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# EVALUATION OF SELECTED TECHNICAL PROPERTIES OF BITUMEN BINDERS MODIFIED WITH SBS COPOLYMER AND CRUMB RUBBER

# OCENA WYBRANYCH WŁAŚCIWOŚCI TECHNICZNYCH LEPISZCZY ASFALTOWYCH MODYFIKOWANYCH KOPOLIMEREM SBS I MIAŁEM GUMOWYM

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# Abstract

Good quality bitumen used in the production of bitumen-aggregate mixtures is a binder with high stiffness and elasticity at high operating temperatures occurring in summer and adequate flexibility during exposure to sub-zero temperatures. Currently, one of the best technological solutions to improve the viscoelasticity of bitumen and the resistance to ageing is their modification with various types of additives. The paper presents the results of penetration tests as a function of temperature, softening point (ring and ball method) and strain energy at various temperatures of road bitumen modified with SBS (styrene-butadiene-styrene) copolymer, crumb rubber and simultaneous bitumen modification with SBS copolymer and crumb rubber. The obtained results allowed to assess temperature sensitivity, resistance to changes in selected technical properties of the tested binders as a result of technological ageing process with RTFOT (Roler Thin Film Oven Tester) method and to evaluate changes in their technical properties in relation to the 50/70 base bitumen. Multivariate analysis of variance (MANOVA) was used to analyse the impact of the modification type on the test results of technical properties (significance of the impact of the considered factors on the level of technical properties).

Keywords: road bitumen, modified binders, viscoelasticity of bitumen, technological ageing process

# Streszczenie

Dobrej jakości asfalty stosowane do produkcji mieszanek mineralno-asfaltowych to lepiszcza o dużej sztywności, a zarazem sprężystości w wysokich temperaturach eksploatacyjnych występujących latem oraz odpowiedniej elastyczności podczas oddziaływania temperatur ujemnych. Obecnie jednym z najlepszych rozwiązań technologicznych polepszającym właściwości lepkosprężyste asfaltów oraz polepszającym odporność na starzenie jest ich modyfikacja różnego rodzaju dodatkami. W artykule przedstawiono wyniki badań penetracji w funkcji temperatury, temperatury mięknienia PiK oraz energii odkształcenia w różnych temperaturach badania asfaltów drogowych modyfikowanych kopolimerem SBS (styren-butadien-styren), miałem gumowym i jednoczesnej modyfikacji asfaltu kopolimerem SBS i miałem gumowym. Uzyskane wyniki badań pozwoliły na ocenę wrażliwości temperaturowej, odporności na zmiany wybranych właściwości technicznych badanych lepiszczy w wyniku procesu starzenia technologicznego metodą RTFOT (Roler Thin Film Oven Tester) oraz na ocenę zmian ich cech technicznych w odniesieniu do asfaltu bazowego 50/70. Do analizy wpływu rodzaju modyfikacji na wyniki badań cech technicznych (istotność wpływu rozważanych czynników na poziom cech technicznych) wykorzystano analizę wariancji wieloczynnikowej ANOVA.

Słowa kluczowe: asfalty drogowe, lepiszcza modyfikowane, właściwości lepkosprężyste asfaltów, starzenie technologiczne



# 1. INTRODUCTION

The operating properties of bitumen-aggregate mixtures (BAM) are mainly determined by the bitumen used in their production. The binders used to produce BAM should have an appropriate viscoelasticity range. Good quality bitumen is a binder with high stiffness and elasticity at high operating temperatures occurring in summer and adequate flexibility during exposure to sub-zero temperatures. Currently, one of the best technological solutions to improve the viscoelasticity of bitumen and the resistance to ageing is bitumen modification with various types of additives. The technical literature describes many modification methods of bitumen binders, among others: polymers, plastomers, rock asphalt, fly ash, compounds of metallic and organo-metallic salts, latex, rubber, synthetic wax, natural rubber latex, sulphur, lime or nanoparticles [1-5]. However, the most effective improvement results of bitumen technical properties were achieved using polymer additives (especially SBS copolymer) [6, 7] and crumb rubber [8-10].

The dynamically progressing increase in the number of motor vehicles, both in Poland and the world, has caused that, from the point of view of environmental protection, the disposal of worn automotive components, including tyres, has risen to the rank of a very important ecological issue. It has been established that there are about 240 thousand tons of worn car tyres every year in Poland and this number is constantly growing [11]. The need for rapid disposal of stored automotive rubber waste has initiated the development of many technologies leading to their effective recycling. Materials obtained from the processing of worn car tyres are widely used in road construction. Many technologies using rubber waste to build roads are known. One of the ways to reuse rubber waste from worn car tyres is to modify bitumen with crumb rubber. Crumb rubber is a material obtained by grinding worn tyres into particles of less than 1 mm.

