The Properties of Asphalt Binder Modified with WMA Additives

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Abstract

Asphalt binder is very important in ensuring the durability of asphalt surfaces. Unfortunately, as an organic material, it is subject to the ageing process. During its transport from petrochemical plants and the production of asphalt mixture, it is exposed to high temperatures. The process of asphalt oxidation takes place, as a result of which its viscoelastic properties are reduced. This stage of ageing is called technological ageing (short-term ageing). However, during the operation of the asphalt surface, it is subject to the action of atmospheric factors such as wind, water and solar radiation. Operational ageing takes place (long-term ageing), the asphalt binder becomes more rigid. As a result of asphalt binder ageing, the operational durability of the road surface is reduced. In order to limit this negative process, various types of ageing inhibitors are used for asphalt binders, which can be chemical and mineral materials or natural and synthetic oils. In the tests, two WMA (Warm Mix Asphalt) additives were used in order to limit the ageing of asphalt binder - synthetic wax and tall oil amidopolamine, which were added to the asphalt binder at the same time. Synthetic wax was used in the amount of 1.0% w/w, 1.5% w/w, 2.0% w/w and 2.5% w/w and tall oil amidopolamine in the amount of 0.2% w/w, 0.4% w/w and 0.6% w/w in relation to the amount of asphalt binder. The influence of technological and operational ageing on the change of physic-mechanical and rheological properties of the bitumen composition with both WMA additives. Based on the analysis of the obtained research results, the optimum content of WMA additives in bitumen was determined and the effect of their synergy was demonstrated, which ensures the limitation of the influence of short-term and long-term ageing on the bitumen properties. Therefore, the required durability of the asphalt surface will be ensured in the long term of operation.

Keywords: *bitumen, WMA additives, RTFOT, PAV*

1. Introduction

As an organic material, bitumen ages as a result of many external factors. During production and transport from the petrochemical plant to the asphalt mix plant, as well as during the manufacturing of the mineral asphalt mixture, bitumen is exposed to high temperatures of about 180°C and oxygen. Consequently, oxidation processes of the binder components occur due to the oxygen in the atmosphere [1, 2]. This reduces the viscoelastic properties of the bitumen. It becomes more rigid. This stage of aging is called technological aging, or short-term aging. Conversely, during pavement operation, the asphalt in the mineral asphalt mixture is affected by climatic factors such as wind, water, and solar radiation. These factors cause further aging of the bitumen, called operational aging, or long-term aging. As a result of aging, the properties of asphalt decrease, reducing the service life of the pavement and causing cracks to appear. Recently, much research has been conducted on using various substances to slow the aging process. The effectiveness of these substances varies greatly. Currently, the aging process of bitumen is reduced by adding various organic and inorganic chemical compounds called aging inhibitors to the binder [3-6]. These compounds can be natural or synthetic [7-10] and include methyl esters of vegetable or animal fatty acids [11-15], cellulose, and lignin [16, 17]. One of the most effective anti-aging organic compounds is the SBS copolymer [18-20], which is often used as an asphalt modifier to improve its standard and rheological properties. Research on developing an additive that meets asphalt's high anti-aging requirements is ongoing. However, special attention should be paid to ensure that the anti-aging asphalt additives also meet the requirements for lowering the technological temperatures of bitumen and the resulting asphalt mixture.

This is because, in addition to ensuring high-quality asphalt and preventing aging, the goal is to reduce the energy intensity of technological processes in the road construction industry. This primarily includes producing a mineral-asphalt mixture, which has an average temperature ranging from 165 to 180°C, depending on the type of asphalt used. This issue is significant because over 90% of the world's road surfaces are constructed using bitumen technology. Additionally, producing high-temperature asphalt mixtures emits greenhouse gases, especially CO_2, which play an important role in climate warming [21, 22]. Therefore, research is being carried out on new asphalt technologies in which the bitumen mix is produced at about 135°C. This technology is referred to as warm mix asphalt (WMA) and is characterized by a reduced temperature of about 35-40°C compared to traditional technology. To produce WMA, various types of bitumen additives [23-25] or foaming with water [26] are used. These solutions should ensure the quality of both the binder and the mineral asphalt mixture produced with it. Commonly used additives include SBS copolymer [27–31], synthetic wax [32–38], various chemical compounds [39–48], vegetable or synthetic oils [49–54], and rubber [55, 56].

The research concerned the possibility of using WMA additives to improve the binder's physical, mechanical, and rheological properties, but the additives did not always slow down or reduce the binder's aging process. The studies generally involved using only one WMA additive with bitumen [45, 50, 56].

