Optimization of the Synthetic Wax Content on Example of Bitumen 35/50

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Abstract

The paper presents the results of the bitumen studies, including the U.S. MSCR procedures describing bitumen creep under dynamic loading. It permits predicting the possibility of the damage in bituminous layers at high ambient temperatures and at low and high shear stresses. In addition, the study included such features as: the breaking point temperature, the complex modulus $G^*$, the viscosity at low shear rates. The tests were performed generally for bitumen 35/50. The test results for asphalt 50/70 are only informative. Bitumen 35/50 (50/70) was modified with synthetic wax derived from the Fischer-Tropsch synthesis (F-T) in amounts of 1.5%, 2.5%, 3.0% and 4.0%. The assessment of the influence of the synthetic wax on basic properties of the bitumen has been enriched by means of quantitative analysis of the morphology of the modified bitumen by means of the fluorescence microscope. A summary of the entire analysis was performed using multiparameter optimization which purpose was determining the optimal content (range) of the synthetic wax including its high stiffness and maintaining the satisfactory flexibility at a low temperatures.

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1. Introduction

Currently, one of the major challenges which must be faced in today's society is efficient and cost-effective energy management with the significant reduction of fuels. Production of asphalt mixtures requires energy both in the production process as well as bitumen production in the asphalt plants. In the U.S., the production of the bitumen and bituminous mixtures was the second most energy-intensive manufacturing industry [1]. Another problem associated with bituminous mixtures is their durability and susceptibility to permanent deformation. The main reasons for them are: exceeding a permissible individual wheel-pavement contact stress, a rise in the number of vehicles, decreasing loading frequency in urban traffic conditions combined with a high axle load [2]. The limitation of deformations may be achieved through correctly designed mineral mix, as well as an application of bitumen hard enough to guarantee an increase in the structural density of the bituminous mixture. But on the other hand, the application of that kind of a binder requires the use of high technological temperatures of over 155 °C under the WT-2/2010 polish guidelines to reach densities in the range from 2 to 20 Pas. This range of viscosity is required during the manufacturing process and subsequent placing of the bituminous mixture in order to ensure it the right compaction ratio. However, such a high temperature decisively deteriorate their low-temperature behavior and promotes ageing process of bitumen. Ideally, the performance of WMA should be the same as that of HMA, both structurally and functionally [3], [4].

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One of solutions of this problem is the modification of the bitumen by means of organic additives, which could be aliphatic synthetic waxes. It needs to be noted that the wax components in the bitumen are not new. The issues of optimal modification of modern wax incorporated into the bitumen in terms its rheology were frequently taken up. [5–7]. Generally waxes have been divided into two groups: paraffins in bitumens and modified synthetic waxes [5]. Macro-crystal paraffin waxes with the melting point below 70 °C are responsible for deterioration of bitumen functional properties [8]. Aliphatic synthetic waxes obtained in the Fischer-Tropsch (F-T) process distinguish themselves by a little bit different effect. Their structure differs from the paraffin wax in bitumens. Moreover, they work together in a different way with the bitumen [9]. F-T waxes crystallize as micro-crystal structures and composed of molecules with a large number of atoms coming up to 100 [10]. Owing to their morphology they have a noticeable effect on bitumen rheological properties increasing the binder dynamic density below 100 °C and simultaneously increase in the softening point [11] as well. Synthetic waxes can be considered as a very fine filler exerts an influence of stiffness of the bitumen. However, F-T synthetic waxes decrease in the binder viscosity at the temperature over 100 °C, lowering the compaction temperature of a bituminous mixture up to 30 °C.

2. Wax modified bitumen morphology

Morphological research on the bitumen 35/50 with the 1.5% and 4% modifier content provides an object material. The keynote was indication the character of quantitative and qualitative differentiation of the wax in the bitumen. The experiment was conducted by means of epi-fluoroscent microscope. All samples, before substantially tests, have been cooled down to the temperature of +15 °C. Wax modified bitumen pictures were presented in Fig. 1.

![Wax modified bitumen morphology](image)

Fig. 1. The pictures of 35/50 bitumen modified F-T wax in amount of 1.5% (picture a) and in amount of 4% (picture b) along with quantity analysis of wax forms length

The quantity analysis of wax length forms lighten up with UV light (measured using of AxioVision 4.8 programme) has been submitted of analyze of variance. It was found that the amount of synthetic wax has the significant influence on length of wax forms (p-value < 0.001). The distribution of test results were presented in Fig. 2.

