The Effect of Synthetic Wax and Technological Aging on the Rheological Properties of Polymer Modified Asphalt Binders

Marek Iwański[1*)], Małgorzata Cholewinska[2)]

[1*)] Faculty of Civil Engineering and Architecture Kielce University of Technology, Al. Tysiąclecia P.P, 7, 25-314 Kielce, Poland, iwanski@tu.kielce.pl; ORCID 0000-0002-3183-0141

[2)] Faculty of Civil Engineering and Architecture Kielce University of Technology, Al. Tysiąclecia P.P, 7, 25-314 Kielce, Poland, mcholewinska@tu.kielce.pl; ORCID 0000-0001-8369-3428

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Abstract

The intensive development of transport and especially the increase in the number of trucks with a general traffic structure makes it necessary to use polymer-modified asphalts binder in the production of asphalt mixtures. They are characterized by high physical and mechanical parameters and can meet the requirements that are imposed on them. However, as a result of technological ageing during the production of the asphalt mixture and operational ageing, the binder parameters are reduced. In order to slow down this process, various types of additives are used, which act as ageing inhibitors. These types of materials include low-viscose additives, which include Fisher-Tropsch synthetic wax. At the same time, such additives affect the reduction of technological temperatures of the production and incorporation of the asphalt mixture, which is very beneficial in terms of environmental protection. There are reductions in greenhouse gas emissions and the energy consumption of the production of the asphalt mixture is reduced. The synthetic wax F-T was used in the tests, which was dosed to the PMB 45/80-65 polymer asphalt binder in the amount of 0.0%, 1.5%, 2.5% and 3.5%. Then the binder was subjected to technological ageing (short-term ageing). In the first stage of the tests, the penetration at 25°C, softening temperature and critical temperatures during the low-temperature parameter testing were determined. In the second stage, the following rheological characteristics were tested in the range of linear and non-linear viscoelasticity: zero shear η_0, dynamic modulus $|G^*|$, phase shift angle δ and MSCR parameters. Based on the analysis of the test results, it was found that the synthetic wax reduced the susceptibility of the polymer asphalt at medium and high operating temperatures and affected the reduction of its ageing rate while increasing the elastic part of the dynamic modulus. Synthetic wax increased the zero shear viscosity level of the binder, which will have a beneficial effect on reducing the deformation rate of the asphalt mixture and ensure the required operational durability of the surface.

Keywords: *polymer modified asphalt binders*, *short-term ageing, synthetic wax, physico-mechanical properties*

1. Introduction,

The road industry faces many challenges today. One of these challenges is ensuring the required service life of asphalt pavement. The increasing loads on roads and the growing proportion of heavy vehicles in overall vehicle traffic make it necessary to use polymer modified bitumen in mineral asphalt mixtures. This increases its resistance to permanent deformation. Unfortunately, a disadvantage is that they must be heated to about 180°C during the production of the mineral-asphalt mixture.

An important problem associated with using bitumen in mineral asphalt mixture technology is its aging. During production, transportation from the petrochemical plant to the asphalt mix plant, and manufacturing, the binder is affected by high temperatures and oxygen in the air. As a result, unfavorable changes occur in the bitumen, it oxidizes and stiffens, which can adversely affect the quality of asphalt pavements. This stage of asphalt aging is called technological or short-term aging [1]. However, research shows that it plays a significant role in the bitumen aging process. To counteract aging, various types of natural [2-5] and artificial [6-10] additives are added to the asphalt to slow or even significantly reduce aging [11-14].

