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Investigation for Modification of Road Bitumen with Nanocarbon Materials

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Abstract

This paper presents methods for the production of nanoscale coke powders for bitumen modification. The chemical vapor deposition (CVD) method enables the production of nanopowders with particle sizes ranging from 14 to 30 nm at temperatures of 1000–1200 °C. Bitumen modification was performed by adding nanoscale coke powders, in accordance with the requirements of Kazakhstan standard 1373–2013. The nanosized powders were obtained by CVD in an inert nitrogen atmosphere with a specific surface area of 24.8 m^2/g. Modification of bitumen with nanoscale coke yields grades with high thermal stability, ensuring optimal structural strength and durability of the pavement. Rheological studies show that nanoscale coke acts as an active structure-forming agent rather than an inert filler. The restriction of asphaltene mobility leads to a consistently reduced phase angle (δ) and the absence of a transition to a viscosity-dominated regime. The samples are characterized by the lowest phase angle values across the entire range of the complex modulus. This indicates the formation of a highly developed elastic-viscous structure due to the presence of nanosized coke, which promotes the formation of a percolating physical network and restricts the mobility of the bituminous system. The observed behavior indicates the dominance of the elastic component and the increased structural organization of the resulting bituminous binders.

1. Introduction

Recently, numerous studies have been published on the use of coke as a modifier in road construction materials [1–7]. Study [8] demonstrated the feasibility of using non-target fractions and high-sulfur cokes as additives in the production of asphalt concrete mixtures. Owing to its nature, coke possesses high adsorption capacity, and its use as an adsorbent is a promising area of research. Petroleum coke has been identified as a high-molecular-weight, spatially cross-linked, metal-containing, and polydisperse polymer. Consequently, its use as an additive in bituminous materials and asphalt concrete mixtures is expected to improve certain characteristics. The carbene groups in the coke are high-molecular-weight substances, while the carbides are a cross-linked three-dimensional polymer, a significant portion of whose carbon atoms are incorporated into condensed aromatic structures. The addition of these compounds to bituminous binders provides high tensile strength. New bonds form between the rigid blocks of the cross-linked polymer and the polydisperse system in the bitumen. These bonds do not break at high temperatures as the mobility of the macromolecules increases, allowing the cross-linked polymer to become irreversibly incorporated into the spatial structure of the bituminous binder. This explains the reduced bleeding of the bituminous binder with coke [9].

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Among nanomaterials, carbon nanotubes and fullerenes have been proposed as modifiers for bitumen and asphalt concrete due to their unique physicochemical properties, which enable the targeted formation of the composition and structure of road construction composite materials. A nanostructuring modifier for asphalt concrete described in [10] consists of bitumen uniformly dispersed in 0.2–10% carbon nanotubes, 10–20% technical carbon, and 1–20% organoclay. The dispersion process was carried out in a bath under ultrasonic irradiation for 10 min at 180 °C. The test results showed an increase in the compressive strength of the asphalt concrete from 5.6 to 8.6 MPa, which is explained by the nanostructured system's tendency to reduce free energy due to a synergistic reinforcing effect. The disadvantages of the nanomodifier are its high content in the asphalt concrete mixture and the need for additional ultrasonic treatment to ensure complete distribution of the modifier in the bitumen.

To prepare the ArmBit modifying nano-additive for bitumens and asphalt concrete, the ArmCap composition has been proposed, containing multiwalled carbon nanotubes and nanodiamonds. This modification is distinguished by the fact that the bitumen directly absorbs the nanoparticles immediately after their formation, which prevents their aggregation for more than six months. Nanomodifier with concentration of 0.005% in asphalt concrete demonstrated improved physical and mechanical properties compared to the regulatory document requirements [11]. The action mechanism of the nanomodifier is explained by the interaction of long-chain bitumen molecules in the formation of a strong spatial network. The addition of the modifier to the asphalt concrete matrix initiates a specific physical-chemical process of converting the surface energy of nanoscale particles, which leads to the ordering and strengthening of the bitumen systems.

