Evaluation of Foaming Performance of Bitumen Modified with the Addition of Surface Active Agent

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Abstract. The article presents the analysis of the performance of foamed bitumen modified using surface active agents. Although, bitumen foaming permits production of asphalt concrete and other asphalt mix types without using chemical additives in significantly reduced temperatures, the decrease in processing temperatures still impacts the adhesion performance and bitumen coating of aggregates in final mixes. Therefore, in some cases it may be feasible to incorporate adhesion promoters and surface active agents into warm and half-warm mixes with foamed bitumen to increase their service life and resilience. Because of the various nature of the available surface active agents, varying bitumen compatibility and their possible impact on the rheological properties of bitumen, the introduction of surface active agents may significantly alter the bitumen foaming performance. The tests included basic performance tests of bitumen before and after foaming. The two tested bitumen were designated as 35/50 and 50/70 penetration grade binders, which were modified with a surface active agent widely used for improving mixture workability, compactibility and adhesion in a wide range of asphalt mixes and techniques, specifically Warm Mix Asphalt. Alongside to the reference unmodified bitumen, binders with 0.2%, 0.4% and 0.6% surface active agent concentration were tested. The analysis has shown a positive influence of the modifier on the foaming performance of both of the base bitumen increasing their maximum expansion ratio and bitumen foam half-life. In the investigations, it was found that the improvement was dependent on the bitumen type and modifier content. The improved expansion ratio and foam half-life has a positive impact on the aggregate coating and adhesion, which together with the adhesion promoting action of the modifier will have a combined positive effect on the quality of produced final asphalt mixes.

1. Introduction

Industry in Europe and worldwide is undergoing radical changes resulting in both the improved sustainability of modern societies and the reduced impact on the environment. Striving to contribute to the trends set by subsequent conventions on climate change and by the legal standards of the EU internal market, the road building industry has increased its awareness and capacity to deliver expected environmental benefits. New technologies are aimed at the maximized use of environmentally compatible secondary and waste materials and at the overall reduction in energy intensity of construction processes [1-7]. Normally, high processing temperatures are necessary to
produce bituminous road paving materials so large energy savings are expected from the lowering of production and paving temperatures.

The idea of using lower temperatures for producing bituminous mixtures is not new. First and the most drastic attempts were made in 1956 by Prof. Ladis Csanyi at Iowa State University USA. The novel bitumen foam was used as a binder. Since then foaming has been used in various countries, including USA, Australia and Europe – mainly for producing cold mix asphalt for low volume pavements and lower portions of the pavement structure. Other approaches to low temperature bitumen mix production are based on chemical additives added to bituminous binders to change their properties at an adequate stage of production or in-service. The additives include surface active agents, emulsifiers, zeolites and waxes [7-10]. Germany has been successfully applying waxes to lower the mix temperature and to improve workability and compaction of bituminous mixtures for the last 20 years. It was not until 15 years ago that processing temperature reduction (during production, hauling, placement and rolling temperatures) became a priority. Given the move towards more common use of the technology, the performance of warm asphalt mixes needs to be thoroughly studied.

This paper reports the results from testing foaming properties of 35/50 and 50/70 road paving bitumen modified with an addition of a surface active agent. These binders are commonly used for paving the roads under medium traffic loads [11]. Adequate foaming performance is of primary importance for the durability of pavement constructed with foamed warm mix asphalts. The bitumen foaming technique has a direct effect on the aggregate-binder bond and on the mechanical properties of the resulting mixture. Significant factors affecting the lifespan of a pavement include proper coating of aggregate grains with bitumen and ensuring an adequate level of adhesion between these two components [12, 13].

2. Materials and research methodology

2.1. Experimental program

The tests aimed at determining the effect of surface active agents added during the WMA production process on the variability of the properties of 35/50 and 50/70 bitumen types. The tests were performed both before the modification of the binder with the chemical additive prior to foaming and after these processes.

The results were investigated in scope of the intended use in production of warm bituminous mixes. The additive content in both binders was ranging from 0.2% to 0.6% with an increment of 0.2% by mass. The bitumen samples were prepared according to EN 12594. The testing was divided into two stages. Basic parameters of the road bitumen were determined before foaming during the first stage of the tests. In the second stage, the performance of the bitumen foam was measured.

