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TEMPERATURE EFFECT ON THE PROCESS OF ROAD BITUMEN MODIFICATION WITH CARBONIZED RESIDUE FROM THE WASTE TIRES PYROLYSIS

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Abstract. The possibility of utilizing solid carbonized residue (CR) obtained in the pyrolysis of waste automobile tires (WT) was analyzed. On average, as a result of WT pyrolysis, about 36 wt. % of carbonized residue is obtained, which can serve as an adhesion additive and/or aging inhibitor in modifying petroleum bitumen. The CR obtained at a small-tonnage industrial plant and bitumen of the BND 70/100 brand produced by PJSC "Ukrtatnafta" were analyzed. The influence of the carbonized residue on the operational properties of modified bitumen was established for different ratios of raw materials (BND 70/100 bitumen):CR, and at different mixing temperatures. According to the obtained results, the optimal amounts of CR and temperatures for modifying petroleum bitumen were proposed.

Keywords: waste tires, pyrolysis, carbonized residue, bitumen modification, adhesion additive, carbon black.

1. Introduction

Given the decline in global oil reserves, using alternative energy sources is becoming a pressing issue. The oil refining industry is gradually adapting to cheaper and heavier feedstocks due to the depletion of expensive and light oil. This prompts a gradual transition to unconventional feedstocks such as waste and biomass.[1] It is worth noting that the reuse of industrial waste (waste tires, oils, plastics, etc.) for energy production can reduce the negative impact on the environment.[1-6] Particularly promising in this aspect is the use of used automobile tires (WT), which are considered the most common solid waste in the world generated due to vehicle operation.

Given current trends that show a rapid increase in tire production, with the global industry expected to produce more than 2,700 million tires by the end of 2027, the issue of tire recycling is becoming increasingly important.[7]

Typically, most tires have a service life of fewer than four years, after which they turn into waste that takes up significant landfill space. It is known that about 5 million tonnes of tires are disposed of globally each year, while a much larger amount of WT is accumulated.[7-10]

Today, about 52% of WT waste is recycled globally, 43% is used for energy production, and the remaining 5 % is landfilled or incinerated.[11,12] Waste tires require large landfills for disposal, as they are bulky, biodegradable, and can remain in landfills for up to a hundred years. This method of waste disposal is also expensive and environmentally hazardous.[13,14] Uncontrolled tire accumulation poses significant risks to public health and the ecosystem, causing a specific type of environmental pollution known as black pollution.[14]

Nearly 1.5 billion waste tires are discarded at the end of their useful life each year, and this figure is on the rise and could reach 5 billion by the end of 2030. In China, for example, 210 million tonnes of WT are discarded annually,[15] and their disposal has already become a serious environmental issue. In the EU, about 18% of the waste tires were disposed of or reused, 38% were recycled, and 40% were used for energy production.[15] The total average amount of WT generated annually is shown in Fig. 1.

It is known that the main components of waste tires are rubber, fillers, soot, steel, sulfur, zinc oxide, process oils, vulcanization accelerators, etc. The relevant waste management includes collection, transportation, processing, and disposal (including landfilling). In general, the disposal of waste tires and other polyisoprene-based products is problematic.[16] The main ways of disposing and recycling waste tires include landfilling, reuse, incineration, use as a raw material for pyrolysis, carbon black production, and use in civil engineering.[15]

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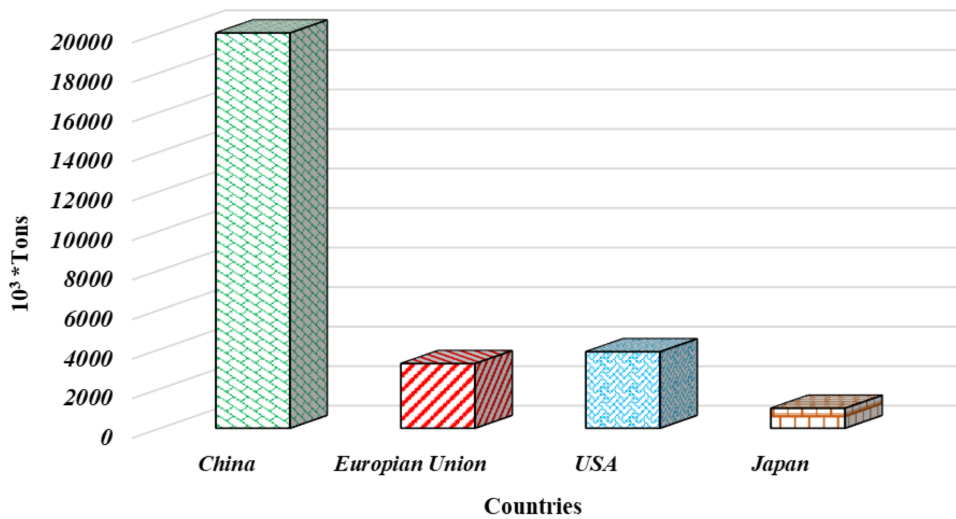

Fig. 1. The number of waste tires generated per year in some countries

In addition, some waste tires are used to prepare modified bitumen.[4,17] At the same time, the high energy intensity of WT has led to its reuse in various industries. Processes such as gasification,[18] combustion,[19] and pyrolysis[20,21] of WT allow maximizing resource utilization while reducing dependence on traditional fossil fuels. The methods of waste tire disposal and their typical applications are shown in Fig. 2.

