

BULLETIN OF THE POLISH ACADEMY OF SCIENCES TECHNICAL SCIENCES DOI: 10.24425/bpasts.2025.153833

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# Three-factor assessment of rutting factor of modified asphalt binders and mastics in the ageing process

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**Abstract**. This paper discusses the research and multivariate analyses of rheological properties of asphalt binders and mastics containing various mineral fillers and SBS copolymer-modified bitumen. In the experimental design employed, three types of mineral filler were used: limestone, basalt or granodiorite. For the purpose of determining the effect of ageing processes on the rheological properties of the obtained mastics, all asphalt binders tested were subjected to short-term (technological) ageing simulated under laboratory conditions using the RTFOT method. The main aim of this study was to evaluate the rutting resistance of modified binders and asphalt mastics depending on such factors as: the type of mineral filler, the percentage of SBS copolymer, temperature, and the influence of the ageing process. A three-way ANOVA was used to test for rutting resistance. The results of laboratory tests and statistical analyses made it possible to assess the significance of the factors examined in the research on the rutting resistance of asphalt binders and mastics, both before and after short-term ageing.

**Key words:** polymer modified bitumen, rutting factor, asphalt mastic, maintenance parameter, Multiple Stress Creep and Recovery test (MSCR), multi-factorial experimental design

# 1. Introduction

The predominant technology applied to pavement construction in the European Union is asphalt. Pavement utilising this technology are susceptible to viscoplastic deformation under load. The increasing expectations of the EU, in particular with regard to ecology, are forcing the Polish road infrastructure to adapt to these standards. The most commonly used material in road pavement construction is asphalt mixture, produced and laid in form of Hot Mix Asphalt (HMA). HMA consists of asphalt binder, aggregate, filler and additives. On average, asphalt binder accounts for merely an average of about 5% of the weight of HMA, yet its influence on the properties of mixtures is decisive under European asphalt road operating conditions. The construction of modern pavements that are durable and resistant to deterioration requires the use of highquality binders that are applied in the correct proportion in the HMA. Among other requirements, asphalt binders should conform to a wide range of operating temperatures. The operating temperatures range from -40°C to +80°C. The standard performance characteristics of binders are inadequate for application across such a wide temperature range, necessitating the development of high-quality binders through modification processes. Consequently, investigating the rheological properties of modified binders is of critical importance. Furthermore, in light of progressively stringent environmental regulations, it is essential to identify asphalt

binders that meet these escalating requirements. Among the most commonly utilized bitumen modifiers are polymers, particularly styrene-butadiene-styrene (SBS). The most popular modified bitumens are: polymer modified bitumen (PMB), highly modified asphalt binder (HiMA), and rubber-modified asphalt binder [1, 2]. During the mixing process of the components comprising asphalt mixture (HMA), a binder with altered properties is formed, known as an asphalt mastic, which contains asphalt binder, mineral filler and fine aggregate fractions. The properties of the mastic rely on the properties of the individual components, in particular the mineral filler used in the HMA, and especially on its structure. Structural characteristics of the mineral filler typically include grain size, void content, specific surface area, methylene blue index and filler density. Monitoring the grain composition of the filler is an essential task during the evaluation process of filler used for HMA production. The functional properties of fillers used in road construction are defined as a group of characteristics related to the behaviour and interaction of the filler in asphalt mastics, and thus in asphalt pavements, on stiffening properties, water resistance or bitumen absorption [3, 4]. The influence of the filler on the stiffening properties of mastic is primarily determined through indirect methods by performing basic tests on mastics and base binders. They are useful in the processes of designing and optimising the asphalt mixture at the initial stage. The stiffening properties have a significant impact on

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workability during HMA production, pavement laying and compaction, as well as the formation of permanent deformations and low-temperature cracking over the service life of the road pavement [3, 5].

The interaction between asphalt binder and mineral filler is largely dependent on the physical properties of the constituent materials in the HMA. According to the papers on the subject [3, 4, 5], the specific surface area and the void content of the filler used come to the fore. The filler is the first component to react with the asphalt binder particles during the HMA production process. The research conducted so far concerned the behavior of asphalt mastic depending on the type and amount of mineral filler. Many types of fillers were used to build asphalt pavements, such as hydrated lime, fly ash, diatomite, Portland cement and limestone filler [6,7,8]. These data showed that the filler used affects the parameters of the obtained mastic. This is due to the fact that the filler, which carries a high surface energy, accumulates positive cation ions, mainly calcium and magnesium, on the surface of the grains. Following the reaction of the cations with the organic acids contained in the asphalt binder, organometallic salts are formed. The strong covalent bonds at the interface between the filler and the asphalt binder lead to the formation of an asphalt mastic. The mastic produced in this manner, which is the suspending medium of the HMA, covers the grains of the mineral aggregate (diversion medium). As a result, the adsorptive properties of the filler should be at an optimum level to prevent an excessive stiffening in the HMA [3]. The rheological properties of mastics are the primary contributor to the behaviour of HMA at operating temperatures. The elastic properties of the mastic ensure that the asphalt pavement is resistant to permanent deformation, pavement fatigue and low-temperature cracking. Song and a team from Southeast University examined mastic containing lime filler and SBS copolymer-modified asphalt binders, using different filler/bitumen F/B (filler/bitumen) ratio values from 0 to 1.8 (by weight) [6]. They carried out a modified test programme in accordance with the MSCR (Multiple Stress Creep and Recovery test) method using shear stresses over a wide range: 0.1; 3.2; 6.4 and 12.8 kPa, and used variable sample heights at the 25 mm spindle (1.0, 1.5 and 2.0 mm). In the end, based on the results of the determination of the parameters of non-recoverable creep compliance (Jnr) and elastic recovery (R) in the MSCR test, they observed that mastics prepared with modified asphalt binders have a higher resistance to rutting. The unmodified bitumen yielded low values of elastic recovery and high values of non-recoverable creep compliance. Based on the results of the elastic recovery difference R<sub>diff</sub>, they calculated the optimum lime filler content in mastic to be 1:1 (by weight) [6]. Furthermore, Cardone and team [7] studied asphalt binders modified with SBS copolymer and used them as foundation for the creation of mastic with lime and basalt fillers. Having examined the properties of asphalt mastic incorporating SBS copolymer-modified asphalt binders, they showed in the Bending Beam Rheometer (BBR) test that the increase in the stiffness modulus in the BBR test for bitumen 50/70 is not dependent on the type of filler used, however, it significantly affects the stiffness of the filler/bitumen/polymer

