

ISSN 2518-1483 (Online),  
ISSN 2224-5227 (Print)

2020 • 1

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ  
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫң

# БАЯНДАМАЛАРЫ

## ДОКЛАДЫ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
РЕСПУБЛИКИ КАЗАХСТАН

## REPORTS

OF THE NATIONAL ACADEMY OF SCIENCES  
OF THE REPUBLIC OF KAZAKHSTAN

PUBLISHED SINCE 1944



ALMATY, NAS RK

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2020 • 1

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ISSN 2518-1483 (Online),  
ISSN 2224-5227 (Print)

Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» Республикалық қоғамдық бірлестігі (Алматы қ.)  
Қазақстан республикасының Мәдениет пен ақпарат министрлігінің Ақпарат және мұрагат комитетінде 01.06.2006 ж.  
берілген №5540-Ж мерзімдік басылым тіркеуіне қойылу туралы күелік

Мерзімділігі: жылына 6 рет.

Тиражы: 500 дана.

Редакцияның мекенжайы: 050010, Алматы қ., Шевченко көш., 28; 219, 220 бөл.; тел.: 272-13-19, 272-13-18,  
<http://reports-science.kz/index.php/en/archive>

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Типографияның мекенжайы: «NurNaz GRACE», Алматы қ., Рысқұлов көш., 103.

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**Доклады Национальной академии наук Республики Казахстан»**

**ISSN 2518-1483 (Online),**  
**ISSN 2224-5227 (Print)**

Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы)

Свидетельство о постановке на учет периодического печатного издания в Комитете информации и архивов Министерства культуры и информации Республики Казахстан №5540-Ж, выданное 01.06.2006 г.

Периодичность: 6 раз в год.

Тираж: 500 экземпляров

Адрес редакции: 050010, г.Алматы, ул.Шевченко, 28; ком. 219, 220; тел. 272-13-19, 272-13-18,  
<http://reports-science.kz/index.php/en/archive>

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Адрес типографии: «NurNaz GRACE», г. Алматы, ул. Рыскулова, 103.

# REPORTS

2020 • 1

OF NATIONAL ACADEMY OF SCIENCES OF THE  
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**Reports of the National Academy of Sciences of the Republic of Kazakhstan.**

ISSN 2224-5227

**ISSN 2518-1483 (Online),**

**ISSN 2224-5227 (Print)**

Owner: RPA "National Academy of Sciences of the Republic of Kazakhstan" (Almaty)

The certificate of registration of a periodic printed publication in the Committee of Information and Archives of the Ministry of Culture and Information of the Republic of Kazakhstan N 5540-Ж, issued 01.06.2006

Periodicity: 6 times a year

Circulation: 500 copies

Editorial address: 28, Shevchenko str., of. 219, 220, Almaty, 050010, tel. 272-13-19, 272-13-18,  
<http://reports-science.kz/index.php/en/archive>

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Address of printing house: «NurNaz GRACE», 103, Ryskulov str, Almaty.

# *Chemistry*

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## **REPORTS OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN**

ISSN 2224-5227

Volume 1, Number 329 (2020), 134 – 138

<https://doi.org/10.32014/2020.2518-1483.17>

UDC 625.7/8:691.16

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## **EFFECT OF ROAD BITUMEN MODIFICATION WITH NANOCARBON POWDER**

**Abstract.** Low temperature characteristics (stiffness and m-value) have been determined on a bending beam rheometer in the paper for a road bitumen of grade BND 70/100 and the same bitumen with adding of a nanocarbon powder in the quantity of 2% by weight. The bitumen has been produced by Pavlodar petrochemical plant (Kazakhstan) from a crude oil of Western Siberia (Russia) by direct oxidation. The bitumen has the following standard characteristics: penetration depth of the needle (penetration) at 25°C: 75 dmm; ductility at 25°C: 118 cm; softening point: 47.5°C; Fraas point: -28.5°C. The nanocarbon powder with sizes of 150-200 nm has been obtained from a coal of the “Saryadyr” deposit (Akmola region, Kazakhstan). A mechanical disperser has been used for dispersing of the coal with sizes up to 50 mm (till 2-3 mm), an aerodynamic mill (till 20 µm) and a reactor with rotating electromagnetic field. Content of carbon and ash in the nanopowder is 92.74% and 2.16% respectively. Average moisture of the nanopowder is 2%. Before testing on the bending beam rheometer the neat bitumen and the nanocarbon bitumen have passed double artificial aging: short-term aging under standard AASHTO T 240-13 and long-term aging under standard ASTM D 6521-08 [14]. It is found out that addition of the nanocarbon powder improves low temperature resistance of the bitumen. The effect of the bitumen modification with the nanocarbon powder is the highest one at very low temperatures. The possibility has been shown for the use of the bitumen of grade BND 70/100 (of more viscous consistence) at its modification with the nanopowder both in the southern regions of the republic and in the northern ones.