The asphalt concretes in patents [12, 13] consisted of crushed stone, sand, bitumen, and a carbon modifier. The 0.03–0.06% nanomodifier added to heated bitumen at 130–140 °C. Mineral powder (MP-1) was also added to the asphalt concrete mixture. Modification result presents compressive strength of the asphalt concrete at 20 and 50 °C increased to 3.9–4.3 MPa and 1.6–1.75 MPa, respectively. The strength of the asphalt concrete mixture increases due to the improvement of the modified bitumen's structure by carbon nanomaterials, which leads to the effective transition of bitumen from a bulk to a film state.

Research [14] proposed the modification of polymer-bitumen binders with carbon nanoparticles, which well linked with polymers but also to the surface of mineral components. Modification of dolomite flour used as a mineral powder with 10% carbon nanoparticles, 3% SBS polymer, and 4–10% plasticizer made it possible to obtain an asphalt concrete binder with properties similar to those of polymer-bitumen binders.

Paper [15] demonstrates that the simultaneous incorporation of SBS polymer and a suspension based on diesel fuel and carbon nanotubes obtained a modified bitumen binder with improved penetration and elasticity. Increasing content of the nanotube led to a decrease in ductility and a drop in elasticity the optimal nanotube concentration is 0.016%. The improvement in performance characteristics is attributed to the participation of active carbon centers in the formation of a three-dimensional cross-linked structure. To prevent agglomeration of the nanotubes, the suspension was prepared using ultrasound, and diesel fuel used as a plasticizer.

The aim of the present study is to investigate the modification technology for bitumen binders modified with high-nanocarbon materials to improve the performance characteristics of asphalt concrete.

2. Materials and Methods

The carbon materials used in this study were obtained from industrial petroleum coke at the Pavlodar Petrochemical Plant (Kazakhstan). The petroleum coke was produced by the coking of heavy oil residues (tars). Form of material present solid carbon material.

Carbon nanomaterials were synthesized from petroleum coke samples using chemical vapor deposition (CVD). The coke samples were processed at temperatures of 1000–1200 °C in an inert atmosphere. The samples were heated to the specified temperatures at a rate of 10 °C/min. A flow of inert gas was fed into the reaction zone at a rate of 1000 ml/min. After reaching each set temperature (1000, 1100, and 1200 °C), the samples were held at that temperature for 30 minutes. The samples were then cooled to room temperature at a rate of 10 °C/min. The nanostructure of the carbon material was characterized using a JEM 100CX transmission electron microscope at an accelerating voltage of 100 kV (in a vacuum, at low temperature). In transmission electron microscopy, the electron beam interacts with the sample, forming an image on a photographic plate and a specialized camera. Sample preparation was performed using the dry method.

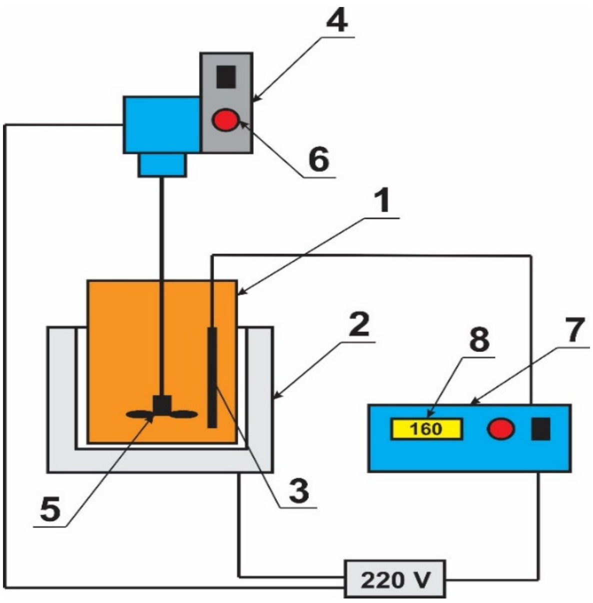

Fig. 1. Schematic diagram of the experimental setup for bitumen modification: (1) – cylindrical reactor; (2) – electric furnace; (3) – temperature sensor (thermocouple); (4) – stirrer; (5) – metal propeller; (6) – stirrer speed controller; (7) – temperature controller; (8) – digital display.

