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EFFECTIVENESS OF A SPECIFIC RECLAIMED ASPHALT PAVEMENT (RAP) REJUVENATING AGENT

SKUTECZNOŚĆ SPECJALISTYCZNEGO PREPARATU CHEMICZNEGO W ODŚWIEŻANIU GRANULATU ASFALTOWEGO

STRESZCZENIE. Materiały stosowane w budownictwie powinny funkcjonować w obiegu zamkniętym, co oznacza ich ponowne wykorzystanie w momencie utraty właściwości w pierwotnym wyrobie budowlanym. Do tego typu materiałów należy zaliczyć mieszanki mineralno-asfaltowe, które w postaci destruktu asfaltowego pozyskiwane są z remontowanych lub przebudowywanych dróg. Ich funkcjonalność w dużym stopniu zależy od właściwości lepiszcza asfaltowego, które w wyniku procesów starzeniowych utraciło swoje właściwości lepkosprężyste. Poprawę tych właściwości można uzyskać poprzez zastosowanie specjalistycznych preparatów chemicznych zwanych rejuwenatorami. W artykule przedstawiono wyniki badań, które podzielono na dwa etapy. W pierwszym etapie badania wykonywano dla lepiszcza asfaltowego. Wykorzystano asfalt drogowy 50/70 przed i po starzeniu (RTFOT + PAV) oraz postarzone lepiszcze z dodatkiem rejuwenatora. Obok badań podstawowych (penetracji, temperatury mięknięcia, zespolonego modułu ścinania i kąta przesunięcia fazowego) oznaczono skład grupowy asfaltów oraz wykonano analizy widmowe. W drugim etapie badania wykonano dla betonu asfaltowego AC 16 W 50/70, pozyskanego z WMB. Oznaczono podstawowe parametry mieszanki referencyjnej, po starzeniu technologicznym oraz eksploatacyjnym oraz z udziałem odświeżacza. W zakresie oznaczeń była gęstość, gęstość objętościowa, odporność na działanie wody, moduł sztywności, trwałość zmęczeniowa oraz parametry niskotemperaturowe mieszanki (TSRST). Przeprowadzone badania granulatu asfaltowego z udziałem estera fosforowego alkoholu olejowego, etoksyłowanego świadczą, że substancja ta wpływa pozytywnie na zmiany właściwości zestarzonych lepiszczy asfaltowych, co pozwala na powodzeniem stosować ją w technologii drogowej.

SŁOWA KLUCZOWE: granulát asfaltowy, asfalt, rejuwenator, moduł sztywności, zmęczenie, TSRST.

ABSTRACT. Contemporary construction materials are expected to be recyclable in a closed-loop system, meaning they should allow reuse, after they have lost their original properties while being part of the original building product. This group of materials should undoubtedly include bituminous mixtures, which may be reclaimed during road renewal or alteration projects, and subsequently reused as Reclaimed Asphalt Pavement (RAP). Noteworthy, RAP performance depends on the properties of the binder it contains, whose visco-elastic behaviour has been affected by the ageing processes. Fortunately enough, these properties of bitumen may be effectively recovered by means of special chemical agents called rejuvenators. This paper presents the results of two-stage research project on this subject. The first stage included testing of the bituminous binder under analysis. It was a 50/70 pen-grade bitumen tested before and after short-term ageing (RTFOT + PAV) and aged binder tested after rejuvenation treatment. Spectroscopic and fractional composition analyses were carried out in addition to determining the basic properties i.e. penetration, softening point, dynamic shear modulus and phase angle. In the second stage of this research project a different material was tested: AC 16 W 50/70 asphalt concrete sourced from a hot-mix asphalt plant. The specimens were made from the fresh mix (control) and the same mix after short- and long-term ageing and after subsequent rejuvenation treatment. The determinations included density, bulk density, water sensitivity, stiffness modulus, resistance to fatigue and low temperature performance determined with the Thermal Stress Restrained Specimen Test (TSRST). The tests of RAP treated with oleyl alcohol ethoxylate phosphate ester carried out as part of this research showed improvement of the performance properties of aged bituminous binders, and thus confirmed the suitability of this agent in road paving applications.

KEYWORDS: reclaimed asphalt pavement (RAP), bitumen, rejuvenator, stiffness modulus, fatigue, Thermal Stress Restrained Specimen Test (TSRST).

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1. INTRODUCTION

Bituminous pavements are exposed to various operational and environmental loads, resulting in different types of distress that appear during the pavement service life. Ageing of the bituminous binder that compromises fatigue cracking or low-temperature cracking resistance of the bituminous layer or causes local ravelling [1–9] (Fig. 1) can be named as one of the causes of this pavement distress. The ageing process involves the action of atmospheric oxygen, evaporation of lighter fractions during production or their absorption in the pores of the aggregate and thixotropic hardening, resulting in increased hardness and brittleness of bituminous binders [10–12]. Chemical reactions between oxygen and the bitumen components and UV radiation are deemed to play the most important role in the above-mentioned process [13–17]. The oxidation rate is the highest during the mixture production, specifically when the binder is mixed with the aggregate [18, 19]. A high production temperature, thin coat of the binder developing on the surface of aggregate which is saturated with heavy metal salts and a high supply of oxygen are the factors that have a bearing on the structure of bitumen [20]. The most prominent changes concern naphthalene aromatics, which become converted to resins and the resins are, in turn, converted to asphaltenes [7, 18, 21, 22]. The properties of bitumen change as a result, which includes increase in viscosity, stiffness, softening and breaking points and a decrease in ductility, phase angle and stress relaxation rate [23–26]. These changes, in turn, lead to pavement distress and the resulting need to remove old (aged) bituminous layers.

Use of RAP i.e. locally obtained milled asphalt having strictly controlled properties should be an option of choice for any road administrator. The amount of RAP added to the fresh mix should be specified depending

on the expected final properties of the mix, the RAP properties and the recycling method used at the Asphalt Mixing Plant (AMP). In Poland the amount of RAP added to fresh mixes during production is in the range of 10–20%, i.e. small. The underlying cause is the method of adding RAP to the mix used in Poland, in which cold RAP is added directly to the mixing plant. This proportion may well be increased (up to as much as 50%), which, however, would necessitate addition to the process of the so-called black drum for drying and pre-heating of the RAP. However, double drum (or barrel) asphalt mixing technology is, as yet, unavailable in Poland [23–25].

A prerequisite to increasing the RAP proportion in fresh asphalt mixes is the requirement to improve the properties of the old bitumen it contains [26–28]. To this end, rejuvenating agents or rejuvenators are added in the process [29–33]. These substances are abundant in oily fractions (saturated and unsaturated fatty acids), naphthalene-aromatics, resinous fractions that may be present alone or in any combination [34–36]. They are added to dried and pre-heated RAP (generally premixed with fresh bitumen) in order to reinstate the dispersed phase content by supplementing the composition with maltenes [37, 38]. This allows to restore the original sol-gel structure, desired in paving applications, to aged bitumen that often have taken gel character to a great extent. An important issue from this point of view is to effectively combine the fresh and the old bitumen into one binder [39–42]. This effectiveness will be reflected, for example, in the low-temperature performance of the mix. As an additional benefit, new generation rejuvenators improve aggregate-binder adhesion, thus increasing water and frost resistance of the final mix containing RAP [25, 29, 32, 43–45].