![Wax modified bitumen morphology](image)

Fig. 1. The pictures of 35/50 bitumen modified F-T wax in amount of 1.5% (picture a) and in amount of 4% (picture b) along with quantity analysis of wax forms length

Generally, it is noticeable, that the length paraffin forms are vary as of the modern wax content increase. The variability level of test results was evaluated by means of determination of confidential intervals for a mean of results at the significance level of 0.05. The increase in the quantity of wax forms with more coarse-grained character in bitumen can appear in a different manner causing the loss of the homogenization of the wax-bitumen phase and also can lead to the formation of low temperature cracking. The increase in loss of homogeneity takes place as the wax content increases. Too short distances among crystal structures of wax F-T cause the reduction of the „lubrication” effect between them and consequently will increase in the brittleness of the bitumen. The similar phenomenon was registered in the work [10].
3. Basic characteristic of the wax modified bitumen

3.1. Penetration index and breaking point temperature

The penetration index reflects an assessment of the bitumen thermal susceptibility and indicates the dynamics of changes of the bitumen stiffness. It has been determined as a result of testing two basic rheological parameters of the bitumen according to the equation:

$$PI = \frac{20 \times T_{R&B} + 500 \times \log P - 1952}{T_{R&B} - \log P + 120}$$

(1)

where: $T_{R&B}$ – softening point temperature [°C],

$P$ – penetration grade at 25 °C [x 0, 1 mm].

The evaluation of the penetration grade at 25 °C was performed according to EN 1426 [27], whereas the softening point temperature conformed to requirements of EN 1427 [28]. Moreover the penetration index results (in the plot) were presented in conjunction with breaking point temperature. The breaking point temperature results are unusually essential for assessing the adverse effect of modifier in WMA technology. The above-mentioned test result were presented in Fig. 3.
measurements of the penetration grade. The test results clearly indicate that the bitumen 35/50 modified with the additive content over 2% tend to the rheological form of gel type. In reference to the bitumen 50/70 the similar level of the penetration index is obtained with increasing modifier content amounted to 0.5% (the penetration index above +1). The quantity growth of the modifier above 2.5% clearly limits the thermal susceptibility and increases the bitumen stiffness. It should be noted that the excessive content of modern wax in bitumen could cause an increase of brittleness of bitumen what was indicated in the work [12].

3.2. Dynamic viscosity tests

The dynamic viscosity test, at first, was aimed at gauging a cohesion force in the bitumen with the synthetic wax. The second meaning of this investigation was the indication of the influence of the concentration of wax on changes into the structure of bitumens. This kind of the research was aimed for determining of minimal temperature in which there is a beginning of wax crystallization process. The investigation was performed according to EN-13702-2:2003. Results of the changing of the dynamic viscosity within the range from 50 °C to 140 °C was presented in Fig. 4.

![Fig. 4. The influence of the synthetic wax on the dynamic viscosity in the range from 50 °C do 140 °C](image)

It should be noted that for both bitumens (35/50 and 50/70), in extended analyze [26], show a two characteristic temperature ranges. The first of them is noticeable below 80 °C to 90 °C and it conformed to onset of a little curve convex upward of dynamic viscosity plot and suggests that there is a early stage of the wax crystallization process. The second temperature range is from 100 °C to 110 °C. Above this temperature range is observed the significant dispersing of the bitumen phase by the synthetic wax giving beneficial and optimal value of dynamic viscosity for proper coating the aggregate by the bitumen. The drop in viscosity value facilitates better coating of the aggregate by bitumen 35/50 in the temperature above of 125 °C. Furthermore, the localization of graph line representing the viscosity changes of 50/70 shows the predominant influence of the type of the bitumen. The bitumen 50/70 with 4.0% of the synthetic wax approximately corresponds to 35/50 bitumen variant containing 2.5% of the synthetic wax.

