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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

IMPROVEMENT OF OIL RHEOLOGICAL PARAMETERS WITH USE OF NANOPARTICLES AND NANOCOMPOSITES AND THEIR APPLICATION TO THE ROAD BITUMEN

Specialty: 2314.01 - Oil chemistry

Field of science: Chemistry

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GENERAL CHARACTERISTICS OF THE WORK

Actuality of the topic and the degree of processing. Interest in the extraction of the heavy oil in connection with reduction of the light oil reserves in the world continues to grow. One of the factors that classify oils as heavy oils is their high viscosity and high paraffin content. It is mainly heavy oil with a viscosity of 30 mPa·s and higher. According to preliminary estimates of specialists, the reserves of heavy oil is more than the reserve of light and low-viscous oils by about 1 trln tons. In the developed countries, such oil is considered not only as oil reserves, but also as a factor of the country's economic development in the coming years.

It is undeniable that the oil reserves in the oil fields of Azerbaijan also have a high viscosity and contain a lot of paraffin. A fraction of the oil extraction rich with heavy paraffin hydrocarbons with high melting temperatures and resinous-asphaltene substances is increased every year. In addition, in connection with acceleration of the oil extraction, the intensive search for new fields is also continuing. Such new oil fields are usually discovered on the borders of the Caspian Sea with other countries and at a sufficient depth. Since these oils possess high viscosity, their extraction, transportation, processing and use cause serious problems. The paraffin deposits are formed on the internal walls of the oil pipelines and working surface of the oil refining devices, which leads to an increase of the costs for elimination of these harmful factors and decrease of profitability of the oil fields.

Now, a search of the optimal methods for elimination of asphalt and paraffin deposits in the oil and decrease of its viscosity is carried out. In addition to the abovesaid one, the intensive investigations for solution of the complex problems using one of the latest achivements in science and technology – nanomaterials are being carried out. In sipte of the fact that the territory of the oil extraction in Azerbaijan is not very large, and in connection with oil extraction for many years, the supply of wells with oil pipelines meets the requirements, the extraction, transportation and use of high-paraffinic high-viscous oil creates great economic difficulties.

That is why, most of the investigations in the oil industry has been devoted to search of ways of increase of the oil fluidity and improve its rheological parameters.

On the other hand, the oils with high resin content have certain structural-mechanical properties in the layers, the viscosity and mobility of this oil depend on sliding stress and pressure gradient, and its filtration in porous media doesn’t subject to Newton and Darcy laws. During exploitation of the oil fields, the pressure along the surface of the oil layers varies widely, in a part of the layer a dynamic pressure gradient of the sliding pressure is low, the oil mobility is very small, and the viscosity is quite high. Due to the presence of asphalt-tar-paraffin compounds in the oil produced in existing oil fields, oil production from these wells is difficult. The additives, especially at low temperatures, including in wells create non-Newtonian properties in the oil. It should be noted that the presence of resin in the oil gives it elasticity, and the availability of paraffin – nonlinear viscosity. It should be remembered that due to the high oil fluidity in high-performance oil wells, the formation of paraffins and other deposits on the walls of lifting and bearing pipes is practically not observed.

For fight with formation and removal of asphalt-resinous paraffin deposits on the walls of the oil pipelines, first of all, the following initial parameters of the oil should be practically investigated: a quantity of hydrocarbons, fractional composition, a quantity of paraffin contained in the oil, its molecular weight, surface tension coefficient, viscosity, the regularities of their changes depending on external factors have been determined. From this point of view, an actuality of the topic of the dissertation devoted to the improvement and management of the rheological parameters of the paraffined oil with use of various technological methods and nanomaterials is undoubted.

Object and subject of the study:

The object of the study was samples of very paraffinic viscous oil extracted at the N. Narimanov NGLU. Nanoparticles of Al (60-80 nm), Cu, and Fe (50 nm) were used to study the effect of nanoparticles on the physicochemical properties and rheological parameters of paraffin oils. For the preparation of nanocomposites, the surfactants Alkan DE - 202, Dissolvan-4411, used as demulsifiers in the oil industry, and sulfonol, widely used in a number of technological operations, were taken. BND 50/70 bitumen was used in the studies. To improve the characteristics of bitumen, amines obtained from local raw materials were synthesized and applied in the form of nanocomposites.

Goals and purposes of the study:

Improvement of the rheological parameters of the paraffined oil with use of nanoparticles and nanocomposites and use of results in making of road bitumen.

For achievement of this purpose it is proposed to solve the following problems:

- Selection of the samples for investigation from various wells and reservoirs for oil storage;
- Development of methods of investigation of the rheological parameters of the oil products: density, viscosity and surface tension coefficients and adjustment of the corresponding devices;
- Selection of nanoparticles influencing on rheological parameters, making of nanocomposites;
- Carrying out of chromatographic and phase analysis of the oil composition taken from various wells and oil reservoirs;
- Investigation of the dependence of viscosity, density and surface tension coefficients of the oil samples on concentration, material and temperature of the metal nanoparticles;
- Investigation of the influence of concentration and types of surface-active substances on oil rheological parameters;
- Achievement of application of results of the investigations for development of road bitumen.

Research methods:

Fractional composition analysis, Perkin Elmer Perkin Elmer AutoSystem XL gas chromatograph, viscometers of different diameters for determining rheological parameters, pycnometers of different sizes for determining specific gravity, and Mi 806pH / TDS manufactured by Milwaukee Instruments for determining the pH of the system. - performed in accordance with the developed program using counters.