Fig. 1. A bituminous pavement affected by cracking and ravelling

2. MATERIALS USED IN THE STUDY

2.1. THE BITUMEN

The effect of the rejuvenator addition on the bituminous binder properties was checked on the basis of 50/70 pengrade bitumen manufactured by one of the Polish refineries. The following series of specimens were subjected to the tests:

- not aged (controls),
- RTFOT+PAV aged,
- RTFOT+PAV aged and rejuvenated by adding 1%, 2%, 3%, 4% and 5% of the rejuvenator used.

2.2. THE REJUVENATOR

Oleyl alcohol ethoxylate phosphate ester was used as the rejuvenator in preparation of the above-mentioned specimens. Its structural formula is represented in Fig. 2. It is an oily substance having min. viscosity of 19 mm²/s at 40°C, consisting predominantly of hydrocarbons having carbon number in the range of C20 to C50.

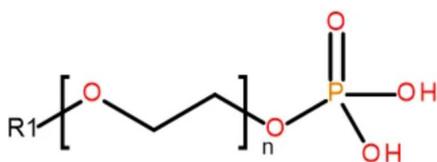


Fig. 2. Structural formula of oleyl alcohol ethoxylate phosphate ester [46]

Its function was to effectively combine the old and fresh bitumen, while restoring the rheological properties of the old binder contained in RAP. This rejuvenator is compatible with most bitumen, including bitumen modified with polymers (SBS, SBR, EVA), ground tyre rubber (GTR), polyphosphoric acid (PPA) and recycled asphalt shingles (RAS).

Its influence on the bitumen properties was checked on 50/70 bitumen samples subjected to RTFOT+PAV ageing. It was added to the bitumen at a rate between 1% and 5%, increased in 1% increments.

2.3. BINDER COURSE ASPHALT CONCRETE

The mixture that was used in this study to check the effectiveness of the analysed rejuvenator was a plant-mixed asphalt concrete (AC 16 W 50/70) intended for laying the binder course on a KR3-4 traffic load duty class road located in Poland. The properties of the analysed bituminous mixtures are given in Table 1 and shown in Fig. 3.

The AC 16 W 50/70 mix was subjected to short-term (STOA) and long-term (LTOA+) ageing. During short-term ageing a loose mixture sample was heated for two hours at 135°C in a forced air drying oven (without delay after sampling at the chosen AMP). Also long-term ageing was carried out on a loose mixture sample. The standard LTOA procedure, in which compacted specimens are heated for 5 days at 85°C, was considered inappropriate for our study purposes, as the small changes in the binder structure it produces may be considered representative of 2–3 year ageing in normal service conditions [55–57]. Then the mix, still loose, was re-heated at 135°C for another 8 hours. This ageing procedure has been found to be more effective [56, 58, 59].

Table 1. Properties of AC 16 W 50/70 mix specified for KR3-4 medium traffic load duty class (based on type testing)

Property	Test method	Unit	Value
Total binder content B	–	%	4.2
Aggregate blend density r_a	PN-EN 1097-6 [47] PN-EN 1097-7 [48] WT-2:2014 [49]	Mg/m ³	2.884
Mix bulk density r_b	PN-EN 12697-6 [50]	Mg/m ³	2.526
Mix density r_{mh}	PN-EN 12697-5 [51]	Mg/m ³	2.666
Void content in the mix VMA	PN-EN 12697-8 [52]	%	5.3
Voids filled with bitumen VFB	PN-EN 12697-8 [52]	%	66.2
Void content in the mix V	PN-EN 12697-8 [52]	%	15.6
Water resistance ($ITSR$)	PN-EN 12697-12 [53]	%	94
Rutting resistance: – Proportional Rut Depth PRD_{air} – wheel tracking slope WTS_{AIR}	PN-EN 12697-22 [54]	% mm/10 ³ cycles	3.3 0.05

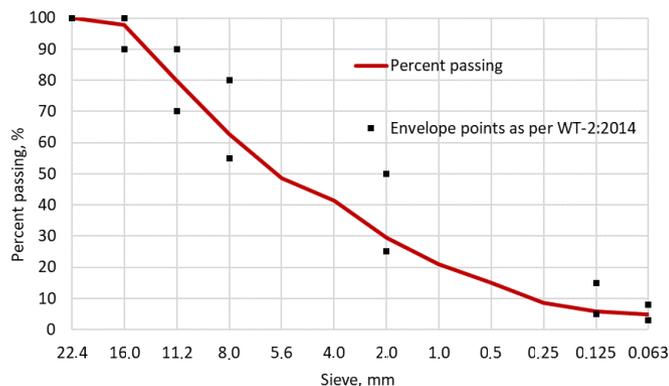


Fig. 3. Aggregate grading curve (AC 16 50/70 specified for KR3-4 medium traffic load duty class)

The tests were carried out on the analysed bituminous mix after STOA ageing (control), after long-term ageing (LTOA+) and after rejuvenating treatment done after STOA<OA+ ageing).

First, the rejuvenator content was optimized based on the stiffness modulus values obtained in the IT-CY test. The rejuvenator was added directly to the mix at the rates of 0.1%, 0.2% and 0.3%. The optimum proportion was 0.2% and this amount of rejuvenator was applied in further part of this research.

3. TEST METHODS

3.1. TESTING OF THE BITUMINOUS BINDERS

The analysed bituminous binders were subjected to specific tests to determine both their basic properties (penetration, softening point) and rheological properties (fatigue cracking critical temperature FCCT determined in the dynamic shear rheometer).

3.1.1. Penetration and softening point

Bitumen penetration was tested as per PN-EN 1426 [60] and the softening point was determined using the ring and ball (R&B) method as per PN-EN 1427 [61]. These two values were used next to calculate the IP penetration index (PN-EN 12591) using equations (1) and (2) [62].

$$A = \frac{\log 800 - \log Pen_{25}}{T_{PIK} - 25}, \quad (1)$$

$$IP = \frac{20 - 500 \cdot A}{1 + 50 \cdot A}, \quad (2)$$

where:

- A – temperature sensitivity,
- Pen_{25} – binder penetration @ 25°C, 0.1 mm,
- T_{PIK} – binder softening point, °C,
- IP – penetration index.

The value of IP allows determining changes of the rheological properties of bituminous binders caused by the changes in their structure.

The fatigue critical temperature FCCT was calculated using the dynamic shear modulus G^* value and phase angle d determined with DSR rheometer as per AASHTO T315 [63]. The tests were carried out under sinusoidal cyclic loading at a 10 rad/sec. loading frequency at four test temperature: 5°C, 20°C, 25°C and 30°C. The objective was to verify, based on the obtained values, the effect of the ageing process and the used rejuvenator on the fatigue cracking resistance of the binder (FCCT at $G^* \cdot \sin \delta = 5,000$ kPa).