Based on preliminary studies [57-59], scientific procedures were undertaken to investigate the possibility of reducing asphalt aging due to the use of synthetic wax and thallium oil amidopolamine. This would guarantee the long-term durability of the pavement, as well as lower the temperature required for manufacturing and incorporating the mineral asphalt mixture.

2. Materials and methods

2.1. Materials

The study used 50/70 bitumen from Orlen Asfalt SA in Płock, which is the basic bitumen used for asphalt mixtures intended for the upper layers of the pavement structure in Poland and in the countries of Central and Eastern Europe. Mixtures of this type are intended for the construction of structural layers of road pavements under 2.5×10^6 to $7{,}3 \times 10^6$ 100 kN equivalent standard loads.

On the other hand, WMA additives were used to modify the properties of bitumen. These included synthetic wax with a reduced carbon footprint, tall oil amidopolyamine, and other materials. The synergy of their interaction with asphalt enables them to regulate the aging of asphalt and counteract adverse changes in its physical and mechanical properties.

2.2 Synthetic wax LC

As one of the additives to the 50/70 bitumen, a new generation of F-T synthetic wax (SW_{LC}) was used, characterized by a reduced carbon footprint compared to the one used so far in road practice. It was introduced into road practice in 2023 as a material that meets the current high environmental requirements (Tab. 1).

Tab. 1. Basic properties of the SW_{LC} synthetic wax [60]

Property	Unit	Value
Appearance	-	Solid, white or yellowish
Flash point	°C	285
Solidification point	°C	95
Density at 25 °C	Mg/m^3	0.9
Viscosity at 135 °C	Pa·s	12
Molecular weight	g/mol	ca. 1000

Synthetic F-T wax differs significantly from the natural paraffin found in petroleum. It is a long-chain hydrocarbon (its hydrocarbon chains contain between 40 and 115 carbon atoms) obtained from CO and H2 in the Fischer-Tropsch synthesis process. It has a more finely crystalline structure than natural paraffin, whose hydrocarbon chains contain between 15 and 50 carbon atoms. Its molecular weight is about 40% higher than that of the natural waxes found in bitumen. The long hydrocarbon chains of synthetic F-T wax crystallise at higher temperatures and the shorter chains crystallise at lower temperatures. Their effect is similar to that of asphaltenes and depends on the reduction of their mobility with respect to the maltene fraction.

2.3. Tall oil amidopolyamines

Tall oil amidopolyamines (TOA) are insoluble in water and soluble in certain organic solvents (e.g. toluene, paraffin, trichloroethylene). They reduce the surface tension of the bitumen binder, which improves the wrapping of the aggregate grains with bitumen during the production of the asphalt mixture. They are used as a surface-active agent (SAA). The basic physical properties of TOA are summarised in Table 2. A characteristic feature of compounds from the polyamine group is the presence of at least two amine groups, shown schematically in Figure 1.

Tab. 2. Properties of the TOA [61]

Property	Unit	Value
Appearance	-	Liquid, brown
Density 20°C	Mg/m^3	0.98
Viscosity at 20°C Brookfield Spindle SC 4-18	m·Ps	538
Pour point	°C	< 20
Flash point	°C	>218

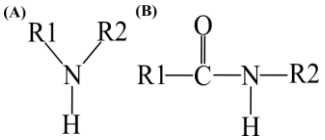
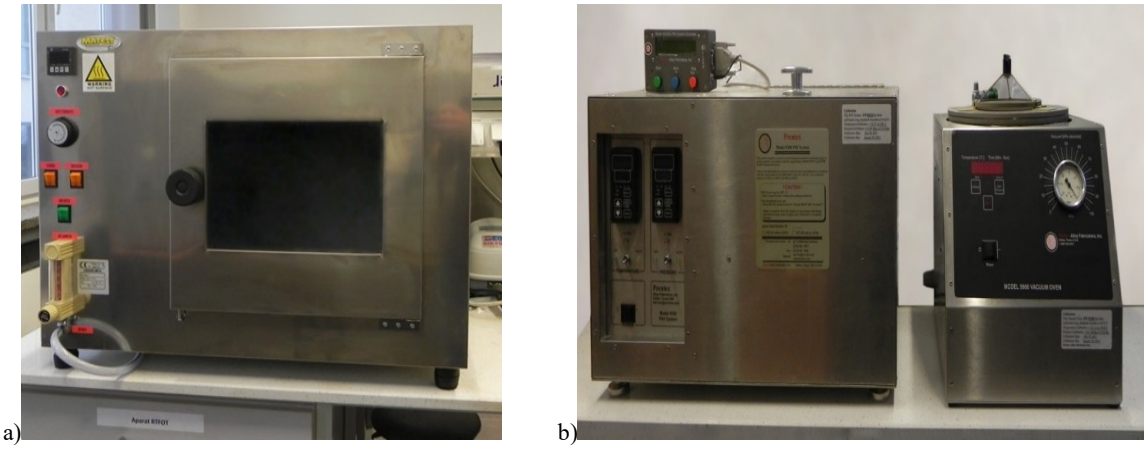


Fig. 1. Chemical structure of amine (A) and amide (B) functional group.

