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Mutual Correlation Between Softening Point and Flow Resistance at Elevated Temperature According to Bitumen Mass in Bitumen Sheets

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Abstract. Bitumen sheets are isolating materials that are used in the building construction, primarily as roof waterproofing, vapour barrier and waterproofing of the substructure. For civil engineering they are applied as the isolation of concrete bridge decks and they are used as the main waterproofing for underground construction. Currently, bitumen sheets mentioned above are modified by polymers of elastomer and plastomer. In the places where the bitumen sheets are exposed to UV radiation or to high temperature, the manufacturer is, according to the product standards, required to declare the flow resistance at elevated temperature. This paper deals with the investigation of the interdependence between the flow resistance at elevated temperature and the softening point of the bitumen matter in these bitumen sheets. In practice, the results of this dependence are often used in the production of bitumen sheets during the inter-operative check, where the softening point value determines the value of the flow resistance at elevated temperature. This allows to determine, whether the produced bitumen sheets will fulfill the values declared by the manufacturer with advance. Since the test for detection of the value of the softening point takes in average 30 minutes then it is possible to react quickly and efficiently during the production and to make the appropriate corrections. Reinforced bitumen sheets which are used for waterproofing of the isolation of concrete bridge decks were selected as the samples from various manufacturers. In practice, this aforementioned dependency is often cited but it is not explicitly documented by a sufficiently large number of samples. The authors of this article found during the previous research that the dependence is influenced by the temperature value – the higher is the temperature, the more obvious is the dependence. For this reason, it is very evident in the case of bitumen sheets modified by polymers of the plastomeric character. The hypothesis, that there exists some relationship between the flow resistance at elevated temperature concerning to bitumen sheets and the softening point of the bitumen matter, was confirmed only partially. The aim of the paper is to confirm or refute the hypothesis even for materials modified by polymers of elastomeric character. For this reason, the number of bitumen sheets modified by polymers of both plastic and elastomeric character was increased. Results were formulated based on the statistical evaluation of data obtained from laboratory measurements.

1. Introduction

This paper deals with the investigation of the interdependence between the flow resistance at elevated temperature [1], [2] for reinforced bitumen sheets and softening points of bitumen matter softening point [3] in these reinforced waterproofing bitumen sheets (bitumen sheets in the next text).



The results of this dependence are frequently used in the production of bitumen sheets during an in-process check in practice, when the softening point value determines the value of the flow resistance at an elevated temperature [1,2]. The flow resistance at elevated temperature is a part of an exit check. It is possible to determine whether the manufactured bitumen sheets meet the values declared by the manufacturer in advance. These values are given in the national standards because the European standards related to the bitumen sheets are merely of a test character and they do not specify the quality requirements. The flow resistance at elevated temperature is determined for the bitumen sheets, which are used in the roof compositions according to the CSN (Czech technical standard) EN 13707: 2014 [4] and the isolation of the bitumen bridge decks according to the CSN EN 14695: 2010 [5]. The requirements for the flow resistance at an elevated temperature in the Czech Republic, [5,6], Slovakia [7] and Germany [8,9] are shown in Table 1.

Table 1. Requirements for the flow resistance at elevated temperature according to [6-10]. This was for the bitumen sheets used for insulating roofs and bitumen bridge decks in monolayer systems. SBS –the mass modified by elastomers, APP – the mass modified by plastomers. Source: authors.

Standard Type of modification of bitumen coating matter	Bitumen sheets requirements [°C]	
	APP	SBS
Roof construction		
ČSN 730605-1: 2014	$\geq + 120$	$\geq + 90$
DIN V 20000-201	$\geq + 130$	$\geq +100$
Bitumen bridge deck		
ČSN 736242:2010	$\geq + 100$	
STN 736242:2010	$\geq + 100$	
DIN V 20000-203	$\geq + 150$	$\geq +110$

The authors of this article found [10] during the previous research that the dependence is influenced by the temperature value. The higher is the temperature, the more obvious is the dependence. For this reason, it is very noticeable in the case of bitumen sheets with the mass modified by plastomers. The aim of the paper is to confirm or refute the hypothesis even for materials modified by polymers of elastomeric character. For this reason, the number of bitumen sheets modified by polymers of both plastic and elastomeric character was increased by another 16 samples of bitumen sheets with the mass modified by elastomers and plastomers.