Basic positions taken out for defense:

− Peculiarities of character of change of the rheological parameters of the paraffined oil depending on concentration of the metal nanoparticles and nanocomposites;

− Influence of external factors on the paraffinization process of the oil products;

− Fractional analysis of the crude oil extracted from various paraffin wells;

− Dependence of the heat-physical parameters of the paraffined oil on concentration of nanoparticels and nanocomposites and influence of external factors;

− Determination of the optimal costs for nanoparticles and nanocomposites providing oil deparaffinization;

− Possibilities of application of the oil sludge in practice.

Scientific novelty of the study:

- Metal nanoparticles and surface-active substances for decrease of viscosity of the heavy oil with high viscosity containing asphalt-resionous-paraffin compounds for preparation of the optimal values of the rheological parameters has been developed;

− The fractionation analysis of highly viscous oil samples with various fields has been carried out, their distributive characteristics have been determined;

− The optimal values of the metal nanoparticles and surface-active substances – nanocomposites corresponding to decrease of density, viscosity, surface tension coefficient of the paraffined oil have been determined;

− The parameteras of activation of the paraffined oil have been determined, their variation characteristics depending on external factors and concentration of nanoparticels have bee determined.

Theoretical and practical significance of the study:

The possibilities of the industrial application of nanoparticles and nanocomposites for purification of the paraffined oil and method of preparation of road bitumen of high quality have been proposed. These results have been also used in LLC "Demirli Yol Tikinti" and in State Agency of automobile roads of Azerbaijan. The acts received on this occasion are stated in the dissertation.

Publications:

On a theme of the dissertation 15 scientific papers and reports at conferences, including 4 in influential foreign journals have been published.

Approbation of work:

The dissertation was discussed at the following conferences:

− VII Traditional International Scientific Conference "Ecology and protection of life activity", Sumgait State University, 7-8 May, 2012;

− VIII Traditional International Scientific Conference "Ecology and protection of life activity" dedicated to the “Year of Industry”, Sumgait State University, 3-4 December, 2014;

− III International Scientific Conference of young researchers dedicated to the 92nd anniversary of National Leader Heydar Aliyev, Baku, 17-18 April, 2015;

− XIX Republican Scientific Conference of doctoral students and young researchers, Azerbaijan State University of Economics, Baku, 7-8 April, 2015;

− I International Scientific Conference of young researchers dedicated to the 94th anniversary of National Leader Heydar Aliyev, Caucasus University (now Baku Engineering University), Baku, 18-19 April 2017;

− Breakthrough research: problems, laws, prospects. Collection of articles of the XIV International Scientific and Practical Conference, Penza, January 27, 2020.

It was discussed at scientific seminars of the chemical and biological faculty of Sumgait State University.

The name of the organization in which the dissertation work was performed: The dissertation work was performed at the Department of" Petro chemistry" of Sumgayit State University.

Personal participation of the author:

the main goal of the study and the tasks set by the author to achieve them are indicated, the research directions are defined, the results are processed, systematized and discussed. The author was also directly involved in the preparation and conduct of laboratory and experimental studies.

Structure and scope of the dissertation:

The dissertation consists of 162 pages, including an introduction, 4 chapters, main results, 21 illustrations, 32 tables and a list of literature from 160 titles used. The dissertation consists of 210307 characters without tables, figures and bibliography.

Work content: In the introduction the actuality of the dissertation theme has been substantiated, the purpose of work, its scientific novelty, practical significance and key questions of defense have been determined. It also presented a brief review of existing theoretical and practical works on the questions considered in the dissertation.

In I chapter of the dissertation the analysis of research works devoted to the oil paraffinization and methods of its elimination has been carried out. It has been also reflected the chemical composition and nature of paraffin deposits, analysis of the current situation in the oil pipelines and installations, information about some methods of fight with paraffin deposits, including chemical methods.

In II chapter of the dissertation the technology of carrying out of the chromatographic analysis of the paraffined oil is described, information about reasons of change of viscosity during extraction, transportation and storage of oil is presented, and also a method used for investigation of the oil surface tension coefficient is described. A use of the metal nanoparticles Fe, Cu and Al in the purification of paraffin oils is not accidental in this work. These substances can maintain a powdery state even in aqueous media, are economically profitable, possess high reserves, and a level of the investigations of new types of nanocomposites based on them is very low.

A density of the copper nanoparticles used in the investigation is 8,94 g/cm^3, melting temperature – 1083°C, boiling temperature – 2517°C, and molar mass – 63,5 g/mol.

A density of the iron nanoparticles used in the investigation is 7,86 g/cm^3, melting temperature – 1535°C, boiling temperature – 2750°C, molar mass – 55,85 g/mol. The iron nanoparticles have been obtained by sol-gel method of colloid chemistry or by precipitation from vapor phase. A density of the aluminum is 2,70 g/cm^3, melting temperature – 660,32°C, boiling temperature – 2319°C, molar mass – 26,98 g/mol.

Chapter III, summarizing the main experimental results of the dissertation, includes an analysis of the fractional composition of the oil samples taken from various oil wells and oil reservoirs, as well as determination of the total quantity of paraffins in the oil composition and their boiling temperatures.

The fractional analysis of the oil sample taken from commodity reservoir showed that the largest quantity of paraffin is observed in C_8 fraction, the percentage of paraffin in C_{15}-C_{19} fractions is vibrated in the range of 3.05-3.35. The temperature dependence of mass fraction of the oil fractions taken from commodity reservoir has been investigated and it has been established that with increase of the molecular weight of the fractions to C_{41}, the boiling temperatures are also naturally increased to 522°C.