4. Visco-elastic properties of the wax modified bitumen

4.1. Low shear viscosity tests (LSV)

Viscosity at the low shear rate (LSV) is an approximate evaluation of the structural zero shear viscosity (ZSV). Due to the limitations of measuring devices and the lack of accurate assessment ZSV to determine the approximate level of intact structure of the bitumen was used LSV parameter [13]. This value characterizes the material when it does not reveal the shear rate dependence. Steady flow state of the viscosity and LSV evaluation were made in compliance with prCEN/TS-15324: 2006. The study, using viscometer oscillatory tests, was performed at 60 °C and the frequency of 0.005 Hz.
However, according to the test results performed by Zoorob [14] the softening point temperature tests do not reflect entirely bitumen stiffness. The result of the softening point of the bitumen gives the dynamic viscosity value equal to 1 kPAs without information about the shear rate. The results [15] also suggests that the best influence on the resistance to the formation of the permanent deformation in asphalt pavement has a LSV parameter for a value greater than 2 kPAs. Above this fixed viscosity values the mixture of MMA is resistant to permanent deformations (from heavy duty traffic). Therefore, the purpose of this study was to determine the content of modifier where the bitumen 35/50 reaches a value of 2 kPa. Test results with confidence interval ranges for mean are presented in Fig. 5.

\[
\begin{array}{|c|c|}
\hline
L_V & LSV 60 \pm 0.95 \text{ Conf. Interv.} \\
\hline
-20000 & 0.152, 0.244 \\
0 & 1E5, 1.2E5 \\
20000 & 1.4E5, 1.6E5 \\
40000 & 1.8E5, 2E5 \\
60000 & \\
80000 & \\
1E5 & \\
1.2E5 & \\
1.4E5 & \\
1.6E5 & \\
1.8E5 & \\
2E5 & \\
\hline
\end{array}
\]

Fig. 5. The influence of the synthetic wax on the low shear viscosity

4.2. Complex modulus tests

Bitumen complex modulus constitutes an full assessment of the susceptibility of the binder as a visco-elastic material. Performed the preliminary analysis concerned an evaluation of the changes of 35/50 bitumen modified with F-T synthetic wax. Basically the study has a bearing on the influence of the phase transition properties of the synthetic wax on bitumen 35/50. The analysis was performed at the frequency 1.56 Hz represents the slow-moving vehicles. Results of changes in the complex modulus as a function of the temperature was presented in Fig. 6.

\[
\begin{array}{|c|c|}
\hline
L_V (F-T wax) & 60 70 80 90 100 115 135 \\
\hline
35/50+0 & 35/50+1.5 \\
35/50+4.0 & 50/70+1.5 \\
50/70+4.0 & L_V (F-T wax) \\
\hline
\end{array}
\]

Fig. 6. The influence of the synthetic wax and the temperature on the complex modulus G*

It should be noted the complex modulus value decreases as the temperature increases. Dynamics of changes in the complex modulus is strictly subordinated to the phase transition of the synthetic wax. In the temperature range up to 80 °C the complex modulus of the wax suggests the behavior similar to the linear visco-elasticity model. In this temperature range, the bitumen has the high level of the complex modulus value. The increasing of the bitumen stiffness is particularly enhanced by modifier content of 4.0%. In the temperature range from 90 °C to 115 °C can be observed the sudden drops in complex modulus of synthetic wax. The effect of the rapid phase change has direct influence on the bitumen complex...
modulus. In the consequences for this range of temperature it may be observed a rapid decrease in the complex modulus of bitumen 35/50. Liquefaction of the bitumen is especially noticeable for bitumen 35/50 with the high dispersed phase content (asphaltenes).

4.3. MSCR tests

Two sorts of bitumen, namely 35/50 and 50/70 with a different consistency, modified with the synthetic wax F-T amounting 0%, 1.5%, 2.5%, 3.0%, 4.0% by weight, have been adopted in testing. The preparation process has involved samples with a mass of 250 g for each level of modification. Then the sample has been heated up to the temperature of 155 °C and retained at it for 30 minutes. Mixing of the bitumen with the synthetic wax has been the next stage. The binder has been agitated in a blender at 400 rpm retaining a constant temperature. The quality assessment has been carried out in accordance with [16]. The MSCR (abbreviation for Multiple Stress Creep Recovery Test of Asphalt Binder) has been carried out under the AASHTO TP70 methodology worked out to verify test results according to SHRP (the complex modulus, phase angle) as an alternative assessment for modified bitumens. That test enables an assessment of the bitumen in non-linear behavior and it distinguishes itself with a high correlation of bitumen test results with test results of compacted bituminous mixtures obtained in the course of tests in a wheel tracker (wheel tracker) [17]. The MSCR has been proposed as a better method for predicting pavement failures caused by high ambient temperatures when bitumens turn into fluids [18]. That method allows to assess the compliance and relaxation of a binder applied in a mixture and it simulated load conditions as near as possible to natural and with a non-linear behavior of the bitumen in a broad range of stress. Binder non-recoverable compliance tests have been carried out for two stress ranges of 100 Pa and 3200 Pa weighing down for 1 second and subsequently the measurement of the bitumen elastic recovery for 9 seconds at the bitumen temperature of 60 °C. The whole cycle of one range of stress have lasted for 100 seconds. Consequently, the value of susceptibility $J_{nr}$ (a non-recoverable part of deformation divided by the applied stress), and the value of elastic recovery $\varepsilon_r$ in % (the relative value of elastic strain in percentages being the ratio of deformation in the first second at the beginning of a cycle to the value of deformation in the tenth second) have been determined.