composite. Moreover, by testing the rheological properties at high temperatures by performing the MSCR test, they found that the addition of filler significantly reduces the value of nonrecoverable creep compliance, thus indicating a higher resistance to permanent deformation of the mastic, which is strongly influenced by the interaction of the filler with the modified asphalt binder [7]. Nevertheless, it must be emphasised that the rheological properties of a mastic, in addition to the viscosity of the base binder used, are also impacted by the structure of the mineral filler used. In paper [4], J. Piłat demonstrated that particular care should be taken in the selection of the filler applied, as this affects the susceptibility of the mastic as a result of varying climatic and loading conditions. The analysis of the HMAs compositions most commonly used in Poland revealed that asphalt binder accounts for a maximum of 15% of the mix volume. On the other hand, in the asphalt mastic obtained, the asphalt binder makes up about 60-80% of the volume, with the filler consisting 20-40%. Thus, it can be noted that the influence of mastic on the HMA final product is very high [8, 9, 10].

This observation justifies the need to assess the rheological properties of the asphalt binders and mastics tested. As a measure for assessing these properties, the authors of this project adopted the RF Rutting Factor. The originality of the research lies in the evaluation of RF values in a three-factor experiment which takes into account filler type, copolymer percentage and temperature, for mastics both before and after ageing. Multivariate analysis of variance (ANOVA) is extensively used in experimental research [11].

Taking into consideration the preservation of natural aggregate deposits, the ecological aspect and the good of the environment, one must emphasise that it is essential to use fillers other than the limestone filler classically employed in Poland. Papers [7, 10] discussed research using fillers such as Portland cement, fly ash obtained from the combustion of hard coal, while dust generated during the dust extraction of aggregates in the production of HMA has been used in [12, 13, 14], and hydrated lime in [9, 10]. In this study, the RF index was evaluated using mineral fillers obtained through the dedusting of basalt and granodiorite aggregates. Rutting resistance was tested with the application of a three-way analysis of variance performed in reference to five levels of SBS copolymer content, three types of fillers and three levels of temperature, before and after ageing.

The paper is structured as follows: section 2 describes the characteristics of the modified asphalt binders and mastics. Section 3 outlines the goals and methodology for rheological properties. Section 4 presents the rutting factor test results, using a three-factor experimental design, before and after ageing. Section 5 summarises key findings, offers conclusions and discusses the article's contribution to research on road pavement durability.

The aim of the research on asphalt mastics is to assess the potential use of mineral fillers, such as dust generated as a by-product of aggregate dedusting during the hot mix asphalt (HMA) production process, as alternative components in asphalt mixtures.

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# 2. The Properties of the Studied Materials

The study used bitumen 50/70 and SBS copolymer-modified bitumen concentrate, as well as three different mineral fillers. Through the mixing of the components in different configurations, asphalt mastic was obtained as end-product. Mastic is an effective binder incorporated in the asphalt mixture and the main determinant of the rheological properties of the resulting HMA [4, 9, 15, 16]. The mastic specimens contained a reference limestone filler (LF) and two mineral fillers differing in acidity, namely granodiorite (GF) and basalt (BF), with grain size below 0.125 mm. The structural properties of the fillers used in the research are shown in Table 1 with a confidence level of 95%. The tests examined SBS elastomermodified bitumen and asphalt mastics. In an effort to analyse the effect of the SBS elastomer content on the rheological properties of the asphalt binders and mastics, the authors developed an asphalt binder modification in laboratory using a polymer modified bitumen concentrate containing SBS Kraton linear block copolymer, with a given copolymer content of (9.0  $\pm 0.2$ )%. Bitumen 50/70 was modified with the application of the compounding method, having been mixed in appropriate proportions with bitumen of given mass content of a block copolymer with a linear SBS structure. The obtained result were modified asphalt binders with a known SBS copolymer content, i.e. 3%, 5% and 7%. In order to evaluate the influence of ageing processes on the properties of modified asphalt binders and mastics, the authors applied a laboratory method simulating short-term (technological) ageing RTFOT (Rolling Thin Film Oven Test), according to EN 12607-1.

