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Composition and rheological characteristics of bitumen in short-term and long-term aging

Состав и реологические характеристики битума при кратковременном и длительном старений

B.B. Teltayev*,
Kazakhstan Highway Research Institute, Almaty, Republic of Kazakhstan
C.O. Rossi,
University of Calabria, Cozenza, Calabria, Italy
S.Zh. *Ashimova*
Kazakhstan Highway Research Institute, Almaty, Republic of Kazakhstan

Д-р техн. наук, президент Б.Б. Телтаев*,
Казахстанский дорожный научно-исследовательский институт, Алматы, Республика Казахстан
PhD, профессор Ч.О. Росси,
Университет Калабрии, г. Козенца, Калабрия, Италия
инженер С.Ж. Ашимова
Казахстанский дорожный научно-исследовательский институт, Алматы, Республика Казахстан

Key words: blown bitumen; short-term aging; long-term aging; dynamic shear rheometer; bending beam rheometer; complex shear modulus; phase angle; stiffness

Ключевые слова: окисленный битум; кратковременное старение; длительное старение; динамический сдвиговой реометр; реометр с изгибаемой балкой; комплексный сдвиговой модуль; фазовый угол; жесткость

Abstract. This paper investigates the impact of sequential short-term and long-term aging of blown bitumen of the grade BND 70/100 on its mechanical characteristics in the temperature interval from 76°C to -36 °C. Group chemical composition of the bitumen has been determined by the method of liquid adsorption chromatography by the chromatograph “Gradient M”. Short-term aging has been performed in the vertical rolling thin film oven (RTFOT) under the standard of AASHTO T 240-08, and the long-term aging - in the pressure aging vessel (PAV) under the standard of ASTM D 6521-08. Mechanical characteristics of the bitumen are complex shear modulus *G** and phase angle *δ* at the mean and high temperatures (from 4 °C to 76 °C) have been measured by dynamic shear rheometer (DSR) under the standard of AASHTO T 315-08. Bitumen stiffness S at low temperatures (from -24 °C to -36 °C) has been measured by bending beam rheometer (BBR) under the standard of AASHTO T 313-08. It has been determined that during short-term aging the content of oils in the bitumen has been decreased for 1.5 %, and the content of asphaltenes has been increased for 2 %. After the long-term aging, performed after the short-term aging, the content of oils in the bitumen has been decreased for 7 %, and the content of asphaltenes has been increased for 6.3 %. The content of resins in the bitumen remains practically constant at both types of aging. At the mean and high temperatures the short-term and long-term aging increase the complex shear modulus up to 2 and 7 200 times respectively and decrease the phase angle at average for 4-6° and 8-10° respectively. At low temperatures the short-term aging and long-term aging increase the bitumen stiffness in 1.5 and 2.5 times respectively.

Аннотация. В настоящей работе исследовано влияние последовательных кратковременного и длительного старений окисленного битума марки БНД 70/100 на его механические характеристики в температурном интервале от 76 °С до -36 °С. Групповой химический состав битума был определен методом жидкостно-адсорбционной хроматографии на хроматографе «Градиент М». Кратковременное старение было осуществлено в вертикальной тонкопленочной вращающейся печи (RTFOT) по стандарту AASHTO T 240-08, а длительное старение - в сосуде высокого давления и температуры (PAV) по стандарту ASTM D 6521-08. Механические характеристики битума – комплексный сдвиговой модуль G* и фазовый угол *δ* при средних и высоких температурах (от 4 °С до 76 °С) были измерены динамическим сдвиговым реометром (DSR) по стандарту AASHTO T 315-08. Жесткость битума S при низких температурах (от -24 °С до -36 °С) была измерена реометром с изгибаемой балкой (BBR) по стандарту AASHTO T 313-08. Установлено, что при кратковременном старении содержание масел в битуме уменьшается на 1,5 %, а содержание

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асфальтенов увеличивается на 2 %. После длительного старения, осуществленного после кратковременного старения, содержание масел в битуме уменьшается на 7 %, а содержание асфальтенов увеличивается на 6,3 %. При обоих видах старения содержание смол в битуме остается практически постоянным. При средних и высоких температурах кратковременное и длительное старения повышают комплексный сдвиговой модуль до 2 и 7 200 раз соответственно и уменьшают фазовый угол в среднем на 4-6° и 8-10° соответственно. При низких температурах кратковременное и длительное старения повышают жесткость битума в 1,5 и 2,5 раза соответственно.

