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Effect of Synthetic Wax and Surface-Active Agent and Aging Process on the Rheological Properties of Polymer Modified Asphalt Binder

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Abstract. In order to ensure the required service life of road pavements, polymer modified asphalts binder are increasingly being used to produce asphalt mixtures. However, as a result of technological aging during the production of the asphalt mixture and operational aging, the parameters of the asphalt binder are reduced. In order to slow down this process, various types of low-viscosity additives are used, which include Fisher-Tropsch synthetic wax and surface-active agents. Such additives also have the effect of lowering the technological temperatures for the manufacture and incorporation of the asphalt mixture. As a result of their use, there is also a reduction in greenhouse gas emissions and energy intensity in the production of the asphalt mixture. In the study, synthetic wax F-T was used, which was dosed at 0.0%, 1.5%, 2.5% and 3.5%, as well as a surfactant at 0.0%, 0.2%, 0.4% and 0.6% into PMB 45/80-65 polymer-modified asphalt binder. The binder was then subjected to aging. An important part of the study was the determination of rheological characteristics in terms of linear and non-linear visco-elasticity, viscosity multiple creep and stress recovery tests (MSCR), oscillatory tests (DSR), and low-temperature characteristics (BBR). Based on the analysis of the test results, the effect of the additives used on the rheological properties of the polymer asphalt was found to be significant. The addition of synthetic wax F-T contributes to a significant increase in the level of stiffness (hardness) of polymer modified asphalt binder after the aging process. In terms of asphalt pavement operation at low temperatures, however, it was noted that long-term aging has a much smaller effect on the stiffness (brittleness) of polymer modified asphalt binder with 0.6% surfactant addition than with 3.5% synthetic wax content. It was also found that after the aging process, polymer asphalt with 3.5% F-T synthetic wax addition contributes to an increase in Jnr3.2 susceptibility and has a comparable value of elastic return R3.2 compared to the base asphalt before aging. On the other hand, the addition of surfactant in the amount of 0.6% causes both a decrease in the value of susceptibility Jnr3.2 and elastic recurrence R3.2. At the same time, it was found that synthetic wax decreased the susceptibility of PMB 45/80-65 at medium and high operating temperatures, and influences a decrease in its aging rate with an increase in the elastic part of the dynamic modulus. Synthetic wax increased the level of zero shear viscosity of the binder which will have a beneficial effect on reducing the deformation rate of the mineral-asphalt mixture and ensure the required service life of the pavement.

Keywords: Polymer modified asphalt binder; synthetic wax F-T; surface active agent; aging process; rheological properties

INTRODUCTION

Currently, the main direction of research work in the road industry is the development and implementation into road practice of mineral-asphalt mixtures at reduced production and embedding temperatures produced by Warm Mix Asphalt (WMA) or Half-Warm Mix Asphalt (HWMA) technology [1-6]. They should be environmentally friendly and have an energy-efficient production process and low greenhouse gas emissions and contribute to the improvement of technological processes of road construction [7]. To this end, it is indispensable to use a low-viscosity binder obtained by a process other than heating it. Two main methods are used to achieve this effect. The first is to add various types of chemical additives to the asphalt, which will provide an asphalt viscosity of 0.2 Ps a to 2.0 Ps a [8 - 10]. The additives used are also designed to provide favorable standard and rheological properties of the asphalt [11-13]. As a result, it is possible to produce mineral-asphalt mixtures whose manufacturing temperature is reduced by about 40ºC [14,15] compared to traditional technology characterized by a temperature range of 160ºC to 180ºC. Also, the temperature at which the asphalt mix is built into the pavement construction layer is lower than the standard temperature. Reducing the temperature of asphalt mixture production also contributes favorably to extending the roadwork season as a result of reducing the temperature gradient between the temperature of the mixture and the temperature of the environment. Reducing the need to heat the mineral-asphalt mixture in the high-temperature range contributes to a reduction in the cost of its production as a result of 15 to 20% associated with energy savings. Also very important is the effect of improving working conditions by reducing the impact of high temperatures on workers directly working in the mineral-asphalt mix plant and during road construction.