2.4 Ageing procedure

The aging of the asphalt binder containing synthetic wax and tall oil amidopolyamine was carried out in two stages. During the first stage, a short-term technological Rolling Thin Film Oven Test (RTFOT) was performed in accordance with EN 12607-1 requirements using an Italian MATEST S.p.A. device (Fig. 3a).

During the second stage, the binder was subjected to long-term aging in a pressure-thermal chamber model 9300 PAV System from Prentexa Alloy Fabricators Inc. z USA (Fig.3b), in accordance with the requirements of EN 14769.



a) b)

Fig. 2 Labortory equipment for bitumen aging; a) RTFOT, b) PAV (Fot. M.M. Iwański)

2.5. Design of experiment

The research plan was predicated on the assumptions of the factorial algorithm of the experimental plan [62]. The properties of 50/70 bitumen with the addition of synthetic wax SW_{LC} and tall oil amidopolyamines on TOA were determined based on a 4x4 factorial plan. The adoption of this experimental plan was due to the amount of dosed synthetic wax SW_{LC} (1.0%, 1.5%, 2.0%, and 2.5% by weight) to asphalt and tall oil amidopolymines (TOAP) at 0.0%, 0.2%, 0.4%, and 0.6% by weight to asphalt, respectively.

The following binder properties were determined as part of the testing program:

- penetration in 25°C (Pen, EN 1426:2015-08),
- softening point ($T_{R\&B}$, EN 1427:2015-08),
- breaking point (T_{Fraass}, EN 12593:2015).

Therefore, to describe the change in the value of parameter A in 50/70 bitumen in relation to the SW_{LC} and TOA content, a statistical model using a second-degree polynomial was adopted. [62]:

$$A = b_0 + b_1 \cdot x_1 + b_2 \cdot x_2 + b_3 \cdot x_1 \cdot x_2 + b_4 \cdot x_1^2 + b_5 \cdot x_2^2 \quad (1)$$

where: x_1 = synthetic wax - SW_{LC} (%), x_2 = tall oil amidopolyamines - TOA (%), b_0-b_5 – regression coefficients

Next, ANOVA analysis of variance was used to estimate the coefficients of the polynomial degree. The approximation of the parameters was based on the least squares method.

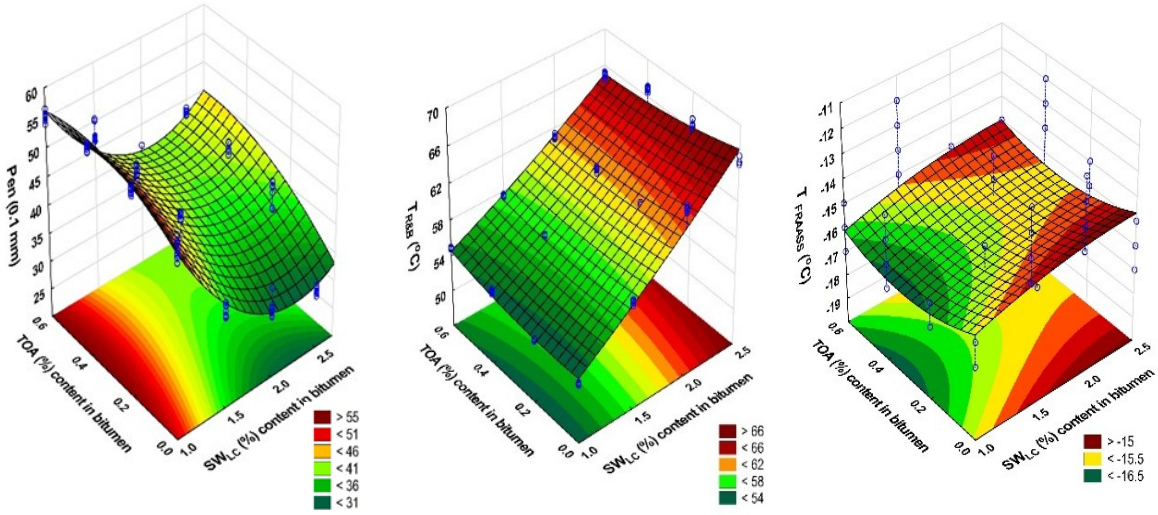
The results of the study were visualized using Statistica software [63].