A laboratory setup was used to modify bitumen with nanocoke. A schematic diagram of the setup is shown in Fig. 1. The setup consists of a cylindrical reactor (1) with a length of 20 cm and an internal diameter of 15 cm. The reactor is heated using an electric furnace (2). The reactor is equipped with a thermocouple (3) connected to a temperature controller (7) for monitoring and maintaining the desired temperature. The mixing speed of the bitumen–nanocoke mixture is controlled by a stirrer (4) equipped with a metal propeller (5) driven by a speed controller (6). The heating power is regulated by adjusting the supply voltage to the furnace through the temperature controller, while the process temperature is displayed on the digital display (8).

Modification of 100/130 road bitumen with nanoscale coke powders in amounts of 0.1, 0.5, and 1% was performed using a propeller mixer at a rotation speed of 750 rpm at a temperature 160 °C for 1 hour. The fatigue strength of the modified bituminous binders was evaluated using the Linear Amplitude Sweep (LAS) test method according to AASHTO T391-20 [16], based on the Viscoelastic Continuum Damage (VECD) model per AASHTO TP101-14 [17]. The tests were conducted on a SmartPave 102e dynamic shear rheometer using an 8 mm diameter parallel-plate geometry at a temperature of 25 °C. Measurements were taken at a fixed strain of 0.1% in the frequency range from 0.2 to 30 Hz. The vibration frequency remained constant (10 Hz), while the strain amplitude increased uniformly from 0.1% to 30% over a specified number of steps. In this way, real-world cyclic loading conditions characteristic of road pavement operation were simulated. The data obtained were processed using the RheoCompass software.

The J_{nr} parameter is the irreversible component of the modulus of compliance, equal to the specimen's deformation following a creep-recovery cycle, divided by the applied stress. The obtained parameters J_{nr}3.2 kPa value is key for classifying the binder (according to the Superpave method), as it determines the binder's resistance to residual deformation the lower the J_{nr}3.2 kPa value, the greater the resistance to rutting. The recovery result $R_{3.2}$, in turn, indicates the effectiveness of the bituminous binder modification. The percentage change in J_{nr} following a stress change from 0.1 to 3.2 kPa measures the binder's sensitivity to increased load, with the requirement that such an increase not exceed 75%.

The Multiple Stress Creep Recovery (MSCR) test allows the assessment of a bituminous binder's ability to resist deformation under repeated loading and to recover its original shape after the load is removed. This analysis is widely used in road construction to evaluate the quality of bitumen and its suitability for use in various climatic conditions. The MSCR test helps determine how well bitumen will resist aging and deformation under the influence of loading and temperature fluctuations, ensuring a longer service life for the pavement. The tests were performed using a dynamic shear rheometer equipped with parallel plates of 25 mm diameter and a gap setting of 1 mm. The test temperature was 58 °C. The load levels of 0.1 and 3.2 kPa resulted in 20.32% and 9.93%, respectively. The test was conducted for Class "V" heavy traffic in accordance with AASHTO M 332-20 [18].

3. Results and Discussion

Chemical vapor deposition promotes the transition of carbon from an amorphous or weakly ordered state to a polycrystalline form with a high degree of layer orientation. The most pronounced increase in specific surface area by BET is observed at a synthesis temperature of 1100 °C from 1.23 to 24.83 m^2/g. This increase may be associated with the formation of a more developed porous structure. Surface area by the Langmuir method and t-Plot:

a similar trend is observed for the surface area calculated by the Langmuir method (with a maximum of 36.25 m^2/g) and the external surface area by t-Plot (23.15 m^2/g). The micropore volume according to t-Plot is a positive value (0.000611 cm^3/g), indicating the development of microporosity. The average adsorption pore diameter according to BET (13.18 Å) and BJH (20.71 Å) may be a consequence of pore expansion and openness. The average particle size of the synthesized nanoparticles was approximately 30 nm. The overall morphology suggests that the carbon nanomaterial is not highly ordered or graphitized, as evidenced by the relatively low contrast and the appearance of more agglomerated and less well-defined structures compared to highly crystalline carbon materials.