1. Introduction

It is well-known that bitumens in the conditions of their storage, preparation of an asphalt concrete mix, its transportation, laying, and compaction and during operation of an asphalt concrete pavement are subject to aging [1–3]. Due to aging bitumens usually become more viscous and more brittle at low temperatures. Therefore, the quantitative evaluation of the impact of bitumen aging on their properties is important for road engineering.

At present many countries of the world use widely three methods of artificial aging for bitumens. The first two of them [4] imitate the aging of a bitumen during preparation of an asphalt concrete mix, its transportation, laying, compaction, and the third method models the aging of a bitumen during operation of an asphalt concrete pavement. The latter two methods have been developed quite recently and included into the known American technical system Superpave [5].

The review of the published works [6–13] has shown that at present the intensive investigation is performed for the impact of bitumen aging on the properties of the bitumens themselves [8–10] and asphalt concretes with their use [11–15]. Meanwhile, practically always bitumen aging has been performed under the methods, included into Superpave. Practically all investigations are experimental ones and the impact of short-term and long-term aging has been evaluated in them on the rheological properties [5, 8–12], structural changes [9], standard characteristics [11, 12] of bitumens and asphalt concretes [9–13].

This paper investigates and gives the quantitative evaluation of the impact of short-term and long-term aging of the blown bitumen of grade BND 70/100 on its group chemical composition and mechanical (rheological) characteristics within the temperature interval from 76 °C to -36 °C by dynamic shear rheomoter (DSR) and bending beam rheometer (BBR).

2. Materials and Methods

2.1. Bitumen

Bitumen of grade BND 70/100 has been selected for experimental research of its rheological characteristics at various temperatures in three conditions: non-aged, after short-term aging (RTFOT) and after long-term aging (RTFOT+PAV). Bitumen has been produced at Pavlodar petrochemical plant from crude oil of Western Siberia (Russia) by method of direct oxidation. Its characteristics satisfy the requirements of the standard of Kazakhstan ST RK 1373-2013 [16]. Grade of bitumen under Superpave: PG 64-40 [5]. The main standard characteristics of the bitumen in the initial condition are shown in the Table 1.

Table 1. Main standard characteristics of bitumen in initial condition

Indicator	Measurement unit	Requirements of ST RK 1373-2013	Values
Penetration depth of the needle, 25°C, l00 g, 5s	0.1 mm	70–100	75
Penetration Index	-	-1.0...+1.0	-0.87
Ductility at the temperature of:			
25°C		≥75	118
0°C		≥3.8	5.2
Softening point	°C	≥ 45	47.5
Fraas brittle point	°C	≤ -20	-28.5
Dynamic viscosity at 60°C	Pa·s	≥145	229
Kinematic viscosity at 135 °C	mm^2/s	≥250	428

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2.2. *Short-term aging*

Short-term aging of the bitumen in the vertical rolling thin film oven have been performed under the standard of AASHTO T 240-08, which models the bitumen aging during preparing of an asphalt concrete mix, its transportation, laying and compaction. The samples of the bitumen were in the oven at the temperature of 150 °C for 75 minutes.

2.3. *Long-term aging*

Long-term aging of the bitumen in the special pressure aging vessel has been performed under the standard of ASTM D 6521-08, which models the bitumen aging during operation of the asphalt concrete pavement. The samples of the bitumen, after the short-term aging, were in the vessel under the pressure of 2070 kPa and at the temperature of 100 °C for 20 hours.