We used a Pareto chart to evaluate the significance of the effect of a particular component (synthetic wax SW_{LC} or tall oil amidopolyamines, TOA) or the interaction between them on the properties of foamed bitumen. This allowed us to determine not only whether a factor was significant but also the strength of a given model parameter's influence and the trend's direction. The values on the Pareto chart corresponded to the standardized relative effect evaluation values and were presented graphically [64].

3. Results and discussion

3.1. The effect of the synthetic wax SW_{LC} and tall oil amidopolyamines *TOA amount on the bitumen physico-chemical properties*

A comprehensive analysis of the change in penetration at 25°C of 50/70 bitumen, softening temperature and Frass temperature in terms of the amount of synthetic wax WS_{LC} and tall oil amidopolyamines TOA is presented by regression models using relationship (1) in the form of response plots generated using Statistica software (Fig. 3).



(A) (B) (C)

$$Pen = 98.260 - 59.805\ SW_{LC} + 13.206\ SW_{LC}^2 + 22.082\ TOA - 36.389\ TOA^2 + 7.224\ SW_{LC}\ TOA$$

$$T_{R\&B} = 45.559 + 6.767\ SW_{LC} + 0.802\ SW_{LC}^2 + 2.694\ TOA + 6.434\ TOA^2 - 4.236\ SW_{LC}\ TOA$$
$$T\ Fraass = -17.576 + 72.344\ SW_{LC} - 0.472\ SW_{LC}^2 - 5.554\ TOA + 7.813\ TOA^2 + 0.122\ SW_{LC}\ TOA$$

Fig. 3. Penetration (A), softening point (B) and breaking point (C) of the bitumen modified with synthetic wax LC and TOA and the representation of the models describing the relationship

Analyzing the correlations shown in Fig. 3, we can conclude that there is a synergistic effect of the SW_{LC} synthetic wax and tall oil amidopolyamines interaction on the penetration, shimmer, and Fraass temperatures at 20°C. The intensity of the effects of the two additives on the analyzed binder properties varies depending on their concentration. Synthetic wax SW_{LC} stiffens the binder, while tall oil amidopolyamines reduce its stiffness. The use of both additives and the occurrence of mini-synergy between them makes it possible to regulate the parameters of the modified binder depending on its purpose and the type of mineral asphalt mixture.

3.2. The effect of the synthetic wax LC and tall oil amidopolyamines TOA on long and short-term bitumen ageing

According to the test plan, the effects of short-term (RTFOT) and long-term (RTFOT+PAV) aging on changes in penetration at temperature (Pen_{25}), softening temperature ($T_{R\&B}$), and Fraassa temperature (T_{FRAASS}) of 50/70 bitumen were examined. Tests were carried out on nine specimens in a given series [62]. The change in Pen_{25} of 50/70 bitumen in terms of SW_{LC} and TOA was characterized using ANOVA. Regression models were generated using Statistica [63] and are summarized in Table 3 and shown graphically in Fig. 4. Conversely, the significance analysis of the effects of SW_{LC} and TOA on Pen $_{RTFOT}$ and Pen $_{RTFOT+PAV}$ of modified asphalt was performed using the Pareto relationship, shown in Figures 4a and 4c.

Tab. 3. Fit function parameters for the Pen_{RTFO} and $Pen_{RTFOT+PAV}$

Model parameters	**Pen_{RTFOT} (%); R^2 = 0.723; MS = 8.618**					**$Pen_{RTFOT+PAV}$ (%); R^2 = 0.825; MS = 1.911**				
	RC	**SE**	**-95% C.Limit**	**+95% C.Limit**	**p_{value}**	**RC**	**SE**	**-95% C.Limit**	**+95% C.Limit**	**p_{value}**
Intercept	50.603	3.018	44.635	56.572	0.000	29.188	1.421	26.377	31.999	0.000
(1) SW_{LC} (L)	-18.689	3.502	-25.614	-11.763	0.000	-7.468	1.649	-10.730	-4.2069	0.000
SW_{LC} (Q)	3.047	0.978	1.112	4.982	0.002	0.797	0.460	-0.114	1.708	0.085
(2) TOA (L)	16.522	5.137	6.364	26.681	0.001	6.203	2.419	1.419	10.988	0.011
TOA (Q)	-43.350	6.11653	-55.444	-31.257	0.000	-14.809	2.886	-20.504	-9.113	0.000
1Lx2L	13.603	1.957	9.733	17.473	0.000	7.574	0.921	5.751	9.397	0.000