TEM analysis of coke-derived carbon nanomaterials revealed the formation of irregular layered carbon structures with nanoscale morphological features, indicating the successful generation of carbon-rich nanostructured materials. In Fig. 2A with 200 nm scale, the nanomaterial exhibits aggregated flake-like particles with heterogeneous morphology, suggesting the formation of disordered carbon sheets and stacked domains typical of partially graphitized carbon. Figure 2B with 50 nm scale shows a higher magnification view of the particle edge, where thin layered fringes and partially transparent regions indicate the presence of few-layer carbon nanosheets. These features suggest local ordering and the development of graphitic domains at the nanoscale. Carbon nanomaterial provides further evidence of compact nanostructured carbon with denser lattice-like contrast and finer textural organization, indicating increased structural ordering at smaller scales. The progressive transition from agglomerated particles to layered edge structures and denser nanoscale ordering demonstrates hierarchical carbon architecture. These observations confirm the formation of defect-rich, partially graphitized carbon nanomaterials derived from coke, which are promising for advanced functional material applications.

The physical and mechanical properties of bitumen samples modified with nanoscale coke powders are presented in Table 1. As the modifier content increases, the penetration value and softening temperature of the bitumen samples modified with nanoscale coke powders change non-monotonically. Bitumen modified with 0.5% nanocoke demonstrated the maximum penetration value (80) and softening temperature (46.5 °C). In terms of physical and mechanical properties. Obtained all samples meets requirement to Kazakhstan standard 1373-2013.

Table 1. Physical and mechanical properties of bitumen binders

Parameters	Nanocoke content, wt. %				Kazakhstan standard requirement 1373–2013
	0	0.1	0.5	1.0	
Penetration at 25 °C	80	76	80	74	71–100
Softening temperature, °C	45.5	47.7	46.5	47.3	Not lower 45

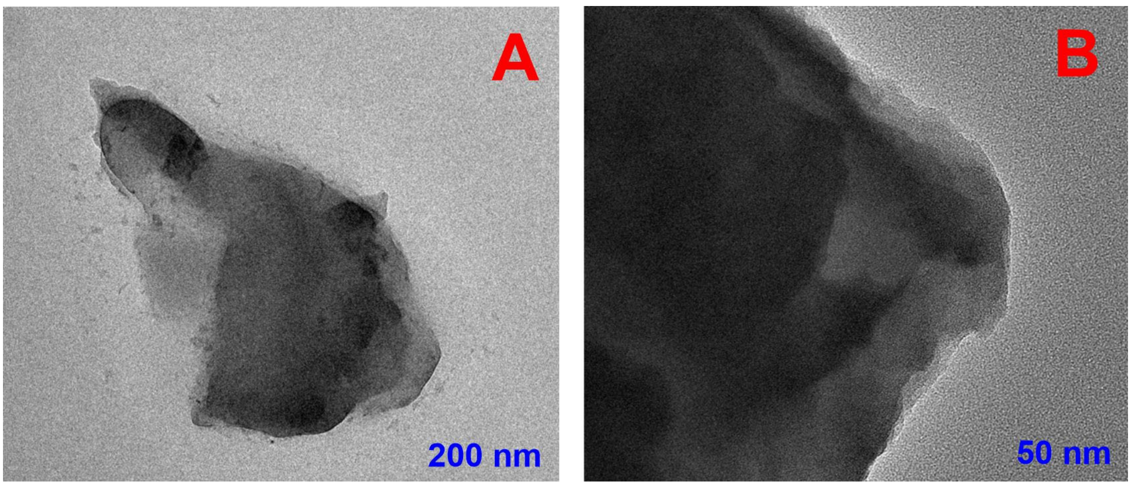

Fig. 2. TEM images of carbon nanomaterials.

Under the Superpave system, bitumens are classified according to a nominal temperature range known as the Performance Grade (PG). Since 2010, the system has been supplemented with a test for Multi-Stress Creep and Recovery, which determines the traffic class suitable for a given bituminous binder. Table 2 lists the requirements for bitumen performance indicators by traffic category and intensity according to the Superpave system. The suitability of a bitumen for a specific traffic category is determined by the $J_{nr}3.2$ index.