2.4. *Dynamic shear rheometer*

The mechanical characteristics of the bitumen at the mean and high temperatures (from +4 °C to +76 °C) have been measured by dynamic shear rheometer (Figure 1) under the standard of AASHTO T 315-08. The samples of the bitumen in the shape of round plate with diameter of 25 mm and thickness of 1 mm have been tested under the impact of sinusoidal varied strain, which has the amplitude of 12 % and frequency of 10 rad/s. Before testing the samples have been kept at the specified temperature not less than for 10 minutes. Shear deformation γ, shear stress τ and the phase angle δ have been measured as the test results.

The value of the complex shear modulus G* of the bitumen has been calculated under the formula [17–19]:

$$G^* = \frac{\tau_{\max} - \tau_{\min}}{\gamma_{\max} - \gamma_{\min}}, \tag{1}$$

where $\tau_{\max} - \tau_{\min}$ are maximum and minimum shear stresses respectively;

$\gamma_{\max} - \gamma_{\min}$ are maximum and minimum shear strains respectively.

The shear stresses τ_{max}, τ_{min} and shear strains γ_{max}, γ_{min} occur on the same plane, and their differences in the values are caused by sinusoidal load.

Figure 1. Dynamic shear rheometer (DSR)

2.5. *Bending beam rheometer*

The mechanical characteristics of the bitumen at low temperatures (-24, -30 and -36°C) have been measured by bending beam rheometer (Figure 2) under the standard of AASHTO T 313-08. The samples of the bitumen for tests had the shape of a beam with dimensions of 6.25x12.5x125 mm. Before testing the samples have been kept at the tested temperature for 60 minutes. In the beginning of the test the load, equal to 980 mN, has been applied automatically for 1 second and it has been kept as the constant one for the following 240 seconds. The maximum deflection of the middle of the beam has been measured automatically.

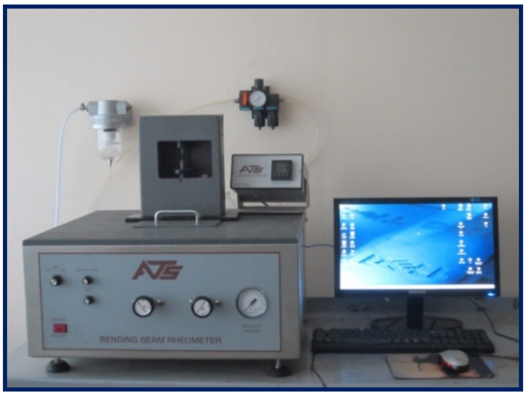

Figure 2. Bending beam rheometer (BBR)

Maximum stress on the bottom surface of the bituminous beam in its middle has been calculated under the formula:

$$\sigma = \frac{3 \cdot P \cdot \ell}{2 \cdot b \cdot h^2} \tag{2}$$

where P is a load, mN;

h, b, ℓ are height, width and length of the beam respectively, mm.

Maximum strain of the bottom surface of the bituminous beam in its middle at the time moment *t* has been calculated under the formula:

$$\varepsilon(t) = \frac{6 \cdot h}{\ell^2} f(t) \tag{3}$$

where $f(t)$ is the maximum deflection of the middle of the bituminous beam, mm.

The stiffness of the bituminous beam at the time moment *t* has been calculated under the formula

$$S(t) = \frac{P \cdot \ell^3}{4 \cdot b \cdot h^3 \cdot f(t)}. \tag{4}$$

2.6. Group chemical composition of bitumen

Group chemical composition of the bitumen has been determined by liquid adsorption chromatography method on the chromatograph “Gradient M” (Figure 3), manufactured by the Institute of petrochemical processing of the Republic of Bashkortostan (Russia). Chromatograph consists of two parts: analytical and detecting. The analytical part is the glass capillary column with the length of 300±5 mm and diameter of 1.2–1.4 mm, filled with the modified silica gel. Separating of the sample into maltenes (oils and resins) and asphaltenes with the use of complicated mixes of the solvents taken in different proportions (isooctane, dichloroethane, diisomayl ether, ethyl acetat, ethyl hydroxide and chlorbenzene). Detection for the groups of chemical compounds has been performed according to their heat conductivity at the temperature of 680 °C.

