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MECHANICAL AND IMAGE ANALYSIS OF ADHESION BETWEEN MINERAL AGGREGATE AND BITUMINOUS BINDER

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Abstract. Asphalt mixtures often fail due to poor interaction between mineral aggregate and bituminous binder. Therefore, many efforts are being made on adhesion improvement between the two materials. In this work, paving grade bitumen 50/70 was doped with two types of adhesion promoters. Asphalt mixtures composed of crushed aggregate Brant coated by binder were made and exposed to stripping water. Then, they were subjected to visual and digital image analysis aiming to quantify aggregate residual bitumen-coated areas. Besides, two cylindrical samples of aggregate were bonded together by a thin film of bitumen doped with adhesion promoters. After solidification of binder, force needed for separation of the two parts was measured. It was shown that residual bitumen-coated areas were increased by 20-30 % by adding adhesion promoters into binder. Mechanical adhesion of thus modified binder to aggregate was increased by up to twice.

Keywords: Bituminous binder, adhesion, adhesion promoters, pull-off tests, image analysis.

1. Introduction

Asphalt mixtures used in construction of road pavements are composite materials consisting of two main phases: mineral aggregate and bituminous binder. Adhesion between them is the key parameter which is responsible for road mechanical properties as well as lifetime. If adhesion is insufficient, the two materials can be disintegrated. Consequently, pavement structure is thus damaged with potholes [1]. Physicochemical interaction between binder and aggregate is complex process based on adhesive bonds. These are influenced by bitumen chemical composition (especially presence of polar components), temperature of materials used, and surface properties of aggregate [2, 3].

It has to be also taken into account that adhesion between binder and aggregate, alongside the parameters mentioned above, is strongly influenced by water. Most of aggregates used for production of road pavements are composed of hydrophilic acid-based minerals like granite and quartz. These exhibit strong affinity for water, higher than for bitumen [4, 5].

Given the fact that aggregate of the most frequented hot-mix asphalt mixtures is during production of asphalt mixtures dried and preheated to high temperatures often exceeding 150 °C [6, 7], the presence of internal water (water contained in row aggregate) can be practically excluded. However, the exposition to external water, ie., water entering pavements because of poor drainage or highly wet subgrade is unavoidable. Consequently, bitumen is thus replaced by water due to its low interaction with aggregate, followed by stripping effect – an adhesion failure between bituminous material and aggregate surface [4, 5].

The whole process of so-called asphalt moisture damaged is illustrated in Figure 1. The figure a) shows theoretical ideal state: aggregate is perfectly coated by thin film of bitumen and is thus protected against water penetration. However, the real state is depicted in figure b) where the continuous layer of bitumen is damaged due to either poor coating process or abrasion against other aggregate particles. Because of traffic loads and phenomenons described earlier, water gradually penetrates the aggregate through the bitumen coat defect and thus separates the bitumen thin film from aggregate surface [8, 9].

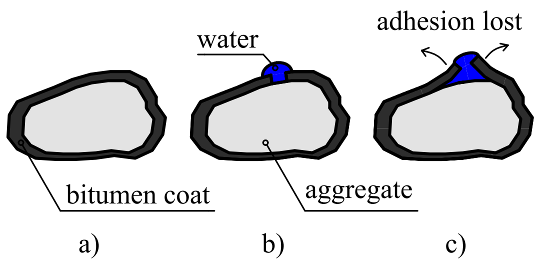

Figure 1. Aggregate coated by bitumen thin film, a) ideal state, b) real state, and c) stripping due to water.

To avoid stripping, different types of adhesion promoters at the form of liquid additives can be added into bituminous binder. The role of such chemical surfactants is to maximize the formation of intermolecular interactions between bitumen and aggregate surface [4, 5] and to reduce aggregate hydrophilicity. These are usually based on alkylsilanes, phospho-

Mark	Binder	Adhesion promoter	Admixture amount [wt. %]
R	50/70	-	0
M1	50/70	unsaturated fatty acids with diethanolamine	0.2
M2	50/70	phosphorus-based	0.3

Table 1. Summarization of three variants of bituminous binder 50/70 used.

rus, chemical surfactants comprising amines, or unsaturated fatty acids combined with diethanolamine. Their amount differed from 0.2 to 0.4 % of binder weight. The choice of a suitable additive depends on the origin of used aggregate, the type of binder, and temperature of asphalt mixture preparation [10, 11].