In addition, fractional compositions of the bitumen were determined in the SARA test and Fourier-transform infrared spectroscopy (FTIR) analyses were also carried out.

3.1.2. SARA test

The fractional compositions of the analysed bitumen were determined using the Thin Layer Chromatography method using Flame Ionization Detector (TLC/FID method). The following set of solvents (eluent) was used to break down the binders into fractions:

- heptane (C7H16),
- toluene (C7H8) ÷ heptane (C7H16) (80%÷20%),
- dichloromethane (CH2Cl2) ÷ methanol (CH3OH) (95%÷5%).

These solvents were used in the order of the increasing eluting strength. After elution, the frame holding the chromarods was placed in the IATROSCAN MK-6 analyser, equipped with the flame ionization detector (FID) [64]. Ten determinations were done for each specimen series.

3.1.3. FTIR spectroscopy analysis

Mid-IR analysis was carried out using transmission spectroscopy method. The spectra were generated for 1% bitumen solution using tetrahydrofuran (THF) as the solvent. Five determinations were done for each specimen. Baseline correction was carried out for the obtained spectra, in line with the RILEM's guidelines developed as part of the TC-272-PIM-TG1 research project [65].

The FTIR spectra were generated for the prepared virgin binder specimens (Sec. 2.1) and for RAP binder reclaimed from the AC 16 W 50/70 mix (from the specimens used to determine the IT-CY stiffness module).

In addition, surface areas were determined for characteristic bands of standardised spectra: $1,700\text{ cm}^{-1}$ (carbonyl bands) and $1,030\text{ cm}^{-1}$ (sulphoxide bands) and $1,460\text{ cm}^{-1}$ and $1,375\text{ cm}^{-1}$ (control bands), which do not change in intensity during ageing. The surface areas under the respective bands (e.g. A_{1700} , A_{1030} , etc.) were calculated by numerical integration, using the trapezoidal rule. The relevant integration limits for the chosen bands were determined by finding the local minima. These surface areas were, in turn, used to determine the I_{csa} values, calculated as the ratio between the total surface areas of the characteristic (i.e. sulfoxide and carbonyl) bands and the control bands. I_{csa} values were calculated as follows:

$$I_{csa} = I_{csr} = \frac{A_{1700} + A_{1030}}{A_{1460} + A_{1375}}. \quad (3)$$

The ageing index I_a values were obtained by comparing the obtained I_{csa} values with the control bitumen index value I_{csr} (3). The index value was calculated as follows:

$$I_a = \frac{I_{csa}}{I_{csr}}. \quad (4)$$

3.2. TESTING OF BITUMINOUS MIXES

The analysed bituminous mixes were tested in two stages. The first stage included determination of their basic physical parameters (density, bulk density, void content) and finding an optimum amount of rejuvenator, based on the stiffness modulus value determined in the IT-CY test. The purpose of the second stage experiments was to determine the optimum rejuvenator application rate. To this end, the following tests were carried out: ITSR – to determine water sensitivity, resistance to fatigue test and TSRST test – to determine low-temperature performance of the mix under analysis.

3.2.1. Physical parameters of the analysed bituminous mixes

The mix densities ρ_{mh} were determined using the volumetric procedure A, as per PN-EN 12697-5 [51]. Bulk density r_b was, in turn, determined using procedure B – SSD, as per PN-EN 12697-6 [50] and void content was determined as per PN-EN 12697-8 [52]. The specimens were compacted using Marshall compactor hammer, with 75 blows applied on each specimen side.

3.2.2. IT-CY stiffness modulus as per PN-EN 12697-26 [66]

Stiffness modulus was determined by applying indirect tension test on 100 mm dia. by 60 ± 2 mm high cylindrical specimens made in laboratory using the Marshall method as per PN-EN 12697-30 [67]. The test temperature was 10°C and cyclic vertical load was applied axially on the specimens (Fig. 4). The horizontal displacement applied on the sample was 5mm (in two perpendicular planes), load increment time was 120 ± 4 ms and loading cycle duration was 3 sec. The test result was a mean of five load pulses.



Fig. 4. Stiffness modulus determination with IT-CY test on AC 16 W 50/70 specimens

3.2.3. Water sensitivity

Water sensitivity of the mixes was checked using the ITSR procedure described in WT-2:2014, Appendix 1 [49]. In this test, the indirect tensile strength is tested under static load at $25 \pm 2^\circ\text{C}$ test temperature on dry and wet samples (ITS_d and ITS_w respectively). The ITSR value itself is calculated as follows:

$$ITSR = \frac{ITS_w}{ITS_s} \cdot 100. \quad (5)$$

3.2.4. Resistance to fatigue as per PN-EN 12697-24 [68]

The resistance to fatigue of the analysed mixes was tested on $50 \times 60 \times 400$ mm prismatic specimens at 10°C test temperature (Fig. 5). The resistance to fatigue values were determined for three different strain levels of 110, 140 and

170 mm/m at 10 Hz test frequency. The initial stiffness module was determined in the 100th load cycle. The fatigue criterion used in this study was the stress and strain state at which the stiffness modulus reached 50% of the initial value.



Fig. 5. 4PB-PR resistance to fatigue test of the AC 16-W-50/70 mix

3.2.5. TSRST test

In this test the specimens are gradually cooled at a rate of 10°C/h during which the specimens are restrained to prevent shrinkage (i.e. the strain equals zero) – Fig. 6. The temperature decrease rate has a critical bearing on the test output [69, 70]. Higher temperature increase gradients produce faster growth of induced stress. The initial test temperature was 20°C. The logged parameters included stress increase over time, the minimum temperature and stress at failure ($T_{failure}$ and $s_{cry, failure}$). The test conditions are shown in Fig. 7 below.

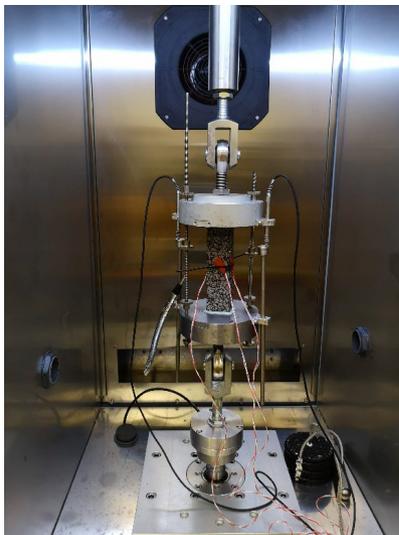


Fig. 6. TSRST test to determine low-temperature performance of AC 16 W 50/70 mix

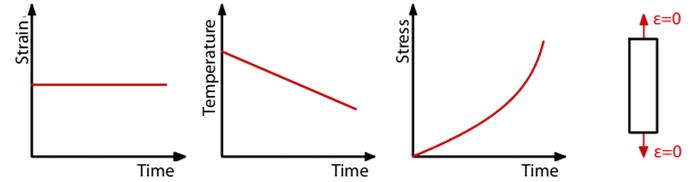


Fig. 7. TSRST results

4. PRESENTATION AND ANALYSIS OF THE TEST RESULTS

4.1. BITUMINOUS BINDERS TEST RESULTS

4.1.1. Results of basic tests

The basic properties of bitumen, including penetration (Pen) and R&B softening point are shown in Fig. 8. As a result of RTFOT+PAV ageing, the properties of 50/70 have changed considerably, coming close to the properties of the RAP binder under analysis. These changes included a drop of penetration by 36×0.1 mm and increase of the softening point by 11.5°C. Then the rejuvenator was added, again causing considerable softening of the previously aged binder 5% proportion of rejuvenator decreased the penetration value by 4.4×0.1 mm and increased the softening point by 1.9°C. These values are close to control bitumen characteristics.

