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Analysis of the effect of aging process on selected F-T wax modified bitumen

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Abstract

Effect of adding different amount of the modern wax (F-T wax) to three bitumens such as 35/50 and 160/220 penetration grade were studied. The main objective of the experiment was to evaluate a changing in rheological properties of the bitumen with a synthetic wax before and after aging process according to RTFOT method and supported by the Multiple Stress Creep Recovery test (MSCR). The experiment was conducted by means of different types of laboratory equipment, such as rotational viscosimeter, ductilometer as well as equipment for determining basic rheological parameters like softening point temperature, penetration grade. Moreover an analysis was enriched by epifluorescence microscope observation of wax forms. The results show that the type of the bitumen had a decisively influence on aging process of the wax modified bitumen. The conducted analysis did not reveal a strong correlation between the modern wax content and aging rate of the bitumen. Additionally, the interaction between a modern wax content and a type of bitumen affected changes in breaking point temperature.

Keywords: MSCR test; aging; F-T wax; rheology.

Nomenclature

J_{nr}	creep compliance (kPa^{-1})
ER	elastic recovery (%)
G^*	complex modulus (Pa)
G''	loss modulus (Pa)
RTFOT	The rolling thin film oven test
MSCR	Multiple Stress Creep Recovery Test
Greek symbols	
δ	phase angle (deg)
η	dynamic viscosity (Pa·s)

1. Introduce

The aging of asphalt mixtures is a change process of physical and mechanical properties in the bitumen [5]. This phenomenon takes place during production, the storage, the transport and the placing of asphalt mixture. In addition, the aging of the bitumen is continued for service life of the asphalt pavement. Mainly, the aging process affects the increase in the stiffness of the binder parallelly with reduction of its plastic parts of complex modulus (G*) which leads to embrittlement of bitumen at low temperatures and affects the reduction in its resistance to climatic factors [4]. Oxidation of the bitumen can be attributed to the mechanism of the radical-type of oxidation (radical hydrocarbon and hydrocarbon radical peroxide). The reaction of the oxygen in the presence of UV radiation and temperature generates permanently in bituminous mixtures, radical products from oxidation process of hydrocarbon compounds [14]. The aging of the bitumen, in terms of the evaporation of the lighter components like oil, takes place in a high temperature and it can be simulated by RTFOT process.

Despite the large amount of information on the beneficial effects of synthetic waxes on the stiffness of asphalt mixture [17] little attention is paid to the presence of a synthetic wax in different bitumens in the interaction with the aging process

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affecting structural properties of the binder. This paper presents an attempt of the interaction analysis of short-term aging of synthetic wax and the bitumen kind and also their influence on the creep parameters using the methodology AASHTO TP-70 (MSCR test).

2. Materials

2.1. Bitumens

In the experiment, non-aged bitumens with penetration grade 35/50 and 160/220 derived from Plock petrochemistry were used. The choice of bitumens with such levels of the consistence at 25ºC was dictated by having two groups of bitumen with a different amount of asphaltenes and maltenes content. Due to the oxidation process, the intensification of changes in bitumen properties is different [5], what we can expect, especially in stiffness the bitumen before and after aging in the presence of the synthetic wax. The presence of a small amount of synthetic wax should invoke an increase in brittleness and therefore the effect of changes in mechanical properties will be more underlined.

2.2. Synthetic wax (Fischer-Tropsch synthesis wax)

In the study one of the representatives of microcrystalline waxes derived from the Fischer-Tropsch synthesis was used. Its structure differs from the bitumen paraffin waxes, thus they differently affect the neat bitumen and its natural incorporated paraffins [11]. FT-waxes crystallize as micro-crystalline structures containing a large number of carbon atoms up to 100 [12]. Thanks to FT-waxes morphology they significantly influence on the rheological properties of the bitumen and they cause an increase in the dynamic viscosity of the binder in the temperature range below 100 °C and subsequently cause an increase in the bitumen softening point temperature [7]. Their presence reminds the presence of very fine filler. At temperatures above 100 °C synthetic waxes reduce rapidly the viscosity of the bitumen lowering the compaction temperature of bituminous mixtures up to 30 °C.

3. Test methods

3.1. Experiment Design

The scope of the experiment assumed division of samples series into two subsets. The grouping factor was the conditioning process (aging asphalt) according to the method *RTFOT*. Measurements of the bitumen properties of each subsets were made using two kinds of bitumen with a penetration grade 35/50 and 160/220. All bitumens were undergone the modification with the synthetic wax in an amount of 3% and 6%. In addition, in studies were took a part bitumens without modification as reference samples. Thereby, each of the two subsets contained nine experimental cases.