The second method of lowering the viscosity of asphalt is by foaming it with water [16 - 18] or by adding zeolite [19 - 20]. At the same time, using water to foam the asphalt more significantly reduces the technological temperature of the asphalt mixture [21]. It should be noted, initially this technology was used only during deep recycling on the cold pavement construction [22, 23, 24]. Over the course of two decades, a wide range of research has been done on obtaining foamed asphalt characterized by high foaming parameters as a result of the use of various additives, such as F-T synthetic wax (SW) [25], chemical additives or surfactants (SAA) [26, 27]. The use of foamed asphalt with additives makes it possible to obtain a recycled mineral-asphalt mixture with high mechanical parameters [28] and the use of waste materials such as mineral dust in its composition [29]. Nowadays it is possible to use water-foamed asphalt also in Half Warm Mix Asphalt [30].

A significant impact on the properties of asphalt and the mineral-asphalt mixture produced with it is caught up with the aging process of the binder, as a result of which asphalt loses its visco-elastic properties and becomes brittle. Two stages can be distinguished in the aging process of asphalt, technological aging - short-term aging [31, 32] and operational aging - long-term aging [33, 34]. The use of various types of additives of natural or artificial origin in WMA or HWMA reduced-temperature asphalt mixture production technology has a beneficial effect on reducing the aging of the binder [35 - 39]. It should be noted that virtually only one type of additive was used to modify the asphalt.

Until recently, only additives were practically used for non-polymer-modified asphalt, which is intended for making mineral-asphalt mixtures for pavements loaded with less than heavy traffic. Attention is now being paid to the need to ensure the durability of pavements loaded with heavy vehicle traffic over the long term. For this type of pavement, polymer-modified asphalts are used as a binder for mineral-asphalt mixtures. Accordingly, research is being undertaken on the feasibility of using polymer-modified asphalts in WMA and HWMA.

Therefore, it is necessary to determine the effect of SW and SAA on the properties of polymer-modified asphalt in the context of its aging process. It is particularly important to determine its rheological properties, which determine the long-term performance of asphalt pavements. Preliminary studies have shown that the effect of synthetic wax F-T and SAA on the standard parameters of polymer-modified asphalt is very good [40].

MATERIALS AND METHOD

Tested Materials

The study used polymer-modified asphalt binder, which is widely used in Central Europe for the production of asphalt mixtures intended for the structural layers of road surfaces with high traffic volumes (Table 1).

Table 1. Basic properties of PMB 45/80-65 polymer modified asphalt [41].

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
Dynamic viscosity at 135°C	EN 12702-2	Pa·s	0.649

The SW and surface active agent were used as asphalt additives (Table 2). The SW and SAA significantly reduce the viscosity of the asphalt and ensure at a high level the adhesion of the binder to the mineral aggregate, guaranteeing adequate durability of the asphalt pavement.

Table 2. Basic properties of the SW and SAA [42, 43].

Property	Unit	Additive SW_{LC}	Additive SAA
Appearance	-	Solid pellets, white or yellowish	Viscous liquid, brown
Flash point	°C	285	>218
Solidification point	°C	95	
Density at 25 °C	Mg/m^3	0.9	
Density at 20 °C			0.88-0.98
Viscosity at 135 °C	Pa·s	12	
Molecular weight	g/mol	ca. 1000	

Experimental program

Polymer-modified asphalt binder was subjected to SW and SAA modification, which were added in 1.5% to 3.5% increments of 0.5% and 0.2% to 0.6% increments of 0.2%, respectively.

