Pyrolytic product used as an alternative binder rejuvenator

Lidija Avsenik[1, a], Marjan Tušar[1, b]

[1] *National Institute of Chemistry, Ljubljana, Slovenia*

[a] lidija.avsenik@ki.si
[b] marjan.tusar@ki.si

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ABSTRACT

Binder rejuvenators are additives which purpose is to return aged bitumen its original properties. That means we can use a higher proportion of reclaimed asphalt pavement (RAP) in asphalt mixture when we add rejuvenator. Till now for rejuvenators were used different materials e.g. flux oil, tertiary amines, emulsions, soft bitumen, and combination of different compounds. In our research binder rejuvenator produced from scrap tires was tested. For producing so called alternative binder rejuvenator (BR) the pyrolysis process will be used. Prior to the production, pyrolysis process was properly modified in order to get the BR with adequate properties. By changing the time and temperature of the pyrolysis process we obtained different pyrolytic products. All of them and their blends with reference bitumen were investigated using standard tests for bituminous mixtures. Results of the tests showed that some of the pyrolytic products could be used as a binder rejuvenator. Test showed that milder terms of pyrolysis are more appropriate for rejuvenator production. Further tests on bitumen, aged bitumen and asphalt mixtures will be done to get more information about this new products.

Keywords: Additives, Ageing, Reclaimed asphalt pavement (RAP) Recycling, Rejuvenators

1 INTRODUCTION

Asphalt mixtures are commonly used for the surface layers on European roads. When the construction of new roads, or the reconstruction of old ones, is in progress we can choose to use virgin material, or we can reuse the materials from used asphalt, namely because the asphalt is a material that can be almost entirely recycled. Recycling is the process in which the reclaimed asphalt (RA) materials substitute the virgin materials. At first, asphalt recycling was considered as an option in the seventies during the oil crisis, when the prices of the bitumen greatly increased. Initially, recycled asphalt materials were primarily used for the road maintenance and the construction of the low-trafficked roads. Today, recycling methods are also considered for the heavy trafficked roads, and a recycling ratio may be close to 100%.

It is known, that during the road's service time, the binder in the asphalt mixture has been aged. The bitumen aging can be divided in two stages: the short-term and the long-term aging. The short-term aging occurs during the mixing and the construction process of the asphalt. The long-term aging continues throughout the lifetime of the pavement [1]. Many factors have influence on the aging process, the greatest impact have the UV radiation and the high temperatures. Two types of aging are known: the reversible and the irreversible aging. The irreversible process causes the chemical changes of the bitumen. The irreversible processes are: the oxidation, the loss of volatile components and the exudation. The reversible process is physical hardening, which causes changes in rheological properties without altering the chemical composition [1], [2], [3]. At ambient temperatures, physical hardening normally is very slow, but it can speed up at low temperatures [4]. Aged bitumen becomes harder, more brittle, its viscosity increase which results in loss of adhesion, ductility, and in the end, chip loss and cracking of asphalt layers [5], [6].

We are able to regain binder's initial properties by adding a rejuvenator to aged binder. Purpose of the rejuvenator is to return aged bitumen its original properties by reconstructing the chemical composition of the aged bitumen [7]. Rejuvenators are designed to soften the existing bitumen on the surface of the RA. Commonly bitumen becomes less viscous, more ductile and its coating properties are restored [8].

Different materials are used as rejuvenators e.g. flux oil, tertiary amines, emulsions, soft bitumen, combination of different compounds. In our research, binder rejuvenator was produced by pyrolyse of scrap tires as raw material. Today only a small percentage of a srap tires are landfilled, majority of them is recovered as a material [9]. Properties of the pyrolytic products, and their blends with bitumen were studied and compared to the properties of the reference bitumen made from the crude oil.