Where: Q – quadratic, L – linear, RC - Regression Coefficient, SE - Std. Error,

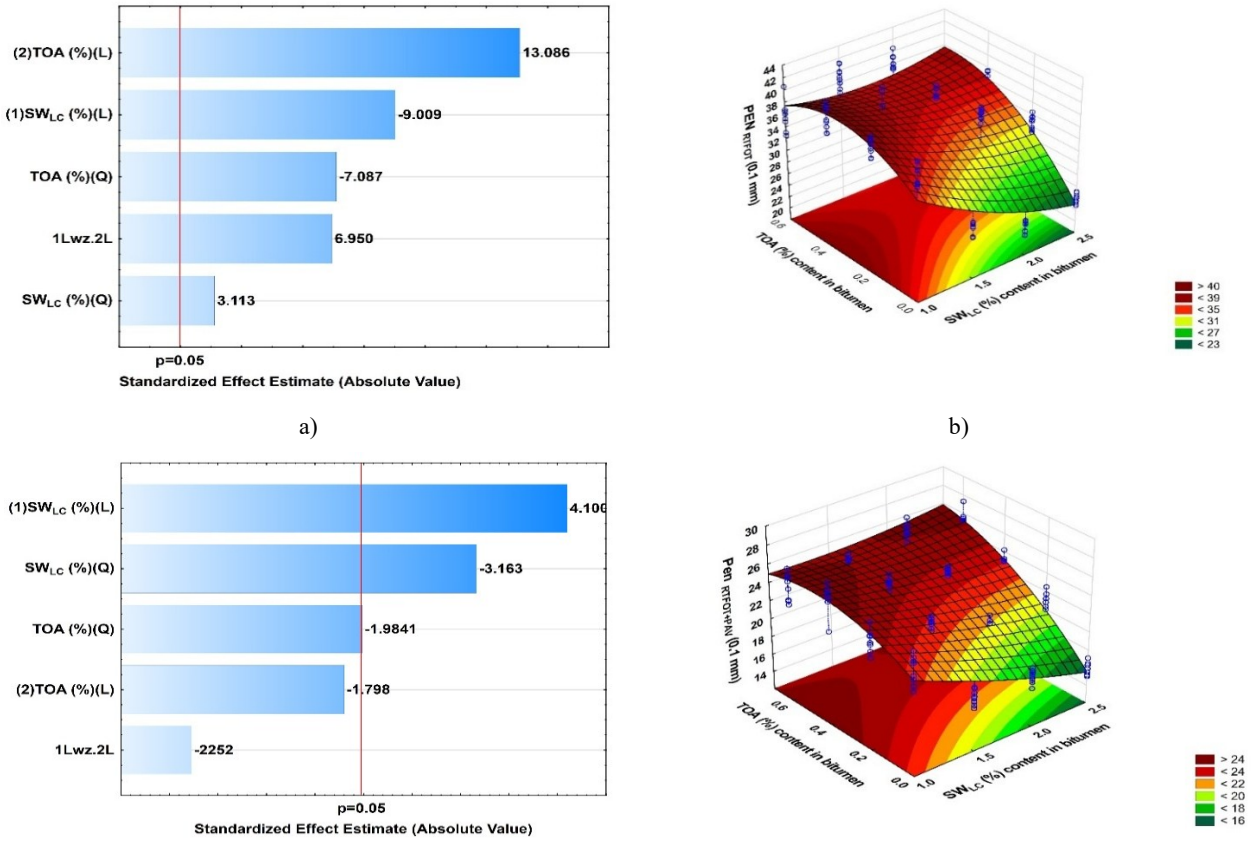


Fig. 4. Influence of WMA additives on bitumen characteristics; a) Pareto relationship for Pen_{RTFOT}, b) Pen_{RTFOT}, c) Pareto relationship for $Pen_{RTFOT+PAV}$, d) $Pen_{RTFOT+PAV}$

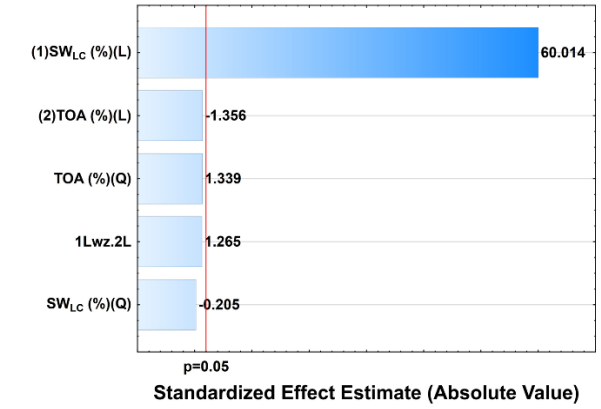
Based on the Pareto analysis in Fig. 4a, it can be concluded that SW_{LC} and TOA both significantly affect the change in penetration after RTFOT aging and that their effects on penetration after aging are synergistic. However, only SW_{LC} synthetic wax significantly affects the change in penetration after RTFOT+PAV aging.

