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ADHESION BETWEEN DIFFERENT ASPHALT BINDERS AND AGGREGATE TYPES EVALUATED USING BOILING WATER STRIPPING TEST IN SCOPE OF WMA FOAMING TECHNIQUE

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Abstract. This study examines adhesion mechanisms in binder–aggregate systems designed to simulate warm-mix asphalt incorporating water-foamed bitumen. The experimental program involved limestone, gabbro, melaphyre and quartzite aggregates, along with four paving-grade asphalt binders (50/70 and 45/80-55) originating from two different refineries. Adhesion performance was assessed using the boiling water stripping test, with residual binder coverage quantified through digital image analysis. In addition, dynamic viscosity measurements of the asphalt binders at relevant temperatures were conducted, and the petrographic analysis of the composition of aggregates was conducted. The samples produced with conventional (liquid) binders were mixed at temperatures representative of hot-mix asphalt production while reduced temperatures typical of warm-mix asphalt technology (20 °C lower) were used with the foamed asphalt binders. With the limestone and gabbro aggregates most of the experiments yielded over 90% residual binder coverage after the boiling test. The melaphyre and quartzite aggregates revealed on the other hand significant differences between the performance of the binders in terms of their type, origin and their form. The foamed binders with lowered mixing temperatures produced on average approx. 5% point lower residual binder coverage in melaphyre and quartzite while in limestone and gabbro the average difference was only 1% point. The source refinery of the asphalt binder significantly affected adhesion outcomes; however, the supplementary rheological and chemical analyses did not fully account for the observed discrepancies. Overall, the findings suggest that the foaming process itself may enhance the water resistance of asphalt binder-aggregate systems.

Keywords: adhesion, foamed bitumen, warm-mix asphalt, boiling water stripping test, digital image analysis, dynamic viscosity, petrographic analysis.

1. Introduction

Warm-mix technologies enable substantial reductions in emissions and energy consumption associated with road construction (D'Angelo et al., 2008). Warm-Mix Asphalt (WMA) is typically produced at temperatures 20–30 °C lower than comparable hot-mix asphalt. Adequate mixture workability and compactability can be achieved using various approaches, including asphalt binder additives (Behnood, 2020; Remišová & Holý, 2017), asphalt binder foaming (Iwański et al., 2020, 2021), asphalt mix additives (Chomicz-Kowalska et al., 2020, 2021; Ghabchi et al., 2014; Woszuk et al., 2017), binder fluxing (Pucułek et al., 2020), and combinations of these methods (Chomicz-Kowalska et al., 2020; Rubio et al., 2012). Mechanical water foaming, in particular, enables the production of warm-mix asphalt without additional additives, whereas other WMA technologies typically incorporate antistripping measures. This provides an opportunity to investigate the moisture resistance of such mixtures without the confounding influence of antistripping agents. Several studies on warm-mix asphalt produced using mechanical water foaming have reported that these mixtures may be more susceptible to moisture damage (Bairgi et al., 2021; Cucalon et al., 2014; Rahman et al., 2021). However, some investigations (Shu et al., 2012) have shown that plant-produced foamed WMA exhibits moisture susceptibility comparable to hot-mix asphalt, in contrast to laboratory-produced warm mixes. It has also been demonstrated that the extent of aging can significantly affect the moisture resistance of warm-mix asphalts, indicating that adequate laboratory aging (Xu et al., 2017) or summer aging prior to the winter period (Cucalon et al., 2016) may be sufficient to improve this property. Although moisture susceptibility tests conducted on compacted asphalt mixture specimens generally represent the performance of the final mixture well, their results may be influenced by additional factors. Typically,

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they include degree of compaction, gradation, mixture composition, and the specific methoology used. These factors can be excluded in model studies, such as binder-aggregate affinity tests conducted using various methods (Paliukaitė et al., 2016).

Based on the reviewed literature, this study investigated the influence of asphalt binder water foaming on the stripping resistance of different combinations of aggregates and asphalt binder under controlled conditions. Stripping resistance was assessed using the boiling water stripping method, with the results quantified through digital image analysis. The boiling water method was selected due to its insensitivity to the mechanical properties of aggregates (in contrast to the rolling bottle method). The study utilized different aggregate types, which are regarded classically as ranging from basic to acidic and paving grade as well as polymer modified bitumen from two different suppliers were used. To quantify the area of the aggregates covered by the remaining bitumen film, a digital image analysis approach was used.

