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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

BIOCOMPONENT BASED EMULSIFIED DIESEL FUEL RESEARCH AND PRODUCTION

Speciality: 2314.01 – Petroleum chemistry

Field of science: Technical

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The work was performed at the «Renewable Fuels» Laboratory of the Institute of Petrochemical Processes named after Y.H. Mammadaliyev of the Ministry of Science and Education of the Republic of Azerbaijan

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GENERAL DESCRIPTION OF WORK

The relevance of the theme and the degree of development. Nowadays irreversible changes in the environment are the main focus of world's scientists, and various measures are being implemented in developed countries to prevent these negative changes. So, according to research, one of the main roles in environmental pollution is played by vehicle emissions. For this reason, modern oil refining in the world's leading countries is developing in the direction of the production and use of more environmentally friendly engine fuels. The work carried out in this area is focused on the research of alternative energy sources with the aim of reducing the dependence on mineral resources and minimizing the impact on the environment.

The advantage of alternative energy sources, including biofuels that is widely used in transportation, is theirs regeneration and environmental safety. Since they are obtained from renewable materials, they do not contain sulfur and aromatic hydrocarbons, and therefore their combustion products are safer than mineral fuels. More ever they decompose safely within 2 months when they fall into the environment. In addition, biofuels are neutral as a source of greenhouse gases, because the amount of carbon dioxide released into the atmosphere during their production and consumption is equal to the amount of CO_2 gas absorbed from the atmosphere by plants. An example of this is bioethanol as a free fuel type, as well as an additive to gasoline, which today has already led to a reduction of 8 million tons of greenhouse gases (CO_2 equivalent) per year, which is equivalent to the annual emissions of 1.21 million cars.

However, monoatomic alcohols (methanol, ethanol, etc.), which are widely used as additives to gasoline fuels, were also desired as oxygen additives for diesel fuels. On the other hand, the presence of an oxygen atom in an alcohol molecule reduces the amount of harmful substances in the exhaust gases derived from internal combustion engines. Thus, compared to mineral diesel fuels, alcohols with a high vaporization temperature (the vaporization temperature of ethanol is 870 kJ/kg, while the vaporization temperature of petroleum-based diesel fuel is 230-250 kJ/kg) reduces the maximum temperature of

combustion and reduces the amount of nitrogen oxides released into the atmosphere.

One of the factors that improve the composition of flue gases formed during combustion is the low boiling point of the alcohols. Thus, the low boiling point ensures rapid evaporation of alcohol from the fuel mixture and creates additional turbulence in the mixture of petroleum-based diesel fuel with the used alcohol. This effect, noted for a wide range of emulsion fuels, also improves engine economic performance. Depending on the nature of alcohols and the amount of water they contain, they form a completely transparent homogeneous solution or emulsion with mineral diesel fuel. Additionally, the use of different emulsifiers is important to ensure the stability of the emulsified fuel obtained with alcohols, and in this case, the indicators of the toxic waste gases and fuel consumption for obtained ternary mixtures should be reviewed.

Taking into account the above, the synthesis of emulsifiers that ensure the stability period of emulsion diesel fuels, the study of the quality indicators of obtained emulsified diesel fuels, their use and the composition of smoke gases formed during combustion is an urgent problem and requires solution.

The object and subject of research. The subject of the dissertation work is to study the characteristic indicators of the component obtained of diesel fuel derived from Baku crude oils with 1-10% mass pure and 5-15% mass water-containing C_1-C_4 alcohols by conventional mixing and ultrasonic vibrations with a frequency of 20 kHz. Thereafter the synthesis of emulsifiers that ensure the stability period of emulsion diesel fuels, the study of the quality indicators of emulsified diesel fuels and the composition of smoke gases formed during combustion. The subject of the study to produce commodity diesel fuels based on partially renewable raw materials by meeting the quality requirements of modern environmental regulations.

The aim of the work. The main purpose of the work is production of emulsified diesel fuels via various mixing methods, research of their quality indicators and combustion products. It considers obtaining diesel fuel produced from a mixture of Baku crude oils containing 1-10% mass pure and 5-15% watered C_1-C_4

alcohols through conventional mixing and ultrasound mixing that meets diesel quality and flue gas content requirements of modern standards.

The main provisions for the defense. In the completed dissertation work, the quality and purity of feedstock and products were determined through IR-, 1H, 13C NMR spectra and relevant ASTM and GOST standards.

The main provisions put forward in the dissertation for defense are the followings:

- obtaining of commercial diesel fuel with pure methanol, ethanol, i-propanol and n-butanol alcohols with 1-10% mass by conventional and ultrasonic mixing methods, determination of quality indicators and combustion products;

- synthesis of 1-acylate-propanol-2,3 monoether, 1,3-dimethylol-1,3 dimethyl linolenacyclohexanol-2 ethers, oxypropylene esters of natural petroleum acids, oxypropylene esters of vegetable fatty acids and methyl esters of vegetable oils as emulsifiers for obtaining 1-10% mass emulsified diesel fuel with methanol, ethanol, i-propanol and n-butanol alcohols containing 5-15% mass water in the components of commercial diesel fuel;

- synthesis of emulsified diesel fuel with mixture of 5-15% mass water-retaining methanol, ethanol, i-propanol and n-butanol mixtures with 1-10% mass mixtures of commercial diesel fuel via conventional and ultrasonic mixing methods. Alcohol:emulsifier ratios of 1:0.25-1:0.5–1:1 studied for quality indicators, as well as combustion products;

- testing of acylate-propanol -2,3 mono ether synthesized as an emulsifier, oxypropylene esters of natural petroleum and vegetable fatty acids as multifunctional additives that simultaneously improve resource-saving and lubricating properties of diesel fuel.

The scientific novelty of the work is:

- the commodity diesel fuel obtained from Baku crude oil was mixed with C_1-C_4 alcohols as pure and 5-15% mass water contained alcholos by conventional and ultrasonic mixing at a frequency of 20 kHz. 1-10% mass emulsified diesel fuels were prepared at 25°C and 0°C. The stability period, quality indicators and composition of smoke

gases formed during combustion of the obtained compounds were studied.

• the stability period of compounds obtained by adding 0.5% mass of methyl esters of cotton seed oil acids to 1-10% mass mixtures of commercial diesel fuel with pure methanol, ethanol, i-propanol and n-butanol alcohols. Alcohol:ether ratio of 1:0.5-1:1 selected, quality indicators and composition of smoke gases formed during combustion were studied.

