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Assessment of polymer-modified bituminous binders with enhanced resistance to thermooxidative and ozone aging

Abstract: The article explores the evaluation of aging processes in bitumen materials to develop innovative binders with improved resistance to aging, based on commercially available products. The study incorporates RTFOT, PAV, and ozone aging in a controlled simulation chamber to mimic real-world conditions. Rheological properties were assessed using a Dynamic Shear Rheometer (DSR), and chemical changes in the binders were analyzed using FT-IR spectroscopy. Furthermore, the newly developed binders were classified based on standard test methods. The results highlight the importance of selecting precise test methods in developing high-performance binder formulations with improved durability. The new formulations suggest an average 15% improvement in resistance to both thermo-oxidative and ozone aging compared to the reference binders.

Keywords: improved aging resistance, ozone aging, polymer-modified bitumen, thermooxidative aging.

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1. INTRODUCTION

Aging resistance of bituminous binders refers to the ability of the material to preserve its properties after it was initially produced [1]. In case of bitumens in particular, the process of aging is generally described in two distinct categories:

- Short Term Aging (STA): during the preparation of asphalt mixture and road construction,
- Long Term Aging (LTA): during the rest of the service life of a pavement.

In the typical method of binder production, the most direct process of chemical change is a result of reaction between oxygen (O_2) in air and reactive compounds in the bitumic mixture at elevated temperatures. These processes involve mainly oxidation by oxygen in air, molecular rearrangements (such as chain scission) and condensation reactions. Additional aspects, such as evaporation of volatile compounds also have a significant effect on overall properties of binders [2].

The process of oxidation of vacuum residue in a predominant method for production of various bitumen grades, in which the desired physical properties – dictated by normative requirements – are obtained by controlled oxidation of bitumen with air at high temperatures (200-300°C).

Short Term Aging is mostly attributed to additional oxidation that occurs mainly in the process of mixing binder with mineral aggregate, at elevated temperatures and in the presence of air. As such, the impact of this process has to be taken into account in road construction. STA is normally simulated using Rolling Thin Film Oven Test (RTFOT), and the extent to which parameters of binder can change is normalized [3, 4].

The most common method for long-term aging is the Pressure Aging Vessel (PAV) method. As another variant of thermooxidative method, where a sample is subject to air under increased pressure, it allows to simulate degradation of properties after construction. Literature sources estimate that PAV aging is equivalent to about 10-15 years of service [5], depending on conditions. Parameters of binders after PAV aging are provided by manufacturers as a part of product specification and normatively required in most countries.

The thermooxidative mechanism has been of great interest to researches for quite some time now, resulting in many observations and theories regarding the degradation of binders in presence of oxygen [5]. The detailed discussion of these finding won't be elaborated on in this paper, but one common factor is all works on subject identify the presence of molecular oxygen (O_2) in air as a direct initiator of the thermooxidative degradation [6].

Furthermore, in order to better understand how the presence of oxygen affects the organic framework of bitumen, impact of other reactive forms of oxygen was evaluated, mainly ozone (O_3) and various nitrogen oxides (NO_x). The systematic approach to such testing for eventually formulated by Mirwald et al. as the Viennese Binder Aging method (VBA) [7] – whereby the binder is subject to mixture of reactive oxygen species (ROS), consisting of ozone and different nitrogen oxides, produced by static electricity discharge generator at 80°C.

Study by Primerano et al. [8] conducted additional analysis, by selectively exposing bitumen samples to O_3, N_O and NO_2. Although the study was not able to show the direct correlation between the concentration of ozone and degree of degradation, in the chosen concentration range of between about 1200 to 6000 ppm, it provided direct evidence that "ROS play an important role and should not be disregarded when looking at aging of bituminous materials".

In this paper we aim to evaluate the impact of ozone on the modified bitumens, of which desirable properties are a result of synergistic interaction between the polymeric network and the bituminous matrix [9]. Ozone has long been identified as one of main contributing factors to degradation of butadiene-based elastomers, analyzed in detail in the field of polymer chemistry [10]. Resistance to ozone cracking is an important property of vulcanized rubbers and is generally tested according to the ISO 1431-1:2022 standard [11]. Since Styrene-Butadiene-Styrene (SBS) type elastomers are commonly used for binder modification, the goal of the study is to asses whether there is larger destructive impact on modified bitumens as a result of introduction of elastomer susceptible to ozone degradation.

In order to better understand the conducted analysis, the use of Fourier Transform Infrared Spectroscopy (FT-IR) is proposed to monitor various products of oxidation of binder in the carboxyl and carbonyl range (1600-1800 cm^{-1}) of the spectrum [12]. Recent development of quantitive or semi-quantitive method for evaluation of aging in bitumens using FT-IR spectroscopy has pushed towards higher degree of standardization, with the aim to provide more accurate results and allow for better comparison of data between researchers [13-17]. The semi-quantitive analysis is conducted with the use of carbonyl index, where the progress in recent literature has been taken into account.

2. MATERIALS AND METHODS

2.1. MATERIALS

Commercial binders of three different specifications were used as a reference material in this study, consisting of asphalts with different content of Styrene-Butadiene-Styrene (SBS) copolymers:

- 50/70 Unmodified Bitumen,
- 45/80-55 Polymer Modified Bitumen (about 2% to 4% SBS),
- 45/80-80 Highly Modified Bitumen (> 5% SBS).

