



THE COMPARATIVE STUDIES OF THE PROPERTIES OF JOINT SEALANTS PRODUCED BY MANUFACTURERS AND IN LABORATORY CONDITIONS WITH THE USE OF HIGHLY MODIFIED BITUMEN

BADANIA PORÓWNAWCZE WŁAŚCIWOŚCI MAS ZALEWOWYCH WYTWARZANYCH PRZEZ PRODUCENTÓW I W WARUNKACH LABORATORYJNYCH Z WYKORZYSTANIEM ASFALTU WYSOKOMODYFIKOWANEGO

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Abstract

Joint sealants produced on the basis of modified bitumen are an effective mean for protection of expansion joints on bridges and for repair of cracks in various road surfaces. A comparative study was performed to evaluate seven hot-applied joint sealants obtained commercially and three joint sealants produced in laboratory conditions with different contents of highly modified asphalt binder (40 to 100%). The basic properties of the joint sealants and asphalt binders were evaluated, including penetration, softening point, breaking point and elastic recovery. Additionally, Fourier infrared spectroscopy (FTIR) method was used to evaluate the chemical composition of the asphalt binders. The variability of the basic properties of joint sealants was estimated in the range from -77.1% to 43.6% in relation to the base asphalt binder. It has been established that the addition of crumb rubber, hydrated lime and rapeseed oil may be viable in controlling the parameters of the joint sealants.

Keywords: hot-applied joint sealant, highly modified bitumen, expansion joint, hydrated lime, crumb rubber

Streszczenie

Zalewy szczelin produkowane na bazie asfaltów modyfikowanych są skutecznym rodzajem zabezpieczenia przerw dylatacyjnych na obiektach mostowych oraz naprawy uszkodzeń różnych typów nawierzchni drogowych. Badaniami porównawczymi objęto siedem mas zalewowych stosowanych na gorąco, wytworzonych przez krajowych i zagranicznych producentów, oraz trzy masy zalewowe wytworzone w warunkach laboratoryjnych o różnej zawartości wysokomodyfikowanego lepiszcza asfaltowego (40 do 100%). Ocenie poddano podstawowe cechy lepiszczy asfaltowych oraz parametry wyprodukowanych na ich bazie mas zalewowych, obejmujące: penetrację w 25°C, temperaturę mięknięcia, temperaturę łamliwości Fraassa i nawrót

sprężysty. Dodatkowo, do porównania składu chemicznego lepiszczu asfaltowych wykorzystano metodę spektroskopii fourierowskiej w podczerwieni (FTIR). Oszacowano procentowy zakres zmienności podstawowych właściwości mas zalewowych w relacji do bazowego lepiszczu asfaltowego w granicach od -77,1% do 43,6% w relacji do bazowego lepiszczu asfaltowego. Ustalono, że istotnym regulatorem parametrów mas zalewowych mogą być dodatki odpadów gumowych, wapna hydratyzowanego oraz oleju rzepakowego.

Słowa kluczowe: zalewa szczelin na gorąco, asfalt wysokomodyfikowany, szczelina dylatacyjna, wapno hydratyzowane, odpady gumowe

1. INTRODUCTION

One of the basic requirements for sealing expansion joints is the use of a highly flexible sealant made on the basis of petroleum based bitumen with the addition of elastomeric polymers (such as SBS, crumb rubber), fillers and surface-active substances [1-6]. Cold and hot-applied sealants are a commonly used type of protection of expansion joints on structures with reinforced concrete and steel structures, as well as in asphalt concrete, concrete or epoxy surfaces. Joint sealants are responsible for the transfer of deformations between the expansion joints due to its deformability over a wide range of temperatures. The dimensions of the expansion joint are selected individually for each structure. The bituminous expansion joint is a surface element that not only transmits the load of heavy vehicles' wheels, but also compensates vertical and horizontal deformations caused by the expansion joint's edge movement in the construction e.g. a road bridges [7-9].

The paper concerns a selection of the properties of hot-applied joint sealants, classified as the high-extension type N1 (elastic) or low-extension type N2 (normal), in accordance with the standard EN 14188-1 [10]. The selection of the joint sealant type, in terms of its viscoelastic properties, as well as the method of application should result from the role and function that it will perform during the service life of the road or bridge surface. As construction materials, joint sealants must meet the requirements of the applicable standards and guidelines [2, 10, 11]. The current standard [10] does not introduce material restrictions on the composition of the joint sealants, but it provides a specific set of requirements that should be met by the final product.

Crack sealing with hot-applied sealants based on modified bitumen is also a widely used technology for the surface repairs of the upper layers of road surfaces, made of either asphalt mixtures or cement concrete. Preventing the degradation of road structures requires the immediate and permanent maintenance of their discontinuities, which occur during pavement

service [12]. The properties of applied joint sealants must be adapted to the nature of the job, the location where they will be used in the surface structure, and the effects of external factors [13-15].

Phenomena such as aging, fatigue and thermal stability of joint sealants based on bitumen can cause failures or even secondary cracking. The use of non-modified asphalt binders in crack sealants provides a cheap solution for the maintenance of cracking pavements; however, such formulations are prone to debonding and poor low- or high-temperature performance. Therefore, it is necessary to prepare joint and crack sealants with adequate performance.

Highly modified bitumens are characterized by a large amount (typically 7 to 8 percent) of specialized styrene-butadiene-styrene block copolymers, ensuring their compatibility with the base bitumen. In pavement engineering, various technological solutions are used in the field of asphalt modification, and the use of additives for mineral and asphalt mixtures depends on their purpose in the road surface construction, the environmental conditions, and the pro-ecological requirements [16-20]. Therefore, modified asphalt binders are used to produce and investigate joint and crack sealant [1, 3, 21-25]. Styrene-butadiene-styrene (SBS) and crumb rubber (CR, 10, 15, 20%) have been utilized by Gong et al. [26] to modify sealants with bitumen to overcome the disadvantages of the poor high-temperature and rheological properties of sealants. It has been shown that the SBS/CR-modified asphalt sealant has a greater viscosity and higher temperature deformation resistance.

Gnatenko et al. [27] in their study confirmed that to reduce brittleness and increase the flexibility of joint sealants and mastics at low temperatures, it was necessary to use a plasticizer. On the other hand, improving their performance at high temperatures required the use of thermoplastic elastomers made of SBS, latexes, mineral fillers (5, 10 and 15%), or fine crumb rubbers (3, 5, 15 and 20%), which are similar to those used in road paving asphalt binders [19, 20].

As shown above, joint sealants are complex preparations and given the broad range of base asphalt binders, modifiers, fillers, and other non-soluble additives. So far, no clear guidelines have been developed regarding the material composition and detailed requirements for the properties of individual components of joint sealants.

Joint and crack sealants may be produced in very diverse combinations of components:

- one-component, exclusively of modified or highly modified bitumen;
- two-component with the use of virgin components, produced from asphalt binders and mineral fillers in various proportions;
- multi-component with the use of virgin components (asphalt binders and mineral fillers) and waste materials (crumb rubber, aggregate dusts) in various proportions.

Due to the observed failure modes in asphalt expansion joints [3, 9, 30] and road surface repairs after sealing [12, 24] it is necessary to conduct further research on the properties and composition of joint sealants in terms of their applicability in various climatic conditions and traffic loads.

The lack of conclusive requirements for the components of asphalt-based sealant indicates the need to conduct investigation in this area and to search for additives that are an important regulator of their properties.

The aim of the planned research and analyses was:

- Verification of the possibility of reducing the content of asphalt binder in multi-component hot-applied joint sealant in relation to used in industry products up to 40%.
- Evaluation of the influence of hydrated lime and rapeseed oil as regulators on the variability of the properties of joint sealants.