In addition to the stripping tests, mineralogical characterization of the aggregates was conducted and basic rheological characterization of bituminous binders was performed to identify potential sources of variability in stripping behavior. Dynamic viscosity was evaluated in view of its relevance to surface energy-based adhesion mechanisms and its contribution to mechanical resistance to flow during boiling.

2. Tested materials and methodology

2.1. Experimental program

The presented results included three factors in the investigation of the effects on the asphalt binder-agregate adhesion:

- type of asphalt binder:
  - PGB – 50/70 paving grade bitumen,
  - PMB – 45/80-55 polymer-modified bitumen,
- different refineries form which the asphalt binder was sourced:
  - O,
  - L,
- mixing temperature and form of the binder:
  - liquid-HMA,
  - foamed-WMA,
- aggregate type:
  - limestone,
  - gabbro,
  - melaphyre,
  - quartzite.

The aggregates used in the study were selected based on their compatibility with asphalt binders – from the limestone clasically understood as most compatible with asphalt binders, to quartzite yielding difficulties in coating and moisture resistance without adhesion promoters. Two intermediate aggregates were also selected: melaphyre (classified often as a basalt type rock) and gabbro (recognized as a granite type rock).

The mixing temperatures were dependent on the type of the binder and the presumed production technique. For the foamed asphalt binders a WMA production technique was presumed and mixing temperatures decreased by 20 °C in relation to HMA temperatures were used to simulate the effects of WMA mix production (shown in Table 1).

Table 1. Mixing temperatures used in the study

Form of the asphalt binder	PGB (50/70)	PMB (45/80-55)
Mixing temperature (°C)		
Liquid – HMA	150	165
Foamed – WMA	130	145

2.2. Dynamic viscosity testing of asphalt binders

The dynamic viscosity was measured in accordance to EN 13302 using a TA Instruments DHR-2 dynamic shear rheometer with concentric cylinders accessory as shown in Figure 1.

Figure 1. Dynamic shear rheometer with a concentric cylinder accessory used for evaluation of dynamic viscosity of the asphalt binders

The dynamic viscosity measurements were conducted at a 1/s shear rate from the highest to the lowest temperature.

2.3. Petrographic analysis of the aggregates

A particle size fraction of 8/11 mm aggregates was subjected to the petrographic analysis, the same as the used in boiling tests. The examination of aggregate samples using a transmitted-light polarizing microscope was focused on the determination of mineralogical composition (qualitative and quantitative analysis), but also included evaluation of size and arrangement of constituents and their type and degree of mineral alteration.

The petrographic analysis in case of all aggregates was conducted on sets of two thin sections, prepared by an experienced technician in accordance with the recommendations given in Section 5.3 of RILEM AAR-1.1 (Nixon & Sims, 20216). The thin sections were prepared as follows: selected representative aggregate grains were embedded in a resin with a known refractive index and bonded to a microscope slide. Thin slices were then cut from the grains, and their thickness was reduced to 0.02 mm by grinding and polishing. The prepared specimens were examined using a JENAPOL (Carl Zeiss Jena) petrographic transmitted-light polarizing optical microscope equipped with a Nikon DS-Fi1 digital camera (5-megapixel resolution) and the NIS-Elements BR 3.2 image analysis software (Nikon Corporation). The volumetric proportion of aggregate constituents was determined using the planimetric method by counting 600 points on each thin section. The points were counted along six measurement lines, parallel to each other and to the longer edge of the thin section.

2.4. Sample preparation and boiling water stripping test

The experimental protocol adopted in this study followed the procedure specified in EN 12697-11 (European Committee for Standardization [ECS], 2020, Section 7: boiling water stripping method).

Sample preparation began with the foaming of the asphalt binders. Bitumen was foamed using a Wirtgen WLB10S laboratory foamer with a foaming water content of 2%. The foamed binders were transferred to glass containers and were allowed to cool to ambient temperature. The non-foamed binders were also poured into identical containers and cooled under the same conditions.