Another challenge for the road construction industry is reducing energy consumption by using mineral-asphalt mixtures at lower manufacturing and incorporation temperatures. These materials are environmentally friendly because lowering the asphalt's temperature reduces greenhouse gas emissions, primarily CO_2, and provides many social and economic benefits [15-16]. Implementing asphalt mixtures with reduced production temperatures, i.e., WMA technology mixtures, positively impacts the entire road construction cycle. It extends the road construction season into late autumn by reducing the temperature gradient between the mixture and the surrounding temperature. This results in longer cooling times and a longer compaction cycle for the structural layer of asphalt pavements. To reduce the technological temperatures of asphalt mixtures, low-volume modifiers are used, including Fisher-Tropsch synthetic wax (SW) [17-23], various chemical additives [24-28], and surfactants [29-30]. Other technologies include dosing zeolite into the asphalt mixture and foaming bitumen with water [31-32] and zeolite [33-34], which was previously used for cold recycling of lower pavement construction layers [35-39].

First, low-viscosity additives were individually dosed into the bitumen [40]. Preliminary studies showed that using two additives, synthetic wax and talc oil aminopolyamine, achieves a favorable effect in limiting the aging of bitumen [41-42]. These studies were performed on ordinary bitumen. It is also important to determine the effect of these additives on polymer modified bitumen and demonstrate the possibility of modifying it and reducing the technological temperatures for producing a mineral asphalt mixture.

2. Materials and Research Methodology

2.1. Tested Materials

The study used PMB 45/80-65 bitumen, which is commonly used in Central and Eastern European countries to produce mineral asphalt mixtures for wearing and binder layers in road pavement structures. Table 1 shows this binder's basic values.

Tab. 1. Basic properties of PMB 45/80-65 polymer modified asphalt binders.

Property	Test Method	Unit of Measure	Result
Penetration at 25°C	EN 1426	0.1 mm	45.3
Softening point $T_{R\&B}$	EN 1427	°C	74.7
Fraass breaking point	EN 12593	°C	-17.8

Synthetic wax is used as an additive in bitumen, which occurs naturally in solid form as very fine granules.

Synthetic wax significantly reduces the viscosity of asphalt. It also ensures a high level of adhesion between the bitumen and the aggregate, guaranteeing the durability of the asphalt pavement. The properties of Fisher-Tropsch synthetic wax (SW) are summarized in Table 2.

Tab. 2. Characteristics of the Fisher-Tropsch synthetic wax (SW) [43]

Property	Unit of Measure	Value
Appearance	-	solid pellets, white or yellowish
Flash point	ºC	285
Solidification point	ºC	95
Density at 25 °C	Mg/m^3	0,9
Molecular weight	g/mol	ca. 1000

In accordance with EN 12594, the samples were prepared for testing by heating PMB 45/80-65 to a temperature 90°C higher than its softening temperature. This was followed by the separation of 1,200 g samples of PMB 45/80-65 for each amount of SW that was dosed. This volume was sufficient to perform all tests on the bitumen binder with a given amount of SW before and after RTFOT ageing. The samples were then returned to the dryer and the set temperature was maintained for 30 minutes. Next, the PMB 45/80-65 bitumen was mixed with an appropriate amount of SW to increase homogeneity using a variable-speed electric mixer. This process lasted for three minutes at a constant temperature of 300 rpm. The resulting asphalt binder samples with SW were then assessed for quality in accordance with EN 12594.

2.2. Experimental program

PMB 45/80-65 bitumen was modified with SW, added in 0.5% increments from 1.5% to 3.5%.

The study investigated the effect of SW on the following properties of the binder:

- penetration in 25ºC (Pen, EN 1426:2015-08), which is a measure of the consistency of the binder,
- softening point ($T_{R\&B}$, EN 1427:2015-08),
- Fraassa breaking point (T_{Fraass}, EN 12593).

The rheological properties of the bitumens were also determined. These properties include:

- complex viscosity η*,
- zero shear viscosity η_0,
- dynamic modulus |G*|,
- phase shift angle δ,
- irreversible creep susceptibility J_{nr},
- elastic recovery R.

All the parameters tested were determined in two stages:

- before ageing:
- after short-term ageing by RTFOT (Rolling Thin Film Oven Test) in accordance with EN 12607-1.

The Christensen Anderson Marasteanu (CAM) model [44-45] described by equation (3) was used to describe the behavior of asphalt binders with SW added during the aging process.