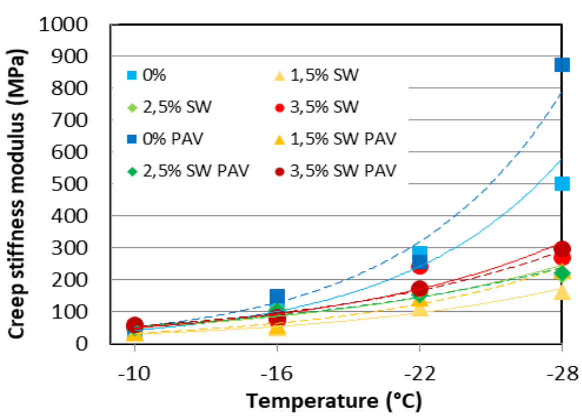
The scope of the study of the effect of SW and SAA dosed into the binder was to examine the following properties of the binder: S and m-value parameter in the bent beam rheometer – BBR (EN 14771), composite viscosity η*, zero shear viscosity η0, dynamic modulus |G*|, phase shift angle δ, elastic recurrence R and irreversible creep susceptibility Jnr.

All tested parameters were determined before and after ageing using the RTFOT (Rolling Thin Film Oven Test) method in accordance with the PN-EN 12607-1 standard and the RTFOT+PAV (Pressure Aging Veesel) method in accordance with the PN-EN 14769 standard. The results obtained on 9 samples were subjected to static analysis in order to determine the significance of the effect of SW and SAA on the properties of PMB 45/80-65 polymer modified asphalt using analysis of variance (ANOVA) [44, 45].

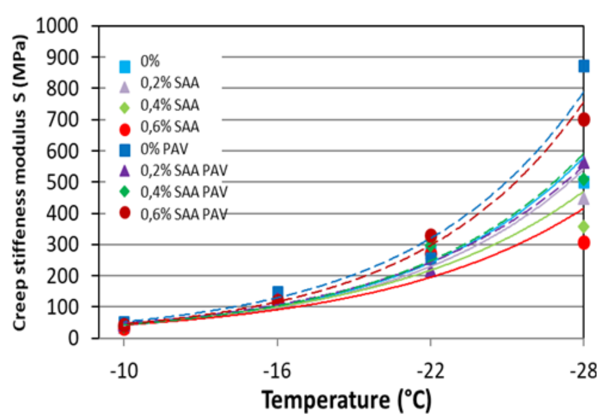
RESULTS AND DISCUSIONS

Creep Stiffness of Polymer Modified Asphalt Binder with SW and SAA

Figure 1 and Figure 2 present the results of testing the rheological properties at low operating temperatures [39] of polymer-modified asphalt binder with SW at 1.5%, 2.5%, and 3.5% and SAA at 0.2%, 0.4%, and 0.6% before and after long-term aging (RTFOT+PAV).



a)

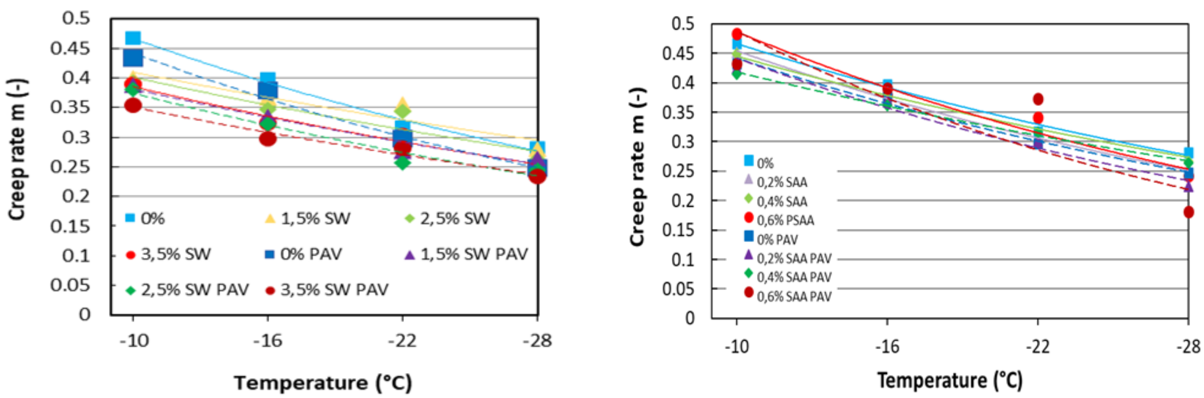


b)