2 MATERIALS AND METHODS

2.1 Materials

All investigated pyrolytic products were obtained by the pyrolyse of the scrap car tyres. The pyrolysis process is a thermochemical decomposition of organic material at elevated temperatures in the absence of oxygen. It involves the simultaneous change of the chemical composition and the physical phase, and is irreversible. The pyrolytic products were generated by the condensation of the vapours from a batch reactor, where pyrolysis took place at different temperature and it lasted for different time. In Table 1 are presented the produced pyrolytic products. The exact temperatures and times at which the pyrolyse was conducted are not given as they are business secret. But from Table 1 it is clear for which product the temperature was elevated/reduced regarding to reference temperature ($T_{ref} \pm Y$), and how the time was prolonged/shortened regarding to reference time ($t_{ref} \pm X$). The last three pyrolytic products (9 th ,10 th and 11 th) were added oils from the previous pyrolysis processes, and different proportions of the rubber from the scrap car tires. The oils were extracted from the water cooled condensator.
Depending on time and temperature of pyrolysis, we can categorize the pyrolytic products, from the ones which were produced under the harshest terms, to the ones which were produced under the mildest terms. This is presented in Table 1. Pyrolytic product 4, which is labeled with number 1, in the column of the terms of the pyrolsis process, was produced at the harshest conditions, and the pyrolytic product 11 was produced at the mildest conditions.

Table 1: List of the investigated pyrolytic products

	Duration of the pyrolyse t_{ref} + X [min]	Temperature of pyrolyse T_{ref} + Y [°C]	Remarks	Terms of the pyrolyse process - from the harshest (1) to the mildest (11)
Pyrolytic product 1	t_{ref} + 5	T_{ref} +20	-	4
Pyrolytic product 2	t_{ref}	T_{ref} +60	-	3
Pyrolytic product 3	t_{ref}	T_{ref} -5	-	5
Pyrolytic product 4	t_{ref} + 5	T_{ref} +80	-	1
Pyrolytic product 5	t_{ref} -1	T_{ref} +80	-	2
Pyrolytic product 6	3* t_{ref}	T_{ref} -12	-	7
Pyrolytic product 7	3*t_{ref}	T_{ref} -80	-	9
Pyrolytic product 8	0	T_{ref} +40	-	6
Pyrolytic product 9	30*t_{ref}	T_{ref} -20	Added different oils from the previous pyrolysis processes and different proportions of the rubber from the scrap car tires	8
Pyrolytic product 10	Firstly: 3* t_{ref} and T_{ref} -150; secondly: 3* t_{ref} and T_{ref} -30; thirdly: 3* t_{ref} and T_{ref} -10			10
Pyrolytic product 11	Firstly: 3* t_{ref} and T_{ref} -150; secondly: 3* t_{ref} and T_{ref} -28; thirdly: 3* t_{ref} and T_{ref} -18			11

Bitumen of the penetration grade 50/70 was used as a matrix of the blends.
Bitumen was mixed with the pyrolytic products, which were obtained by the pyrolysis. All the blends were laboratory produced by adding a controlled quantity of the pyrolytic product to the bitumen. The blends of bitumen and pyrolytic product were all produced by mixing two of the components in ratios of 1:1 to produce a blended sample of 60 g total mass (30 g by 30 g). The blending process consisted of heating both components to mixing temperature for 90 minutes and pouring the required masses into a small container. The two components were then manually stirred together for approximately 60 s to produce a uniformly distributed binder blend. The blends were then poured into sample containers, and stored at 5 °C prior to testing. Only the last three samples (11_5%, 11_10%, and11_20%) were also prepared in smaller concentrations. Concentrations of the different samples are presented in Table 1.