The conducted research has included measurements of the following parameters:
- susceptibility $J_{nr,100}$ (at stress of 100 Pa),
- susceptibility $J_{nr,3200}$ (at stress of 3200 Pa),
- elastic recovery at the deformation $R$ [%].

The values of the tested parameters have been estimated basing on changes of deformation of a bitumen sample in a loading-unloading process. Fig. 7 illustrates an exemplary graph of deformation changes of the bitumen 35/50 and 50/70 with the 2.5% modifier content for stresses of 100 Pa and 3200 Pa.

![MSCR Graph](image)

Fig. 7. Strain changes of the bitumen depended on dynamic stress level

An increase in the modifier amount brings about a decrease in the bitumen susceptibility. In that case, as it grows, when the process of bitumen structuring with F-T wax crystals sets in motion, it should be expected at least the linear growth of rut resistance [12] and a rise of the resilient stiffness modulus of the asphalt concrete. The tendency of changes for two ranges of stresses is similar. The test results of 35/50 binder creep compliance $J_{n100}$ at stress 100 Pa and $J_{n3200}$ at 3200 Pa were presented in Fig. 8.
Error bars represent 95% confidence interval for the mean. The compliance of bitumen is increased at high stress level 3200 Pa. Differentiation between values registered for bitumen 35/50, coming out for the high value stresses, may be caused by disturbing the bitumen structure [13]. In any case, the creep compliance of the modified bitumen 35/50 within a given range is lower with the increase of concentration of synthetic wax. Observing the bitumen compliance it should be noted that the concentration of synthetic wax approximately of 2.5% in the bitumen gained creep compliance level (Jnr3200) approximately of 0.5 kPa$^{-1}$, which conforms a heavy traffic load [16].

The second binder parameter was the elastic part of deformation R100 at stress 100 Pa and R3200 at 3200 Pa (Fig. 9).

The tested bitumens have sustained a similar growing trend of the elastic recovery with low stresses. The value of elastic recovery has increased along with the bitumen type. This fact fully correlate with results of the compliance and it is associated with an increase in the modulus. It extend the size of stress in which the bitumen is in the viscoelasticity range [12]. The higher elastic recovery of the bitumen 35/50 involves the higher level of structural density and the higher level of a maximal shearing stress causing a disturbance of the equilibrium state of the bitumen colloidal system. Therefore it is safe to say that proportioning of the synthetic wax F-T by an appropriate quantity may significantly improve the bitumen rut resistance endowing bituminous mixtures a more elastic character at the temperature 60 ºC which is regarded as the highest summer pavement temperature.
5. Optimal solution of synthetic wax content

Finding the optimal solution depends on the boundary conditions (the criteria to be met by bitumen). The optimization object was the bitumen 35/50. The main objective of this analyze was to gain the increase in stiffness and maintaining of bitumen flexibility at low temperatures as well. For this purpose, the bitumen 35/50 was modified with the different content of the synthetic wax. The domain of experiment with the synthetic wax (L_V) was in the range of 1,5%, 2,5%, 3,0%, 4,0%. Criteria for parameters, which should be applied, depends on the desired final result and the criteria required by European standards. For this purpose, a utility function algorithms "distance-weighted least squares smoothing procedure" [19] was used. Utility function varies in the range <0,1>. The values that are close to 0 are not allowed, and close to 1 are treated as desire. Each variable, used for the optimization, has been standardized in the range of 0-1. Determination of optimum solutions of desire function was evaluated by means of intervals proposed in scope [20]. The result of the utility function below 0,36 is considered as unacceptable. The list of the criteria used to obtain the optimum solution is presented in Table 1.