**Keywords:** Bitumen, nanocarbon powder, bending beam rheometer, stiffness, m-value.

**Introduction.** Multi-year investigations of Kazakhstan Highway Research Institute [1-5] have shown that climatic conditions of Kazakhstan require the improvement of operational characteristics for bitumens. One of the ways for the increase of bitumen resistance to sharp continental climatic conditions is their modification with different polymers [6-7]. Our works [8-10] have shown the possibility for the increase of road bitumen characteristics at low temperatures by their modification with nanocarbon powder. This paper is continuation of our above works and contains additional results for improvement of low temperature resistance for road bitumen modified with nanocarbon powder.

### **Materials and Methods**

**Bitumen.** A bitumen of grade 70/100 produced by Pavlodar petrochemical plant which is traditionally used in road construction in Kazakhstan has been accepted for investigation. Characteristics of the bitumen comply with the requirements of the standard of Kazakhstan ST RK 1373-2003 [11] (Table 1).

Table 1- Main standard indicators for the bitumen of grade BND 70/100

Penetration depth of the needle at 25° C, 0.1 mm	Ductility at 25° C, cm	Softening point, ° C	Fraas point, ° C
75	118	47.5	-28.5

**Nanocarbon powder.** A nanopowder with sizes of 150-200 nanometers (nm) has been produced from a coal of the “Saryadyr” deposit belonging to “Corporation “On-Olzha” LLP (Akmola region, Kazakhstan). Indicators of the nanopowder quality after purification are shown in Table 2.

Table 2 - Indicators of the nanopowder quality

Indicator	Value
Sizes, nm	150-200
Content of carbon, %	92.74
Ash content, %	2.16
Volatile substances, %	3.10
Moisture, %	2.00

**Production of the nanocarbon powder.** The nanocarbon powder has been produced by three-staged dispersion of the coal rock. On the first stage the grains of the coal rock with sizes up to 50 mm have been dispersed on a mechanical dispergator till sizes of 2-3 mm (coal dust). On the second stage the coal dust has been dispersed till size of 20  $\mu\text{m}$  in an aerodynamic mill. On the third stage a special reactor is used to obtain the carbon powder up to sizes 200 nm where an electromagnetic field is rotated with the speed of 50-3000 rotations per second.

**Preparation of the nanocarbon bitumen.** First the carbon nanopowder was dispersed in kerosene with the purpose of uniform distribution provision of the nanopowder particles in the bitumen through the impact on ultrasound with the frequency of 90 kHz for 5 min at a room temperature. Then the dispersion (kerosene + nanopowder) was added into the bitumen at the temperature of 160° C and continuous mixing for 30 min.

**Low temperature characteristics of the nanocarbon bitumen.** At present in many countries of the world in accordance with the Technical System Superpave the low temperature resistance of bitumens and bituminous binders is evaluated under the results of their testing on a bending beam rheometer (BBR) according to the standard ASTM D 6648 [12]. Before testing bitumens and bituminous binders pass double artificial aging: short-term aging under standard AASHTO T 240-13 [13] and long-term aging under standard ASTM D 6521-08 [14].

**Results and Discussion.** Figures 1 and 2 show the graphs for variation of stiffness (S) and m-value of the bitumen and the nanocarbon bitumen in time at the temperatures of -24.2° C, -30.3° C and -35.9° C constructed according to the results of their testing in BBR. As it is seen from Figure 1 the nanocarbon powder reduces considerably the bitumen stiffness at all the considered temperatures and the time intervals, i.e. it increases essentially its low temperature resistance.