Table 2: Blends and their ratio between reference bitumen and pyrolytic product

Ratio between components	Reference bitumen B 50/70	Sample
Pyrolytic product 1	1:1	B + 1
Pyrolytic product 2	1:1	B + 2
Pyrolytic product 3	1:1	B + 3
Pyrolytic product 4	1:1	B + 4
Pyrolytic product 5	1:1	B + 5
Pyrolytic product 6	1:1	B + 6
Pyrolytic product 7	1:1	B + 7

Pyrolytic product 8	1:1	B + 8
Pyrolytic product 9	1:1	B + 9
Pyrolytic product 10	1:1	B + 10
Pyrolytic product 11	1:1	B + 11
Pyrolytic product 11	1:20	11_5%
Pyrolytic product 11	1:10	11_10%
Pyrolytic product 11	1:5	11_20%

2.2 Methods

For characterizing the samples we used the standard testing methods, normally used for the bituminous mixtures, defined in the European standards. The softening point, the ductility and the Fraass breaking point were determined in order to make the comparison between the bitumen, the pyrolytic products and their blends.

2.2.1 Viscosity

With the rotational viscometer Haake RS50, the viscosity, the mixing temperatures and the compaction temperatures were determined.

2.2.2 Ring and ball test

For assessing the softening properties, the Ring and Ball method was used as determined in the EN 1427:2007 [10]. The softening point is determined by heating the samples casted in brass ring to the point where the steel ball, lying on the top of binder, envelop in and eventually fall through a binder for a prescribed distance of 25 ± 0.4 mm.

2.2.3 Fraass test

The brittleness at the low temperatures of the binders was determined by the Fraass breaking point according to the EN 12593:2007 [11]. A binder sample is applied to a metal plate at an even thickness. This plate is submitted to a constant cooling rate and flexed repeatedly until the binder layers breaks. The temperature at which the first break occurs is reported as the Fraass breaking point.

2.2.4 Ductility test

The tensile properties of the binders were determined by the force ductility method in accordance to the EN 13589:2008 [12]. After casting binder, the moulds are transferred to the traction plates and then stretch at the prescribed speed at 25 °C up to the elongation 1500 mm. If the specimen breaks before it reaches the prescribed elongation, the brittle break has occurred.

3 RESULTS AND DISCUSSION

3.1 Viscosity

The rotational viscometer test method is presently considered to be the most practical means of determining the viscosity of the bitumen. The instrument allows the testing of the binders over a wide range of the temperatures [13]. The viscosity is the ability of a liquid to resist the flow. Therefore, the binders with a high viscosity have difficulty flowing, while the binders with low viscosity tend toward the state of a Newtonian liquid [14]. Determination of the mixing and compaction temperature for each pyrolytic product was done by measuring the viscosity using the rotational viscometer Haake RS50. The mixing, and the compaction temperatures, for the asphalt mixtures are determined at the elevated temperatures from the plain binder viscosity at 0.170 ± 0.02 Pas and 0.280 ± 0.03 Pas respectively [15]. Viscosity values are not constant as they are affected by many conditions. The most important are temperature and shear rate.
The results of the mixing and compaction temperatures are given in Table 3. As we can see, the mixing and the compaction temperatures of the pyrolytic products are higher than the reference bitumen's. We can assume that the pyrolytic products have higher viscosity. Also the homogeneity of the sample influenced the viscosity. More the sample is inhomogeneous, more its viscosity increase. Pyrolytic products 2, 9, 10, and 11 were inhomogeneous.

Pyrolytic product 11 was extremely inhomogeneous (containing visible small particles) and it has the highest mixing temperature.

Table 3: The mixing and the compaction temperatures

	Mixing temperature (η=0.17 Pas)	**Compaction temperature (η=0.26 Pas)**		**Mixing temperature (η=0.17 Pas)**	**Compaction temperature (η=0.26 Pas)**
Reference bitumen and pyrolytic products	[°C]	[°C]	Blends	[°C]	[°C]
B 50/70	141.2	121.9	B + 1	152.1	125.8
1	153.6	127.1	B + 2	146.0	123.3
2	192.9	159.9	B + 3	143.6	121.9
3	169.2	141.2	B + 4	146.6	123.3
4	185.6	156.8	B + 5	152.1	128.0
5	187.8	158.6	B + 6	142.2	119.2
6	169.3	138.8	B + 7	140.1	118.2
7	139.4	116.4	B + 8	150.7	129.1
8	177.6	153.1	B + 9	158.6	129.3
9	190.6	150.3	B + 10	135.3	113.8
10	155.7	121.5	B + 11	140.3	105.2
11	202.3	154.4	11_5%	140.4	118.5
			11_10%	134.8	111.8
			11_20%	129.6	102.5