• 1,3-dimethylol-1,3 dimethyl linolen acyclohexanol-2 ether (SAS1), stearic acid (SAS2), as well as oxypropyl esters of cotton oil acids (SAS3), 1-acylate-propanol -2,3 mono ester (SAS4), oxypropyl esters of petroleum acids (SAS5) were synthesized and studied as emulsifiersin order to obtain stable emulsion fuel with 95-85% mass C_1-C_4 alcohols of commodity diesel fuel

• commodity diesel fuel with 95-85% purity C_1-C_4 alcohols and synthesized emulsifiers with alcohol:emulsifier ratio of 1:0.25-1:1 studied, methods of low energy (SAS4 emulsifier with high HLB) and ultrasonic mixing, the nanoemulsion diesel fuels with high stability period were obtained and their stability period, quality indicators and composition of smoke gases formed during combustion were studied

• C_1-C_4 absolete alcohols as well as synthesized emulsifiers SAS3 and SAS5 have been studied as multifunctional additives to diesel fuel to improve resource saving and lubrication properties

Practical value. Compared to mineral-based diesel fuel, the application of emulsion diesel fuels can be recommended because its technical and economic feasibility. CDF (commodity diesel fuel) mixed with 95-90% purity C_1-C_4 alcohols and synthesized emulsifiers (e.g., SAM1- 1,3-dimethylol-1,3 dimethyl linolenacyclohexanol-2 ether) in a certain ratio (e.g., 1:0, 25) were recommended for use, where the stability periods at 25°C are 150-100 days and 180-125 days in conventional and ultrasonic mixing respectively. In the low temperature (0°C) regime, it varies between 120-75 and 150-85 days. The reduction in the content of combustion products is 8-47% for CO, 7-33% for NO_x, 8-23% for SO_x and 24-27% for smokiness. Thus, it is possible to expand the possibilities of use of emulsion diesel fuels in the future by preparing different proportions of components of a

number of emulsifiers synthesized for this purpose, including anhydrous and aqueous alcohols, in order to reduce their effects on the environment and dependency on mineral resources.

Publications. Based on the dissertation materials, 18 scientific papers were published, including 8 articles, 8 theses and 2 abstracts of plenary conferences. Scientific articles on the topic of the dissertation were published in the following journals: "Processes of Petrochemistry and oil refining", "Chemical problems", "World of Petroleum Products", "Oil Refining and Petrochemistry", "Oil Industry of Azerbaijan", "News of Azerbaijan Higher Technical Schools", Sumgait State University "Scientific news".

Approbation of work. The main results of the dissertation were discussed at national and international conferences, including Current Problems of Chemistry, XI International Scientific Conference (Baku, May 15-16, 2019), VII International Scientific and Technical Conference, Alternative Sources of Raw Materials and Fuels (AIST-2019) (28-30 May 2019., city Minsk, Belarus), "Prospects of innovative development of chemical technology and engineering" International Scientific Conference (Sumgait, November 28-29, 2019), "Actual problems of modern chemistry" International conference dedicated to the 90th anniversary of ANAS NKPI (Baku, October 2-4, 2019), XIII International Scientific Conference "Actual Problems of Chemistry" dedicated to the 96th anniversary of the birth of national leader H. Aliyev (Baku, April 15-19, 2019), International Conference on Actual Problems of Chemical Engineering APCE-2020 (Baku, December 24-25, 2020), International Scientific Conference, Perspectives of Innovative Development of Chemical Technology and Engineering, (Baku, November 28-29, 2019), THERMAM 2020 9th Rostock International Conference: "Technical Thermodynamics: Thermo physical properties and Energy Systems" (Rostock, Germany, October 15, 2020), "Modern Chemistry Problems and Development Trends" Republican Scientific-Practical Conference (Baku, December 12, 2020), "Prospects of Innovative Development of Chemical Technology and Engineering" International Scientific Conference (Sumgait, November 28-29, 2019), Actual Problems of Modern Natural and Economic Sciences, International It was presented at

scientific conferences such as Scientific conference (Ganja, November 12, 2020).

Place of dissertation work. The dissertation work was carried out at the "Renewable fuels" laboratory of the Institute of Petrochemical Processes of the Ministry of Education and Science of Azerbaijan and Azerbaijan State Oil and Industry University.

Personal participation of the author. In the dissertation, determination of the purpose and directions of the work in the conducted research, implementation of experiments and analysis and generalization of the obtained results belong to the author.

The structure and scope of the dissertation. The dissertation work consists of introduction, 4 chapters, 45 figures, 63 tables, conclusions, 162 literature references and printed on 209 pages. The volume of the work includes characters for Introduction 11226, Chapter I 57296, Chapter II 22879, Chapter III 26957, Chapter IV 69795, Conclusions 2848, in total 191001 characters, excluding tables, pictures and bibliography.

The introduction of the dissertation presents the relevance of the topic, the purpose of the dissertation, the tasks set, the scientific novelty of the work, the practical significance, and the areas of application of target products.

In the first chapter, requirements for fuels and their combustion products, the main methods of obtaining diesel fuels that meet standards, an overview of the literature published in the local and foreign press is reflected.

In the second chapter, the quality indicators of the used raw materials, the methods of obtaining emulsified fuels of diesel fuel with alcohols, the synthesis methods of emulsifiers and the IR- and 1H, 13C NMR spectra that determine the degree of purity of the obtained products are provided.

In the third chapter, obtaining of 1-10% mass mixtures of diesel fuels with 99.5-99.9% purity C_1-C_4 alcohols, as well as studying the composition of the flue gases generated from mixture of commercial diesel fuel and C_1-C_4 alcohols, individual and mixed use of methyl esters of cottonseed oil acids studied.

In the fourth chapter, obtaining of emulsion fuels with

commodity diesel fuel and C_1-C_4 alcohols and surfactants, also using C_1-C_4 alcohols of commodity diesel fuel and 1,3dimethylol-1,3 dimethyl linolenacyclohexanol-2 ether as a surfactant, emulsion diesel fuel using C_1-C_4 alcohols and oxypropylene esters of fatty acids as surfactants, using C_1-C_4 alcohols and oxypropylene esters of petroleum acids as surfactants, and comparative analysis of quality indicators and combustion products studied. At the same time, a technical and economic evaluation of the process of production of emulsified diesel fuel performed that recommended for use. Thus, the cost of commercial diesel fuel obtained from the 1:0.25 ratio of 95-85% purity C_1-C_4 alcohols of diesel fuel and SAS1 emulsifier (1,3-dimethylol-1,3 dimethyl linolenacyclohexanol-2 ether), energy carriers and economic indicators such as sales value were analyzed.

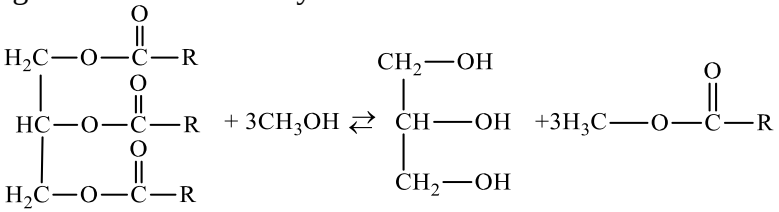
The dissertation ends with the conclusions drawn on the basis of the research carried out in the work and the list of references used.

MAIN CONTENT OF WORK

Synthesis of emulsifiers for diesel fuel and C_1-C_4 alcohol mixtures

In this research work, various emulsifiers were synthesized in order to obtain emulsified diesel fuel by adding anhydrous and aqueous C_1-C_4 alcohols as oxygen additives to commodity diesel fuel and to improve the stability indicators of mixture.

The synthesis of methyl esters of fatty acids was carried out according to the following transesterification reaction of triglycerides of vegetable oils with methyl alcohol:



The reaction was carried out in a three-necked flask equipped with a thermostat, a stirrer, and a thermometer. During the process, a measured amount of alcohol (alcohol/oil ratio is 6:1) and KOH in the amount of 0.5% based on the oil as a catalyst are placed in a glass flask, the reaction solution is heated to 65°C, a calculated amount of vegetable oil is added dropwise through a separatory funnel, and nitrogen is added with constant stirring. The process continues for 5-6 hours. After the reaction is complete, stirring is stopped and the mixture is kept still to separate the biodiesel and glycerol layers.