Figure 3. Liquid adsorption chromatograph

3. Results and Discussion

3.1. Group chemical composition of bitumen

Group chemical compositions of the investigated bitumen in three conditions has been shown in the Figure 4, where the impact of aging on the chemical composition is clearly seen: one can consider that the content of resins is practically constant at double aging (short-term and long-term); short-term and long-term aging decrease the content of oils for 1.5 % and 5.5 % respectively and increase the content of asphaltenes for 2.0 % and 4.3 % respectively. In general, the sequential double aging decreased the content of oils for 7.0 %, and the content of asphaltenes has been increased for 6.3 %.

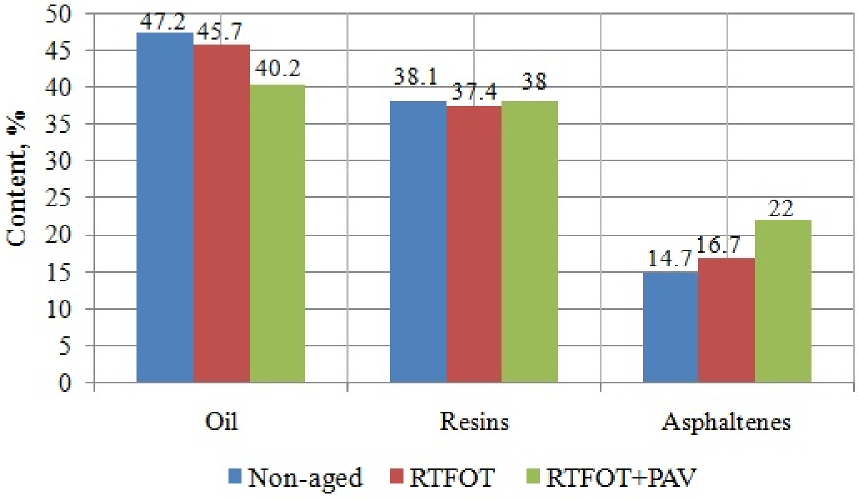

Figure 4. Group chemical composition of bitumen

3.2. Mechanical characteristics

3.2.1. At the mean and high temperatures

The graphs for dependence of the complex shear modulus G* and phase angle *δ* of the bitumen in three conditions on the temperature have been represented in Figures 5 and 6. It is seen that the aging changes the mechanical characteristics of the bitumen. Thus, after the short-term aging G* has been increased at the temperatures of 4 °C and 76 °C in 1.3 and 2.1 times respectively. And the impact of long-term aging on G* was great: the increase of G* was 5 300 and 7 200 times at the temperatures of 40 °C and 76 °C respectively.

In semi-logarithmic coordinates the temperature dependences of G* of the bitumen in the initial condition and after the short-term aging are nearly the straight lines. These straight lines are nearly parallel, i.e. the thermal sensitivity of the bitumen G* is practically similar in the specified conditions. Dependence of G* on the temperature after the long-term aging is of some other nature: within the range of temperatures from 26 °C to 76 °C it is a straight line, but with the less thermal sensibility; from 4 °C to 13 °C it is also described by the equation of straight line, but with considerably less thermal sensitivity; and temperature range from 13 °C to 26 °C is a transition one, within which the non-linear decrease of thermal sensitivity of the bitumen G* occurs.

Thus, the short-term aging of the bitumen, during which the content of oils has been decreased for 1.5 %, and the content of asphaltenes has been increased for 2 %, determined the increase of shear modulus G* at the mean and high temperatures in 1.3 and 2.1 times; long-term aging, resulting in the oil content decrease for 7 %, and the asphaltenes content increase for 6.3 %, has lead to the increase of G* at high temperatures up to 7 200 times.

The graphs in Figure 6 show clearly the impact of temperature and aging on phase angle *δ*. The phase angle is an important mechanical characteristic of the viscoelastic materials [20–22]. It shows the ratio of the elastic and non-elastic deformations. Its value varies from 0° to 90°. For the pure elastic material it is equal to 0° and for pure plastic material it is equal to 90°.