Additionally, two experimental compositions of the same normative parameters with Improved Ageing (IA) resistance were prepared using modified recipe and provided for comparison. These binders are referred to as IA compositions: 45/80-55 IA and 45/80-80 IA, respectively.

2.2. METHODS

2.2.1. RTFOT and PAV aging

The standardized Rolling Thin Film Oven Test (RTFOT) and Pressure Aging Vessel (PAV) were used to simulate the STA and LTA respectively. RTFOT was conducted according to EN 12607-1 at the temperature of 163℃ for 75 minutes [18]. PAV was performed at 100℃ for 20 hours, at the pressure of 2.1 MPa according to EN 14769:2023 [19].

2.2.2. Ozone aging

Samples of bitumen after RTFOT of 50.0 g ± 0.5 g were poured on circular plates with a diameter of 15 cm. The temperature of the binder was such that it allowed for the warm mixture to spread evenly on the dish. The ozone simulation was conducted using ANSEROS SIM 7300TH Ozone Testing Chamber, at 10.0 ppm of O_3 for 72 hours at the temperature of 60°C. Considering that the chamber generates ozone from air using electric discharge generator, a non-negligible amount of various nitrogen oxides (NO_x) is likely present. The exact concentration of NO_x was not measured during the experiment, but it is considered to be about 10 times lower than O_3 concentration similar that in other experiments with comparable setups [7]. The humidity in testing chamber was not controlled. After aging the qualitative *IR* spectra directly from surface of each sample were collected. The *IR* measurements used for the calculation of carbonyl index were measured after samples were homogenized, by brief heating to temperature of about 180°C and then mixing thoroughly.

2.2.3. Standardized testing of bitumen properties

All unaged bitumen samples were subjected to assessment required by the PN-EN 14023 normative classification [20], a Polish standard, based on the European norm. The Ball and Ring Test for the softening point was conducted according to PN-EN 1427 [21], and the Penetration Test was done according to PN-EN 1426 standard [22]. Storage stability of binders was assessed based on PN-EN 13399 standard [23]. Elastic recovery was measured in the temperature of 25°C, according to PN-EN 13398 [24].

2.2.4. DSR analysis

Rheology of all bitumen samples selected for the experiment was analyzed according to the PN-EN 14770 [25] standard using Anton Paar Physica MCR 101 dynamic shear rheometer (DSR). The parallel plate setup was used, with the 25 mm diameter shearing plates and the gap of 1 mm. The complex shear modulus G^* and phase angle δ were measured, for each sample before and after aging, at temperatures of 10°C, 25°C, 60°C and 80°C at a constant oscillation speed of 10 rad/s. The ranges of applied deformations were determined taking into account the stiffness of the tested materials, in a way that allows tests to be carried out in the linear range of viscoelasticity of binders.

Specific aging indices based on Complex Modulus G^* and Phase Shift Angle δ were used to analyze the aging of binders after individual aging stages: Complex Modulus Aging Index CMAI (1) and Phase Angle Aging Index PAI (2):

$$CMAI = \frac{G^*_{aged}}{G^*_{unaged}}, \tag{1}$$

$$PAI = \frac{\delta_{aged}}{\delta_{unaged}}. \tag{2}$$

2.2.5. Fourier-Transform Infrared Spectroscopy

All spectra used in the study have been registered using Perkin-Elmer Spectrum Two FT-IR spectrometer with ATR diamond crystal, at resolution of 4 cm^{-1} and four scans in the range from 4000 to 450 cm^{-1}. The binder sample has been applied directly onto the crystal and pressed down with use of the pressure screw. Baseline correction was performed for the post-processing of the spectra using a set point approach at 3100, 1753, 930 and 660 cm^{-1} [14]. The range of aging indices found in recent literature was tested, with the baseline integration of the carbonyl range of 1722-1678 cm^{-1} appearing to provide most accurate results. Spectra used for calculation of the carbonyl indices were used without further normalization. The aliphatic range of $–CH_2$ and $–CH_3$ bending vibrations at 1518 cm^{-1} to 1330 cm^{-1} was selected based upon literature and used without further modification [14]. The semi quantitive carbonyl index was calculated according to (3):

$$I_{\mathrm{C=O}} = \frac{A_{\mathrm{C=O}\ (1722-1678)}}{A_{\mathrm{ref}\ (1518-1330)}} \cdot 1000\ [\mathrm{a.u.}], \tag{3}$$

where:

$I_{\mathrm{C=O}}$ – carbonyl index,

$A_{\mathrm{C=O}}$ – area of carbonyl range,

A_{ref} – area of reference range.

Difference of carbonyl index, between the aged sample and the unaged bitumen of the same grade was calculated using (4):

$$\Delta I_{\mathrm{C=O}} = I_{\mathrm{C=O}\ (aged)} - I_{\mathrm{C=O}\ (unaged)}. \tag{4}$$

3. RESULTS AND DISCUSSION

3.1. PARAMETERS OF BINDERS BEFORE AGING

All binder compositions in the study were subjected to analysis based on normative classification, based upon PN-EN 14023 standard [20]. Parameters for all unaged bitumens presented in this study are compared in Table 1.

The IA binder compositions follow the same normative classification as their reference equivalents. Storage stability tests indicate that modified compositions do not undergo phase separation.