Table 3 presents the results of tests on modified bitumens for creep and recovery under repeated loading. The values of irreversible creep compliance at low (0.1 kPa) and high (3.2 kPa) J_{nr} load levels characterize the amount of permanent deformation that remains after the load is removed. The lower the value, the better the bitumen resists rutting. Comparing J_{nr} at different load levels allows for an assessment of the bitumen binders sensitivity to stress changes. The percentage change in J_{nr} between two load levels reflects the bitumen sensitivity to load; a high percentage indicates greater sensitivity to load.

The percentage of deformation recovery at low (0.1 kPa) and high (3.2 kPa) load levels (R) reflects the elastic component of bitumen behavior. The higher the value, the greater the amount of elastic deformation that is recovered. The percentage change in R between the two load levels indicates the extent to which recovery capacity decreases as the load increases.

Among the samples of bitumen modified with nanocoke, the bitumen with a 1% addition of nanocoke present the lowest values of non-recoverable creep compliance at low (0.1 kPa) and high (3.2 kPa) load levels; however, the J_{nr}, diff value of the original bitumen (20.41%) was slightly higher than that of the bitumen modified with 0.5% nanocoke (19.43%). Bitumen binders maximum R value under low and high loads is observed with the addition of 1% nanocoke, which is also confirmed by the minimum R_{diff} value of 79.62%. According to AASHTO M 332-20, a $J_{nr}3.2$ value in the range of 2.0–3.0 kPa^{-1} corresponds to the “S” grade as per Table 3 and is recommended as a binder in road pavements for standard traffic.

Figure 3 shows the dependencies of shear strain on loading time, which allow for a clear comparison of the stability and strength characteristics of bitumens with carbon additives. In the elastic range up to 5% deformation, all curves rise almost identically, indicating similar initial elastic behavior. However, the modified bitumen with 0.5% nanocoke already slightly outperforms the others at this stage, indicating the highest initial elasticity.

All specimens present similar linear behavior, indicating similar elastic characteristics in the initial loading phase. Figure 4 shows the sample containing 1% nanocoke exhibits the highest stiffness, a finding also confirmed by the VECD parameter (a damage modeling system for viscoelastic continuous media) – model C_0 = 2.03. The addition of coal leads to a significant increase in strength, particularly at a dosage of 1%. After reaching a maximum, all samples droped in stress a sign of structural failure. The neat bitumen and the 0.1% nanocoke additive degrade slowly and uniformly, whereas the 0.5% and especially the 1% nanocoke additives lose strength more abruptly, indicating increased brittleness value.

Table 2. Requirements for bitumen properties by traffic category and intensity according to the Superpave system

Cycling	Load	Requirement for $J_{nr}3.2$, kPa^{-1}
S (standard)	<10 million axles and standard traffic	≤ 4.0
H (intense)	10–30 million axles and low-speed traffic	≤ 2.0
V (very intense)	>30 million axles or parking	≤ 1.0
E (extremely intense)	>30 million axles and parking	≤ 0.5

Table 3. Test results for MSCR bituminous binders

Nanocoke content, wt.%	$R_{0.1}$, %	$R_{3.2}$, %	R_{diff}, %	$J_{nr}0.1$, kPa^{-1}	$J_{nr}3.2$, kPa^{-1}	J_{nr}, diff, %	Cycling
0	6.53	0.00	99.98	3.3487	4.0735	21.65	S
0.1	10.50	1.43	86.39	2.2045	2.7263	23.67	S
0.5	7.63	0.92	87.92	2.5124	3.0005	19.43	S
1.0	10.77	2.19	79.62	1.8052	2.1737	20.41	S