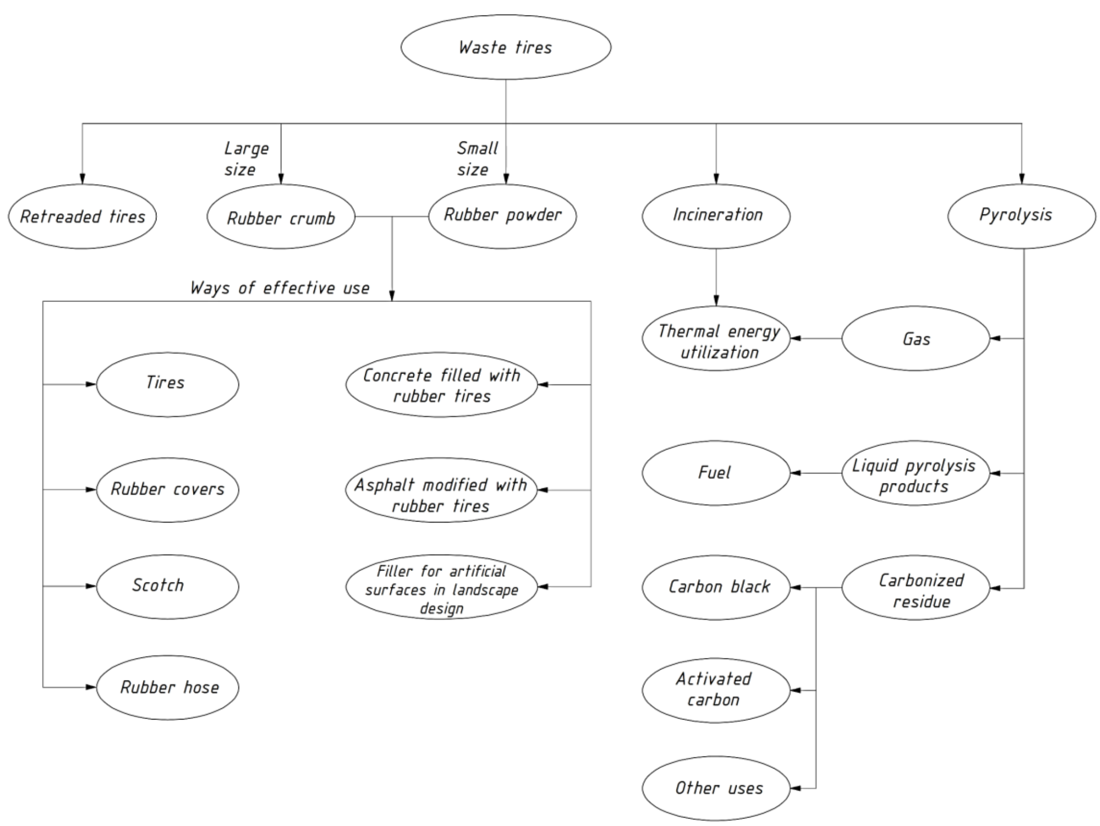

Fig. 2. Methods of disposal of waste tires and their common application areas

Pyrolysis is one of the most promising and environmentally sustainable ways to solve the problem of waste disposal. This process is an effective alternative to landfilling and incineration. It allows for converting waste into valuable products, such as liquid fuels and chemical compounds with significant industrial value.[2,5] As a result of the WT pyrolysis process, a mixture of pyrolysis gases (1.7-77.0 wt. %), liquid pyrolysis products (14.9-63.7 wt. %), and solid carbonized residue (CR) in the amount of 21.6-69.0 wt. %.[1,2] The carbonized residue is a by-product of the WT pyrolysis process. It consists of soot (80-90 wt. %) and inorganic substances (10-20 wt. %), always in rubber compounds used in the production of tires.

Thus, about 36 wt. % of the solid (carbonized) residue from the WT pyrolysis process can be used in further processes to produce carbon black, activated carbon, *etc*.[2,22] There is also the possibility of using the carbonized residue as an oil adsorber, insulator, or concrete component.[23]

Deeply carbonized activated residue (DCAR) has also been tested as a filler to improve the thermal stability of bitumen. It has been found that adding DCAR positively affects the rheological properties of bitumen and its aging.[24] However, it is not clear how the DCAR obtained at relatively low temperatures, which has not undergone modification, affects the performance properties of bitumen (primarily, adhesive properties and aging resistance). In addition, deep-ground DCAR can be used in quantities exceeding 5 wt. % was investigated in the work.[24]

Therefore, these studies aimed to establish the possibility of using the «standard» carbonized residue as an adhesive additive and/or aging inhibitor in modifying BND 70/100 bitumen, which was added in amounts of 0.5-5.0 wt. %.