Our research is directed to investigate the effect of the pyrolytic product on the reference bitumen. On Figure 1 relative values of the mixing and the compaction temperatures of the blends regarding to the reference bitumen are presented. Figure 1 shows how the pyrolytic product decreased or increased the mixing, and the compaction, temperature of the reference bitumen. If the relative value is higher than 1, the temperature of blend is higher than B 50/70's temperature. If the value is less than 1 the temperature of blend is lower than B 50/70's temperature.

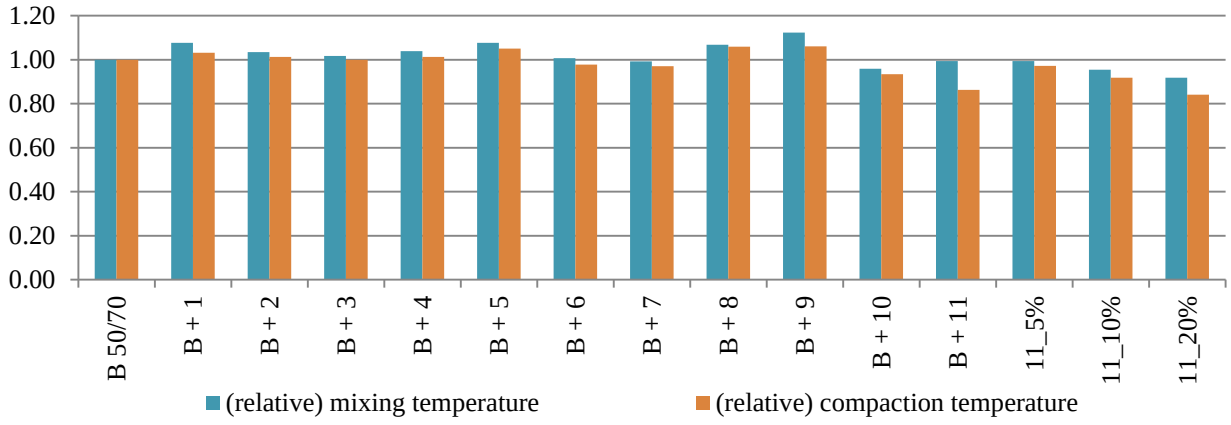

Figure 1: Relative mixing and compaction temperatures of the blends regarding to the reference bitumen B 50/70

Although the pyrolytic products all had higher compaction and mixing temperatures, their blends with bitumen had lower temperatures, except for B+5, B+8 and B+9. B+1, B+2, B+3 and B+4 had approximately the same temperature. Blends with the pyrolytic product 10 and 11 had significantly lower temperatures. It is interesting, that

increasing the proportion of product 11, from 5 % to 20 %, is lowering the mixing, and the compaction temperature. Blend with concentration 50 % (B+11) had the mixing temperature similar to B 50/70, compaction temperature is smaller.

Viscosity of the blends was calculated at three different temperatures and was measured at the constant shear rate. The results are presented in Table 4. As mentioned before, the temperature is very important factor, which influences the viscosity. From Table 4 it is clear that the viscosity of the bitumen and the blends is decreasing with increasing temperature.