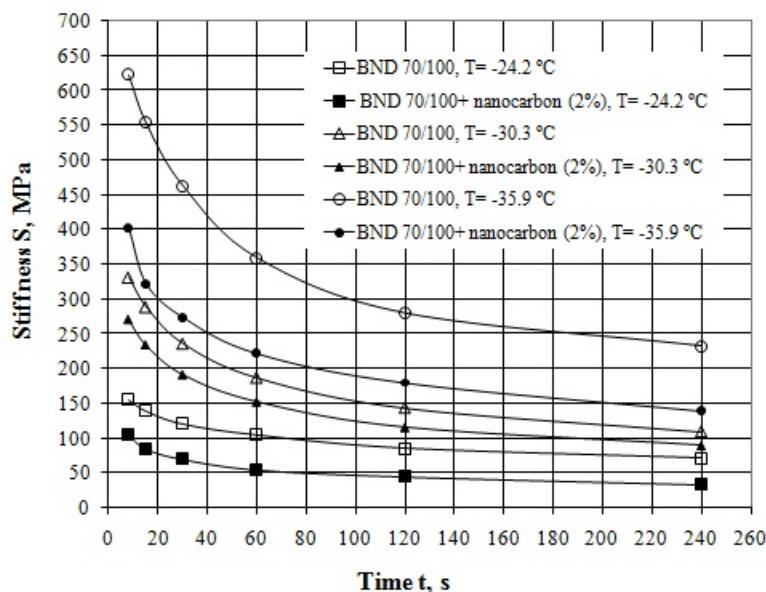


Figure 1 - Graphs for stiffness variation of the bituminous binders in time at low temperatures

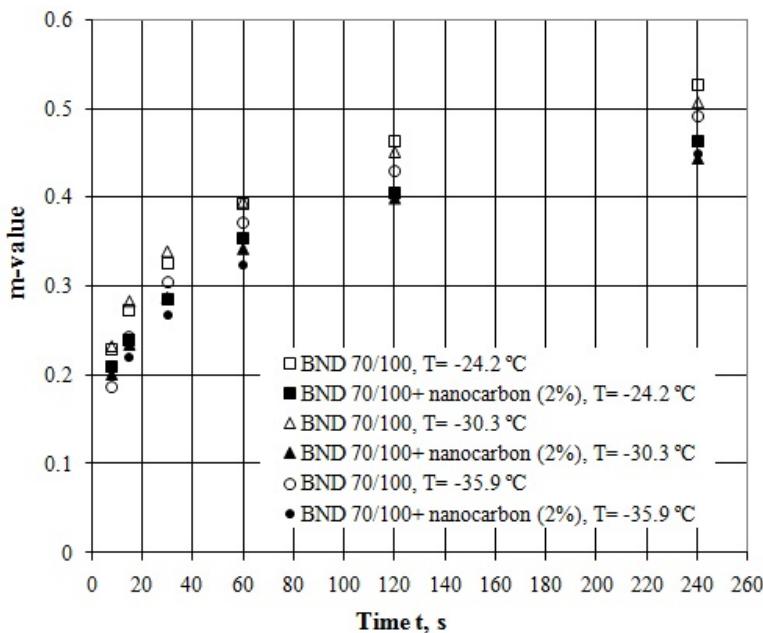


Figure 2 - Graphs for m-value variation of the bituminous binders in time at low temperatures

As it is known [15], in the Technical System Superpave the load duration equal to 60 s is a characteristic one, and the values of stiffness and m-value for bituminous binders at duration of  $t=60$  s are accepted as the standard ones.

Table 3 presents the stiffness values of the bitumen and the nanocarbon bitumen at  $t=60$  s.