The aggregates and asphalt binders prior to mixing were heated to the adequate mixing temperatures specified in the experimental programme increased by 5 °C.

Mixing was performed in pre-heated steel bowls using glass rods. The binder was added at 2% by aggregate mass, corrected for aggregate density. Mixing continued until complete coating of all particles was achieved, which consistently required less than 3 min. The resulting mixtures were then spread on a silicone rubber mat, the coated particles were separated using a glass rod, and the material was left to cool at ambient temperature.

From each coated aggregate batch, three test specimens of 200 g ± 0.5 g were prepared for boiling. The boiling water stripping tests were conducted in accordance with EN 12697-11 (ECS, 2020). Upon completion of the boiling procedure, the samples were transferred onto silicone rubber mats, the particles were separated again and the material was left to cool and dry for a minimum of 24 h.

2.5. Image acquisition of bitumen coated aggregates and analysis

Based on preliminary trials and the experience reported by other researchers (Källén et al., 2016; Komačka et al., 2019), a procedure for acquiring high-quality photographic data was established. The main principles of this methodology included:

1) image acquisition under repeatable and controlled artificial lighting using a ring light;
2) the use of a distinct background color to facilitate background removal during post-processing, if required;
3) arranging the aggregate particles without gaps in order to minimize background reflections;
4) photographing each sample twice – first in its initial orientation and subsequently after flipping it upside down – to increase the amount of sample area per specimen and reduce potential bias related to the initial particle arrangement.

As shown in Figure 2a, the digital images of the aggregate samples were captured using a consumer mirrorless camera equipped with an APS-C CMOS III sensor (Fujifilm XT-2). The camera was mounted on a tripod and positioned perpendicular to the sample surface resulting in a repetitive image capture (Figure 2b).

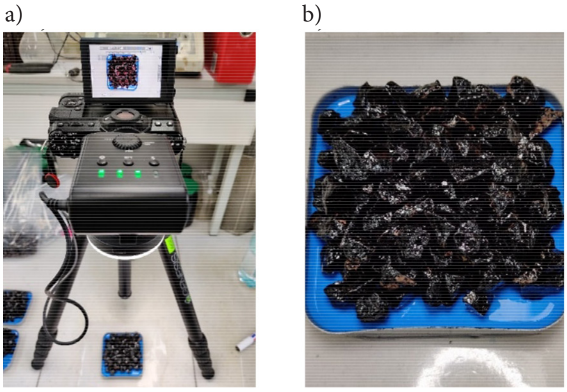

Figure 2. Photographic setup used to acquire the images: a) tripod setup; b) sample prepared for image capture

The lens was set to a focal length of 55 mm and an aperture of f/11 to reduce geometric distortion and ensure sufficient depth of field. Two accessories were mounted in front of the lens: a circular polarizing filter to control surface reflections from the bitumen film and a ring light serving as the primary illumination source. Exposure parameters were selected to avoid highlight and shadow clipping, and a reference target was included to normalize exposures during post-processing.

Aggregate specimens previously subjected to the boiling water stripping tests were carefully arranged on small trays filled with silicone rubber in two to three layers. This arrangement was selected to ensure that the background material did not produce reflections on the asphalt binder coating the particles (Figure 2b). Each specimen was photographed once, then flipped upside down, and the particle arrangement was adjusted as needed to acquire a second image. Following image acquisition, the samples were packaged and archived.

The images were preprocessed prior to the primary analysis to standardize the exposures and crop the central region of the sample. Analysis was conducted using a custom Python script utilizing the OpenCV library. The original RGB (red, green, blue) images were converted to the HSV (hue, saturation, value) color space, which is widely employed in computer vision and image processing applications owing to its closer alignment with human color perception (Ibraheem et al., 2012; Phuangsaijai et al., 2021). The main part of the image manipulation involved image segmentation through identification and validation of distinct regions via color thresholding and masking: the background, asphalt binder-coated aggregates and stripped (exposed) aggregate surfaces as shown in Figure 3.