- Comparison of the basic characteristics of joint sealants produced by manufacturers and in laboratory conditions in relation to the same properties of base bitumen.
- Estimation of the percentage range of variability of the properties of joint sealants in relation to the base asphalt binder.

It has been established that it is possible to regulate selected properties of the joint sealants by changing the chemical composition and proportions of its components (fillers, hydrated lime, rapeseed oil, crumb rubber). It was found that it was difficult to ensure the required standard parameters of joint sealants for the content of asphalt binder below 50%, even when using highly modified bitumen.

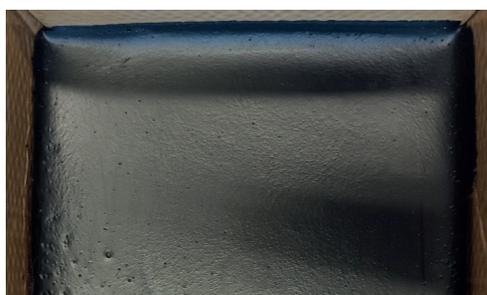
2. MATERIALS AND METHODS

2.1. Materials

2.1.1. Origin of materials

The investigations covered hot-applied joint sealant of various characteristics, obtained commercially (Fig. 1a), as well as produced in laboratory conditions (Figs. 1b, 1c). The sealants produced by commercial manufacturers, were marked with MS symbols (MS1 – MS7), while those produced in laboratory conditions were marked as LS (LS1 – LS3). Seven hot-applied asphalt-based joint sealants were used, obtained from 4 selected domestic and foreign manufacturers. According to the manufacturers, the intended use of the sealants included: sealing cracks, horizontal gaps, expansion joints and technological joints in road surfaces of all traffic load categories. Three joint sealants LS1 – LS3 were produced with the use of new components (bitumen and fillers).

a)



b)



c)



Fig. 1. Photographs of investigated hot-applied joint sealant: a) produced by manufacturer (MS3); b) during manufacturing with homogenizer Unidrive X100 under laboratory conditions [31]; c) in laboratory utensil after homogenization (LS1)

2.1.2. Joint Sealants Produced by Manufacturers

According to the manufacturers’ declarations, the joint sealants selected for the investigation complied with the requirements of EN 14188-1 [10] standard for the following types:

- Flexible (high-extension) – type N1 (MS1, MS2, MS4);
- Normal (low-extension) – type N2 (MS3, MS5, MS6, MS7).

Selected declared properties are presented in Table 1.

On the basis of the solvent extraction of the MS1 – MS7 sealants and subsequent EDX spectroscopy (Quanta Feg 250 SEM and EDS spectrum), their material and chemical composition was identified [36]. It was found that among the investigated sealants there are various compositions:

- one-component, exclusively of highly modified bitumen (MS1);
- two-component, constituting a composition of asphalt binders and mineral fillers (MS7);
- multi-component with the use of new components (asphalt binders, mineral fillers) and waste materials (rubber crumbs) in various proportions (MS2 – MS6).

The occurrence of rubber particles of different size, origin and method of production (cryogenic and mechanical method) in investigated joint sealants (MS2 – MS6) with different content percentages (8.7-19.3%) was found. Selected images of the crumb rubber used in MS3, MS5 and MS6 are presented in Figure 2.

Table 1. Selected characteristics of joint sealant declared by manufacturers

Material Properties	Characteristics of Investigated Joint Sealant and Declaration by Manufacturers							Testing Method
	MS1	MS2	MS3	MS4	MS5	MS6	MS7	
Type of hot-applied joint sealant	N1	N1	N2	N1	N2	N2	N2	EN 14188-1 [10]
Density at +25°C, in Mg/m ³	No dec.*	1.15 ±0.05	1.12	1.15 ±0.05	1.2	No dec.	1.2 ±0.1	EN 13880-1 [32]
Softening point, ring and ball, in °C	No dec.	98 ±8	≥85	98 ±8	102	No dec.	≥85	EN 1427 [33]
Cone penetration at 25°C, in 0.1 mm	40 to 130	60 ±10	40 to 100	60 ±10	54	40 to 100	40 to 100	EN 13880-2 [34]
Penetration and recovery at +25°C, in%	≥60	65 ±5	<60	65 ±5	59	≤60	≤60	EN 13880-3 [35]

* No dec. — No declaration by manufacturer

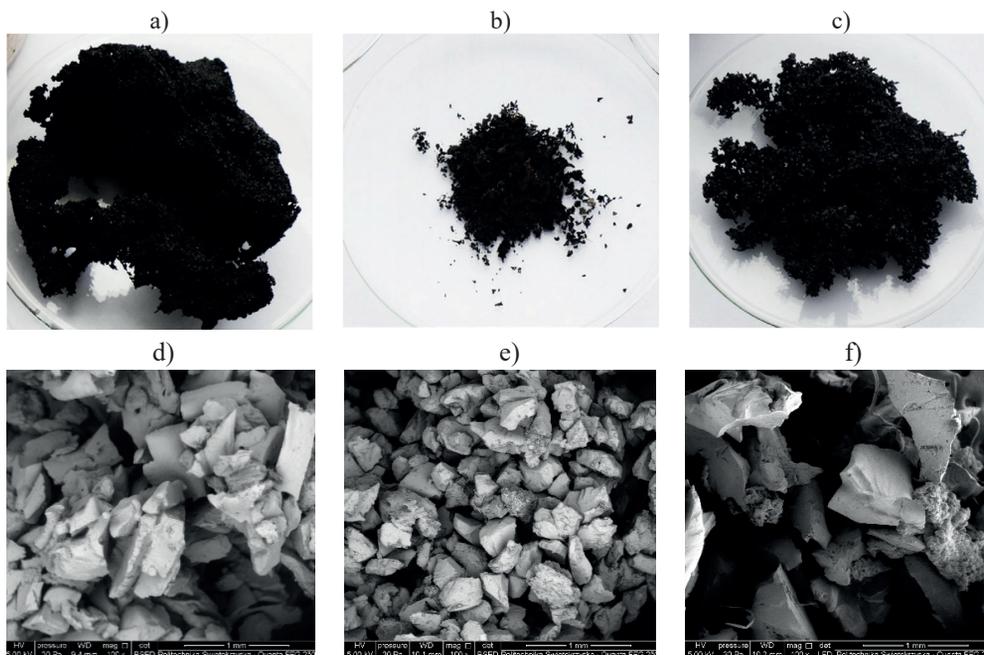


Fig. 2. Images of the rubber crumbs recovered from the selected joint sealants: a), b), c) MS3, MS5, MS6 – without magnification; d), e), f) MS3, MS5, MS6 – magnification × 100 by scanning electron microscope

The variation in the content of the rubber waste components, as well as the method of producing crumb rubber, significantly affects the physicochemical properties of rubber–asphalt binders [37]. Rubber crumbs were utilized for modifying the asphalt-based sealants in order to obtain greater viscosity and higher temperature deformation resistance. The use of rubber also increases the material's resistance to low temperatures. Therefore, it should be assumed that it is an important regulator of the properties of joint sealant.

Mineral filler that passed through a 0.063 mm sieve has been identified in different contents (10.6 to 50.2%) in the joint sealants from manufacturers (MS2 –MS7). The analysis of the chemical composition with the use of EDX spectroscopy showed the presence of calcium or silicon with the highest percentage in the investigated materials.

2.1.3. Joint Sealants Produced under Laboratory Conditions

The following materials were used in the production of the joint sealants LS1 – LS3 under laboratory conditions:

- highly modified bitumen 65/105-80 according to PN-EN 14023:2011/Ap1:2014-04 (National Annex) [38];
- lime filler;
- hydrated lime (calcium hydroxide).