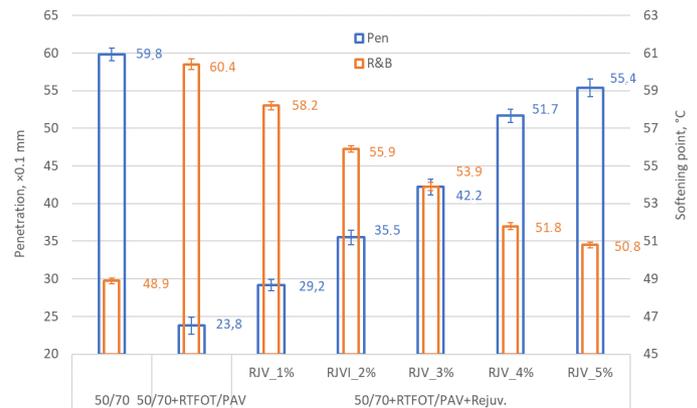


Fig. 8. Penetration and softening point of 50/70 bitumen used as a control, after RTFOT+PAV ageing, followed by rejuvenating treatment with the chosen rejuvenator

The bituminous binder penetration index was determined on the basis of the obtained penetration and softening point values (Fig. 9). The lowest IP (-1.1) value was obtained for the control bitumen, which increased to -0.5 as a result of ageing. The addition of 5% of rejuvenator

decreased the IP value to -0.8. This may be the effect of oily fractions (including saturated and aromatic hydrocarbons) introduced into the aged bitumen composition, noting that this does not bring back the original situation in this respect. That said, a lower IP value indicates a lesser temperature sensitivity of the rejuvenated bitumen, as compared to the control.

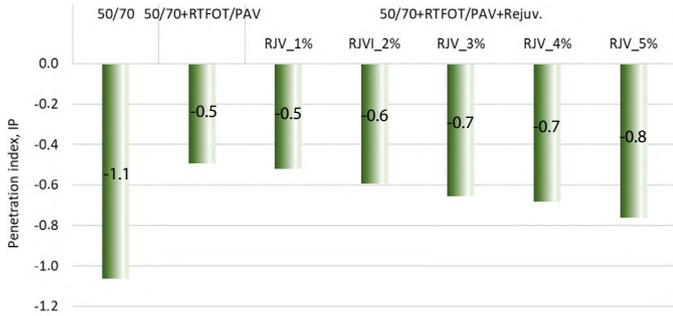


Fig. 9. IP value of 50/70 control bitumen, after RTFOT+PAV ageing, followed by rejuvenating treatment

4.1.2. Rheological tests

The values of dynamic shear modulus G^* and shift angle δ obtained in testing of the analysed bituminous binders are given in Fig. 10 and Fig. 11. Figure 12 gives the obtained fatigue critical cracking temperatures (FCCT), i.e. the temperatures at which $G^* \cdot \sin\delta = 5,000$ kPa.

The ageing process (RTFOT+PAV) increases the dynamic shear modulus G^* and decreases the phase angle δ . FCCT increases by over 4°C as a result. The rejuvenating treatment, in turn, improves the bitumen performance by decreasing G^* , increasing δ and decreasing FCCT. A decrease of FCCT, as compared

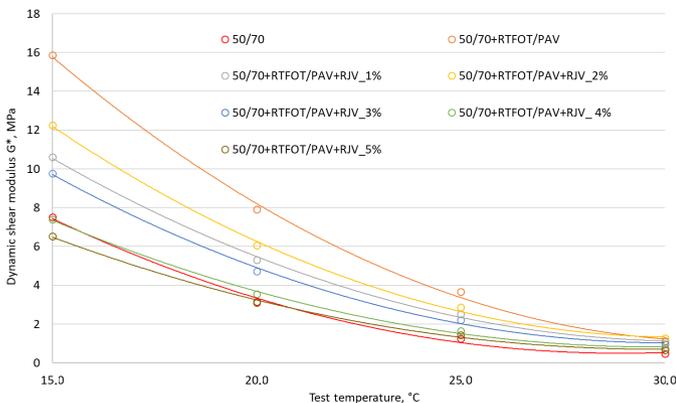


Fig. 10. Dynamic shear modulus measured in DSR for the analysed 50/70 bitumen: control, after RTFOT+PAV ageing and after rejuvenating treatment

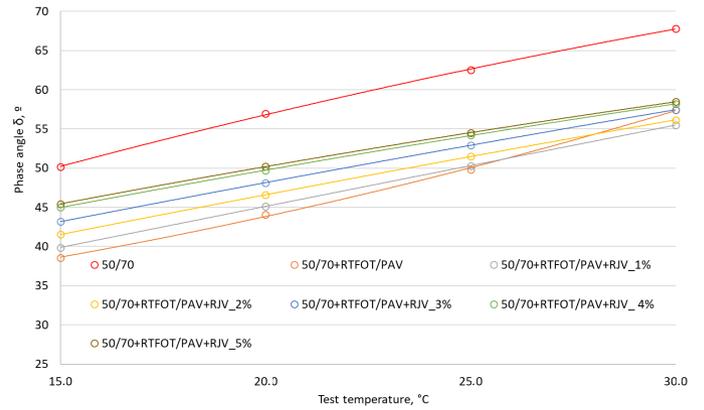


Fig. 11. Phase angle measured in DSR for the analysed 50/70 bitumen: control, after RTFOT+PAV ageing and after rejuvenating treatment

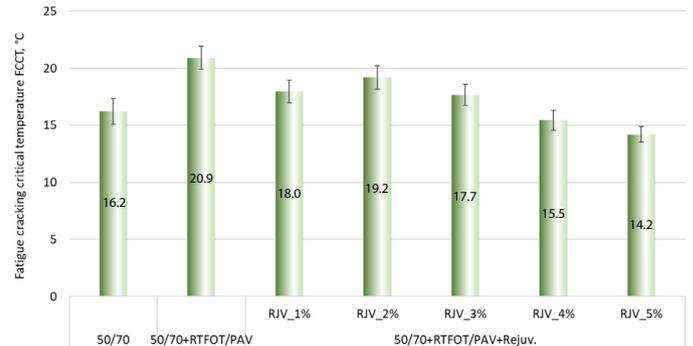


Fig. 12. Fatigue cracking critical temperature FCCT for the analysed 50/70 bitumen: control, after RTFOT+PAV ageing and after rejuvenating treatment

to the control, was noted already at 4% rejuvenator application rate. On the other hand, the rejuvenating treatment did not improve the phase angle δ , up the control values, even when added at 5% application rate. The difference in this value remained at ca. 5°C and 10°C at the test temperatures of 15°C and 30°C respectively.