RTFOT aging decreases the penetration of bitumen modified with WMA additives. This trend intensifies after RTFOT+PAV aging. As a result of the aging process, the binder becomes more rigid. Conversely, the aging process of both RTFOT and RTFOT+PAV slows down the rate of penetration reduction as a result of the synergy of SW_{LC} and TOA in the range of 1.0% and more than 0.2%, respectively, and the bitumen becomes less stiff.

The change in the softening temperature value of 50/70 asphalt in terms of SW_{LC} and TOA was characterized using analysis of variance (ANOVA). Regression models using relation (1) were generated in the form of response plots using Statistica [63] and are summarized in Table 4 and shown graphically in Fig. 5. Additionally, the significance of the effect of SW_{LC} and TOA on the $T_{R\&B\ RTFOT}$ and $T_{R\&B\ RTFOT+PAV}$ of the modified bitumen was analyzed using the Pareto relation [64], as shown in Figures 5a and 5c.

Tab. 4. Fit function parameters for the $T_{R\&B\ RTFO}$ and $T_{R\&BRTFOT+PAV}$

Model parameters	$T_{R\&B\ RTFOT}$ (%); R^2 = 0. 963; PE MS = 0.64519					$T_{R\&B\ RTFOT+PAV}$ (%); R^2 = 0.972; PE MS =0. 454				
	RC	SE	-95% C.Limit	+95% C.Limit	p_{value}	RC	SE	-95% C.Limit	+95% C.Limit	p_{value}
Intercept	52.484	0.825	50.851	54.117	0.000	59.509	0.509	58.501	60.516	0.000
(1) SW_{LC} (L)	7.175	0.9582	5.280	9.070	0.000	7.851	0.591	6.682	9.020	0.000
SW_{LC} (Q)	-0.055	0.2677	-0.584	0.474	0.837	-0.851	0.165	-1.177	-0.524	0.000
(2) TOA (L)	-2.937	1.405	-5.716	-0.157	0.0385	3.796	0.867	2.082	5.510	0.000
TOA (Q)	2.242	1.673	-1.066	5.551	0.182	-9.921	1.032	-11.962	-7.880	0.000
1Lx2L	0.677	0.535	-0.381	1.736	0.2079	0.768	0.330	0.115	1.421	0.021



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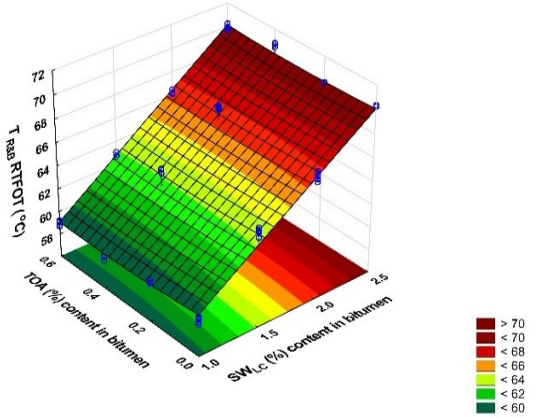

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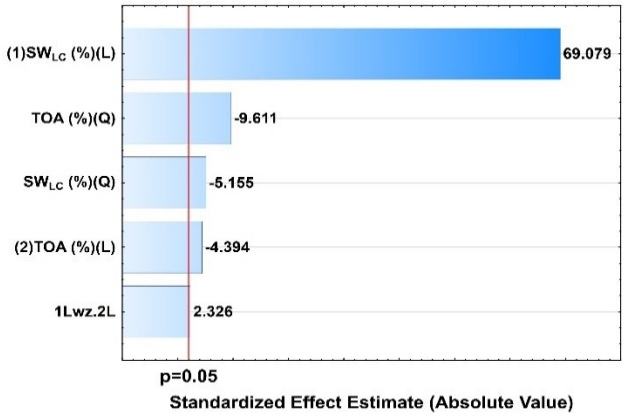

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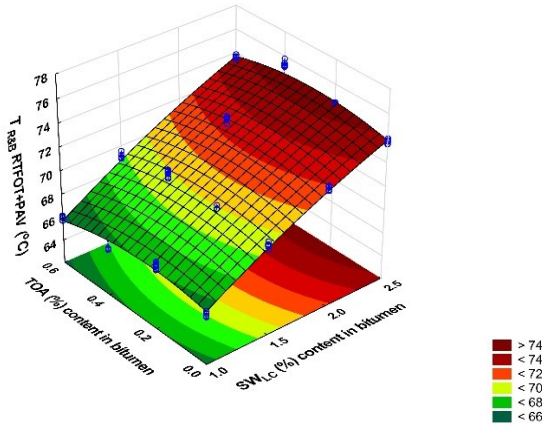