The highly modified bitumen 65/105-80 is characterized by a high content of SBS polymer in the amount of about 7.0-7.5% m/m. Such significant amount of this component results in the formation of a continuous polymer network in the binder structure, which is not present in typical polymer bitumen. This improves the properties of the asphalt at high and low temperatures. Its main advantages include increased resistance to cracking, permanent deformations and fatigue. These features contribute to the applicability of the 65/105-80 bitumen as the basic component for flexible hot-applied joint sealants.

The most important properties of 65/105-80 asphalt binder, declared by its manufacturer, are presented in Table 2.

In order to ensure the high quality of the joint sealant, a mixed filler was used in the study, which contained hydrated lime (calcium hydroxide) in the amounts of 10, 20 and 30% interchangeably with lime filler.

Three types of joint sealant were produced, differing in the content of mineral filler used in the amounts of 20, 40 and 60%.

Table 2. Selected characteristics of the highly modified asphalt binder (65/105-80) declared by manufacturer

Property of asphalt binder	Needle penetration at 25°C	Softening point, ring, and ball	Fraass breaking point	Elastic recovery at 25°C
Unit	0.1 mm	°C	°C	%
Test method	EN 1426 [39]	EN 1427 [33]	EN 12593 [40]	EN 13398 [41]
Declared value	65 to 105	≥ 80	< -18	≥ 80

2.2. Research Methodology

The comparative analyses covered the properties of ten joint sealants produced by commercial manufacturers (MS) and produced in laboratory conditions (LS) with varying content of asphalt binder (40 to 100%) and other components. Previous investigation in this area [36, 39] has been compared, verified and extended.

The testing methodologies included the solvent extraction of the investigated joint sealant MS1 – MS7 with the use of tetrachloroethylene as a solvent:

- The determination of the soluble binder content (EN 12697-1).
- The recovery of soluble bitumen with the use of a rotary evaporator (EN 12697-3+A1).

Distillation conditions used in the testing methodologies for recovery of soluble bitumen were as follows:

- Solvent: Tetrachloroethylene,
- Boiling Point: 121°C,
- Temperature (First Phase): 110°C,
- Pressure (First Phase): 40 kPa,
- Temperature (Second Phase): 110°C,
- Pressure (Second Phase): 40 kPa,
- Extra Temperature: 180°C.

Samples of LS1 – LS3 were prepared in laboratory conditions. The mineral sample filler was dried at 105°C for 24 h in order to eliminate the moisture it contained. The filler was then dosed in the required proportion into the asphalt binder and mixed at 500 rpm for 30 minutes at a constant temperature of 160°C.

Table 3 presents the basic compositions of the analysed joint sealants.

Each of the LS sealants was produced in laboratory conditions in three variants with different content of hydrated lime in the mineral filler, i.e.:

- 0% (LS1/0, LS2/0, LS3/0),
- 10% (LS1/10, LS2/10, LS3/10),
- 20% (LS1/20, LS2/20, LS3/20),
- 30% (LS1/30, LS2/30, LS3/30).

Table 3. Compositions of the investigated joint sealants

Component materials of joint sealant	Mass share of components in sealant (%)									
	MS1	MS2	MS3	MS4	MS5	MS6	MS7	LS1	LS2	LS3
Soluble asphalt binder (MS sealant)/ Asphalt binder (LS sealant)	100	71.3	59.0	69.8	70.7	72.8	49.8	80.0	60.0	40.0
Mineral filler (≤ 0.063 mm)	0	14.2	21.7	15.6	10.6	18.5	50.2	20.0	40.0	60.0
Mineral and rubber dust (> 0.063 mm; ≤ 0.19 mm)	0	5.5	12.4	4.1	3.9	0	0	0	0	0
Rubber powder/crumbs (> 0.19 mm)	0	9.0	6.9	10.5	14.8	8.7	0	0	0	0
Total	100	100	100	100	100	100	100	100	100	100

The testing methodologies included:

- Fourier transform infrared spectroscopy (FTIR) using the attenuated total reflectance (ATR-FTIR) method for comparative analyses of asphalt binders used for the production of joint sealants;
- Basic properties of the joint sealant and (soluble) asphalt binder:
 - Needle penetration at 25°C acc. EN 1426 [39],
 - Softening point, ring, and ball acc. EN 1427 [33],
 - Fraass breaking point acc. EN 12593 [40],
 - Elastic recovery at 25°C acc. EN 13398 [41].

In order to determine the range of variability of the basic properties of joint sealants in relation to the base asphalt binder, the percentage differences in the values of individual parameters were determined.

3. RESULTS AND DISCUSSION

3.1. Comparative analyses of asphalt binders used in joint sealants

3.1.1. Conventional properties of asphalt binder used in joint sealants

The comparative evaluations of the penetration at 25°C, the softening point, the Fraass breaking point, and the elastic recovery were performed. Table 4 shows the test results of the asphalt binders used in investigated joint sealants.

The analysis of the results in Table 4 shows that the asphalt binders extracted from the MS1 – MS7 joint sealants did not relate clearly to the types provided in the national annexes (PL) of EN 12591 [42] and EN 14023 [38], which are used for paving asphalt binders. This result may have been affected by the influence of the modification of the varying content of crumb rubber or other substances.

The results of the elastic recovery test at 25°C in the case of sealants MS1, MS2 and MS3 – MS7 indicated a very high rate of elastomeric modification similar to that seen in the highly modified bitumens produced in Poland according to the national annex of the PN-EN 14023 standard [38]. The MS3 sample failed before the end of the test but showed an almost immediate return to its original shape. The needle penetration tests indicated significant variability in the consistency of the asphalt binders (58.6 to 136.3 \times 0.1 mm). All the analysed binders are characterized by high resistance to low-temperature cracking, expressed by the Fraass breaking point parameter (approx. -25°C). Asphalt binders used in MS1, MS2, MS3 – MS6 sealants had similar average values of softening point, ranging from 95.3°C to 101.4°C. Slightly lower values were found in binders recovered from the MS3 sealants (81.3°C) and those used in LS1 – LS3 (89.0°C). The lowest average softening point was obtained for the MS7 joint sealant (63.1°C).

On the basis of the analysis of the properties of highly modified bitumen 65/105-80, used in LS1 – LS3 joint sealants, it can be concluded that it is characterized by very favorable parameters, which recommend its use as a basic component for flexible hot-applied joint sealant. The high elasticity of the binder ensures the proper operation of the elastic joint sealants, allowing for the transmission of the significant deformations and strains caused by both vehicle traffic and climatic factors. Excellent low-temperature and high-temperature properties will ensure the required durability of the joint sealant, which is very important in the case of making expansion joints and sealing gaps in the surface.

Table 4. Test results of asphalt binders used in investigated joint and crack sealants

Joint sealant	Property of asphalt binders (mean value ± standard deviation)			
	Needle penetration at 25°C	Softening point, ring, and ball	Fraass breaking point	Elastic recovery at 25°C
Unit	0.1 mm	°C	°C	%
Test method	EN 1426 [39]	EN 1427 [33]	EN 12593 [40]	EN 13398 [41]
Valid N	5	4	3	2
MS1	58.6 ± 3.1	95.3 ± 0.7	< -25.0	99.0 ± 0.5
MS2	136.3 ± 3.7	96.8 ± 0.7	-24.7 ± 0.6	100.0 ± 0.0
MS3	79.6 ± 2.2	81.3 ± 0.4	< -25.0	133 mm; 179 mm*
MS4	134.2 ± 2.8	98.5 ± 1.0	< -25.0	100.0 ± 0.7
MS5	63.0 ± 1.9	101.4 ± 1.8	-24.7 ± 0.6	96.0 ± 0.0
MS6	102.2 ± 4.1	96.2 ± 3.3	< -25.0	97.0 ± 1.4
MS7	93.7 ± 0.9	63.1 ± 0.3	< -25.0	98.0 ± 2.8
LS1 – LS3	73.0 ± 2.1	89.0 ± 0.5	-25.0 ± 0.6	95.0 ± 0.0

* The sample failed before the end of test.