On the basis of results of the chromatographic analysis a dependence of mass fraction and boiling temperatures of the fraction on content of hydrocarbons in the samples of the paraffined oil taken from wells 429 and 411 (OGPD named after N. Narimanov) shows that as well as in the samples taken form oil reservoirs, a maximum quantity of C_8 fraction in this oil is equal to 3,55%. It has been determined that the boiling temperatures for C_5-C_{40} fractions vary between 36-522°C. The average boiling temperature was 343°C [6,12].

As it was known, the oil density is one of the most important parameters characterizing the quality of the crude oil and it is strongly depends on the quantity of resins, asphaltenes and paraffins in its composition, temperature, quantity of dissolved gases and pressure. Consequently, for extraction, transportation, processing and, in the end, the oil sale, the oil density must be in accordance with certain standards of. For this reason, the change of the oil density and surface tension coefficient depending on temperature has been investigated. The investigations were carried out in the interval of temperatures 17-67°C, where surface tension coefficient decreased from 29,68 mN/m to 25,4 mN/m. Unlike surface tension coefficient, the relatively weak decrease of density in the investigated temperature range observed.

The similar investigations have been also carried out on the oil samples subjected to the action of the iron nanoparticles with size 50 nm [8]. It has been detected that with increase of concentration of the iron nanparticles from 0 to 0,001% the surface tension coefficient decreased sharply from 29,67 mN/m to 26,4 mH/m, the subsequent increase of concentration to 0,01% didn’t practically influence on surface tension. This means that the surface tension coefficient of the oil sample gets the minimum value at low limits of the iron nanoparticles – at value 0,001%.

It has been detected that the surface tension coefficient of the paraffined oil taken from tested well, before action of the iron nanoparticles with temperature rise decreased from 29,7 mN/m to 26,3 mN/m, after treatment of the oil with iron nanoparticles a decrease is 25,5 mN/m to 25,4 mN/m. It should be noted that in the investigated interval of temperatures it is observed a decrease of value of the oil surface tension coefficient, subjected to action of the iron nanoparticles.

No anomalies were observed depending of the oil density on concentration of the iron nanoparticles, with increase of the concentration of nanoparticles in the oil composition to 0,005%, its density increased from 861 kg/m^3 to 871 kg/m^3, and with increase of this concentration to 0,01% decreased to 867 kg/m^3.

The influence of surface-active substances with various composition and in different quantities on rheological parameters of the paraffined oil samples has been also investigated. It has been detected that the surface tension coefficient is sharply decreased with increase of a quantity of Alkanes (Alkane-202 reagent) in the sample composition to 0,0025% of the concentration of nanoparticles and then remains constant independently of a quantity of Alkanes. The surface tension coefficient shows the minimum value in a quantity of Alkanes corresponding to 0,0025%.

With increase of a quantity of Alkanes in the sample composition to 0,001%, the oil density is slightly increased and then remains stable independently of the subsequent increase of a quantity of Alkanes. The dependence of the surface tension coefficient and density on the additives concentration in addition of other surface-active substances – sulfonol and dissolvan (Dissolvan-4411) to the paraffined oil has been investigated. The analysis of the results showed that the characters of changes of the surface tension coefficient and density depending on the quantity of additives are the same.

A viscosity is one of the most sensitive oil parameters and strongly depends on influence of the external factors. Taking into account this, the study of the dynamic and kinematic viscosity coefficients of the paraffin oil has a scientific and practical importance, as well as the study of changes of these coefficients in the paraffin oils depending on the temperature and temperature of the nanoparticles.

The dependence of surface tension coefficient and density on additives concentration in addition of other surface-active substances – sulfonol and dissolvan to the paraffined oil has been also investigated. The analysis of the results showed that the characters of changes of both the surface tension coefficient and the density depending on the quantity of additives are the same.

A viscosity is one of the most sensitive oil parameters and strongly depends on influence of the external factors. Taking into account this, the study of the dynamic and kinematic viscosity coefficients of the paraffined oil has a scientific and practical importance, and also it is important to investigate the changes of these coefficients depending on the concentration of nanoparticles in the paraffined oil and temperature [3, 11].

The results of change of the viscosity coefficients depending on concentration of the iron nanoparticles with size of 50 nm and temperature are presnted in Fig. 1.

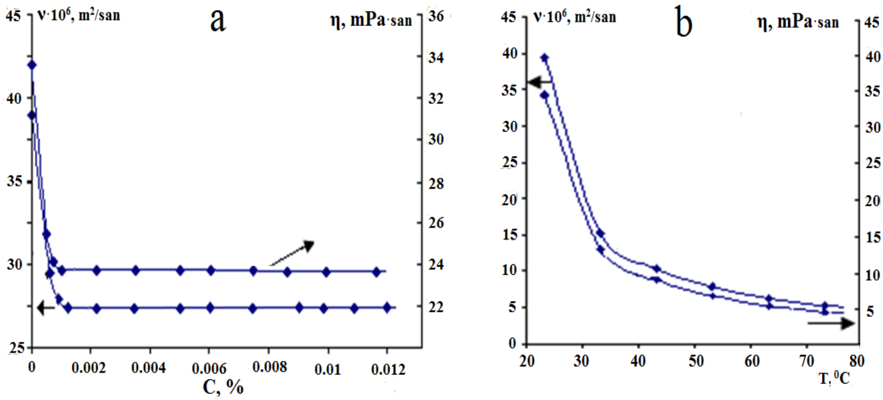

Figure 1. Temperature dependence of kinematic (ν) and dynamic (η) viscosity coefficients before (a) and after (b) nanoaction

As can be seen from Fig.1a, with increase of a quantity of the nanoparticles in the mixture to 0,001, the kinematic viscosity is sharply decreased from 38,5 to 27,5, then with subsequent increase of a quantity of Alkane addition this index is not practically changed.