The CAM model is an attempt to more accurately describe the stiffness of both unmodified and modified bitumen by the following relationship [44-46] (1):

$$|G^*| = G_g \cdot \left[1 + \left(\frac{f_c}{f}\right)^v\right]^{-\frac{w}{v}} \quad (1)$$

where: G^* - complex shear, G_g - glassy modulus, f_c – the frequency at the cross point (Hz), f – measurement frequency, *w, v* – experimental curve fitting parameter pf the CAM model.

The CAM model defines the rheological index R as (2):

$$R = \frac{log2}{k} \quad (2)$$

The test results obtained were subjected to a static analysis to determine their reliability and establish the significance of the effect of the SW on the properties of the 50/70 bitumen, using an analysis of variance (ANOVA) [47].

In order to ensure the reliability of the results obtained for the properties of PMB 45/80-65 bitumen modified by the addition of SW, each parameter was determined using 9 samples [48].

The study results were analysed using Statistica software [47]to ensure their reliability and determine significant relationships between the tested binder parameters and the amount of SW used.

3. Results and discussion

3.1. The effects of the synthetic wax F-T content on basic bitumen properties

The first parameter analysed in terms of the effect of synthetic wax and ageing on PMB 45/80-65 bitumen was penetration at 25°C. The results are shown in Fig. 1.

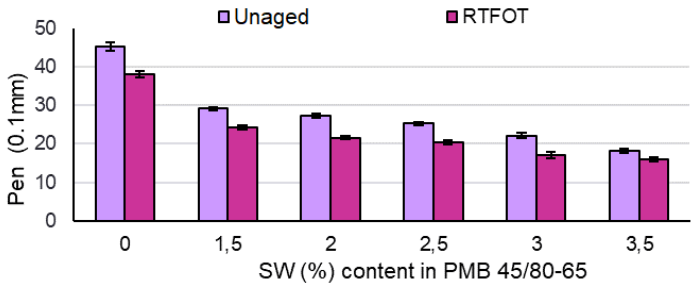

Fig. 1. Effect of synthetic wax and RTFOT ageing on the penetration of PMB 45/80-65 bitumen

Adding synthetic wax to the range of its callus lowers the penetration of PMB 45/80-65 bitumen. It hardens. The ageing of RTFOT and RTFOT+PAV affects this trend. To accurately assess the changes in the properties of PMB 45/80-65 bitumen when synthetic wax is added, and bearing in mind that the tested bitumen binder interval values had a normal distribution, the results were subjected to multivariate analysis of variance (ANOVA). Two factors were extracted for the analysis:

- the type of viscosity-reducing binder agent (synthetic wax).
- the ageing stage (ageing).

Additionally, the interaction effect between the ageing stage and the modifier type (ageing*modifier) was analysed. The results of the significance of the effect of the considered factors on the penetration level are presented in Table 3..

Tab. 3. Variance Analysis - Penetration

Factor	Univariate Significance Tests; Sigma-Restricted Parameterization Effective Hypothesis Decomposition			
	Pen (0.1·mm) SS*	**Pen (0.1·mm) MS_{Error}****	**Pen (0.1·mm) F*****	**Pen (0.1·mm) p-value********
Intercept	150951,7	150951,7	4836,884	0,00
SW	9703,9	9703,9	310,938	0,00
Agging	1880,9	940,5	30,135	0,00
Agging*SW	92,7	46,4	1,485	0,23
Error	4306,8	31,2		

* SS - Sum of Squares, **MS_{Error} - Mean Squared Error, ***F - MSE_{efect}/MS_{error};

Based on the results of the analysis of variance, it can be concluded that both synthetic wax and ageing stage had a significant effect on the level of variation of the penetration trait (p-value < 0.05). However, the interaction effect between ageing stage and modifier type was not statistically significant, with a confidence level above 0.05 (p-value = 0.23). Synthetic wax (MS = 9703.9) made the greatest contribution to explaining the variation in terms of mean square error (MS).