3.1.2. The chemical analyses of the asphalt binder used in joint sealants with the use of FTIR method

The extracted soluble asphalt binders from MS2 – MS7 sealants and highly modified bitumens used in MS1 and LS1 – LS3 sealants were evaluated using FTIR spectra.

Figure 3 presents the 1900 cm⁻¹ to 500 cm⁻¹ absorption bands of the overlaid spectra of the extracted asphalt binder (MS2 – MS7) and modified bitumen (MS1, LS1 – LS3).

The FTIR spectrum for highly modified bitumen 65/105-80 used in LS1 – LS3 sealants is most similar to the asphalt binders used in sealants: MS1, MS2, MS4 and MS5. This indicates the presence of similar chemical compounds in them. Three of these materials (MS1, MS2, MS4) were extracted from the hot-applied joint sealants, classified as high-extension type N1 (flexible), in accordance with the standard [10].

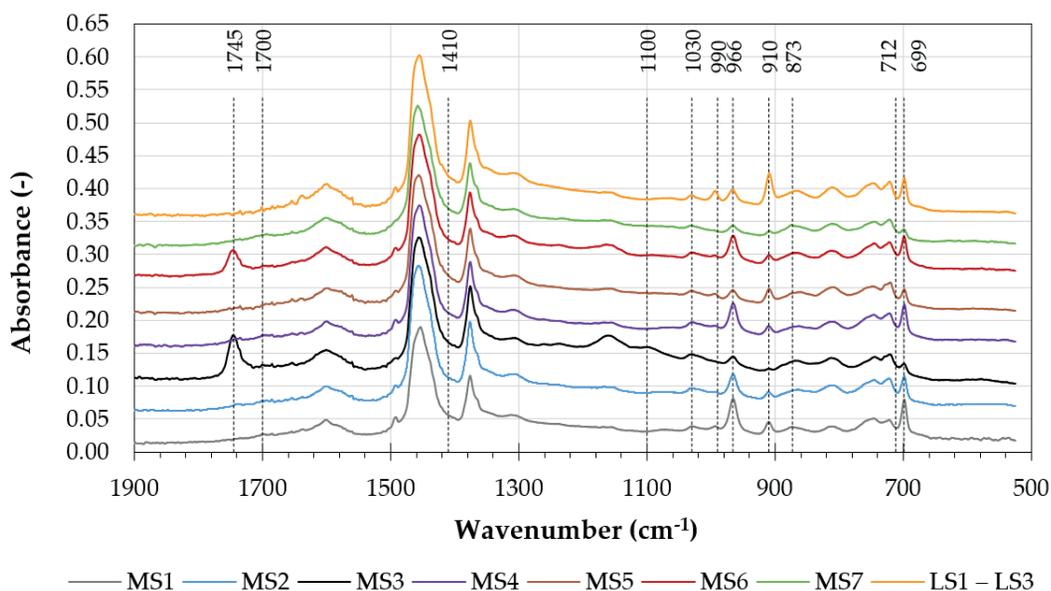


Fig. 3. FTIR spectra of the asphalt binders used in investigated joint sealants

All the FTIR spectra of the extracted asphalt binders and new highly modified bitumen show low, comparable peak heights in the 1700 cm⁻¹ and 1030 cm⁻¹ wavenumber regions associated with carbonyl and sulfoxide compounds, indicating that they were not exposed to a significant amount of oxidation.

All of the bitumen binders exhibited significant peaks at 966 cm⁻¹, indicating the presence of butadiene structures. Clear peaks at 990 cm⁻¹ and 910 cm⁻¹, which are characteristic of vinyl groups, and at 699 cm⁻¹, which is characteristic of styrene structures, have been identified in most binders (except MS7). These responses indicate the presence of styrene-butadiene rubber in the samples of the tested soluble material. Additionally, the asphalt binder extracted from the MS3 and MS6 joint sealants registered prominent peaks at the 1745 cm⁻¹ wavenumber, which is typically seen in bio-oil derivatives (e.g., fatty acid methyl esters) and may correspond to the stretching of the -C=O in ester

functional groups. The MS3 and MS6 bitumens were used in hot-applied joint sealants, classified as low-extension type N2 (normal), in accordance with the standard EN 14188-1 [10].

The above confirms the correct choice of 65/105-80 bitumen for the production of joint sealant with the same properties and application as for the N1 type with high-extension (flexible).

3.2. Comparative analyses of basic properties of joint sealants in relations to the base asphalt binder

3.2.1. The results of basic properties of joint sealants in relations to the base asphalt binder

The evaluations of the penetration at 25°C, the softening point, the Fraass breaking point, and the elastic recovery were performed to measure the effects of different types and compositions of joint sealants on their basic properties. Table 5 presents the results obtained for the investigated joint sealants.

Table 5. Test results of investigated joint sealants

Joint and crack sealant	Property of joint sealant (mean value ± standard deviation)			
	Needle penetration at 25°C	Softening point, ring, and ball	Fraass breaking point	Elastic recovery at 25°C
Unit	0.1 mm	°C	°C	%
Test method	EN 1426 [39]	EN 1427 [33]	EN 12593 [40]	EN 13398 [41]
Valid N	5	4	3	2
MS1	58.6 ± 3.1	95.3 ± 0.7	< -25.0	99.0 ± 0.5
MS2	69.5 ± 1.6	100.3 ± 0.4	< -25.0	96.0 ± 0.7
MS3	36.2 ± 2.5	108.9 ± 0.7	-24.3 ± 0.6	62.0 mm; 48.0 mm*
MS4	65.5 ± 0.9	109.1 ± 0.6	< -25.0	98.0 ± 0.0
MS5	46.3 ± 1.8	104.8 ± 0.4	-21.3 ± 1.5	94.0 ± 0.0
MS6	56.9 ± 5.7	103.2 ± 3.8	< -25.0	97.0 ± 0.0
MS7	78.3 ± 1.6	86.4 ± 1.1	< -25.0	91.0 ± 0.0
LS1/0	66.5 ± 3.0	85.75 ± 0.9	-22 ± 1.3	-
LS1/10	64.86 ± 2.9	87.02 ± 1.0	-21 ± 1.2	94.0 ± 0.5
LS1/10_oil	104.80 ± 1.2	84.82 ± 1.1	-23 ± 1.1	97.5 ± 0.0
LS1/20	63.33 ± 3.0	87.45 ± 1.1	-19 ± 0.6	-
LS1/30	62.0 ± 3.1	88.85 ± 1.2	-18 ± 0.8	-
LS2/0	47.5 ± 1.2	85.9 ± 1.1	-20 ± 1.1	-
LS2/10	46.0 ± 1.5	87.49 ± 1.2	-20 ± 1.4	-
LS2/20	43.25 ± 1.4	88.58 ± 0.9	-18 ± 0.7	-
LS2/30	40.0 ± 1.3	90.46 ± 0.7	-18 ± 0.6	-
LS3/0	21.0 ± 1.1	92.98 ± 1	-19 ± 1.0	-
LS3/10	19.5 ± 0.9	95.26 ± 1.1	-18 ± 0.7	-
LS3/20	17.67 ± 1.2	96.65 ± 0.9	-17 ± 0.4	-
LS3/30	16.75 ± 1.2	97.99 ± 1.1	-17 ± 0.3	-

* The sample failed before the end of test

According to standard [10], the minimum softening point for joint sealants is 85°C.