Table 4: Viscosity of the blends

		Viscosity	
	T = 60 °C	**T = 100 °C**	**T = 150 °C**
	[Pas]	[Pas]	[Pas]
B 50/70	209.37	2.03	0.12
B + 1	71.50	1.56	0.23
B + 2	104.19	1.67	0.17
B + 3	110.84	1.67	0.15
B + 4	92.59	1.59	0.17
B + 5	99.02	3.36	0.18
B + 6	61.04	1.31	0.14
B + 7	71.39	1.21	0.13
B + 8	234.85	2.60	0.21
B + 9	56.31	1.74	0.29
B + 10	30.91	0.90	0.12
B + 11	6.93	0.43	0.15
11_5%	73.03	1.35	0.12
11_10%	31.14	0.90	0.09
11_20%	9.01	0.53	0.09

Figure 2 shows relative values of the viscosity, at different temperatures, regarding to the reference bitumen. The viscosity of all blends, except B+8, is lower than half of the viscosity of the references bitumen at 60 °C. B+8's viscosity is higher than the references bitumen, at mentioned temperature. At 100 °C the viscosity of the blends had increased, both, absolute values and relative values, comparing to the reference bitumen. B+5 and B+8 are the only blends that had the viscosity higher than reference bitumen, other's viscosity was smaller. At 150 °C all blends viscosity was at least as high as the reference's bitumen, except for the smaller proportion blends of the pyrolytic product 11 (11_10% and 11_20%).

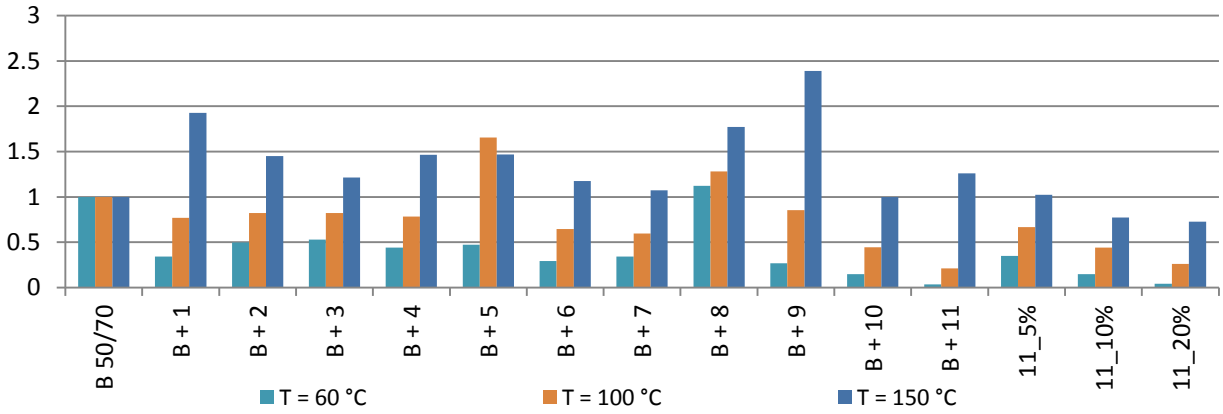

Figure 2: Relative viscosity values of blends at different temperatures regarding to the reference bitumen B 50/70

3.2 The softening point temperature and the Fraass temperature

Pyrolytic products 2, 3, 4, 5, and 8 had higher softening point than the reference bitumen. The highest softening point had products 4 and 5, the lowest had the pyrolytic product 10. For pyrolytic product 11 the softening point could not be measured, because the sample was in liquid state at the ambient temperature.
Pyrolytic products 2, 5, 8, and 11 had higher Fraass breaking point, other products had lower values. The Fraass breaking point for the product 8 is a positive temperature, which was expected, because the product was brittle at ambient temperature.
All the results are presented in Table 5.