After separating the obtained products into two layers (lower glycerol, upper layer vegetable oil esters), the lower layer is taken by draining, the upper layer remaining in the separating funnel is washed from traces of the catalyst until a neutral reaction. Then the obtained product was dried with a rotary evaporator and the degree of purity of the obtained ethers was determined by means of 1H, 13C NMR spectra (Fig. 1):

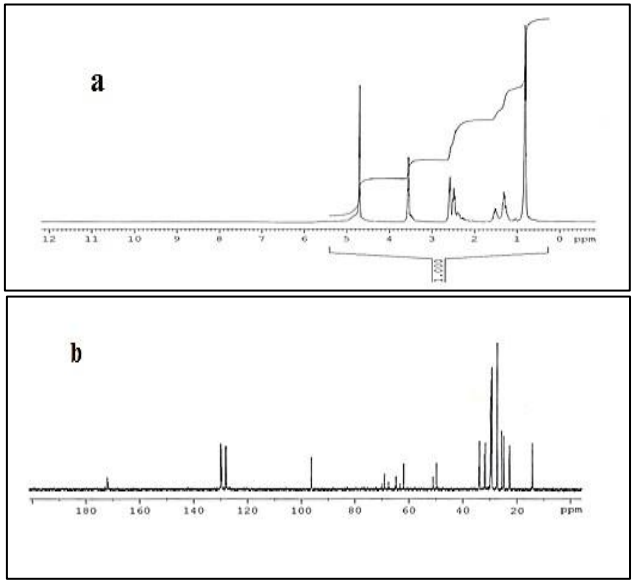
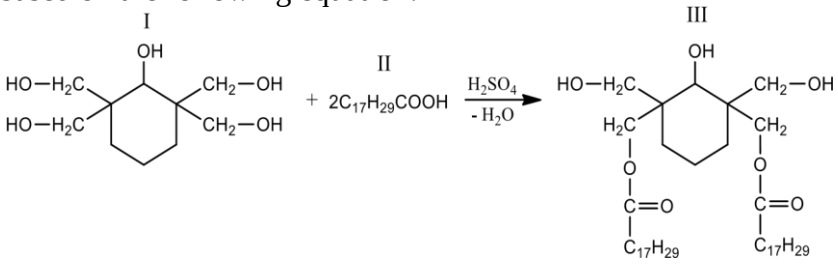


Fig. 1. ′H (a) and 13C NMR (b) spectra of methyl esters of cottonseed oil acids

The reaction for obtaining 1,3-dimethylol-1,3 dimethyl linolenacyclohexanol-2 ether (SAS1) used as an emulsifier was carried out based on the following equation:



Here I – 1,1,3,3-tetramethylol-cyclohexanol-2; II-linolenic acid; III- 1,3-dimethylol-1,3 dimethyl linolenacyclohexanol-2 ester (SAS1).

10 g of I (0.046 mol), 25.62 g (0.092 mol) of II in a three-necked reaction flask equipped with a heater, a stirrer, a thermometer, a condenser, and a counter-cooler were added to 0.5 g of solid sulfuric acid as a catalyst (calculated as 2% by acid), as a solvent 100 ml of toluene is placed. The reaction was carried out for 48 hours. During

this time, 1.7 g of water was separated from the reaction. Then, the reaction product is cooled to room temperature, neutralized in a separatory funnel, and washed. With the help of a water pump, the solvent toluene is distilled and separated from the ether. The degree of purity and structure of the obtained dried ether was determined by the IR spectrum (Fig. 2):

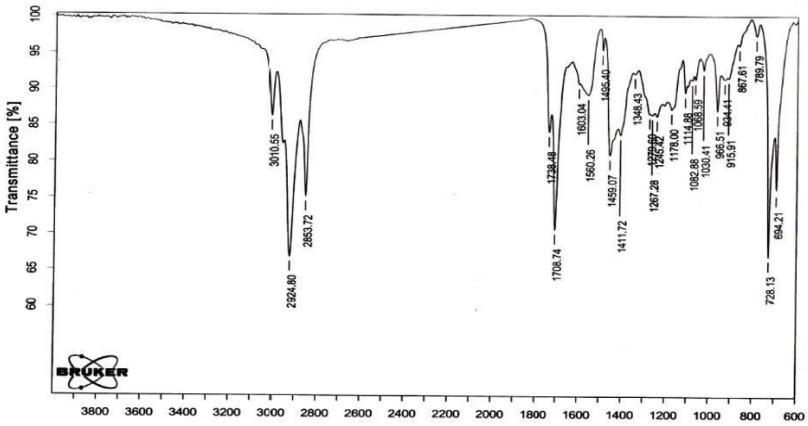
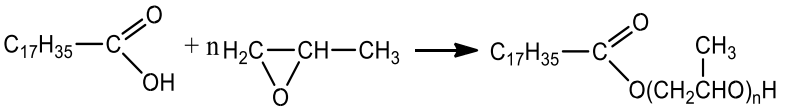


Fig. 2. IR spectrum of emulsifier SAS1

Obtaining of SAS2 emulsifier (oxyporopropylene esters of fatty acids) was carried out at 120-150°C in a 8218/8c hammered chrome-nickel steel autoclave equipped with a heating jacket rotating at 50-86 rpm. Stearic acid was used as a fatty acid.

The reaction of stearic acid with oxypropylene was carried out using potassium hydroxide as a catalyst (when 3% by mass was taken based on the acid) in a ratio of 1:5-1:6:



The degree of purity of the obtained complex ether was studied by the IR-spectroscopy method (Fig. 3):

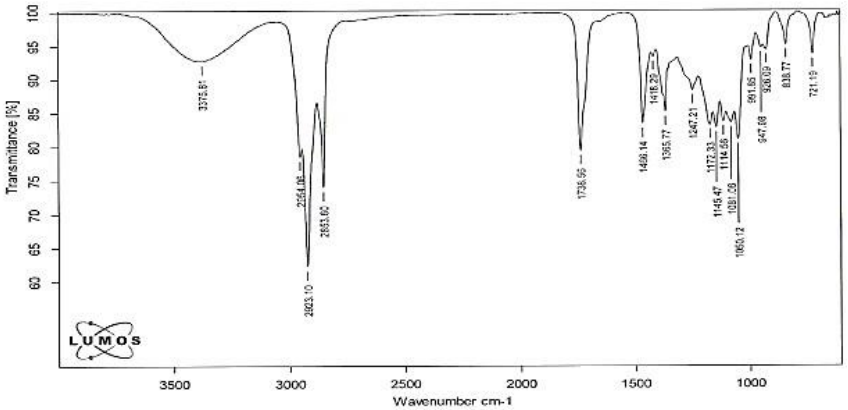


Fig. 3. IR spectrum of SAS2 emulsifier

In order to obtain SAS3 emulsifier (oxypropylene esters of cottonseed oil acids), hydrolysis of cottonseed oil was carried out by a known method in the 1st stage, and a mixture of acids was obtained. For this, 5N NaOH solution was added to cotton oil heated to 50-80°C, and the hydrolysis process was carried out for 5-6 hours. After that, the fatty acids were separated by adding 20% HCl to the obtained mixture, and in the next step, the oxypropylene ester of stearic acid was separated in an autoclave made of 8218/8c hammered chrome-nickel steel and equipped with a heating jacket, rotating at 50-86 round per minute, 120-150°C, cotton of fatty acids with oxypropylene in a ratio of 1:6, using sodium hydroxide as a catalyst (when 3% by mass of the acid is taken). The degree of purity of the received esters was checked by means of the IR spectrum (Fig. 4):