3.2. IMPACT OF AGING ON BITUMENS WITH DIFFERENT POLYMER CONTENT

3.2.1. Scope of analysis

In order to assess the impact of the ozone aging method proposed in this study it was presented in conjunction with established aging methods of RTFOT and PAV. The short-term aging is represented by at RTFOT stage, with long-term being simulated by RTFOT + PAV and RTFOT + O_3 tests, on all three binder compositions with different polymer content. The samples before aging and after each aging stage were analyzed using DSR and FT-IR spectroscopy.

3.2.2. DSR analysis of the reference binders

The DSR analysis of complex modulus G^* and phase angle δ after each stage of aging allows for direct comparison of changes observed as a result of RTFOT, RTFOT + O_3 and RTFOT + PAV simulations. The measurements were taken at 10°C, 25°C, 60°C and 80°C covering a wide range of temperatures corresponding to real-world conditions. Fig. 1 shows G^* for each grade of binder at different stages of aging.

Table 1. Normative parameters of binders before aging

Parameter	Sample				
Type of bitumen	50/70	45/80-55	45/80-55 IA	45/80-80	45/80-80 IA
Penetration at 25°C [0.1 mm]	63	61	62	53	59
Softening point [°C]	50	66.4	61.6	91	90.5
Elastic recovery at 25°C [%]	–	90	86	93	86
Storage stability test Softening point difference [°C]	–	0.4 Top: 70.6 Bottom: 70.2	1.0 Top: 67.2 Bottom: 68.2	0.0 Top: 93.0 Bottom: 93.0	0.0 Top: 93.5 Bottom: 93.5
Storage stability test Penetration difference [0.1 mm]	–	0 Top: 65 Bottom: 65	0 Top: 69 Bottom: 69	−1 Top: 58 Bottom: 59	−1 Top: 64 Bottom: 65

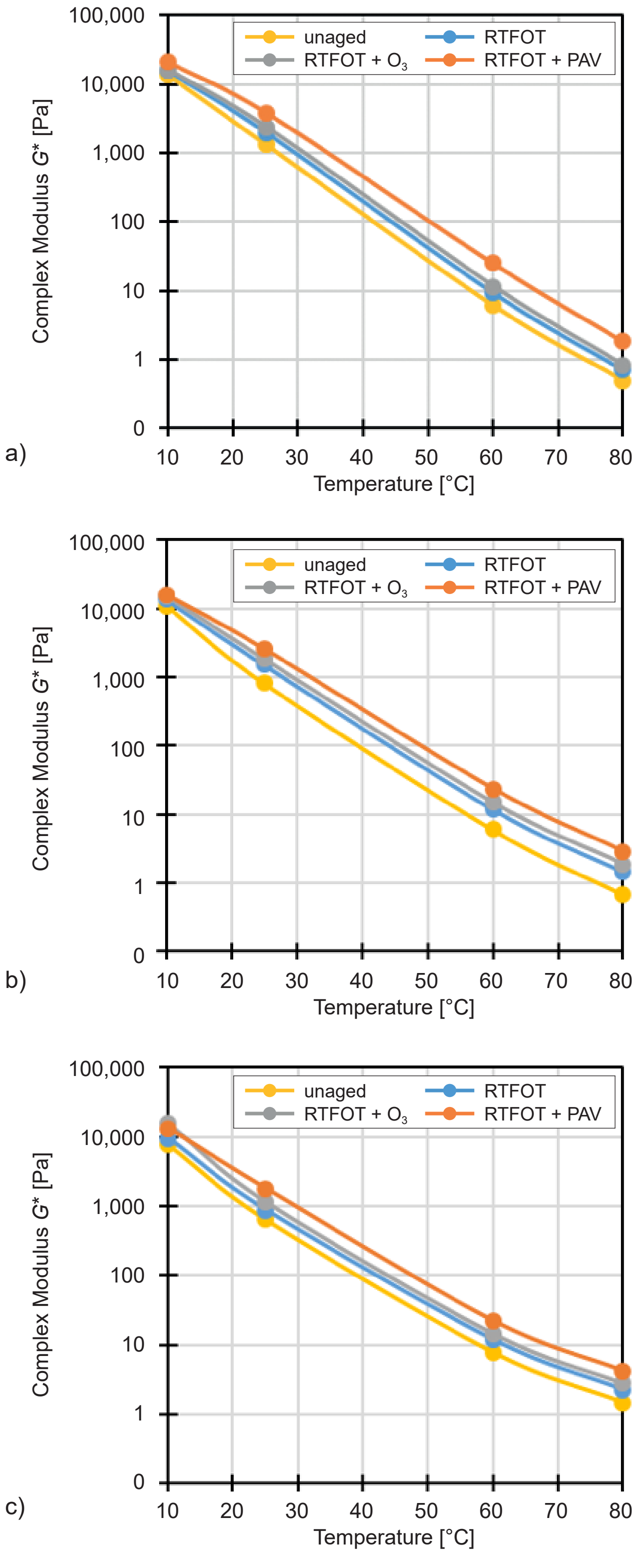

Fig. 1. Complex modulus G^* at each aging stage:
a) 50/70, b) 45/80-55, c) 45/80-80

Across different degrees of modification the complex modulus of unaged binders follows a similar trend with regards to the temperature. The impact of simulated aging is clearly visible, with an increase in G^* corresponding to the hardening of the material during RTFOT, RTFOT + O_3 and PAV simulations. This change can be quantitatively measured using the calculated CMAI index, provided in Fig. 2.