4.1.3. SARA analysis

The fractional composition of the analysed bitumen was determined with thin-layer chromatography, using flame ionization detector (TLC-FID analysis). The results are shown in Fig. 13.

The original fractional composition of the analysed bitumen has changed due to high temperature and access of air, i.e. in the process of ageing. The most pronounced changes after RTFOT+PAV were observed in the content

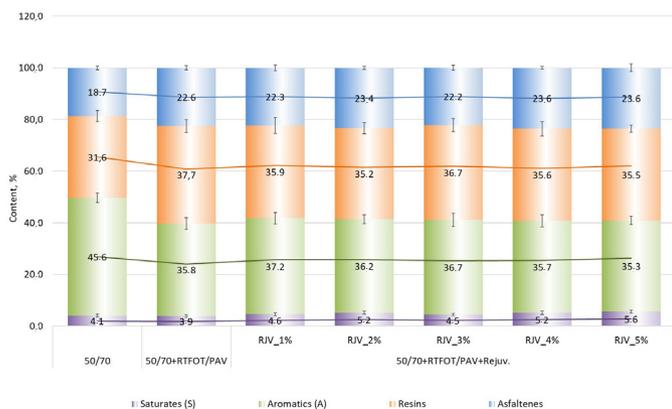


Fig. 13. Fractional composition of 50/70 control bitumen, after RTFOT+PAV ageing and after rejuvenating treatment

of aromatic hydrocarbons, resins and asphaltenes. The aromatics content decreased by about 10%, while the proportions of resins and asphaltenes increased by ca. 6% and ca. 4%, respectively. Rejuvenation brought only small changes in this respect, relating mainly to the content of saturated hydrocarbons (*S*) and resins (*R*). Adding rejuvenator at 5% application rate increased the *S* content by ca. 1.7%, at the same time decreasing the *R* content by ca. 2.2%.

SARA analysis was carried out also for reclaimed (RAP) bitumen prepared for stiffness modulus determination. The results of fractional composition analysis are represented in Fig. 14 below.

The greatest fractional composition changes concerned the aromatics, saturated hydrocarbon and asphaltene fractions. After STOA/LTOA+ the content of aromatics decreased by ca. 10%, while the content of resins

and asphaltenes increased by ca. 8.6% and ca. 2.1%, respectively. The rejuvenating treatment had no apparent effect on the fractional composition of the analysed bitumen. A higher, i.e. 6% application rate increased the saturated hydrocarbons content by ca. 2%, decreased the amount of resins by ca. 4% and increased the amount of asphaltenes by ca. 1.8%.

The TLC-FID results, while providing information on the fraction composition of bitumen do not allow a reliable assessment of the rejuvenating treatment effectiveness.

4.1.4. FTIR spectroscopy analysis

The output of the mid-range IR analysis of the analysed bitumen using the transmission method (FTIR) is shown in Fig. 15. Figure 16, in turn, shows the spectra of the bitumen extracted from the mixes (RAP bitumen) and the control. They show the range where the greatest changes were observed, i.e. the carbonyl and sulphoxide bands ($1,700\text{ cm}^{-1}$ and $1,030\text{ cm}^{-1}$, respectively).

Note that lab aged binders (RTFOT/PAV) feature considerably smaller spectral changes (in 1700 cm^{-1} and $1,030\text{ cm}^{-1}$ bands, as compared to the same bitumen extracted from the mix). Apparently, laboratory ageing falls short of simulating the processes taking place in bituminous mixes in reality. This observation is supported by the ageing index values calculated with equations (3) and (4), as represented in Fig. 17 and Fig. 18 below.

As shown in Fig. 17, the ageing degree achieved by lab ageing of 50/70 bitumen (RTFOT+PAV) reached the value of 2.2 (value of ageing index I_a). The added rejuvenator failed to noticeably lower this value. As a matter of fact, the I_a values did actually increase, thus giving no indication of the rejuvenating treatment effectiveness.

On the other hand, for RAP binders the changes in I_a appear adequate. LTOA+ ageing increased the I_a value of 3.1 after STOA up to 7.9. Next, rejuvenator was added at a rate of 0.3% of the mix weight and the I_a value decreased back to 5.0 as a result.

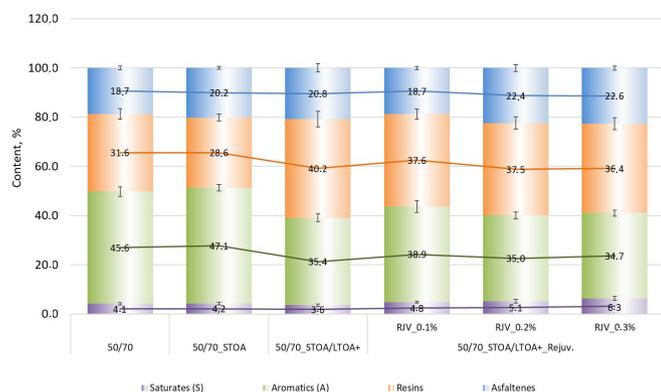


Fig. 14. Fractional compositions of 50/70 bitumen and of the bituminous binders extracted from the samples prepared for the IT-CY test