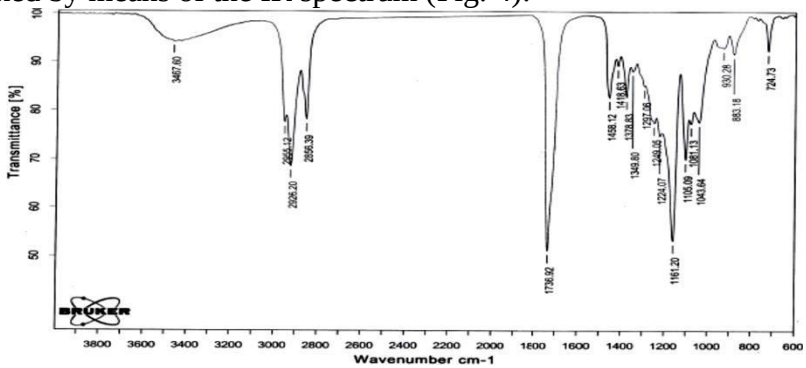
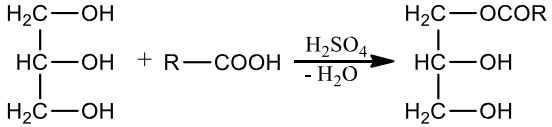


Fig. 4. IR spectrum of SAS3 emulsifier

The synthesis of SAS4 emulsifier (1-acylate-propanol-2,3) was carried out according to the following reaction:



0.1 mol of glycerin, 0.1 mol of acetic acid, 2% sulfuric acid (calculated by acid), 150 ml of benzene or toluene, xylene, CCl4 (carbon chlorine-4) are placed in the reaction flask and the reaction is carried out for 5-6 hours. As a result of the process, 1.8-2 g of water is obtained from the reaction. The end of the reaction is determined by the fact that the acid number does not change. Two layers form in the reaction flask. The upper layer is separated from the lower layer in a separator sleeve. The upper layer is neutralized, washed and the solvent is distilled off from ether. The obtained ether is obtained by ordinary and pressure distillation, depending on the molecular weight and boiling point. The degree of purity and structure of the obtained ether was determined by the IR spectrum (Fig. 5):

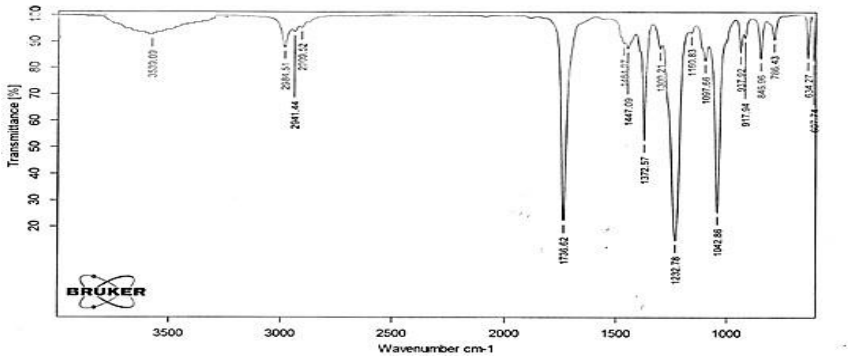
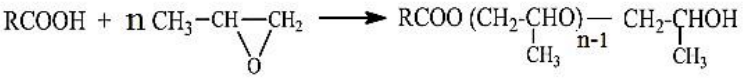


Fig.5. IR spectrum of emulsifier SAS4

The obtanining of SAS5 emulsifier (oxypropylene esters of natural petroleum acids) was carried out according to the following equation:



The process was carried out in a rotary 8218/8c forged chrome-nickel steel autoclave equipped with a heating jacket at 50-86 rounds per minute.

For this, 200-300 ml of raw materials with different proportions are placed in an autoclave with a capacity of 1 liter. During the thermocatalytic conversion of the raw material, a calculated amount of catalyst is added to the autoclave at the same time, and when the temperature rises to 50°C, the motor starts to rotate the autoclave. The temperature is raised to 120-140°C and at this time the pressure increases to 10-12 atm and the process continues for 5 hours under these conditions. The degree of purity of the obtained ethers was checked by means of the IR spectrum (Fig. 6):

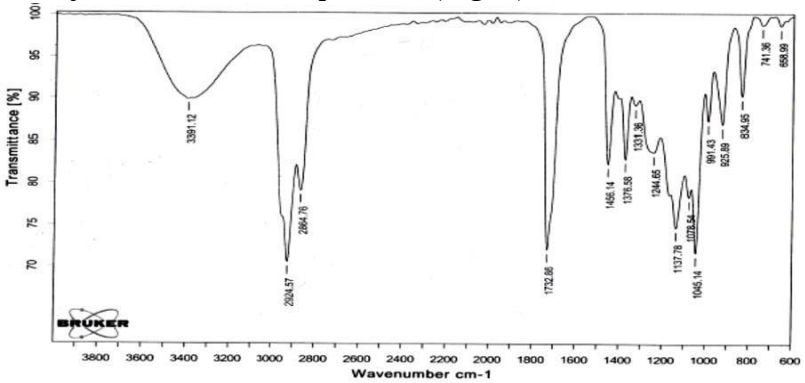


Fig.6. IR spectrum of emulsifier SAS5

Synthesis of 1-10% mixtures of diesel fuels with C_1-C_4 alcohols and research as fuel

1-10% mixtures of C_1-C_4 alcohols with diesel fuel at 0.5-0.1% moisture content were prepared and the stability of the obtained compounds, depending on the temperature, time and method of preparation, was studied. Experiments were performed at room temperature (25°C) and at 0°C. The preparation of diesel fuel/alcohol mixtures was carried out using a mixer with a stirring speed of 600 rpm and a Helshier UIP2000hd ultrasonic sonotrode with a frequency of 20 kHz. To evaluate the time stability of the obtained compounds,

the particle sizes of the alcohols in diesel fuel were determined by taking Dynamic Light Scattering LB (Horiba, UK) spectra. An argon laser with a variable intensity (λ = 650 nm) and a scattering angle of 173° was used to measure the dimensions in a wide range.

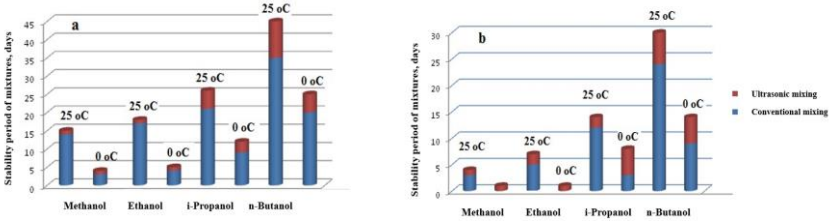


Fig.7. Comparison of stability period of C_1-C_4 alcohols with diesel fuel compounds for 5% (a) and 10% (b) mixtures

From the obtained results, it is known that the increase in the stability period of the compounds directly depends on the length of the alkyl radicals of the alcohols used. At the same time, the increase in the stability time in mixing through the ultrasonic sonotrode is more observed in the use of i-propanol and n-butanol. Although mixtures with various completely anhydrous alcohols are clear stable during the first few days of preparation, later these mixtures turn into slightly coalescing solutions, and then these mixtures can be considered emulsions of diesel fuel with alcohols. After a certain time, the emulsions separate and form a two-phase mixture (diesel fuel and alcohol phases). This fact can be explained by the growth of alcohol particles in the diesel fuel until the alcohol particles completely split into two phases, as determined by dynamic light scattering.

