Petroleum & Coal
ISSN 1337-7027

Available online at www.vurup.sk/petroleum-coal
Petroleum & Coal 57(6) 650-660, 2015

CRITERIA FOR EVALUATION OF UNMODIFIED AND MODIFIED BITUMENS

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Received December 3, 2015; Accepted December 28, 2015

Abstract

Unmodified and modified bitumen were evaluated by determination of softening point, penetration and dynamic viscosity. Bitumen was modified by crumb rubber. Properties of modified and unmodified bitumen were compared. The effectiveness of bitumen modification is reflected in properties change of the prepared mixture in comparison to the properties of unmodified bitumen. Determination of penetration and softening point gives possibility of calculation of the temperature susceptibility and penetration index of unmodified and modified bitumen. Softening point, penetration, penetration index, viscosity and relative viscosity increment were investigated as the performance criteria for evaluation of bitumen modified by crumb rubber. Results showed that the values of viscosity and relative viscosity increment are the most suitable for modifier effectiveness evaluation.

Keywords: Bitumen; crumb rubber; penetration index; temperature susceptibility; viscosity; relative viscosity increment.

1. Introduction

Modified bitumens are widely used in road mixtures. The changes of modified bitumen properties are connected with the interactions of the polymer with compounds present in bitumen. The polymeric modifiers have significant effect on the cost of the bitumen mixture. An extremely important factor for the modified bitumen preparation is the optimal concentration of the modifier. Modifier in optimal concentration in bitumen improves the properties of the final product to the required quality at minimum costs. The change of the modified bitumen properties is monitored by the measurements of the chosen physical variables. The determination of the softening point temperature (tRB) [1] and penetration [2] are common methods for the bitumen properties evaluation in practice. Laboratory methods include determination of the resistance to hardening under the influence of heat and air [3-5] determination of the affinity between aggregate and bitumen [6] and especially rheological measurements [7-10].The bitumen character and efficiency of modification can be assessed based on the standard parameters which involve temperature dependences of viscosity parameters.

Pfeiffer and Van Dormaal [11] have introduced for bitumens a temperature susceptibility *A* as the difference of logarithms of bitumen penetration at two different temperatures recalculated to the temperature change of 1°C (equation 1):

$$A = (\log pen_1 - \log pen_2)/(t_1 - t_2) \tag{1}$$

where pen_1 is penetration at temperature t_1 and pen_2 is penetration at temperature t_2. Penetration index (*PI*) was defined using temperature susceptibility (equation 2).

$$PI = (20 - 500 \cdot A)/(1 + 50 \cdot A) \tag{2}$$

The value of penetration index is used for classification of bitumen to three groups by character of the internal structure. Bitumens which have $PI > 2$ are referred as gel-type. When the value of penetration index is $PI < -2$ bitumens are classified as sole-type. Road bitumens have usually *PI* in the range of -2 to +2 inclusive. This group of bitumens is classified as sole – gel type [15-16].

Pfeiffer and van Dormaal [11] found that most of bitumens have penetration of 800 penetration units (p.u.) at the softening point temperature. In equation (1) they replaced temperature t_2 by the softening point temperature t_{RB} and penetration pen_2 at the temperature $t_2 = t_{RB}$ by the value of 800. According to this finding it is possible to calculate the penetration index PI_{RB} using the penetration measure at temperature 25°C (pen_{25}) and using the softening point temperature t_{RB} according to equation (3).

Pfeiffer and van Dormaal [11] have accepted assumption that at the softening point temperature the most bitumen has penetration of 800 penetration units (p.u.). According to the authors the temperature t_2 in equation (1) is substituted by the temperature of softening point t_{RB}. The value of 800 p.u. is given for penetration pen_2 at the temperature t_{RB} According to this finding it is possible to calculate the penetration index PI_{RB} using the penetration at temperature of 25°C (pen_{25}) and the softening point temperature t_{RB} according to equation (3).

$$PI_{RB} = (1952 - 500 \cdot \log(pen_{25}) - 20 \cdot t_{RB}) / (50 \cdot \log(pen_{25}) - t_{RB} - 120) \quad (3)$$

