# RHEOLOGICAL PROPERTIES OF THE BITUMINOUS BINDER EXTRACTED FROM SMA PAVEMENT WITH HYDRATED LIME

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**Abstract.** The durability of pavement layers depends on the type of bitumen and changes in its material structure during service life. In 1999, while rebuilding and modernizing road infrastructure in Kielce (Poland), a stone mastic asphalt wearing coarse layer with hydrated lime was placed on one of the town's main streets. Stone mastic asphalt mixture contained 6.2% of D70 bitumen (currently 50/70) and 4% SBS polymer under the trade name Kraton 1101 CM. The hydrated lime was dosed into the stone mastic asphalt mixture to replace 30% of the filler mass. Pavement surface condition after 12 years of service life was very good. In 2011, bitumen samples were extracted from stone mastic asphalt and tested. The tests were performed on the samples that contained fatty amine and hydrated lime as adhesive agents, obtained from stone mastic asphalt wearing course layer in the rut paths and from between the area limited by rut paths. The hydrated lime additive was found to have a positive effect on rheological properties of the recovered bitumen providing resistance to the water and frost.

**Keyword:** binder rheological parameter, hydrated lime, recovered binder, polymer SBS, stone mastic asphalt pavement.

## 1. Introduction

Asphalt layers of road structure have to be resistant to the impact of heavy traffic loads and climatic factors (Lamontagne *et al.* 2001). Durability of the layers depends on the type of bituminous binder used and its changes during service life (Grabowski, Słowik 2002) as well as the kind of aggregate. During production of the stone mastic asphalt a special attention should be paid to some factors which make contribution to the increase in fuel consumption (Zaumanis *et al.* 2012). Therefore, to increase the reliability of pavement, a proper range of bitumen plastic work has to be ensured as well as a slow-down in the aging process (Gawel *et al.* 2016). Modifiers provide a suitable solution (Judycki, Jaskuła 2001; Zaumanis *et al.* 2012). First attempts to apply hydrated lime in bitumen date back to the early twentieth century (Kennedy 1984). It was used as an adhesive agent for mineral mixtures with a high content of $SiO_2$ to improve water resistance and frost resistance of pavement (Little, Petersen 2005). The addition of hydrated lime was found to reduce the aging process by limiting the increase in bitumen stiffness (Plancher *et al.* 1976). Real life simulation tests performed mainly under laboratory conditions helped to observe how this additive affected the properties of bituminous mixtures (Lesueur, Little 1999). So far,

major research effort has been devoted to discovering the influence hydrated lime has on the neat bitumen properties in the asphalt concrete. Few tests have been designed for the stone mastic asphalt (SMA) mixtures with polymer modified bitumen (Mouillet *et al.* 2008). The interaction between hydrated lime and the modified bitumen (SBS polymer modified bitumen) used in SMA mixtures has to be studied further.

In 1999, during road rebuilding and modernization, a SMA wearing coarse layer with hydrated lime was placed on one of the main streets in Kielce (Poland). The SMA mixture contained 6.2% of D70 bitumen (currently 50/70) and 4% SBS polymer known as Kraton 1101 CM. Hydrated lime was dosed into the SMA mixture to replace 30% of filler mass. The mineral mixture was made of quartzite sandstone containing 95% $SiO_2$. The experiment was designed to examine both the SMA mixture performance and the bituminous binder (D70) properties. Two parameters were controlled: the content of the SBS polymer and the content of hydrated lime (Iwański *et al.* 2012b). The reference SMA layer was placed with the fatty amine additive. The following surface conditions were monitored: evenness, roughness, rutting, raveling, stripping, and cracking. The pavement surface condition after 12 years of

service life was very good. It is noteworthy that the layers with the hydrated lime additive retained better parameters than other layers (Iwański *et al.* 2012b; Shafiei, Latifi Namin 2014).

## 2. The testing methodology for the bitumen rheological properties extracted from SMA pavement

In 2011 tests were performed on the bitumen that was extracted (recovered) from SMA pavement. The bitumen was recovered from two sections that contained hydrated lime (HL) and the reference adhesive additive – fatty acid amine (A). The recovered binders from SMA mixtures were obtained in the area of the wearing course layer from the wheel paths (RD) and from the site unaffected by rutting (from between the wheel paths) (N).

The binder was recovered from the 2 cm thick top surface layer subjected to the most adverse aging process due to the action of solar radiation, de-icing solutions (salt) used in winter, and other climatic factors. In the preparation process, the samples with a mass of 250 g for each level of modification were separated on the basis of their location. Then they were heated up to the temperature of 155 °C and kept at this temperature for 30 minutes. The binder was agitated in a blender at 400 rpm at the constant temperature. Test properties of the bitumen extracted from SMA pavement were the average value of the tested samples distribution and were treated as independent variables. The scope of tested structural properties

of the binder covered the following parameters: softening point temperature, penetration grade at 25 °C, breaking point temperature, zero shear viscosity (ZSV), dynamic viscosity versus temperature, complex modulus $G^*$ with phase angle, creep compliance at low shear stress Jnr 100 (100 Pa) and at high shear stress Jnr 3200 (3200 Pa) and the non-recoverable part of deformation at low (R100) and high (R3200) shear stress.

