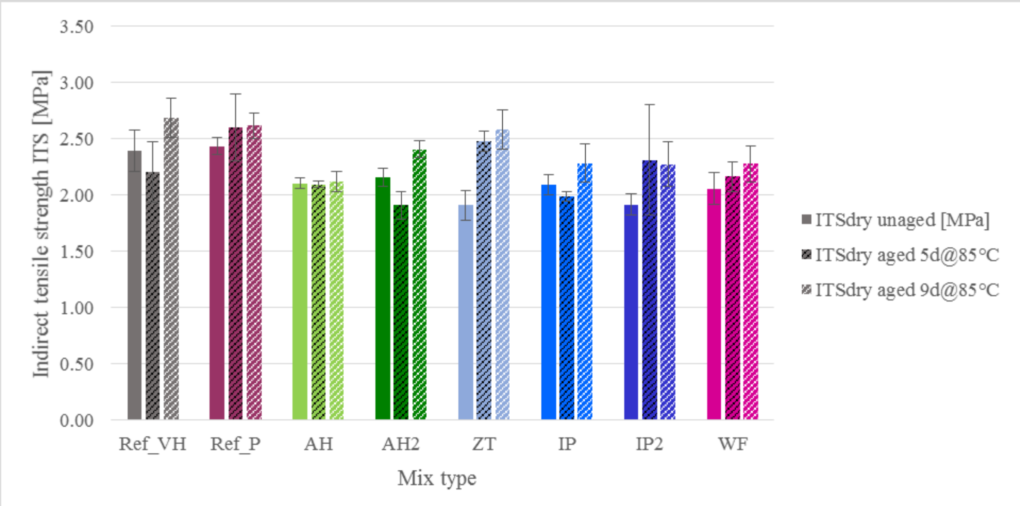

Figure 1: Comparison of indirect tensile strengths in specimens left in air

The last columns (white shading) indicate the results of the second method of laboratory ageing where the asphalt mix was distributed evenly on a pre-prepared mat where the mix was aged in the climatic chamber with forced circulation at 85°C for 9 days. When the time was up, test specimens were prepared with the mix on the Marshall compactor (2x25 impacts) and subsequently used to determine moisture susceptibility. The last series of results shows that the reference mix had the highest indirect tensile strength of 2.68 MPa, the mix with micro-filler and ZT additive scored almost identical result. For the other mixes, the results of indirect tensile strength were lower when compared to the reference mix. When the variants were compared to one another, it is obvious that the ageing of bulk asphalt mixes affects the subsequent test specimen compaction and strengths in comparison to unaged test specimens where they score 10 % more (or, in the case of ZT about 35 %). The ageing of test specimens as such does not demonstrate the same tendencies in all cases which might be caused by the laboratory ageing method applied where the specimens are placed in a climatic chamber at higher temperature; this might cause deformation and the specimens may lose the original volume.

6.2.2 Evaluation of moisture susceptibility of test specimens, including the one freezing cycle option

The effect of adhesion promoters on asphalt mix resistance to negative effect of water was compared for all seven types of the mixes designed. The results of the testing according to EN 12697-12 are show in Figure 2 for unaged mix, in Figure 3 for aged test specimens (5 days at 85 °C) and in Figure 4 for aged bulk asphalt mix (9 days at 85 °C). The results were viewed from several perspectives. The first question concerned the effect of the adhesion promoter applied from the point of view of moisture susceptibility, i.e. whether the ITS ratio improves or not. It was also assessed whether the indirect tensile strength ratios meet the minimum required value for ITSR as stipulated by the national annex to standard CSN EN 13108-1 for asphalt mixes AC_{bin} (at least 80 %). If evaluating test specimens exposed to adverse effects of water with one freeze cycle neither US standard AASHTO T 283-03 nor the national annex to standard CSN EN 13108-1 stipulates a minimum ITSR threshold. For the purposes of experimental comparisons, such threshold was set to 70 % within the study.

When comparing indirect tensile strengths of unaged specimens (see Fig. 2) it can be noted that the specimens exposed to adverse effects of water reached the threshold of ITSR = 0.80 (according to CSN EN 13108-1) in almost all variants. Focusing on comparison of individual variants, the mixes with higher content of AH additive recorded a reduction in ITSR to 0.65. Contrary to the expectations, the results demonstrated that in this case, the application of adhesion promoter had no impact on improving moisture susceptibility in comparison to the reference mix. The conclusions are confirmed by the values measured in test specimens exposed to adverse effects of water, including the freezing. In that case, the ratio of indirect tensile strengths was set to 0.79 for the reference mix; the same was achieved by the variants with micro-filler and ZT or WF additive. For the remaining variants, the indirect tensile strength ratio decreased almost to 0.70 % which was determined as the threshold.