Penetration index calculated from penetration at 25°C and t_{RB} often occurs as a quality parameter of bitumen [12-13,17-18]. The verification of the assumption that the penetration at temperature t_{RB} has value 800 p.u. is possible through comparison of the measured temperature t_{RB} and the calculated temperature t_{800} (t_{800} = temperature at which bitumen acquires penetration 800 p.u.). Temperature t_{800} is calculated from temperature susceptibility (A), which is obtained from equation (1) at a suitable temperature difference interval, for example 35- 25°C. Formula for calculation of t_{800} is shown in equation (4).

$$t_{800_{Pen}} = t_1 + (1/A) * \log(800/pen_1) = 25 + (1/A) * \log(800/pen_{25}) \quad (4)$$

Relationship between penetration and viscosity represents a frequent topic of investigation. Dependencies of penetration on bitumen viscosity [19-21] are the result of comparison of the forces acting against the movement in the fluid. Temperature susceptibility stands for the slope of the logarithmic dependence of penetration on temperature. Linear dependencies of viscosity of petroleum fractions on temperature are also defined by logarithmic dependences on absolute temperature [22-23]. Therefore the temperature dependencies of viscosity and penetration of bitumen are linear in a relatively narrow temperature range [20,25-26]. The interactions of polymer with solvent in practice observed not only as changes of viscosity with temperature [27-28], but mainly as the relative viscosity or as relative viscosity increment [29-31]. Viscosity ratio η_{rel} is defined as the ratio of viscosity of the polymer-solvent mixture η and of the viscosity of the pure solvent η_0 (equation 5).

$$\eta_{rel} = \eta / \eta_0 \quad (5)$$

The relative viscosity increment η_i of a modified bitumen mixture is defined by equation (6).

$$\eta i = (\eta - \eta_0)/\eta_0 = (\eta/\eta_0) - 1 \quad (6)$$

The relative viscosity increment expresses a measure of the polymer contribution to the viscosity of the mixture and can be expressed as percent value. The viscosity ratio increment can be used for characterization of interaction degree between the bitumen and the modifier such as crumb rubber (CR).

As it was mentioned above penetration index is frequently used in research papers [12,15,18] as a parameter for evaluation of the bitumen temperature susceptibility. A significant drawback of the published results is the absence of the probability intervals, within which the calculated penetration index can be observed. As it is indicated in report [20] penetration indices do not always follow the expected relation between penetration index and the change in bitumen properties.

The aim of the study was to compare the effect of changes in penetration and softening point on the values of temperature susceptibility and penetration index of bitumens taking into account the repeatability of the methods. The suitability of penetration and softening point for evaluation of the degree of interactions between crumb rubber and bitumen was compared with relative viscosity increment.

2. Experimental

The penetration measurements were performed on Analis (P731) equipment. Penetration measurements were performed according to European standard EN 1426 and three repeated injections were done with each sample. Softening point was measured according the standard EN 1427 by using the Petrotest Instrument (SUR). The standard prescribes two parallel measurements. The instrument RV2 HAAKE was used for the measurement of dynamic viscosity. The Broockfield viscosity standard B200 (accuracy: ±1% of viscosity value) was used for determination of maximal standard deviation. The average dynamic viscosity 218,3 mPa.s of the standard B200 was calculated from eight experiments and it reached the maximal standard deviation ±1,3%. Two measurements of dynamic viscosity were performed with each sample using the MV-II system. The system was tempered by heating circulator Julabo MA-4.

2.1 Bitumen

Bitumen used in this study was obtained from two different countries. Bitumen 1 (B1) was from Slovnaft, a.s. refinery in Slovak Republic. Bitumen 2 (B2) was obtained from a Canadian refinery. Both bitumens come from one-time sampling flow prepared into 8 kg trays. Sub-samples were prepared by the same manner from the 8 kg trays after heating to 135° C during 4 hour. Table 1 shows the content of sulfur and the group composition of the bitumens samples.

Table 1 Composition of bitumens matrixes

Samples	x (% m/m)				
	x_S	x_{P+N}	x_{Ar}	x_{PS}	x_{Asph}
B1	2.99	11.9	33.1	45.0	9.9
B2	5.13	10.3	42.9	16.6	30.1

x_S – mass fraction of sulfur, x_{P+N} – mass fraction of paraffinic and naphthenic compounds, x_{Ar} – mass fraction of aromatic compounds, x_{PS} – mass fraction of polar substances

2.2 Crumb rubber

Bitumen mixtures were prepared with crumb rubber (V.O.D.S., a.s. Košice, Slovak Republic). Five fractions of different particle sizes (table 2) were prepared. The crumb rubber fraction Fr5 was used for determination of repeatability of mixing crumb rubber with bitumen B1.