2.2. Bitumen properties

Laboratory tests were carried out on paving grade petroleum binders recommended for the production of hot bituminous mixes intended for pavement structural layers by Polish Technical Requirements WT-2 [14].

In the first stage of testing, the basic properties of the binders were measured before and after modification with the surface active agent. The performed tests included: penetration at 25 °C (Pen) according to EN 1426, softening point temperature ($T_{R&B}$) according to EN 1427 and breaking point temperature ($T_{Fraass}$) according to EN 12593. The penetration index ($PI$) was also determined as an additional parameter. The $PI$, computed based on $T_{R&B}$ and Pen, is the measure of bitumen temperature susceptibility and indicates stiffness change rate at varied temperature.
The overall aim of the tests was to determine the effect of the surfactant dosing on the changes in the properties of 35/50 and 50/70 penetration binders. The significance of the obtained results in respect to the *Surfactant content* factor was carried out using one-way ANOVA.

2.3. Foamed bitumen properties

The second stage of the tests involved determining parameters of the bitumen foam (*ER* – expansion ratio and *HL* – half-life) [15] produced from the base 35/50 and 50/70 penetration bitumens, and binders modified with the surfactant additive. The characteristics were measured in laboratory conditions with use of the WLB 10S by Wirtgen GmbH foamed bitumen.

Expansion ratio is the ratio between maximum foam volume and the volume of original bitumen (before foaming); it shows the volume increase during foaming, whereas the half-life is the time that it takes for the foam to collapse to half of its maximum volume, measured in seconds. The expansion ratio is an indirect measure of the viscosity of the foam and provides an indication of how well the binder will disperse in the mineral mix to produce a uniform structure. The half-life is a measure of the stability of the foam and provides an indication of the speed of its collapse. There is usually an inverse relationship between these two parameters, where increasing the content of foaming water causes an increase in the expansion ratio and decrease in the half-life [16].

The properties of the foam were measured at a water content ranging from 1.5% to 4.0% added at 0.5% steps. The amounts of the foaming water used and the conditions of the foam production were selected based on the authors’ own experience [17-19] and that of other researchers [20-22].

Analysis of the results consisted of determining the optimal properties of the foam while manipulating the levels of factors under test (*Foaming Water Content - FWC, Surfactant content*). To provide a reliable evaluation, the foamed bitumen properties were determined four times at each *FWC* level.

2.4. Surface active agent

The surface active agent (Cecabase® RT) used in the tests is manufactured by CECA (France, ICSO CHEMICAL PRODUCTION and CECA Arkema Group), and its composition is subject to patent protection [23]. The liquid substance, produced mostly of waste materials (60%), is added to bitumen. It consists of emulsifiers, surface active agents and polymers [24]. A small amount of this surfactant (from 0.2% to 0.5% by mass of bitumen) is capable of reducing the mix production temperature by about 30-40 °C [23, 24].

In the laboratory tests, the additive was added at 0.2%, 0.4% and 0.6% by mass of the binder directly to the bitumen before foaming (i.e. to the bitumen heated in the WLB 10S laboratory unit).

3. Results and discussion

3.1. The effects of the surface active agent content on basic bitumen properties

Figures 1a-d show the effect of the surfactant content on changes of selected road paving bitumen parameters. Figures 1a-e illustrate the mean values of the parameters together with the 95% confidence interval. The computed penetration index values are displayed in Figure 1d. Table 1 compiles the results of significance tests (one-way ANOVA) for the effect of *Surfactant content* on the distribution of the parameters being tested.
Table 1. Significance analysis (One-way ANOVA) of the effect of surfactant content on the properties of bitumen 35/50 and 50/70.