In the study, needle penetration and elastic recovery were used to assess the characteristics of joint sealant. The applicable standard [10] does not specify the requirements for these tests, but this characteristic was relied on because the purpose of the tests was to compare the same properties of the base bitumen and the joint sealants. For sealants, the required standard test is cone penetration, which should be 40 to 130 × 0.1 mm for the N1 type of sealant and 40 to 100 × 0.1 mm for the N2 sealant type. Elastic recovery is an important parameter that determines the elastic properties of the tested asphalt binder.

The Fraass breaking point parameter is not required according to standard [10], but it is still valuable for the assessment of the elasticity of the tested material at low temperature. Conventional asphalt bridge expansion joints used in low-temperature regions generally show precocious cracking (within the first 2 years) [3]. The manufacturer of sealant MS7 declared compliance with the requirements of the EN 12593 [40] standard with regard to the Fraass breaking point by providing a value of $\leq -30^{\circ}\text{C}$.

On the basis of the analysis of the test results for the LS1 – LS3 joint sealant (Table 5), it can be concluded that the amount of limestone filler has a significant impact on the quality of the sealant, apart from the content of highly modified bitumen 65/105-80.

The use of 60% limestone filler in LS1 – LS3 joint sealants results in an adversely large increase in its brittleness temperature according to Fraass, a significant decrease in needle penetration and an increase in the softening point. The addition of hydrated lime has also a significant impact on the analyzed parameters of the sealants. As its content in the filler increases, the sealant stiffens and is characterized by a decrease in penetration at 25°C and an increase in the softening point. However, with an increase in the concentration of hydrated lime, there is an increase in the brittleness temperature according to Fraass, which is an unfavorable phenomenon, especially with its content of 30%. This type of joint sealants will be very stiff in the pavement working conditions, which in turn will cause its cracking and loss of durability. Lower content of lime filler in the joint sealants has a positive effect on its test parameters. Analyzing the results of the research, it can be concluded that hydrated lime can act as a regulator of the properties of the joint sealants, with its content up to 20% in the filler. The results of the

determinations for the LS1 – LS3 sealants also show that the binder content should not be less than 60% during the production of the sealant. A lower binder content has been used in the MS7 sealant produced by the commercial manufacturer, however, it is a hot-applied sealant, classified as low-extension type N2 (normal), in accordance with the standard EN 14188-1 [10], for which lower requirements are imposed.

The joint sealant containing 80% of highly modified bitumen and 20% mixed filler containing 90% limestone powder and 10% hydrated lime (LS1/10) was qualified for the next stage of the investigating. The sealant with such a composition is characterized by the best parameters among all those performed in the first stage of testing. In order to better understand its properties, an elastic recovery test was also performed.

An attempt was also made to reduce the cracking temperature according to Fraass breaking point with the addition of rapeseed oil in the amount of 3% in relation to the total weight. In this way, a new mixture was obtained, which was subjected to the same tests as the LS1/10 joint sealant without additives, namely penetration, softening point, Fraass breaking point, and elastic recovery. The new sealant was marked with the symbol LS1/10_oil.

The properties of joint sealants in relation to the parameters of the base asphalt binder were presented in Figures 4-7.

For the majority of sealants produced in laboratory conditions (LS1 – LS2) worse low-temperature properties were obtained (higher temperatures according to Fraass) than sealants from manufacturers (MS1 – MS7). The differences were up to 8°C. Unfavorable penetration properties (mean values in the range from 16.8 to 21.0 × 0.1 mm) were obtained for the LS3 sealant.

The penetration and softening point tests indicated that the MS3 sealant was the hardest in consistency from sealants produced by manufacturers, while MS7 was the most susceptible to high temperatures. Among the joint sealants developed in the laboratory, LS3 had the hardest consistency for all 4 variants of hydrated lime content.

For MS3 and LS3, the observed needle penetration results were less than 40 × 0.1 mm, indicating that the cone penetration required by standard [10] was also not guaranteed.

The obtained joint sealants were characterized by high elasticity, as evidenced by the elastic recovery of almost 100% (Fig. 7, Table 5). This means that they

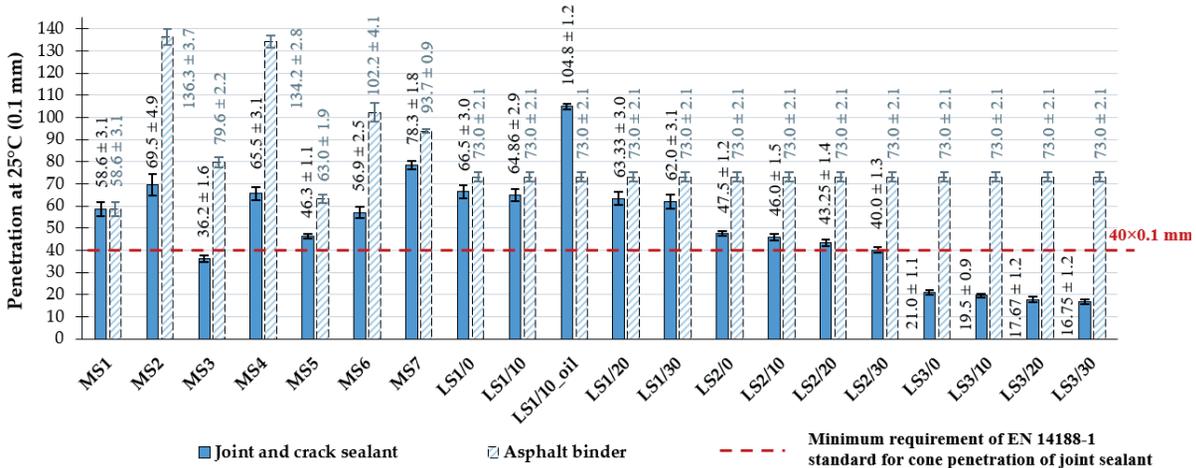


Fig. 4. Results of needle penetration at 25°C of the investigated joint sealants and asphalt binders used in sealants (mean value ± standard deviation)

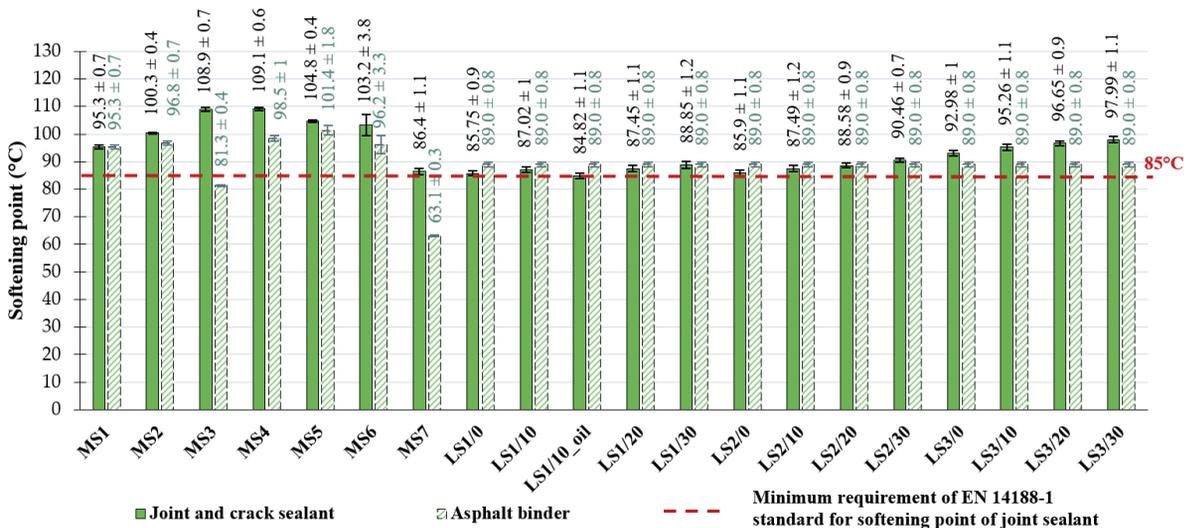


Fig. 5. Results of softening point of the investigated joint sealants and asphalt binders used in sealants (mean value ± standard deviation)