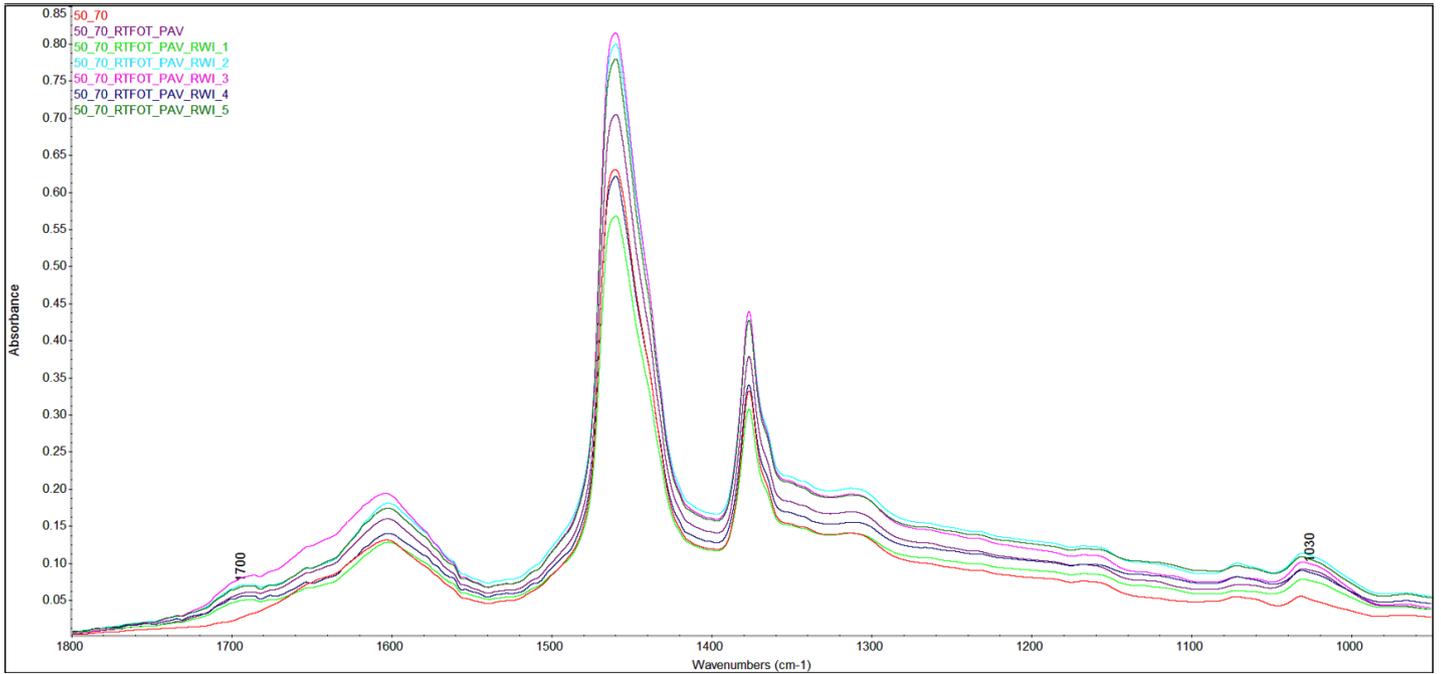


Fig. 15. Spectra of 50/70 bitumen: control, after RTFO+PAV ageing and after rejuvenating treatment

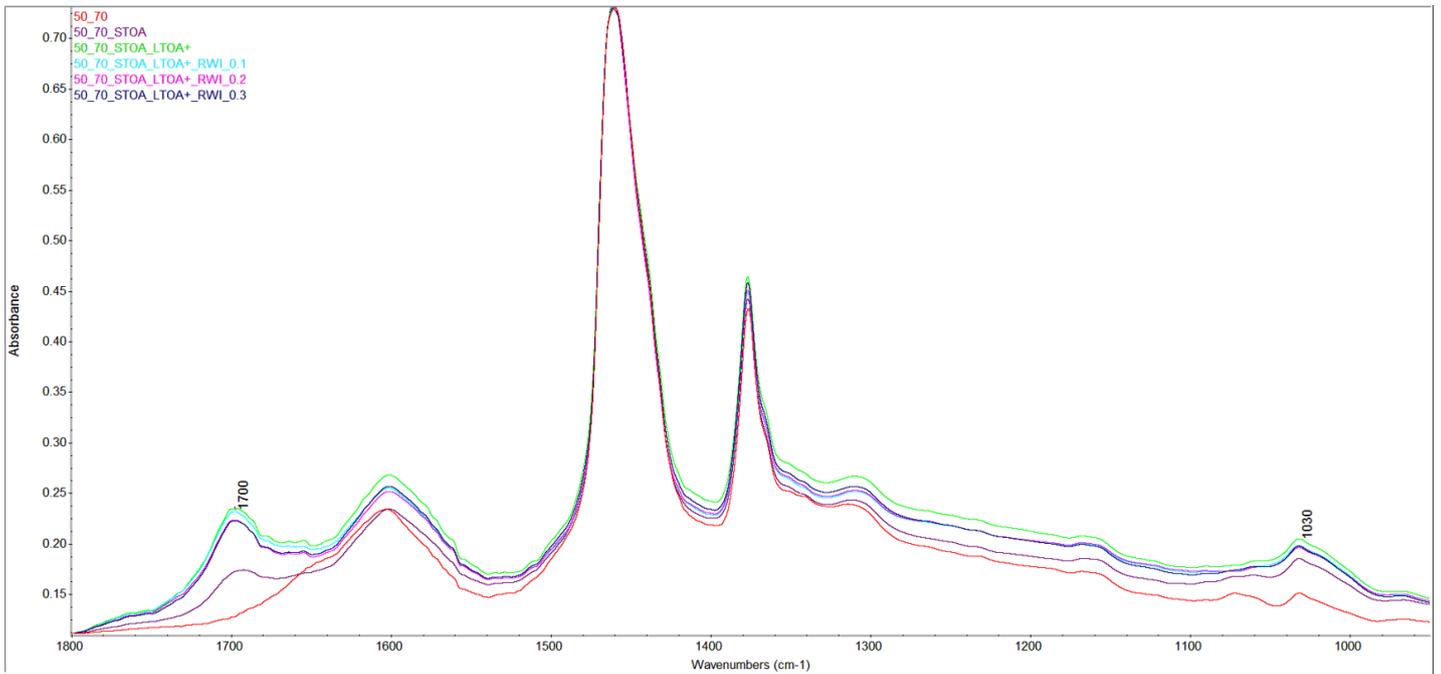


Fig. 16. Spectra of 50/70 bitumen: control and extracted from mixes

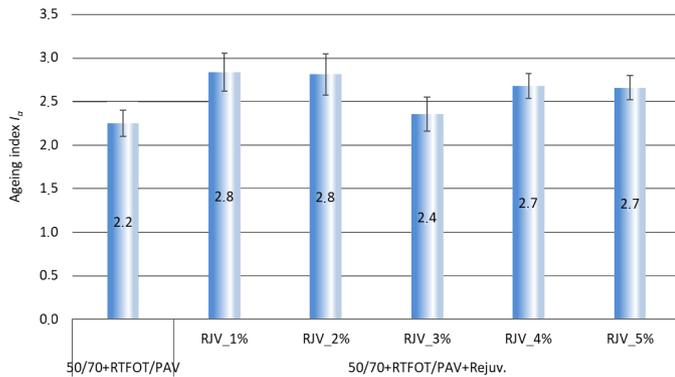


Fig. 17. 50/70 bitumen ageing index: control, after RTFOT+PAV ageing and after rejuvenating treatment

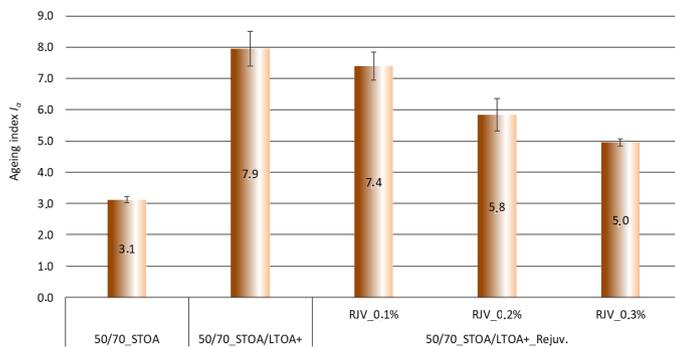


Fig. 18. 50/70 bitumen ageing index: control and extracted from RAP

4.2. BITUMINOUS MIX TEST RESULTS

4.2.1. Physical properties

The physical properties determined as part of this research included density, bulk density and void content. Bulk density was determined on the specimens prepared for IT-CY tests (stiffness modulus determination). The obtained bulk densities are presented in Fig. 19 and in Fig. 20.