2. Materials and methodology

2.1. Used material

Thirty-four samples were selected for testing. They are used for monolayer applications for the insulation of bitumen bridge decks in the Czech and Slovak Republics. Nineteen samples have coarse-grained gritting, nine have fine-grained gritting and six samples are without gritting. There are twelve samples with the mass modified by plastomers and fourteen with the mass modified by elastomers in the selected file. The basic bitumen sheets characteristics are described in Table 2. All samples had a supporting insert made of polyester, which has been impregnated with the bitumen matter.

Table 2. Selected characteristics of the test samples. Legend: HP – coarse gritting, JP – fine-grained gritting, N – the finish of the geotextiles weighing about 20 [g/m²], PES – polyester fleece, P – mass modified by plastomers, E – mass modified by elastomers. Source: authors.

Number of test sample	2	3	4	6	7	8	9	10
Upper surface adjusting	HP	N	HP	N	JP	HP	JP	HP
Bitumen matter type	P	P	E	P	E	P	P	P
Supporting insert, area weight [g/m ²]	230	200	230	200	250	220	230	230
Thickness [mm]	5.20	5.24	5.3	4.83	5.10	5.50	4.85	5.16
Application location (state)	CZ/SK	CZ/SK	SK	SK	SK	SK	SK	CZ/SK
Number of test sample	15	16	17	18	22	23	25	26
Upper surface adjusting	HP	JP	HP	HP	HP	HP	HP	N
Bitumen matter type	E	E	P	E	P	P	E	P
Supporting insert, area weight [g/m ²]	230	230	230	250	250	280	280	250
Thickness [mm]	5.46	4.80	5.67	5.05	5.22	4.80	5.18	4.75
Application location (state)	CZ	CZ	CZ	SK	SK	CZ/SK	SK	SK
Number of test sample	29	30	31	32	33	34	35	39
Upper surface adjusting	N	HP	HP	HP	HP	HP	JP	N
Bitumen matter type	P	P	P	E	E	E	E	P
Supporting insert, area weight [g/m ²]	250	250	280	250	250	230	250	280
Thickness [mm]	5.04	4.81	5.23	5.40	5.34	4.81	4.99	5.12
Application location (state)	CZ	CZ	SK/CZ	SK	SK	CZ	CZ	CZ
Number of test sample	41	42	43	44	45	46	47	
Upper surface adjusting	N	HP	HP	HP	HP	HP	JP	
Bitumen matter type	E	P/E	P	P/E	E	P	P	
Supporting insert, area weight [g/m ²]	230	230	250	230	230	250	230	
Thickness [mm]	5.41	5.41	4.90	4.97	4.50	4.92	5.19	
Application location (state)	CZ	CZ	CZ	CZ	CZ	CZ	CZ/SK	

2.2. Methodology for the statistical evaluation

We have focused on the linear dependence in order to determine the interdependence of the flow resistance at elevated temperature on the softening point. The tightness of the linear sheet was determined by calculating the Pearson Correlation Coefficient. We have performed a test for the significance of the correlation coefficient and we have determined that there is an estimate of a 95% confidence interval for the correlation coefficient. Then we created a linear regression model for dependence of the flow resistance at elevated temperature on the softening point. After that, we formed the regression equation and conducted tests regarding the significance of the regression coefficients, the materiality test of the model and the calculation of the coefficient of determination. All tests and interval estimates were performed on a standard significance level $\alpha = 0.05$.

3. Test results

3.1. The softening point

The value of the softening point of the bitumen coating matter ranged from 107 °C to 153.5 °C. As expected, the mass modified by plastomers reached high temperatures from 120.5 °C to 154.5 °C. The mass modified by elastomers reached temperatures from 118 °C to 135.5 °C. The overview of complete results is shown in Table 3.