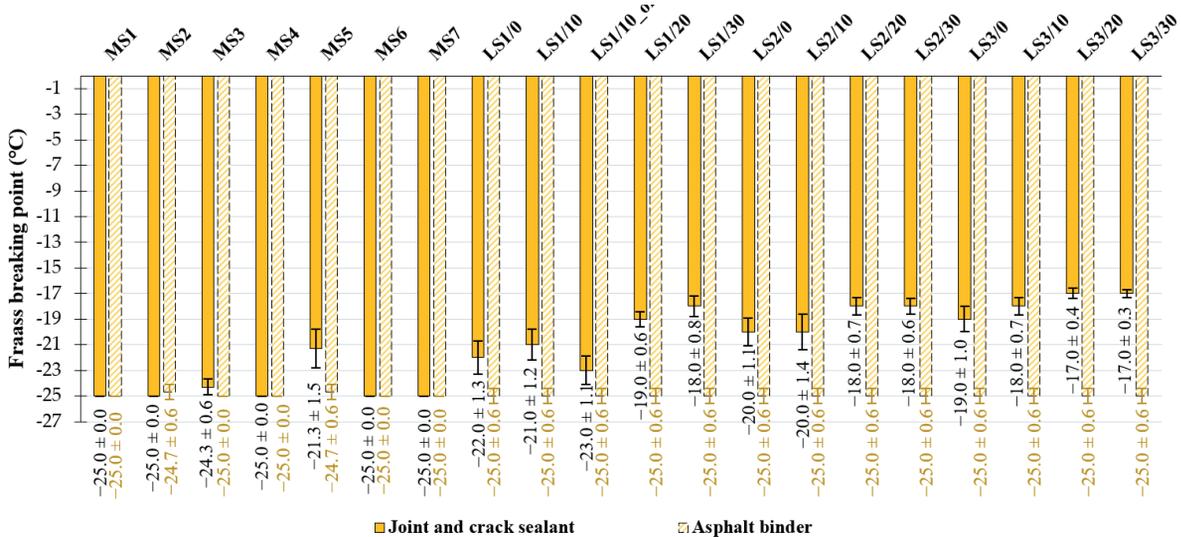


Fig. 6. Results of Fraass breaking point of the investigated joint sealants and asphalt binders used in sealants (mean value ± standard deviation)

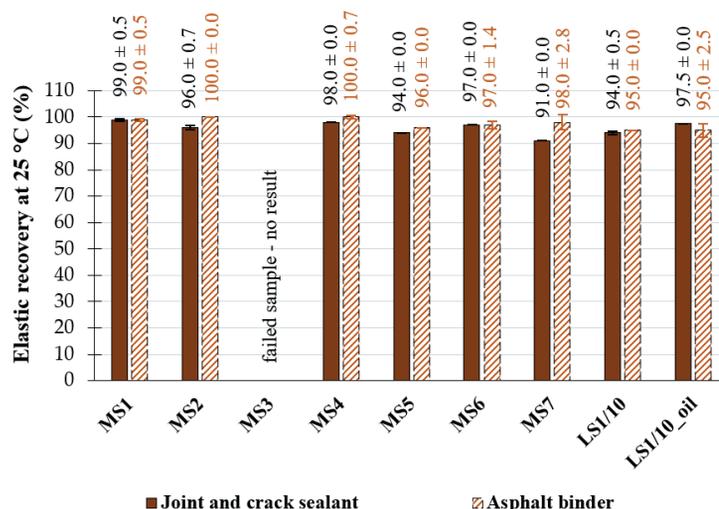


Fig. 7. Results of elastic recovery at 25°C of the investigated joint sealants and asphalt binders used in sealants (mean value ± standard deviation)

can undergo very large deformations without failure, and after reducing (or disappearing) of the stresses transmitted by the structure – they will return to their original shape. In relation to the base bitumen, no significant decrease in this parameter was observed, regardless of the composition of the joint sealant.

The addition of 3% rapeseed oil (in relation to the total weight) results in a favourable reduction of its Fraass temperature by about 2°C (Fig. 6, Table 5). At the same time, a reduction in the softening point was achieved.

A decrease in the average softening point (84.8°C) for the LS1/10_oil sealant was slightly below the parameter required by standard [10]. This indicates the need to extend the scope of investigating for joint sealants with the addition of rapeseed oil taking into account the service life.

3.3. The range of variability of the properties of joint sealants in relation to the base asphalt binder

In order to determine the range of variability of the basic properties of joint sealants in relation to the base asphalt binder, the differences in the values of individual parameters were determined (Table 6). The percentage changes in the values shown in Figures 8 and 9 were also estimated.

The largest range of variability of the investigated parameters was characterized by needle penetration, the differences in relation to the base bitumen ranged from -66.8×0.1 mm (MS2) to 31.8×0.1 mm (LS1/10_oil). Joint sealant with rapeseed oil was the only joint sealant for which an increase in penetration relative to the asphalt binder was recorded.

For the softening point, the largest change in values was recorded for the MS3 sealant (increase

by 27.6°C). For Fraass breaking point, the maximum difference in average values was 8°C for the LS3/20 and LS3/30 sealants.

No significant differences were found between elastic recovery of sealants and base bitumen. The maximum reduction of this parameter concerned the MS7 sealant with a high content of mineral filler of approx. 50%.

In terms of penetration, the highest percentage decrease in value in relation to the asphalt binder was obtained for the MS3 joint sealant (54.5%) among the sealants obtained from manufacturers. In the case of sealants produced in laboratory conditions, the percentage changes in penetration values reached 77.1% for the LS3/30 sealant with the highest content of mineral filler. Smaller percentage differences concerned the softening point and ranged from 0% to 36.9% for MS1 – MS7 and from -13.2% to 10.1% for LS1 – LS3 sealants.

Figure 10 presents the dispersion and nature of changes in the percentage values of the studied parameters depending on the content of asphalt binder in the joint sealants.

With the lowest content of asphalt binder (40%) in the joint sealant, the percentage differences in the properties of the tested sealants in relation to the asphalt binder ranged from -71.2% to -77.1% for needle penetration, 4.5% to 10.1% for softening point and from -24% to 32% for Fraass breaking point. For comparison, with a higher content of asphalt binder in the mass (approx. 60-70%), these differences were characterized by values in the range of -54.5% to -26.5% for penetration, -3.6% to 33.9% for softening point and from -28.0% to 1.2% for Fraass breaking point.