2. Experimental

2.1. Materials

The carbonized residue (fr. 0.56-0.75 mm) of the WT pyrolysis process was obtained in a small-scale industrial unit according to method[25] described by the authors previously. The characteristics of the CR properties are given below in Table 1.

Table 1. Characteristics of the carbonized residue obtained from the WT pyrolysis process

Index	Unit of measurement	Value	Method
Total moisture content	%	1.8±0.2	ISO 589:2015[26]
Ash on dry conditions,	%	9.6±0.2	GOST 11022-95[27]
Volatile matter content: in the dry condition on dry ashless condition	%	 2.9±0.2 3.2±0.2	ISO 562:2015[28]
Dry sulfur content	%	2.09±0.05	ISO 351:1996[29]
Lower heat of combustion in dry combustion	kJ/kg	30297±146	ISO 1928:2006[30]
Lower heat of combustion in operating conditions	kJ/kg	29707±146	ISO 1928:2006[30]
Content of carbon in the dry condition	%	86.56±0.15	ISO 625:1996[31]

Oxidized petroleum bitumen BND 70/100, which was selected at PJSC Ukrtatnafta (Kremenchuk refinery), was used as a raw material for the modification process. Its main characteristics are given in Table 2.

Table 2. Physicochemical characteristics of the initial bitumen

Index	Unit of measurement	Value	Method
Penetration at 25 °C	dmm	76	EN 1426:2015[32]
Softening point	°C	49.6	EN 1427:2015[33]
Ductility at 25 °C	cm	66	According to[34]
Elastic recovery at 25 °C	%	15.5	EN 13398:2018[35]
Adhesion to gravel	mark	3.5	DSTU 8787[36]
Adhesion to glass	%	59	DSTU 9169:2021[37]
Resistance to hardening at 163 °C (RTFOT method):			EN 12607-1:2015[38]
mass change	wt. %	0.11	
softening point after RTFOT	°C	58.2	
penetration at 25 °C after RTFOT	dmm	38	
softening point change	°C	8.6	
retained penetration	%	50.0	

2.2. Methods

Determination of the quality characteristics of the initial and modified bitumen samples was carried out according to the methods given in the relevant regulatory documents.[32-38]

The modification process of the initial oxidized BND 70/100 bitumen was carried out at a laboratory installation with the addition of CR as a modifier according to the following procedure:

– the specified amount of bitumen was heated to the required process temperature (± 5 °C) in a laboratory setup consisting of a metal container, an electric stove, and equipped with a mechanical stirrer and a thermometer;

– switch on the stirring and fix the stirrer speed at N = 1000 rpm;

– add the specified amount of modifier (CR) to the heated bitumen, recording the beginning of the modification process, the duration of which was τ = 3.0 hours;

– switch off heating and stirring after the process was completed;

– modified samples were analyzed using standardized methods.

The modification conditions were selected based on the authors' previous studies.[39]

Based on the characteristics of the obtained products, conclusions were made about their compliance with the requirements of regulatory documents in the bituminous road construction materials field.

3. Results and Discussion

According to the methodology described above, the process of modifying the initial oxidized bitumen of BND 70/100 grade using carbonized residue was carried out. The ratios of bitumen:CR components were 1:0.5; 1:1.0; 1:3.0; 1:5.0 wt. %.

The mixing of the mixture components was carried out at different process temperatures, in particular at 120, 170, and 190 °C, in order to establish the optimal modification conditions.

The research results are presented below in Figs. 3-8, as well as in Tables 1-3.

The following conclusions can be drawn according to the above results, Figs. 3-8. It was found that the best penetration values (Fig. 3) are observed for samples modified with CR in the amount of 0.5 wt. % per raw material at temperatures of 120 and 170 °C, while the further addition of CR slightly reduces this indicator. At the same time, when samples with CR in the amounts of 1.0 – 3.0 wt. % per raw material were modified at a temperature of 120 °C, the penetration value remains approximately constant. In contrast, with a further increase in the process temperature and the amount of CR, the penetration decreases slightly.