3.2. Modification of the bitumen by synthetic wax

The first step was the separation of the bitumen sample for each dosage level, in an amount of 250 g. Each samples was heated up to 155°C and maintained at this temperature for 30 minutes. The intermixing of the bitumen with the synthetic wax has been the next stage to increase its homogenization at a constant temperature and rotation speed 400 rpm. The quality evaluation was made according to EN 12594:2004 and the information in the study [6].

3.3. Bitumen properties

Basic properties of the bitumen were a preliminary study on an influence of the synthetic wax on bitumen properties. The following tests were considered: penetration grade according to EN 1426, softening point temperature according to EN 1427 and breaking point temperature according to EN 12593. At this stage, as a rheological tests, an assessment of the dynamic viscosity as a function of temperature was carried out. The dynamic viscosity versus temperature was examined in accordance to EN 13702-2:2009. The final result, screened in the graph, represents the average of three independent simple viscosity measurements. They did not differ more than 5%. The bitumen sample with a synthetic wax was added to the cylinder in an amount of 52g.The value of the dynamic viscosity at specific temperatures was determined after reaching the bitumen equilibrium state. The equilibrium state was determined as a period of 15 minutes and after this time the measurement of viscosity was done. Due to the non-Newtonian behavior of some actual bitumen the dynamic viscosity was tested at a shear rate of 0.5 s^{-1}. This was the shear rate which corresponds to a shear stress at which the steady flow starts up especially for bitumen at temperatures below 80ºC. The temperature measurement range was from 60°C tests to 140°C in steps of 10 °C.

3.4. MSCR test

The *MSCR* (abbreviation for Multiple Stress Creep Recovery Test of Asphalt Binder) has been carried out after the AASHTO TP70 methodology worked out to verify the test results according to *SHRP* (the complex modulus, phase angle) as an alternative to modified bitumens, especially to those modified with polymers. This test states a simplified analysis of creep-relaxation curve [13]. MSCR test enables the assessment of non-linear bitumen behavior and it distinguishes itself with a high correlation with bitumen test results of compacted bituminous mixtures obtained in the course of tests in a wheel tracker (rutometer) [9]. The MSCR has been proposed as a better instrument for predicting pavement failures brought about by high ambient temperatures when bitumen turns into dense fluids [10]. That method allows to assess the creep compliance and stress relaxation of a binder applied in a mixture in simulated load conditions as near as possible to natural in a broad range of stress. Binder susceptibility tests by means of measuring irreversible deformations have been carried out for two stress ranges of 100 Pa and 3200Pa weighing down for 1 second, followed by 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 has lasted for 100 seconds. Consequently, the value of creep compliance J_{nr} (a non-reversible part of deformation divided by the applied stress) in kPa^{-1}, and the value of elastic recovery *ER* 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.

3.5. RTFOT methodology

The rolling thin film oven test (RTFOT) measures the effect of heat and air on moving film of bituminous binder (EN 12607-1:2009 Bitumen and bituminous binders – Determination of the resistance to hardening under the influence of heat and air - Part 1: RTFOT method). The amount of 35g bitumen sample was placed in a glass bottle. The glass containers were placed in a carriage such that the axis of revolution was horizontal was subjected to hot air action. The temperature was maintained at 163°C and the carriage was rotated in the oven at a rate of 15 rpm for 85 min. This thin bitumen film was exposed to the oxidation process similar to conditions experienced in the production of hot mix asphalt concrete (aging).

4. Test results analyze

4.1. Investigating the effect of aging process on basic bitumen properties

In the first stage of the study all cases of the synthetic wax modified bitumen before and after short-term aging process were analyzed. The scope of research concerns the analysis of the basic properties such as penetration grade (*PG*), softening point temperature ($T_{R\&B}$) and temperature of breaking point (T_{FRAASS}). The purpose of these measurements was the initial diagnosis of the impact of the aging process non-aged subsets of bitumen samples (*Neat*) and subjected to the process of aging (*RTFOT*). The interactions between properties, above mentioned, were presented in uniform scale in Figure 1.