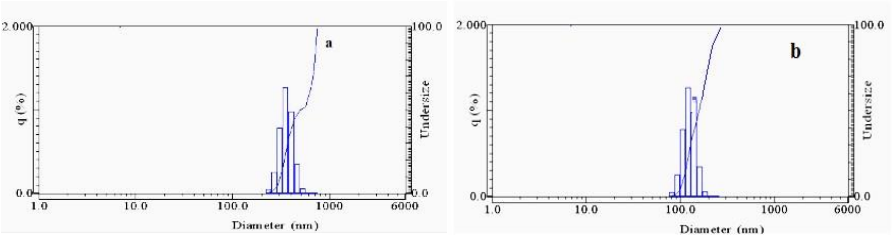


Fig.8. Particle sizes of methanol (a) and n-butanol (b) in 5% mixtures with diesel fuel

Apparently, the alkyl radical of methanol is shorter than the radical of n-butanol, and therefore it dissolves worse in diesel fuel than n-butanol, methanol molecules do not share evenly in diesel and they form agglomerates under different conditions. Butanol molecules share equally in diesel fuel and are further away from each other, and their ability to form agglomerate is less than that of methanol.

In the next stage of the research, the quality indicators of the 1,3,5,7,10% compounds of the used C_1-C_4 alcohols with commercial diesel fuels were determined, and it was found that the most stable and qualitative mixtures close to traditional diesel fuel are n-butanol of diesel fuel obtained from a mixture with alcohol.

Synthesis of 1-10% mixtures with C_1-C_4 alcohols and methyl, ethyl and butyl esters of cottonseed oil acids (ME COA, EE COA, BE COA)

At the next stage of the research, ethyl and butyl esters of cottonseed oil acids were studied as additives that increase the stability period of the 5% mixture of Commodity diesel fuel (CDF) with C_1-C_4 alcohols. It was found that the stability period of the 5% mixture of CDF with methanol does not depend so much on the length of the alkyl radical of the methyl-ethyl-butyl ethers of added cottonseed oil acids, and it can be recommended for use:

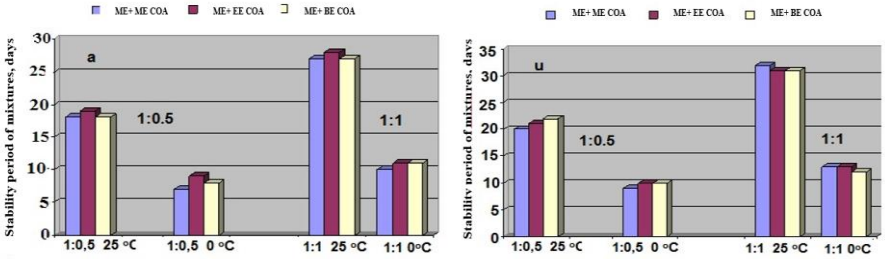


Fig.9.CFD Comparative stability time of alcohol:ether mixtures obtained by conventional (a) and ultrasonic (u)

The quality indicators of these mixtures were also studied, and the results for diesel fuel obtained in a 1:1 ratio of n-butanol and ME COA are given below:

Table 1

Quality indicators of 1-10% compounds of CDF with n-butanol and ME COA in a ratio of 1:1

Quality Indices	EN-590	CDF	CDF + (n-Butanol:ME COA 1:1)				
			1%	3%	5%	7%	10%
Density at 20°C, кg/m3, max	860,0	847,4	847,9	847,1	846,1	845,7	844,6
Fractional composition, °C							
50% distilled at	280	280	279	279	279	276	275
90% distilled at	350	340	340	340	337	335	334
96% distilled at	360	355	354	355	357	353	348
Flash point, °C, min	55	74	72	69	56	49	46
Kinematic viscosity at 20°C, mm^2/s, max	2-6	3,20	3,25	3,21	3,18	3,15	3,10
Pour point, °C, max	-10(-35)*	-31	-32	-32	-32	-31	-31
Cloud point, °C, max	-25(-10)*	-20	-21	-21	-21	-21	-21
Test on a copper plate at 100°C, 3 h	+	+	+	+	+	+	+
Aromatics	15,0	18,0	17,4	17,1	16,7	16,2	16,15
Acidity, mg KOH/100 sm3 fuel, max	5	1,5	1,7	1,7	1,8	1,7	1,9
Iodine number, g I/100 g fuel, max	6	0	1,0	1,8	2,6	2,8	3,4
Total sulphur content, % wt, max	0,005	0,0112	0,0111	0,0111	0,0110	0,0098	0,0096
The actual amount of tar in 100 cm3 of fuel, mg, max	25	18	17,9	17,5	16,7	16,0	15,7
Low heating value, kJ/kg, min	-	42880	42910	42900	42760	42850	42700
Cetane number, min	51	46	45,5	45,5	44	42,5	40,5

As seen from the table, the amount of aromatic hydrocarbons and sulfur compounds remains unchanged for the mixtures of cottonseed

oil acids with methyl esters and methanol of CDF, the actual amount of resins observed in 100 cm3 is slightly higher compared to the ratio of alcohol:esters of 1:0.5 indicators are observed, and also increase relatively at low combustion temperatures.

Thus, 1-10% mixtures of methanol-ethanol-i-propanol-n-butanol and methyl esters of cottonseed oil acids prepared in the ratio of 1:0.5 and 1:1 meet the requirements of the EN-590 standard.

The study of the cetane number and the composition of the smoke gases of the C_1-C_4 alcohols and methyl esters of cottonseed oil acids and the methyl esters of CDF was carried out at the TDF-69 single-chamber diesel engine stand at the Oil Refinery named after H. Aliyev, and the reduction of the smoke gases by % is given below:

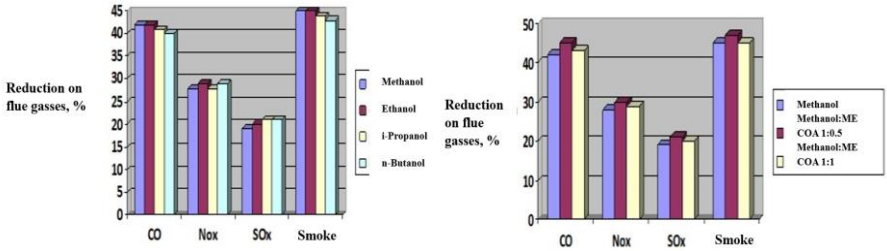


Fig.10. Comparison of reduction of flue gases for mixtures of CDF with C_1-C_4 alcohols and methanol, ME COA

As seen from the data, regardless of the nature of the used alcohols, as the concentration of methyl esters of cottonseed oil acids in diesel fuel increases, carbon monoxide in the combustion products of the obtained compounds decreases by 7-45%, the amount of nitrogen and sulfur oxides by 5-29% and 7-22% respectively, and overall smokiness decreases by 4-45%.