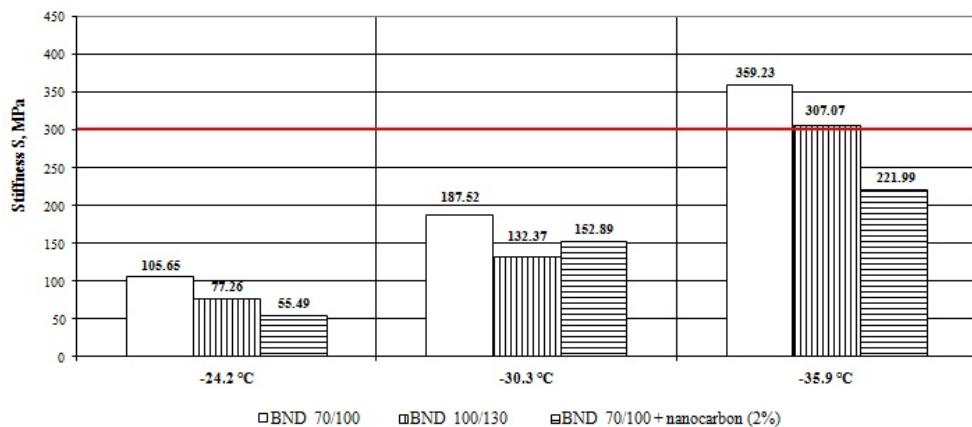
Table 3 - Stiffness values of the bitumen and the nanocarbon bitumen at  $t=60$  s

Temperature, ° C	Stiffness S, MPa		Difference $\Delta S$ , MPa
	Bitumen	Nanocarbon bitumen	
-24.2	105.65	55.49	50.16
-30.3	187.52	152.89	34.63
-35.9	359.23	221.99	137.24

As it is seen from Table 3, the effect of modification with the nanocarbon powder is high at very low temperatures (-36° C).

In the Technical System Superpave it is accepted to consider bituminous binders as the resistant ones at low temperatures, which have m-value higher than 0.3 at  $t=60$  s. The nanocarbon bitumen complies with this requirement at all the considered temperatures.

The bitumen of grade 70/100 is recommended to use in road construction in southern regions of Kazakhstan, and for northern regions - to use the bitumens of grade 100/130. Figure 3 shows the stiffness values at  $t=60$  s for the bitumens of grades 70/100 and 100/130 and the nanocarbon bitumen (BND 70/100 + nanocarbon powder – 2%). It is seen that at the temperature of -36° C the nanocarbon bitumen has the stiffness of (232 MPa) considerably lower (lower for 85 MPa) than the bitumen of grade BND 100/130 (307 MPa). This established fact shows the possibility for the use of the bitumen of grade BND 70/100 by modification with the nanopowder in the northern regions of the republic as well. Meanwhile, low temperature resistance of this nanocarbon bitumen remains considerably high compared with the bitumen of grade 100/130.

Figure 3 - Stiffness values of the bituminous binders at low temperatures  $t=60$  s

### Conclusion.

1. A nanopowder with sizes of 150-200 nm has been obtained from Kazakhstan coal by a mechanical dispersator, an aerodynamic mill and a reactor with a rotating electromagnetic field.
2. Addition of the nanocarbon powder in the quantity of 2% into a road bitumen improves considerably its low temperature resistance. The effect of the bitumen modification with the nanocarbon powder is the highest one at very low temperatures (-36° C).
3. The possibility has been shown for the use of the bitumen of grade BND 70/100 at its modification with the nanopowder both in the southern regions of the republic and in the northern ones.