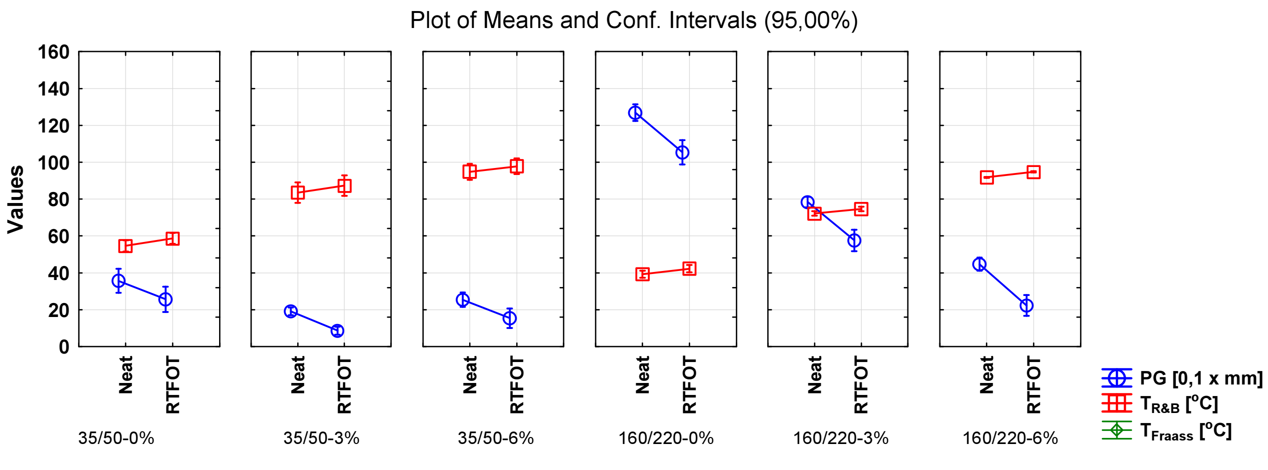


Fig 1. The interaction graph between three variables such as: Penetration Grade (*PG*), softening point temperature ($T_{R\&B}$) and Fraass breaking point temperature (T_{FRAASS})

The results indicate that the aging process in relation to the bitumen was noticeable. However, its intensity is much faster in the soft variant of tested bitumen 160/220. For bitumen 35/50, analyzing the increase in the softening temperature and the penetration grade, the increase in the stiffness is slower than in the bitumen 160/220. With respect to the breaking point temperature parameter the growth rate in stiffness was comparable regardless to bitumen kind. However, the precise evaluation of the stiffness of bitumen cases, after aging, is ambiguous due to the permissible tolerance in range ±2 °C. The accompanying chemical reaction conversion of the compounds of bitumen and evaporation of light oils were much more expressive for the soft bitumen which malthene fractions comprise larger portion than in the bitumen 35/50. This type of bitumen contains more asphaltenes fraction and therefore there is less risk of increase in bitumen stiffness by condensation of aromatic compounds. However, it should be noted that the increase in wax content in bitumen does not affect aging of bitumen. Accordingly, the rise in stiffness of bitumen depends on a kind of bitumen. Knowing that the distribution of synthetic wax occurs at temperatures above 180°C its effect during the aging process will be negligible.

4.2. Dynamic viscosity data from temperature sweep test

The next analyzed parameter was the evaluation of changes in dynamic viscosity as a function of temperature. This parameter allows for the comparison of the dynamic viscosity behavior of different bitumen, in this case a synthetically wax-modified bitumen in a steady flow state, which is at a fixed orientation of molecules (Newtonian fluid). Accordingly, the study was performed at a constant shear rate and a non-Newtonian flow should be expected especially in case of using hard bitumen [3]. The aging process and the presence of a slight quantity of synthetic wax in the bitumen will affect the structure and shape of the molecules in the asphalt mixture. This fact will be mapped in the evaluation of the level of energy required to overcome the intermolecular forces which block sliding of the layers of the liquid [2]. The dependence of dynamic viscosity versus temperature is shown in Figure 2.