Asphalt mastic was formed by mixing asphalt binder with mineral fillers at a filler to bitumen volume ratio of F/B=0.60. Example nomenclature used in the research is as follows: 3%SBS – means asphalt binder modified with an SBS copolymer content of 3%, 5%SBS+LF means asphalt mastic produced using asphalt binder modified with an SBS copolymer content of 5% and limestone filler, 7%SBS RTFOT+GF means asphalt mastic produced with 7%SBS asphalt binder modified with an SBS copolymer content of 7% after RTFOT ageing and granodiorite filler.

TABLE 1. STRUCTURAL PROPERTIES OF THE FILLERS

Structural properties	Unit	LF	BF	GF	Requirements
Specific surface area acc. to <i>Blaine method</i>	Pw[cm <sup>2</sup> /g]	4469 ±35	1806 ±32	2232 ±29	-
Void content acc. to Rigden method	RV [%]	36.5 ±1.3	42.5 ±0.2	42.6 ±0.4	28-45
Density	ρ <sub>f</sub> [g/cm <sup>3</sup> ]	2.71 ±0.04	2.76 ±0.03	2.59 ±0.03	Declared by the manufacturer
Methylene blue indicator	MB <sub>F</sub> [%]	2.3 ±0.2	3.6 ±0.2	2.0 ±0.2	≤10
Increase in softening	∆T <sub>R&amp;B</sub> [°C]	19.9 ±0.9	19.7 ±0.1	15.9 ±0.4	8-25

## 3. Purpose and Methodology of the Studies

The primary objective of the study was to evaluate the rheological properties of the tested asphalt binders and mastics over a wide range of operating temperatures, based on the results of tests conducted by use of a Dynamic Shear Rheometer (DSR), and to assess the impact of the short-term (technological) ageing process of asphalt binders simulated in the laboratory method on changes in the properties of asphalt mastic. The authors carried out an assessment of the resistance of the pavement to the values of the operational parameter, i.e. permanent deformation of the asphalt binders and mastics used in the HMA. A three-factor analysis of the Rutting Factor (RF) was conducted on the basis of prepared material samples before and after ageing with varying SBS copolymer content in the modified bitumen, varying filler acidity and varying measurement temperatures. This analysis was performed in a balanced experiment with four repeated measurements for all level combinations of factors considered.

The research programme involved testing and evaluating the behaviour of asphalt binders and mastics at operating temperatures. The tests were conducted by use of a rheometer DSR of the Physica MCR-101 type. The tests were carried out at varying temperatures; a measuring system composed of two parallel measuring plates with a diameter of Ø8 mm was used. Samples of the test materials, ranging in weight 75-80 mg for asphalt binders, 120-125 mg for granodiorite mastics, and 125-130 mg for limestone and basalt mastics, were weighed on a laboratory balance and rounded to the nearest 0.001 g. The samples filled a gap of 1.5 mm between the moving and stationary plates. A variable measurement temperature was applied, ranging from +82°C to -36°C, with a 1°C decrease in temperature every 1 min. Tests were carried out at an angular frequency of generated oscillations of 10 rad/s and at a logarithmically variable value of oscillation amplitude, starting from 10 mrad at 82°C to 0.1 mrad at -36°C [4, 8, 17, 18, 19]. The research programme also entailed performing the MSCR test (Multiple Stress Creep Recovery). It is a cyclic stress-strain creep test performed by means of a parallel plate system with a diameter of 25 mm and a measuring gap height of 1 mm. The test used a variable temperature, i.e. between 82°C and 46°C with a 6°C increment. At each set temperature, the test consisted of the following stages: creep cycle (loading the asphalt binder sample for a period of 1 s), stress-relieving cycle (unloading the asphalt binder sample for a period of 9 s). A single cycle lasted 10 s and was repeated 10 times. The test was performed at three different shear stress values of 0.1 kPa, 3.2 kPa and 10 kPa. The test began with the lowest applied shear stress value (0.1 kPa), and the specimen being subjected to 10 cycles of loading and unloading, before the stress value was increased and an analogous test was carried out for each applied stress level and measurement temperature. The parameters determined in the MSCR test, i.e. the non-recoverable creep compliance Jnr and the percent recovery R, correlate closely with the rutting resistance of the HMA [13, 14, 20, 21, 22].

To assess the resistance to rutting of the HMA, the authors used the rutting factor determined for the tested asphalt binders and mastics in the high operating temperature range [22], expressed by the formula:

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$$RF = \frac{|G^*|}{\sin\delta}$$

where:

 $|G^*|$  – dynamic shear modulus, Pa  $\delta$  – phase angle,  $^\circ$ 

# 4. Analysis of Test Results

The rheological characteristics of the examined asphalt binders and mastics were determined by performing tests using a rheometer DSR.