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Fig. 5. Influence of WMA additives on bitumen characteristics; a) Pareto relationship for $T_{R\&B\ RTFOT}$, b) $T_{R\&B\ RTFOT}$, c) Pareto relationship for $T_{R\&B\ RTFOT+PAV}$, d) $T_{R\&B\ RTFOT+PAV}$

The Pareto analysis, shown in Fig. 5a, indicates that SW_{LC} significantly affects the change in softening temperature after RTFOT aging. In contrast, the effects of SW_{LC} and TOA synthetic waxes on the change in softening temperature after RTFOT+PAV aging

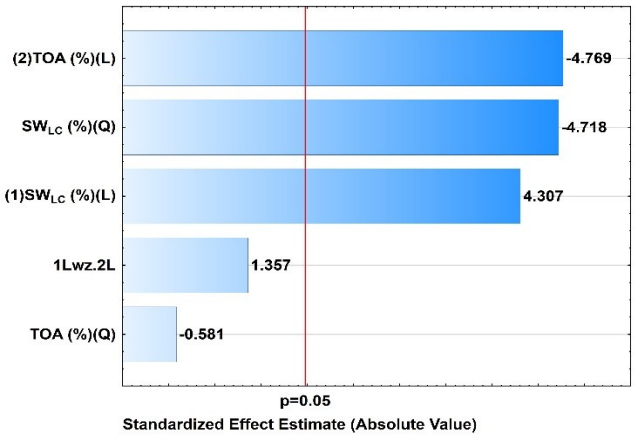
are significant, and a synergistic effect of the two WMA additives on this binder characteristic is observed.

RTFOT aging increases the softening temperature of bitumen modified with SW_{LC} and TOA additives. This trend intensifies after RTFOT+PAV aging. At the same time, the favorable synergistic effect of SW_{LC} and TOA is observed within the dosage range of 1.0% to 1.5% SW_{LC} and the entire TOA dosage range, respectively. The softening temperature increment is much smaller than in the rest of the WMA additive dosage range.

The change in Fraass temperature values of 50/70 bitumen with respect to the amount of SW_{LC} and TOA was characterized using analysis of variance (ANOVA). Regression models using relation (1) were generated in the form of response plots using Statistica software [63] and are summarized in Table 5 and shown graphically in Fig. 6. Conversely, the significance analysis of the effects of SW_{LC} and TOA on the $T_{FRAASS,\ RTFOT}$, and $T_{FRAASS\text{-}RTFOT+PAV}$ of the modified asphalt was performed using the Pareto relationship, as shown in Figures 6a and 6c.

Tab. 5. Fit function parameters for the $T_{FRAASS\ RTFO}$ and $T_{FRAASS\ RTFOT+PAV}$

Model parameters	**$T_{FRAASS\ RTFOT}$ (°C); R^2 = 0.322; PE MS = 33.354**					**$T_{FRAASS\ RTFOT+PAV}$ (°C); R^2 = 0.70; PE MS = 1.662**				
	RC	SE	-95% C.Limit	+95% C.Limit	p_{value}	RC	SE	-95% C.Limit	+95% C.Limit	p_{value}
Intercept	-19.96	1.325	-22.582	-17.339	0.000	-18.868	1.597	-22.027	-15.708	0.000
(1) SW_{LC} (L)	7.575	1.538	4.533	10.616	0.000	6.7561	1.853	3.090	10.422	0.000
SW_{LC} (Q)	-2.027	0.429	-2.877	-1.178	0.000	-1.6389	0.518	-2.663	-0.614	0.001
(2) TOA (L)	-3.395	2.256	-7.857	1.065	0.134	3.2208	2.719	-2.156	8.598	0.238
TOA (Q)	-1.562	2.685	-6.873	3.748	0.561	-6.4236	3.237	-12.825	-0.022	0.049
1Lx2L	1.166	0.859	-0.532	2.866	0.176	-0.2333	1.036	-2.281	1.815	0.822



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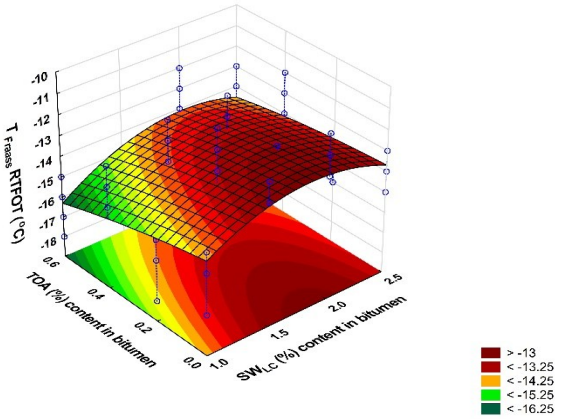