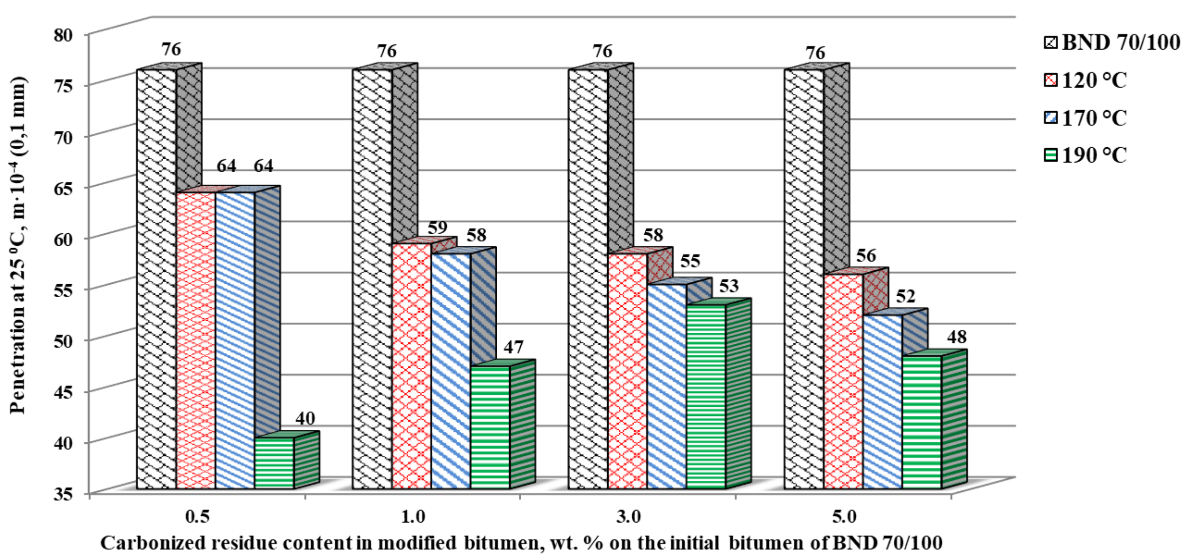

Fig. 3. Dependence of penetration on the modification temperature and the content of CR in the modified bitumen, wt. % on the initial bitumen of the BND 70/100
(following the requirements[40] for BND 50/70, the penetration value at 25°C should be 51-70 and the requirements[41] for BND-A 40/60, the penetration value at 25°C should be 40-60)

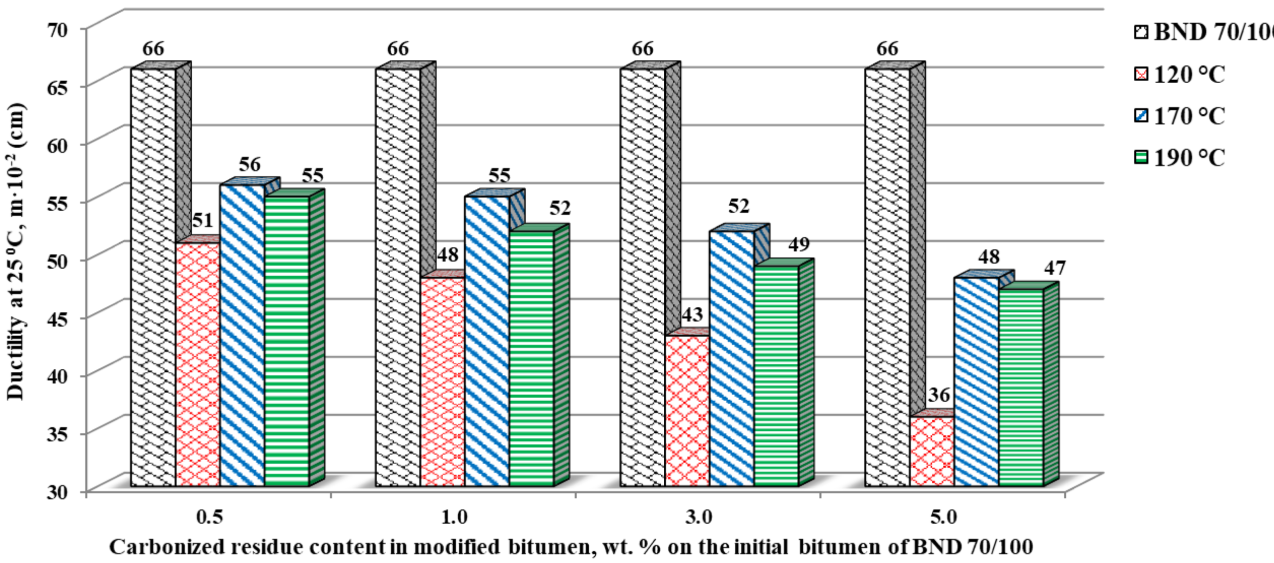

Fig. 4. Dependence of ductility on the modification temperature and the content of CR in the modified bitumen, wt. % on the initial bitumen of the BND 70/100 (following the requirements40 for BND 50/70 the ductility at 25 °C should be ≥50, and the requirements41 for BND-A 40/60, the ductility at 25 °C should be ≥45)