FIGURE 1. Values of stiffness modulus S of asphalt binder before and after long-term aging with: a) SW; b) SAA

Based on the characteristics presented in Figure 1, it can be concluded that lowering the test temperature causes an increase in the stiffness modulus S in each of the tested modified asphalt binders, regardless of the viscosity reducing agent used. The presence of SW in the reference polymer asphalt, regardless of the test temperature, caused a significant decrease in the stiffness modulus S. The greatest decrease was observed at 1.5% in the PMB 45/80-65 reference asphalt. Long-term aging contributes to the continuation of this trend. It should be noted that the greatest increase in the stiffness modulus S after RTFOT+PAV aging was observed for the base asphalt. As the SW concentration in the modified asphalt binder increases, smaller changes in the S parameter value are observed during aging.

The addition of surface-active agents contributed to a decrease in the modulus of stiffness S in all dosage and temperature variants. This is beneficial, since too much stiffness of asphalt at low temperature affects the formation of cracks. Long-term aging of PAV resulted in an increase in S-modulus values in all asphalt binders with SAA (the largest for PMB 45/80-65 asphalt with 0.6% SAA added).



a) b)

FIGURE 2. Values of parameter m of modified asphalts binder before and after long-term aging with: a) SW, b) SAA

The stiffness change characteristics (m parameter) presented in Figure 2 allow us to conclude that as the test temperature decreases, the rate of change in the stiffness of the modified asphalt during loading decreases. The presence of SW in the reference asphalt contributed to the lowering of the m parameter characteristics. Increasing the SW concentration in the asphalt binder resulted in a lower value of the m parameter. The addition of SAA to polymer-modified asphalt caused a slight decrease in the value of the m parameter compared to the asphalt without the additive. After operational aging, the values of the m parameter in all tested asphalt binders with the addition of SAA also decreased slightly. This value is within the measurement accuracy limits of the device.

Influence of Aging on Zero Sher Viscosity of Polymer Asphalt Binder with SW and SAA

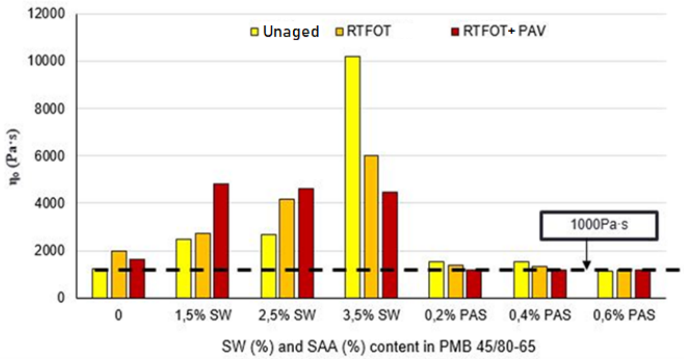
A temperature of 60°C was adopted as the reference temperature characterizing the pressure of the asphalt pavements. The zero shear viscosity was determined by the creep method in accordance with the PKN-CEN/TS 15325 standard and the study [46, 47]. The zero shear viscosity was determined according to equation (1):

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

where:
K ϵ (0;1), m ϵ (0;70)- experimental parameters specific to the fluid,
η_o - zero shear viscosity.

Figure 3 presents the results of viscosity tests η_0 at 60°C before and after the aging processes for polymer-modified asphalt with SW and SAA.

Increasing the amount of SW to polymer-modified asphalt results in an increase in the value of the η0 parameter both before and after RTFOT and RTFOT + PAV processes. In contrast, at 3.5% SW content, there is a subsequent decrease in the viscosity η0 after aging.