The research conducted in Poland and abroad [12-14] have shown that this additive improves the rheological properties of the binder and particularly extends its temperature range of viscoelasticity bitumen-rubber-aggregate [15].The are characterized by improved properties in road operating temperature range: higher fatigue life, improved water resistance and high rutting resistance. The improvement of technical properties of bitumenaggregate mixtures depends on the amount and quality of the rubber additive used in their production and the type of bitumen-aggregate mixture [16]. Bitumenrubber-aggregate mixtures applied to structural layers of roads are characterized by good ability to dampen vibrations caused by vehicle traffic and to reduce traffic noise [17].

The purpose of the conducted tests and analyses was to assess the temperature sensitivity of modified bitumen binders, to analyse changes in selected technical properties of the binder as a result of the ageing process with the RTFOT method and the impact assessment of the type of modifying additive on the technical properties of the binders modified in relation to the 50/70 base bitumen. Multivariate analysis of variance (MANOVA) was used to analyse the impact of the modification type on technical properties of the analysed bitumen binders.

# 2. TESTED BITUMEN BINDERS

The following bitumen binders were used for laboratory tests:

- 50/70 bitumen.
- elastomer bitumen: 50/70 bitumen modified with 5% SBS copolymer (5%) (mark: S-5),
- bitumen-rubber binder: 50/70 bitumen modified with crumb rubber (10%) (mark: G-10),
- rubber-elastomer bitumen binder, obtained by simultaneous modification of 50/70 bitumen with crumb rubber (10%) and SBS (2%) (mark: S-2+G-10).

Bitumen binder samples were prepared in accordance with PN-EN 58 and PN-EN 12594 standards. The bitumen binder modification process involved heating the 50/70 bitumen to  $180^{\circ}$ C ( $\pm 5^{\circ}$ C), then adding the appropriate amount of modifiers. To distribute the additives evenly in the bitumen, the components were mixed in the dryer at a constant velocity of 300 rpm. The effective mixing time was 1 hour for each modification type.

#### 3. TEST METHODOLOGY

For the assessment of the technical properties of modified binders the following laboratory tests were performed before and after the technological ageing

- penetration at test temperatures of 5°C, 15°C and 25°C according to EN 1426,
- softening point with ring and ball method, according to EN 1427,
- strain energy at test temperatures of 5°C, 15°C and 25°C according to EN 13703,

The technological ageing process simulation on bitumen binders in laboratory conditions was



performed with the RTFOT method according to the PN-EN 12607-1 standard.

# 4. TEST RESULTS AND ANALYSIS

#### 4.1. Penetration

The modified bitumen and 50/70 bitumen penetration mark results at test temperatures of 5°C, 15°C and 25°C, before and after the technological ageing process, are shown in Figures 1 and 2.

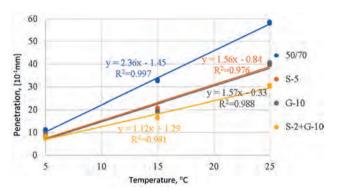


Fig. 1. Penetration test results

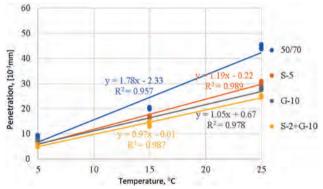


Fig. 2. The penetration test results of binders subjected to RTFOT technological ageing process

On the basis of the obtained results of the 50/70 bitumen, elastomer bitumen (S-5), rubber-bitumen binder (G-10) and rubber-elastomer bitumen binder (S-2+G-10) penetration tests before and after RTFOT technological ageing process at various temperatures, it was found that the modifying additives in the form of SBS copolymer and crumb rubber reduce the bitumen penetration in the entire range of analysed temperatures. It proves that the modifying additives affect the stiffening of the modified binders. The modified binders are much less sensitive to temperature variations in relation to the 50/70 bitumen, which is confirmed by the lower slope value "a" in the given linear functions. It was found that the lowest temperature sensitivity among the analysed bitumen binders is characterized

by rubber-elastomer bitumen binder (S-2+G-10) (trend line slope factor value a = 1.12), followed by elastomer bitumen (S 5) (a = 1.56), rubber-bitumen binder (G 10) (a = 1.57) and 50/70 bitumen (a = 2.37). The penetration results of the tested bitumen binders after RTFOT technological ageing process (Fig. 2) indicated that the process of short-term ageing process reduced their temperature sensitivity.