Production of emulsion diesel fuel using C_1-C_4 alcohols with CDF and 1,3-dimethylol-1,3 dimethyl linolenacyclohexanol-2 ether (SAS1) as surfactant

CDF containing 1-10% mixtures of 5-15% watered methanol, ethanol, i-propanol or n-butanol were prepared, and at the same time,

in order to obtain a stable emulsion, the compounds obtained by adding different amounts to the SAS1 CDF/methanol mixtures, the stability periods, fuel quality indicators including combustion gases were identified and compared. In addition, the hydrophilic-lipophilic balance (HLB) of the obtained substance was determined according to equation (1):

$$H = 7 + \sum_{i-1}^{m} H_i - 0.475 \cdot n,$$

Here: m-the number of hydrophilic groups in the molecule, coefficient for hi- i-hydrophilic group: 2.1 for the -COOH group: 1.9 for the -OH group; -0.475 for -CH, CH2, CH3, =CH- groups, n- is the number of lipophilic groups in the molecule:

HLB_{SAS1}=6*(-0,475)+3*1,9+7+2,1+36*(-0,475) 2*2,1=8,55

At the same time, mathematical modeling of the process of obtaining emulsified fuels with SAS1 was carried out.

To develop a process regression model, the functional relationship between process parameters was determined and used for process prediction.

The output function (duration of the obtained mixture as a stable mixture, days) can be described as a linear polynomial:

$$Y_k = a_0 + \sum_{i=0}^{n} a\, a_i \times z_i + \sum_{\substack{i=0 \\ i \neq g}}^{n} a\, a_{ij} \times z_i \times z_j\ ;\ \ k = 1,2,3 \quad (1)$$

Here; Y_k - output parameters (95%, 90%, 85% for 3 concentrations of alcohols), Z_i-factors, a_i - coefficient of the regression equation.

"S-plus 2000 professional" program was used to determine the coefficients of equation (1). This program is used for automated mathematical processing of experimental data obtained by "Mathwork" company. The coefficients of the regression equation are presented.

The assessment of the significance of the regression coefficients was confirmed by the significant correlation coefficient, the student's criterion, and also the experimental error of approximation:

$$ti = \frac{|ai|}{\sqrt{S2a}} \quad (2)$$

where S2a is the experimental error.

Coefficients are tested for significance by determining the values of S2ac and inserting it into Equation 2 (2). Thus, the calculations showed that the coefficients a13 and a23 are insignificant in all three cases, so they can be excluded from the equations. In this case, the final equations can be described as follows:

Y_1 =116,7-6,94*Z1 + 3,38*Z2+1.41*Z3 +3,75*Z1*Z2 .....(3)
Y_2=89,56– 7,59*Z1 + 19,0*Z2+1,746*Z3 +1,88*Z1*Z2 .....(4)
Y_3= 76,74-7,8*Z1 + 7,7*Z2+1.267*Z3 +3,617*Z1*Z2 .....(5)

The correctness of the obtained model was checked by means of the Fischer criterion:

$$F = \frac{S2qal}{S2tək} \quad (6)$$

Here S_{qal} is the residual variance and Stack is the repeatable variance and is calculated according to the following equations:

$$S2\ qal = \frac{1}{N-l} \sum_{n} (Ypk - Ytk)\ 2 \quad (7)$$

$$S2\ tək = 1/(m-l) \sum_{1}^{m} (Ypk - Ytk)\ (Yp0\text{-}Yt0)^2 \quad (8)$$

where N is the total number of experiments,

l- the number of significant coefficients of the regression Ykh- the calculated value of the output parameter

Ykt is the experimental value of the output parameter

Using the obtained numbers in equation (7) and (8), we get:

$S2_{1qal}$=6,67 $S2_{2qal}$ =5,85 $S2_{3qal}$ =7,66
$S2_{1tək}$ = 1,2 $S2_{2tək}$ = 0,95 $S2_{3tək}$ =1,5

The correctness of the model is determined by the number F. Using the values of S2i $_{qal}$ and S2i $_{tək}$ in equation (6), we find:

F1p=5,55 F2p=6,15 F3p=5,1

If the level of significance is 5%, the table value of Fisher's value is Fc =8.9. When Fp<Fc, the statistical model adequately describes the process being studied and can be used to determine optimal process parameters.

Regression equations not only allow predicting the values of the response function for the given conditions of the experiment, but also provide the necessary information to choose the optimal mode of the technological process.

To solve the optimization problem, the Matlab-6.5 program, which includes modern algorithms of the linear programming problem, was applied.

As an optimization criterion, the maximum functional Y1= F max = f(Z1,Z2,Z3), which indicates the maximum number of days was taken which the mixtures work stably.

Calculations show that Z1 is 1% wt.mass when equal, when the ratio of methanol to SAS1 is equal to 1: 0.25 (Z2) and the temperature of Z3 is equal to 25 °C, the period of use of the received fuel as a stable fuel is 150 days. The mathematical model can be used to predict the process of obtaining stable diesel fuels using C_1-C_4 alcohols and various SASs.

Thus, the stability periods of the compounds were studied and determined that adding 0.25% of SAS1 to the diesel fuel/methanol mixture with respect to alcohol for a 1% mixture of 95% methanol in CFD increases the stability period of the obtained compounds up to 150 days. The same result is observed when the mixture is kept at a low temperature and when the content of methanol in diesel fuel is increased to 3%. In both cases, the obtained compounds are completely transparent. Considering that the stability time of those mixtures is up to 21 minutes without using SAS1, there is no doubt that SAS1 is an effective emulsifier.

Regarding the size of the particles, the size of the particles in the compound obtained by ultrasonic vibrations with a frequency of 20 kHz is 20-100 nm, and the size distribution of the particles in the nanoemulsion obtained at this time is narrower. In this case, as the amount of SAS1 increases, the amount of small-sized particles increases, and the average diameter of the particles changes over time

(Fig. 11):

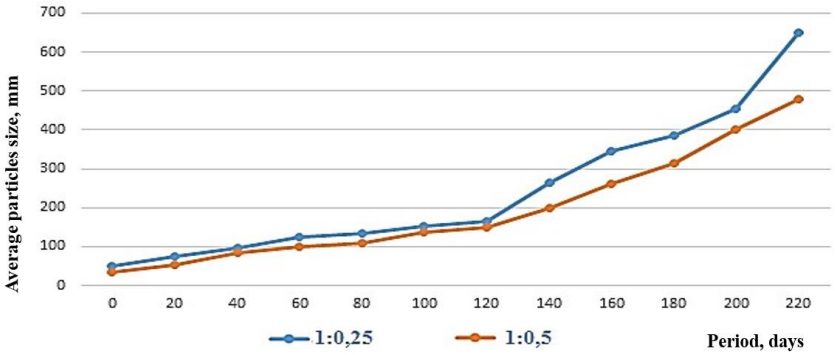


Fig.11. Variation of particle sizes with time in ultrasonic mixing of alcohol:SAS1 ratio of 1:0.25 and 1:0.5

Low combustion heat for mixtures of 95-85% n-butanol with SAS1 in a ratio of 1:0.25 with diesel fuel is 42285-42080 kJ/kg. At the same time, the cetane number decreases when alcohols are added, at least when n-butyl alcohol is used. Other indicators also meet normative standards:

Table 2

Quality indicators of 95-85% N-Butanol:SAS1 1:0.25 ratio of 5% compounds of CDF

Quality Indices	EN-590	CDF	CDF+ 5% n-Butanol		
			95%	90%	85%
1	2	3	4	5	6
Density at 20°C, кg/m3, max	860,0	847,4	846,0	846,4	847,2
Fractional composition, °C					
50% distilled at	280	280	276	277	277
90% distilled at	350	340	334	336	337
96% distilled at	360	355	349	350	350
Flash point, °C, min	55	74	56	57	57
Kinematic viscosity at 20°C, mm2/s, max	2-6	3,20	3,14	3,15	3,16
Pour point, °C, max	-10(-35)*	-31	-32	-32	-33
Cloud point, °C, max	-25(-10)*	-20	-21	-21	-22
Test on a copper plate at 100°C, 3 h	+	+	+	+	+
Aromatics	15,0	18,0	16,4	16,2	16,2

continuation

1	2	3	4	5	6
Acidity, mg KOH/100 sm3 fuel, max	5	1,5	1,3	1,4	1,3
Iodine number, g I/100 g fuel, max	6	0	0	0	0
Total sulphur content, % wt, max	0,005	0,0112	0,0111	0,0111	0,0110
The actual amount of tar in 100 cm3 of fuel, mg, max	25	18	16,0	15,6	15,3
Low heating value, kJ/kg, min	-	42880	42285	42195	42080
Cetane number, min	51	46	44	44	44

As for the composition of combustion products, the reduction of carbon monoxide is 8-47%, nitrogen and sulfur oxides are 7-33% and 8-23% respectively and the reduction of smoke is 24-27%. Ultrasonic mixing results an additional 2-5% reduction of these oxides for 1:0.25 ratio of 90% alcohols with SAS1 to commodity diesel fuel compounds. In general, for 1-10% mass C_1-C_4 alcohols of commodity diesel fuel and compounds obtained in a ratio of 1:0.25 with SAS1, the smokiness of exhaust gases decreases by 12-48%.

Production of emulsified diesel fuel using oxypropylene esters of C_1-C_4 alcohols and fatty acids (SAS2/SAS3) as surfactants

Oxypropylene esters of fatty acids-oxypropylene ester of stearic acid (SAS2) as an emulsifier for the mixture of CDF with C_1-C_4 alcohols, as well as oxypropylene esters of a mixture of fatty acids obtained from the hydrolysis process of cottonseed oil (SAS3) were prepared and studied in different proportions. The hydrophilic balances of emulsifiers were calculated:

HLB_{SAS2}=15(-0,475) +7+2,1+3,28*(-0,475) +1,9=2,32

For oxypropylene esters of cottonseed oil acids (SAS3), the HLB is taken to be approximately 2.7 (due to the presence of alkyl radicals of different C16-C18 lengths).

The stability indicators of the compounds obtained by conventional and ultrasonic mixing methods are given below (Fig.12):

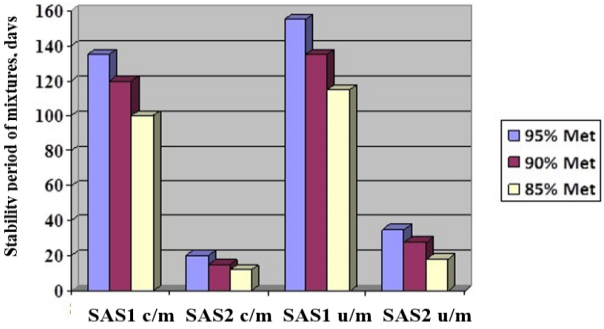


Fig.12. Comparative stability times of compounds using SAS1 and SAS2 emulsifiers

As can be seen from the figure, the stability time of compounds obtained using SAS2 are 2.7-6.2 times less than the stability times of compounds obtained using SAS1. It was found that the CDF of 90% methanol and 5% compounds obtained at alcohol:SAS1 ratios of 1:0.25 and 1:0.5 compared to the particle sizes obtained in the presence of SAS2 emulsifier even at alcohol:SAS2 ratios of 1:1 particle sizes are almost 2-10 times larger. That is, the SAS2 emulsifier is not able to form a complete nanoemulsion even with an alcohol:SAS2 ratio of 1:1 and ultrasonic mixing.

SAS3 was studied as an emulsifier to obtain a stable compound with 95-85% C_1-C_4 alcohols of commodity diesel fuel, and the alcohol:SAS3 ratio of CDF at 1-10% with 95-85% purity C_1-C_4 alcohols in ratio of 1:0.25-1:0.5. 1-acylate-propanol -2,3 mono ether (SAS4) emulsifier was tested as an emulsifier providing a phase change in obtaining compounds with nanosized particles in ratio of 1:1. In order to obtain a stable emulsion with methanol containing 5-15% water in the diesel fuel, the stability times of the compounds obtained by adding 0.5% to the SAS4 CDF/methanol mixture were determined. Hydrophilic-lipophilic balance was calculated:

$$HLB_{SAS4}=3(-0,475)+7+2,1*2+1,9*2=13,1$$

It was found that the addition of 0.5% of SAS4 to the mentioned mixtures leads to a practically 2-2.5 times increase in the stability times of the obtained compounds. Comparative rates of Ostwald ripening in emulsified fuels obtained with the use of SAS3 and

SAS3+SAS4 emulsifiers of 95% ethanol and 5% mixture of commodity diesel fuel are shown in Fig.13:

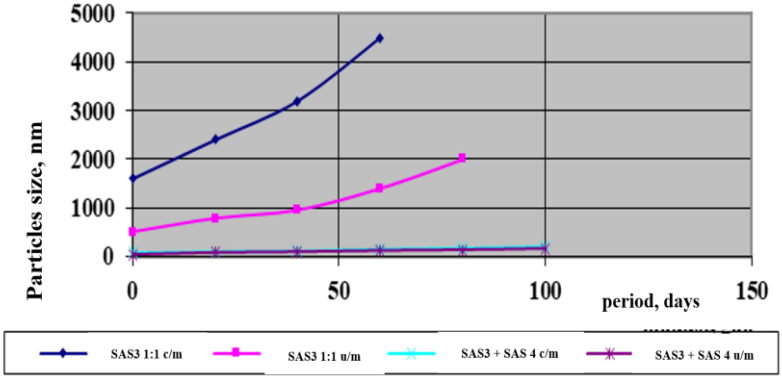


Fig.12. Comparative rates of Ostwald ripening using SAS3 and SAS3+SAS4 emulsifiers

Studies have shown that SAS2 and SAS3 can be used as emulsifiers to obtain stable emulsified fuel with 95-85% purity C_1-C_4 alcohols of commodity diesel fuel, and CDF with 95-90% purity C_1-C_4 alcohols and SAS3 emulsifier. Compounds taken in a 1:1 ratio can be recommended for use. Also, when 5% is added to the C_1-C_4 anhydrides alcohols with CDF, the composition, the diameters of the corrosion spots of the mixtures are reduced by 10-29%. The addition of SAS3 and SAS3/SAS4 emulsifiers causes a decrease in the diameters of the corrosion spots of the received diesel fuels by 21-42 and 34-58%.

Production of emulsified diesel fuel using CDF with C_1-C_4 alcohols and oxypropylene esters of petroleum acids (SAS5) as surfactants

In order to obtain a stable emulsion with methanol that containing 5-15% water in the diesel fuel, the stability times of the compounds analyzed by adding to the SAS5 CDF/methanol mixture in the ratio of 1:0.25-1:0.5-1:1. The stability times of the compounds via ultrasonic mixing are slightly increased and for mixtures with 1,3,5,7% alcohol:SAS5 in the ratio of 1:0.25-1:1 it is 52-78, 46-70, 25-50, 20-34 days, and for a 10% mixture, it increases only 2 days and makes

12-14 days. Approximately the same stability times are observed using 95-85% ethanol, i-propanol and slightly more n-butyl alcohol, but they differ sharply from the stability times of the compounds obtained using SAS1 (Fig. 14).

