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# Rheological Properties of Oxidized Bitumen with Polymer Additive

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**Abstract:** In the present study rheological properties of bitumen of grade BND-90/130 obtained from crude oil of Western Siberia (Russia) by the direct oxidation method and polymer binder, obtained by adding in pure bitumen the polymer Elvaloy 4170 are investigated. Binders in initial state and after short-term aging at high and average temperatures were tested on the Dynamic Shear Rheometer (DSR) and at low temperatures after double aging-on the Bending Beam Rheometer (BBR). The obtained results showed that in all cases of testing operational properties of polymer-bitumen binder is significantly better than pure bitumen.

Key words: Bitumen, polymer, dynamic shear rheometer, bending beam rheometer

#### INTRODUCTION

Bitumen is one of the main materials for road hot mix asphalt preparation which is widely used now around the world. But, as we know, hot mix asphalt has a number of disadvantages. So, on hot mix asphalt pavement there can be residual deformations at high temperatures. At low temperatures occur transverse temperature cracks. Under multiple actions of truck wheels loadings appear fatigue cracks. A number of the above mentioned defects can lead to destruction of pavement structure.

Currently, it is well known that the mechanical behavior of hot mix asphalt due to the rheological properties of bitumen. Therefore, to improve operational behavior of hot mix asphalt improves properties of bitumen. One of the effective ways on increasing of bitumen stability to mechanical and climatic influences is modification by a polymer. Bitumen modification by adding polymer in it, is practiced enough long in Europe and the USA. But in road construction of Europe and the USA residual bitumen are commonly used.

Mechanical properties of bitumen strongly depend on temperature. The climate of Kazakhstan is sharply continental. A number of scientific researches are devoted to the accounting of climatic features of Kazakhstan (Teltayev and Kaganovich, 2011, 2012a; Teltayev and Aitbayev, 2014a, b). This study is a continuation of experimental studies devoted to the evaluation of thermorheological properties of bitumen binders (Teltayev and Kaganovich, 2012b).

In the present study, mechanical properties of bitumen of grade BND-90/130 obtained from crude oil of Western Siberia (Russia) by the direct oxidation method and polymeric binder, obtained by adding in pure bitumen the polymer Elvaloy 4170 are investigated. Mechanical

behavior of binders at high and average temperatures were tested on the DSR and at low temperatures-BBR.

#### MATERIALS AND MEHODS

**Materials:** In the present study, properties of pure bitumen of grade BND-90/130 and the same bitumen modified by the polymer Elvaloy 4170 have been investigated.

Bitumen of grade BND-90/130 made in pavlodar petrochemical plant from crude oil of Western Siberia (Russia) by the direct oxidation method. Its characteristics meet requirements of the Kazakhstan standard (ST RK 1373-2005, 2005). Basic standard indicators of the bitumen are given in Table 1.

Polymer-bitumen binder, researched in this study, was prepared in laboratory by adding in pure bitumen of grade BND-90/130 the polymer Elvaloy 4170 in amount of 1.4% from the mass of bitumen.

For bitumen modification a special mixer was used. Sample of bitumen warmed up to the temperature of 180°C, stirred with a mixer (at rate of 200 rpm) to create a small vortex. Bitumen sample has been sustained

Table 1: Basic standard indicators of pure bitumen of grade BND-90/130

		Requirements	
	Measuring	of ST RK	
Indicator	unit	1373-2005 (2005)	Values
Penetration, 25°C, 100 g, 5 sec	0.1 mm	91-130	98.00
Penetration index PI	-	-1.0+1.0	-0.96
Ductility:	cm		
-25°C		≥65	139.00
-0°C		≥4.0	5.50
Softening temperature	$^{\circ}\mathrm{C}$	≥43	45.30
Fraas temperature	$^{\circ}\mathrm{C}$	≥-20	-24.60
Dynamic viscosity, 60°C	Pa•s	≥75	174.20
Kinematic viscosity	$\mathrm{mm^2~sec^{-1}}$	≥180	409.00