The dependence of the dynamic viscosity coefficent (η) on Alkane concentration is also shown in Fig.1a. One can see that a sharp decrease η is observed with increase of concentration to 0,001, with subsequent increase of concentration of the nanoparticles to 0,01 the dynamic viscosity coefficent remains constant. We'll note that the results of the investigation of dependence of the viscosity coefficients of the paraffined oil sample on the temperature showed that with temperature rise from room temperature to 27°C, the kinematic viscosity coefficient decreased sharply from $40 \cdot 10^{-6}$ m^2/sec to $15 \cdot 10^{-6}$ m^2/sec, then with temperature rise to 67°C, it is decreased little, to $5 \cdot 10^{-6}$ m^2/sec. The temperature dependence of the kinematic viscosity coefficient was also similar.

It has been investigated the viscosity indicators of the paraffined oil products in addition of Alkanes to them, the results have been shown in Fig. 2.

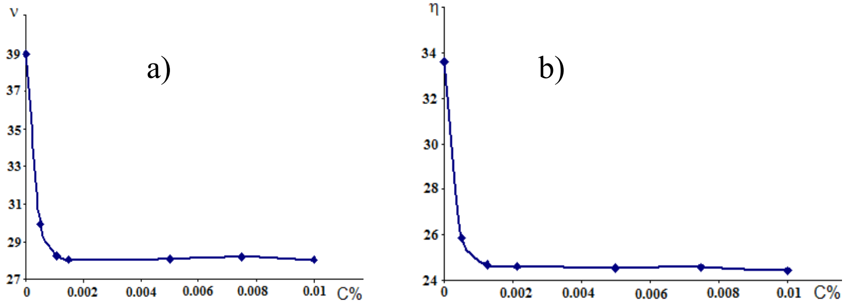

Figure 2. Dependence of kinematic (a) and dynamic (b) viscosity coefficients of the mixture paraffined oil+alkane on alkane concentration

It has been established that when a content of Alkane additions in the oil reaches 0,00075%, the kinematic viscosity coefficient is decreased from $38{,}94 \cdot 10^{6}$ m^2/sec to $29{,}02 \cdot 10^{-6}$ m^2/sec, and the subsequent increase of the Alkane concentration practically doesn't influence on change of the viscosity. The kinematic viscosity coefficient in these Alkane concentrations decreased from 33,58 mPA·sec to 25,04 mPA·sec.

The dependence of the viscosity coefficient on concentration of additives – dissolvan and sulfonol has been investigated and it has been detected that with increase of concentration to 0,00075% the kinematic viscosity coefficient is decreased from $38{,}94 \cdot 10^{-6}$ m^2/sec to $29{,}12 \cdot 10^{-6}$ m^2/sec, and the kinematic viscosity coefficient is decreased from 33,64 mPA·sec до 25,16 mPA·sec. The subsequent increase of concentration has a little effect on change of the viscosity coefficients.

It was known, the paraffined oils have a high viscosity. To explain this, the mixture was considered as a colloidal system (Fig.

3), and it turned out that at a low paraffin concentration the colloidal particles in the oil composition are located at a great distance from each other, as a result of which they interact weakly and the viscosity of such system is low [7].

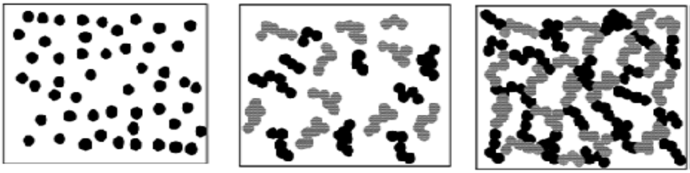

Figure 3. Various microstructures existing in the system depending on particles concentration of a colloidal system

IV chapter of the dissertation has been devoted to the study of possibility of use of the oil residues in practical matters. In the first paragraph of the chapter, the influence of polymers and surface-active substances (surfactants) on properties of viscous bitumens has been investigated.

As is known, the polymers technically used for making of bituminous-polymer composites must meet the following requirements:

− must be not decomposed under the conditions of making of the asphalt concrete mixture;

− in the making of the asphalt concrete mixtures on equipment and under temperature conditions corresponding to traditional technologies must be mixed with bitumen;

− in hot weather, should increase the resistance of bitumen in the road pavement to displacement from voltage, and at low temperatures to increase its hardness;

− the chemical and physical parameters must be stable, retain their inherent properties for a long time, and not lose quality when used.

The various additives and technologies were used to solve this problem.

The surface-active substance (SAS) obtained as a result of the reaction of naphthenic acid and triethanols at 160-180°C was taken as I additive. SAS obtained in a toluene medium consists of a salt of naphthenic acid. This SAS is a viscous liquid of brown color with acid number 5-9 mg KOH/g, a water content 0.1-0.2% and low specificity [13].

With the aim of addition of the polymer to the bitumen and ensure its complete distribution, a polyethylene solution has been prepared in an organic solvent (II additive). Cyclohexane was used as a solvent. In the solvents, polyethylene swells and dissolves in the temperature range from 70 to 80°C. In making of a bituminous polymer mixture, the components are mixed for 1 h at temperature higher the softening temperature of the bitumen by 10°C, then the temperature is gradually increased to 100-120°C, intensively mixed and cooled. The content of polyethylene in the made bituminous-polymer mixture is 3.0-5.0%.