Table 2 Crumb rubber fractions of different particle sizes

Fraction of crumb rubber	Sieve designation		Dosage to bitumen
	Mesh	mm	% m/m
Fr 1	8 to 14	2.380 to 1.410	5.0 ± 0.1
Fr 2	16 to 18	1.190 to 1.000	5.0 ± 0.1
Fr 3	20 to 30	0.841 to 0.595	5.0 ± 0.1
Fr 4	45 to 70	0.354 to 0.210	5.0 ± 0.1
Fr 5	100 to 170	0.160 to 0.090	5.0 ± 0.1

2.3 Mixing of bitumen with crumb rubber

Equipment IKA-ULTRA-TURRAX® T18 with extension S 18N-19G was used for mixing crumb rubber with bitumen. The samples were melted at temperature 135 ± 1°C and were poured off to five test containers. Each sample contained 200 ± 5 g of bitumen and was mixed with crumb rubber. Each dose of crumb rubber was calculated to the actual mass of bitumen. Crumb rubber content in all bitumen samples was 5 ± 0.01 % m/m. The mixing process was carried out at a temperature of 150 ± 3°C and with duration of 60 min. The operating speed was 15 000 rpm. The samples without crumb rubber were mixed at the same conditions as the samples with crumb rubber. After the mixing process each sample was analyzed according to the European standard methods for the determination of penetration [1], softening point [2] and viscosity [7].

3. Results and discussion

Bitumen properties were evaluated considering three parameters: penetration [1], softening point [2] and viscosity [7]. The possibility of using the measured parameters as criteria for evaluation of properties of crumb rubber and bitumen mixtures was reviewed.

3.1 Softening point, penetration, temperature susceptibility and penetration index

The results of the standard parameters determination for samples B1 and B2 are shown in Table 3. Penetration was determined at two different temperatures (25°C and 35°C). Standard deviation of the penetration and softening point measurements does not exceed standard permitted limits. The softening point temperature t_{RB}, at which it is assumed that a viscosity of bitumen becomes 800 p.u can be compared with the temperature t_{800}. The temperature t_{800} was calculated using the temperature susceptibility *A* and the penetrations at 25°C and 35°C using equation (3). The temperature difference between calculated t_{800} and softening point t_{RB} was greater than 7°C for both bitumens. Premise for achieving penetration of 800 penetration units at softening point is so called in question.

Table 3 Penetration and softening point of the bitumen samples

Measurement No.	B1				B2			
	Penetration [0.1mm] at 25°C	Penetration [0.1mm] at 35°C	t_{RB} [°C]	t_{800} [°C]	Penetration [0.1mm] at 25°C	Penetration [0.1mm] at 35°C	t_{RB} [°C]	t_{800} [°C]
1	102	230	44.1	50.3	205	438	32.1	42.9
2	99	224	44.3	50.6	208	437	32.8	43.1
3	98	230	44.4	49.6	205	429	32.9	43.4
4	103	216	43.9	52.7	212	441	32.9	43.1
5	107	216	44.6	53.6	193	417	32.6	43.5
6	106	222	45.8	52.3	189	428	33.8	42.7
7	109	218	44.4	53.8	208	415	33.8	44.5
8	105	222	43.9	52.1	199	422	33.3	43.5
9	108	216	43.7	53.9	200	425	33.0	43.4
$\bar{x}$	104	222	44.3	52.1	202	428	33.0	43.3
$\pm\sigma_i$	4	6	0.6	1.6	8	9	0.5	0.5
$\pm\sigma_{\bar{x}}$	1	2	0.2	0.5	3	3	0.2	0.2
$\pm\Delta X_{max}$	3	4	0.5	1.2	6	7	0.4	0.4

The measured values of the penetration and of the softening point temperatures were used to calculate the temperature susceptibility and penetration index of bitumen (Table 4).