Table 6. Test results of differences in properties of the sealant in relation to the asphalt binder

Joint and crack sealant	Content of asphalt binder	Differences in mean properties in relation to the asphalt binder			
		Needle penetration at 25°C	Softening point, ring, and ball	Fraass breaking point	Elastic recovery at 25°C
Unit	%	0.1 mm	°C	°C	%
Test method	–	EN 1426	EN 1427	EN 12593	EN 13398
Valid N	–	5	4	3	2
MS1	100.0	0.00	0.00	0.0	0.0
MS2	71.3	-66.80	3.50	-0.3	-4.0
MS3	59.0	-43.40	27.60	0.7	0.0
MS4	69.8	-68.70	10.60	0.0	-2.0
MS5	70.7	-16.70	3.40	3.4	-2.0
MS6	72.8	-45.30	7.00	0.0	0.0
MS7	49.8	-15.40	23.30	0.0	-7.0
LS1/0	80.0	-6.50	-3.25	3.0	–
LS1/10	80.0	-8.14	-1.98	4.0	-1.0
LS1/10_oil	80.0	31.80	-4.18	2.0	2.5
LS1/20	80.0	-9.67	-1.55	6.0	–
LS1/30	80.0	-11.00	-0.15	7.0	–
LS2/0	60.0	-25.50	-3.10	5.0	–
LS2/10	60.0	-27.00	-1.51	5.0	–
LS2/20	60.0	-29.75	-0.42	7.0	–
LS2/30	60.0	-33.00	1.46	7.0	–
LS3/0	40.0	-52.00	3.98	6.0	–
LS3/10	40.0	-53.50	6.26	7.0	–
LS3/20	40.0	-55.33	7.65	8.0	–
LS3/30	40.0	-56.25	8.99	8.0	–

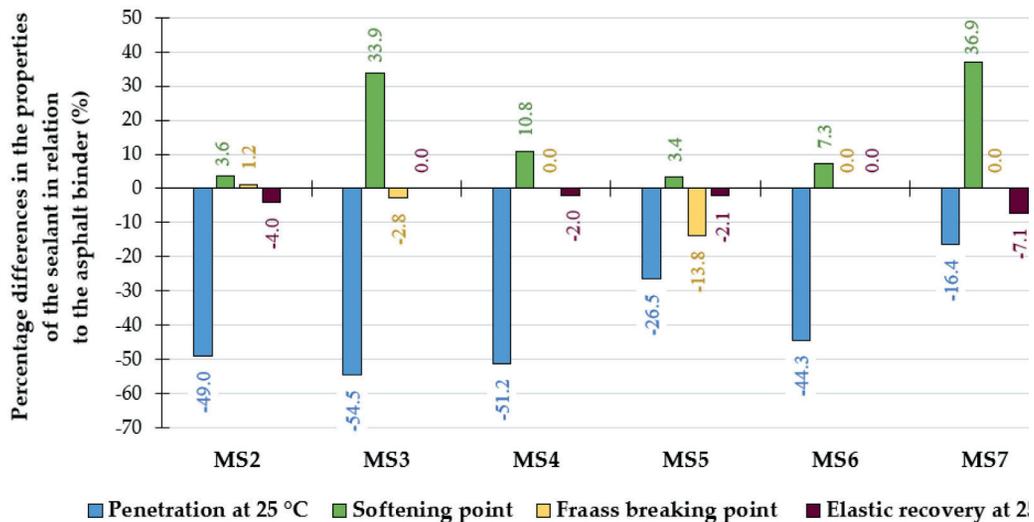


Fig. 8. Results of percentage differences in properties of the MS2 – MS7 sealants in relation to the asphalt binder

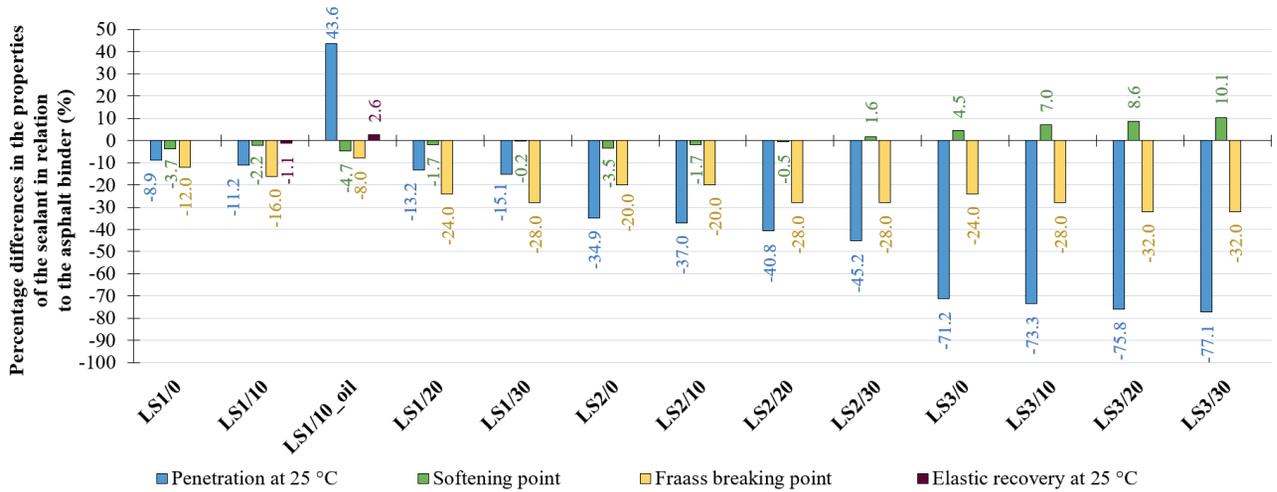


Fig. 9. Results of percentage differences in properties of the LS1 – LS3 in relation to the asphalt binder

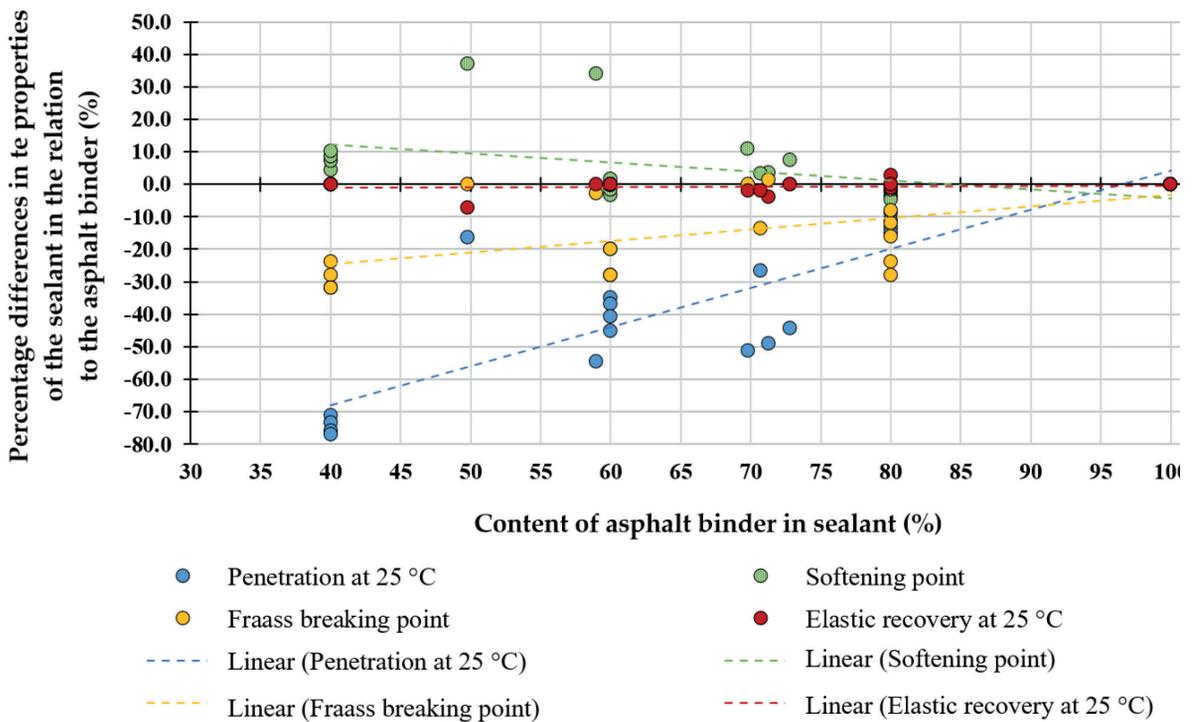


Fig. 10. Results of percentage differences in properties of the sealant in relation to the asphalt binder depending on the content of the asphalt binder

With the increase in the content of asphalt binder in the sealants, smaller percentage differences in the properties of the joint sealant in relation to the asphalt binder were observed. It was found that the greatest influence of asphalt binder content on percentage differences in the properties of the joint sealants in relation to the asphalt binder concerns penetration, while the smallest – elastic recovery, despite the different properties of the other components of the sealant.