## 3. Basic rheological properties of the bitumen extracted from SMA pavement

Basic properties of the binder recovered from SMA pavements after 12 years of their service, including penetration grade at 25 °C, softening point temperature, Fraass temperature, and the binder ductility are compared with the reference bitumen (denoted by "C") in Fig. 1.

The test results indicate that the bitumen properties deteriorated during the operation period in comparison to the neat bitumen that had been applied 12 years earlier. However, the intensification of changes in the recovered bitumen properties referred to the type of adhesive agent such as hydrated lime and the fatty amine and varied with the test sites location. Slight changes in the properties of the SMA-recovered bitumen occurred in the wheel path cores. Weaker influence of the aging process on the bitumen properties was observed in the bitumen with hydrated lime. Hydrated lime acted as an antioxidant and slowed down the aging process much more effectively. Only in the
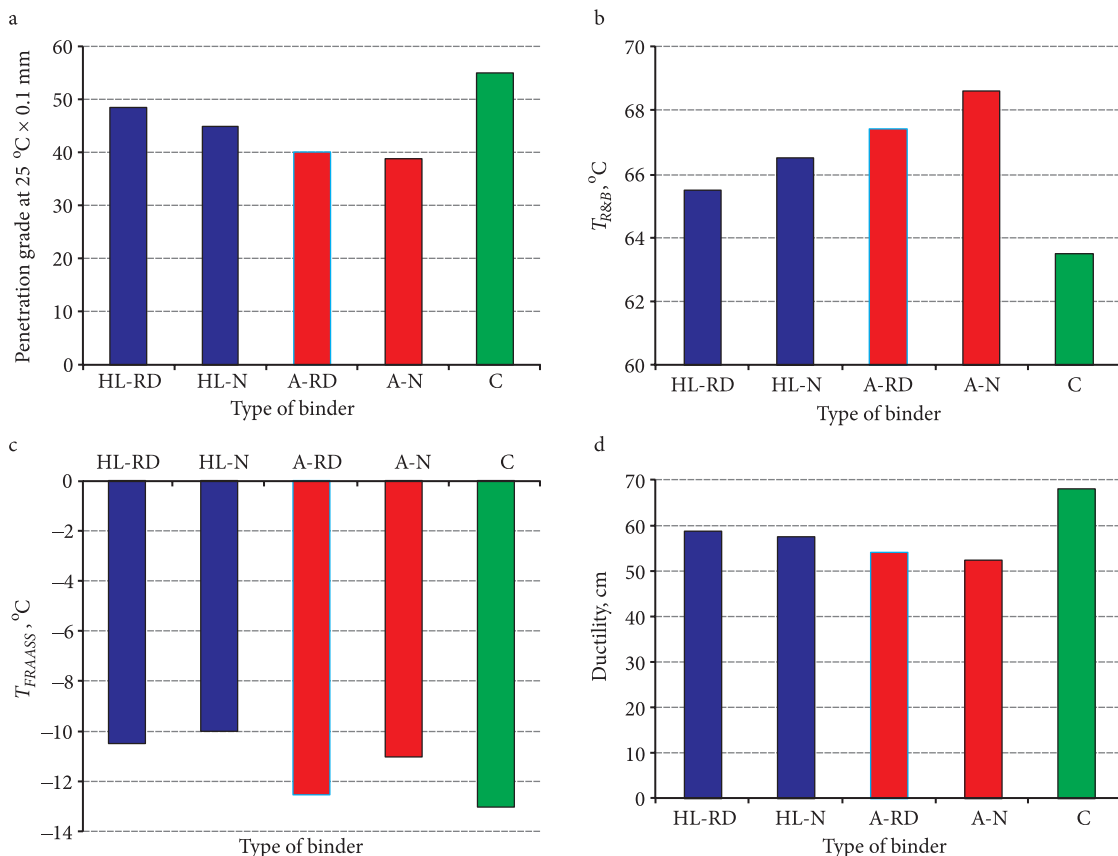


**Fig. 1.** Properties of the bitumen extracted from SMA after 12 years in operation: a – penetration grade at 25 °C; b – softening point temperature; c – breaking point temperature; d – bitumen ductility

case of the breaking point temperature measured according to Fraass test an application of the fatty amine to the bitumen is much more beneficial. For a comprehensive assessment of the penetration grade and the softening point temperature of the recovered bitumen, the dimensionless penetration index was used, $PI = f(T_{R\&B}, Pen)$. The recommended value of this index, according to the U.S. requirements, is in the range from –1.5 to 0.7 (Błażejowski *et al.* 2011). This index range provides a good compromise between the behavior of the bitumen at low temperatures and the bitumen stiffness required in the road surface. The penetration index, which expresses a relation between the softening point temperature and the penetration grade, can be drawn as the following formula (1) (Gawel *et al.* 2001):

$$PI = \frac{20 \cdot T_{R\&B} + 500 \cdot \lg P - 1952}{T_{R\&B} - 50 \cdot \lg P + 120}, \qquad (1)$$

where *PI* – penetration index; $T_{R\&B}$ – softening point temperature, °C; *P* – penetration grade at 25 °C, 0.1mm.