The other sample of bituminous-polymer mixture has been made with use of oligomers obtained as a side product during production of polyethylene (III additive). An oligomer at room temperature is a mass similar to soft paraffin, a part of it is liquid, and for this reason its addition to the bitumen is simplified. As in the other samples, the oligomer is added to the bitumen in a quantity of 3-5%. Besides oligomer, SAS, polyester resin and phosphoric acid are added to the bitumen. In the mixture, the components on the basis of oligomers were taken in the following mass ratio: Polyethylene oligomer : polyester resin: phosphoric acid: SAS=1:0,4:0,05:0,2.

Here, the mixture consisting of amides of fatty acids and imidazoline, synthesized at 180°C on the basis of fatty acids and polyethylene polyamine has been used as a surface-active substances. As IV additive, the composition obtained as a result of treatment of this mixture of amide and imidazoline compounds with sulfur powder 70-75°C has bee used.

V additive has been made as a composition consisting of mixture of polyethylene and IV additive.

The influence of prepared additives on viscous road bitumen has been investigated in the laboratory. The softening temperatures on method "ring and ball", extensibility, depths of the needle penetration, adhesiveness with rock material (adhesion) and penetration index for samples of the tested bitumen and samples with additives have been calculated. The reagents were added to the bitumen in a quantity of 0,5-3,0%. The results of the investigations are presented in Table 1. As can be seen from Table, the softening temperatures of the tested bitumen and bitumen samples with additives on the basis of surface-active substances meet the requirements to viscous road bitumen BNB 50/70 according to **TŞ** AZ 3536601.242-2015. A comparison of the obtained results shows that depending composition on samples and their quantity it is observed a temperature rise to +2,6°C in comparison with indices of the tested bitumen. On the contrary, an addition of the composition of surface-active substances with polymer materials to the bitumen favors the decrease of the softening temperature of the mixture. An addition of 1,5-3,0% of the polymer composition-SAS to the bitumen decreases the softening temperature of the bitumen by 5°C in comparison with tested sample. As can be seen from the results, I additive at concentration 0,5% favors the increase of depth of the needle penetration. Since the addition of 0,5% of I additive to the bitumen favored the increase of depth of the needle penetration in comparison with 65-70-75 (0,1 mm) observed in the tested sample and achievement of values 90-95-98, respectively.

The subsequent increase of a quantity of the additive stabilized the depth of the needle penetration in the bitumen on a mark of 60-65-70 (0,1 mm). After introduction of 35,0% of the solution of high-pressure polyethylene in cyclohexane (II additive) in a quantity of 3,0%, the depth of the needle penetration increased to 85-92 (0,1 mm). The presence of phosphoric acid in addition to the polymer in III additive favored the concentration of the bitumen and decrease of the depth of the needle penetration. Since, in this case, the depth of the needle penetration was 63-64 (0,1 mm).

Table 1.

Results of the investigation of influence of additives from composites of surface-active substances and polymers on viscous road bitumen

Name of indices	Requirements to the Road bitu-men BNB 50/70 according to TŞ AZ 3536601.242-2015	Test bitumen	A quantity of additives, %								
			I (SAS on the basis of naphthenic acids)			II HPPE/ cyclohexane)	III (oligomer/ L3603/ H_3PO_4 /SAS	IV SAS (fatty acids+PEPA)+S_8		V(polymer SAS-Fatty acids+ PEPA+S_8)	
			0.5	0.75	1.0	3.0	3.0	0.5	1.0	1.5	3.0
Softening tempera-ture on method “ring”and “ball”,°C	49-54	49.6-50.0	50.1;50.3	49.6; 49.8	50.4;50.9	44.9; 45.0	46.0; 46.7	50.7; 51.2	52.5; 52.6	46.0; 46.3	45.1; 45.3
Tension indicator, at 25°C, cm	>55	90-96	100-100	95-100	60-62	45-47	52 -56	71-72	70-71	53-62	51-60
Depth of the needle penetra-tion, 0,1 mm, at 25°C, mm	51-70	65-75	90-98	60-70	60-70	84-92	63-64	-	-	95-96	94-95
Adhesi-veness with rock material	II	III	II	II	II	III	III	II	II	III	III
Penetra-tion index	-0.2 ÷-0.6	-0.2÷-0.6	0.3 ÷ 0.7	-0.4 ÷-0.8	-0.4 ÷-1.0	-1.3÷-1.04	- 0.4 ÷-1.0	-	-	-0.63÷-0.51	-0.95 ÷ - 0.85

I – excellent, II – good, III – unsatisfactory

In the mixture of toluene and tsiclohexane composition obtained as a result of the combined treatment of surfactants based on fatty acids and polyethylene polyamine gray, an increase in the depth of the needle compared to the test sample caused 1.5 and 3.0. % bitumen.

In the test sample, this value is 65-70-75 (0.1 mm), and the needle penetration depth from the addition of V is 94-96 (0.1 mm).

The influence of reagents on extensibility of the bitumen samples has been investigated in ductilometer at 25°C in accordance with GOST 11505 – 75. The obtained results show that an extensibility index of the samples with concentarions of I additive 0,5 and 0,75% is higher than the corresponding tested sample values. In other cases an addition of the reagents to the bitumen negatively influences on change of extensibility and an elasticity of the samples with polymer additives is lower that in the tested sample.

One of the key indices of the bitumen is its adhesion to the rock materials. This index determines the durability of asphalt-concrete pavement, as well as the road pavement during its exploitation. As a result of the study of additives, it has been established that the adhesion of bitumen to the rock material is increased in all investigated concentrations of I and IV (SAS) additives, but the addition of the polymer composites decreases this index of bitumen. On the basis of the results of the investigation, the adhesion of bitumen samples with I and IV additives to the rock material has been well evaluated.

Thus, the results show that the surface-active substances synthesized on the basis amines – amides of oirganic acids and esters in addition to the viscous road bitumen in concentartion 0,5-1,0% increase its adhesion to rock materials and also elasticity.