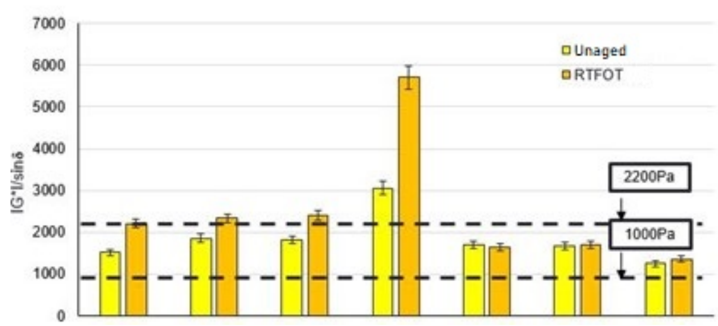


FIGRUE 3. Zero shear viscosity η_0 at 60°C before and after RTFOT and RTFOT+PAV of polymer modified asphalt with SW and SAA additives

Regarding PMB 45/80-65 with the addition of SAA, the change in the value of parameter η_0 is different - the increase in the amount of modifier reduces the value of zero shear viscosity. This trend takes place before as well as after the aging process. Most likely, this is related to the fact that the asphalt binder is plasticized by the presence of hydrocarbon chain], which cause slippage at the asphaltene micelle interface. It should also be noted that despite the aging process, the value of zero shear viscosity η_0 is still at a similar level to that of the reference binder with the low-shear modifier SAA before the aging process. This indicates that the aging process causes little change in the SW and SAA modified asphalt.

Effect of the Aging Process on Polymer Asphalt Binders with the Addition of SW and SAA in Terms of the Parameter |G*|/sinδ

Using a DSR dynamic shear rheometer in accordance with AASHTO T 315 and EN 14770, the values of dynamic modulus |G*| and phase shift angle δ were also determined.
The results of the results of determining the |G*|/sinδ parameter according to the SHRP methodology at 1.59 Hz (10 rad/s) and at 60°C for SW and SAA binders before and after RTFOT are presented in Figure 4.



FIGRUE 4. Effect of aging process and type of modifier on the results of parameter |G*|/sinδ according to SHRP methodology at 1.59Hz and at 60°C

According to Superpave's requirements, it is recommended that asphalt binders at the expected highest operating temperature in asphalt pavement be characterized by values of the parameter |G*|/sinδ >1000 Pa for unaged asphalt and |G*|/sinδ >2200Pa for asphalt after technological aging – RTFOT.

The results of the analysis indicate that all variants of polymer asphalt modification before the aging process met the requirements defined by the rutting parameter |G*|/sinδ, relative to the criteria marked on Fihure 4 with a dashed line (>1000Pa). With regard to SW-modified asphalts, the effect of its presence resulted in a slightly greater increase in the parameter |G*|/sinδ than in the case of the determinations of binders with SAA. After the RTFOT aging process, only asphalts modified with SW polymer (|G*|/sinδ >2200Pa) met the requirements according to Superpave. In their case, the effect of the increase in the parameter |G*|/sinδ compared to the state before the aging process is clear. On the other hand, polymer asphalt binders with SAA exhibit a comparable value of the tested parameter as before the aging process and do not meet the requirements of Superpave (|G*|/sinδ <2200Pa). This may indicate that the asphalt mixture layer made with SAA agent is likely to be characterized by the premature appearance of viscoplastic deformation.

Black's Curves for the Viscoelastic Properties of Binders with the Addition of SW and SAA

The effect of the aging process on the properties of polymer-modified asphalt with various modifiers in WMA technology is presented using Black curves. They show the dependence of the |G*| on the δ of the binder. They also demonstrate the rheological character of polymer-modified asphalt and enable graphical comparison of different variants of asphalt binder modification. Furthermore, this type of analysis allows for inferences about the viscoelasticity of polymer asphalt modified with temperature-reducing inhibitors.

Plots of the Black curves for polymer-modified asphalt with SW and SAA additives before and after aging are presented in Figure 5.