Table 4 The values of temperature susceptibility, penetration index of bitumen B1 and B2

Measurement No.	B1				B2			
	A	PI	A_{RB}	PI_{RB}	A	PI	A_{RB}	PI_{RB}
1	0.0353	0.85	0.0468	-1.03	0.033	1.33	0.0833	-4.20
2	0.0355	0.82	0.0470	-1.06	0.0322	1.49	0.0750	-3.70
3	0.0371	0.52	0.0470	-1.05	0.0321	1.52	0.0749	-3.70
4	0.0322	1.50	0.0471	-1.07	0.0318	1.58	0.0730	-3.60
5	0.0305	1.88	0.0446	-0.72	0.0335	1.22	0.0813	-4.10
6	0.0321	1.52	0.0422	-0.36	0.0355	0.81	0.0712	-3.40
7	0.0301	1.98	0.0446	-0.72	0.0300	2.00	0.0665	-3.10
8	0.0325	1.43	0.0467	-1.01	0.0326	1.40	0.0728	-3.60
9	0.0301	1.98	0.0465	-0.99	0.0327	1.38	0.0753	-3.70
$\bar{x}$	0.0328	1.39	0.0458	-0.89	0.0326	1.41	0.0748	-3.68
$\pm\sigma_i$	0.0026	0.54	0.0017	0.24	0.0015	0.32	0.0050	0.33
$\pm\sigma_{\bar{x}}$	0.0009	0.18	0.0006	0.08	0.0005	0.11	0.0017	0.11
$\pm\Delta X_{max}$	0.0020	0.42	0.0013	0.19	0.0011	0.24	0.0039	0.25

The temperature susceptibility A is usually calculated at temperature difference Δt = 10°C using penetrations measured at 25°C and 35°C. In this case the value of penetration index PI calculated from the relation (2) is really the function of penetration. Influence of the penetration at 35°C and 25°C at the maximum deviation of the penetration index PI can be quantified using the partial derivatives of the function $PI = f(pen_{25}, pen_{35})$. Partial derivatives of this function are expressed by the formulas (7) and (8):

$$\partial(PI)/\partial(pen_{35}) = -150/2.3026 * pen_{35} * (1 + 5\log(pen_{35}) - 5 * \log(pen_{25}))^2 \quad (7)$$

$$\partial(PI)/\partial(pen_{25}) = 150/2.3026 * pen_{25} * (1 + 5\log(pen_{35}) - 5 * \log(pen_{25}))^2 \quad (8)$$

On the other hand the temperature susceptibility A_{RB} is calculated using penetration at 25°C and temperature difference $\Delta t = t_{RB} - 25$. The value of the penetration index PI_{RB} calculated using the relation (3) is not very penetration function. Penetration index PI_{RB} is besides the penetration also a function of the softening point. Influence of the softening point and penetration at 25°C at the maximum error of the penetration index PI_{RB} can be quantified using the partial derivatives of the function $PI_{RB} = f(t_{RB}. pen_{25})$. Partial derivatives of this function are expressed by the formulas (9) and (10):

$$\partial(PI_{RB})/\partial(pen_{25}) = 1500 * \log(pen_{25}) - 37600/2.3026 * pen_{25} * (50 * \log(pen_{25}) - t_{RB} * 120) \quad (9)$$

$$\partial(PI_{RB})/\partial(t_{RB}) = -1500 * \log(pen_{25}) + 4352/(50 * \log(pen_{25}) - t_{RB} - 120) \quad (10)$$

The use of derivatives of the equations (2) and (3) as follows (7),(8),(9),(10) and the use of maximum deviations of penetrations and softening points permits calculation of total maximum deviations of penetration indices of PI and PI_{RB}. Similarly, the parameters softening point t_{RB} and penetration at 25°C contribute to the total maximum error PI_{RB}. Table 5 shows the total maximum errors of penetration index PI and PI_{RB} as well as overall maximum deviations (α = 0.05; t_{n-1} = 2.306).

Table 5 Maximal deviations of penetration indices PI a PI_{RB}

Samples	Δ_{max}	PI Contribution (%)		Δ_{max}	PI_{RB} Contribution (%)	
		pen_{25}	pen_{35}		pen_{25}	t_{RB}
B1	0.31	38.4	61.6	0.46	38.9	61.1
B2	0.31	35.5	64.5	0.18	18.6	81.4

From the calculated values of maximum deviation of penetration indices it is visible, that penetration at 35°C exhibits a higher contribution to the deviation of PI. Penetration index PI as a function of the penetration represents the rate of penetration change with temperature in the given temperature range. The softening point contributes essentially to the maximum deviation of penetration index PI_{RB}. Since the value of penetration 800 p.u. does not correspond to the softening point, it is not possible to consider PI_{RB} as a measure of penetration change with temperature in the temperature interval $\langle 25; t_{RB} \rangle$. The penetration index calculated repeated determination of penetration and the softening point are shown in Table 4. The maximum errors calculated from the values of penetration indices (Table 4) are in good agreement with maximum deviations determined by the partial derivatives.