Fig.1. Relationship between the dynamic shear modulus and temperature for asphalt binders and mastics containing 3%SBS



Fig.2. Relationship between the dynamic shear modulus and temperature for asphalt binders and mastics containing 7%SBS



Fig.3. Relationship between the phase angle and temperature for asphalt binders and mastics containing 3%SBS



Fig.4. Relationship between the phase angle and temperature for asphalt binders and mastics containing 7%SBS

Figures 1-2 present the temperature dependence of the dynamic shear modulus in the defined operating temperature range, i.e. from -36°C to 82°C, which was determined under sinusoidal kinematic forcing at a constant oscillation frequency  $\omega = 10$  rad/s. The rheological properties of asphalt binder as a viscoelastic material vary depending on the temperature and the applied load. Following the determination of the dynamic shear modulus |G\*| and the phase angle  $\delta$ , it is possible to evaluate the viscoelastic properties of the tested asphalt binders and mastics. In order to avoid the formation of permanent deformations in asphalt pavements at high operating temperatures during the summer, it is advisable to ensure a high value of the dynamic shear modulus and a low value of the phase angle, i.e. the tested



Fig.5. RF rutting factor values determined at 70°C

material should contain a higher proportion of the elastic part than the viscous one [10, 23, 24].

The results of the temperature dependence study for the dynamic shear modulus are shown in Fig. 1-2 on a semilogarithmic scale for two modified asphalt binders used as an example, with 3% SBS and 7% SBS content, both before and after RTFOT ageing. In each case analysed, an increase in test temperature results in a decrease in the dynamic shear modulus value. In all cases, the presence of filler affected the viscoelastic characteristics of the mastic. The greatest stiffening effect was observed when the granodiorite filler was employed, whereas the results of the dynamic shear modulus calculation for the basalt and limestone filler almost overlap. This demonstrates the positive effect of the basalt filler on the rheological properties of the mastic over a wide range of temperatures, extending over the operating temperature range of the pavement, in a range that is similar to the one for limestone filler, commonly used in HMA.

Figs. 3-4 offer examples of graphs illustrating the temperature dependence of the phase angle  $\delta$  for asphalt binders and mastics with 3% SBS and 7% SBS in the modified bitumen. The first observation to make is that for the low temperature range of the test, the value of the phase angle tends asymptotically towards a similar value for the mastic (each case studied) and the baseasphalt binder. This may be due to the fact that the stiffness of the asphalt binder is dominant at low temperatures and thus affects the rheological properties of the mastic [8, 10]. At  $-36^{\circ}$ C phase angle values of less than 10° were obtained. It can



therefore be inferred that all the tested materials develop properties similar to elastic bodies. In contrast, at the high temperature range (above 40°C), a large variation in phase angle values was observed. It was noted that an increase in the SBS copolymer content leads to a significant reduction in the value of the phase angle in the high temperature range. This is most evident for the modified bitumen containing 9% SBS. which reached  $\delta$ =19.9° at 82°C; however, it reached a maximum value of 56.7° at 29°C. A similar relationship can be noted for mastic types produced with 9% SBS content modified bitumen, as in the high temperature range they reach  $\delta$  values ranging from 28.8° for the 9%SBS+LF mastic to 40.1° for the 9%SBS RTFOT+GF mastic. It can therefore be concluded that they exhibit viscoelastic properties with a dominant elastic part. In addition, the reduction of phase angle values in mastic is enhanced by modification with SBS copolymer.

The analysis of Fig. 5 has shown that, as the SBS copolymer content increases in the case of unaged asphalt binders and mastics, the rutting factor value rises from 0.51 kPa for bitumen 50/70 to 26.75 kPa for 9%SBS, and for different mastics from 2.62 kPa for 50/70+BF to 34.12 kPa for 9%SBS+LF. An upward trend can also be noted for each of the mastics examined. The highest RF values among the analysed mastics were reached in almost all instances by the granodiorite filler, the exception being the 9%SBS+GF mastic. By contrast, the examination of RTFOT-aged bitumens indicates that an increase in copolymer content does not result in an increase in rutting factor values, which is especially noticeable for modified asphalt binders containing 5% SBS, 7% SBS and 9% SBS. The upward trend continues for the mastics examined, the exception being the 9%SBS+BF asphalt mastic, as it reached an RF value that was lower by 0.72 kPa after being subjected to RTFOT ageing compared to the material before ageing. To summarise, it was observed that an increase in the SBS copolymer content in the asphalt mastic has a very positive effect on the increase in the rutting factor value of the mastic for each mastic analysed in the study. The resulting rutting factor values in the temperature of 70°C were compared with the Superpave criteria, i.e. RF > 1.0 kPa for unaged bitumen and  $RF>2.2\ kPa$  for RTFOT-aged bitumen. Bitumen 50/70 and 3%SBS both before and after RTFOT ageing do not satisfy these requirements. The remaining modified bitumens and mastics meet the above requirements by a wide margin.

In order to examine the influence of the primary and interaction factors on the rutting factor, the authors carried out a three-factor analysis of variance RF determined at three temperatures of 60°C, 70°C and 80°C in the DSR rheometer. The RF was subjected to an extensive statistical analysis over the upper operating temperatures which can be observed on a pavement during the summer, when the HMA is most susceptible to permanent deformation.

The analysis in question offers an answer to the following question: Are the differences between the expected RF values attributable to random causes only, or are they of systemic nature?

RF measurements are classified according to three independent factors:

A five-level factor SBS determining the SBS copolymer content of the tested material: 0%, 3%, 5%, 7%, 9%.

A three-level factor determining the mastic examined: LF=limestone filler, BF=basalt filler, GF=granodiorite filler.