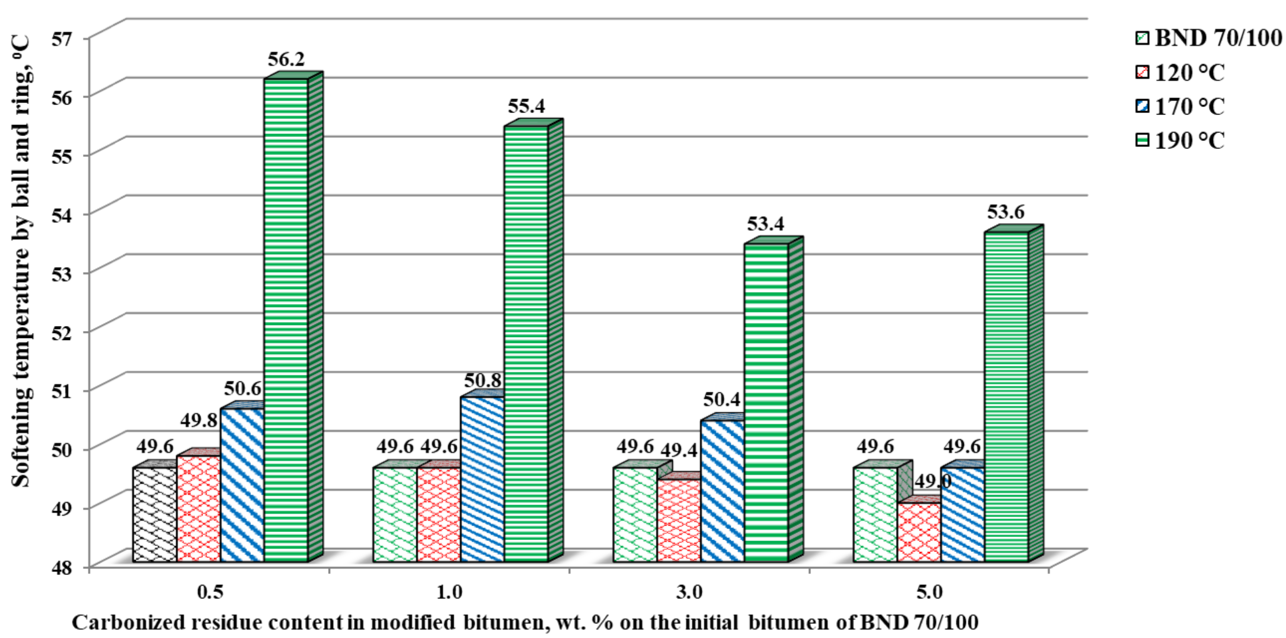

Fig. 5. Dependence of softening temperature on the modification temperature and the content of CR in the modified bitumen, wt. % on the initial bitumen of the BND 70/100 (following the requirements40 for BND 50/70 the softening point should be within 49-55 °C and the requirements41 for BND-A 40/60, the softening point should be within 51-57 °C)

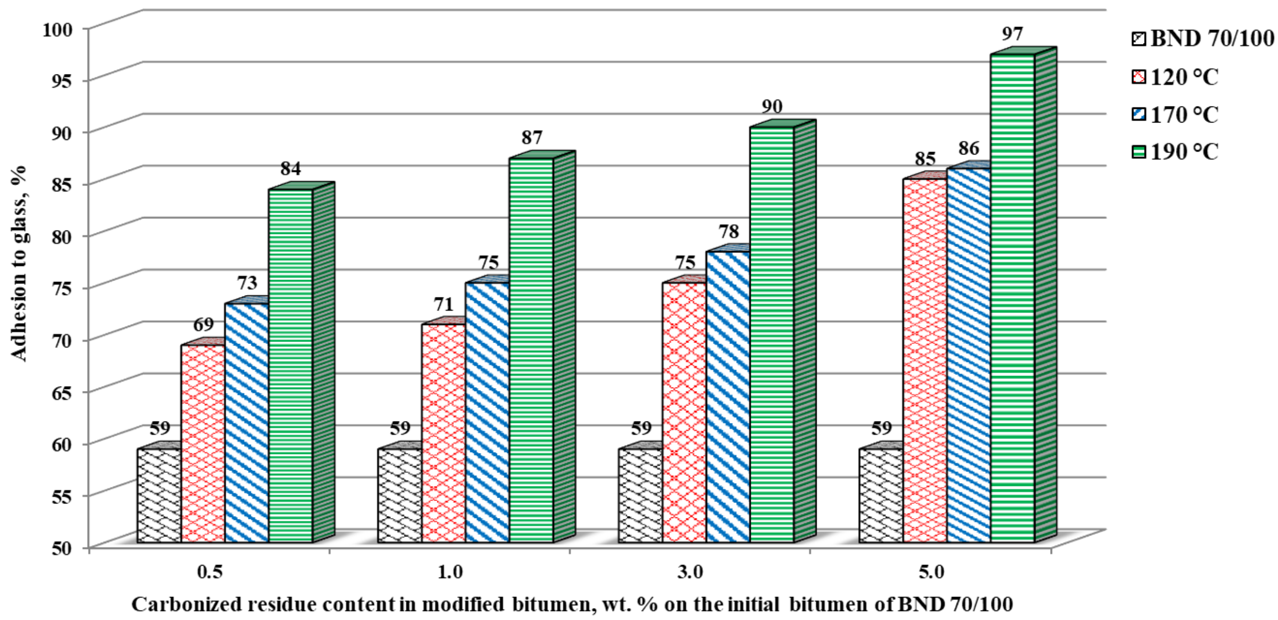

Fig. 6. Dependence of adhesion to glass on the modification temperature and the content of CR in the modified bitumen, wt. % on the initial bitumen of the BND 70/100 (following the requirements40 for BND 50/70, the value of adhesion to the glass surface should be ≥ 25, while following the requirements41 for BND-A 40/60 – ≥75)