Table 3. The test results of the determination of softening point – the Ball and Ring Method according to [3] for the corresponding samples. Source: [10] and authors.

Number of test sample	2	3	4	6	7	8	9	10	14
Softening point [°C]	153	141	122.5	128.5	125	107	145.0	153.5	127
Number of test sample	15	16	17	18	22	23	25	26	28
Softening point [°C]	124.5	128.5	120.5	131	153	153	135.5	144.5	122
Number of test sample	29	30	31	32	33	34	35	39	40
Softening point [°C]	141.5	153	154.5	126.5	118	119.5	126.5	125.5	150
Number of test sample	41	42	43	44	45	46	47		
Softening point [°C]	129	126.5	127	127	116.5	139.5	150.5		

3.2. The flow resistance at elevated temperature

The flow resistance at elevated temperature reached the temperature from 100 °C to 160 °C. As expected, the mass modified by plastomers reached a high temperature, i.e. from 110 °C to 160 °C. The mass modified by elastomers reached the temperature from 100 °C to 130 °C. Complete results can be found in Table 4.

Table 4. The test results of the flow resistance at elevated temperature [1] for the corresponding sample. Source: [10] and authors.

Number of test sample	2	3	4	6	7	8	9	10	14
Flow resistance [°C]	155	155	120	115	125	120	160	160	125
Number of test sample	15	16	17	18	22	23	25	26	28
Flow resistance [°C]	100	110	125	130	160	160	125	140	110
Number of test sample	29	30	31	32	33	34	35	39	40
Flow resistance [°C]	155	150	155	125	115	115	110	115	155
Number of test sample	41	42	43	44	45	46	47		
Flow resistance [°C]	120	125	155	120	105	130	160		

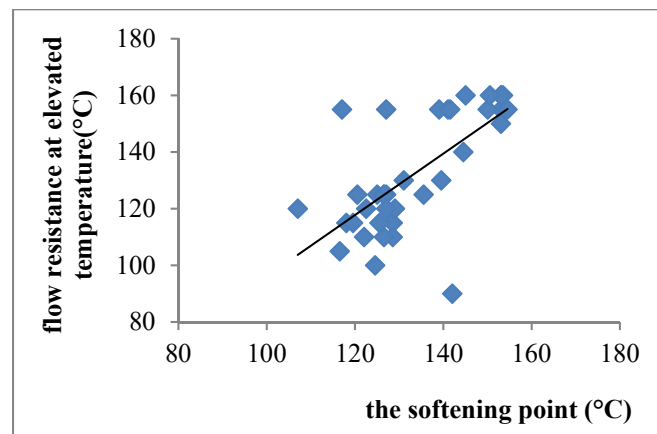
3.3. Results of statistical evaluation

The calculations and tests were realized in three steps. In the introduction, we analysed all of the measured samples. In the second step, we conducted the evaluation separately for the bitumen sheets with the mass modified by plastomers and for the bitumen sheets with the mass modified by elastomers. In the third step, we divided samples of bitumen sheets into two groups according to the production site. The first group collected bitumen sheets produced in Europe with the exception of Italy and the second group included bitumen sheets produced in Italy. All tests and interval estimates were performed on a standard significance level $\alpha = 0.05$.

We calculated the correlation coefficient $r = 0.68$ and interval estimation with values (0.46; 0.82) for the dependence of the flow resistance at elevated temperature on the softening point which was applicable to all of the samples. The regression model was given by the regression equation in the form $y = 1.08x - 12.13$, wherein the variable x presents the softening point and y evaluates the flow resistance at elevated temperature, see Figure 1.