Table 1 presents an impact assessment of the modification method on the results of the bitumen binder penetration tests. The analysis of MANOVA multivariate analysis of variance was used for the impact assessment. The sum of the effect squares (SS), the mean sum of the error squares (MS), the result of the analysis of variance (F) and the probability level (p-value) were determined. The groups of the same number characterized by a set of values with a standard distribution of their values were compared.

The results of multivariate analysis of variance prove that the modifier type has a significant impact on penetration (significance level p < 0.05). The interaction between temperature and the modifier on the penetration value also proved important. It was found that the best solution is the simultaneous modification with SBS copolymer and crumb rubber (MS = 2697.61), followed by: modification with crumb (MS = 1366.86), modification with SBS copolymer (MS = 1279.32). Based on the test results after RTFOT technological ageing process, it appears that the best solution is a modification with crumb rubber (MS = 1686.48), followed by a modification with a SBS copolymer (MS = 1490.29), and a simultaneous modification with a SBS copolymer and crumb rubber (MS = 1014.31).

# 4.2. Softening point (ring and ball method)

Figure 3 shows the mean softening point (ring and ball method) values of modified bitumen and 50/70 bitumen before and after technological ageing process.

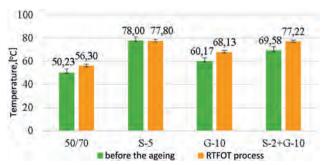


Fig. 3. Results of softening point test of bitumen binders before and after RTFOT technological ageing process



Table 1. Multivariate analysis of variance of modifier type impact on penetration

Modifier type	Ageing	Factor	Sums of Squares	Mean Squares	F-ratio	p-value
SBS	before RTFOT	Temperature	10827.91	5413.95	45659.9	<0.05
		Amount of SBS	1279.32	1279.32	10789.4	<0.05
		Temperature* Amount of SBS	430.94	215.47	1817.2	<0.05
		Error	4.27	0.12		
	after RTFOT	Temperature	6415.05	3207.52	47602.8	<0.05
		Amount of SBS	1490.29	1490.29	7276.4	<0.05
		Temperature* Amount of SBS	290.67	145.33	2156.9	<0.05
		Error	2.43	0.07		
		Temperature	10813.79	5406.89	40004.0	<0.05
	TFOT	Amount of crumb rubber	1366.86	1366.86	10113.0	<0.05
	before RTFOT	Temperature* Amount of crumb rubber	447.94	223.97	1657.1	<0.05
Crumb rubber		Error	4.87	0.14		
Crumb	after RTFOT	Temperature	5868.62	2934.31	38836.4	<0.05
		Amount of crumb rubber	1686.48	1686.48	9085.7	<0.05
		Temperature* Amount of crumb rubber	407.01	203.51	2693.5	<0.05
		Error	2.72	0.08		
	before RTFOT	Temperature	8602.07	4301.04	31691.8	<0.05
		Amount of SBS+crumb rubber	2697.61	2697.61	19877.1	<0.05
SBS + Crumb rubber		Temperature* Amount of SBS+crumb rubber	1012.29	506.15	3729.5	<0.05
		Error	4.89	0.14		
	after RTFOT	Temperature	5455.60	2727.80	39965.4	<0.05
		Amount of SBS+crumb rubber	1014.31	1014.31	14860.8	<0.05
		Temperature* Amount of SBS+crumb rubber	528.52	264.26	3871.7	<0.05
		Error	2.46	0.07		



Table 2. Multivariate d	analysis of varia	ince of modifier to	vne influence on	hitumen hinde	er softening point

Modifier type	Ageing	Factor	Sums of Squares	Mean Squares	F-ratio	p-value
SBS	before RTFOT	Amount of SBS	7502.42	3751.24	10116.75	<0.05
		Error	6.73	0.46		
	after RTFOT	Amount of SBS	5447.95	2724.06	14217.89	<0.05
		Error	3.44	0.26		
Crumb rubber	before RTFOT	Amount of crumb rubber	2141.84	1070.92	8518.73	<0.05
		Error	2.26	0.13		
	after RTFOT	Amount of crumb rubber	1327.34	663.67	3850.01	<0.05
		Error	3.10	0.17		
SBS+crumb rubber	before RTFOT	Amount of SBS+crumb rubber	1328.93	1328.93	6452.62	<0.05
		Error	2.47	0.21		
	after RTFOT	Amount of SBS+crumb rubber	1615.73	1615.73	6500.05	<0.05
		Error	2.98	0.25		