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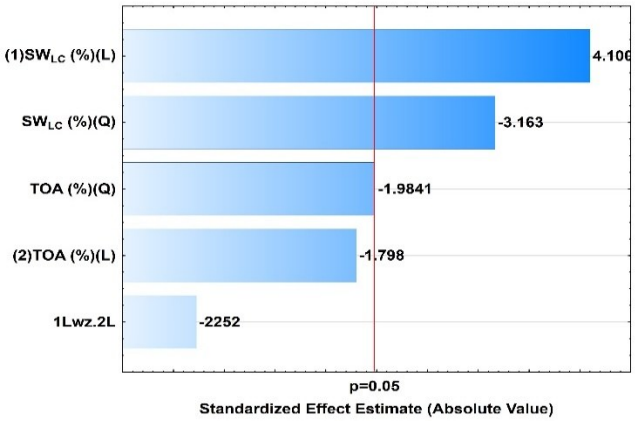

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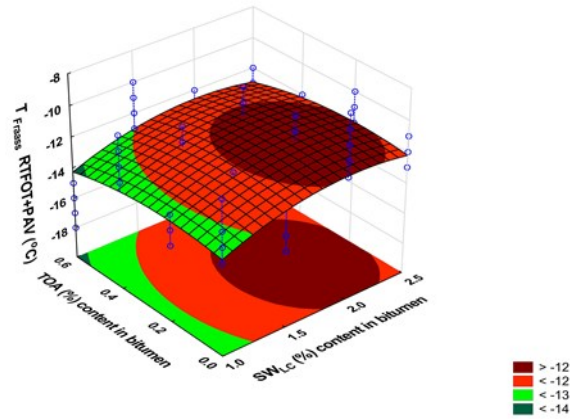

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Fig. 6. Influence of WMA additives on bitumen characteristics; a) Pareto relationship for $T_{FRAASS\ RTFOT}$, b) $T_{FRAASS\ RTFOT}$, c) Pareto relationship for $T_{FRAASS\ RTFOT+PAV}$, d) $T_{FRAASS\ RTFOT+PAV}$

The Pareto analysis, shown in Fig. 6a, indicates that both the TOA and the SW_{LC} significantly affect the change in Fraass temperature after RTFOT aging. However, it should be noted that the effect of TOA is more significant than that of SW_{LC}. Conversely, in the RTFOT+PAV aging process, WMA additives significantly affect the inverse relationship.

RTFOT aging increases the Fraass temperature of bitumen modified with SW_{LC} and TOA additives. At the same time, a favorable synergistic effect of SW_{LC} and TOA is observed at a dosage of 1.5% SW_{LC} and 0.4% TOA, respectively. A similar trend is observed after RTFOT+PAV aging: a further increase in Fraass temperature. A favorable synergistic effect of WMA additives is observed over the entire range of TOA and synthetic wax dosage, from 1.0% to 1.5%.

In summary, the RTFOT and RTFOT+PAV aging processes cause significant changes in penetration values at 25°C, softening temperature, and Fraass temperature. The binder becomes more rigid. However, it should be noted that the favorable synergistic effect of the synthetic wax, SW_{LC}, and TOA slows down the binder's aging rate. Based on the evaluation of the test results, the optimal composition of WMA additives to bitumen occurs at a SW_{LC} dosage of 1.5% and a TOA dosage of 0.4%.

4. Conclusion

Based on the study of the effects of synthetic wax (SWLC) and tall oil amidopolyamine (TOA) on the properties of bitumen 50/70 with the RTFOT and RTFOT+PAV aging processes, the following conclusions can be drawn:

1. The RTFOT aging process alters the properties of bitumen 50/70 containing SWLC synthetic wax and TOA tall oil amidopolyamine. During RTFOT+PAV service aging, the tendency to change the properties of asphalt with WMA additives continues.
2. The intensity of the effect of the RTFOT and RTFOT+PAV aging processes on the change in the properties of the bitumen depends on the content of the SWLC synthetic wax and the TOA tall oil amidopolymine in the binder.
3. The effect of RTFOT and RTFOT+PAV aging on the change in the properties of bitumen is reduced by the synergy of SWLC and TOA at optimal contents of 1.5% and 0.4% by weight of the binder, respectively.
4. Synthetic wax SWLC and tall oil amidopolyamine TOA, when applied to bitumen in optimal amounts, act as aging inhibitors.

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