The effect of synthetic wax on the TR&B shimmer temperature of PMB 45/80-65 asphalt was then analysed (see Fig. 2).

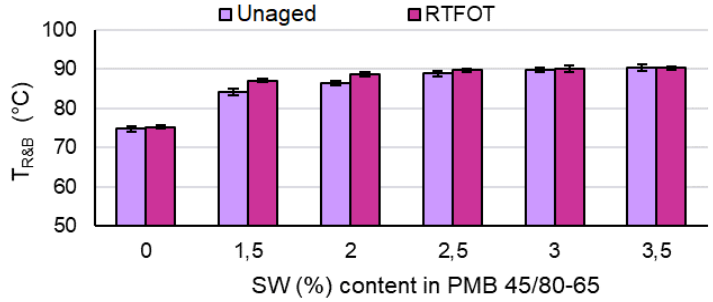

Fig. 2. Effect of synthetic wax and RTFOT ageing on the softening temperature of PMB 45/80-65 bitumen

Based on the test results, it can be concluded that using synthetic wax F-T significantly increases the softening temperature by around 15°C at a content of 3.5%, compared to the reference asphalt. The ageing process did not significantly impact the softening temperature of asphalts modified with synthetic wax.

The results of the assessment of the significance of the effects of SW and RTFOT ageing on the softening point ($T_{R\&B}$) are shown in Table 4.

Tab. 4. Variance Analysis - Softening point

Factor	Univariate Significance Tests; Sigma-Restricted Parameterization Effective Hypothesis Decomposition			
	$T_{R\&B}$ (°C) SS	**$T_{R\&B}$ (°C) MS**	**$T_{R\&B}$ (°C) F**	**T_{PiK} (°C) p - value**
Intercept	935766,0	935766,0	63777,19	0,00
SW	4760,3	4760,3	324,44	0,00
Agging	42,0	21,0	1,43	0,24
Agging*SW	18,4	9,2	0,63	0,54
Error	2024,8	14,7		

For this parameter, SW has a significant effect on the change in softening temperature (p-value <0.05). The other factors, namely ageing stage and the interaction between ageing stage and SW, are not statistically significant. Synthetic wax made the greatest contribution to explaining the variation with respect to the mean square error (MS), as with penetration (MS = 4760.3).

The next basic parameter considered was fracture temperature according to Fraass. According to EN 12593, this is the lowest temperature at which a bitumen layer, when applied to a steel plate and cooled at a constant rate of 1°C/min, will scratch or crack when bent. This parameter indicates the approximate transition temperature of the bitumen to a state corresponding to the brittle-elastic model and suggests the brittleness of the binder at low temperatures.

The results of determining the fracture temperature of synthetic wax-modified asphalts before and after short- and long-term ageing are shown in Fig. 3.

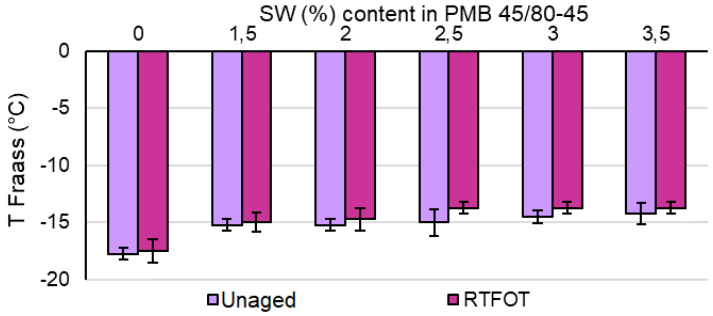

Fig. 3. The dependence of the fracture temperature of PMB 45/80-65 bitumen on the amount of SW synthetic wax, before and after the RTFOT process

As shown in Fig. 6.6, the fracture temperature results indicate that the fracture temperature value increases with the amount of SW additive. At a 3.5% share in the bitumen binder, the decrease in fracture temperature is approximately 3.5°C. Technological ageing increases the fracture temperature, thereby contributing to the susceptibility of pavements to low-temperature cracking. However, this increase is small, with PMB 45/80-65 bitumen modified with viscosity-reducing agents exhibiting an increased fracture temperature of up to 1.5°C compared to the unaged binder after long-term ageing. The results of the multivariate analysis of variance for the fracture temperature attribute (T_{Fraass}) are presented in Table 5.