| Feature | Bitumen type | Effect          | Univariate Tests of Significance
|---------|--------------|-----------------|----------------------------------|
|         |              |                 | Sigma-restricted parameterization |
|         |              |                 | Effective hypothesis decomposition |
|         |              | Intercept       | SS  | DF | MS  | F       | p       |
| Pen     | 35/50        | Surfactant content (%) | 14.75 | 3 | 4.92 | 2.57 | 0.103389 |
|         | 50/70        | Intercept       | 52212.25 | 1 | 52212.25 | 30563.27 | 0.000000 |
|         |              | Surfactant content (%) | 13.25 | 3 | 4.42 | 2.59 | 0.101679 |
| TR&B    | 35/50        | Intercept       | 47306.25 | 1 | 47306.25 | 20642.73 | 0.000000 |
|         | 50/70        | Surfactant content (%) | 20.25 | 3 | 6.75 | 2.95 | 0.075969 |
|         |              | Error           | 27.50 | 12 | 2.29 |
|         |              | Intercept       | 42539.06 | 1 | 42539.06 | 17755.43 | 0.000000 |
|         | 50/70        | Surfactant content (%) | 19.19 | 3 | 6.40 | 2.67 | 0.094873 |
|         |              | Error           | 28.75 | 12 | 2.40 |
| TFraass | 35/50        | Surfactant content (%) | 8.187 | 3 | 2.729 | 2.77 | 0.088501 |
|         | 50/70        | Intercept       | 3660.25 | 1 | 3660.25 | 3029.172 | 0.000000 |
|         |              | Error           | 14.50 | 12 | 1.208 |

The results in Figure 1a-d and Table 1 indicate that the effect of the surfactant used at the specified amounts was similar for both bitumen types. The p-values obtained for the F statistic (Table 1) higher than the assumed significance level (α=0.05) show that the Surfactant content factor had no statistically significant effect on the values of 35/50 and 50/70 bitumen parameters, as confirmed by minor changes in the values of those parameters and by relatively large changes in the test results.

However, the analysis of these relationships leads to the conclusion that for both bitumen types, the increase in the surface active agent content (within the tested range 0.2% to 0.6%) increased the penetration by an average of 3 (0.1mm), while the softening point decreased by about 2 °C, making the bitumen being tested softer. The results obtained for the breaking point temperature indicate that the addition of the surface active agent reduced its value from -9.5 °C to -11.0 °C for 35/5 and from -14.0 °C to -16.5 °C for 50/70, thereby improving their resistance to negative temperatures. The final parameter tested to classify the bitumen in terms of temperature sensitivity was the penetration index. The PI values decreased with an increase in the amount of surfactant added to 35/50 and 50/70 and fall within the <-1; 0> bracket. The PI of the bitumen used in the road building industry ranges from +2.0 to -2.0, but the recommended PI values are in the range -1.0 to +1.0. The PI values obtained from the tests, -0.9 and -0.8 for 35/50 and 50/70, respectively at the maximum surfactant content (0.6%) keep the bitumen within the sol-gel rheological type limits.
3.2. The effects of the surfactant content on foamed bitumen properties

As mentioned earlier, the second stage of the testing involved analysis of two bitumen foam parameters, expansion ratio ($ER$) and half-life ($HL$). To properly evaluate the properties of the foam, the parameters were measured four times at varying $FWC$ levels (1.5%, 2.0%, 2.5%, 3.0%, 3.5%, 4.0%).

In the first stage of the investigation, the results obtained helped develop relationships (Figure 3) between the parameters ($ER$, $HL$) and the change in the level of the water added during foaming. The resulting characteristics were the basis for determining the optimum foaming water content for each binder type at the intersection of the $ER$ and $HL$ curves in line with the recommendations [25].

Figures 2a-d illustrate the measurement results for the characteristics of the bitumen foam, depending on the bitumen type (35/50, 50/70), surfactant amount (0.2%, 0.4% and 0.6%) and foaming water level. Table 2 summarizes bitumen foam parameters ($ER$, $HL$) at optimal $FWC$ dosage.
Analysis of the foamed bitumen’s physical properties shows that the addition of the surface active substance (from 0.2% to 0.6%) had a positive effect on changes observed in the foaming parameters of bitumen composites made with 35/50 and 50/70 binders. At the fixed optimal foaming water content, the values of ER and HL increased with increasing amount of surfactant used.

The composites made with the softer binder (50/70) had higher expansion ratios than those made with the harder binder (35/50) at all surfactant dosage levels. This relationship confirms previous findings observations [17, 19] and those of other researchers [26, 27], indicating that softer binders have better foaming properties. An inverse relationship was observed for the foam HL in which case
the harder bitumen type, 35/50, and 35/50 with 0.2%, 0.4% and 0.6% of the surfactant had longer half-lives, indicating that the foam decayed at a slower rate.