With using of adhesion promoters, a new question about their efficiency arises: How do they improve adhesion between the two materials? Generally speaking, two evaluating basic methods are known [12]: (i) loose asphalt mixture tests and (ii) mechanical analysis. The first of them is based on subjective visual or digital image analysis [13, 14]. Asphalt mixture is made according to relevant technical standard [6, 15] and then exposed to hot stripping water or abrasion for certain period. After that, the stripped aggregate areas are quantified and assessed. These methods are indirect and it is therefore clear that adhesion between the two materials can not be quantified from the mechanical point of view. The second mentioned method – mechanical analysis – is then divided into two subcategories as indirect and direct. Indirect testing is based on mechanical behavior evaluation of asphalt mixture specimens. In recent technical practice, we are talking primarily about tensile strength tests of prismatic or cylindrical asphalt mixture specimens. The durability and resistance of compacted specimens is checked before and after water exposition [16]. It is obvious that the indirect testing can be influenced by many variable factors, for example by mixture inhomogeneity, aggregate shape, etc.

The all shortcomings listed above can be overcome using the direct mechanical adhesion analysis. Aggregate-bitumen bonds can be determined by means of peel or pull-off tests. In case of the first mentioned, polymeric or metal belt is bonded using bitumen as the adhesive thickness-controlled layer on preheated prismatic sample of aggregate. After cooling of the specimen and solidification of bitumen, the belt is peeled off under angle of 90° (to aggregate surface plane), while the fracture development is observed [17, 18]. Pull-of tests are based on the same general principles. Thin layer of bitumen is sandwiched between two cylindrical aggregate samples. One part of aggregate is after technological pause pulled off, aiming to evaluate interfacial bond strength between the two materials [18]. Such a method seems to be the most effective of all mentioned, because it combines only two main materials and provides the most valuable data from the perspective of interface interaction.

Although the pull-off method is not widespread in the Czech Republic, we decided to use it in order to determine bond strength between mineral aggregate and differently modified bituminous binder. To extend such obtained results, we made asphalt mixtures and subjected them to standard loose asphalt mixture tests.

2. Materials

2.1. Aggregate and bituminous binder

For purposes of this study, aggregate from Brant quarry was chosen. It is composed primarily of granite porphyry, or porphyry microgranite (quartz, potassium feldspar, and mica rocks), therefore it is hydrophilic and thus more susceptible to water damage, with poor adhesiveness to binder and elevated potential of stripping failures. Its surface can be characterized as porous and its composition as relatively homogeneous. Paving grade bitumen 50/70 was used, standardized by [19].

2.2. Adhesion promoters

To ensure stronger adhesive bonds between both materials, two types of liquid-form chemical-based additives were used. These were applied at the trace amount of 0.2 and 0.3 wt. % of bitumen according to recommendations of their manufacturers. Reference and two modified binder variants were used, as summarized in Table 1. In the case of mixture marked as M1, reactive products of unsaturated fatty acids with diethanolamine were used. The additive can be characterized as brown viscous fluid, which is recovered by condensation of unsaturated fatty acids at high temperature with disposal of water. The acids are dissoluble in ethanol and acetone and can be emulsified in water. In the bituminous binders, such additives are soluble at standard blending/mixing temperatures. The additive according to the producer shall be resistant to long-term heating (150 hours) at 150 to 160 °C without any changes in its activity. The second mixture, M2, was doped with a new generation of phosphorus-based adhesion promoters. This type is specific due to versatile application and improved thermal stability allowing to resist higher temperatures.

2.3. Sample preparation

Cylindrical specimens having 42±0.2 mm in diameter were drilled from block of aggregate using table grill equipped with diamond tipped tile drill bit. Then, such obtained samples were cut off to 25 mm length using saw Struers Secotom-15 with diamond cutting blade to ensure perfectly planar cut, perpendicular to longitudinal axis of the specimens. Two thus obtained specimens were then preheated to 150±5 °C and sandwiched with 1 mm thick layer of the bitumen with the same temperature. The thickness of the bitumen layer was ensured by applying its properly weighed amount. After that, the sandwiched specimens were tempered to 15±1 °C.

According to [6], crushed aggregate of fraction 8/16 mm (in sum ca. 300 g, temperature 160±5 °C) was coated by the bituminous binder (12±0.3 g, temperature 170±5 °C). After 24 hours of samples storage, these were submerged into stripping water having 60 °C for following 60±5 minutes. Then, they were subjected to visual and digital image analysis as described in separate paragraph below.