The penetration indices PI of bitumen B1 and B2 are outside the range of the values required in standard [34-36]. The penetration index PI_{RB} of bitumen B1 fits to the desired interval, but, the value PI_{RB} of bitumen B2 is outside the interval introduced by standard. Comparison of penetration index PI and penetration index PI_{RB} shows different values in the case of both tested bitumens. The penetration indices and temperature sensitivities shown in Figure 1 confirm that two chosen bitumen of different gradations have a quite near values of penetration index PI but have very different values PI_{RB}. The penetration index and temperature susceptibility values are indicated in Figure 1 with the interval of maximum errors calculated using the partial derivatives of the corresponding functions.

The standard for road bitumens [34], for hard industrial bitumens [35] and for multigrade road bitumens [36] requires the value of penetration index in the range from $\langle -1.5; 0.7 \rangle$. These standards include the method for determination of penetration index. The formula for the penetration index calculation specified in the standards [34-36] coincides with the equation (3).

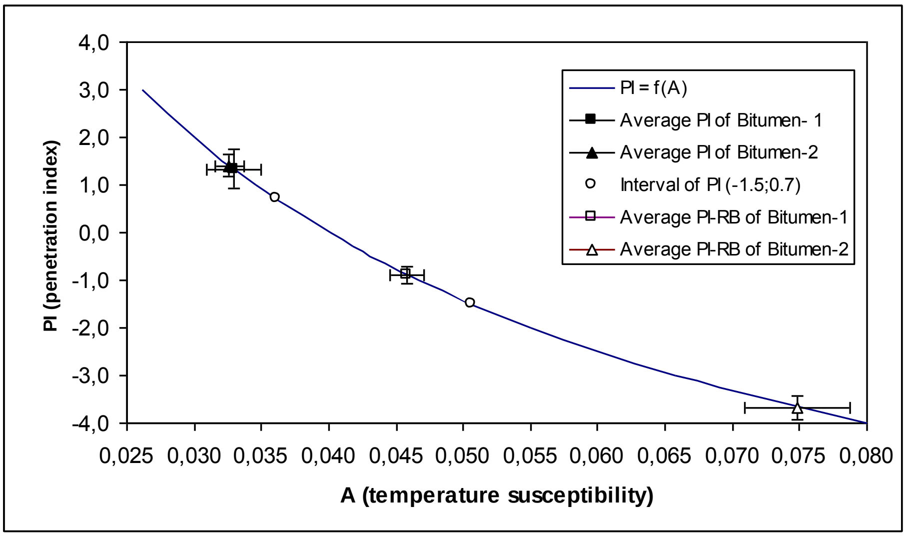

Figure1 Dependence of *PI*=f(*A*) in penetration index interval <-4.3>

Different methods for calculation of the temperature susceptibility and penetration index provide inconsistent results. The calculation of *PI*, which is based on two penetrations measured at various temperatures, is affected by the accuracy of the penetration measurement and by the difference of applied temperatures. Value of the softening point affects the numerical value of the penetration index PI_{RB} more than penetration at 25°C.

Literary sources use for penetration indices identical name and identical symbol *PI*, however they are calculated by various methods according the equations (2) and (3). The symbols *PI* and PI_{RB} used in this text are appropriate as well as terminological distinction is advisable. A suitable name for a PI_{RB} is penetration - softening point index [32-33].

3.2 Assessment of crumb rubber impact on the properties of bituminous mixtures using penetration, softening point and penetration index

Different bituminous compositions were prepared by mixing of various particle sizes of crumb rubber with the bitumen B1 and B2. The temperatures of softening point and the penetrations of the prepared samples were determined.

Precision of parameters (pen_{25}, t_{RB}) measurements for bitumen modified with crumb rubber was examined by estimation of the parameters for mixtures prepared by repeated mixing of the crumb rubber (fraction F5 - particle size 20-30 mm) in the amount of 5 % m/m and bitumen B1. Experimental conditions of mixing are given in the experimental section. The experimental results of the determination of the softening point, penetration at 25°C and the calculated penetration index PI_{RB} are shown in Table 6.