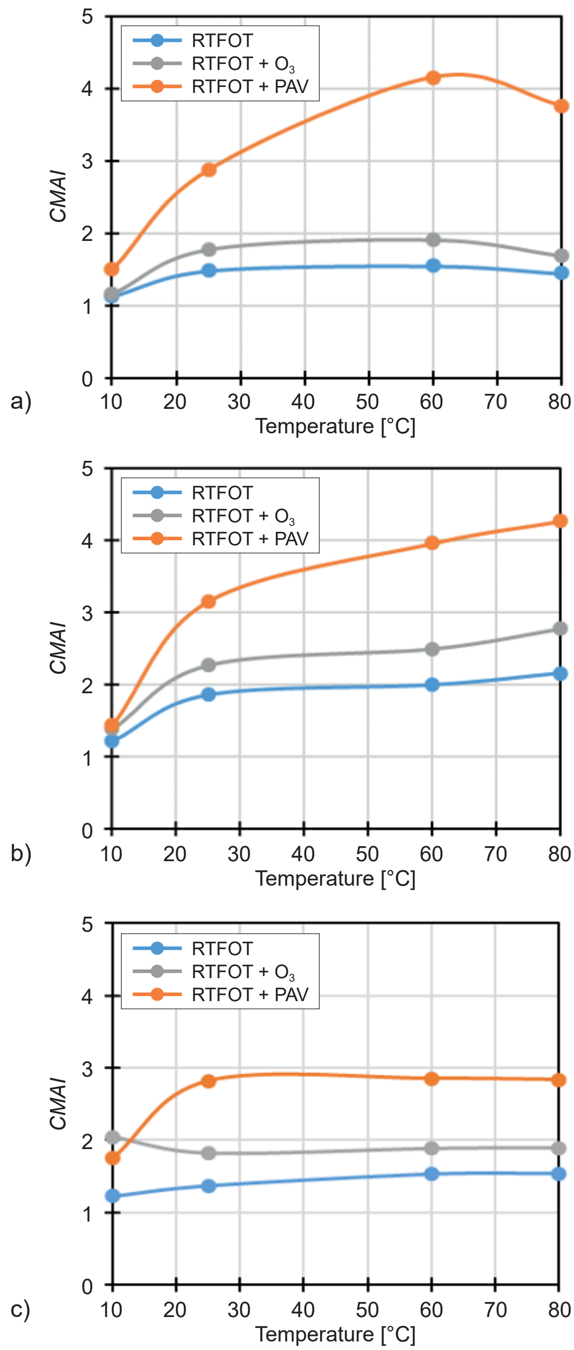

Fig. 2. *CMAI* at each aging stage: a) 50/70, b) 45/80-55, c) 45/80-80

The *CMAI* indicator describes a relative impact of aging in terms of complex modulus. The value of 1.0 corresponds to G^* of the unaged binder, with higher index meaning more pronounced stiffening of the material as a result of aging. The RTFOT aging in most temperatures resulted in average *CMAI* value of about 1.5 for 50/70 and 45/80-80 binders, and the subsequent ozone chamber aging increased the *CMAI* to factor in the range of 2. In all materials RTFOT + PAV resulted in highest relative G^* values, exceeding those of RTFOT + O3 by a factor of about two times. Another crucial parameter in describing the viscoelastic properties of binders is phase angle, shown in Fig. 3.

The phase angle δ is considered to indicate the balance between elastic and viscous behavior of certain material. A low phase angle (close to 0 degrees) indicates a predominantly elastic or solid-like behavior, while a phase angle closer to 90 degrees indicates a predominantly viscous or liquid-like behavior. This is clearly visible when comparing the unmodified 50/70 binder to its modified cunterparts – in modified bitumens the elastic behavior is present even at high temperatures – accounting for much lower values of δ. The lowest value of δ across the temperature range is measured in 45/80-80 binder, as a direct result of introduction of SBS polymer in a relatively large amount. The plots at different stages of aging also illustrate the significant change in phase angle as a result of both RTFOT, ozone and PAV. The biggest change is visible after RTFOT + PAV procedure, with RTFOT + O_3 resulting in a minor increase over the RTFOT simulation alone.