3. Experimental methods

3.1. Pull-off tests

Sandwiched cylindrical specimens, 5 pieces for each mixture, were fixed to clamping chuck composed from two mirror half circles. A space between the chucks and testing specimens was filled with rubber seal in order to ensure strong adhesion between them. Then, thus fixed specimen was placed to steel cage ensuring its direct guiding during loading. The cage is movable in the direction of specimen axis. It is equipped with tensile sticks from the both sides. These are used for fixing the whole construction in a loading frame, as captured in Figure 2. Sandwiched specimens were loaded through the construction by tension using a loading frame MTS Alliance RT/30. The experiment was displacement controlled at the constant rate of 0.5 mm/min.

Figure 2. Construction for pull-off tests fixed in loading frame. In the middle, there is a sandwiched specimen placed.

3.2. Visual and image analysis

Following [6], the conventional visual analysis was based on subjective assessment of asphalt mixtures by two evaluators, who classified the rate of stripped areas of aggregate.

In order to minimize "human factor" and inaccurate evaluators assessment, semi-automatic digital image analysis was employed. Asphalt mixtures were captured using DSLR camera Canon EOS 6D (full-frame sensor) equipped with lens Canon EF 100mm f/2.8L Macro IS USM. Such device allowed to capture even insignificant details being necessary for proper assessment. The samples were lighted by daylight only. Consequently, thus obtained images were subjected to evaluating software enabling entropy-based image segmentation. Thanks to local entropy calculation, the software is capable to assess roughness of the texture. The areas with high entropy are considered to belong to aggregates while low entropy areas represent the bituminous matrix [14].

4. Results and discussion

4.1. Pull-off tests

Force as a function of displacement obtained during pull-off testing is for all three types of sandwiched specimens shown in Figure 3. All curves were calculated as arithmetic mean from 5 independent measurements. A significant difference was detected in comparison of specimens with modified and reference bitumen. Both modified specimens (M1 and M2) showed bonding strength approximately twice as higher as those containing reference binder (R). Moreover, reference samples showed perceptibly less gradual increase in recorded force in initial stages of loading. Comparing both samples sandwiched with doped bitumen to each other and considering measurement deviations, it can not be concluded which adhesion promoter is better.

4.2. Visual and image analysis

Visual analysis of two independent operators revealed that bitumen surface coated area reached on 60 % only in the case of the reference mixture (R), with belonging verbal classification as "unsatisfactory" according to [6]. Unlike both modified mixtures (M1 and M2) exhibited the area about 80 % – "satisfactory" classification (see Table 2).

Entropy-based image segmentation, however, provided quite different results than visual analysis. Concretely, reference mixture (R) exhibited 67.1 % of coated areas, while M1 and M2 94.7 and 96.0 %, respectively (Table 2). Given the fact that the semi-automatic analysis is considered to be more accurate than the subjective one, such obtained results are assumed to be more valuable and conclusive. Moreover, as it is shown in Figure 4, the green colored regions, marking the coated areas, prove that the amount of stripped areas is negligible.

Regardless to differences between results from both methods, it can be concluded that adhesion promoters significantly contributed for improving in adhesion between mineral aggregate and bituminous binder.

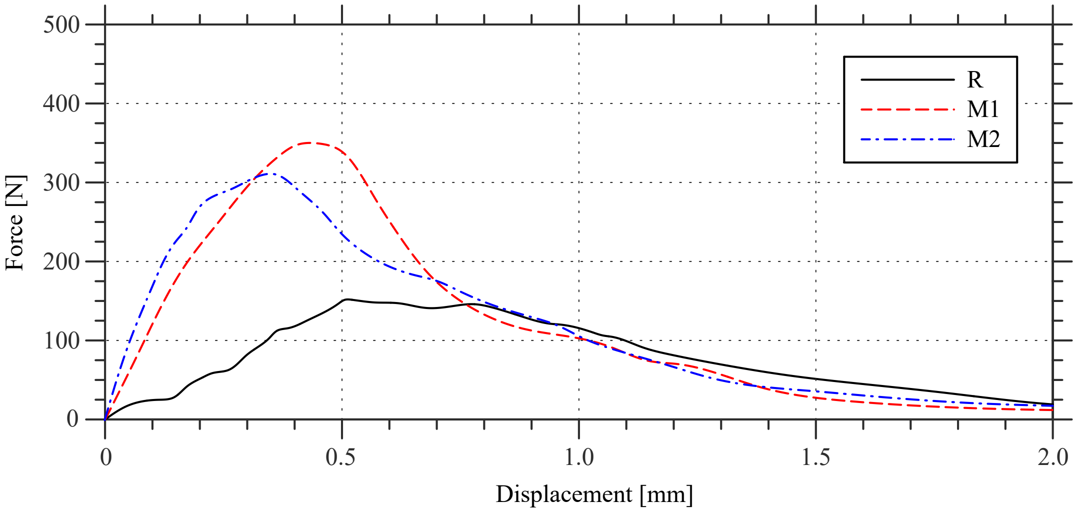

Figure 3. Force as a function of displacement revealed during pull-off experiment.