3.4. Discussion

Joint sealants are complex formulations and, given a broad range of base asphalt binders, modifiers, fillers, and other non-soluble additives, their composition may vary widely, while still fulfilling the performance specifications. It is possible to regulate the selected properties of the joint sealant by changing the chemical composition and the proportion of its components (fillers, hydrated lime, rapeseed oil, rubber crumb). It is also possible to search for

further material and technological solutions in order to improve the quality and durability of joint sealants used for extension joints and crack repairs.

The tests showed similar properties of the MS and LS sealants in terms of elastic recovery at 25°C (91 to 99%), despite the different contents and chemical compositions of their individual components, the results indicated a very high degree of elastomeric modification of the bitumens used in the sealants and/or the effect of rubber modification, regardless of the sealant type (normal or elastic). The positive effects of the use of polymer SBS and/or crumb rubber in joint sealants were also indicated in other studies [6, 27, 43].

The determined values of the softening point corresponded with the European standard [10] requirements for most investigated hot-applied joint sealants ($\geq 85^\circ\text{C}$). The lowest softening points were determined for the joint sealant in which 3% rapeseed oil was used (84.8°C) and were slightly below the value required by the standard [10].

The needle penetration tests indicated significant variability in the consistency of the joint sealants (16.75 to 104.8×0.1 mm). It should be noted that cone penetration for joint sealants is characterized by lower values than needle penetration, ranging from a few to a dozen or so percent in relations to the needle penetration of the same sealant, as shown by previous studies [36]. The MS3 and LS3 sealants met neither the requirements of the applicable European standard [10] nor the manufacturer's declaration for minimum cone penetration (40 to 100×0.1 mm). Thus, they will not meet the standard requirements [10] in this area.

In the case of LS3 sealant, this is due to the high content of mineral filler (60%).

The percentage range of variability of the basic properties of joint sealants was estimated in the range from -77.1% to 43.6% in relation to the base asphalt binder, differentiated by their composition and the analyzed property.

On the basis of the tests performed, it can be concluded that it is advisable to use highly modified bitumen 65/105-80 as a binder for the joint sealants intended for filling gaps in the pavement or for asphalt expansion joints. It is characterized by parameters comparable to the requirements for the joint sealants currently used in industry and the standard [10], while attention should be paid to the content of filler and other components.

The analysis of the test results showed that, depending on the needs, it is possible to regulate the properties of the sealant by changing the proportions of its components

– binder and filler. Increasing the filler content in the composition of the sealants causes a decrease in penetration (the sealant is more rigid), an increase in the softening point and a slight increase in the Fraass breaking point. Hydrated lime also plays an important role in the composition of the filler, with the help of which the stiffness of the sealant can be adjusted. On the other hand, by increasing or decreasing the binder content of the sealant, its consistency and thus its intended use can be controlled. If it is to fill narrow gaps in the pavement, it should have a more liquid consistency, and therefore a higher binder content, and in the case of using expansion joints to cover expansion joints, its amount should be lower. Hydrated lime used in these investigated sealants may act as an active filler that has antioxidant and other effects, as other studies show [22-24].

On the basis of highly modified asphalt 65/105-80 and lime filler, the flexible joint sealants were made, which can be used for:

- execution of bituminous expansion joints on bridge structures;
- filling expansion joints in asphalt and concrete pavements, also in concrete floors with large spans;
- filling gaps between concrete or stone elements and mineral and asphalt mixture;
- sealing joints between the pavement and devices built into it, such as manhole covers, drain grates, etc.;
- filling and sealing linear cracks and damaged technological joints in asphalt pavements.

A visible discrepancy was observed between the Fraass breaking point performance of the asphalt binders extracted from the MS and LS joint sealants. The differences between the Fraass breaking points of the MS joint sealants and the asphalt binders extracted from them were in most cases indistinguishable. On the other hand, these differences in LS joint sealants were significant and amounted to 8°C increase in this property. Similar difference was observed in case of the MS5 sealant. In case of the LS joint sealants, this result could be attributed to the extraction process and its effect on the polymer network contributing to the performance of the highly modified asphalt binder used in these formulations. Based on this observation, a additional study into the effects of extraction process on the properties of different highly modified asphalt binders should be carried out.

Sealants developed in laboratory conditions (LS) had less favorable properties in terms of resistance to low temperatures (higher Fraass temperature values) than those produced by commercial manufacturers, which indicates the need to expand the scope of

further research in this area. The lack of conclusive requirements for the components of hot-applied joint sealant indicates the need to conduct investigation in this area and to search for additives that are an important regulator of their properties.

4. CONCLUSIONS

The present study considered the basic performance parameters of seven hot-applied sealants produced by domestic and foreign manufacturers and three sealants obtained in laboratory conditions with different contents of highly modified asphalt binder (40 to 100%) and other components. Based on the study, the conclusions were as follows:

- The percentage content and properties of the base bitumen have a decisive influence on the properties of the joint sealants. In order to obtain favorable performance parameters of the joint sealant type N1 (flexible) acc. to European standard [10] it is recommended to use highly modified bitumens with polymers and/or with the use of other substances, e.g. crumb rubber, in order to achieve greater viscosity and higher temperature deformation resistance.
- It was found that it was difficult to ensure the required standard parameters of joint sealants for the content of asphalt binder below 50%, even when using highly modified bitumen. In this case, it is advisable to consider replacing part of the mineral filler with rubber waste, however, this requires verification on the basis of further laboratory tests.
- The addition of hydrated lime and rapeseed oil may be an important regulator of the parameters of joint sealants, in addition to the content and properties of modified bitumen.
- On the basis of the test results for the joint sealant produced in laboratory (LS1 – LS3), it can be concluded that the amount of limestone filler has a significant impact on the quality of the sealant, apart from the content of highly modified bitumen 65/105-80. The joint sealant containing 80% highly modified bitumen and 20% mixed filler containing 90% limestone powder and 10% hydrated lime (LS1/10) was characterized by the best parameters.
- An attempt was also made to reduce the Fraass breaking point with the addition of rapeseed oil in the amount of 3% in relation to the total weight. Due to the fact that the softening point was too low in relation to the requirements of the standard [10] and the needle penetration was significantly increased in relation to the base bitumen, the possibility of reducing the oil content in subsequent laboratory tests should be considered.
- With the increase in the content of asphalt binder in the sealants, smaller percentage differences in the properties of the joint sealant in relation to the asphalt binder were observed. It was found that the greatest influence of asphalt binder content on percentage differences in the properties of the joint sealants in relation to the asphalt binder concerns penetration, while the smallest – elastic recovery, despite the different properties of the other components of the sealant.
- In terms of penetration, the highest percentage decrease in value in relation to the asphalt binder was obtained for the MS3 joint sealant (54.5%) with the highest rubber content among the sealants obtained from commercial manufacturers. In the case of sealants produced under laboratory conditions, the percentage changes in penetration values reached 77.1% for the LS3/30 sealant with the highest content of mineral filler. Smaller percentage differences concerned the softening point and ranged from 0% to 36.9% for MS1 – MS7 and from -13.2% to 10.1% for LS1 – LS3 sealants.

The analysis of the test results showed that, depending on the needs, it is possible to regulate the properties of the sealant by changing the proportions of its components – binder and filler. The lack of conclusive requirements for the components of asphalt-based sealant indicates the need to conduct further research on the properties and composition of joint sealants in terms of their applicability in various climatic conditions and traffic loads. In further studies it would be advisable to extend the analyses with additional [10] standard tests for sealants, e.g. related to cone penetration, penetration and recovery (resilience) and adhesion to the surface.

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