Figure 3. Results of the boiling tests using limestone and gabbro aggregates (13) quartzite aggregate with O-PGB liquid binder (HMA) – 84.8% residual binder coverage (left – image before segmentation, right – image after segmentation)

Pixel counts for each masked region were then determined. The corresponding areas derived from paired images – acquired before and after sample inversion were added, enabling calculation of the asphalt binder coverage on the visible aggregate surface.

3. Results

3.1. Results of dynamic viscosity of the asphalt binders

The dynamic viscosity of the investigated asphalt binders was determined at several temperatures, with three replicates for each temperature. These included both the temperatures corresponding to the conditions used for coating aggregate samples with asphalt binder (according to the experimental design) and the temperature of 100 °C. The temperature of 100 °C corresponds to the boiling point of water and thus reflects the conditions applied in the boiling test used for adhesion assessment. The figures represent mean values of the dynamic viscosity and 95% confidence intervals.

With respect to the dynamic viscosity results obtained at the temperatures used for mixing the asphalt binder with aggregate samples (Figure 4), it was found that the viscosity of all investigated binders ranged from approximately 0.25 to 1.1 Pa·s. For all binder types, water-foamed asphalt binders exhibited significantly higher dynamic viscosity, which can be attributed to the test temperature being 20 °C lower than that used for the corresponding binders in the liquid form. For both binder producers, polymer-modified binders showed higher dynamic viscosity values than paving-grade binders. The paving-grade binders from both producers exhibited nearly identical dynamic viscosity values. In contrast, for polymer-modified binders, the binder supplied by the producer denoted as L-PMB showed slightly lower dynamic viscosity than the O-PMB binder.

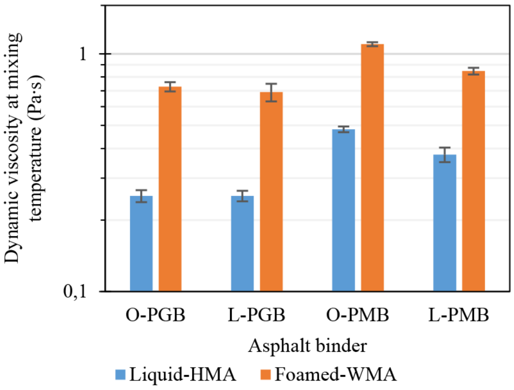

Figure 4. Dynamic viscosity of the investigated asphalt binders at the respective mixing temperatures

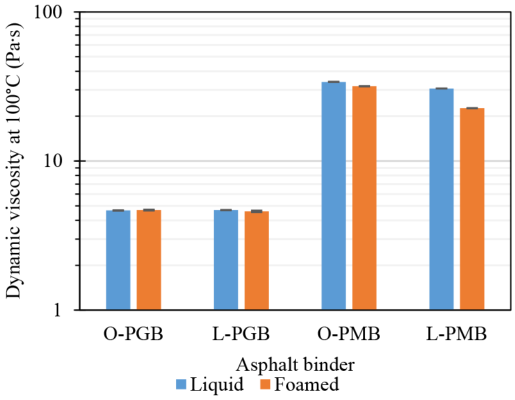

Figure 5. Dynamic viscosity of the investigated asphalt binders at the temperature of the boiling test

For the tests conducted at 100 °C (Figure 5), substantially larger differences were observed between the dynamic viscosity values of paving-grade and polymer-modified binders. The paving-grade binders from both producers – regardless of whether they were tested in the foamed or liquid form – exhibited a dynamic viscosity of approximately 4.7 Pa·s. For polymer-modified binders, similarly to the results obtained at lower temperatures, the L-PMB binder showed lower viscosity than the O-PMB binder. Moreover, at 100 °C, polymer-modified binders tested in the water-foamed form exhibited significantly lower dynamic viscosity.

3.2. Results of the petrographic analysis of the aggregates

The mineral composition of four aggregate types (limestone, gabbro, melaphyre, and quartzite) was evaluated based on petrographic and quantitative analysis and is shown in Table 2.