Changes in Penetration Index of the SMA-recovered bitumen are presented in Fig. 2.

It should be noted that the presence of fatty amines liquefies the bitumen and triggers the change towards the rheological kind of the bitumen – the "sol" type. This type of the binder displays excessive sensitivity to the rutting formation in the pavement. In the case of the recovered binder with hydrated lime (HL) the reduction in the susceptibility of SMA was observed. The bitumen taken from between the wheel paths had a less beneficial index penetration value than the samples taken from the area of mix rutting. Regardless of the sampling location, the bitumen with HL had a lower thermal susceptibility than the binder with the fatty amine. Therefore SMA mixture that contains the bitumen modified with hydrated lime will be more resistant to the formation of permanent deformation such as rutting.

It should be concluded that the breaking point temperature of the extracted bitumen with the amine additive was lower than that of the bitumen with hydrated lime. The Polish standard, however, allows breaking point temperature differences between two samples to reach 2 °C.

The variations between different quality groups were assessed using the analysis of variance (ANOVA).

The results of the evaluation of significant impact of the selected factors (type of hydrated lime, fatty acid amine, location – in the wheel paths, in between the wheel paths) on the breaking point temperature are shown in Table 1.

The type of adhesion agent (*p*-value = 0.045) had a significant effect on the results of the breaking point temperature. However, the decision is not clear because this value is close to the limit of the significance level that amounted to α = 0.05. Other parameters did not play a significant role. According to the analysis results, the level of the breaking point temperature with fatty amines is about 1.5 °C lower than the value for the bitumen with hydrated lime. Such a difference is less than the acceptable tolerance (±2 °C for the breaking point temperature), thus the difference is not significant.

## 4. Dynamic viscosity of extracted bitumen as a function of temperature

Dynamic viscosity is the one of the fundamental properties, which enables determination of optimal temperatures for bituminous mixtures performance and compaction. Dynamic viscosity reflects internal friction, which confirms the existence of cohesion forces between constituents of the bitumen when they shift to each other (Gawel *et al.* 2001). The experiment was conducted using a rotary viscometer "Rheotest 2" with a cylinder mark H2. All bitumen viscosity tests were performed at a shear rate of 1 s$^{-1}$. The viscosity measurement was carried out over the temperature range from 60 °C to 135 °C with steps of 10 °C in most cases. The bituminous binder in operational
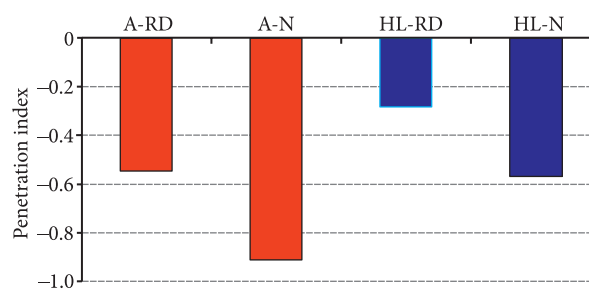


**Fig. 2.** Changes in penetration indexes of SMA-recovered bitumen

**Table 1.** The evaluation of significant influence of tested factors on the breaking point temperature of the extracted bitumen

| Effect | Degr. of freedom | Univariate results for each DV over-parameterized model type III decomposition | | | |
|---|---|---|---|---|---|
| | | $T_{FRAASS}$, °C | | | |
| | | SS | MS | F | p |
| Type of additive | 1 | 6.750 | 6.75000 | 5.61039 | 0.04534 |
| Location | 1 | 0.750 | 0.75000 | 0.62338 | 0.45256 |
| Type of additive * Location | 1 | 0.750 | 0.75000 | 0.62338 | 0.45256 |
| Error | 8 | 9.625 | 1.20313 | | |
| Total | 12 | 1404.625 | | | |

temperatures should behave as a shear thinning non-Newtonian fluid (Grabowski, Słowik 2002). The results of dynamic viscosity tests at temperatures between 60 °C and 135 °C are presented in Fig. 3.