A three-level factor T determining the measurement temperature:  $60^{\circ}$ C,  $70^{\circ}$ C and  $80^{\circ}$ C.

A full three-factor ANOVA model was applied for both sets of rutting factor measurements

$$Y_{ijkr} = \mu + \alpha_i + \beta_j + \gamma_k + (\alpha\beta)_{ij} + (\alpha\gamma)_{ik} + (\beta\gamma)_{jk} + (\alpha\beta\gamma)_{ijk} + \epsilon_{ijkr}$$
(2)

where:

 $Y_{iikr}$  is the value of the RF variable,

*i* represents group of factor Filler,  $i \in \{LF, GF, BF\}$ ,

*j* represents group of factor SBS,  $j \in \{0, 3, 5, 7, 9\}$ ,

*k* represents group of factor Temperature,  $k \in \{60, 70, 80\}$ ,

r represents the replication number, r = 1, 2, 3, 4.

There are a total of  $n = 3 \cdot 5 \cdot 3 \cdot 4 = 180$  observations of RF.

 $\mu$  is the overall mean,

 $\alpha_i$  are the deviations of groups of factor Filler from the overall mean  $\mu$  due to factor Filler,

 $\beta_j$  are the deviations of groups in factor SBS from the overall mean  $\mu$  due to factor SBS,

 $\gamma_k$  are the deviations of groups in factor Temperature from the overall mean  $\mu$  due to factor Temperature,

 $(\alpha\beta)_{ij}$  is the interaction term between factors Filler and SBS,  $(\alpha\gamma)_{ik}$  is the interaction term between factors Filler and Temperature,

 $(\beta \gamma)_{jk}$  is the interaction term between factors SBS and Temperature,

 $(\alpha\beta\gamma)_{ijk}$  is the second-order interaction between the three variables Filler, SBS and Temperature,

 $\epsilon_{ijkr}$  are the random disturbances. They are assumed to be independent, normally distributed, and have constant variance.

In order to check for normal distribution, the Kolmogorov-Smirnov and Shapiro-Wilk tests were carried out, and the Bartlett, Hartley and Cochran tests were used to test for homogeneity of variance of combinations of factor levels. Overall, the majority of the measurement data groups fulfilled these criteria. However, there were several groups of data that did not meet the assumed requirements. These groups require a separate analysis of the cause of this phenomenon.

The hypotheses about the equality of the mean responses for groups of factor Filler are

 $H_0: \alpha_{LF} = \alpha_{GF} = \alpha_{BF}, H_1:$  at least one  $\alpha_i$  is different,  $i \in \{LF, GF, BF\}.$ 

The hypotheses about the equality of the mean response for groups of factor SBS are

 $H_0: \beta_0 = \beta_3 = \beta_5 = \beta_7 = \beta_9, \ H_1: \text{ at least}$ one  $\beta_i$  is different,  $j \in \{0, 3, 5, 7, 9\}.$ 

The hypotheses about the equality of the mean response for groups of factor Temperature are

 $H_0: \gamma_{60} = \gamma_{70} = \gamma_{80}, H_1:$  at least one  $\gamma_k$  is different,  $k \in \{60, 70, 80\}.$ 



The hypotheses about the interaction of the factors are

 $H_0: (\alpha\beta)_{ij} = 0, H_1: \text{ at least one } (\alpha\beta)_{ij} \neq 0,$  $H_0: (\alpha\gamma)_{ik} = 0, H_1: \text{ at least one } (\alpha\gamma)_{ik} \neq 0,$  $H_0: (\beta\gamma)_{jk} = 0, H_1: \text{ at least one } (\beta\gamma)_{jk} \neq 0,$ 

 $H_0: (\alpha\beta\gamma)_{ijk} = 0, H_1:$  at least one  $(\alpha\beta\gamma)_{ijk} \neq 0$ , In this notation parameters with two subscripts, such as  $(\alpha\beta)_{ij}$ , represent the interaction effect of two factors. The parameter  $(\alpha\beta\gamma)_{ijk}$  represents the three-way interaction.

TABLE 2. COMPILATION OF THE ANOVA CALCULATION RESULTS FOR MASTIC BEFORE AGEING

Analysis of Variance							
Source	Sum Sq.	d.f.	Mean Sq.	F	Prob>F		
Filler	1.32848e+08	2	6.6424e+07	5.24	0.0064		
SBS	1.81209e+10	4	4.53023e+09	357.58	0		
Temperature	6.37214e+09	2	3.18607e+09	251.48	0		
Filler:SBS	8.55032e+08	8	1.06879e+08	8.44	0		
Filler:Temperature	4.81882e+07	4	1.2047e+07	0.95	0.4368		
SBS:Temperature	5.69427e+08	8	7.11783e+07	5.62	0		
Filler:SBS:Temperature	5.35036e+07	16	3.34398e+06	0.26	0.9981		
Error	1.71033e+09	135	1.26691e+07				
Total	2.78624e+10	179					