Fig. 1: Dynamic shear rheometer

Table 2: Basic standard indicators of polymer-bitumen binder

Indicator	Measuring unit	Requirements of (ST RK 1025-2004, 2004)	Value
maicator	um	(ST KK 1023-2004, 2004)	Value
Penetration, 25°C,	0.1 mm	≥60	80.0
100 g, 5 sec			
Ductility: - 25°C	sm	≥25	49.0
Softening temperature	$^{\circ}\mathrm{C}$	≥60	67.0
Fraas temperature	$^{\circ}\mathrm{C}$	≥-22	-25.1

at mixture temperature within 10 min, then slowly (speed about 10 g min<sup>-1</sup>) added in bitumen preselected amount of polymer. Continuous mixing process of polymer-bitumen binder lasted for 2 h, the next 12 h it has been conditioned at constant temperature of 180°C for 10 min. The obtained polymer-bitumen binder meets requirements of the Kazakhstan standard (ST RK 1025-2004, 2004). Basic standard indicators of the polymer-bitumen binder are shown in Table 2.

### Methods

**High temperature behavior:** High-temperature behavior of the pure bitumen and the Polymer Modified Bitumen (PMB) was investigated by testing them on DSR (Fig. 1) according to the standard (AASHTO T 315-08, 2008). DSR was made by Bohlin Instruments Company, USA. Binders were tested in initial state and after aging in the Rolling Thin Film Oven (RTFO) (short aging) according to the standard (AASHTO T 240-08, 2008) in temperature interval from +46 to +88°C. In tests on binder sample in circular plate form with 1 mm thickness and 25 mm diameter the shear stress has been applied which varied in time on the sinusoidal law. Deformation frequency was  $\omega = 10 \text{ rad sec}^{-1}$ . To achieve thermal equilibrium the binder sample previously maintained on testing temperature at least 10 min. Shear deformation and phase angle were measured.

The value of the complex shear modulus of binder was calculated by equation:



Fig. 2: Bending beam rheometer

$$G \; \textcolor{red}{*} = \frac{\tau_{_{max}} - \tau_{_{min}}}{\gamma_{_{max}} - \gamma_{_{min}}}$$

where,  $\tau_{\text{max}}$ ,  $\tau_{\text{min}}$  is the maximum and minimum shear stress, respectively,  $\gamma_{\text{max}}$ ,  $\gamma_{\text{min}}$  is the maximum and minimum shear deformation, respectively.

**Medium temperature behavior:** Medium temperature behavior of the binders was investigated by testing on the DSR according to the standard (AASHTO T 315-08, 2008) after their double aging: firstly in RTFO according to the standard (AASHTO T 240-08, 2008), then in accelerated aging vessel according to the standard (ASTMD6521-08, 2008) in temperature interval from +4°C to +40°C. Deformation frequency was  $\omega = 10$  rad sec<sup>-1</sup>. Binders test samples had a circular plate form with 2 mm thickness and 8 mm diameter.

Low temperature behavior: Low temperature behavior of binders was investigated on the BBR (Fig. 2) according to the standard (AASHTO T 313-08, 2008). BBR was made by Applied Test Systems Inc., USA. Binders were tested after their double aging according to the standards (AASHTO T 240-08, 2008; ASTM D6521-08, 2008) at temperatures of -18, -24, -30 and -35°C. Binders samples for testing were in form of a beam with the size of 6,25×12,5×125 mm. Before testing the samples were kept at test temperature for 60 min. At the beginning of the test load is applied automatically by the value of 980 mN within 1 sec and it remains constant during the next 240 sec. Then it is automatically unloaded and the stiffness and relaxation rate are calculated.

Binder stiffness is determined by equation:

$$S(t) = \frac{PL^3}{4bh^3\delta(t)}$$

where, S(t) is binder creep stiffness at the time point t, P is constant load, L is distance between supports of beams, b, h is width and thickness of beam, respectively,  $\delta(t)$  is deflection of the middle of beam.