Results of dynamic viscosity versus bitumen temperature extracted from SMA indicated that there was a significant increase in viscosity at temperatures over 90 °C (Fig. 3).
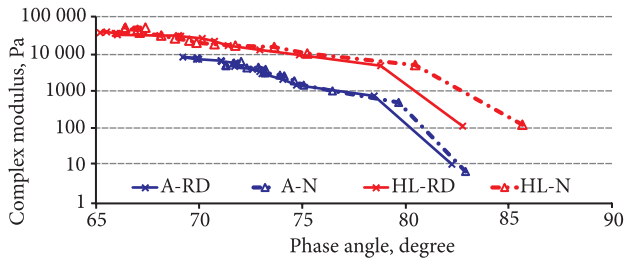


**Fig. 3.** The dynamic viscosity versus temperature of the bitumen extracted from SMA pavement at temperature from 60 °C to 135 °C
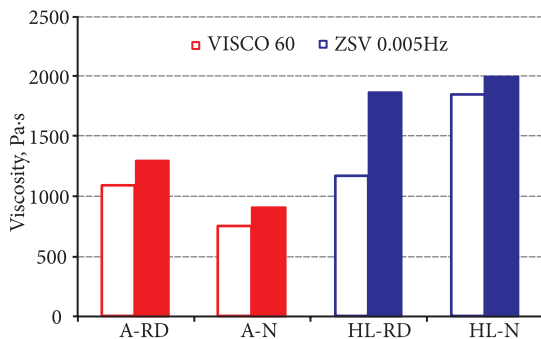


**Fig. 4.** Zero shear viscosity (ZSV) of extracted binder at temperature of 60 °C at frequency $f = 0.005$ Hz in comparison with dynamic viscosity at 60 °C (VISCO 60)

The hydrated lime and SBS polymer changed the bitumen structure making it more stiff and elastic. The results of dynamic viscosity of the recovered binder with the addition of fatty amine did not invoke such effect. Dynamic viscosity values varied depending on sampling locations. This may indicate a different aging process of the asphalt surface. The research was extended to add a zero shear viscosity (ZSV at 0.005 Hz). This parameter represents a behavior of the bitumen in linear visco-elasticity. The results of the zero shear viscosity tests are shown in Fig. 4.

Identification of significant factors that affected the dynamic viscosity and ZSV at 60 °C are shown in Tables 2 and 3.

All the factors had a significant influence on the dynamic viscosity variation ($p$-value < 0.05). Logical conclusion indicates strong impact of the considered factors on this parameter (VISCO60). The presence of hydrated lime caused an increase in dynamic viscosity value close to 80% in relation to the reference bitumen (with the amine). This effect definitely contributes to the reduction of the plastic deformation in the SMA pavement. With regard to the evaluation of "location" parameter, 18% increase in dynamic viscosity value was observed in samples taken from out of area of mix rutting (N). The effect of the aging process of samples located in the wheel paths (RD) relates to permanent compaction process caused by traffic thus contributing to the low variation in the dynamic viscosity results.

The ZSV (zero shear viscosity) parameter was correlated with the location and the type of additive ($p$-value < 0.05), and although the influence of the location factor was the lowest, it was still significant. The hydrated lime (HL) incorporated into the bitumen causes the increase in structural viscosity of approximately 90% in comparison

**Table 2.** The evaluation of the influence of the qualitative factors on the dynamic viscosity (VISCO60) at 60 °C

| Effect | Degr. of freedom | Univariate results for each DV over-parameterized model type III decomposition | | | |
|---|---|---|---|---|---|
| | | VISCO60, Pas | | | |
| | | SS | MS | F | p |
| Type of additive | 1 | 1 028 431 | 1 028 431 | 670.9436 | 0.00000 |
| Location | 1 | 91 351 | 91 351 | 59.5968 | 0.00006 |
| Type of additive * Location | 1 | 766 591 | 766 591 | 500.1204 | 0.00000 |
| Error | 8 | 12 262 | 1 533 | | |
| Total | 12 | 19 679 006 | | | |

**Table 3.** The evaluation of the influence of the qualitative factors on the zero shear viscosity (ZSV) at 60 °C

| Effect | Degr. of freedom | Univariate results for each DV over-parameterized model type III decomposition | | | |
|---|---|---|---|---|---|
| | | ZSV, Pas | | | |
| | | SS | MS | F | p |
| Type of additive | 1 | 2 077 421 | 2 077 421 | 915.0374 | 0.00000 |
| Location | 1 | 52 391 | 52 391 | 23.0765 | 0.00135 |
| Type of additive * Location | 1 | 206 168 | 206 168 | 90.8103 | 0.00001 |
| Error | 8 | 18 163 | 2270 | | |
| Total | 12 | 30 039 740 | | | |

with the bitumen with the amine content (A). In this case the variation in the test results of samples taken from the rut paths (RD) looks smaller than in the samples that were taken from the area between the rut paths (N).

## 5. Complex modulus and phase angle of extracted bitumen

Complex modulus G* and phase angle δ characterize the visco-elastic behavior of a material (Sybilski 2000). Parameter G* describes complex resistance of the material to permanent deformation by repeated shear stress. The material consists of an elastic and viscous part of the complex modulus, and their interaction is described by the phase angle δ. The difference between the assessments of phase angle value of these two additives is approximately close to 30%. The illustration of the influence of additives on the visco-elastic behavior of the extracted bitumen is presented in Fig. 5 (for complex modulus G*) and in Fig. 6 (for phase angle).