Table 5: Results of the R&B, the Fraass and the ductility tests

					Ductility (EN 13589)		
	R&B (EN 1427)	**R&B mixing law (EN 13108-1)**	**Fraass (EN 12593)**	**Temperature range (R&B-Fraass)**	**Elongation**	**Force**	**Energy**
	[°C]	[°C]	[°C]	[°C]	[mm]	[N]	[J/cm2]
B 50/70	50.1	-	-10.8	60.85	1500.01	0.97	0.07
1	43.3	-	-13.5	56.8	235.5	0.1	0
2	58.4	-	2.4	55.95	186.92	1.01	0.06
3	54.1	-	-18	72.1	136.61	0.34	0.02
4	60.7	-	-11.5	72.15	256.72	1.41	0.1
5	60.0	-	-7.5	67.5	353.82	1.41	0.12
6	46.9	-	-20.3	67.15	231.93	0.27	0.02
7	45.5	-	-13.6	59.05	826.55	0.41	0.02
8	64.5	-	1.4	63.1	1308.85	12.59	0.91
9	41.7	-	-20	61.7	145.03	0.14	0.01
10	30.2	-	-22.9	53.1	202.12	0.04	0.00
11	-	-	-0.6	-	-	-	-
B + 1	43.0	46.7	-12.8	55.75	902.44	0.31	0.02
B + 2	46.0	54.2	-8.4	54.4	534.14	0.52	0.03
B + 3	46.7	52.1	-18.3	65	334.53	0.58	0.04
B + 4	46.5	55.4	-11.4	57.9	1500.01	0.96	0.06
B + 5	46.2	55.0	-10.4	56.55	1492.35	0.82	0.05
B + 6	44.5	48.5	-18.3	62.75	759.86	0.43	0.03
B + 7	45.3	47.8	-14.1	59.35	1500.01	0.72	0.04
B + 8	51.9	57.3	-5.6	57.5	1500.01	3.15	0.22
B + 9	37.8	45.9	-23.6	61.4	1500.00	0.67	0.10
B + 10	36.9	40.1	-21.9	58.75	1500.01	0.61	0.06
B + 11	-	-	-24	-	-	-	-
11_5%	42.5	-	-16.7	59.2	1342.9	2.6	0.23
11_10%	36.3	-	-22.5	58.75	746.04	0.45	0.03
11_20%	22.3	-	-23.2	45.5	-	-	-

Figure 3 shows the relative values of the softening point and the Fraass breaking point comparing to the reference bitumen. The pyrolytic products lowered the softening point of the blends. All the blends had lower softening point than the reference bitumen. For our product this is a good feature, as we want to use it as a rejuvenator. That means the pyrolytic product could lower the high softening point of the extracted bitumen in reclaimed asphalt. The blend containing 50 % of pyrolytic product 11 was in liquid state at ambient temperature, so the measurement of softening point was not possible.

The softening point of the blends can be calculated according to the mixing law determined in EN 13108-1:2006/AC:2008 [16]. The values are calculated by the following equation ″Eq (**1**)″:

$$T_{R\&B,m} = \frac{B_0}{100} T_{R\&B,m} + \frac{B_{PP}}{100} T_{R\&B,PP} \qquad (1)$$

Where:

B_0, B_{PP}	are the percentages of the the bitumen and the pyrolytic product in the blend
$T_{R\&B,0}$, $T_{R\&B,PP}$, $T_{R\&B,m}$	are the temperatures of the softening point for the bitumen, the pyrolytic product and the blend

The results of the calculated softening points are presented in Table 5. For all pyrolytic products the calculated softening point is higher than the measured. The equation 1 is actually just an arithmetic mean of the pyrolytic product's softening point and the references bitumen's softening point, because the ratio between pyrolytic product and the reference bitumen is 1:1. The calculated and measured values did not match well. That means the blends are not just mix of the two components, but also some reactions happened between those two components.
Also on Figure 3 the relative values of the Fraass breaking point are presented. Almost all pyrolytic products lowered the Fraass breaking point (B+1, B+3, B+4, B+6, B+7, B+9, B+10, all blends containing pyrolytic product 11), the exceptions were pyrolytic products 2, 5, and 8, whose blends had higher Fraass point. Lowering Fraass breaking point means, that the temperature range is extended.