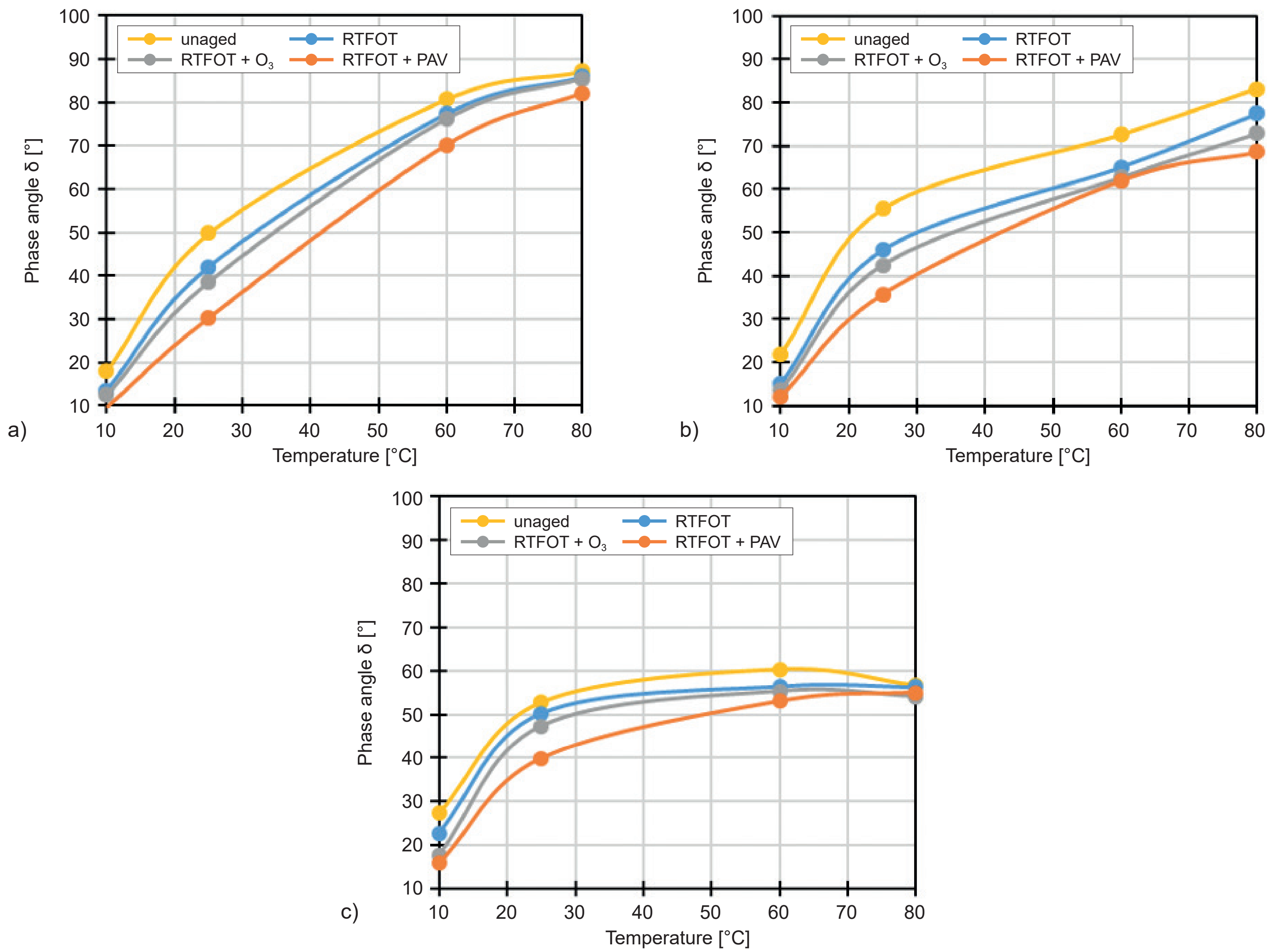

Fig. 3. Phase angle δ at each aging stage: a) 50/70, b) 45/80-55, c) 45/80-80

3.2.3. Change in FT-IR absorption spectra of aged binders

The analysis of infrared spectra provides crucial information on chemistry of bitumens, with distinct absorption bands for different chemical bond types and functional groups present in the material. Comparison of FT-IR measurements, after each stage of aging, allows for the qualitative analysis of polymer modified binders as well as to conduct a semi-quantitive calculation of carbonyl index that corresponds to the increase in carbonyl bonds as a direct result of progressive oxidation of the material as a result of aging. Figs 4 and 5 show the comparison of FT-IR spectra in the 1900 cm^{-1} to 1500 cm^{-1} range for 45/80-55 and 45/80-80 binder respectively, after each aging simulation.

The carbonyl band, with maximum at around 1700 cm^{-1} has been identified as a main indicator of progressing oxidation, resulting in formation of the carbonyl and carboxylic compounds. The measurement directly from the surface of the sample exposed to ozone reveals disproportionally high degree of oxidation, with broadening of the band into the carboxylic region of about 1750 cm^{-1}. It is important to note that the surface spectrum was processed using a modified baseline approach, and as such can be used only to qualitatively assess the impact of degradation. In case of both binders the trend in terms of the absorption in carbonyl region was determined to be RTFOT + PAV > RTFOT + O_3 > RTFOT. These spectra were registered from homogenized materials and processed in the same way, allowing for calculation of semi-quantitive carbonyl indexes. The difference between iCO of the unaged material, and the index after aging, is calculated in Fig. 6.