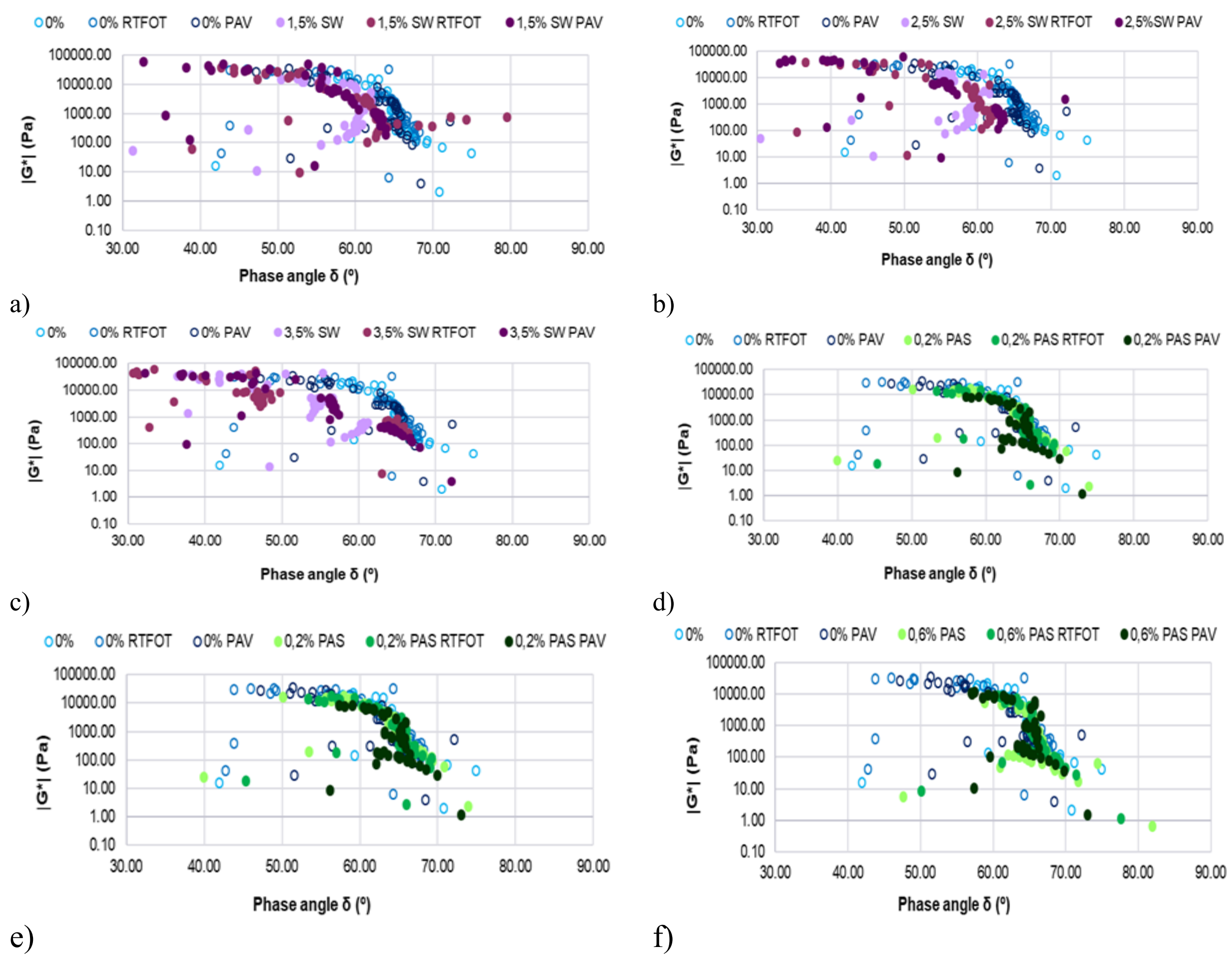


FIGURE 5. Black Curves for polymer asphalt before and after aging process: a) 1.5%SW in PMB; b) 2.5%SW in PMB; c) 3.5%SW in PMB; d) 0.2%SAA in PMB; e) 0.4%SAA in PMB; f) 0.6%SAA in PMB.

Based on the analysis of the test results presented in Figure 5a, Figure 5b and Figure 5c, it can be concluded that as the SW concentration in the reference binder increases, the phase shift angle δ decreases. Therefore, in asphalt, the elastic part G' of the dynamic modulus dominates. However, in unaged binders, the amount of SW and the polymer content will contribute to an increase in the dynamic modulus |G*|. Based on the test results, it can be concluded that the difference in the phase shift angle between the reference asphalt and the aged RTFOT+PAV asphalt with 3.5% SW is approximately 5° (Figure 5c). It should be noted that the results of determining the dynamic modulus |G*| polymer-modified asphalt + 3.5% SW, after aging RTFOT+PAV, are arranged along a vertical line.