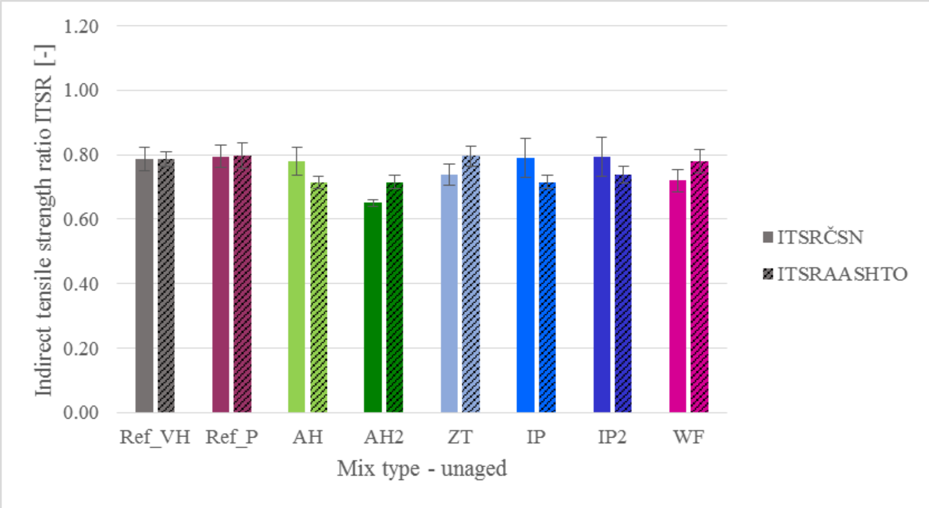

Figure 2: Results of moisture susceptibility test; unaged asphalt mixes

Figure 3 gives an overview of the ITSR results for test specimens exposed to ageing for 5 days at 85 °C. Focusing on the ITSR according to CSN EN standard, it can be noted that the best results were scored by the variant with a higher content of AH additive. The ITSR amounts to nearly 0.98 while a similarly high value was achieved by the AASHTO method although the method is based on less favourable conditions affecting the asphalt mix under observation. The mix option with micro-filler reached a 0.85 limit which meets the minimum threshold required. Out of the remaining mixes, the variant with a lower proportion of AH additive and the variant with Impact come close to the threshold. It is obvious that a higher dose of the additive with the aggregate concerned has no potential to improve moisture susceptibility as is also confirmed by the results according to AASHTO. The poorest results in ITSR were recorded by the asphalt mix with WF additive where the ratio came to 0.37 for the European approach and the ratio according to the American test method came to 0.29, i.e. almost fifty per cent less than the value of the reference mix. The aforementioned result is at least noteworthy; it might point out some restrictions of the test method. When compared to WF additive, the variant with ZT additive scored better (0.52 vs. 0.40); however, the mixes do not meet the required minimum ITSR thresholds anyway.

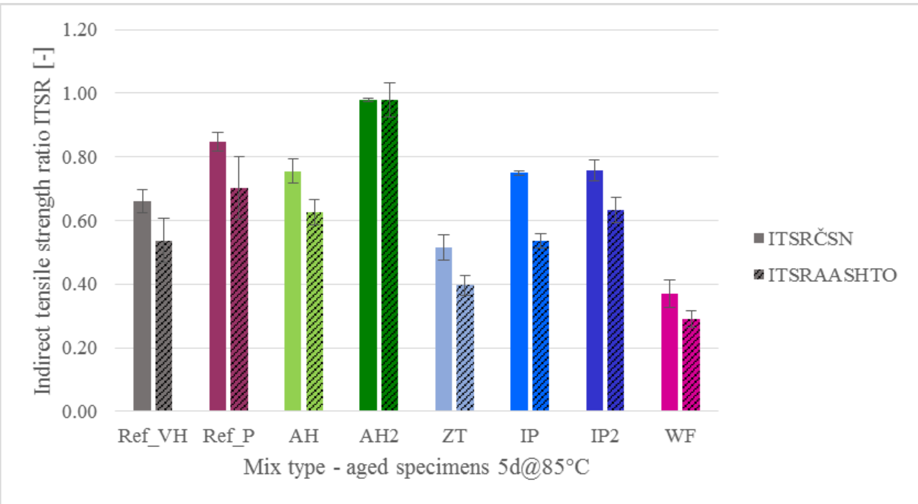

Figure 3: Results of moisture susceptibility test; aged asphalt test specimens 5d @85°C

Focusing on the findings for test specimens made of aged bulk asphalt mix, the results in Fig. 4 are of interest. The moisture susceptibility results determined according to CSN EN methods and the US test method yield almost identical ITSR values, despite the fact that the latter method exposes the test specimens to less favourable conditions. When compared to the reference mix where the ITSR amounts to 0.71 and 0.68, we can note that neither Impact 8000 in a content of 0.3 and 0.6 %, nor WF additive delivered any improvement in adhesion; the ITSR ratio amounts to 0.60 on average. An improvement was recorded in the case of micro-filler application and for AH additive content of 0.3 %. In these cases, the ITSR amounts to roughly 0.90. Higher content of AH additive resulted in further improvements.