An addition of modifiers and stabilizers to the bitumen during the asphalt manufacturing process reduces the freezing temperature, the decrease of the bitumen viscosity and, as a consequence, uniform distribution of bitumen on the surface of solid fillers, prevents the "aging" process of the asphalt at the first stage.

The influence of amine compounds of the natural organic acids as adhesion additives to the bitumen for improvement of quality of road asphalt-concrete pavements has been investigated [10].

It was known that during oil processing in the fractions of ligroin, kerosene and gasoil there are technical petroleum acids C_{10}-C_{14}. At the synthesis of amine compounds of naphthenic acids, the oil acids obtained in the purification of the kerosene fraction and having an acid number 268.7 mg KOH/g were used. A part of the substance (I reagent), obtained as a result of the reaction with technical petroleum acid, was treated with 37% formaldehyde solution at temperature 5-10°C and formed the group –N–CH2OH (III reagent) in a moleule.

The sulfur powder in a quantity 8% forming an emulsion in 80% of water (IV reagent) was added to the part of the reagent obtained on the basis of fatty acids. The sulfur powder has the following granulometric composition:

Particles with size up to 6 mcm	-	54 - 70%
Particles with size up to 13 mcm	-	16 - 44%

The sulfur powder contains 0,05% of SAS – synthanol DC-10.

By addition of the prepared reagents to the viscous road bitumen in a quantity of 0,5-1.5% at 130-140°C with stirring for 20-30 minutes, the bituminous compositions were made and their softening temperatures on method "ring and ball" and extensibility were determined. The adhesiveness of the bitumen samples with additives and tested bitumen with rock material was determined by a method of "passive" adhesion. For this purpose, the bitumen samples were soaked at 130-140°C into a rock material with size of 2-5 mm, then held in boiling distilled water on a metal grid for 30 minutes, after extraction from boiling water, the samples were kept in cold water for 2-3 minutes and the adhesiveness of the bitumen with rock material in comparison with the tested sample was investigated. The results of bituminous compositions obtained at various concentrations of the synthesized reagents are presented in Table 2. It has been detected that I, III and IV reagents in the investigated concentrations have a positive effect on the adhesion of bitumen to the rock material and increase it from "satisfactory" to "good”.

It has been established that although II additive in a quantity 1,0% has a good effect, its subsequent increase (1,5%) favors the weakening of adhesion, and at the same time increases the softening temperature of the bitumen at concentrations 0,5-1,0%. This index at concentration 1.0-1.5% of I reagent is 0,6-2,6°C, at concentration 1,0% of II reagent – 1,6-2,4°C and at concentration 0.5-1.0% of IV reagent –0,7-2,6°C. At concentrations 0.5-1.0% of III reagent the softening temperature of the bitumen corresponded to index of the tested sample. The study of the sample extensibility of modified bitumen on ductilometer in accordance with GOST-11505-75 showed that due to influence of additives at 25°C their extensibility is in the level of requirements to road bitumen (Table 2).

Table 2

Results of the investigation of bitumen composites

Reagent	Reagent concentration, %	Indices of bitumen		
		Softening temperature on method "ring and ball", °C	Tension indicator, at 25°C, cm	Adhesiveness with rock material
Requirements to road bitumen BNB50/70 in accordance with TŞ AZ 3536601.242-2015	-	49 – 54	>55	II
Tested bitumen	-	49.6 - 50	90 - 96	III
I	1.0	50.5-50.6	77-78	II
	1.5	51.8-52.6	61-62	II
II	0.5	48.9-49.1	-	III
	1.0	51.6-52.4	48-50	II
	1.5	48.8-49.9	-	III
III	0.5	46.8-47.8	72-80	II
	1.0	49.7-49.9	60-72	II
	1.5	50.1-50.7	40-44	II
IV	0.5	50.7-51.2	71-72	II
	1.0	52.5-52.6	70-71	II

I – excellent,   II – good,   III – unsatisfactory

Reagents I and IV proved to be more effective based on the characteristics of the modified bitumen samples. Taking into account the fact that the reagents are synthesized on the basis of the oil acids having local production base and fatty acids obtained during the production of the cotton oil, there are perspectives for their further use in the asphalt-concrete production.

The quality of asphalt coatings can be improved by adding a number of modifiers. Polymeric additives affect the asphalt's penetration ability, softening temperature, strength, viscosity and heat resistance. At the same time, asphalt coatings sometimes begin to collapse sooner than expected. The collapse of the pavement is usually "sweating" of bitumen, softening of asphalt in hot weather, formation of cracks, and grinding of particles. Therefore, improving the quality of asphalt compositions is always on the agenda.

A new direction in this area is the use of the achievements of nanotechnology. Research has shown that 60-80 nm Al nanoparticles increase the performance of SAS, affect oil viscosity, accumulation intensities of paraffin and salt, but also show high activity in very small concentrations [18].

In addition, the surface tension of surfactants with Al nanoparticles in a wide range of composite concentrations in aqueous solutions of sulfonol was studied (Table 3).

Table 3

Influence of Al-nanowire particles (60-80 nm) on the surface tension of SAS solutions

Density of nanoscale particles in solution,% mass	Surface tension of Sulfonol + Al-nanoparticles, 10^{-3}N / m	Reduction of surface tension,%
-	18.5	-
0.0005	7.5	59.5
0.001	5.7	69.2
0.005	6.5	64.9
0.01	7.3	60.5

The results show that in the studied compounds, the Al nan nanoparticles reduce the surface tension of the aqueous solutions of SAS to 70.0%. However, the amount of nanoparticles in the SAM solution is more than 0.001%, which reduces the effectiveness of nanoparticles to the surface tension of the studied solutions.