Tab. 5. Variance Analysis - Fraassa breaking point

Factor	Univariate Significance Tests; Sigma-Restricted Parameterization Effective Hypothesis Decomposition			
	T_{Fraass} [ºC] SS	T_{Fraass} [ºC] MS	T_{Fraass} [ºC] F	T_{Fraass} [ºC] p-wartość
Intercept	37152,56	37152,56	24624,63	0,00
SW	222,51	222,51	147,48	0,00
Agging	12,17	6,08	4,03	0,02
Agging*SW	1,56	0,78	0,52	0,60
Error	208,21	1,51		

Regarding fracture temperature, the interaction effect between ageing stage and modifier type was not significant at the 0.05 confidence level (p-value = 0.59). Also, the interaction between these factors contributed the least to explaining the variation with respect to the mean square error (MS). This may be related to the large reading tolerance of ±2°C for this study.

3.2 Examining the rheological properties of the asphalt binder

All tests on the rheological properties of PMB 45/80-65 bitumen, depending on the amount of SW, were carried out using a dynamic shear rheometer (DSR). These tests were performed at temperatures of 40°C, 60°C and 80°C, within a frequency range of 0.01 to 10 Hz, in accordance with EN 14770.

Zero shear viscosity was determined using the creep method in accordance with PKN-CEN/TS 15325. This is characterised as a value extrapolated from the shear rate or for frequencies approaching zero. Therefore, the zero shear viscosity value of SW modified PMB 45/80-65 bitumen was assigned the low shear viscosity (LSV) readout. The frequency at which the plateau level (η_0) was determined was set to 0.005 Hz (i.e. a shear rate of 0.0032 s^{-1}). The viscosity value, η_o, was obtained using a modified Cross model, referred to as the Cross/Sybilski model [49]. Its form was determined by equation (3):

$$\eta^* = \frac{\eta_o}{1+K\omega^m} \tag{3}$$

gdzie:
$K \in (0;1)$, $m \in (0;70)$- liquid-specific experimental parameters,
η_o - zero shear viscosity.

Figure 4 shows the results of the viscosity tests (η_0) at 60°C, both before and after the RTFOT process, for 45/80-65 bitumen with SW synthetic wax.

The critical value, of non-aged bitumen, for the parameter η_0 is the 1000 Pas level indicated by the dashed line. Below this value, the occurrence of the third stage of the rutting process is very often observed in the mineral-asphalt mixture. The results of the test results show that all variants of the SW modification of PMB 45/80-65 bitumen before and after RTFOT) have a viscosity value η_0 > 1000 Pas. Thus, greater resistance to the rutting process of the mineral asphalt mixture will be ensured.

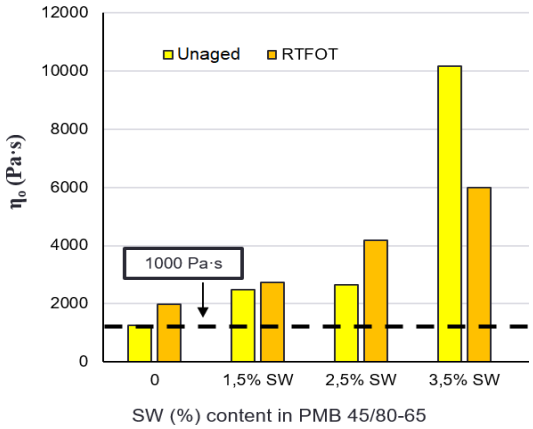

Fig. 4. The zero-shear viscosity (η_0) at 60 °C before and after the RTFOT ageing of PMB 45/80-65 asphalt with SW

On the other hand, the influence of the ageing process on the properties of PMB 45/80-65 bitumen with SW addition can be represented by Black curves. They characterise the dependence of the dynamic modulus |G*| on the phase shift angle δ. They also show the rheological character of the binder and allow a graphical comparison of the different variants of asphalt modification. They also allow inferences to be made about the viscoelasticity of PMB 45/80-65 with SW addition.