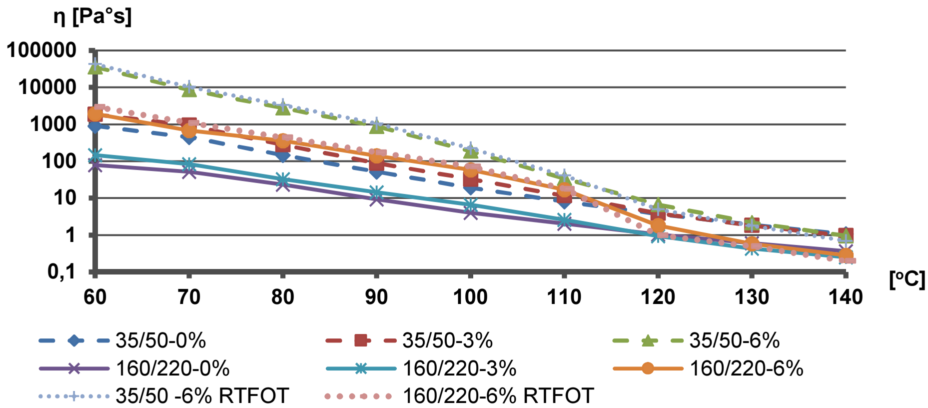


Fig. 2 The dynamic viscosity *η* versus temperature *T*

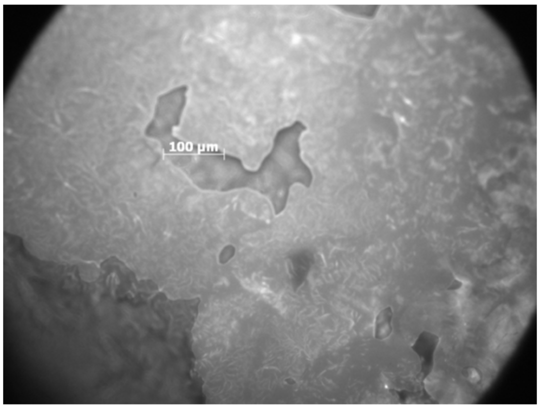
On the graph of changes in dynamic viscosity as a function of temperature, for clarity, the cases of bitumen results 35/50 and 160/220 after aging with the addition of 6% synthetic wax were given. It should be noted that as the amount of synthetic wax increased the dynamic viscosity increased rapidly in the range from 60 °C to about 115 °C. At temperatures above 115 °C the dynamic viscosity drops in value. The temperature of 115 °C is an approximate in which a dynamic transition of wax phase starts up. It is connected to the exceeding a stress yield point of the synthetic wax. This fact indicates that mixture compaction below that temperature (yield point temperature) makes difficult and decreases a workability of bituminous mixture. The increase in dynamic viscosity in relation to the unmodified bitumen, after aging, fully correlates with an increase in the softening point temperature of bitumen increasing the level of bitumen stiffness at operational temperatures. Conversion reactions of the bitumen compounds and expansion of the asphaltenes micelles lead to the increase in molecular weight of the bitumen. Therefore, the mobility of the bitumen is decreased. The lack of maltenes compounds and their rapid loss in the bitumen cause the increase in stiffness of the bitumen. In the pictures (Figure 2) the dynamic viscosity of the bitumen 160/220 (soft), after aging, (*RTFOT*) and its modification with synthetic wax in the amount of 6% correspond to the bitumen 35/50 modified with 3% before a short-term aging. Changes of viscosity as a function of temperature is described in line with the Arrhenius formula:

$$\eta = A \cdot e^{\frac{E}{RT}}, \qquad (1)$$

where: *A* – constance that is characteristic for the specific liquid; *E* – activation energy; *T* – temperature.

It should be noted that the parameter *A* in formula (1) is strongly associated with a molecular weight of the bitumen kind. Bitumen fraction enriched with synthetic wax is characterized by an increase in this parameter. A similar relation appeares in the aging process. Due to the wax compound is not disintegrated in the temperature range thus the increase in the molecular weight of bitumen is related to the existence of two factions in the bitumen: asphaltene micelles and synthetic wax content. In addition, the curvature of the dynamic viscosity graph and the related activation energy *E* parameter, dynamic viscosity results indicate that the wax modified bitumen requires a lot of energy to reach a state of Newtonian flow. After the destruction of the undisturbed bitumen state the rapid decrease in dynamic viscosity appears [8]. The increase in the stiffness of bitumen 160/220 with 6% of synthetic wax content in relation to the wax content of 3% may lead to very high shrinkage and high brittleness of bitumen. This paradox (for soft asphalt) may be caused by the fact that the aging of the soft bitumen progresses quicker. Therefore, it leads to uncontrolled aggregation of the wax crystal forms caused by lack of maltenes compounds during conversion reactions (especially resin) [16]. It also brought about an increase in the thixotropy of bitumen. In addition, photographs of the bitumen were taken after bitumen aging (*RTFOT*) 35/50 and 160/220 modified 6% by synthetic wax. Photographs are shown in Figure 3.

a) b)