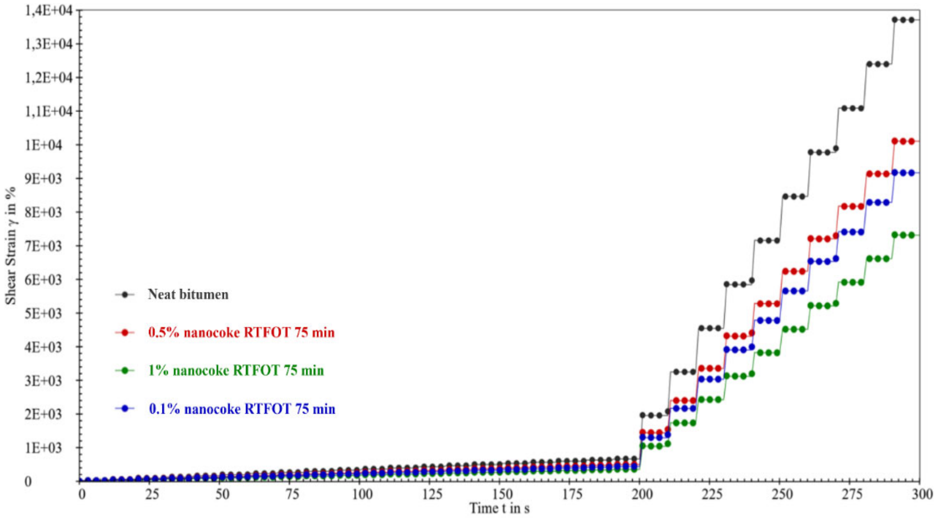

Fig. 3. Dependence of shear strain on loading time

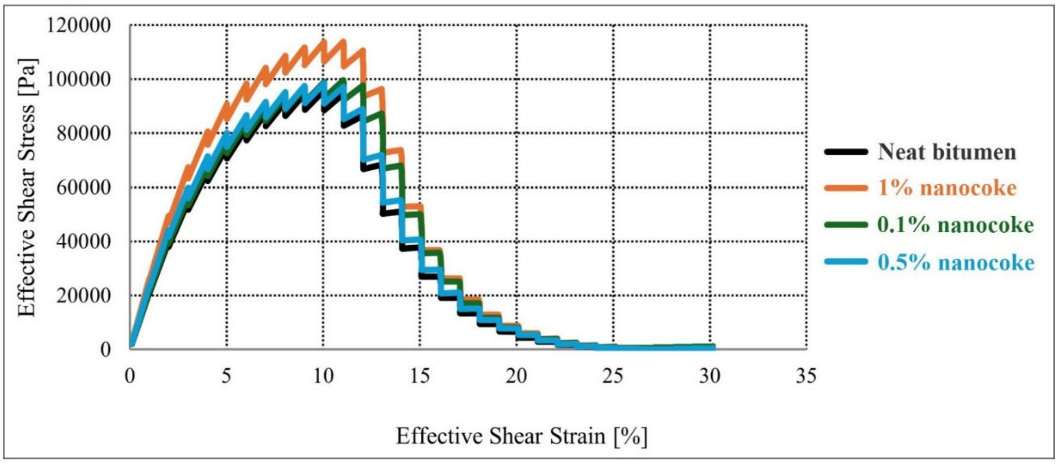

Fig. 4. Dependence of effective shear stress on effective shear strain.

The values of the C_0 parameter (Table 4) increase from the initial bitumen sample 1.71 to the modified samples, reaching a maximum for the bitumen with a 1% nanocoke additive (C_0 = 2.03). This indicates an increase in deformation resistance during the initial loading stage, which can be interpreted as an improvement in the elastic properties of the bitumen binder due to modification. This suggests that the nanoparticles lead to the formation of a stiffer structure in the bitumen binder. During modification, carbon nanoparticles with a high specific surface area interact with asphaltene aggregates and the polar components of bitumen, leading to the formation of a more structured dispersed system. With sufficient dispersion, the particles create a spatially distributed network of physical-mechanical contacts that facilitates stress redistribution and reduces their local concentration in the asphaltene phase. This limits the mobility of the dispersion medium, increases the effective stiffness of the system, and enhances