The results indicate that the use of hydrated lime causes an increase in complex modulus of the bitumen (bitumen 50/70 modified with 4% SBS polymer) at 60 °C in comparison with the reference bitumen (bitumen 50/70 with the fatty acid amine additive). First of all it should be noted that the binder recovered from the SMA mixture containing hydrated lime, regardless of the sampling method, is characterized by the sevenfold increase in the complex modulus. The application of hydrated lime in the SMA mixture ensures greater resistance to rutting formation in comparison with the fatty acid amine in SMA. This level of stiffness of bitumen for the case with H-L provides low susceptibility to the permanent deformation according to assumptions of SHRP programme (Sybilski 2000: 50).

The results of the analysis indicate the shift towards more elastic behavior of the bitumen. This is caused by the presence of hydrated lime in the bitumen. Bitumen samples with hydrated lime are characterized by at least a lower value of phase angle. For a cases A-RD and HL-RD average of result of A-RD is a slightly lower than bitumen denoted as HL-RD. The differences are within the measured error and it is difficult to decide their significant variation. Furthermore, the results of bitumen with hydrated lime point to higher complex modulus value. The high level of G* and lower phase angle δ suggest an increase in the elastic part of the complex modulus (G') of the tested bitumen. Such behavior of extracted bitumen is connected with a change in the bitumen structure.

## 6. MSCR tests for extracted bitumen

The MSCR (Multiple Stress Creep Recovery Test of Asphalt Binder) was conducted under the AASHTO TP70 standard designed to verify the test results according to SHRP (complex modulus, phase angle) as an alternative assessment for modified bitumen. The test enables

evaluation of the bitumen non-linear behavior (Cardone *et al.* 2015; Iwański, Mazurek 2012a). Additionally, it characterizes a high correlation between rheological bitumen test results and those of compacted bituminous mixtures obtained from the rutting assessment in wheel tracker (Zoorob, Oliveira 2012). The MSCR has been proposed as a better method for predicting pavement failures caused by high ambient temperatures when bitumens turn into fluids (Kim 2009). The method allows assessing the compliance and relaxation of a binder applied in a mixture and simulates load conditions as near as possible to natural with a non-linear behavior of the bitumen in a broad range of stress. Binder non-recoverable compliance tests were carried out for two stress ranges of 100 Pa and 3200 Pa weighing down for 1 second, with subsequent measurement of the bitumen elastic recovery for 9 seconds at the bitumen temperature of 60 °C. The whole cycle of one range of stress lasted 100 seconds. Consequently, the value of creep compliance Jnr (a non-recoverable part of deformation divided by the applied stress), and the value of elastic recovery $\varepsilon_r$ in % (the relative value of elastic strain in percentages being the ratio of deformation in the first second at the beginning of a cycle to the value of deformation in the tenth second) were determined.

The following parameters were measured:
- Creep compliance denoted by Jnr100 (at stress of 100 Pa);
- Creep compliance denoted by Jnr3200 (at stress of 3200 Pa);
- Elastic recovery at deformation *R*, %.

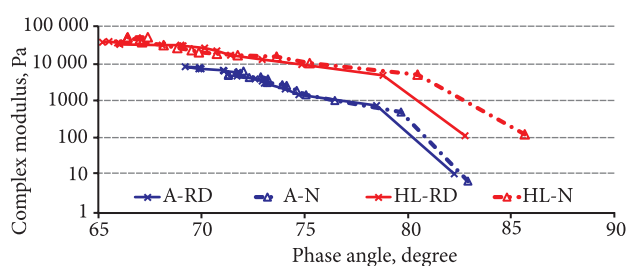**Fig. 5.** Complex modulus G* versus phase angle of extracted bitumen from SMA pavement at the temperature of 60 °C

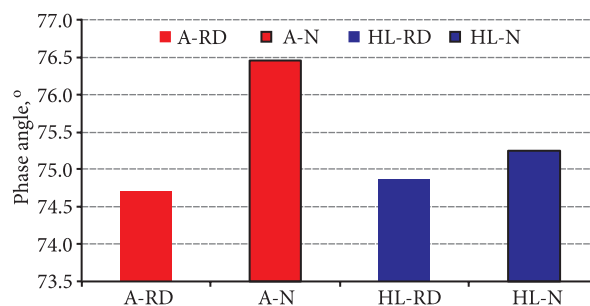**Fig. 6.** The phase angle value δ of extracted bitumen at frequency of 1.56 Hz and temperature of 60 °C

The results were calculated on the basis of deformation changes during loading/unloading stage of the bitumen extraction (Fig. 7).