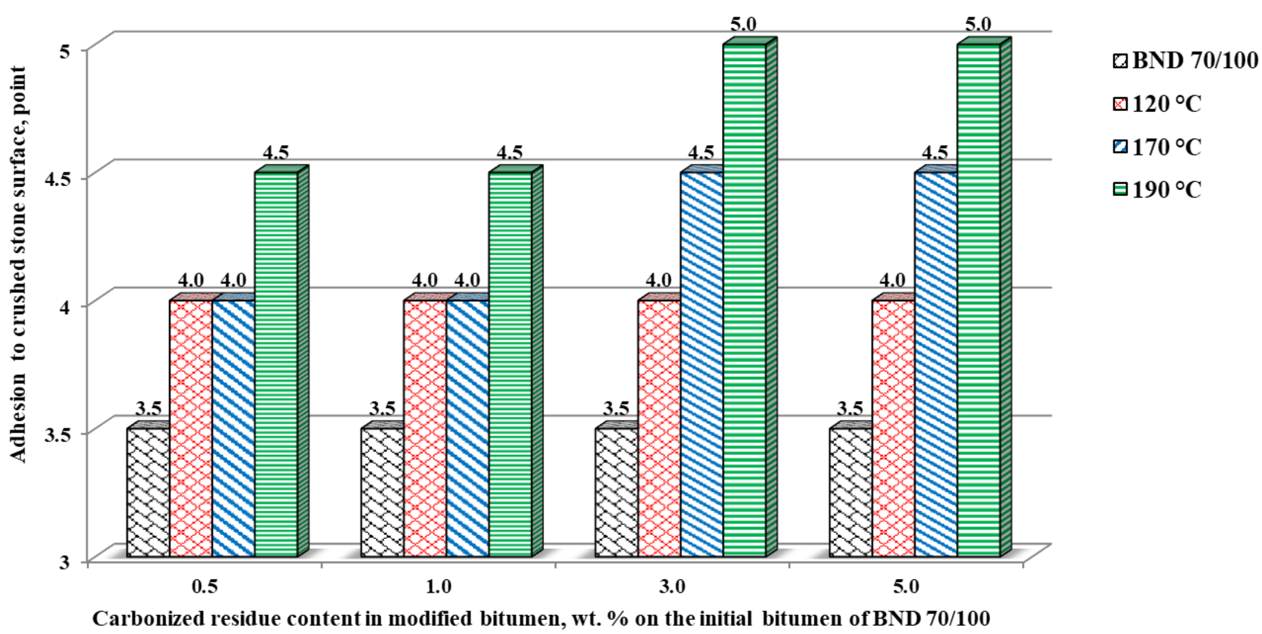

Fig. 7. Dependence of adhesion to crushed stone surface on the modification temperature and the content of CR in the modified bitumen, wt. % on the initial bitumen of the BND 70/100 (following the requirements41 of BND-A 40/60, the value of adhesion to the crushed stone surface should be ≥ 5 points)

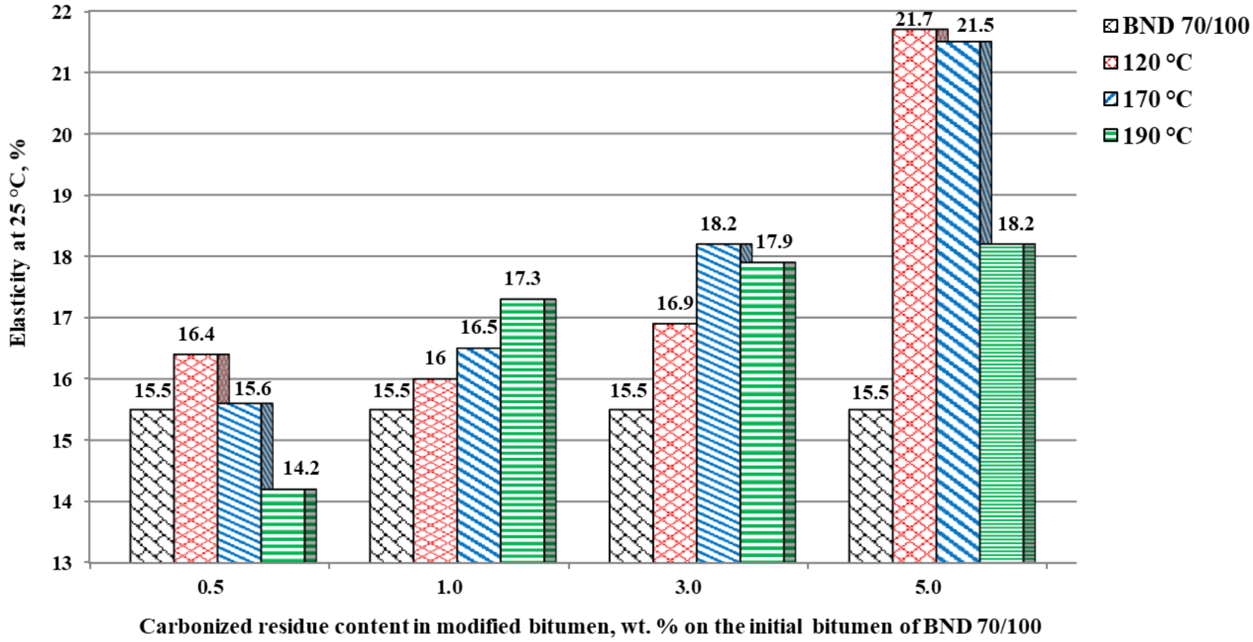

Fig. 8. Dependence of elasticity on the modification temperature and the content of CR in the modified bitumen, wt. % on the initial bitumen of the BND 70/100