It is known that the surface tension is due to the excess of free energy on the surface that affects the molecules within the bipolar environment at the expense of non-compensating molecular shear forces. When nanoparticles interact at the molecular level with SAM on the surface, it appears that the effect of uncontrolled molecular forces on the SAM molecules is reduced, thus reducing the free energy surplus on the surface, thus reducing the surface tension.

The study of the effect of 0.001% Al nanoparticles (60-80 nm) on the surface tension of sulfonol solutions with different solids (0.004-0.032% mass) showed that the surface tension increased as the sulfonol content increased in the investigated compounds 9.5 · 10-3N / m · By 10-3 N / m, in other words, the surface tension of the sulfonol solutions with the corresponding solubility without nanostructures is reduced by 69.2 - 80.9% (Table 4).

Table 4

SAS - Solution of Al-nanohouse particles (60-80 nm)
Effect of Sulfonol on the surface tension

Density,% mass		Surface tension, 10^{-3} N / m		Reduction of surface tension,%
Sulfonol	Al Nano (60-80 nm)	Sulfonol	Sulfonol + Al-Nano	
0.002	0.001	40.6	12.0	70.4
0.004	0.001	31.4	9.5	69.7
0.008	0.001	18.4	5.7	69.2
0.016	0.001	16.6	4.0	75.9
0.032	0.001	14.7	2.8	80.9

At the same time, research has shown that when 0.5 to 1.0% of the bitumen is added to the oil-soluble non-ionic surface-active substance, its physical and chemical parameters - solidification, adhesion to solid surfaces are improved, while other indicators for road closure are improved. Answer. In this regard, the combined effects of surface-active matter and Al-nanoparticles on the performance of bitumen were studied. BNB 50/70 bitumen was used in the research. The results obtained are given in Table 5 and 6.

Table 5

Test results of bitumen with SAS (1.0%) and Al nanoparticles (0.05%)

№	The name of indicators	Requirements for road bitumen BNB 50/70 according to TC AZ 3536601.242-2015	Indicators of test bitumen sample	Actual indicators of bitumen sample with SAS	Actual indicators of bitu-men sa-mple with SAS + Al-nano-particles
1	The softening temperature according to the globe and hoop, ° C	49 – 54	50	48	49.5
2	Depth of penetration of needle (penetration), 25ºC, 0.1 mm	51 – 70	90	70	55
3	Extraction at 25ºC, cm	>55	100	85	75
4	Sticking to shattered	II	III	II	II

I - excellent, II - good, III - unsatisfactory

Table 6

Test results of bitumen with SAS (1.0%) and Al nanoparticles (0.1%)

№	The name of indicators	Requirements for road bitumen BNB 50/70 according to TC AZ 3536601.242-2015	Indicators of test bitumen sample	Actual indicators of bitumen sample with SAS	Actual indicators of bitumen sample with SAS + Al-nano-particles
1	The softening temperature according to the globe and hoop, ° C	49 – 54	50	49	50
2	Depth of penetration of needle (penetration), 25ºC, 0.1 mm	51 – 70	90	70	65
3	Tension indicator, at 25ºC, cm	>55	100	85	95
4	Sticking to shattered	II	III	II	II

I - excellent, II - good, III - unsatisfactory

The results show that the addition of 0.05% Al-nanoparticles to the bitumen-SAS composition caused the change in performance compared to the test sample. Thus, stretching index of the bitumen sample extracted by the additives was reduced to 39–41 cm at 25°C and the depth of the injection of 0.1 mm needle was 55–60 and the softening temperature was 49.0 - 49.5°C for method “spine and ring”. At the same time, there has been an improvement in the adhesion of the bitumen - the improved adhesion of bitumen with the test sample and additives.

Increase in aluminum metal nanoparticles by 0.1% was mainly influenced by bitumen sample extraction. As the amount of al-nanowire particles increased from 0.05% to 0.1%, the bitumen extraction at 25°C increased from 39-41 cm to 65-74 cm. Other indicators, particularly the adhesion of the bitumen, were observed to be better than the test sample. It should be noted that SAS improved adhesion to the bitumen samples compared to the test sample.

Different surfactants are used to enhance the adhesion of the asphalt. In addition, asphalt is added to the bitumen, which is dried in powder to minimize the effects of water. Although this may solve the problem of water effects in the use of a number of traditional asphalt mixtures, most commonly made asphalt mixtures do not accept these supplements. In this case, additives that modify the rheology of the asphalt - soluble and insoluble additives are used.

Different clays, cellulose fibers, silicon 4-oxide, and others, such as insoluble in bitumen and distributed in its volume, regulate the rheology of low-soluble olufaltin. substances are used. The range of rheological modifiers in asphalt is broader. These types of supplements include resins of different origin, fatty acids and their soaps, saturated alcohols, hydrogenated fats, natural resin and other substances. These substances form homogeneous mixtures as a solution of asphalt at hot mixture temperatures, providing the strength of the incident. Fatty acids and technical oil acid salt - aluminum technical oil acid as a soluble component in asphalt - were prepared and added to bitumen.

The fatty acids were prepared by the treatment of sodium salts - soapstock at 85 - 90°C with the purification of cotton oil. The acids obtained were mainly composed of unsaturated acids - linolen (43.0%) and olein (31.0%). In addition, it contains palmitin (24.0%) and stearin (2.0%) acids.