The relaxation rate of binder is calculated by equation:

$$m(t) = \frac{d \lg S(t)}{d \lg t}$$

where, m(t) is the relaxation rate of binder at the time point t, lg S(t) is decimal logarithm of bitumen stiffness at the time point t, lg t is decimal logarithm of time t.

#### RESULTS AND DISCUSSION

# High temperature behavior

Complex shear modulus: In Fig. 3 and 4 the graphs of dependence of the complex shear modulus G\* of binders in initial state and after short aging (RTFO) from temperature are shown. We can see that the complex shear modulus value in considered temperature interval decreases with temperature increase according to the

exponential law. In initial state of pure bitumen the maximum value of G\* at temperature of 46°C is 13,69 kPa and the minimum value at temperature of 88°C is 0,093 kPa, i.e., G\* varies in 147.2 times in considered temperature interval, or in 3.5 times on each 1°C. In initial state of polymer-bitumen binder the maximum value of G\* at temperature of 46°C is 21,94 kPa and the minimum value at temperature of 88°C is 0,487 kPa, i.e., G\* varies in 45,1 times in considered temperature interval or in 1.1 times on each 1°C. Thus, we can say that at high temperature polymer-bitumen binder with Elvaloy 4170 in initial state has significantly high complex shear modulus and significantly lower (in 3.2 times) temperature sensitivity in comparison with pure bitumen.

After short term aging (PAV) the maximum value of G\* of pure bitumen at temperature of 46°C is 24,69 kPa and the minimum value at temperature of 88°C is 0,152 kPa, i.e., G\* varies in 162,4 times or in 3.7 times on each 1°C. The maximum value of G\* of polymer-bitumen binder at temperature of 46°C is 27.6 kPa and the minimum value at temperature of 88°C is 0,739 kPa, i.e., G\* varies in 37,3 times or in 0.9 times on each 1°C. In other words, at high temperatures after short aging polymer-bitumen

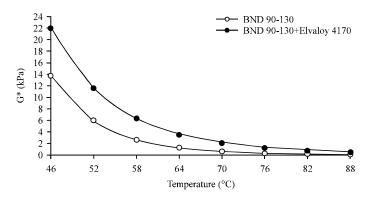


Fig. 3: Dependence of the complex shear modulus of binders in initial state from temperature

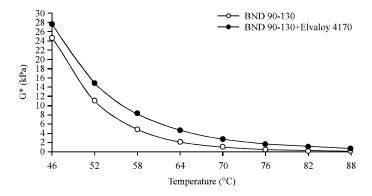


Fig. 4: Dependence of the complex shear modulus of binders after short aging (RTFO) from temperature

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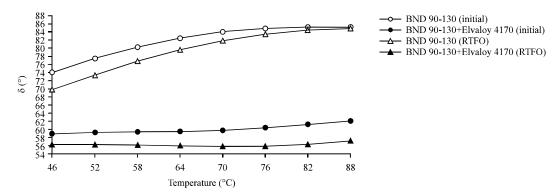


Fig. 5: Dependence of the phase angle of binders in initial state and after short term aging (RTFO) from temperature

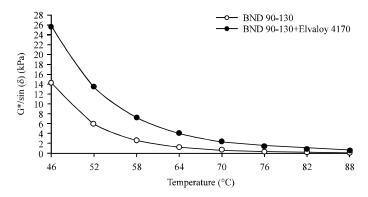


Fig. 6: Dependence of the rutting indicator of binders in initial state from temperature

binder with Elvaloy 4170 has higher complex shear modulus and significantly lower (in 4.1 times) temperature sensitivity in comparison with pure bitumen.

**Phase angle:** Another important characteristic of the mechanical behavior of viscoelastic materials is the phase angle. It shows ratio of elastic and inelastic deformation. Its value varies from 0-90°. For purely elastic bodies it is equal to 0° and for purely plastic bodies is 90°.