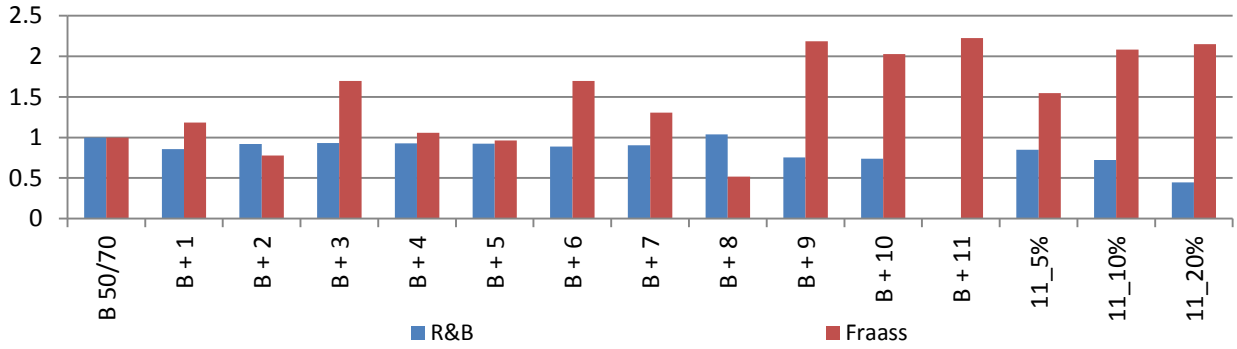

Figure 3: Relative values of the Ring & ball test and the Fraass test of the blends regarding to the reference bitumen B 50/70

3.3 Tensile properties

We performed the ductility test in order to asses the tensile properties of the pyrolytic products and the blends. The reference bitumen reached the elongation of 1500 mm. All pyrolytic products broke before they reached the whole elongation. The pyrolytic product 8 was the closest to reach the reference bitumen, with elongation 1308,85 mm. Also the pyrolytic product 8 had the highest force, measured at the test, which is 12,59 N. All other values of force measurements were in the range around 1 N or even smaller. The ductility test was performed at 25 °C for the pyrolytic products (and their blends) from 1 to 8. For others the test temperature was lowered, to 15 °C. Pyrolytic product 11 and its blends, B+11 and 11_20%, could not be tested even at the temperature 15 °C, so we did no performed test for those three samples. The results of all ductility tests are given in Table 5.

Figure 4 shows us the relative values of the elongation and the force, from the ductility tests of the blends, in comparison to the reference bitumen. The pyrolytic products 4, 5, 7, 8, 9, 10, and 11 (in adequate concentration) retained the elongation of the reference bitumen in the blends. All other pyrolytic products shortened the elongation of the blends.

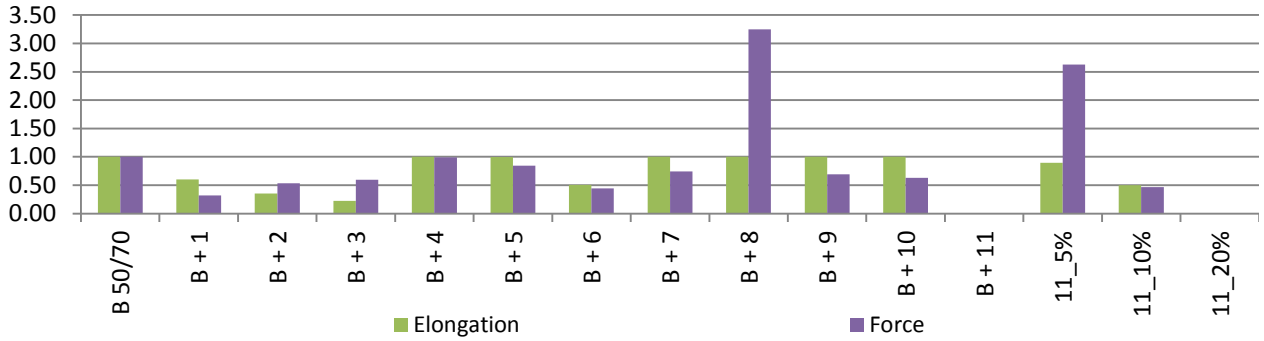

Figure 4: Relative values of the ductility test of the blends regarding to the reference bitumen B 50/70

4 CONCLUSIONS

We were testing the pyrolytic products as a possible rejuvenator for the binders in the reclaimed asphalt. We tested eleven different pyrolytic products, produced at different terms of the pyrolysis process.