On the basis of the test results of the softening point of 50/70 bitumen, elastomer bitumen (S-5), rubber-bitumen binder (G-10) and rubber-elastomer bitumen binder (S-2+G-10) before and after RTFOT technological ageing process, it was found that the modifying additives in the form of SBS copolymer and crumb rubber significantly increase the softening point temperature in relation to the reference 50/70 bitumen. This is a very positive effect as it can be expected that bitumen-aggregate mixtures with these binders will have a much higher rutting resistance than 50/70 bitumen mixtures. It was found that elastomer bitumen (S-5) (increase by 27.7°C) shows the highest increase in softening point in relation to the 50/70 bitumen, followed by rubber-elastomer bitumen binder (S-2+G-10) (increase by 20°C) and rubber-bitumen binder (G-10) (increase by 9.8°C). The results of the softening point of the tested bitumen binders after RTFOT technological ageing process indicated that the short-term ageing process increases the softening point of rubber-elastomer bitumen binder, rubber-bitumen binder and 50/70 bitumen, but practically does not affect the softening point change of elastomer bitumen (a slight decrease in ring and ball method by 0.2°C).

Table 2 presents an impact assessment of the modification method on the results of the bitumen binder softening point tests.

The results of multivariate analysis of variance prove that the modifier type has a significant impact on softening point (significance level p < 0.05). It was found that the best solution is the modification with SBS copolymer (MS = 3751.24), followed by: simultaneous modification with SBS copolymer and crumb rubber (MS = 1328.93), modification with crumb rubber (MS = 1070.92). The same conclusions can be formulated on the basis of the test results after RTFOT technological ageing process: modification with SBS copolymer (MS = 2724.06), simultaneous bitumen modification with SBS copolymer and crumb rubber (1615.73) and crumb rubber (MS = 663.67).

# 4.3. Strain energy

The modified bitumen and 50/70 bitumen strain energy mark results at test temperatures of 5°C, 15°C and 25°C, before and after the technological ageing process, are shown in Figures 4 and 5. The strain energy is a very good parameter for the quality assessment of bitumen binders [18]. Bitumen with high strain energy is considered to be structurally



more consolidated binders. This test also reflects the proper polymer cross-linking in elastomer bitumen. Bitumen-aggregate mixtures with binders of high strain energy are characterized by higher resistance to the fatigue crackage under road operation conditions.

The test strain energy testing process provides for the sample elongation to 400 mm. It should be noted that the rubber-bitumen binders (G-10) and 50/70 bitumen at test temperatures of 5°C and 15°C have not reached this length both before and after the technological ageing process. The value of the integral (surface area) was adopted for the analysis in the force-elongation constant until the samples break.

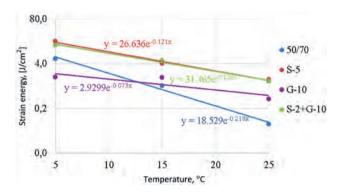


Fig. 4. Strain energy test results

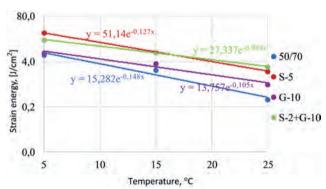


Fig. 5. The strain energy test results of binders subjected to RTFOT technological ageing process

On the basis of the obtained strain energy test results of the 50/70 bitumen, elastomer bitumen (S-5), rubber-bitumen binder (G-10) and rubber-elastomer bitumen binder (S-2+G-10) before and after RTFOT technological ageing process at various temperatures, it was found that the modifying additives in the form of SBS copolymer and crumb rubber increase the bitumen strain energy in the entire range of analysed temperatures. The modifying additives improve structural consolidation of the modified binders. Elastomer bitumen and rubber-elastomer bitumen binder, followed by a rubber-bitumen

binder (except for the marks at 5°C) and 50/70 bitumen are characterised by the highest marked strain energy. The strain energy test results of the bitumen binders after RTFOT technological ageing process (Fig. 5) proved that the process of short-term ageing process increases their strain energy.