Table 5. The tests of significance and interval estimates for coefficients

Estimate of coefficients	Test statistic	<i>p</i> -value	Upper limit of confidence interval	Upper limit of confidence interval
-12.13	-0.458	0.65	-65.88	41.63
1.08	5.489	$3,64 \cdot 10^{-6}$	0.68	1.48

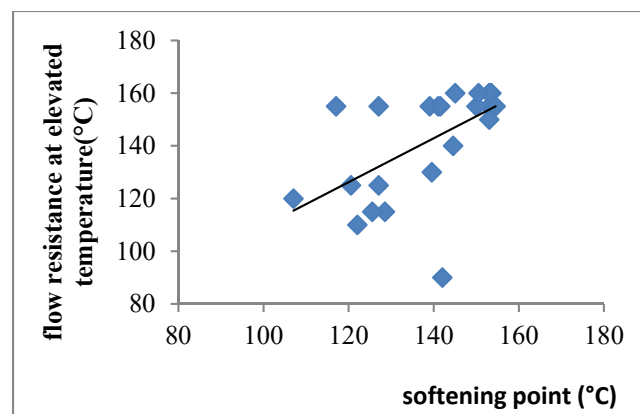
**Figure 1.** Regression - all samples. Source: authors

The coefficient of the determination R^2 has the value of 0.447 and the significance of the model is determined on the basis of the test statistics by the value $F = 30.14$ and $p\text{-value} = 3.64 \cdot 10^{-6}$.

Further, we calculated the correlation coefficient $r = 0.56$ and the interval estimation with values (0.19; 0.79) for the dependence of the flow resistance at elevated temperature on the softening point which was applicable to all of bitumen sheets with the mass modified by plastomers. The regression model was then given by the regression equation in the form $y = 0.83x + 26.26$, wherein the variable x presents the softening point and y evaluates the flow resistance at elevated temperature, see Figure 2 and Table 6.

Table 6. The tests of significance and interval estimates for coefficients (plastomers)

Estimate of coefficients	Test statistic	<i>p</i> -value	Upper limit of confidence interval	Upper limit of confidence interval
26.26	0.71	0.49	-51.25	103.77
0.83	3.11	0.005	0.28	1.39

**Figure 2.** Regression – bitumen sheets with the mass modified by plastomers. Source: authors

The coefficient of the determination R^2 has the value of 0.28 and the significance of the model is determined on the basis of the test statistics by the value $F = 9.69$ and p -value = 0.005.

We calculated the correlation coefficient $r = 0.5$ and the confidence interval is (-0.09; 0.84) for the dependence of the flow resistance at elevated temperature on the softening point which was applicable to all of the sheets with the mass modified by elastomers. The regression model was then given by the regression equation of the form $y = 0.83x + 12.37$, wherein the variable x represents the softening point and y evaluates the flow resistance at elevated temperature, see Figure 3 and Table 7.

Table 7. The tests of significance and interval estimates for coefficients (elastomers)

Estimate of coefficients	Test statistic	p -value	Lower limit of confidence interval	Upper limit of confidence interval
12.37	0.22	0.83	-113.57	138.31
0.83	1.85	0.09	-0.17	1.84

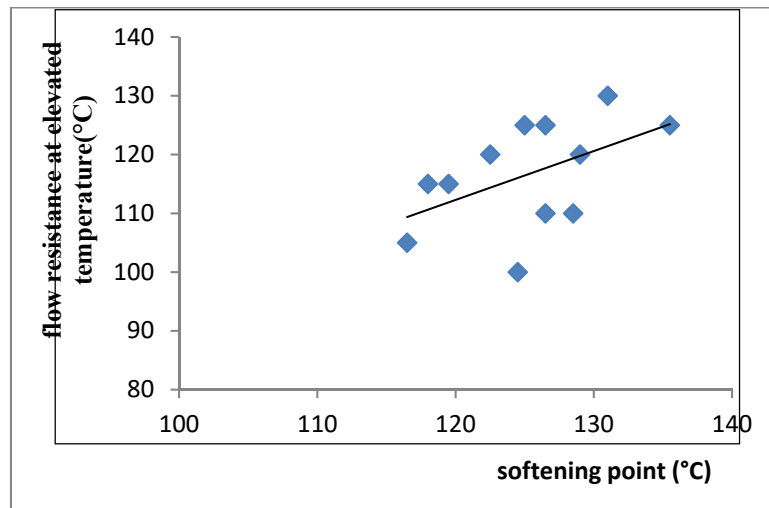


Figure 3. Regression – bitumen sheets with the mass modified by elastomers. Source: authors.