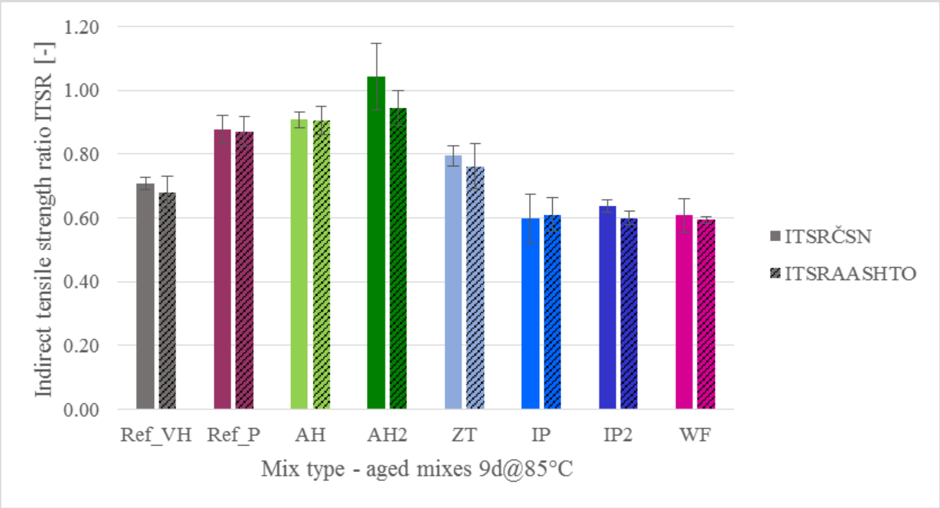

Figure 4: Results of moisture susceptibility test; specimens from aged bulk asphalt mix 9d @85°C

7. DISCUSSION

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8. CONCLUSION

The issue of adhesion between aggregate and bituminous binders as well as the closely related aspect of durability tested by asphalt mix resistance to water remains one of the longest studied asphalt mix phenomena. Similarly to the ageing of bituminous binders and asphalt mixes, we still cannot describe and verify all phenomena and causes affecting the adhesion as such with clarity and certainty since there are both physically-mechanical and chemical aspects of the issue as such. Moreover, if the two phenomena are combined they allow us to reflect the real-life conditions much better; however, the problem becomes more complex at the same time.

The presented study delivered results which make it obvious that the adhesion test as such provides quick information on the quality of aggregate particle coating by bitumen; the disadvantage is its considerable dependence on the evaluator's subjective impression and the fact that the method applied is quite difficult to relate to any real-life effects in the pavement structure. The subjective evaluation aspect can surely be eliminated by more exact and more demanding methods (e.g. determining the contact angle or surface free energy), the problem of making the test conditions more realistic should be taken into account at least by considering the ageing aspect, but also by the possible effects of not only the water-higher temperature combination but a more complex water-frost-temperature-time system, too. Due to that, the aim was to take into account at least the time (ageing) aspect in the partial assessment.

For the bitumen-aggregate adhesion assessment it is recommended:

(a) to use test methods eliminating subjective evaluation;
(b) to use modified ageing test protocols (e.g. 3x RTFOT or 3x TFOT) to prove long-term stability and functionality of adhesion promoters.

The issue of asphalt mix ageing and water susceptibility is similarly complex. Firstly, we have to say that regardless of the interpretation of what period is simulated by the specific ageing method, it is appropriate to assess aged asphalt mixes. The effect undoubtedly occurs in the pavement structure and it is unimportant, from the perspective of life and effect of the adhesion promoters, what occurs e.g. during the first year; the important aspect is how effective it will be on the overall behaviour of the mix after 5 or more years. Following conclusions can be made:

(a) Comparing unaged mixes and the two options with ageing either bulk mix or test specimens the used adhesion promoters seems to show different behaviour in each of the cases.
(b) The ageing of compacted test specimens according to the test method defined in prEN 12697-52 is probably better in simulating the real conditions in the pavement although, on the other hand, the level of compaction of the asphalt mix within the pavement is higher than the selected approach with 2x25 blows which is considered for the purposes of simulating accelerated water impact. The results showed better a difference between the tested mix variants, nevertheless, the question still is if this can be related to the different effect of the used additives.
(c) If Marshall test specimens aged it is recommended to encase them preferably by sturdy metal mesh to avoid their deformation.
(d) In the case of bulk asphalt mixes, the results seemed more consistent; this approach to ageing is simpler to perform and the test specimens are not deformed at all. On the other hand, the bitumen film ages evenly throughout the mix; this does not reflect the likely situation of a compacted asphalt layer. Therefore, what is simulated by this approach is a matter of interpretation. Last but not least, particularly in areas where cold and

hot climates alternate, a change of the existing methodology of moisture susceptibility evaluation must be supported and a freezing cycle added to the test as this has an impact on the pavement structure and affects the durability of the asphalt mix.

Finally we wish to emphasise that this study did not intend to classify the effectiveness of various additives or compare pulverized limestone and chemical additives at all. Therefore, no attention was paid to the chemical composition of individual additives and to the fact whether additives with similar proportions of active ingredients were chosen. For such purposes, the study would have had to be defined in a different manner. Therefore, it would be wrong and contrary to our approach to stipulate any conclusions on the advantages of individual additives based on this study. The selected test methods clearly show that such an evaluation is ambiguous at least.

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