Regardless of the sampling location, the bitumen derived from SMA mixtures with hydrated lime was characterized by a lower level of strains than the samples of the bitumen with fatty amines. This affects the level of bitumen creep compliance, specified as Jnr. The bitumen samples with hydrated lime addition had a lower level of the creep compliance than the bitumen samples with fatty amine. This result was observed regardless of the level of stress. Due to small differences between the means of selected cases to identify significant contrast between them, in the follow-up part of this report, an analysis of variance for these parameters will be performed. Results of creep compliance (Jnr), tested for 100 Pa and 3200 Pa
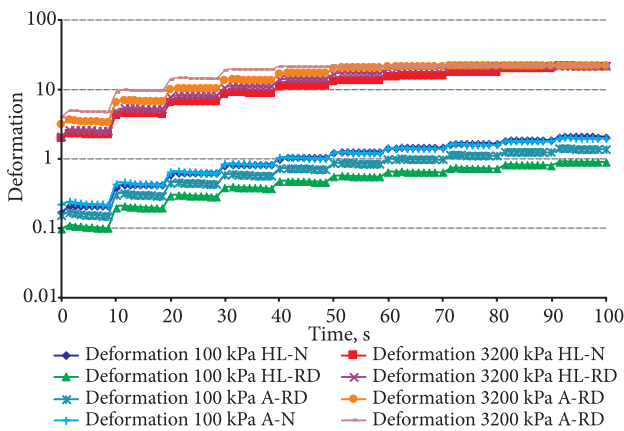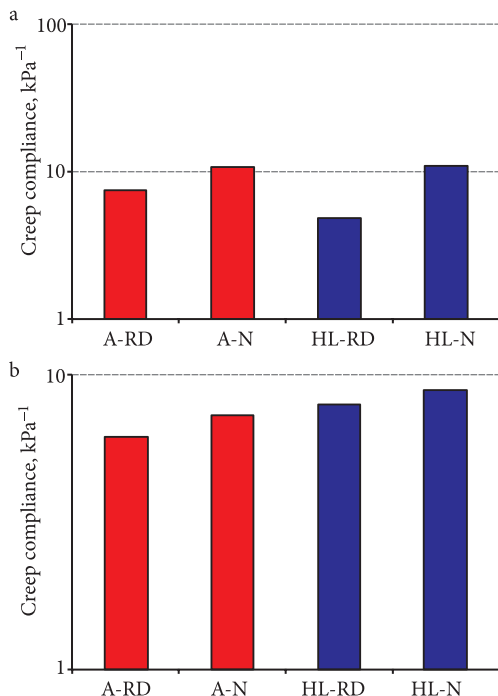
shear stress levels are shown in Fig. 8. However, the results of elastic recovery tests conducted for 100 Pa and 3200 Pa shear stress levels are shown in Fig. 9.

The complementary element to analyze is the elastic recovery (R) according to the MSCR test specification (Fig. 9). In relation to low shear stress all samples gained approximately the same level of strains. Test results obtained at high level of stress revealed some differences. For the purpose of finding variations between results it was necessary to perform an analysis of variance. The results of elastic recovery for specific group samples are presented in Fig. 8. The results indicate small differences between samples of the bitumen with hydrated lime. This suggests that hydrated lime had a beneficial influence on the structure of bitumen and prevented it from turning it into a more brittle material. The presence of hydrated lime also stopped the thixotropic effect in the bitumen.

The analysis of variance results for the influence of additives (hydrated lime, fatty amine) and location (in the wheel paths, between the wheel paths) on the creep compliance and the elastic recovery are presented in Figs 10–13.

The analysis of the test results for both cases of shear stress reveals that the location and additive type have a significant influence on the creep compliance. Both, for low and high level of shear stress in samples with hydrated lime, 11% and 20% decrease in the creep compliance value in relation to samples with the amine was recorded. The location effect indicated that the bitumen samples taken from the wheel paths at low shear stress (100 Pa) had lower creep compliance. The high stress caused an inverse action in bitumen samples taken from the area between the wheel paths, which exhibited lower creep compliance.



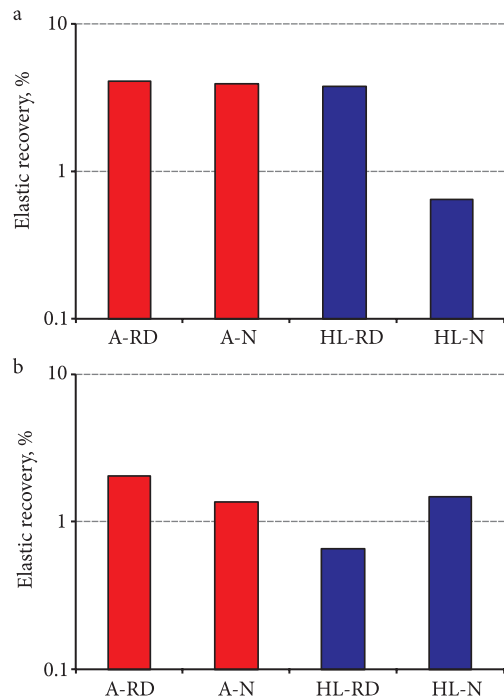**Fig. 7.** Strain changes of the extracted bitumen from SMA pavement samples dependent on dynamic stress level