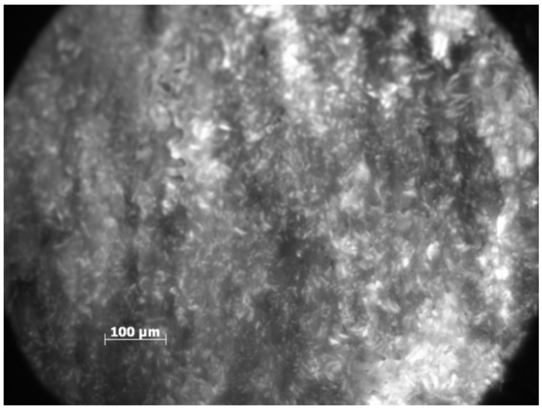


Fig. 3 UV microscopic photograpiesof : a – bitumen 35/50-6% ater *RTFOT*; b – bitumen 160/220 - 6% after *RTFOT*

In the enclosed photographs a varied aggregation of microcrystalline wax forms can be seen. In the case of the bitumen 160/220 the aging process reduced the level of resins playing a role as the stabilizer. Therefore, the soft bitumen (160/220) modified with the synthetic wax, after aging, may exhibit a higher thixotropy and exist as the harder bitumen.

4.3. Multiple Stress Creep Recovery Test (MSCR)

The *MSCR* tests were performed for two stress ranges namely 100Pa and 3200Pa. The first level represents the behavior of bitumen in the linear visco-elasticity stress range, where the relation between *G** (storage modulus) and the deformation is constant. The level of shear stress 3200Pa reflects the conditions of heavy load traffic. This range represents also the state after exceeding a yield stress of the bitumen. At this juncture bitumen behavior is close to Newtonian fluid model. It should be noted that the process of the rapid growth in the rutting surface at a high temperature refers to the situation in which the bitumen behaves as visco-elastic material with the supremacy of viscous part of the complex modulus. Bitumen stiffness is highly shear stress dependent. Therefore, the assessment of the susceptibility of the bitumen for both stress levels sheds a light on the non-elastic behavior of bitumen in bituminous mixtures. Moreover, the evaluation of creep compliance of the bitumen for a high level of stress allows for assessment of the bitumen in terms of the decrease in its elasticity under the effect of high stress. Deformation of some cases of synthetic wax modified bitumen with an applied shear stress 3200 Pa are presented in Fig. 4.