Long-term ageing increased the air voids by 2.1%, as compared to the control one. The rejuvenating treatment improved the compaction capacity, as shown by a 0.5% (i.e. slight) increase in the void content after adding the rejuvenator at a 0.3% rate.

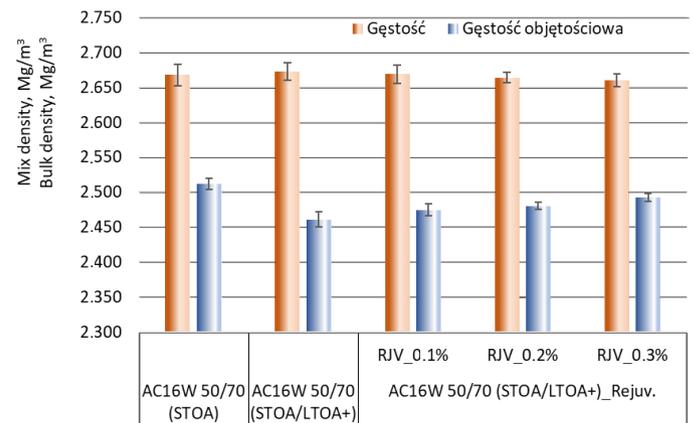


Fig. 19. Density and bulk density of AC 16 W 50/70 mix: control (after STOA), after long-term ageing (STOA<OA+) and after rejuvenating treatment

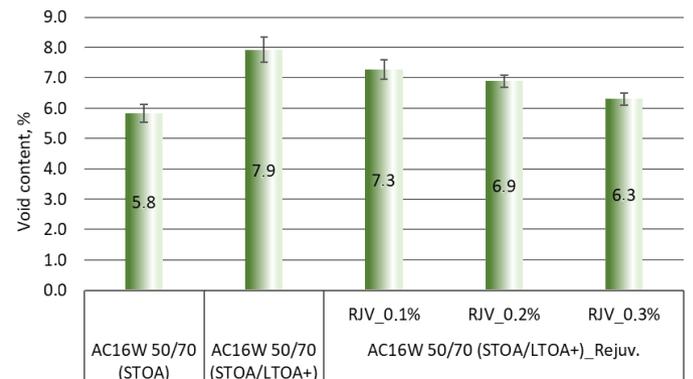


Fig. 20. Void content in AC 16 W 50/70 mix: control (after STOA), after long-term ageing (STOA<OA+) and after rejuvenating treatment

4.2.2. IT-CY test

The purpose of IT-CY test was to find the rejuvenator application rate at which the mix after STOA<OA+ ageing would have the stiffness modulus closest to the control one, i.e. the mix subjected to short-term ageing only. The test results are represented in Fig. 21 below. The rejuvenator was added at three rates: 0.1%, 0.2% and 0.3% of the mix weight.

The LTOA+ ageing process increased the mix stiffness, as evidenced by the increase in IT-CY values by over 2,300 MPa. The rejuvenating treatment, in turn, reduced the increased stiffness bringing it close to the control, when the rejuvenator was added at 0.2% rate.

Therefore, 0.2% application rate was applied from this point on.

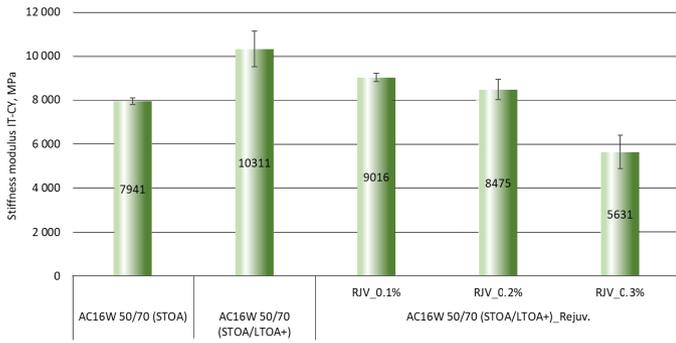


Fig. 21. Stiffness modulus of AC 16 W 50/70 mix after STOA ageing (control), after STOA<OA+ ageing and after rejuvenating treatment

4.2.3. ITSR test to check water sensitivity

The tensile strength values obtained in the ITS test on dry (ITS_D) and wet specimens (ITS_W) and ITSR values are given in Fig. 22 below.

The test results showed that LTOA+ process was effective in causing severe ageing, as evidenced by increased stiffness and much higher water sensitivity indicated by lower value of ITSR (74%). Rejuvenating treatment decreased the stiffness and improved water sensitivity (ITSR = 81%) yet the application rate of 0.2% was found to be too low.

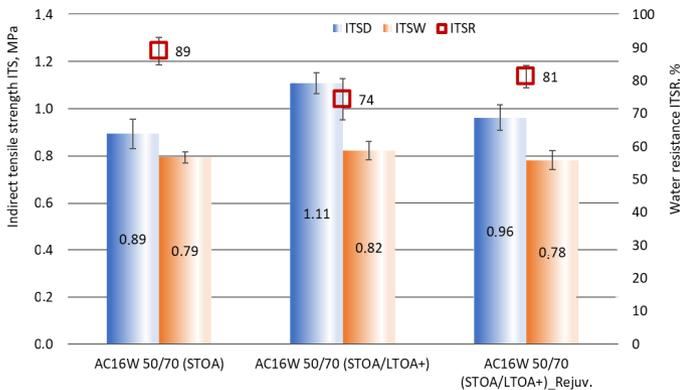


Fig. 22. Water resistance of AC 16 W 50/70 mix after STOA ageing (control), after STOA<OA+ ageing and after rejuvenating treatment

4.2.4. 4PB-PR resistance to fatigue test

The test results (Fig. 23) show that the applied ageing process had a severe impact on the resistance to fatigue of the mix under analysis. This decreased resistance to fatigue was improved by the rejuvenating treatment (application rate: 0.2%), yet the achieved improvement

is considered insufficient. The obtained strain value after 1 million load cycles was 130 $\mu\text{m}/\text{m}$ for the control mix, which decreased by ca. 15 $\mu\text{m}/\text{m}$ after the rejuvenating treatment.