The coefficient of the determination R^2 has the value of 0.18 and the significance of the model is determined on the basis of the test statistics by the value $F = 3.41$ and p -value = 0.09.

The correlation coefficient for the dependence of the flow resistance at elevated temperature on the softening point for bitumen sheets made in Europe except Italy was determined in the value $r = 0.83$ and the confidence interval is (0.53; 0.94). The regression model is then given by the regression equation of the form $y = 1.04x - 9.79$, wherein the variable x represents the softening point and y evaluates the flow resistance at elevated temperature, see Figure 4 and Table 8.

Table 8. The tests of significance and interval estimates for coefficients (bitumen sheets made in Europe except Italy)

Estimate of coefficients	Test statistic	p -value	Lower limit of confidence interval	Upper limit of confidence interval
-9.79	-0.36	0.72	-68.49	48.90
1.04	5.08	0.00027	0.60	1.49

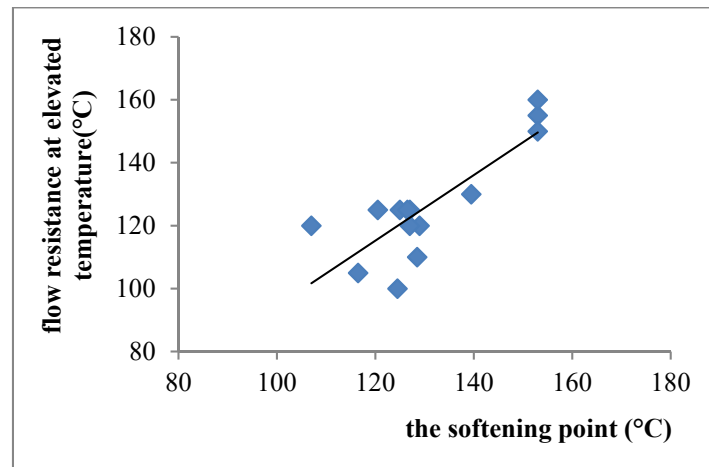


Figure 4. Regression - bitumen sheets made in Europe except Italy. Source: authors.

The coefficient of the determination R^2 has the value of 0.68 and the significance of the model is determined on the basis of the test statistics by the value $F = 25.85$ and $p\text{-value} = 0.000269$.

The correlation coefficient for the dependence of the flow resistance at elevated temperature on the softening point for bitumen sheets made in Italy was determined in the value $r = 0.34$ and the confidence interval is (-0.26; 0.75). The regression model is then given by the regression equation of the form $y = 0.72x + 39.83$, wherein the variable x represents the softening point and y evaluates the flow resistance at elevated temperature, see Figure 5 and Table 9.

Table 9. The tests of significance and interval estimates for coefficients (bitumen sheets made in Italy)

Estimate of coefficients	Test statistic	$p\text{-value}$	Lower limit of confidence interval	Upper limit of confidence interval
39.83	0.49	0.63	-138.32	217.98
0.72	1.20	0.26	-0.60	2.05

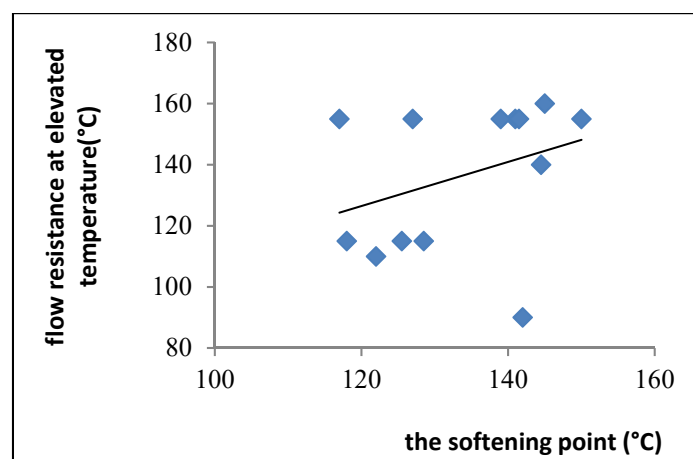


Figure 5. Regression – bitumen sheets made in Europe except Italy. Source: authors.