The Black curves are based on the results of dynamic binder tests carried out at 40°C, 60°C and 80°C in the frequency range from 0.01 Hz to 10 Hz.

Graphs of the Black curves of PMB 45/80-65 bitumen with synthetic wax additive before and after RTFOT ageing are shown in Figure 5.

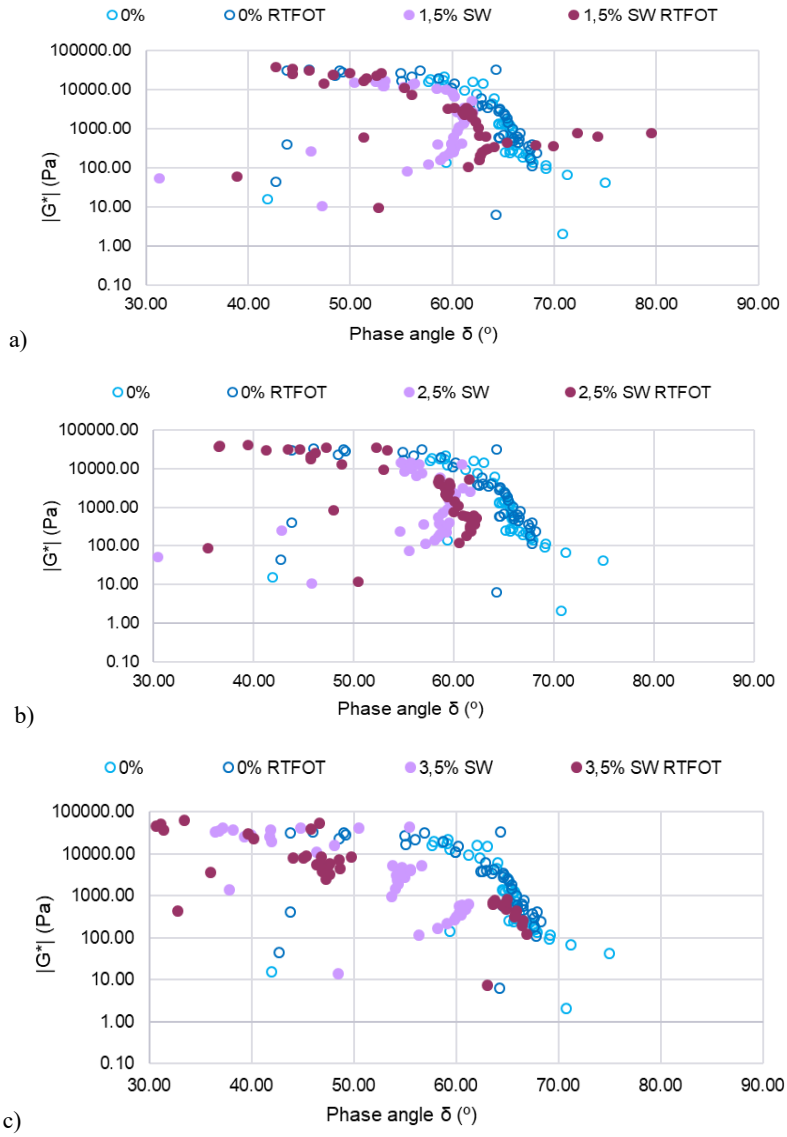

Fig. 5. Black curves show the PMB 45/80-65 bitumen before and after RTFOT ageing: a) PMB+1,5%SW; b) PMB+2,5%SW; c) PMB+3,5%SW.