Different results were found for polymer modified asphalt binders with SAA (Figure7.4d),e),f)). During tests with PMB 45/80-65 modified asphalt with SAA, it was observed that its increase does not cause a significant change in the phase shift angle δ. After the aging process, the SAA-modified PMB 45/80-65 polymer asphalt has similar properties to the reference asphalt. Compared to the asphalt binder not subjected to SAA modification, the course of the Black curve of PMB 45/80-65 polymer modified asphalt with SAA modifier coincides with the results of the initial asphalt after aging. However, it was also noted that as the amount of SAA additive in the polymer-modified asphalt increases, there is a decrease in the dynamic modulus |G*| compared to the reference asphalt. Therefore, it should be concluded that the mineral-asphalt mixture made with SAA-modified PMB 45/80-65 polymer asphalt will be more susceptible to permanent deformation. This is not favorable from the point of view of using it for asphalt pavement working at high temperatures (in summer).

Influence of Aging Binder with SW and SAA Additive on Cyclic Creep and Recovery

The multiple stress creep test (MSCR) was conducted at a temperature of 60°C (the expected highest operating temperature of the asphalt pavement) and a shear stress of 3.2 kPa, representing the nonlinear viscoelasticity (N-LVE) behavior, on a properly configured dynamic shear rheometer (DSR), in accordance with AASHTO TP 70 and EN 16659.

In Figure 6 and Figure 7, which show the dependence of R3.2 (%) on Jnr3.2 (kPa-1), a red dashed line is marked to distinguish modified from unmodified asphalts binder. This line was developed based on experimental studies and is determined using the function y = 29.371*(Jnr 3.2) - 0.2633 [47].

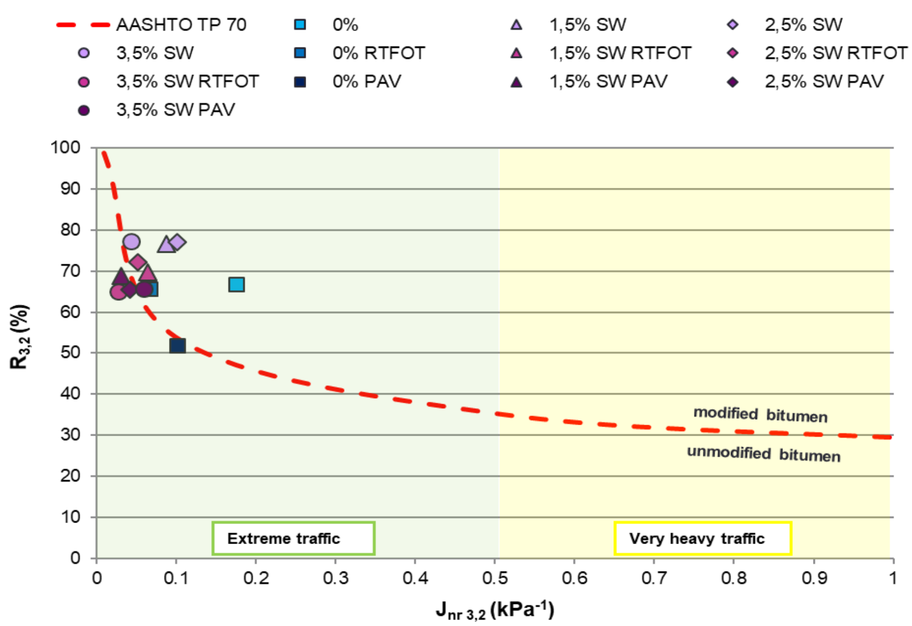


FIGURE 6. Results of tests of polymer-modified asphalt with SW for R as a function of Jnr at 3.2 kPa load at 60°C before and after aging