The coefficient of the determination R^2 has the value of 0.12 and the significance of the model is determined on the basis of the test statistics by the value $F = 1.44$ and $p\text{-value} = 0.255822$.

4. Discussions

4.1. The softening point and the flow resistance at elevated temperature

Based on the results [10], the samples were divided into two groups according to the modification of bitumen. In the case of the mass modified by plastomers, twenty samples of thirteen have a total higher flow resistance at elevated temperature, and seven samples have a higher temperature of the softening point. In more detail, the results are analyzed in Figure 6. In the group of bitumen sheets with the mass modified by elastomers, thirteen samples of a fourteen have higher temperature of the softening point and in one case the value is the same. See Figure 7.

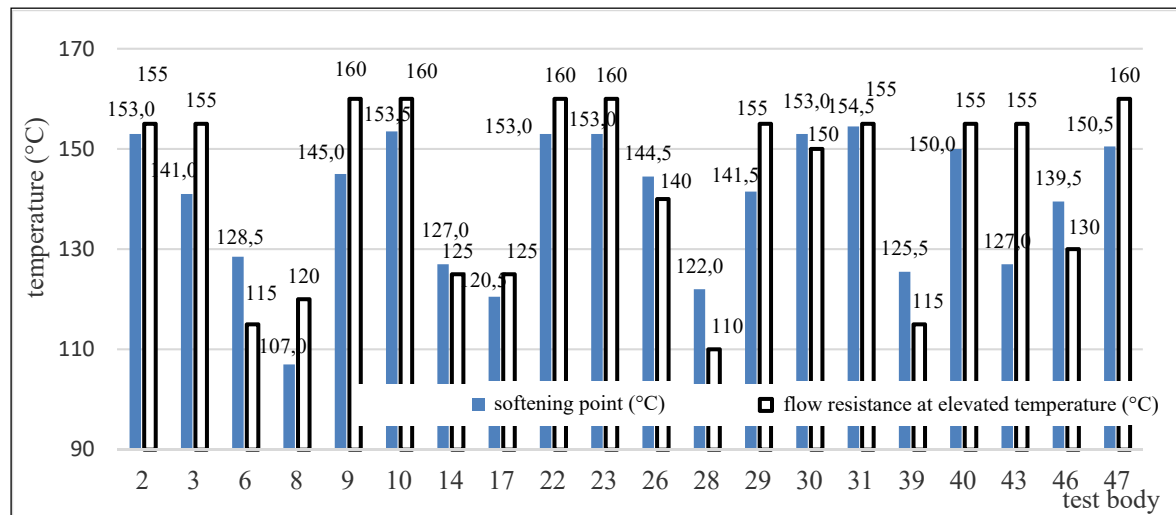


Figure 6. Results of the flow resistance at elevated temperature test [1] and a softening point [3] of bitumen sheets with the mass modified by plastomers. Source: authors

Noteworthy is the following fact that in the group of bitumen sheets with the mass modified by plastomer: in four cases of six cases, the softening point value is higher than the flow resistance at elevated temperature. This is a temperature in the value to 125 °C. This temperature approaches the lower limit for bitumen sheets with the mass modified by plastomer, see Table 1. This fact just made us carry out the statistical evaluation in three steps in total.