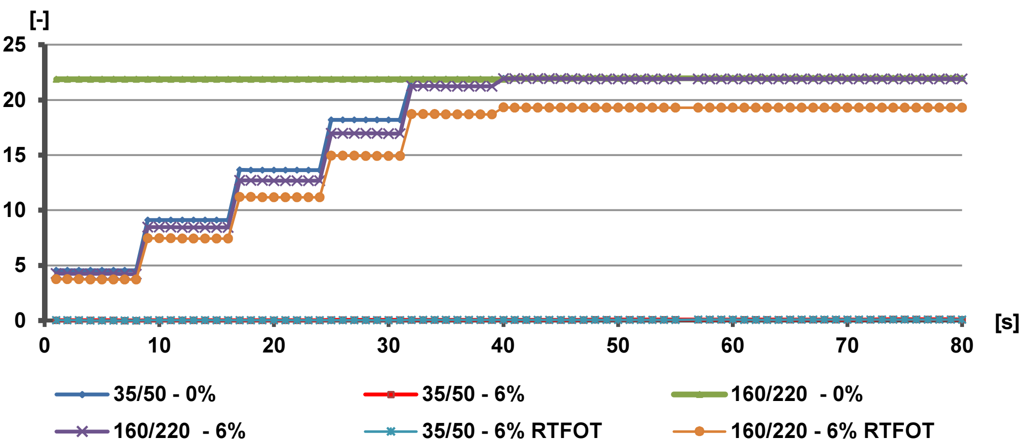


Fig. 4 Deformation versus time for shear stress 3200Pa

The test results shown in the graph reveal that for a given stress the bitumen 160/220 is within steady state flow and reaches a Newtonian fluid. This state of affairs confirms low level of its softening point which equals to 40°C. In the case of the bitumen 35/50 modified with synthetic wax in an amount of 6%, regardless of the aging process, the level of total strains is small as well as the level of non-recovered strains. It should be noted that the aging process in relation to the bitumen 160/220 modified with synthetic wax in an amount of 6% causes a decrease in the level of deformation in the graph and certainly can be found in creep compliance of the bitumen. Considering the bitumen 35/50, before aging, the strain level is comparable to non-aged bitumen 160/220 with the addition of 6% synthetic wax. After fourth loading cycle both bitumen obtained deformation limit for the measuring device which indicates reaching a steady flow of bitumen. Despite the fact that the deformation character of non-aged bitumen such as 160/220-6% and 35/50 are at comparable levels and the softening point temperatures are different and range between 60 °C and 80 °C. Therefore, the softening temperature cannot be paramount parameter for determining the bitumen susceptibility of rutting process. The complexity of the phenomena accompanying the determination of softening point and its usefulness revealed Zoorob *et al*. [18]. On the basis of the deformation for each 10 load cycles two rheological characteristics are determined. The first deals with a size of non-recovered deformation in a single load cycle and thus a level of elasticity. It was denoted as creep compliance J_{nr}. The second parameter describes the level of elastic deformation which returned after the specified stress and is marked as percentage of elastic recovery *ER*.

The experiment consists three factors such as: binder kind, aging and synthetic wax content. Evaluation of the effect of these factors is determined by multivariate analysis of variance. The results of analysis, with respect to parameter *p*-value was shown in Table 1.

Table 1. Multivariate analysis of variance

Effect	Univariate Results for Each DV Sigma-restricted parameterization Effective hypothesis decomposition			
	Jnr_{100} [kPa^{-1}] *p*-value	Jnr_{3200} [kPa^{-1}] *p*-value	ER_{100} [kPa^{-1}] *p*-value	ER_{3200} [kPa^{-1}] *p*-value
Intercept	**< 0.0001**	**< 0.0001**	**< 0.0001**	**< 0.0001**
Aging	0.071396	**0.014039**	0.263488	0.195921
Bitumen kind	**< 0.0001**	**< 0.0001**	**< 0.0001**	**< 0.0001**
WC	**< 0.0001**	**< 0.0001**	**< 0.0001**	**< 0.0001**
*Aging*Bitumen kind*	0.220072	0.124498	0.775685	0.205722
*Aging*WC*	0.628467	0.604184	0.822711	0.310237
*Bitumen kind*WC*	**< 0.0001**	**< 0.0001**	**< 0.0001**	**< 0.0001**

Analysis of variance was carried out at a significance level of 0.05. All the results of the parameter p-value less than 0.05 imply the adoption of the alternative hypothesis on the differentiation between levels of factors. Furthermore, the analysis included an assessment of the interaction significance between the independent variables (factors). The results of analysis point out that the effect of aging (*Aging*) has a significant influence on the results of the creep compliance at a stress 3200Pa. With regard to the level of stress of 100Pa, an ascertainment of the aging impact on the level of the creep compliance of the bitumen was not clear. In other cases the aging effect varied the test results of J_{nr} but only for the measurement error approximately 20%. This fact is associated with a slight disproportion between test results before and after aging process and the mean of estimated error. This result also points out that changes of parameters J_{nr} and *ER* for both bitumen after aging are proportionally similar. In contrast, it should be noted that both the type of bitumen (bitumen kind) and the level of bitumen modification (*WC*) caused a significant impact on the distribution of creep parameters.

Only in the case of interaction analysis between factors: the amount of synthetic wax (*WC*) and bitumen (bitumen kind) significantly affected the creep parameter according to the *MSCR* methodology. For other cases of interactions between considered parameters in which the aging process took part the significant differences for values of J_{nr} and *ER* parameters were not obtained. Particularly relevant is the fact that there was the lack of interaction between the amount of synthetic wax and aging process. All the information reflects the interaction graph in Figure 5.