The optimum ductility values (Fig. 4) are observed for samples modified with CR of 0.5-3.0 wt. % of raw materials at a temperature of 170 °C, as well as for samples modified with CR in the amount of 0.5-1.0 wt. % of raw materials added at a temperature of 190 °C. The ductility values of the samples obtained at 120 °C do not meet the requirements. It is worth noting that CR was added to 5.0 wt. % of raw materials at different temperatures does not significantly affect the properties of bitumen.

As a result of determining the softening temperature (Fig. 5) of the modified samples, it was found that optimal values were obtained following the requirements for all samples modified CRs at a temperature of 170 °C.[40,41] It was found that at a temperature of 120 °C, there is a gradual decrease in the values of the softening point, which, when added at 5.0 wt. % CR per raw material is at the limit of the recommended requirements (49 °C).[40,41] In comparison, at a process temperature of 190 °C, the optimal softening temperature values are observed only for samples modified with CR in the amount of 3.0 and 5.0 wt. %.

According to the results obtained (Fig. 6), high values of adhesion to glass are observed for all values of the studied temperatures when adding CR in the amounts of 0.5-5.0 wt. % of the raw material. It is worth noting that particularly high values of adhesion to glass are observed for the tested samples at a temperature of 190 °C, but at the same time, the addition of CR in the amount of 0.5-1.0 wt. % of the raw material at 120 °C and 0.5 wt. % of the raw material at 170 °C is less effective.

As a result of determining the adhesion to crushed stone, according to Fig. 7, it was found that only with the addition of CR in the amount of 3.0 and 5.0 wt. % by raw material and at a temperature of 190 °C, modified bitumen that meets the standard's requirements can be obtained.[41] Thus, CR can be considered as an effective adhesive additive to bitumen in the amount of 0.5-3.0 wt. % at 190 °C.

According to Fig. 8, the highest elasticity values were observed for the tested samples modified with CR in amounts of 5.0 wt. % of raw materials at temperatures of 120 and 170 °C.

As a result of the analysis, regularities were obtained based on the following assumptions. It was found that with an increase in temperature and the amount of modifier, the interaction of the studied bitumen with the CR is better. As can be seen, according to the above results, at a modification temperature of 120 °C, the obtained modified bitumen meets the requirements only in terms of such indicators as penetration and elasticity. In contrast, establishing the optimal value of the number of CR at this temperature is complicated. The properties of modified bitumen improve somewhat when the process temperature increases to 170 °C, for which, in addition to the specified penetration and elasticity, the softening point and ductility values are also at the required level. The properties of samples modified with CR were improved by 3.0 and 5.0 wt. % of raw materials at 190 °C can be observed more clearly. It is worth noting that at this ratio of components and process temperature, it is possible not only to qualitatively utilize the solid carbonized residue of the pyrolysis of WT, which has minimal use, but also to improve the adhesive properties of the binder. At the same time, to ensure all the necessary properties of the binder modified with CR in the amount of 3.0 and 5.0 wt. % by weight of raw materials at a temperature of 190 °C, it is worth considering the possibility of additional plasticizers.

Table 3. Changes in the properties of bitumen obtained as a result of their modification by CR (t = 120 °C, τ = 3 h) after heating (RTFOT method)[38]

Index		Initial bitumen BND 70/100	Bitumen with CR additives, wt. % CR per raw material			
			0.5	1.0	3.0	5.0
Change in properties after heating	softening point, °C	58.2	57.2	56.8	56.4	55.4
	softening point change, °C	8.6	7.4	7.2	7.0	6.4
	mass change, wt. %	0.11	0.057	0.100	0.157	0.168
	retained penetration, %	50.0	60.9	67.8	74.1	80.3
	penetration at 25 °C after RTFOT, dmm	38	39	40	43	45

Table 4. Changes in the properties of bitumen obtained as a result of their modification by CR (t = 170 °C, τ = 3 h) after heating (RTFOT method)[38]

Index		Initial bitumen BND 70/100	Bitumen with CR additives, wt. % CR per raw material			
			0.5	1.0	3.0	5.0
Change in properties after heating (RTFOT method)[36]	softening point, °C	58.2	58.8	58.8	58.1	57.1
	softening point change, °C	8.6	8.2	8.0	7.7	7.5
	mass change, wt. %	0.11	0.10	0.106	0.11	0.124
	retained penetration, %	50.0	60.9	74.1	76.4	78.8
	penetration at 25 °C after RTFOT, dmm	38	39	43	42	41