Mark	Coated area by visual analysis (%)	Classification by visual analysis	Coated area by DIC analysis (%)	Classification by DIC analysis
R	60	D-E: unsatisfactory	67.1	D: unsatisfactory
M1	80	C: satisfactory	94.7	A: good
M2	80	C: satisfactory	96.0	A: good

Table 2. Summarization of binder coated area results obtained by both visual and digital image (DIC) analysis.

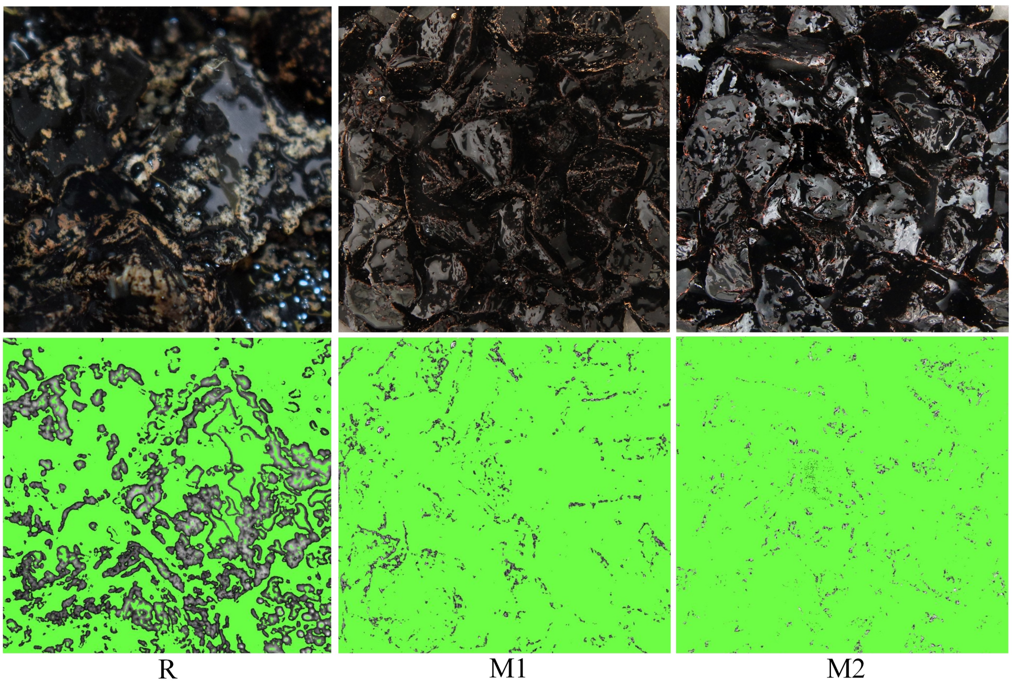

Figure 4. Images of aggregate coated by binder (top) and highlighted coated areas as a result form DIC analysis (bottom).

5. Conclusions

Adhesion between paving grade bitumen 50/70 and mineral aggregate from Brant quarry was analyzed by means of visual, digital image, and mechanical analysis. In order to increase an interaction between the two materials, bitumen was doped with adhesion promoters based on unsaturated fatty acids with diethanolamine and phosphorus-based at the amount of 0.2 and 0.3 wt.% of binder.

Asphalt mixtures composed from crushed aggregate (8-16 mm) coated by binder were exposed to stripping water. Consequently, areas coated by binder were quantified. Then, the adhesion was analyzed mechanically using pull-off tests, when two cylindrical parts of aggregate were sandwiched with 1 mm layer of bitumen binder and consequently pulled-off.

It was shown that adhesion between the two materials was increased using adhesion promoters by ca. 20 and 30 % according to visual and digital image analysis, respectively. Pull-off tests revealed that mechanical adhesion between aggregate and modified bitumen was increased by up to twice.

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