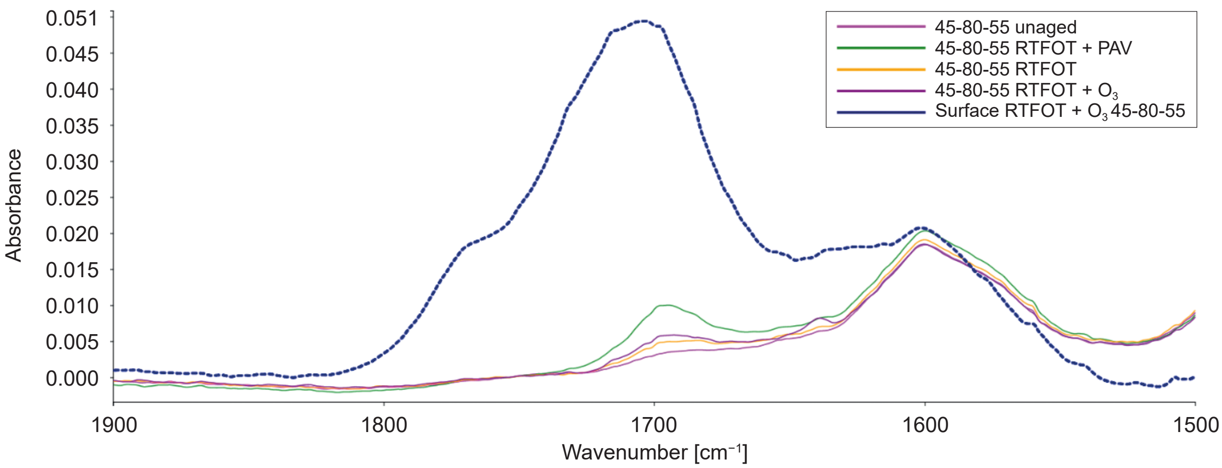

Fig. 4. Spectra of polymer modified binders in 1900-1500 cm^{-1} region for 45/80-55 grade

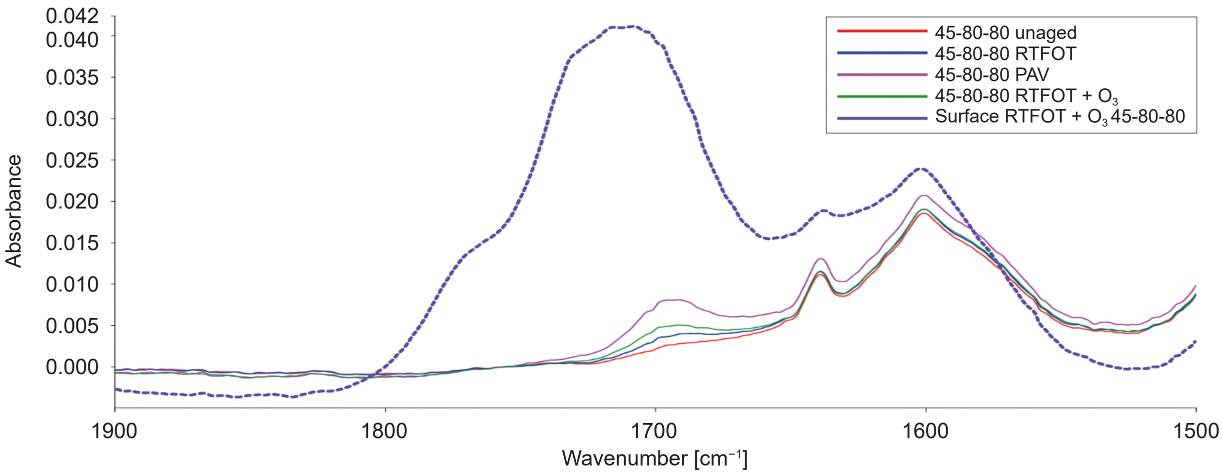

Fig. 5. Spectra of polymer modified binders in 1900-1500 cm^{-1} region for 45/80-80 grade

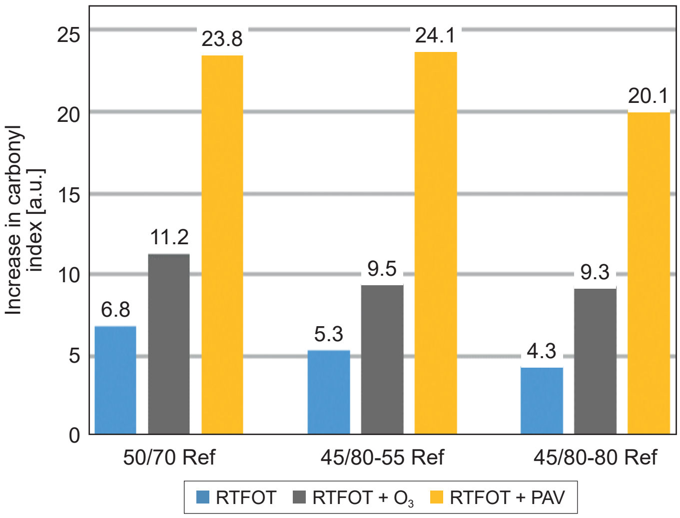

Fig. 6. Difference in carbonyl index after each stage of aging for reference samples

The values of carbonyl index allow for monitoring of the degree of oxidation of selected bitumen stages, after each aging simulation. For RTFOT and RTFOT + O_3 aging the magnitude of oxidation is similar across the grades with different polymer content, with the lower values across binders with increasing polymer content. The oxidation during the ozone aging process is clearly visible, with the proposed method resulting in additional increase of carbonyl index of similar level to that of RTFOT aging, and about 20% to 25% of that of PAV procedure.

3.3. AGING RESISTANCE OF COMPOSITIONS WITH IMPROVED AGING PERFORMANCE (IA)

3.3.1. IA compositions

The IA variants are bitumen compositions of the same normative grade as the reference materials, prepared according to modified composition with a goal to improve aging resistance of binders. The IA variants are evaluated following the similar procedure to the reference binders, including RTFOT, O_3 and PAV accelerated aging. The surfaces of modified bitumen samples, after RTFOT + O_3 aging, are shown in Fig. 7.

The first step in analysis of the impact of ozone aging on the tested samples was visual observation of binders after the ozone test in environmental chamber. The oxidation is expected to occur directly at the surface, which may be indicated by the matting of the part of the binder exposed to ozone.