Table 6 Parameters of crumb rubber Fr5 – bitumen B1 mixture at six times repeated sample preparation

No.	t_{KG} [°C]	Penetration at 25°C [0.1 mm]	PI_{RB}
1	44.0	85	-1.59
2	46.2	89	-0.78
3	45.8	99	-0.58
4	42.3	92	-1.94
5	44.5	89	-1.30
6	43.4	86	-1.75
$\bar{x}$	44.4	90	-1.32
$\pm\sigma_x$	1.5	5	0.54
$\pm\sigma_i$	0.6	2	0.22
$\pm \Delta X_{max}$	1.5	5	0.57

The maximum deviations (sα = 0.05, t_{n-1}=2.5706)) in the determination of softening point and the penetration were higher compared with those in the relevant standard methods. The determination of these parameters confirmed that the process steps of mixing the crumb rubber in bitumen affect the value of the maximum measurement error. The increase measurement error of parameters caused by mixing bitumen with crumb rubber is small compared to the original bitumen. According to the standard EN 1427 the repeatability determination of penetration in modified bitumen considered indicative. Since our results show that the relative maximum error of determination of the softening point is ± 1.5°C, which represents ± 3.4 % and the deviation of penetration is ± 5 p.u. which represents ± 6 %, the parameters may be usable in evaluating the efficiency of additives.

The maximal error $\pm\sigma_x$ of penetration index PI_{RB} calculated from repeated measurements (Table 6) at an average value PI_{RB} is 0.54, which represents 43%. Maximal deviation of penetration index PI_{RB} determined by derivation of relations (9) and (10) shown in Table 4 has a value ± 0.46, which represents ±35 %. These values of deviations are limiting for use of PI_{RB} as an indicator of changes in the properties of bituminous mixtures with crumb rubber.

Table 7 and Table 8 show that the addition of crumb rubber particles of various sizes into the bitumen sample B1 and B2 led to the changes in the basic parameters. The addition of crumb rubber increased softening point but the decrease in particle size of the crumb rubber was not reflected in the increase of softening point. The value of penetration decreased as the result of crumb rubber presence and also as the result of particle size decrease.

Table 7 Standard parameters and penetration indices of bitumen B1 mixed with crumb rubber

Sample	t_{RB} [°C]	Penetration [0.1mm]		PI	PI_{RB}
		at 35°C	at 25°C		
B1 – Fr1	44.5	220	100	1.06	-0.96
B1 – Fr2	46.9	197	96	1.71	-0.34
B1 – Fr3	46.1	187	90	1.59	-0.77
B1 – Fr4	44.1	182	84	1.20	-1.59

Table 8 Standard parameters and penetration indices of bitumen B2 mixed with crumb rubber

Sample	t_{RB} [°C]	Penetration [0.1mm]		PI	PI_{RB}
		at 35°C	at 25°C		
B2 – Fr1	38	286	169	4.03	-1.67
B2 – Fr2	39.9	270	166	4.59	-0.90
B2 – Fr3	39.7	272	159	3.82	-1.15
B2 – Fr4	40.4	267	148	3.15	-1.13

Calculated values of penetration indices PI and PI_{RB} bitumens B1 and B2 give discontinuous function in dependence on changes in the particle size of the crumb rubber. Similarly, no functional interdependencies between penetration index PI and PI_{RB} were shown. Our observations indicate that the penetration index is not suitable for monitoring the effect of the particle size of the crumb rubber on the properties improvement of the bitumen grades modification.

3.3 Assessment of crumb rubber impact on the properties of bituminous mixtures using dynamic viscosity and relative viscosity increment

The viscosity as a parameter of the modifier mixture in bitumen is part of the evaluation of those systems. Precision of viscosity measurements for bitumen modified with crumb rubber was examined by estimation of the viscosity for mixtures prepared by repeated mixing of the crumb rubber (fraction F5 - particle size 20-30 mm) in the amount of 5 % m/m and bitumen B1. Experimental conditions are given in the experimental section. Results of dynamic viscosities at 125 and 135 ° C and calculated relative increments of viscosity are given in Table 9.