As mentioned in introduction, when binder ages, it become more brittle and its viscosity increases. That means the effect of reduced viscosity is a good indicator, that pyrolytic product could be used as a rejuvenator. Also lowering the softening point is useful for aged binders, where the softening point has increased. That means that hard, aged binder again becomes softer.

Pyrolytic product 11 was produced at the mildest terms of pyrolysis, and for final product, oils from previous pyrolsis, and rubber from scrap tires were added. Because of these oils the product was liquid at ambient temperature and we had problems performing standard tests on samples, which included too much proportion of pyrolytic product 11. Only at concentration of 5 % all the tests were possible to conduct. At this small concentration, the pyrolytic product 11 retained mixing temperature and decreased compaction temperature, softening point, and Fraass breaking point. The viscosity of 11_5% is lower at 60 °C and 100 °C, but higher at 150 °C than reference's bitumen. The pyrolytic product 10 was also produced at mild terms of the pyrolysis. Results show it decreased the mixing and compaction temperature, and viscosity of the reference bitumen. Also the softening point and the Frass breaking point were decreased compared to the reference bitumen. At the same time the elongation was preserved. The viscosity at 60 °C and 100 °C had decreased, at 150 °C was the same as of the reference bitumen. The pyrolytic product 10 also had addition of oils and rubber. The pyrolytic product 7 was produced at the mildest terms of the pyrolyse, if we exclude products with added oils and rubber. Blend B+7 had the same mixing temperature as reference bitumen, but its compaction temperature is lower. The softening point and the Fraass point have decreased, the elongation was the same as the reference's bitumen, but the measured force was lower. The viscosity was lower than reference's bitumen at first two temperatures, 60 and 100 °C, but was higher than reference's bitumen at 150 °C. On the other side of the pyrolsis terms we had the pyrolytic product 4. It was produced under the harshest terms. The mixing and compaction temperatures of blend with reference bitumen had increased comparing to pure bitumen, but only for 4 % and 1 % respectively. The softening point had lowered, as it had the Fraas point, but again only for 7 % and 6 % respectively. The elongation, and the force, at the ductility test of the blend B+4 were the same as for the reference bitumen. The viscosity of the blend had decreased at 60 and 100 °C, but it had increased at 150 °C. All those results show the pyrolytic products produced at milder terms of pyrolysis have better predispositions, than the pyrolytic products produced at harsher terms. If we add oils and rubber to the pyrolytic product we can reduce the concentration of pyrolytic product in the blend.

Besides decreasing the dependency on the crude oils, the new product could also have a positive effect on the environment since it was produced from the waste material and its use would increased the use of the reclaimed asphalt. Binder is the most expensive component of the asphalt mixture, but using RA and rejuvenator means we could lower the content of fresh binder. Also, lowereing the mixing and compaction temperatures is cost efficient, saves energy and is more ecological. Lower compaction temperatures means longer transportation distance and time [17]. This is because the asphalt can be produced at ordinary temperatures and transpoted further than normally. The temperature of asphalt will be lower at the destination, but the asphalt containing rejuvenator can be compacted as good at lower temperatures, as reference asphalt at higher temperatures. Lower temperature on the other hand means less energy consumption in the production of asphalt mixtures, and thus reduced greenhouse gas emissions. When installing asphalt, this means less emission of bitumen fumes and vapours, and thus less harmful to the health of

workers. Equally important is that by using RA we preserve nature, reduce usage of virgin raw materials and reduce waste. All the above mantioned are showing positive effect on ecology. But we must be aware, that the product itself must be further tested to confirme it is not harmful for human and environment.
More tests on the pyrolytic product as an additive, rejuvenator in our study, (rheology tests, affinity tests) and also tests of asphalt mixtures containing RA and rejuvenator (pyrolytic product) are planned in the future to get more information. Additional testing will also ensure, that the pyrolytic product doesn't contain any dangerous substance e.g. PAHs.

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