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## ЖОЛ БИТУМЫН НАНОКӨМІРТЕК ҰНТАҒЫМЕН МОДИФИКАЦИЯЛАУДЫҢ ӘСЕРІ

**Аннотация.** Бұл мақалада МЖБ 70/100 маркалы жол битумы мен осы битумның салмағы бойынша 2% мөлшерде нанокөміртек ұнтағын қосқандагы төменгі температуралық сипаттамалары (қаттылық және т-көрсеткіш) иілгіш білікті реометрде анықталды. Битум Павлодар мұнай-химия зауытында (Қазақстан) Батыс Сібірдің (Ресей) шикі мұнайынан тіке тотықтыру әдісімен өндірілді. Битумның стандарттық сипаттамалары: 25°С-дагы иненің ену терендігі (пенетрация): 75 дмм; 25 °С-дагы созылуы: 118 см; жұмсару температурасы: 47,5°C; морттық температурасы: - 28,5°C. Өлшемдері 150-200 нм нанокөміртек ұнтағы «Сарыадыр» кен орнының (Ақмола облысы) көмірінен альянды. Өлшемі 50 мм-ге дейін көмірді ұнтақтауга механикалық диспергатор (2-3 мм-ге дейін), аэродинамикалық диірмен (20 мкм-ге дейін) және айналма магнит өрісті реактор пайдаланылды. Наноұнтақтағы көмертек пен күлдің мөлшері тиісінше 92,74% және 2,16%. Наноұнтақтың орташа ылғалдылығы 2%. Иілгіш білікті реометрде сынақтан өткізуін алдында таза битум және нанокөміртекті битум екі сатылы жасанды ескіртуден өтті: AASHTO T 240-13 стандарты бойынша қыска мерзімді ескіртуден және ASTM D 6521-08 стандарты бойынша ұзак мерзімді ескіртуден. Нанокөміртек ұнтағын қосу битумның төменгі температуралық тұрактылығын едәуір жақсартатындығы анықталды. Битумды нанокөміртек ұнтағымен модификациялаудың әсері өте төменгі температураларда барынша үлкен. МЖБ 70/100 маркалы (тұтқырылығы жоғары) битумды нанокөміртек ұнтағымен модификациялау арқылы Қазақстанның өңтүстік аймақтарымен қатар солтүстік аймақтарында да пайдалану мүмкіндігі көрсетілген.

**Түйінді сөздер:** Битум, нанокөміртек ұнтағы, иілгіш білікті реометр, қаттылық, т-көрсеткіш.

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## ЭФФЕКТ МОДИФИКАЦИИ ДОРОЖНОГО БИТУМА НАНОУГЛЕРОДНЫМ ПОРОШКОМ

**Аннотация.** В настоящей статье на реометре с изгибаемой балкой определены низкотемпературные характеристики (жесткость и m-value) дорожного битума марки БНД 70/100 и этого битума при добавке

наноуглеродного порошка в количестве 2% по массе. Битум был произведен Павлодарским нефтехимическим заводом (Казахстан) из сырой нефти Западной Сибири (Россия) способом прямого окисления. Битум имеет следующие стандартные характеристики: глубина проникания иглы (пенетрация) при 25°C: 75 дмм; растяжимость при 25°C: 118 см; температура размягчения: 47,5°C; температура хрупкости: -28,5°C. Наноуглеродный порошок с размером 150-200 нм получен из угля месторождения «Сарыадыр» (Акмолинская область, Казахстан). Для измельчения угля с размером до 50 мм использованы механический диспергатор (до 2-3 мм), аэродинамическая мельница (до 20 мкм) и реактор с врачающимся магнитным полем. Содержание углерода и золы в нанопорошке составляет соответственно 92,74% и 2,16%. Средняя влажность нанопорошка составляет 2%. Перед испытанием на реометре с изгибаемой балкой чистый битум и наноуглеродный битум прошли двойное старение: кратковременное по стандарту AASHTO T 240-13 и длительное по стандарту ASTM D 6521-08. Установлено, что добавка наноуглеродного порошка существенно улучшает низкотемпературную устойчивость битума. Эффект модификации битума наноуглеродным порошком наиболее высок при очень низких температурах. Показана возможность использования битума марки БНД 70/100 (более вязкой консистенции) при его модификации наноуглеродным порошком, как в южных, так и в северных регионах республики.

**Ключевые слова:** Битум, наноуглеродный порошок, реометр с изгибаемой балкой, жесткость, m-value.

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**ISSN 2518-1483 (Online), ISSN 2224-5227 (Print)**

<http://reports-science.kz/index.php/en/archive>

Редакторы: М. С. Ахметова, Г. Б. Халидуллаева, Д. С. Аленов

Верстка на компьютере А.М. Кульгинбаевой

Подписано в печать 12.02.2020.

Формат 60x881/8. Бумага офсетная. Печать – ризограф.  
12,12 п.л. Тираж 500. Заказ 1.