TABLE 3. COMPILATION OF THE ANOVA CALCULATION RESULTS FOR MASTIC AFTER AGEING RTFOT METHOD

Analysis of Variance								
Source	Sum Sq.	d.f.	Mean Sq.	F	Prob>F			
Filler	1.32303e+08	2	6.61516e+07	3.14	0.0465			
SBS	1.79929e+10	4	4.49824e+09	213.44	0			
Temperature	2.60405e+10	2	1.30203e+10	617.82	0			
Filler:SBS	1.12487e+09	8	1.40609e+08	6.67	0			
Filler:Temperature	1.63559e+08	4	4.08896e+07	1.94	0.1073			
SBS:Temperature	1.44484e+09	8	1.80605e+08	8.57	0			
Filler:SBS:Temperature	1.94444e+08	16	1.21528e+07	0.58	0.8969			
Error	2.84507e+09	135	2.10746e+07					
Total	4.99386e+10	179						



Fig.6. Correlation of rutting factor RF with applied filler X1, SBS copolymer content X2 and measurement temperature X3 for unaged mastics



**Fig.7.** Correlation of rutting factor RF with applied filler X1, SBS copolymer content X2 and measurement temperature X3 for RTFOT-aged mastics

The results of the study to determine the pre-ageing RF rutting factor using the RTFOT method in the three-factor design examined provide the foundation for ascertaining the significance of the influence of the three factors (SBS content, filler type and temperature) on the pre-ageing RF variable RTFOT, Table 2.Given that the calculated significance level

values (value p=Prob>F) are less than the calculated significance level value ( $\alpha$ =0.05), the authors confirmed a significant effect of the levels of each of the SBS, F and T factors on the tested rutting factor. In contrast, no significance was found for the influence of three factors on the rutting factor (p=0.9981). The results of the determination of the RF rutting factor after the base asphalt binder being subjected to RTFOT-ageing support the existence of a significant interaction between the SBS content and the filler used or the measurement temperature. In this experimental design, similarly to the case before ageing, no significant interaction was found between the three factors (p=0.8969), Table 3.

The obtained ANOVA results presented in Table 2 refer to the mastic before ageing. The following conclusions for the main effects can be drawn from these results. The Filler (type) factor is statistically significant, as p=0.0064. This means that the levels of the Filler factor differ significantly in terms of their effect on the rutting factor (RF). The SBS (content in asphalt binder) factor is highly statistically significant, as p=0.0000. This indicates significant differences between the levels of the SBS factor. The Temperature factor is also highly statistically significant. This indicates a large effect of temperature on the rutting factor. In contrast, the results of the two-way interaction test indicate that the interaction between Filler and SBS is significant, p=0.0000. This indicates that the effect of Filler type on the rutting factor depends on the level of SBS content. Similarly, the interaction between SBS content and Temperature is significant. This indicates that the effect of SBS content depends on the level of temperature. In contrast, the interaction between Filler type and Temperature is not statistically significant (p=0.4368). This indicates that the effect of the Filler factor on the rutting factor does not vary significantly with temperature. It is also revealing that the threeway interaction is not statistically significant (p=0.9981). This indicates that no specific pattern of interaction between the three factors significantly affects the rutting factor. To summarize the case of mastics containing asphalt binders before ageing, the ANOVA suggests that the effects of Filler type, SBS content, and Temperature on the rutting factor are independent in most cases, but that SBS content and Temperature may modulate each other's effects.

Interestingly, the results of the three-way ANOVA for the mastics containing asphalt binders after aging shown in Table 3 lead to the same conclusions as for the unaged ones. This means that the aging process did not change the significance of the effects on the rutting factor of both the main and interaction factors.

Figures 6 and 7 collectively present the correlations of RF with respect to the filler used X1, the SBS copolymer content X2 and the test temperature X3 in the tested mastics both before and after RTFOT ageing. The analysis of the first variable X1





before and after RTFOT ageing shows that granodiorite filler exhibits the highest RF values at different SBS copolymer contents (the exception being 9%SBS), but also exhibits RF values close to LF at varying measurement temperatures. The examination of the second variable X2, namely the SBS copolymer content of the tested mastic, indicated that a higher copolymer content in the mastic results in better rutting resistance both before and after RTFOT ageing. The third variable is the measurement temperature X3, as with increasing measurement temperature the susceptibility to permanent deformation also increases.

Rutting resistance can also be evaluated by means of the MSCR test, informed by parameter J<sub>nr</sub>. It is very common for researchers to question the existence of a correlation between RF rutting factor and resistance to permanent deformation formation in HMAs [6, 10, 24], in particular for polymermodified bitumen. Therefore, ASTM D7405 standard was introduced in 2010 to address MSCR testing using a DSR rheometer. Parameters obtained in the test are  $J_{nr}$  and R. According to [7, 24, 25, 26, 27], for polymer-modified asphalt binders, and even more so for asphalt mastics, the research indicated a strong correlation between parameters J<sub>nr</sub> and R, and the resistance to permanent deformations of HMA. Consequently, the decision was made to check the relationship between the RF rutting factor values and parameter  $J_{nr}$  at 70°C for the tested asphalt binders and mastics before and after RTFOT ageing. The relationship was shown in Figures 8 and 9. In the case of SBS copolymer-modified asphalt binders not subjected to ageing, it can be observed that this is a linear relationship ( $R^2=0.99$ ); an increase in the SBS copolymer content results in an increase in the rutting factor and a decrease in the value of parameter J<sub>nr</sub>. A strong relationship between these two parameters also exists in mastics, the highest for granite-filled mastic ( $R^2 = 0.95$ ) and the lowest for basalt-filled mastic ( $R^2 = 0.84$ ) respectively.