Analysis of the characteristics shown in Fig. 5 indicates that increasing the amount of SW in the PMB 45/80-65 binder decreases the phase shift angle δ. Consequently, the elastic component G' of the dynamic modulus becomes dominant in the binder. This demonstrates that, in unaged binders, the synthetic wax content increases the dynamic modulus value, |G*|. PMB 45/80-65 binders with SW addition exhibit a characteristic curvature, indicating favourable rheological properties. Based on this, it can also be concluded that, at high test temperatures, the level of elastic recoil will increase. The ageing process causes the phase shift angle in PMB 45/80-65 bitumen with SW to increase to a level corresponding to that of unmodified bitumen.

To analyze the MSCR (Multiple Stress Creep Recovery) characteristics showing irreversible creep susceptibility (Jnr) and elastic recovery (R), see Fig.6 and Fig. 7, the effect of the SW additive on the Jnr and R parameters of PMB 45/80-65 asphalt at 60 °C with a shear stress of 3.2 kPa is shown before and after RTFOT ageing.

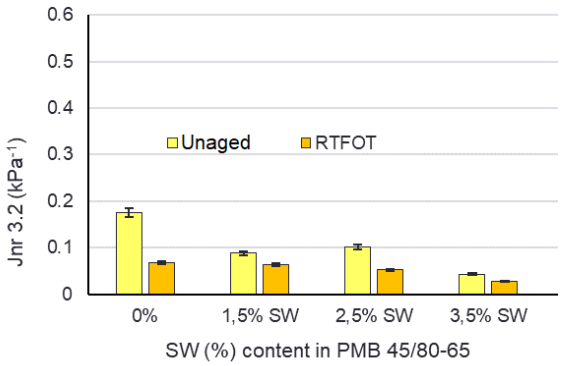

Fig. 6. J_{nr} bitumen binder with SW, before and after RTFOT ageing at 60°C, at a shear stress of 3.2 kPa

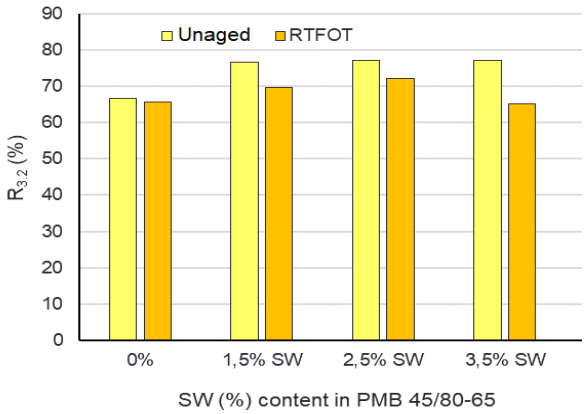

Fig. 7. The elastic recovery (R) of asphalt binders with SW, before and after RTFOT at 60°C and a shear stress of 3.2 kPa

The addition of SW to bitumen PMB 45/80-65 results in a lower Jnr3.2 susceptibility value after RTFOT ageing compared to bitumen without the addition. This is an advantageous result, as mineral-asphalt mixes produced using PMB 45/80-65 with SW will be more resistant to rutting than mixes using reference bitumen.

Figure 7 shows that the PMB 45/80-65 binders with SW obtained higher values for the tested parameter than the reference bitumen in the $R_{3.2}$ rutting tests. In all cases tested, the RTFOT ageing process reduces the elasticity of the asphalt binder. In summary, it can be concluded that adding SW to PMB 45/80-65 improves the rutting resistance of mineral-asphalt mixtures, making them more elastic at 60°C, which is considered the maximum pavement temperature.

Another analysed rheological characteristic of PMB 45/80-65 bitumen with the addition of SW was the CAM model. Figures 8, 9 and 10 present a graphical interpretation of its parameters (f_c and W) and the determined rheological index (R).