The evaluated limestone was dominated by carbonate minerals: calcitic micrite (43.61%), calcitic sparite (22.83%), and bioclasts (17.01%), totaling approximately

83.5% carbonate phase. Quartz content was low (6.09%), while clay minerals and iron compounds accounted for 9.83%. The high $CaCO_3$ content results a strongly basic surface chemistry, promoting durable chemical bonding with polar groups in asphalt (Petersen et al., 1982; Zhang et al., 2015).

The gabbro aggregate contained significant mafic content of minerals: pyroxenes (24.9%), magnetite (8.2%), amphibole (8.9%), and olivine (1.3%). Plagioclase which may be of sodic (acidic and neutral do acidic) or calcic (basic and neutral to basic) type constitutes 30.9% of the aggregate. Biotite was determined at 20.5% with minor quartz component (5.3%). This material is classified as basic/mafic (low SiO_2, high FeO + MgO).

Table 2. Mineral composition of the aggregates used in the study

Minerals	Limestone	Gabbro	Melaphyre	Quartzite
Mineral composition of aggregates (%)				
Plagioclase	0	30.9	43.8	0
Pyroxenes	0	24.9	32.7	0
Olivine	0	1.3	3.9	0
Magnetite	0	8.2	19.6	0
Amphibole	0	8.9	0	0
Biotite	0	20.5	0	0
Quarzite	6.09	5.3	0	72.1
Calcitic micrite	43.61	0	0	0
Bioclasts	17.01	0	0	0
Calcitic sparite	22.83	0	0	0
Chalcedony	0.63	0	0	0
Clay minerals and iron compounds	9.83	0	0	0
Iron oxides and hydroxides	0	0	0	10.1
Micas	0	0	0	2.9
Clay minerals	0	0	0	7.2
Silica	0	0	0	7.7

Melaphyre featured high mafic mineral content: pyroxenes (32.7%), magnetite (19.6%), and olivine (3.9%). Plagioclase constituted 43.8%, which due to their high content may determine the character of the investigated melaphyre aggregate based on their type. Although typically, this type of aggregate is classified as neutral, in other studies it was shown that its adhesive performance with asphalt binders may be poor (Komačka & Remišová, 2019; Remišová, 2004).

Quartzite was dominated by quartz (72.1%) plus additional silica (7.7%), yielding approximately 80% SiO_2. Remaining components included Fe oxides/hydroxides (10.1%), clay minerals (7.2%), and micas (2.9%). The high quartz content results in an acidic surface resulting in weak asphalt bonding and elevated stripping susceptibility (Guo et al., 2020).

3.2. Results of the boiling water stripping tests

The results of boiling water stripping tests are presented in Figures 6–7, representing mean values of the residual binder coverage and 95% confidence intervals.

Limestone exhibited excellent adhesion under all conditions. Residual binder coverage ranged from 92% to 99%, with the majority of values between 95% and 98%. No statistically significant differences were observed between HMA and foamed WMA, between PGB and PMB, or between the two asphalt binder sources.

Gabbro also demonstrated very good overall affinity to the investigated binders, with residual binder coverage ranging from 77% to 99%. The only notable reduction occurred with foamed WMA famed O-PGB binder mixed with the aggregates at lowered temperature (77%). All other combinations exceeded 89%, and both PMB variants (O-PMB and L-PMB) residual near-complete coverage (>97%) for both HMA and WMA. The effect