**Fig. 8.** Test results of the creep compliance extracted bitumen according to MSCR procedure: a – Jnr 100 Pa; b – 3200 Pa



**Fig. 9.** Test results of elastic recovery according to MSCR procedure: a – R 100 Pa; b – 3200 Pa
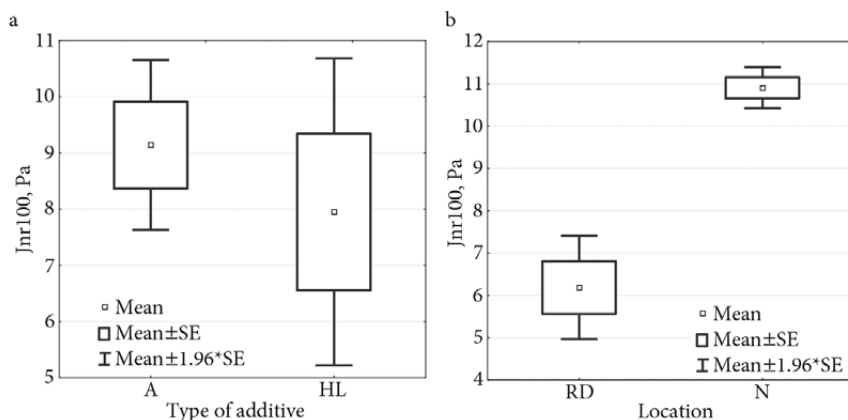
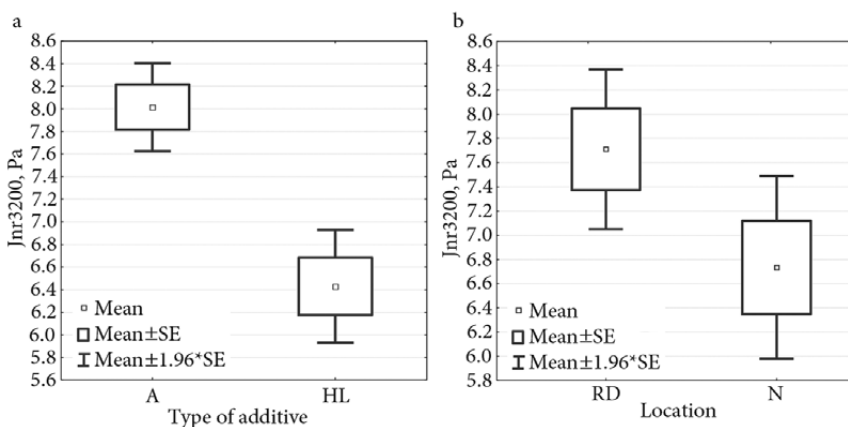**Fig. 10.** Creep compliance (Jnr100) test results distribution at temperature 60 °C: a – additive; b – location



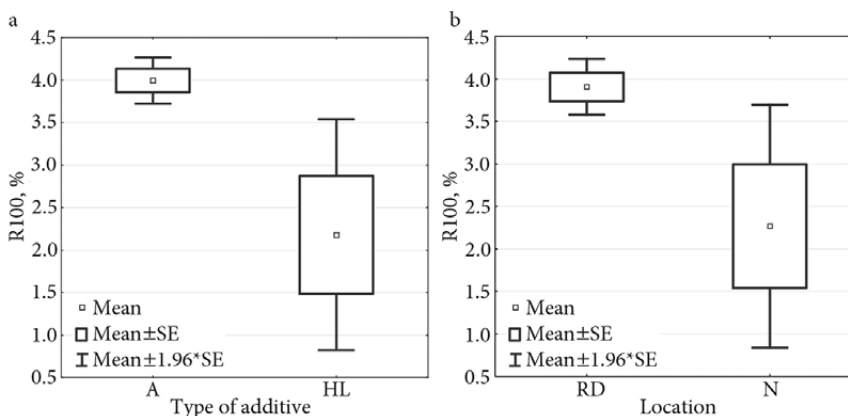**Fig. 11.** Creep compliance (Jnr3200) test results distribution at temperature 60 °C: a – additive; b – location