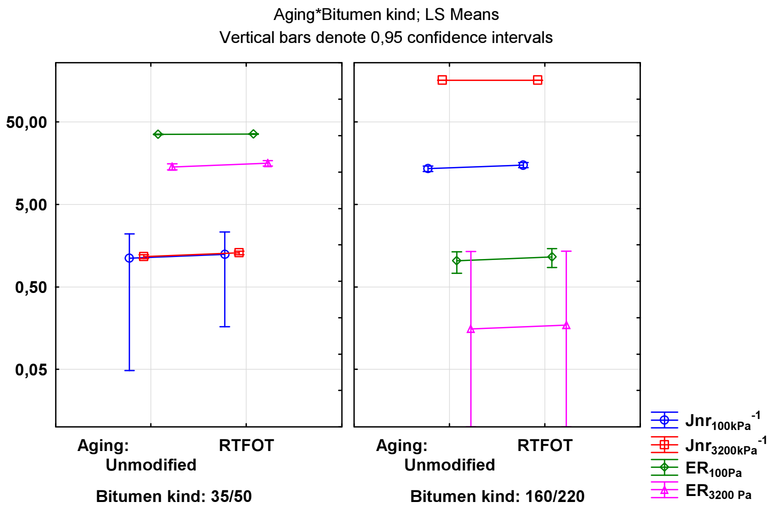


Fig. 5. Interactions graph between factors such as *Aging* and *Bitumen kind*

The conclusion is, that during the aging process of the bitumen there are not chemical changes observed in the wax as well as the synthetic wax is not chemically broken up at the aging temperature amounted to 163°C. Furthermore, the impact of the aging process and the amount of synthetic wax on creep parameters of bitumen should be treated as two separate effects. Changes in the creeping process according to *MSCR* will depend on factors such as: the kind of bitumen, synthetic wax content and the shear stress level. At the current stage of research and at a similar level changes in J_{nr} and *ER* it is difficult to make a final decision, at a significance level of 0.05, that there is an interaction between the kind of bitumen and aging process. However, increasing slightly to 0.3 significance level causes that the effect of the interaction between the aging and kind of bitumen could be registered. Therefore, further experiment should be extended for greater cases of bitumen from different origins.

Comparing the results of creep compliance J_{nr} and elastic recovery *ER* along with the validating curve of the polymer quality modification of bitumen [1] it is possible to obtain information on the behavior of the bitumen at high shear stress state (3200Pa). Curve $ER = f(J_{nr})$ represents the quality of polymer modified bitumen in the end of the use. Summary results are shown in Fig. 6.

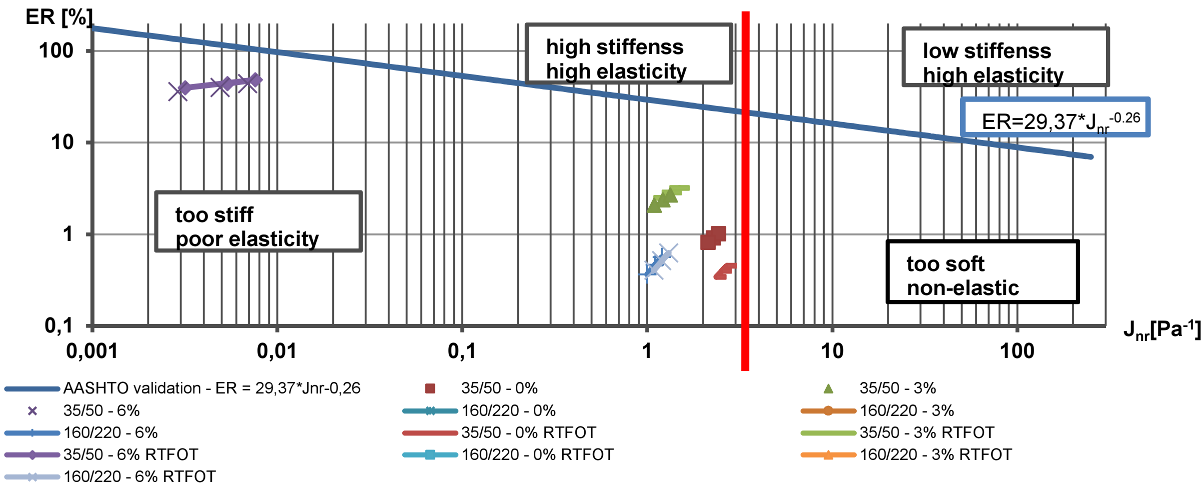


Fig. 6 The plot elastic recovery *ER* versus creep compliance J_{nr} with AASHTO TP 70-09 specification chart (for 3200 Pa shear stress).

It should be noted that for a given stress level the bitumen modified with wax cannot be related to polymer modified bitumen. The increase in wax content in the bitumen (over 3%) causes that the bitumen corresponds to the elastic-brittle model much stronger than elastic one. The embrittlement increase enhances the aging process. Therefore, an excessive amount of wax may reduce the flexibility of the modified bitumen at a low temperature leading it to asphalt pavement cracking. This phenomenon is more observable in the case of high thixotropic material behavior (after *RTFOT* aging).