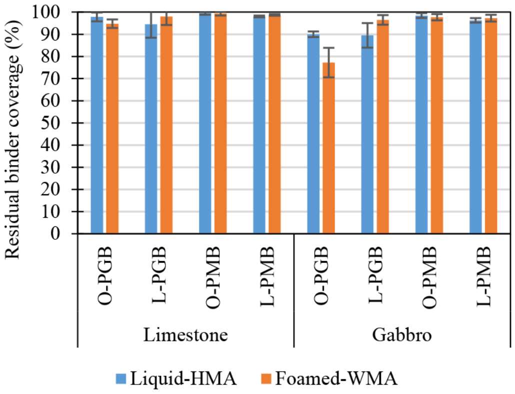

Figure 6. Results of the boiling tests using limestone and gabbro aggregates

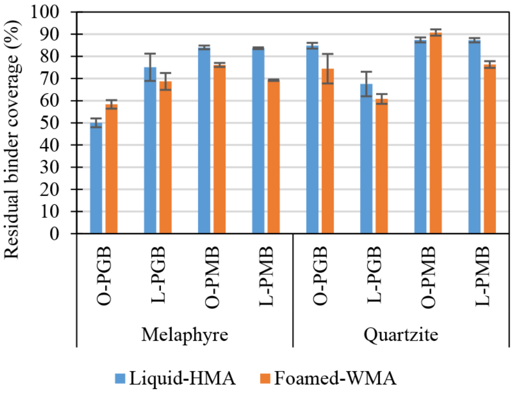

Figure 7. Results of the boiling tests using melaphyre and quarzite aggregates

of asphalt binder source was minor and limited to the foamed O-PGB system; the L-source PGB performed markedly better in the same technology.

The residual binder coverage achieved with melaphyre aggregate showed not only greater variability, but also in general significantly lower values ranging from 50% to 84%. The lowest values were recorded for the O-source PGB (≈50% for liquid HMA and ≈58% for foamed WMA). Switching to the L-source and, especially, the use of polymer modified bitumen significantly improved performance, raising the binder residual coverage to 69–84%. Foamed WMA performed worse compared to conventional HMA in all cases but the O-PGB binder.

Quartzite produced similarly low affinity results (61–92%). Both paving-grade bitumens (O-PGB and L-PGB) yielded only 61–91% coverage, confirming the high stripping susceptibility of this aggregate. Polymer modification markedly enhanced adhesion, increasing residual coverage to 75–92% for both O-PMB and L-PMB. No clear systematic difference was observed between the two asphalt binder sources, and foamed WMA performed similarly to liquid HMA with a slight tendency of lower adhesion in case of foamed WMA binders.

By viewing the results of boiling water tests together with the dynamic viscosity results and the petrographic analysis a few observations can be made:

- the use of PGB (50/70) binders consistently resulted in lower residual binder coverage compared to the PMB binders (45/80-55); this effect can be partially attributed to their lower viscosity; similarly, the L-PMB binder had lower viscositiy in all instances than O-PMB and consistently yielded lower residual binder coverage,
- the results of binder stripping tests were significantly influenced by the source of the asphalt binders, which taking into account their similar viscosities (e.g., O-PGB vs. L-PGB with melaphyre and quartzite aggregates) points to other factors affecting the binder-aggregate interactions – particularly different chemical compositions of these binders,
- the melaphyre aggregate performed poor in the adhesion tests; assuming that the plagioclase contained in the aggregates was the acidic type, the rock contained approx. 63% of acidic minerals, which is similar to the amount of approx. 80% and could contribute to the observed performance.

4. Conclusions

The adhesion ranking observed in the presented data reflects the transition from strongly basic (carbonate-rich) to acidic (silica-rich) character of the aggregates (Zhang et al., 2020). Limestone and gabbro achieved excellent performance irrespective of binder type or production method (77% to 99% residual binder coverage), while melaphyre and the acidic quartzite performed significantly worse (50% to 91% binder coverage). The melaphyre aggregate, conventionally considered as neutral, exhibited the worst performance, with clear benefits from both the L-source asphalt binder and PMB.

The foamed WMA technology produced measurably decreased but comparable adhesion levels to conventional HMA in the great majority of cases, indicating that the foaming process itself does not compromise binder-aggregate bonding when appropriate binders are selected. With the limestone and gabbro aggregates the WMA experiments yielded on average 1% lower residual binder coverage, while in the melaphyre and quatzite aggregates the difference increased to 6%. It should be noted, that decreased coating temperature was also used in these instances.

Regarding the effects of asphalt binder source only minor differences between the two asphalt binder sources (<1% difference on average) were observed only in specific combinations (particularly O-PGB with gabbro and melaphyre in WMA), most likely reflecting subtle variations in asphalt binder acidity or polar-group content. The different types of asphalt binders performed significantly differently, with PMB binders yielding on average 10% higher residual binder coverage compared to the PGB ones.

These findings emphasise the critical importance of matching aggregate mineralogy with binder selection and use of adhesion promoters. The results also support the suitability of foamed warm-mix technology for a wide range of aggregates when combined with properly chosen binders.

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