Table 4. Rheological characteristics of modified bitumens

Nanocoke content, wt.%	C_0	C_1	C_2	α (slope)	α/(1+α)	Model error
0	1.71	0.115	0.414	2.48	0.713	0.31
0.1	1.71	0.110	0.414	2.55	0.718	0.23
0.5	1.86	0.137	0.392	2.54	0.718	0.42
1.0	2.03	0.128	0.404	2.59	0.721	0.38

resistance to microcrack initiation during the initial loading stage, which is reflected in an increase in the C_0 parameter. The C_1 parameter determines how rapidly stiffness decreases as damage intensity increases. For most bitumen samples, C_1 decreased within a narrow range of 0.115 to 0.137. Bitumen with 0.5% nanocoke present the highest value of 0.137; while this indicates high initial stiffness, this material may lose it faster than others under cyclic loading. The C_2 parameter values indicate a change in the distribution of microcracks, making the failure process slightly more stable in the later stages.

Thus, bitumen samples modified with nanocoke present higher initial stiffness values (C_0), which potentially increases their resistance to deformation. However, at the same time, the failure coefficient (C_1) increases, indicating a decrease in fatigue resistance under prolonged loads. This is particularly evident in the bitumen sample containing 0.5% nanocoke, which shows the highest stiffness and susceptibility to damage. An optimal balance between stiffness and resistance to fatigue failure is observed in bitumen samples containing 1% and 0.5% nanocoke, whereas bitumen with 0.5% nanocoke may be effective under conditions of short-term high loads but not for long-term loading.

Bitumen binder with nanocoke up to 1%, the formation of new bonds between particles and asphaltene components increases. This limits the mobility of the maltene phase and increases the modulus (C_0) at small deformations. However, excessive structuring reduces the bitumen matrix's ability for viscoelastic relaxation and stress redistribution. Under conditions of prolonged cyclic loading, this leads to a higher concentration of local stresses and accelerated microcrack development, which is reflected in an increase in the C_1 coefficient and a decrease in fatigue resistance. The behavior characteristics of modified bitumens within the VECD model are presented in Table 5.

Table 5. Behavior characteristics of modified bitumens within the VECD model

Nanocoke content, wt.%	Strength	Behavior during a Violation	α/(1+α)
0	Low	Smooth Degradation	0.713
0.1	High	Sharper Decline	0.718
0.5	Medium	Smooth Degradation	0.718
1.0	Highest	Sharpest Decline	0.721

The addition of nanocoke increases the strength of bitumen by raising its peak stress and modulus of elasticity. Based on the analysis results, the sample containing 0.1% nanocoke is shown to be the optimal option in terms of the combination of load-bearing capacity, plasticity, and damage resistance. This composition demonstrates high peak stress, a low C_1 value indicating slow failure, and the smallest model error, confirming its stability.

The introduction of nanocoke into bitumen slows down the degradation of the bitumen structure. The modified compositions present higher fatigue durability. This is due to the improved distribution of particles within the binder volume without the formation of large agglomerates, which could act as stress concentrators. Coke particles are characterized by high adsorption capacity and retain a greater amount of bitumen on their surface than other known additives. Carbenes and carbides in the coke composition are rapidly reduced after thermal treatment and return to their original state. Nanocoke in bitumen change internal structure and resistance to microcracks. It is supposed that nanocoke particles in the asphalt concrete mixture form a dense and strong skeletal network, increasing compressive strength at high temperatures.

Nanocoke, like other carbon-based fillers (carbon nanotubes, graphene, carbon black), enhances the elastic component of bituminous material, promotes the formation of fractal or percolating networks at the microscale, and manifests itself in a decrease in the phase angle δ, especially during the transition from the viscous to the elastic regime.

Figure 5 shows, in the Van Gurp-Palmen plot, a downward shift of the δ curve compared to unmodified bitumen and an expansion of the region where δ is consistently below the baseline. This indicates the formation of an elastic-viscous structure caused by interactions between nanocoke particles and with the bitumen matrix, which enhances the material's structural integrity and elastic properties.