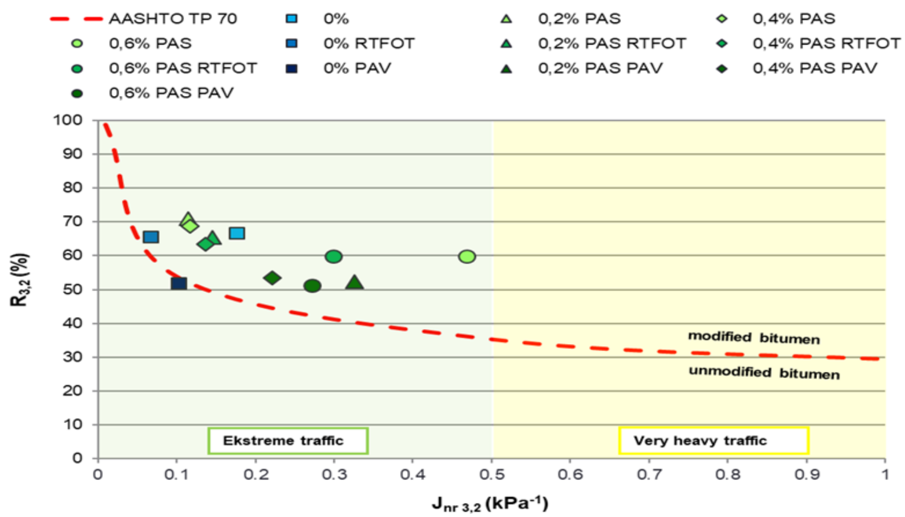


FIGURE 7. Results of tests of polymer-modified asphalt with SAA for R as a function of Jnr at 3.2 kPa load at 60°C before and after aging

Based on the characteristics shown in Figures 6 and 7 regarding the dependence of R3.2 on Jnr3.2, we can see a clear difference between the test results obtained for polymer-modified asphalt with SW and SAA . The addition of SW to polymer-modified asphalt increased the recovery coefficient $R_{3.2}$ compared to the reference asphalt, while simultaneously reducing the compliance of $J_{nr3.2}$. Polymer-modified asphalt with SW additives is characterized by greater elasticity and stiffness.

In the case of PMB 45/80-65 with SAA additive, it was found that it had $R_{3.2}$ recurrence values similar to the base asphalt after the operational aging process. At the same time, an increase in the $J_{nr3.2}$ susceptibility of polymer modified asphalt with SAA after operational aging was also noted regard s of its amount in the base asphalt. This means that the asphalt binder becomes a slightly less elastic material, but clearly more susceptible compared to the reference asphalt. This indicates that asphalt pavements made with such a binder are likely to be more susceptible to the formation of ruts in the pavement.

To verify the degree of modification of the tested binders, Figure 6 and Figure 7 also show the curve marked with a red dashed line. Despite the fact that asphalt binders with SW and SAA additives have different viscoelastic characteristics, almost all tested variants of modification of polymer-modified asphalt meet Superpave's requirements for modification quality.

CONCLUSION

Based on the performed studies of the effect of SW and SAA on the rheological properties of polymer-modified asphalt in terms of short-term and long-term aging, it can be concluded that the SW contributes to a significant increase in the level of stiffness (hardness) of the asphalt binder after the aging process. In terms of asphalt pavement operation at low temperatures, it was found that operational aging has a much smaller effect on the stiffness (brittleness) of polymer modified asphalt with 0.6% SAA addition than with 3.5% SW content.

It was also found that after the aging process, the polymer asphalt with 3.5% SW additive contributes to an increase in $J_{nr3.2}$ susceptibility and has a comparable value of elastic return $R_{3.2}$ compared to the reference asphalt before aging. Meanwhile, the addition of SAA in the amount of 0.6% causes both a decrease in the value of susceptibility $J_{nr3.2}$ and elastic recurrence $R_{3.2}$. This means that asphalt pavements made with SW will be much more resistant to rutting than those formulated with SAA. In contrast, the use of SAA can provide improvements in the resistance of asphalt pavement to low-temperature cracking.

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