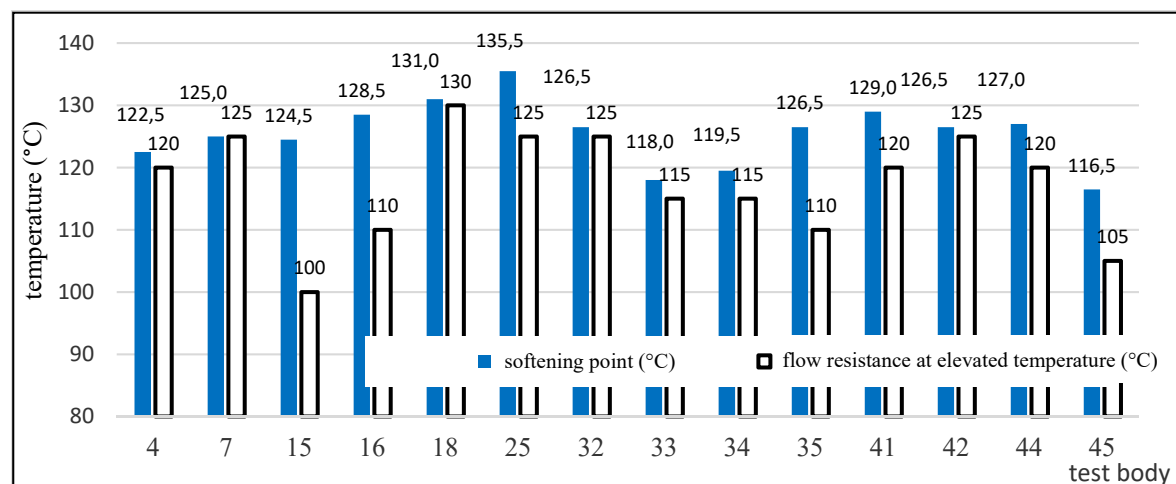


Figure 7. Results of the flow resistance at elevated temperature [1] and the softening point [3] of bitumen sheets with the mass modified by elastomers. Source: authors

4.2. The results of statistical evaluation

In the first model, all bitumen sheets regardless of the type of the bitumen mass were unified into one group. The statistically significant value of the correlation coefficient and the regression model characteristics show that the linear regression model is statistically significant. However, the value of the determination coefficient indicates the high variability of the measured values. Using a model to estimate the flow resistance at elevated temperature at a known softening point value, it is not to be expected that the point estimate obtained by simply substitution to the regression line will directly correspond to the reality. This estimate will be much more reliable by using the interval estimates of these coefficients.

In the second step, bitumen sheets were divided according to the modification of the bitumen cover. For a model with a selection of bitumen sheets with the mass modified by plastomers, the linear regression model is statistically significant, but the confidence values and p -values of the respective tests indicate lower statistical significance. Data variability in this case is manifested very more significantly.

In the case of the model where the selection was limited to bitumen sheets with the mass modified by elastomers, the correlation between softening point and the flow resistance at elevated temperature was statistically insignificant. This was reflected in the compiled linear regression model, which is not statistically significant either. Thus, there is no significant relationship between the softening point and the flow resistance at elevated temperature what is concerning to the mass modified by elastomers.

In the third and fourth steps, bitumen sheets were divided according to the flow resistance at elevated temperature. The temperature in values 130 °C and 150 °C have been marked as the value limits. For the model with the selection of bitumen sheets made in Europe except for Italy, the correlation coefficient was calculated in the value 0.83. Both the significance test and the interval estimate point show to the statistically significant linear dependence. The linear regression model has a coefficient of determination equal to 0.68, which is a much better result. The model as a whole is statistically significant, only the absolute member of the regression equation is statistically insignificant. The regression coefficient representing the slope of the regression line is 1.04, indicating that for this group of bitumen sheets the flow resistance at elevated temperature and softening point are very close to the values, which on average differ in the order of several percent. Bitumen sheets produced in Italy had the correlation coefficient only 0.34 and the test evaluated it by statistically insignificant. Therefore, the linear regression model also appears to be statistically insignificant. This shows the low value of the coefficient of determination (0.12) and the value of the p -value tests of individual coefficients and the significance of the whole model, all of which are significantly greater than the significance level of 0.05. Therefore, the linear dependence of the flow resistance at elevated temperature on the softening point is not shown for Italian bitumen sheets.

5. Conclusions

The dependence of the flow resistance at elevated temperature and the softening point of bitumen sheets were only demonstrated in the case of bitumen sheets with the mass modified by plastomers and bitumen sheets manufactured outside Italy. During the research it was found and confirmed that the dependence is influenced by the temperature value. The higher is the temperature, the more obvious is the dependence. For this reason, bitumen sheets with the mass modified by plastomers are significantly manifested. Thus, the hypothesis was only partially confirmed.

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