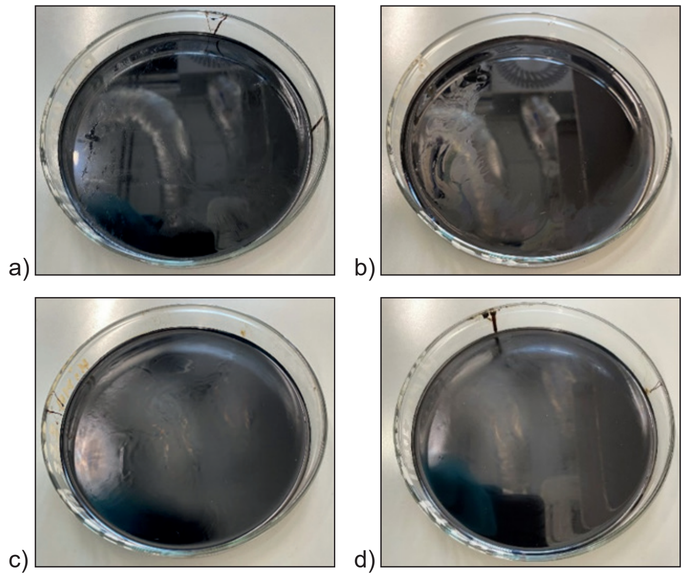

Fig. 7. Surfaces of reference and IA compositions after RTFOT + O_3 aging for: a) 45/80-55 Ref, b) 45/80-55 IA, c) 45/80-80 Ref, d) 45/80-80 IA

3.3.2. *CMAI* indicators of the IA binders

CMAI indicators for improved aging variants for 45/80-55 grade polymer modified binder and the 45/80-80 grade highly modified binder are compared to the reference materials in Figs 8 and 9.

Across bitumen grades of 45/80-55 and 55/80-80 both IA compositions are characterized by significantly lower *CMAI* index across different all tested aging methods. The results indicate lesser degree of hardening of the material, with the *G** values being closer to those of an unaged sample. The only exception to this trend is when the 45/80-80 binder was measured at 10°C – this observation should not be discarded, and could possibly be explained by the higher impact of ozone on the polymer network which is crucial for the properties in lower temperatures. The hypothesis would require additional verification however, but could indicate a condition where ozone aging impacts the modified bitumen in a different way than RTFOT + PAV.

The characteristic lower values of *CMAI* index for the IA compositions are consistent across all aging stages (RTFOT, RTFOT + O_3, RTFOT + PAV) in temperatures above 25°C, with the degree of improvement proportional to the extent of aging. The result indicate that the IA compositions, in both variants, are on average 15% more resistant to long term and ozone aging.