Table 1. Desirability function settings for each dependent variable

| Variable (criteria) | Desirability function parameters | Desirability function settings for each dependent variable |
|---------------------|----------------------------------|----------------------------------------------------------|
| Pen (Penetration grade) (EN 1426) | 35* | 1.0 | 37.5* | 0.5 | 50* | 0 |
| T_R&B (Ring and Ball Temperature) (EN 1427) | 60* | 0 | 72.5 | 0.5 | 89.0 | 1.0 |
| Fraass (breaking point temperature) (EN 12591) [29] | -12* | 1.0 | -7.5* | 0.5 | -5.0* | 0 |
| G' – 60 (elastic part of complex modulus G* according to SHRP) | 1000* | 0 | 36565 | 0.5 | 72444 | 1.0 |
| PI (Penetration Index) [21] | 0* | 1.0 | 1.0* | 0.5 | 2.0* | 0 |
| n60 (dynamic viscosity at 60°C) [29] | 220* | 0 | 7097 | 0.5 | 13740 | 1.0 |
| n135 (dynamic viscosity at 135°C) [29] | 0.7* | 1.0 | 1.0 | 0.5 | 2.0 | 0 |
| LSV60 (low shear viscosity at 135°C and 0.005Hz) [11] | 2000* | 0 | 74639 | 0.5 | 147911 | 1.0 |
| Jnr3200 (creep compliance at shear stress of 3200 Pa and at 60°C) [16] | 0.5* | 1.0 | 2.0* | 0.5 | 4.0* | 0 |

L_V, M_V, H_V - low, medium, high value respectively;
D.V - Desirability value;
* - to which criteria concerns (the rest are taken from experiment domain limits);

The main purpose was obtaining the bitumen with the lower penetration grade than the reference bitumen (35/50 without modification), and the higher softening point temperature than 60 ºC. In addition, the wax modified bitumen had to be characterized by a proper module G' (part of the elastic modulus G*) and the satisfactory level of a flexibility at low temperatures. The multicriteria optimization result is presented in Fig. 10.

The first column in multicriteria analyze (Fig. 10) represents results of the effect of the wax content on specific parameters (L_V). The second column represents a range of desired values from 0 to 1 subordinated for a specific parameter (according to Table 1). The last row in located in the first column denoted "Desirability" constitutes a summary result of the wax influence with its optimal content. Multicriteria optimization result shows that the best solution for modification bitumen 35/50 is the application of the synthetic wax in amount of 3,0%. For this wax content value the utility function (U) amounted to 0,549. This result indicates that this value is within the range of acceptable solutions <0,36 – 0,63> [20]. However, from the point of view of the application, the optimal solution of the wax dosage is in the range between 2,5% and 3,0%. In this range, it is possible to obtain a material with the optimal parameters. Above the modifier content of 3,0% it was occurred decline in the value of the utility function. This is due to a drastic drop in the breaking point temperature of the bitumen. Therefore, the increase in stiffness of the bitumen affects decreasing the compliance of bituminous mixes and
in parallelly high probability of the low-temperature cracking. Considering the optimal dosage range of modifier the length of paraffinic forms in the bitumen phase (35/50) should be in the range from 7 to 10 microns. Such dispersion of the modern wax should ensure the homogeneity of the composition of the bitumen and achieving its required rheological properties.

![Profiles for Predicted Values and Desirability](image)

**Fig. 10. Multicriteria analysis of binder modification**

6. Conclusions

On the base of test results the following conclusion can be drawn.

- The use of synthetic wax causes an increase in penetration index regardless of the type of the bitumen;
- The synthetic wax content excessive causes an increase in the breaking point temperature of the bitumen;
- The presence of the synthetic wax and its transition directly affects the complex modulus of the neat bitumen;
- Tests of dynamic viscosity throughout a temperature sweep starting at 60 °C revealed the liquefaction effect of the bitumen at the temperature of 125 °C which contributes the better compaction of asphalt layers;
- LSV viscosity test at 60 °C indicates that the use of the synthetic wax over 2% results in significant reduction in the plastic deformation of the bitumen;
- MSCR tests at 3200Pa stress revealed that bitumen 35/50 containing 2.5% of the synthetic wax can be used for heavy traffic roads;
- Multicriteria optimization algorithms showed that the best properties of bitumen 35/50, at the set criteria, has been achieved for the concentration of the wax within the scope of 2.5% – 3%;
- Decreasing trend utility function suggests that the increase in the synthetic wax modifier above 3.0% adversely affects the properties of the neat bitumen;
- The optimal amount of the modifier of bitumen 35/50 corresponds to the paraffinic structures length within the range from 7 to 10 microns, thus ensuring the adequate level of the "lubrication" effect between crystals in the bitumen.
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