Al-naphtenat was developed on the basis of sodium naphthenates ("alkaline waste") obtained during purification of fuel fractions from the naphtha acid at the H.Aliyev refinery. The process is easy and is done by mixing alkaline salts with alkaline solution under normal conditions. The obtained Al-naphtenate collapses due to insolubility in water and is purified by filtration.

As can be seen from the table, the addition of Al-naphenate to the system significantly affected the performance of bitumen. Thus, the softening temperature of the bitumen sample with a 3.0% Al-naphthaene contribution decreased from 50-550C to 46.8-46.9°C, while there was a decrease in penetration and tension. Despite the fact that the softening temperature has been reduced, the depth of injection of the needle does not indicate that the bitumen itself has increased due to the contribution. Changes in the performance of bitumen are more pronounced during the subsequent increase in Al-naphthaene.

The results show that the addition of 0.05% aluminum nanoparticles to the bitumen-SAS composition caused a change in performance compared to the test sample. Thus, the bitumen sample extraction was reduced to 39-41 cm, with a 0.1 mm needle penetration depth of 55-60 at 25^0C and a softening temperature of 49.0 - 49.5^0C for "spine and ring".

It has been revealed as a result of the investigations that in addition of 0,5-1,0% of nonionic surface-active substances soluble in the oil to bitumen, its physical-chemical properties – extensibility and adhesiveness with solid surface are improved and simultaneously the other indices meet the requirements for road bitumen. In this regard, the combined influence of surface-active substance and Al nanoparticles on chracteristics of the bitumen has been studied. BNB 50/70 bitumen was used in the investigations (Table 7).

The obtained results show that an addition of 0,05% of Al nanoparticles to composition of the bitumen-SAS led to the change of indices in comparison with tested sample. So, an extensibility of the bitumen sample with additives decreased to 39-41 cm, depth of the needle penetration 0,1 mm at 25°C decreased to 55-60, and the softening temperature on “ring and ball” to 49,0 – 49,5°C.

Table 7

Test results of the bitumen with addition of SAS (1.0%) and Al nanoparticles (0.05% and 0.1%)

№	Name of indices	Requirements to road bitumen BNB50/70 in accordance with TŞ AZ 3536601.242-2015	Indices of the sample of tested bitumen	Factual indices of the sample of bitumen with SAS	Factual indices of the sample of bitumen with SAS + Al nanoparticles
1	Softening temperature on method "ring and ball", °C	49 – 54	50 – 55	48 - 49	49.0-49.5
2	Depth of the needle penetration into bitumen, 25°C, 0.1 mm	51 – 70	85 - 90	60 - 75	55 – 60
3	Tension indicator, at 25°C, cm	>55	100	85 – 86	74 -78
4	Adhesiveness with crushed stone	II	III	II	II

I – well, II – good, III – unsatisfactory

At the same time, it was not observed the improvement of the bitumen adhesiveness with crushed stone – the bitumen adhesion of the tested sample and with additives was the same. An increase of a content of the aluminum nanoparticles to 0,1% mainly influenced on extensibility of the bitumen sample. With increase of a quantity of Al nanoparticles from 0,05% to 0,1%, the bitumen extensibility at 25°C increased from 39-41 cm to 65-74 cm. The other indices and especially the bitumen adhesiveness with crushed stone improved. It should be noted that in the bitumen samples with addition of SAS, the bitumen adhesiveness with crushed stone also improved in comparison with the tested sample.

BASIC RESULTS

1. For the first time, the chromatographic fractional analysis of the crude oil samples taken from various fields (wells) has been carried out [7,14].
2. The dependence of the density, surface tension coefficient, dynamic and kinematic viscosity coefficients of paraffinized petroleum products on the density of iron and copper nanoparticles with an average diameter of ~ 50 nm was studied, the number of nanoparticles corresponds to the optimal values of these parameters were determined [3,6,7,8,9,11,12,14,16].
3. After exposure to iron nanoparticles (with average particle diameter ~ 50 nm) on paraffin oil samples taken from the commercial reservoir of OGPD named after N. Narimanov and various wells, the surface tension values (10-11)%, the kinematic and dynamic viscosity coefficients (37.2-41.6)%), after exposure to copper nanoparticles (average diameter ~ 50 nm), the surface tension of these oil samples decreases by 5.6%, the kinematic and dynamic viscosity coefficients decrease by 18%, and the densities practically do not change. [3,8,11].
4. It was found that it is possible to prevent the formation of paraffin deposits at low oil temperatures by using iron and copper nanoparticles with an average diameter of ~ 50 nm. [8,9].
5. The method of the oil deparaffinizing with use of nanocomposites containing surface-active substabces and metal nanoparticles has been developed. It has been detected that almost unchanged density of the oil samples under the influence of nanocomposites consisting of a combination of 0,0011% of the mass ratio of Alkanes and 0,00075% of the mass ratio of the iron nanoparticles (average particles diameter ~50 nm), a decrease of the surface tension coefficient by 11%, and the kinematic and dynamic viscosity coefficients by 25,5% allow to assume that the nanocomposites made from combination of Alkanes and the iron nanoparticles with size of 50 nm have the highest efficiency [7,14].

6. Based on the experimental values of the rheological parameters of oil, changes in its thermophysical parameters - entropy, enthalpy, and Gibbs energy-are studied, and the influence of external factors is studied [12].
7. It has been establihed that a role of the paraffin component in the formation of the asphalt-resin-paraffin deposits in the oil systems is higher than a role of the resins and asphaltene. The resin decelerates the crystallization and paraffin formation in the carrier systems, while the asphaltenes play a role of crystallization centers [10].
8. It was found that it is possible to regulate and improve the properties of high-viscosity bitumens used in road surfaces by using a mixture of surfactants of different composition and amino compounds of organic acids [10,13].

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