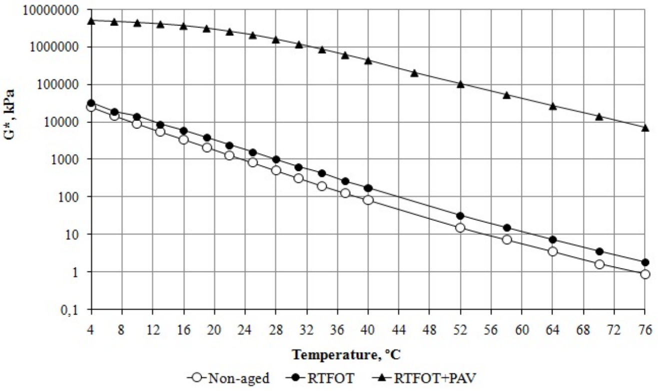

Figure 5. Dependence of complex shear modulus of bitumen on temperature

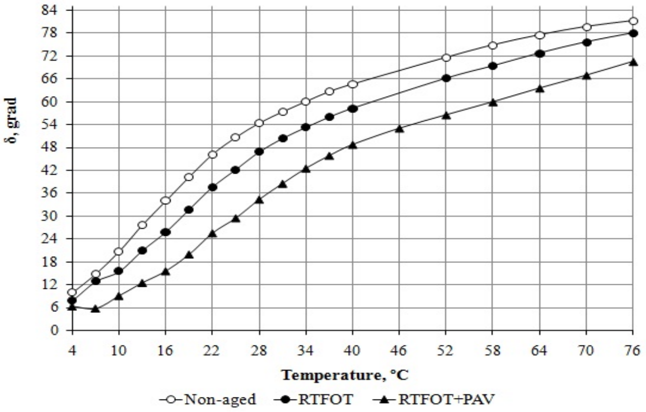

Figure 6. Dependence of phase angle of bitumen on temperature

It can be said that the short-term and long-term aging in the whole considered interval of temperatures (from 4 °C to 76 °C) decrease the phase angle of the bitumen at average for 4-6° and 8-10° respectively.

As could be expected, δ increases with the temperature increase. Temperature dependence of δ can be considered as bilinear one. There is a transition section in each condition of the bitumen (non-aged, RTFOT, RTFOT+PAV) between the first (at mean temperatures) and the second (at high temperatures) linear sections. The first linear section is characterized by higher indicator of thermal sensitivity of δ, and the second one has some lower thermal sensitivity. Smooth non-linear decrease for the indicator of thermal sensitivity of the phase angle occurs within the transition section. As it is seen from the Table 2, with the increase of aging level of the bitumen the position and characteristic (mean) temperature of the transition section on the temperature dependence of the phase angle shifts towards higher temperatures, and the width of the section has been decreased.

Table 2. Characteristics of transition section in temperature dependence of phase angle of bitumen

Condition of bitumen	Characteristics of transition section			
	initial temperature, °C	final temperature, °C	width of section, °C	conventional temperature of transition, °C
Non-aged	24	52	28	30
RTFOT	28	50	22	35
RTFOT+PAV	36	40	4	38

The works [9, 11–14] according to the results of experimental investigations determine that at high temperatures the short-term, as well as the long-term aging, increase complex shear modulus G* and decrease phase angle δ of the bitumens. But the authors do not mention numerical value for variation of G* and δ of the bitumens, provided by the impact of aging. It is obvious that it is connected with the fact that the issue of impact of aging on mechanical and other properties of bitumens is on the stage of studying and accumulating of experimental data, and researchers refrain themselves from general conclusions.

3.2.2. At low temperatures

The graphs for the stiffness of the bitumen at various low temperatures and conditions according to the aging are represented in Figures 7–9, where it can be clearly seen that the short-term and long-term aging impact essentially on deformability of the bitumen at low temperatures. At all low temperatures and conditions the bitumen stiffness decreases essentially under the exponential law within the time interval from 8 s to 240 s. At small load durations (8 s) the short-term aging increases the bitumen stiffness at the temperatures of -24 °C, -30 °C and -36 °C in 2.2; 1.3 and 1.5 times respectively, and the long-term aging in 2.8; 2.5 and 2.3 times respectively. Averaging the data, mentioned above, one can adopt that at average the short-term and long-term aging at small load durations increase the bitumen stiffness in 1.7 and 2.5 times respectively.