In Fig. 5 the graphs of dependence of the phase angle  $\delta$  of binders in initial state and after short term aging from temperature are shown. As we can see, pure bitumen in considered temperature interval as in initial state and after short term aging has significantly high phase angle than polymer-bitumen binder with Elvaloy 4170. Namely, at pure bitumen the phase angle raises from 74-85° in initial state and from 70-85° after short term aging. At polymer-bitumen binder the phase angle varies from 59-62° in initial state and from 56-57° after short term aging. Avoiding a big error, we can say that short term aging has a little influence in considered temperature interval on a phase angle as initial bitumen and polymer-bitumen binder. At temperature of 88°C pure bitumen shows the mechanical behavior close to ideally

viscous liquid. The phase angle of polymer-bitumen binder in initial state varies only on 3° and after short term aging only on 1°. Analysis of the phase angle showed that in considered temperature interval as in initial state and after short term aging the mechanical behavior of polymer-bitumen binder is significantly elastic than pure bitumen. In other words, at high temperatures resistance to rutting of polymer-bitumen binder is significantly higher than at pure bitumen.

**Indicator of rutting:** According to the Superpave system a bitumen binder ability to resist rutting is estimated by the rutting indicator  $G^*/\sin(\delta)$  which is calculated according to determination results of the complex shear modulus and phase angle with frequency  $\omega = 10 \text{ rad sec}^{-1}$  (Asphalt Institute, 2003).

In Fig. 6 and 7 the graphs of dependence of the rutting indicators of binding in initial state and after short aging from temperature are shown. As expected, these figures clearly show the advantage of polymer-bitumen binder which as in initial state and after short term aging values of rutting indicator significantly higher than at pure bitumen. Thus, at pure bitumen in initial state the maximum value of rutting coefficient at temperature of

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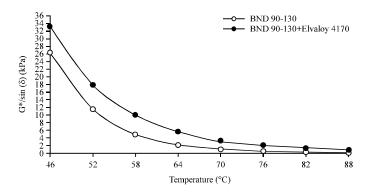


Fig. 7: Dependence of the rutting indicator of binders after short term aging (RTFO) from temperature

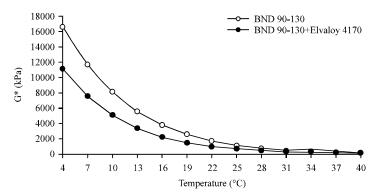


Fig. 8: Dependence of the complex shear modulus of binders after double-aging (RTFO+PAV) from temperature

46°C is 14.23 kPa and at polymer modified binder is 25.6 kPa. At temperature of 88°C, they are, respectively equal to 0,093 kPa and 0,551 kPa. Comparison of results shows that ability to resist rutting of polymer-bitumen binder at temperature of 46°C and 88°C, respectively in 1,8 and 5,9 times higher, than at pure bitumen.

After short term aging at pure bitumen the maximum value of  $G^*/\sin(\delta)$  at temperature of 46°C is 26,3 kPa and at polymer-bitumen binder is 33,13 kPa. At temperature of 88°C, they are 0,153 and 0.878 kPa. After short aging an ability to resist rutting at polymer-bitumen binder at temperatures of 46 and 88°C, respectively in 1,3 and 5,7 times higher, than at pure bitumen.

In the Superpave system (AASHTO M320-03, 2003; Asphalt Institute, 2003) it is accepted that hot mix asphalt pavement had a low propensity to rutting, the rutting indicator  $G^*/\sin(\delta)$  of binder at calculated summer temperature should be not less 1 kPa in initial state and at least 2.2 kPa after short term aging.

It is established that according to the rutting indicator a pure bitumen meets the Superpave system requirements in initial state and after short term aging up to the temperature of 65 and 64°C, respectively and polymer-bitumen binder up to the temperature of 80 and 76°C, respectively.

## Medium temperature behavior

Complex shear modulus: In Fig. 8 the graphs of dependence of the complex shear modulus G\* of binders after double aging from temperature are shown. We can easily notice that in considered temperature interval the complex shear modulus of binders varies significantly. With temperature increase it decreases exponentially.

At pure bitumen the maximum value of G\* at temperature of 4°C is equal to 16583,0 kPa and the minimum value at temperature of 40°C is 139,78 kPa, i.e., G\* varies in 118,6 times in considered interval of temperatures or in 3,3 times on each 1°C.