Literature data [19, 20] indicate that the primary mechanism of the bitumen-nanocoke reaction involves nanocoke actively adsorbing resins, polar aromatic oils, and heteroatomic compounds (S, N, O) without chemical bonding, but forming a spatial network. Here, nanocoke acts as a hub for asphaltene aggregation, forming an "asphaltene-nanocoke-asphaltene" framework, increasing the shear modulus and thermal stability. In our case, at high temperatures, radical stabilization is also possible on the nanocoke surface, reducing the rate of oxidative degradation. The Van Gurp-Palmen plot

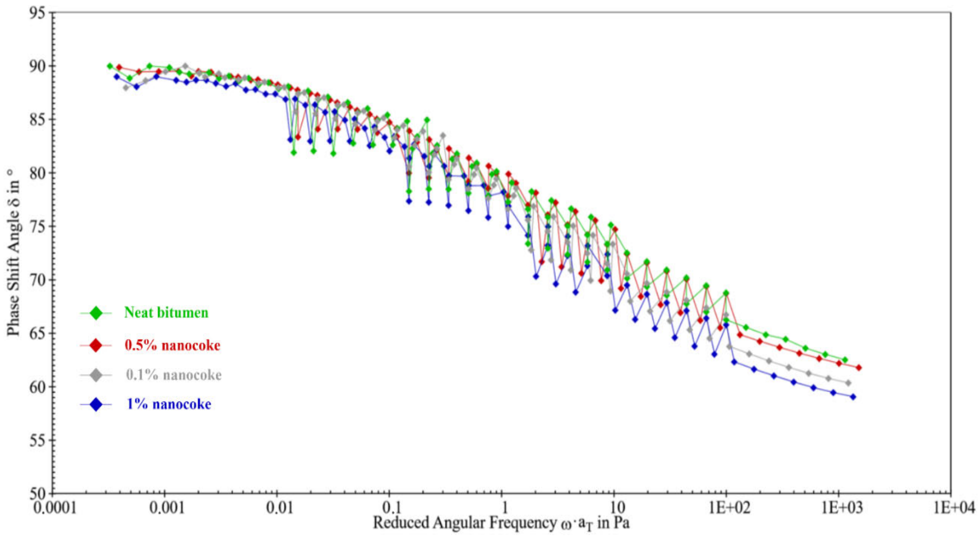

Fig. 5. Dependence of the phase shift angle on the complex shear modulus.

toward smaller phase angles and the appearance of a δ plateau indicate the formation of a stable spatial structure of bitumen due to the adsorption-asphalt and surface-radical interactions of nanocoke. The Van Gurp-Palmen plot is a structure-invariant representation of viscoelastic properties, where the curve shape directly reflects the presence of asphaltene nanoaggregates and network structures formed by nanofillers. Changes in phase angle, such as a decrease in phase angle and the appearance of bends, are interpreted as the result of increased intermolecular and interfacial interactions in the bitumen system.

4. Conclusion

Nanocoke particles were synthesized by chemical vapor deposition in an inert nitrogen atmosphere at 1100 °C for 30 minutes. The synthesized nanoparticles had sizes ranging from 14 to 30 nm, a BET specific surface area of 24.8 m^2/g, a Langmuir method surface area of 36.2 m^2/g, and an average BET adsorption pore diameter of 13.1 Å.

Bitumen was modified with nanocoke samples, and the physical and mechanical properties of the resulting bituminous binders were evaluated in accordance with standard requirements. Bitumen modified with 0.1–1% nanocoke met the requirements of Kazakhstan Standard 1373–2013 with respect to its main physical and mechanical characteristics.

It was established that the bituminous binder containing 0.1% nanocoke is optimal in terms of fatigue stability and lower model error. A nanocoke content of 0.5 wt.% may be suitable for applications subjected to high short-term loads; however, it is less suitable for long-term cyclic loading conditions, as confirmed by the experimental results. Bitumen modified with 0.5% nanocoke retains its structure better under large deformations but is inferior in strength is suitable for elastic operating conditions. Bitumen containing 1% nanocoke possesses an optimal balance between strength and resistance to failure and is the most promising for durable pavements and this content optimal in terms of creep resistance and stiffness. Bitumen modified with 1% nanocoke powder can be recommended for the construction of Class I and II highways in regions of Kazakhstan with a sharply continental climate. This will ensure increased shear stability and resistance to rutting under extreme summer temperatures and high axle load from heavy-duty vehicles.

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