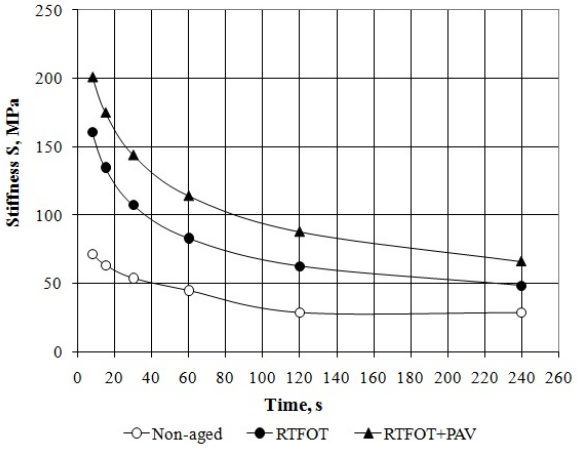

Figure 7. Time dependence of bitumen stiffness at the temperature of – 24 °C

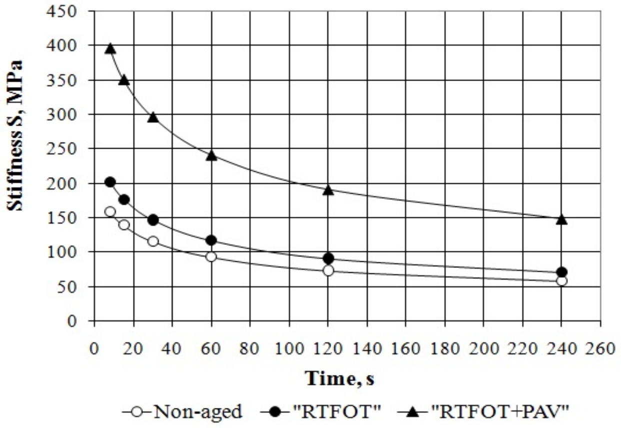

Figure 8. Time dependence of bitumen stiffness at the temperature of – 30 °C

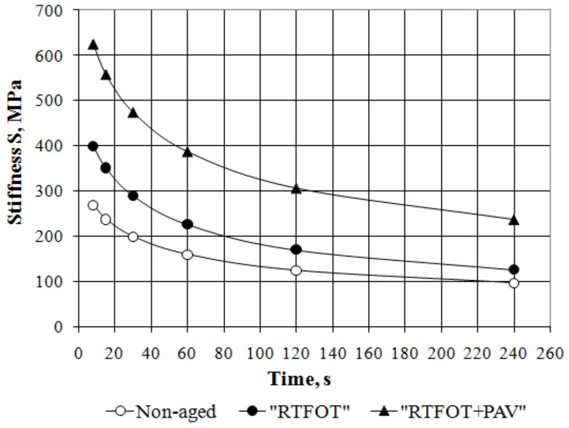

Figure 9. Time dependence of bitumen stiffness at the temperature of - 36°C

As it is known, one of the modern methodological systems in the world, aimed at more differentiated recording of the climatic conditions when defining the operational grade for the road bitumens, is Superpave [2]. The stiffness has been adopted at load duration of 60 seconds in Superpave as one of the bitumen indicators, characterizing its stability at low temperatures. For the tested bitumen the values of specific stiffness (at 60 s) at various low temperatures and aging conditions have been shown in Figure 10. The Figure shows clearly the cooperative effect of short-term and long-term aging and low temperature on specific stiffness of the bitumen: the specific stiffness has been increased with the aging level increase. For example, at the temperatures of -24 °C, -30 °C and -36 °C the short-term aging increases the specific stiffness of the bitumen in 1.8; 1.3 and 1.4 times respectively, and the long-term aging - in 2.5; 2.6 and 2.4 times respectively. At average the short-term aging and long-term aging increase the specific stiffness of the bitumen in 1.5 and 2.5 times respectively.