Table 9 Dynamic viscosity of bitumen B1 containing 5% m/m of crumb rubber (Fr5)

No.	η [mPa.s] at		η_i [%]	
	135°C	125°C	at 135 °C	at 125 °C
1	372.5	504.9	41.10	29.46
2	377.1	525.9	42.84	34.85
3	408.3	544.2	54.66	39.54
4	357.0	503.8	35.23	29.18
5	403.8	517.8	52.95	32.77
6	380.7	526.4	44.20	34.97
$\bar{x}$	383.2	520.5	45.16	33.46
$\pm\sigma_x$	19.5	15.2	7.38	3.90
$\pm\sigma_i$	8.0	6.2	3.01	1.59
$\pm \Delta X_{max}$	20.7	16.1	7.83	4.13

The calculated maximum errors of viscosity measurement (α = 0.05, t_{n-1} = 2.5706) at a temperature of 135°C respectively 125°C are converging to errors of the individual measurements. These errors represent 5.4% respectively 3.1% on the average viscosity values. The percentage values of deviation of relative viscosity increments have a considerably higher value, 17% respectively 12%.

Our results show that the maximum deviation of relative viscosity increment calculated using partial derivatives $\partial\eta_i / \partial\eta$, $\partial\eta_i / \partial\eta_0$ of equation (6) is high. On one hand it is associated with low relative viscosity increment, which is calculated as a ratio of two numbers that are two orders of magnitude larger. On the other hand, taking into consideration the maximum error calculated from repeated measurements (Table 9), the relative increment of the viscosity could be suitable for evaluation of the effectiveness of bitumen modification with polymers.

Tables 10 and 11 show changes of dynamic viscosities and relative viscosity increments of bitumen B1 and B2 mixed with fractions of crumb rubber of different particle sizes. The viscosity increases with the decrease of dimensions of crumb rubber particles. Increase in viscosity due to the addition of crumb rubber was greater at softer bitumen B2. These results show that the dynamic viscosity and also relative viscosity increment are suitable parameter for monitoring changes in the properties of the bitumen in the mixture with varying particle size of the crumb rubber.

Table 10 The effect of the particle size of the crumb rubber mixed with bitumen B1 on viscosity and on relative viscosity increment

Samples	η [mPa.s]		η_i[%]	
	at 135°C	at 125°C	at 135°C	at 125°C
B1	264	390	-	-
B1 - Fr1	269	395	1.90	1.39
B1 – Fr2	279	431	5.76	10.49
B1 – Fr3	302	454	14.48	16.52
B1 – Fr4	312	481	18.23	23.27

Table 11 The effect of the particle size of the crumb rubber mixed with bitumen B2 on viscosity and on relative viscosity increment

Sample	η [mPa.s]		η_i[%]	
	at 135°C	at 125°C	at 135°C	at 125°C
B2	215	348	-	-
B2-Fr1	243	404	13.12	16.24
B2-Fr2	247	427	14.80	22.96
B2-Fr3	252	440	17.12	26.50
B2-Fr4	300	499	39.79	43.48

The standard deviation limits were calculated for relative increment of crumb rubber - bitumen mixtures. The calculated maximum error range for relative viscosity increment at 125°C is ±7,4% for bitumen B1 and ± 3,6 % for bitumen B2.

Conversion of dynamic viscosity to the relative viscosity increment in allows quantifying the effect of particle size on the properties of crumb rubber bitumen mixture and thereby to better describe and evaluating the modification process. Value of relative viscosity increment tends to increase with decreasing particle size of the crumb rubber in bitumen. For application of the relative incremental viscosity as criteria for the evaluation of modified bituminous mixtures is important reduction of its maximum deviation. It was found that the measurement of viscosity at lower temperature is preferred in that respect. Figure 2 shows the increments of the relative viscosity of the bitumen B1 and B2 mixtures with crumb rubber of different size particles. Intervals of maximum errors of relative viscosity increment (α = 0.05, tn-1 = 2.5706) calculated from the percentage of maximum error η_i (Table 9) are showed in the figure 2.