The strain energy test results indicate that the modified binders are much less sensitive to temperature variations in relation to the 50/70 reference bitumen. It was found that rubber-bitumen binders, followed by rubber-elastomer bitumen binders and elastomer bitumen are characterised by the lowest temperature sensitivity among the analysed bitumen. The 50/70 bitumen showed the highest temperature sensitivity. The test results after RTFOT technological ageing process slightly change the order of temperature sensitivity: Rubber-elastomer bitumen and rubber-bitumen binders, followed by elastomer bitumen are characterised by the lowest temperature sensitivity. As before the ageing process, the 50/70 bitumen showed the highest temperature sensitivity.

Table 3 presents an impact assessment of the modification method on the results of the bitumen binder strain energy tests.

The results of multivariate analysis of variance prove that the modifier type has a significant impact on energy strain (significance level p < 0.05). The interaction between temperature and modifier on the energy strain value also proved important. It was found that the modification with SBS copolymer (MS = 287.65) showed the greatest impact, followed by: simultaneous modification with SBS copolymer and crumb rubber (MS = 1328.93) and modification with crumb rubber (MS = 3.67). The same conclusions can be formulated on the basis of the test results after RTFOT technological ageing process: modification with SBS copolymer (MS = 662.37), simultaneous bitumen modification with SBS copolymer and crumb rubber (289.80) and crumb rubber (MS = 6.97).

# 5. CONCLUSIONS

On the basis of the penetration tests, softening points (ring and ball method) and the strain energy of bitumen modified with SBS copolymer, crumb rubber and the simultaneous modification of bitumen with SBS copolymer and crumb rubber at different temperatures, the following conclusions were formulated:

1. Modified binders subjected to penetration test showed much lower sensitivity to temperature variations compared to 50/70 bitumen, both before and after the technological ageing process.



Table 3. Multivariate analysis of variance of modifier type influence on strain energy

Modifier type	Ageing	Factor	Sums of Squares	Mean Squares	F-ratio	p-value
SBS		Temperature	799.04	399.52	4464.83	<0.05
	a F	Amount of SBS	287.65	287.65	3214.62	<0.05
	before RTFOT	Temperature* Amount of SBS	221.54	110.77	1237.92	<0.05
		Error	2.14	0.08		
		Temperature	1311.18	655.59	3996.95	<0.05
	<b>∟</b> ⊢	Amount of SBS	662.37	662.37	4038.26	<0.05
	after RTFOT	Temperature* Amount of SBS	532.41	266.20	1622.98	<0.05
		Error	3.93	0.16		
		Temperature	164.07	82.03	1818.09	<0.05
	9 F	Amount of crumb rubber	3.67	3.67	81.49	<0.05
_	before RTFOT	Temperature* Amount of crumb rubber	1.96	0.98	22.21	<0.05
Crumb rubber		Error	1.08	0.04		
rumb	after RTFOT	Temperature	189.59	94.79	4916.89	<0.05
J		Amount of crumb rubber	6.97	6.97	361.55	<0.05
		Temperature* Amount of crumb rubber	1.24	0.62	16.26	<0.05
		Error	0.46	0.02		
	before RTFOT	Temperature	434.01	217.00	2489.78	<0.05
SBS + Crumb rubber		Amount of SBS+crumb rubber	144.77	144.77	1660.99	<0.05
		Temperature* Amount of SBS+crumb rubber	58.62	29.3	336.29	<0.05
		Error	2.09	0.08		
	after RTFOT	Temperature	532.11	266.05	6069.32	<0.05
		Amount of SBS+crumb rubber	289.80	289.80	6611.09	<0.05
		Temperature* Amount of SBS+crumb rubber	96.12	48.06	1096.41	<0.05
		Error	1.05	0.04		

The most favourable values were obtained in case of simultaneous modification of 50/70 bitumen with crumb rubber and SBS copolymer.

- 2. The softening point tests proved that modified binders are characterized by a much higher softening point in relation to the reference 50/70 bitumen (before and after RTFOT ageing process). In this test, the most favourable results were obtained using the 50/70 bitumen modification with SBS copolymer.
- 3. On the basis of the strain energy tests, it was found that the highest determined strain energy is characterized by elastomer bitumen and rubber-elastomer bitumen binders. The modified binders showed less sensitivity to temperature variations in relation to the 50/70 bitumen. The rubber-bitumen binder is characterised by the lowest temperature sensitivity among the analysed bitumen before the technological ageing process, after ageing: rubber-elastomer bitumen and rubber-bitumen binders.



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