Smaller values of the coefficient of determination  $R^2$  were recorded for asphalt binders and mastics after RTFOT ageing,



Fig.8. Correlation of parameters  $J_{nr}$  and RF at 70°C for unaged asphalt binders and mastics



Fig.9. Correlation of parameters  $J_{nr}$  and RF at 70°C for RTFOT-aged asphalt binders and mastics

although it should be emphasised that a positive effect of modification with the SBS copolymer on the rutting resistance parameters of the mastics obtained was still observable. In this case, the coefficient of determination  $R^2$  reached the highest value for mastics containing granodiorite filler GF ( $R^2 = 0.79$ ) and the lowest value for mastics containing limestone filler LF ( $R^2 = 0.64$ ). In the case when the number of observations is n = 5, the critical value of the coefficient of determination  $R^2$  (at the significance level  $\alpha = 0.05$ ) is 0.77. Therefore, in two cases: LF RTFOT ( $R^2 = 0.64$ ) and BF RTFOT ( $R^2 = 0.66$ ), no significant correlation was found at  $\alpha = 0.05$ .

# 5. CONCLUSIONS

The results of laboratory testing and subsequent analysis lead to the following conclusions.

#### 1. Viscoelastic Properties of Modified Bitumens

All modified bitumens incorporating SBS copolymer exhibit pronounced viscoelastic behavior across a broad operating temperature range, as demonstrated by dynamic shear rheometer (DSR) tests conducted between  $-36^{\circ}$ C and  $82^{\circ}$ C. Asphalt mastics produced with these modified bitumens exhibit significantly enhanced elasticity compared to unmodified 50/70 road bitumen. This improvement is attributed to the incorporation of SBS copolymer in their structure. Furthermore, basalt and granodiorite fillers incorporated into the mastics display rheological properties comparable to those of traditional limestone fillers.

## 2. Reduced Susceptibility to Permanent Deformation

The inclusion of SBS copolymer markedly decreases the susceptibility of the tested asphalt mastics to permanent deformation. This is evidenced by an increase in the rutting factor (RF) and a reduction in the non-recoverable compliance parameter (Jnr).

## 3. **Influence of Multiple Factors on Rutting Resistance** A comprehensive evaluation of rutting resistance, quantified

via a three-factor analysis of variance, highlights the



significant influence of SBS content, filler type, and temperature on the rutting factor (RF). Additionally, significant interaction effects were observed between various pairs of factors, underscoring the importance of examining their combined impact on rutting resistance.

## 4. Sustainability in Mineral Filler Selection

From an ecological and resource conservation perspective, mineral fillers derived as by-products of aggregate dedusting during the hot mix asphalt (HMA) production process offer a viable alternative as mixed fillers in asphalt mixtures. Their use aligns with the goals of preserving natural aggregate reserves.

#### REFERENCES

- K. Shi, Z. Fu, F. Ma, J. Liu, R. Song, J. Li, J. Dai, Ch. Li, Y. Wen. "Development on the rheological properties and micromorphology of active reagent-rejuvenated SBS- modified asphalt" ACS Sustain. Chemistry&Eng., 10(50), 16734-16751 (2022), DOI: 10.1021/acssuschemeng.2c05008
- P. Ostrowski, A. Zofka, K. Błażejowski, M. Pszczoła. "Asphalt pavement structure optimization with alternative materials". *Arch. Civil Eng.*. 3:191-203. (2024) doi:10.24425/ace.2024.150978.
- 3. W. Grabowski, "Structure and functional properties of mineral fillers used in road construction", *Publishing house Poznan Univ. of Technol.*, Poznan (2007)
- J. Piłat. "The influence of mineral filler properties on shaping the technical characteristics of mineral-asphalt composites", *Publishing house of Warsaw Univ. Technol.*, Warszawa (1994)
- X. Yang, Z. Fan, J. Xu, J. Lin, Y. He, D. Wang, P. Liu . "Investigation of graphite tailings as a substitute for filler in asphalt mastics". *Int. J. Pavement Eng.*, 24(1), (2023) 10.1080/10298436.2023.2172578.
- L. Song, N. Fujian, D. Qian, Z. Zili, M. Xiang. "Effect of filler in asphalt mastic on rheological behaviour and susceptibility to rutting". *Int. J. of Pavement Eng.*; 22(1): 87-96, (2019) 10.1080/10298436.2019.1577423.
- F. Cardone, F. Frigio, G. Ferrotti, F. Canestrari, "Influence of mineral fillers on the rheological response of polymer-modified bitumens and mastics", *J. Traffic Transp. Eng.*, 2(6): 373-31(2015) doi.org/10.1016/j.jtte.2015.06.003
- G. Mazurek. "Linear and nonlinear viscoelastic characteristics of asphalt mastic at high pavement operating temperatures". *Monographs*, *Studies, Dissertations no. M118*, Kielce Univer. of Technol., Kielce (2019)
- G. Liu, T. Yang, J. Li, Y. Jia, Y. Zhao, J. Zhang. "Effects of aging on rheological properties of asphalt materials and asphalt-filler interaction ability". *Construction Building Mater.*, 168: 501-511 (2018) doi.org/
- G. Mazurek, M. Pszczoła, C. Szydłowski. "Non-linear mastic characteristics based on the modified MSCR (Multiple Stress Creep Recovery) test". *Struct. Environ.* 11(1), 23-34 (2019). 10.30540/sae-2019-002
- A. Niewczas, K. Andrzejczak; Ł. Mórawski ; E. Dębicka. "Study of Phase Changes in Operational Risk for Trucks". *Energies*; 17 (9), 2143, (2024) 10.3390/en17092143.
- J. Zhang, G. Liu, C. Zhu, J. Pei. "Evaluation indices of asphalt–filler interaction ability and the filler critical volume fraction based on the complex modulus". *Road Mater. Pavement Des.*; 18(6),1338–1352 (2016), 10.1080/14680629.2016.1218789.
- Y. Bi, F. Guo, J. Zhang, J. Pei, R. Li, "Correlation analysis between asphalt binder/asphalt mastic properties and dynamic modulus of asphalt mixture", *Construction and Building Mater.s*, 276(122256), ISSN0950-0618 (2021) doi.org/10.1016/j.conbuildmat.2021.122256