The literature review has shown that the investigations of the impact of aging on mechanical and other properties of bitumens at low temperatures are strictly limited. Only the work [10] investigates the impact of short-term and long-term aging on stiffness of bitumen PMB 45/80-65, modified by liquid surface substance on the basis of amines within the temperature range from -10 °C to -28 °C. It has been determined that the long-term aging does not almost impact on bitumen stiffness, and the short-term aging increases it slightly: critical temperature, at which the bitumen stiffness is equal to 300 MPa, is increased only for 2.5 °C.

Our work investigates pure (unmodified) bitumen. It is obvious, that modification reduces the process of aging for bitumens, which can be a subject for future investigations.

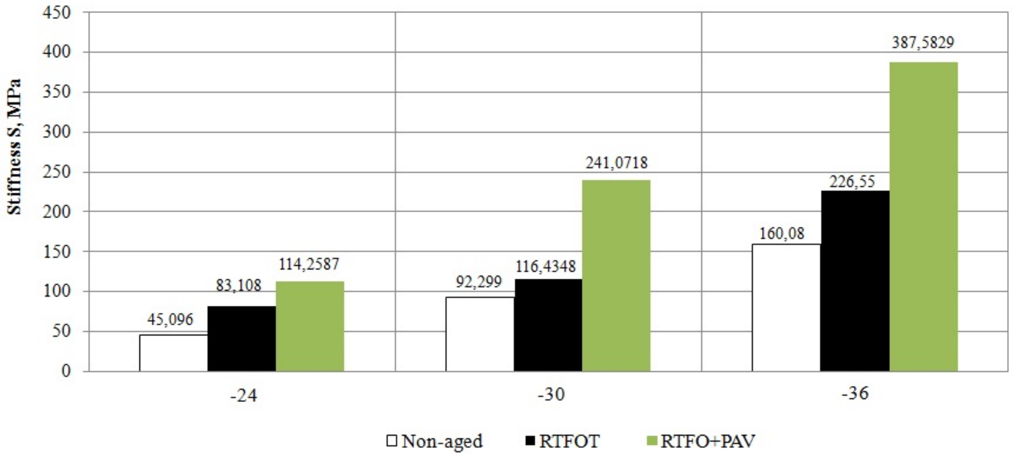

Figure 10. Bitumen stiffness at various low temperatures and aging conditions

4. Conclusion

1. At short-term aging of blown bitumen of grade BND 70/100 the content of oils in it has been decreased for 1.5 %, and the content of asphaltenes has been increased for 2 %. The long-term aging of the bitumen, performed after its short-term aging, compared with its initial condition, decreases the content of oils for 7 %, and the content of asphaltenes increases for 6.3 %. After two types of aging the content of resins in the bitumen remains practically constant.
2. The short-term aging determined the complex shear modulus G* increase at the mean and high temperatures (from 4 °C to 76 °C) in 1.3 and 2.1 times respectively, and the long-term aging increased G* at high temperatures in 7 200 times.
3. The short-term and long-term aging decrease the phase angle δ of the bitumen at the mean and high temperatures at average for 4-6° and 8-10° respectively.
4. The short-term and long-term aging increase the bitumen stiffness S at low temperatures (from -24 °C to -36 °C) in 1.5 and 2.5 times respectively.

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Bagdat Teltayev,*
+7(701)760-67-01; bagdatbt@yahoo.com

Cesare Rossi,
+39(327)833-28-61; cesare.oliviero@unical.it

Saltanat Ashimova
+7(778)955-75-55; salta_32@mail.ru

Багдат Бурханбайулы Телтаев,*
+7(701)760-67-01;
эл. почта: bagdatbt@yahoo.com

Чезаре Оливиеро Росси,
+39(327)833-28-61;
эл. почта: cesare.oliviero@unical.it

Салтанат Жандарбековна Ашимова
+7(778)955-75-55; эл. почта: salta_32@mail.ru

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