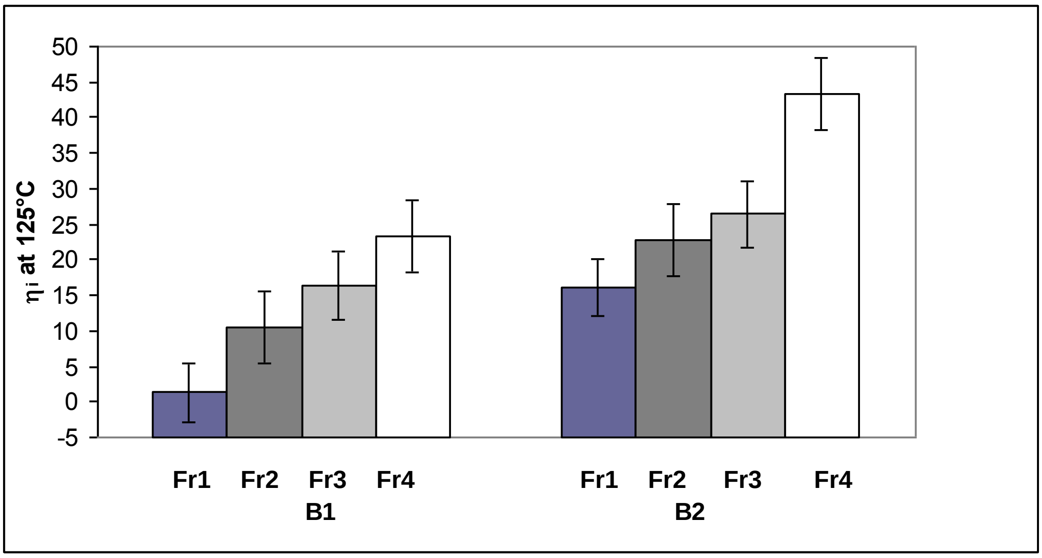

Figure 2. Relative viscosity increment of bitumen B1 and B2 mixed with crumb rubber of various particle sizes

The relative viscosity increment describes differences of interaction of bitumen with crumb rubber. It also reflects the different parameters of bitumen. Relative viscosity increments for various samples are sufficiently different despite the relatively wide interval of error values and can be used to assess the quality of crumb rubber modified bitumen.

4. Conclusions

According to tests, the penetration and softening point, the bituminous products are characterized and sorted into performance classes. These data are part of the basic characteristics of bitumen. Softening point manifested itself as an inappropriate parameter in evaluation of the modification effect of bituminous mixtures with crumb rubber of various particle sizes. The decrease of penetration with the reduction of particle size of the crumb rubber proved to be evident.

Penetration and softening point are used for the calculation of the temperature susceptibility and penetration index. It is necessary to distinguish penetration index *PI* calculated from two penetration data, and penetration index PI_{RB} calculated from the penetration and softening point. Penetration index is not an appropriate parameter for determination of the quality characteristics of bitumen crumb rubber mixtures. This parameter describes the classification of bitumen in terms of sol-gel character. A larger number of measurements of penetration and softening point increase the probability of correct categorization of bitumen. In this study it was shown that the temperature susceptibility and penetration index do not describe sufficiently the changes in bitumen properties mixed with crumb rubber.

Dynamic viscosity is effective and appropriate parameter for monitoring changes in the modification of bitumen with rubber crumb. Changes in dynamic viscosity are more expressed at lower temperature measurements. The bitumen modified with crumb rubber with a smaller particle size reaches a greater change in dynamic viscosity. The calculated relative viscosity increment was shown to be adequate parameter for examination the effect of the particle size of the crumb rubber for evaluation of the quality of the bituminous composition. Its values describe differences in quality parameters of crumb rubber and respond to the different parameters of bitumen.

Symbols

A	temperature susceptibility calculated using penetrations at temperature t_1 and t_2
A_{RB}	temperature susceptibility calculated using penetration 800 p.u. at softening point temperature
pen_1	penetration at temperature t_1
pen_2	penetration at temperature t_2
pen_{25}	penetration at temperature 25°C_1
pen_{35}	penetration at temperature 35°C
PI	penetration index calculated using temperature susceptibility *A*
PI_{RB}	penetration index calculated using temperature susceptibility A_{RB}
t	Temperature, °C
t_{n-1}	critical values of the Student's *t* distribution
t_{RB}	temperature of bitumen softening point
t_{800pen}	temperature at which is penetration of the bitumen 800 penetration units
$\pm X_{max}$	maximal standard deviation
$\bar{x}$	average value

Greek Letters

η	dynamic viscosity, mPa.s
η_{rel}	relative viscosity
η_i	relative viscosity increment, %
η_0	dynamic viscosity of solvent or polymer free bitumen, mPa.s
$\pm\sigma_i$	standard deviation of individual measurements
$\pm\sigma_{\bar{x}}$	standard deviation of the arithmetic mean
$\pm \Delta X_{max}$	maximal standard deviation

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