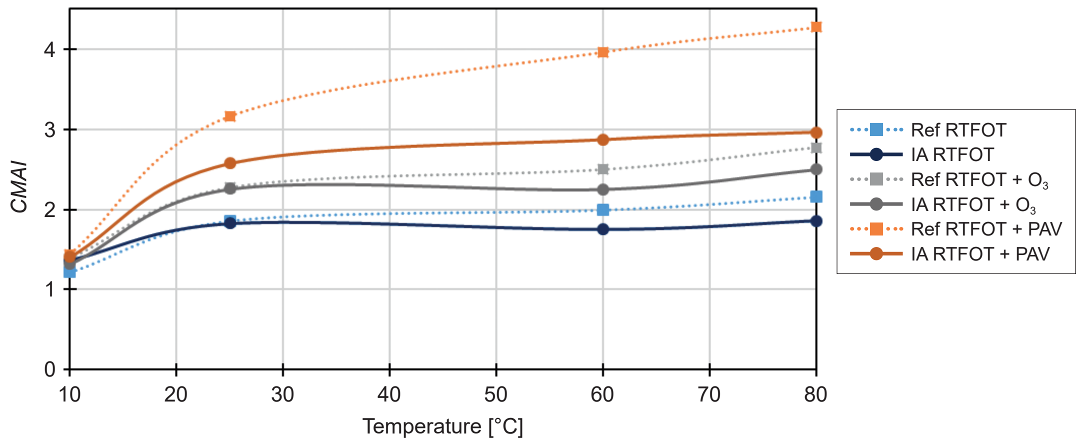

Fig. 8. Comparison of CMAI of reference and IA binders of 45/80-55 grade bitumen

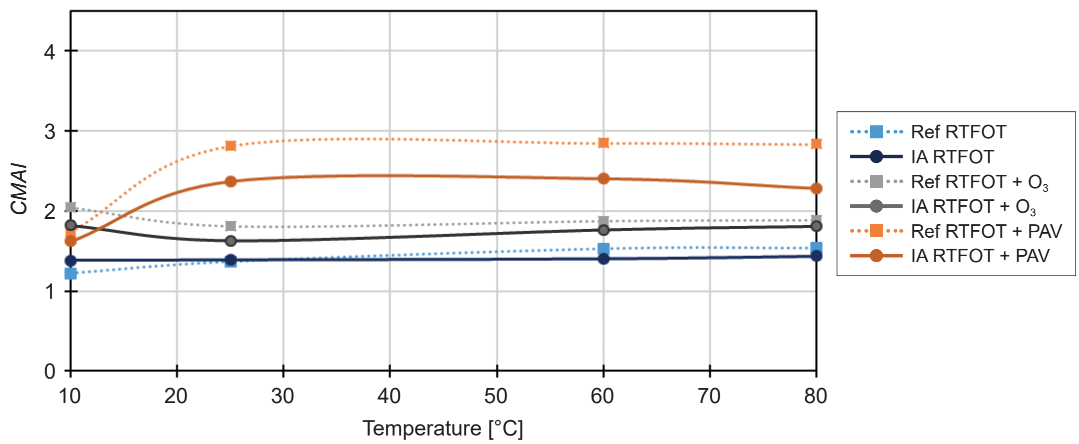

Fig. 9. Comparison of CMAI of reference and IA binders of 45/80-80 grade bitumen

3.3.3. FT-IR analysis of the IA compositions

The analysis of the difference in carbonyl index at each stage of aging, for the reference and IA samples, is shown in Fig. 10.

In terms of carbonyl index the IA compositions follow similar trend to the reference binders, with some of the aging experiments suggesting higher oxidation of IA samples – mainly in case of ozone aging of 45/80-55 binder and RTFOT aging of 45/80-80 binder. This implies that the carbonyl index after RTFOT + O_3 does not directly correlate to the rheological properties measure using DSR in the previous section – specifically in case of IA binders. One possible explanation for that is the introduction of some new components into the bituminous mixture may cause increase in absorption within the carbonyl range, while not contributing to the deterioration of the mechanical properties of those compositions (IA variants).

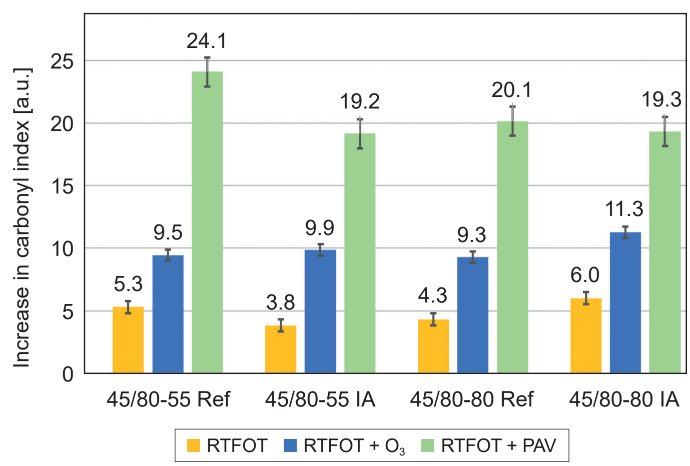

Fig. 10. The difference in carbonyl index by stage of aging, IA and reference compositions

4. SUMMARY AND CONCLUSIONS

The aging behavior of a wide range of materials, consisting of unmodified (50/70), modified (45/80-55) and highly modified (45/80-80) bitumens was studied under the RTFOT and PAV procedures, as well as a variation of ozone aging according to novel procedure with the use of ozone testing chamber. Binders at each aging stage were examined using both rheological approach using DSR as well as analysis of chemical structure with FT-IR. The following conclusions are presented as a result of these experiments:

1. The impact of ozone, alongside other reactive oxygen species, on analyzed binders resulted in consistent oxidation of binders in the study, with resulting impact of both rheological properties and carbonyl index of about 25% that of established PAV procedure. Since the proposed method degrades samples predominantly at the surface, it may be tuned to provide a desired degree of degradation using different ozone concentrations and thickness of the material.
2. The FT-IR analysis revealed the progressing oxidation in bitumens as a result of aging, both in case of modified and unmodified binders. The calculation of carbonyl index using the semi-quantitive approach allowed for accurate modeling of the evolution of carbonyl band, and was found complementary to DSR analysis.
3. Improved aging compositions allowed for about 15% improvement in terms of change of rheological *CMAI* index after aging, in both in case of modified (45/80-55) and highly modified (45/80-80) binder. The obtained effect of improved aging resistance of IA binders requires further research and confirmation on asphalt-mineral mixtures, as well as in real operating conditions.

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Ocena lepiszczy bitumicznych modyfikowanych polimerami o zwiększonej odporności na starzenie termiczne i ozonowe

Streszczenie: W artykule dokonano oceny procesów starzenia lepiszczy asfaltowych w celu opracowania innowacyjnych lepiszczy o zwiększonej odporności na starzenie, opartych na dostępnych komercyjnie produktach. W badaniu zastosowano metody RTFOT, PAV oraz starzenie ozonowe w kontrolowanej komorze symulacyjnej. Właściwości reologiczne materiałów oceniano z wykorzystaniem dynamicznego reometru ścinającego DSR (ang. *Dynamic Shear Rheometer*), a zmiany chemiczne w lepiszczach analizowano przy użyciu spektroskopii FT-IR. Ponadto dokonano klasyfikacji nowo opracowanych lepiszczy w oparciu o normowe metody badań. Wyniki przeprowadzanych analiz uwydatniają znaczenie doboru precyzyjnych metod badawczych w tworzeniu wysokowydajnych formulacji lepiszczy o zwiększonej trwałości. W porównaniu do lepiszczy referencyjnych przygotowane nowe kompozycje sugerują średnio o 15% lepszą odporność na starzenie zarówno termooksydacyjne jak i ozonowe.

Słowa kluczowe: asfalty modyfikowane polimerami, odporność na starzenie, starzenie asfaltów, starzenie ozonowe.