Table 5. Changes in the properties of bitumen obtained as a result of their modification by CR (t = 190 °C, τ = 3 h) after heating (RTFOT method)[38]

Index		Initial bitumen BND 70/100	Bitumen with CR additives, wt. % CR per raw material			
			0.5	1.0	3.0	5.0
Change in properties after heating (RTFOT method)[36]	softening point, °C	58.2	63.2	62.0	57.8	58.4
	softening point change, °C	8.6	7.0	6.6	4.4	4.8
	mass change, wt. %	0.11	0.072	0.086	0.028	0.057
	retained penetration, %	50.0	72.5	65.9	69.8	70.8
	penetration at 25 °C after RTFOT, dmm	38	29	31	37	34

Analyzing the resistance to aging of the initial and modified bitumen samples (change in their properties after heating, see Tables 3-5), it can be stated that the addition of CR in the amount of 5.0 wt. % positively affects the change in the characteristics of modified samples at any process temperature. At the same time, the best results are observed when adding 3.0 and 5.0 wt. % of the CR at a process temperature of 190 °C. In particular, this can be observed by a minor change in the softening point after heating, which is 4.4 and 4.8 °C when adding 3.0 and 5.0 wt. % CR, as well as an increase in residual penetration and lower mass loss after heating compared to the original bitumen.

That is, 3.0 and 5.0 wt. % CR at a temperature of 190 °C should be considered as an inhibitor of bitumen aging.

4. Conclusions

The effect of the carbonized residue obtained in the pyrolysis of WT as an adhesive and/or modifying additive on the performance properties of the oxidized petroleum bitumen of BND 70/100 grade was determined. The carbonized residue is a much cheaper and more environmentally friendly modifier than polymeric modifiers. Using waste conversion products in non-fuel applications will reduce the carbon footprint and promote the development of waste valorization. Accordingly, all this will create additional economic and environmental benefits when laying asphalt pavements.

As a result of the analysis of the obtained patterns, the most clear-cut improvement in the properties of bitumen samples modified with CR in amounts of 1.0-3.0 wt. % of raw materials at a temperature of 170 °C can be observed, which makes obtaining bitumen of BND 50/70 grade possible and has a certain environmental and economic efficiency compared to other industrial additives.

At the same time, CR was added in the amounts of 3.0 and 5.0 wt. % had a positive effect on the raw materials at a temperature of 190 °C, which improves the adhesive properties of bitumen to the values established by regulatory documents for BND-A 40/60 bitumen. At this ratio of components and process temperature, it is possible to utilize WT's solid carbonized pyrolysis residue qualitatively and rationally and obtain a binder with the necessary adhesive properties. Also, at the specified ratio of CR to bitumen, it is possible to obtain a binder that has the necessary resistance to aging, which is manifested in a lower change in the softening point after heating and higher residual penetration values than in the original samples. At the same time, it is essential to add plasticizers to obtain a binder whose characteristics fully meet the requirements of regulatory documents.

Modifying bitumen with carbonized residue makes obtaining two new commercial bitumens possible. By adding 3% by weight of carbonized residue at a temperature of 170°C, commercial bitumen grade BND 50/70 (following DSTU 4044:2019) can be obtained, and in an amount of 3-5% by weight at a temperature of 190 °C, commercial bitumen grade BND-A 40/60 (following SOU 45.2-00018112-067:2011) can be obtained. That is, CR can be utilized in an amount of 3-5% by mass as an additive to road bitumen, thereby reducing the cost of the resulting product and its susceptibility to aging and improving its adhesion to mineral material.

It should be noted that the significant difference in the results obtained at modification temperatures of 190°C (compared to mixing at lower temperatures) suggests a hypothesis regarding the chemical interaction of the CR with bitumen at 190°C. The study of the nature of this interaction and the use of plasticizers should be the subject of further research.

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ВПЛИВ ТЕМПЕРАТУРИ НА ПРОЦЕС МОДИФІКАЦІЇ ДОРОЖНЬОГО БІТУМУ КАРБОНІЗОВАНИМ ЗАЛИШКОМ ПІРОЛІЗУ ВІДПРАЦЬОВАНИХ ШИН

Анотація. *Проведено аналіз можливості утилізації твердого карбонізованого залишку (КЗ), що отримується у процесі піролізу вживаних автомобільних шин (ВАШ). В середньому внаслідок піролізу ВАШ одержується близько 36 % мас. карбонізованого залишку, який може слугувати адгезійною добавкою та/або інгібітором старіння в процесах модифікування нафтових бітумів. Проведено аналіз КЗ, одержаного на малотоннажній промисловій установці, та бітуму марки БНД 70/100 виробництва ПАТ «Укртатнафта». Встановлено вплив карбонізованого залишку на експлуатаційні властивості модифікованих бітумів за різного співвідношення сировина (бітум БНД 70/100) : КЗ та за різних температур змішування. Згідно одержаних результатів запропоновано оптимальні кількості КЗ та температури модифікування нафтових бітумів.*

Ключові слова: *відпрацьовані шини, піроліз, карбонізований залишок, модифікування бітумів, адгезійна добавка, чорний вуглець.*