- F. Li, Y. Yang, L. Wang. "The interfacial interaction between asphalt binder and mineral filler: a comprehensive review on mechanisms, evaluation methods and influence factors". *Int. J. Pavement Eng.*, 23(12), 4291–4305 (2021) https://doi.org/10.1080/10298436.2021.1942468
- J. Zhang, L.F. Walubita, A. N.M. Faruk, P. Karki, G.S. Simate." Use of the MSCR test to characterize the asphalt binder properties relative to HMA rutting performance – A laboratory study". *Construction Building Mater*. 94,218-227.(2015) https://doi.org/10.1016/j.conbuildmat.2015.06.044
- A. Zofka, I. Yut. "Correlation between rheology and chemical composition of aged polymer-modified asphalts". *Construction Building Mater.*; 62,109-117, ISSN 0950-0618, (2024) 10.1016/j.conbuildmat.2014.03.043
- M. Mielczarek, S. Dziadosz, M. Słowik, M. Bilski. "The assessment of the influence of styrene-butadiene-styrene copolymer on the selected rheological properties characterising durability of modified bitumens used in road pavements". *Bull. Pol. Ac.:Tech.*;68(6):1471-1479 (2020). 10.24425/bpasts.2020.135391
- G. Mazurek. "Effect of filler type on non-linear viscoelastic characteristics of asphalt mastic". Arch. Civil Eng.. 67, 2 (2021) 10.24425/ace.2021.137166
- H. Al-Bayati, A.G. Oyeyi, S. Tighe. "Experimental assessment of mineral filler on the volumetric properties and mechanical performance of HMA mixtures". *Civ Eng J*, 6(12), 2312-2331. (2020) 10.28991/cej-2020-03091619
- G. Liu, T. Yang, J. Li, Y. Jia, Y. Zhao, J. Zhang. "Effects of aging on rheological properties of asphalt materials and asphalt-filler interaction ability". *Construction Building Mater.*; 168: 501-511 (2018) doi.org/10.1016/j.conbuildmat.2018.02.171
- M. Mielczarek, S. Fornalczyk, M. Słowik. "Evaluation of the rheological properties of polymer-modified asphalt binders and mastics with organic additive – imidazoline", *Sustainability*, 16(4),1434 (2024) https://doi.org/10.3390/su16041434
- M. Mielczarek, M. Słowik, K. Andrzejczak. "The assessment of influence of styrene-butadiene-styrene elastomer's content on the functional properties of asphalt binders". *Eksploatacja i Niezawodność* - *Maintenance Rel.*; 22 (1): 148–153 (2020), 10.17531/ein.2020.1.17
- Y. Jia, G. Liu, Y. Gao, S. Wang, Z. Li, X. Wang, L. Yan, "Micromechanics prediction of dynamic modulus for asphalt mastic considering filler distribution characteristic: A new perspective", *Construction and Building Mater.s*,364(129916),ISSN0950-0618(2023) doi.org/10.1016/j.conbuildmat.2022.129916
- 24. J. Król, P. Radziszewski, K. Kowalski, M. Sarnowski. "Assessment of an appropriate modifier content in modified bitumen based on the multiple stress creep recovery test", *3rd International Conference on Road and Rail Infrastructure* (2017)
- B.Xing, Ch. Fang, Z. Gan, J. Yang, Z. Li, Y. Lyu, W. Fan. "Evaluation of particle morphology and size of mineral filler and their effects on mastic rheological and creep behavior: Experimental and numerical characterizations" *J. Mater. Res. Technol.*, 30 613-625,ISSN2238-7854, https://doi.org/10.1016/j.jmrt.2024.03.104
- M. Guo, Y. Tan. "Interaction between asphalt and mineral fillers and its correlation to mastics' viscoelasticity" *Int. J. Pavement Eng.*, 22(1), 1-10, (2019) doi.org/10.1080/10298436.2019.1575379
- A. Faheem, H. Bahia. "Modelling of asphalt mastic in terms of fillerbitumen interaction", *Road Mater. Pavement Des.*, 11, 281-303 (2010) doi.org/10.1080/14680629.2010.9690335