At polymer-bitumen binder G\* at temperature of 4°C has the maximum value equal to 11126,0 kPa and at temperature of 40°C has the minimum value equal to 107,21 kPa, i.e., G\* in considered interval of temperature varies in 103,8 times or in 2,9 times on each 1°C.

Modern road experts believe that bitumen binders with less stiffness at average operational temperatures are more resistant to fatigue cracking (Yoder and Witczak, 1975; Papagiannakis and Masad, 2008). At temperatures of 4 and 40°C G\* of polymer-bitumen binder on 33% and 23%, respectively less than G\* of pure bitumen which indicates that it is more resistant to fatigue cracking.

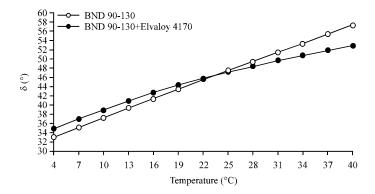


Fig. 9: Dependence of the phase angle of binders after double aging (RTFO+PAV) from temperature

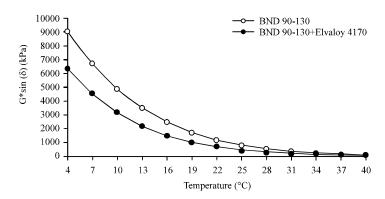


Fig. 10: Dependence of the fatigue cracking indicator of binders after double aging (RTFO+PAV) from temperature

**Phase angle:** Graphs of dependence of the phase angle  $\delta$  of binders after double aging from temperature are presented in Fig. 9. With some minor exceptions we can say that in considered temperature interval the phase angle  $\delta$  of pure bitumen and polymer-bitumen binder is a little different. Varies from 33-35° at temperature of 4°C up to 53-57° at temperature of 40°C. It shows that at temperature variation from 4-40°C an ability to accumulate plastic deformation increases on average in 1,6 times.

**Indicator of fatigue cracking:** According to the Superpave system a bitumen binders ability to resist fatigue cracking is estimated by  $G^{*\bullet}sin(\delta)$  which is calculated according to determination results of the complex shear modulus and phase angle with frequency  $\omega = 10 \text{ rad sec}^{-1}$  (Asphalt Institute, 2003).

Graphs of dependence of the fatigue cracking indicator of binders after double aging from temperature are shown in Fig. 10. We can see that the indicator  $G^{*\bullet}\sin(\delta)$  decreases on exponential dependence with temperature increase. Moreover, in all considered

temperature interval the indicator value of fatigue cracking of polymer-bitumen binder on average in 1.4 times less than of pure bitumen which shows higher fatigue crack resistance of polymer-bitumen binder.

In the Superpave system (AASHTO M320-03, 2003; Asphalt Institute, 2003) is accepted that a hot mix asphalt pavement had sufficient resistance to fatigue cracking, the indicator G\*•sin(δ) of binder at calculated temperature shall be no more than 5000 kPa after double aging. It is established that under the fatigue cracking indicator a pure bitumen and polymer-bitumen binder meet Superpave system requirements up to the temperature of 9,8 and 6,3°C, respectively.

# Low temperature behavior

Creep stiffness: Graphs of dependence of binding stiffness after double aging at different low temperatures are shown in Fig. 11. As we can see, at all considered temperatures stiffness of polymer-bitumen binder is significantly lower than pure bitumen that shows higher resistance to low temperature cracking of polymer-bitumen binder.

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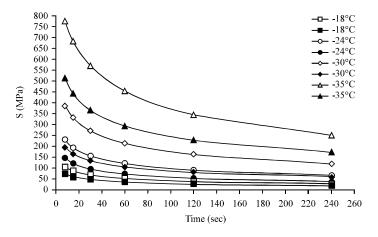


Fig. 11: Binders stiffness after double aging (RTFO+PAV)