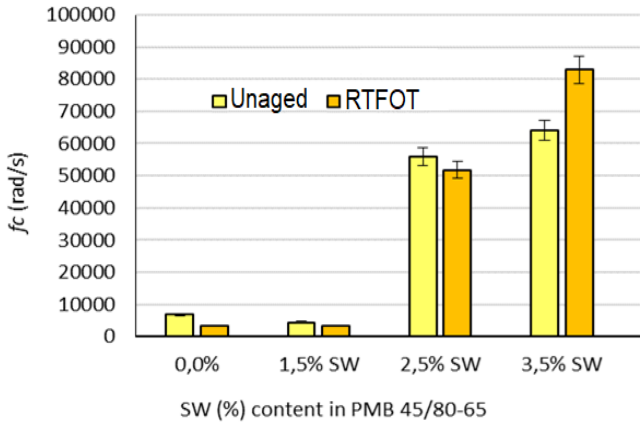

Fig. 8. "Cross-point" frequency f_c values before and after the RTFOT ageing process for PMB 45/80-65 bitumen with an SW additive

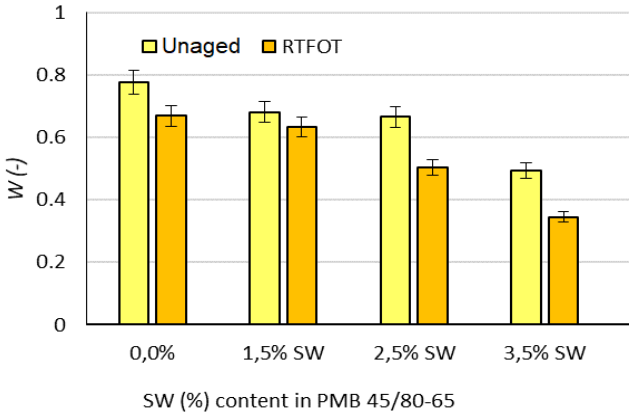

Fig. 9. The W parameter values before and after RTFOT ageing for PMB 45/80-65 bitumen with a Fisher–Tropsch synthetic wax additive

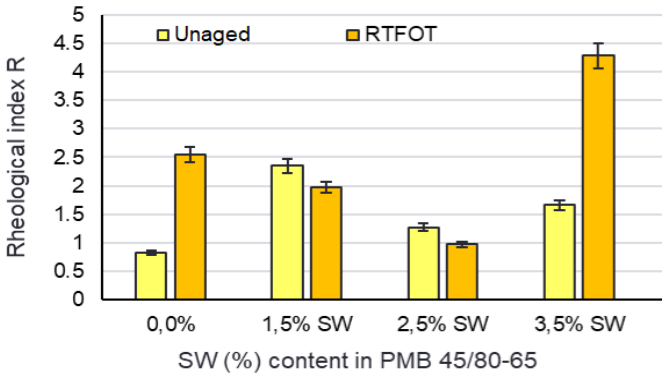

Fig. 10. R-index values for PMB 45/80-65 bitumen with SW additive before and after RTFOT ageing

Analysis of the results shown in Figures 8 and 9 indicates that, as the amount of SW additive increases, the binder stiffens and the frequency range within which stress relaxation occurs decreases.

Conversely, analysis of the rheological R-index characteristics (Figure 10) shows that such an asphalt binder becomes less sensitive to loading time.

4. Conclusion

Based on tests conducted on PMB 45/80-65 bitumen with synthetic wax added, the following conclusions can be drawn regarding the RTFOT ageing process:

1. The addition of synthetic wax significantly affects the basic and rheological characteristics of polymer modified bitumen.
2. The use of synthetic wax favourably modifies the rheological properties of PMB both before and after the RTFOT ageing process, providing the asphalt mixture with greater resistance to permanent deformation.
3. Synthetic wax reduces the effects of the RTFOT ageing process on the properties of polymer modified bitumen by slowing down the ageing of the binder.

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