The optimum foaming water content, determined from the point of intersection of the ER curve and the HL curve, was $FWC=2.5\%$ for each surfactant dosage and bitumen type, except for 35/50, for which FWC was 3.5%.

The foamed neat bitumen failed to meet the requirement for minimum values of foam parameters, as set out for half-warm mixes (ER $\geq 17$ and HL $\geq 13$ seconds [15]). The binders made with the surfactant dosage of 0.4% and 0.6% by mass of the 35/50 binder can be used to produce HWMAs. Adding the surface active agent in the full range of application (from 0.2% to 0.6%) yielded higher parameter values that those recommended. All the foam parameters (high expansion and half-life) will improve the asphalt mixture coating ability, its workability, compaction, and adhesion of the binder to aggregate grains. This is especially important for the mixtures produced and compacted at lower temperatures, as there is a risk that the level of pavement layer compaction can be insufficient. Surface active additives are used in foamed HWMAs as adhesives to secure improved binder-aggregate adhesion effectiveness, especially when the mixture is made with acidic rock aggregates.

Figure 3. Effect of the change in the level of the factors under analysis (Surfactant content, FWC) on $ER$ and $HL$ of the bitumen foam produced with binders 35/50 (a, b) and 50/70 (c, d)
Analysis of the suitability of the binders being tested for use in CMA mixtures indicates that all eight binder types (i.e., with and without surfactants) met the criterion proposed by the Wirtgen Cold Recycling Technology [28] and the Technical Guideline 2 TG2 [29] stating that the minimum allowable values of \( ER \) and \( HL \) should be 8 and 6 seconds, respectively. The use of these binders, however economically inviable they may seem, is justified in cases of adverse weather conditions (e.g., air temperature too low for the in-place deep cold recycling processes), when the foam collapse rate may be faster. Better parameters of the foam (higher expansion and half-life) will ensure adequate coating of the fine mineral particles and uniform mixing of the mineral material. Summing up, the surface active agents used in CMA mixtures are an important factor in the improvement of foamed bitumen properties.

A linear function (Figure 3) was used to describe the relationships between the measured foaming parameters (\( ER \), \( HL \)) of the binders and the amount of water added during the foaming process. The coefficient of determination for the relationships recorded was \( R^2 > 0.92 \) indicating that the proposed functions explain the test results variability to the degree greater than 92%.

The obtained regression models lead to the statement that increasing the water content during the foaming process had a definite effect on the parameters of the binders through increasing the values of their expansion ratios and half-lives. The values of coefficients of determination \( R^2 \) obtained from the analysis indicate a good fit of the mathematical models to the data (\( R^2 > 0.94 \)). The \( p\)-value for the \( F \) statistic obtained from the tests for \( FT \) and \( FWC \) is definitely smaller (\( p\)-value < 0.0001) than the assumed level of confidence (\( \alpha = 0.05 \)), which means that the parameters in question have a significant effect on the changes in characteristics of the foamed bitumens studied.

4. Conclusions
Analysis of the results from basic rheological tests carried out on neat binders (35/50 and 50/70) and on the binders made with the surface active agent leads to the following conclusions:

- the addition of the surface active agent to the binders being tested, 35/50 and 50/70, did not have a significant impact on changes in their fundamental properties, such as penetration at 25 °C, softening point and breaking point;
- increasing the surface active agent content within the range 0.2% to 0.6% resulted in a minor increase in \( PG \) value, whereas \( TR&B \) decreased;
- the addition of the surfactant was a major factor in reducing the Fraass breaking point while increasing its content in 35/50 and 50/70, thereby increasing the resistance of these binders to temperatures below zero;
- the use of surface active agent content within the range tested had a positive influence on the changes in foamed bitumen parameters in the 35/50 and 50/70 bitumen types, by increasing both \( ER \) and \( HL \);
- higher \( ER \) values were recorded for the bitumen composites with the surfactant added to the softer binder (50/70), whereas the increased \( HL \) was recorded for those made with the harder bitumen 35/50;
- the recommended contents of surfactants added to HWMA mixtures are 0.4% and 0.6% in the case of 35/50 and from 0.2% to 0.6% in the case of 50/70. The presence of the surface active agent will improve the bitumen foambility, the coating and compaction of bitumen mixtures, and the adhesion of the binder to aggregate grains.

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