The last evaluation of the test results of creep parameters was a summary of test results at the stress 100Pa.This shear stress level represents the approximate behavior of the bitumen in the linear visco-elastic range (*LVE*). Using the fact that there is a strong correlation between the $1/J''$ (creep compliance) and $G^*/\sin\delta$,especially for non-modified bitumen (with a little elastic recovery) by means of *MSCR* test, it is possible to determine a minimum stiffness of bitumen in terms of its usage in bituminous mixture (rutting test). Therefore, approximate J_{nr} at 100 Pa value less than 1 kPa^{-1} meet the requirements of *SHRP* methodology [15] where for the non-aged bitumen *G''* value automatically should be greater than 1000 Pa. The test results are presented in Fig. 7.

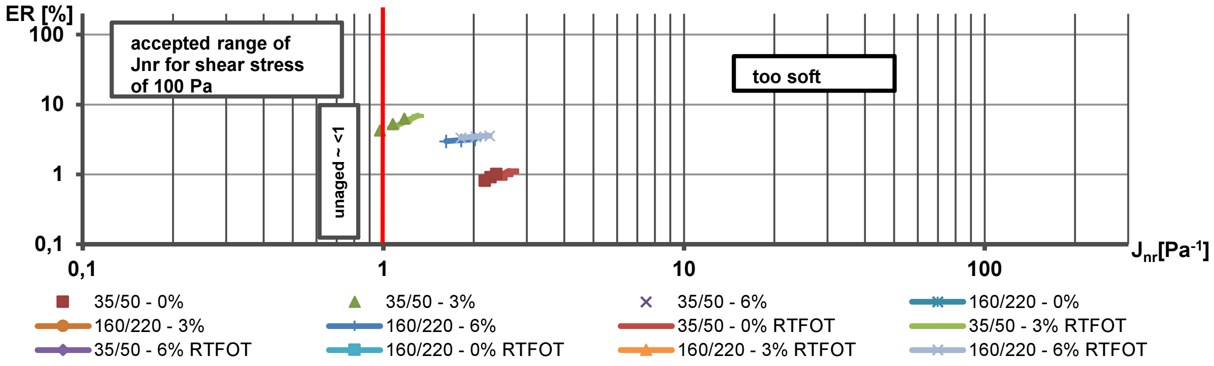


Fig. 7 The plot of elastic recovery changes *ER* [%] vs. creep compliance J_{nr} [kPa^{-1}] at 100Pa shear stress

Summary of the results indicates that, in general, the case of hard bitumen denoted as 35/50 meet the requirements for minimization of permanent deformation at high operational temperatures. The bitumen 160/220 with a high synthetic wax contamination did not meet the minimum requirements for the classification of *SHRP*. Despite the reduction of creep compliance through the modification this bitumen (160/220) still shows the excessive sensitivity to the level of shear stress. This is related to the fact that the linear visco-elastic range is below 50Pa, for this specific bitumen, and excessive thixotropy disturbs homogeneous structure of the modified bitumen. This kind of bitumen quickly turns into liquid (a state of Newtonian fluid).

5. Conclusions

On the basis of the test results the following conclusions can be drawn:

- the aging process influences the changes in basic characteristics of bitumen 35/50 and 160/220;
- the effect of short-term aging and the synthetic wax content causes an increase in the stiffness and brittleness of considered bitumen, lowering its consistency at 25 °C;
- the test of dynamic viscosity versus temperature revealed that the temperature approximately of 115 °C liquefied the bitumen. This phenomenon is caused by the effect of the phase transition of synthetic waxes and their amount in the bitumen;
- aging process raises the level of dynamic viscosity of the bitumen;
- dynamic viscosity of the bitumen increases with respect to the parameter *A* in the Arrhenius model indicating the increase in molecular weight of the bitumen;
- the interaction between the aging process and the amount of synthetic wax was not revealed with respect to the creep properties according to *MSCR* test .Therefore, the presence of a wax may be treated as a very fine filler in the bitumen;
- the kind of bitumen and synthetic wax content significantly contribute to the diversity of the creep compliance J_{nr} and elastic recovery *ER* of bitumen;
- aging process invoke the changes in creep parameters of the bitumen with respect to compliance parameter J_{nr}. Difficulties in resolving the impact of aging on the creep at low shear stresses indicate that this study should be extended to other cases of bitumen;
- analysis of elastic recovery as a function of creep compliance reveal that the bitumen, which undergone to the process of aging with the high synthetic wax content, behaves as the elastic-brittle material;
- bitumen 160/220 in relation to the creep compliance J_{nr} at 100Pa, which is the equivalence of modulus *G''* and despite the high synthetic wax content, did not meet all the requirements connected to providing the rutting resistance of asphalt pavement.

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