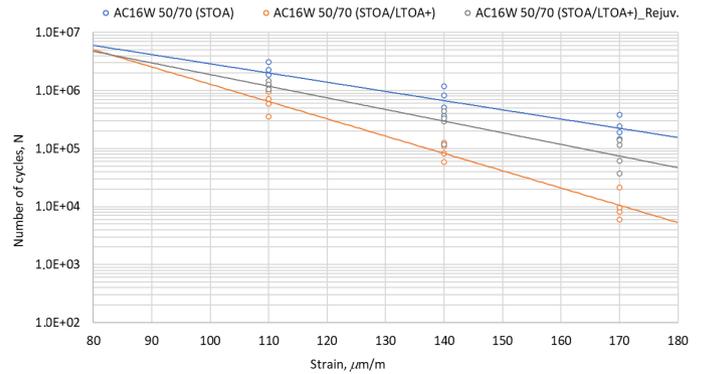


Fig. 23. Resistance to fatigue of AC 16 W 50/70 mix after STOA (control), after STOA<OA+ and after rejuvenating treatment

4.2.5. Low temperature performance properties determined by TSRST test

The low-temperature performance properties of the analysed AC 16 W 50/70 mix are presented in Fig. 24 and Fig. 25.

After the LTOA+ ageing process the failure temperature increased by 6.8°C to -16.9°C, as compared to the control. Next 0.2% of rejuvenator was added, decreasing $T_{failure}$ to -21.0°C. To obtain the failure temperature obtained on the control mix, the above-mentioned application rate should be increased accordingly.

A similar pattern was observed for the maximum measured stress level. The lowest value of 4.07 MPa was obtained for the control one, long-term ageing increased the stress level to 5.97 MPa, and the rejuvenating treatment effectively reduced this value back to 4.90 MPa.

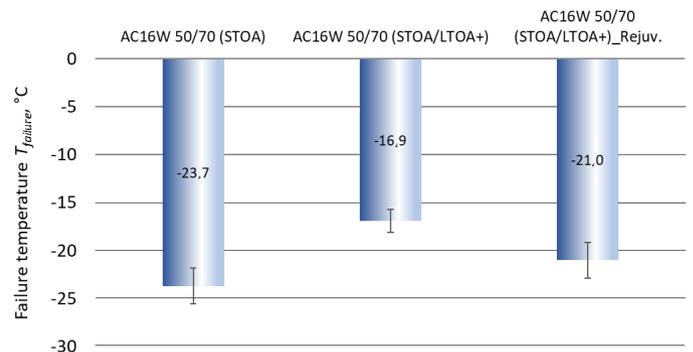


Fig. 24. TSRST failure temperature of AC 16 W 50/70 mix after STOA ageing (control), after STOA<OA+ ageing and after rejuvenating treatment

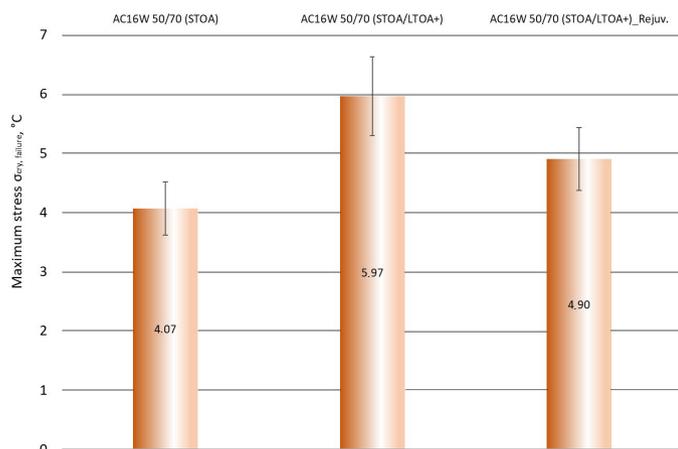


Fig. 25. TSRST maximum stress level of AC 16 W 50/70 mix after STOA ageing (control), after STOA<OA+ ageing and after rejuvenating treatment

5. CONCLUSIONS

Closed-loop economy requires undertaking steps to eliminate or control the amount of generated waste and eliminate or mitigate any environmental impacts of such waste from production, through the useful life and after it has come to an end. The technologies allowing to reuse old asphalt pavements, on equal terms with new paving materials, are very much in line with this policy.

However, one must bear in mind that the performance properties of old pavements have deteriorated over time. The main cause of this deterioration is ageing of bitumen. For successful reuse in large quantities it is, therefore, necessary to restore these lost properties up to the level offered by fresh paving grade bitumen. Special chemical agents, based on esters, oils or imidazolines, are used for this purpose.

This research project, and specifically the results of tests carried out on both the bitumen and bituminous mixes, demonstrated the effectiveness of oleyl alcohol ethoxylate phosphate ester as a bitumen rejuvenating agent. The most important findings of this study are listed below:

1. Addition of min. 5% of the analysed rejuvenator restored the basic performance properties (Pen, R&B) of laboratory aged bitumen. Rejuvenated bitumen is less sensitive to temperature changes than the control one (Fig. 9).
2. The analysed rejuvenator mitigated the impact of ageing (RTFOT/PAV) on the dynamic shear

modulus G^* (Fig. 10) and fatigue cracking critical temperature FCCT (Fig. 12) by bringing these values to the control mix levels. The phase angle δ increased with the increasing rejuvenator application rate, yet the control mix level was not achieved even at the maximum, i.e. 5% application rate (Fig. 11).

3. SARA analysis allows tracking of changes to the fractional composition due to ageing. However, it did not provide a satisfactory assessment of the rejuvenating treatment effectiveness (Fig. 13–14).
4. The effect of rejuvenating treatment was evident in the FTIR spectra but only for RAP bitumen (Fig. 18).
5. LTOA+ lab ageing affected the mix compaction capacity, increasing the void content by 2.1% (Fig. 19 and Fig. 20). Rejuvenating treatment improved this property decreasing the void content by 1.6% (as compared to the control one i.e. the mix after LTOA+ ageing) for 0.3% rejuvenator application rate.
6. Long-term ageing increased the stiffness modulus up to 10,311 MPa i.e. by ca. 2,300 MPa, as compared to the control one. Rejuvenating treatment decreased the stiffness modulus, which has come close to the control one when the rejuvenator was added at 0.2% application rate (Fig. 21).
7. The mix was found to substantially lose its original water resistance as a result of LTOA+ ageing (to 74%) and regained it slightly (yet still below the value obtained on the control one) after addition of 0.2% of the rejuvenator (Fig. 22).
8. Rejuvenating treatment was found to improve the resistance to fatigue of the mix previously subjected to LTOA+ ageing, yet still below the control mix performance in this respect.
9. Ageing affects the resistance to fatigue (Fig. 23) and low temperatures (Fig. 24). These desired properties may be recovered by rejuvenating treatment, yet to reach the control mix performance at least 0.2% of the rejuvenator must be added.

Summing up, we can conclude that both short- and long-term ageing affect the desired performance properties of bituminous mixes. Rejuvenating treatment may effectively reinstate these properties, yet several tests are indispensable to investigate the properties of both the mix and the bitumen it contains.

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