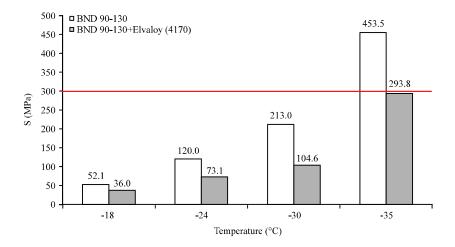


Fig. 12: Dependence of stiffness of binders after double aging (RTFO+PAV) at 60 sec from temperature

On the Superpave system stability of bitumen binders is estimated by two indicators, first is stiffness S on duration of loading 60 sec (AASHTO M320-03, 2003; Asphalt Institute, 2003). Figure 12 shows the values of binders stiffness on duration of loading 60 sec at different temperatures where clearly shown the advantage of polymer-bitumen binder. Advantage of polymer-bitumen binder increases with temperature decrease.

It is considered that binder to had sufficient resistance to a low temperature cracking, its stiffness on duration of loading 60 sec at calculated winter temperature should not exceed 300 MPa. It is established that to this requirement the pure bitumen and the polymer-bitumen binder satisfy up to the low temperatures of -32 and -35°C, respectively. Taking into account for the principle of time-temperature superposition, used in the Superpave system (AASHTO M320-03, 2003; Asphalt Institute, 2003) the pure bitumen and the polymer-bitumen

binder can be used in regions with minimal winter temperature of -42 and -45°C, respectively.

Relaxation rate: The second indicator that the Superpave system evaluate is a bitumen binders sustainability to low temperature cracking, is a relaxation rate (AASHTO M320-03, 2003; Asphalt Institute, 2003). It is accepted that binders to had sufficient resistance to a low temperature cracking, its relaxation rate on duration of loading 60 sec at calculated winter temperature should not be less than 0.3. The values of relaxation rate of the binders on duration of loading 60 sec at different temperatures are shown in Fig. 13. It is established that at all temperatures relaxation rate values of binders are little different. Both binders relaxation rate with temperature decreasing is gradually reduced, but for all considered temperatures it is more than the acceptable value.

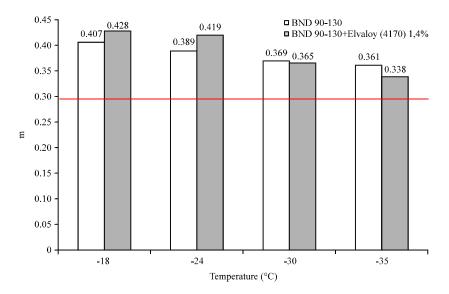


Fig. 13: Dependence of the relaxation rate of binders after double aging (RTFO+PAV) at 60 s from temperature

#### CONCLUSION

In the present study a pure bitumen properties of grade BND-90/130 and the same bitumen modified by the polymer Elvaloy 4170 in amount of 1,4% from the mass of bitumen are investigated. The behavior of bitumen binders in temperature interval from 46-88°C in initial state and after short term aging and in temperature interval from 4-40°C after double aging has been studied on DSR. Low temperature behavior of binders after double aging at temperatures of -18, -24, -30 and -35°C has been studied on BBR.

It is established that in initial state and after short term aging at high temperature polymer-bitumen binder has higher complex shear modulus and lower phase angle than pure bitumen. Temperature sensitivity of the complex shear modulus and phase angle of polymer-bitumen binder significantly (in 3.2 and 4.1 times, respectively) lower in comparison with pure bitumen. Evaluation of rutting indicator under the Superpave system showed that pure bitumen is useful in initial state and after short term aging up to the temperature of 65 and 64°C, respectively and polymer-bitumen binder up to the temperature of 80 and 76°C, respectively.

At temperatures of 4 and 40°C after double aging the complex shear modulus of polymer-bitumen binder is less on 33 and 23%, respectively than pure bitumen. In considered temperature interval the indicator value of fatigue cracking at polymer-bitumen binder on average in 1.4 times less than at pure bitumen. All this facts shows higher stability of polymer-bitumen binder to fatigue cracking.

Research of binders behavior at low temperatures after their double aging showed that according to the Superpave requirements pure bitumen and polymer-bitumen binder can be used in regions with minimal winter temperature of -42 and -45°C, respectively.

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