**Fig. 12.** Elastic recovery R100 test results distribution at temperature 60 °C: a – additive; b – location
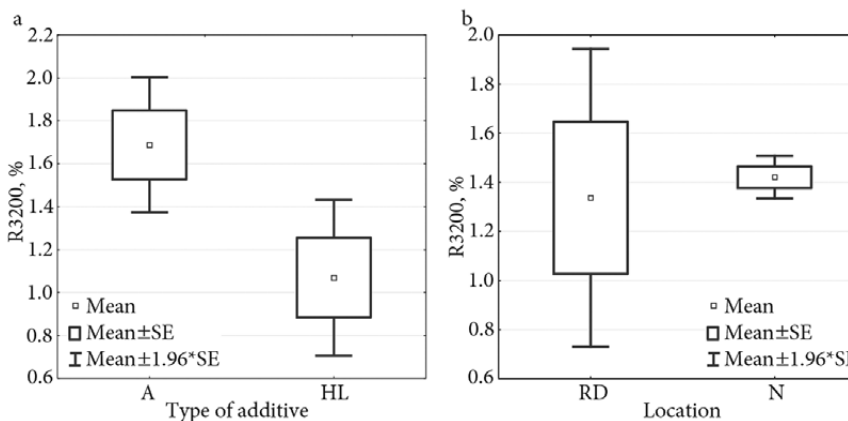


**Fig. 13.** Elastic recovery R3200 test results distribution at temperature 60 °C: a – additive; b – location

The analysis of variance showed that the effect of the "type of additive" factor had a significant influence on the elastic recovery regardless of the stress level. ANOVA algorithms also revealed an effect of the interaction between "location" and "type of additive" factors. In both cases, the changes in elastic recovery level did not exceed 5% and 2% in relation to the initial value. Elastic recovery in the amine modified bitumen was slightly higher than the value recorded for the bitumen with the hydrated lime. The significantly higher value of the elastic recovery samples located in the rut paths confirms the thesis that the pavement surface does not age equally. As for the application of high stress level (320 Pa), the effect of "location" did not have a significant influence that could be connected with the disturbance in the bitumen structure.

## 7. Final analysis

For general assessment of the influence of "location" and "type of additive" on the bitumen rheological properties, the following analysis of variance was used (ANOVA) (Table 4).

To sum up, the effect of the "type of additive" factor turns out to be the most influential factor among all bitumen properties. The effect of "location" also plays an important role in changing bitumen properties. The location effect did not have any influence on properties such as the softening point temperature, the breaking point temperature and the elastic recovery according to MSCR with high shear stress. However it was noticed that the interaction effect (Type of additive * Location) affected bitumen properties except for the breaking point temperature and creep compliance at high shear stress.

## 8. Conclusions

The analysis of the test results for the bitumen extracted from SMA after 12 years of service life allows drawing the following conclusions:

1. The results of the penetration index stands at $IP = -0.5$ which means that the bitumen maintains a gel-sol state with good flexibility at low temperatures. Bitumen samples with amine exhibit a slightly lower level of penetration index which could invoke an increase in susceptibility to permanent deformation of the SMA mixture.

2. The presence of hydrated lime resulted in a significant increase in the softening point temperature up to 2 °C and a reduction in the penetration grade amounted to 3 units (0.1mm). With regard to the effect of location,

the change in value of the penetration grade was approximately 10 units.

3. The presence of hydrated lime in the bitumen resulted in a significant 1.5 °C increase in breaking point temperature. This value is lower than the maximum test error level which amounts to ±2 °C.

4. A plot of the complex modulus G* versus the phase angle revealed an influence of hydrated lime in the bitumen on the increase in G* value. This demonstrates an increase in elastic behavior of the extracted bitumen.

5. The addition of hydrated lime resulted in a significant increase in the dynamic viscosity of the recovered binder in the range from 80% to 90% in comparison with the amine.

6. The addition of hydrated lime resulted in a significant drop in creep compliance of the recovered binder in the range from 11% to 20% in comparison with the amine.

7. Despite of the differences between the results of elastic recovery its maximum value was relatively small.

8. The effects of the location and the interaction between the considered factors proved to have a significant influence on bitumen parameters. Only the breaking point temperature proved to be insensitive to this combination of factors.

9. Tests results showed a significant positive effect of hydrated lime on rheological properties of the recovered binder made from bitumen D70 (50/70) and 4% of the SBS polymer. Hydrated lime had a positive influence on ensuring durability of SMA pavement during 12 years of service life.

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**Table 4.** The collection of statistical analyses of properties of bitumen extracted from SMA pavement (*p*-value < 0.05)

| Effect | Univariate results for each DV over-parameterized model type III decomposition | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | $T_{R\&B}$, °C | Pen, 0.1mm | $T_{FRAASS}$, °C | ZSV, Pas | VISCO60, Pas | Jnr3200, Pa | Jnr100, Pa | R100, % | R3200, % |
| Type of additive | 0.00025 | 0.03752 | 0.04534 | 0.00000 | 0.00001 | 0.00010 | 0.01054 | 0.0001 | 0.00007 |
| Location | 1.00000 | 0.00009 | 0.45256 | 0.00135 | 0.00006 | 0.00001 | 0.00000 | 0.00001 | 0.33338 |
| Type of additive * Location | 0.00012 | 0.00173 | 0.45256 | 0.00001 | 0.00001 | 0.34161 | 0.00394 | 0.00002 | 0.00002 |

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