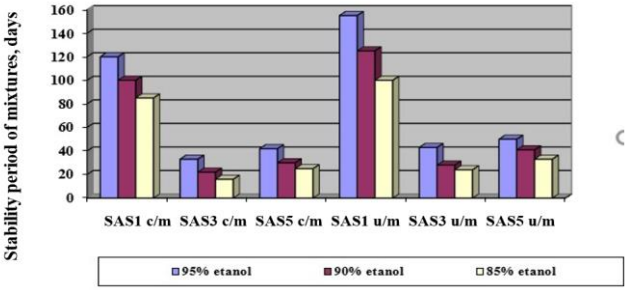


Fig.14. Comparative stability times of compounds using SAS1, SAS3 and SAS5 emulsifiers

As can be seen, the stability times of compounds obtained using SAS5 are 2.7-6.2 times shorter than the stability times of compounds obtained using SAS1, but are approximately the same as those obtained using SAS3. The speed of Ostwald ripening in the obtained compounds is high, and layering occurs in the emulsions after 40-45 days (Fig. 15).

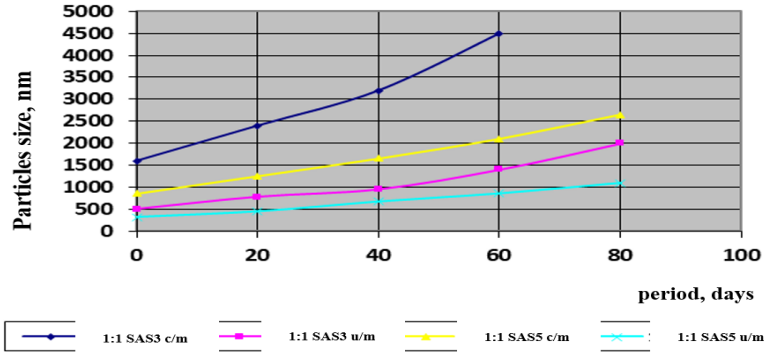


Fig.14. Variation of mean diameter of particle sizes compared to time using CDF with SAS3 and SAS5 emulsifiers

Summarizing the results of the conducted research, it can be concluded that in order to obtain a stable emulsion fuel with 95-85% C_1-C_4 alcohols of commodity diesel fuel, as well as other synthesized

SAS 1-4 emulsifiers, oxypropyl ethers of petroleum acids (SAS5) should be used as an emulsifier.

Technical and economical evaluation of the process of obtaining emulsified diesel fuel

Technical and economical evaluation report prepared for commercial diesel fuel obtained from 1:0.25 ratio of C_1-C_4 alcohols and synthesized SAS1 emulsifier 1,3-dimethylol 1,3 dimethyl linolenacyclohexanol-2 ether. Obtained CDF recommended for use as per study of IPP (Institute of Petrochemical Processes) based on data provided by "Petroleum complex processing and technical-economic justification" laboratory.

The general material balance for the process is given in table 3:

Table 3

Process material balance

Stage I. Synthesis of SAS1 emulsifier		
	gr	%
Feedstock:		
1,1,3,3-tetramethylol-cyclohexanol-2	10,00	28,07
Linolenic acid	25,62	71,93
Total:	35,62	100,00
Product:		
SAS1 – emulsifier (1,3-dimethylol 1,3 dimethyl linolenacyclohexanol-2 ether)	33,62	94,39
Water	1,70	4,77
Losses:	0,30	0,84
Total:	35,62	100,00
Stage II. Production of emulsified diesel fuel		
Feedstock:		
Commodity diesel fuel	8236,90	98,0
1- butanol (95% purity)	134,48	1,6
SAS1	33,62	0,4
Total:	8405,00	100,0
Product:		
Diesel fuel	8362,98	99,5
Losses:	42,02	0,5
Total:	8405,00	100,0

The cost of SAS1 and the wholesale price of emulsion diesel fuel, which is the final product, were calculated taking into account the price of the main raw materials and auxiliary materials, including the indicators of energy carriers. Currently, domestically produced CDF wholesale price is 582 AZN/t, domestic wholesale price including taxes is 828 AZN/t, domestic retail price is 952 AZN/t. Taking those values into account, the result of calculations in 3 options is given in the following table:

Table 4

Calculation of the wholesale price of emulsion diesel fuel

№	Indices	Amount, kg	Price, AZN/kg	Cost, AZN
1	Diesel fuel (with wholesale price within the company)	98	0,582	57,04
	Butanol+SAS1 mixture	2	16,095	32,19
	Total	100	0,892	89,23
2	Diesel fuel (with domestic wholesale price)	98	0,828	81,14
	Butanol+SAS1 mixture	2	16,095	32,19
	Total	100	1,133	113,33
3	Diesel fuel (at retail price)	98	0,952	93,30
	Butanol+SAS1 mixture	2	16,095	32,19
	Total	100	1,255	125,49

Emulsified diesel fuel is calculated in all three variants and the price range is 892-1255 AZN/t. A relatively good result is obtained under option 1, where commodity diesel fuel is taken at the intra-company wholesale price.

If diesel fuel can be taken in distillate form (at a price of 400 AZN/t), then the cost of the final product will be as follows (727 AZN/t). The report is presented in table 5:

Table 5

Calculation of the wholesale price of emulsion diesel fuel

Indices	Amount, kg	Price, AZN/kg	Cost, AZN
Hydrotreated diesel fraction	98	0,400	39,2
Butanol+SAS1 mixture	2	16,73	33,46
Total	100	0,727	72,66

Current calculations have the status of preliminary feasibility study. If the indicators of the process (mode of operation, duration, consumption of energy carriers, prices, etc.) will change, the economic indicators will change accordingly.

CONCLUSIONS

1. 1 -10% compounds prepared for the mixture of CDF, an absolute and 5-15% aqueous C_1-C_4 alcohols (methanol, ethanol, i-propanol and n-butanol) at 25°C and 0°C via conventional mixing and ultrasonic mixing at frequency of 20 kHz. It was determined that as the alkyl radicals of alcohols increase, the stability periods of their compounds obtained with diesel fuel also increase [2,4-5,8,13-15,17].
2. It was found that the stability periods of CDF with C_1-C_4 alcohols for 1-10% compounds are 20-3, 27-5, 30-12 and 67-24 days at 25°C but at 0°C and it is 8-0, 9-0, 14-3, 45-9 days respectively [2-3,13-14,18].
3. It was determined that the stability of the compounds obtained by adding methyl esters of cottonseed oil acids to 1-10% mixtures of CDF with absolute C_1-C_4 alcohols in an alcohol:ether ratio of 1:0.5-1:1 that causes to increase stability period 5-12, 6-16, 7-15 and 8-19 days respectively [2-3,13-14,18].
4. CDF with 95-90% purity C_1-C_4 alcohols and SAS1 (1,3-dimethylol-1,3 dimethyl linolenacyclohexanol-2 ether) emulsifier in a ratio of 1:0.25 are recommended for use, where stability periods are 150-100 days and 180-125 days at 25°C in conventional and ultrasonic mixing respectively. In the low temperature condition, it varies between 120-75 and 150-85 days. The reduction in the content of combustion products is 8-47% for CO, 7-33% and 8-23% for NO_x and SO_x respectively, 24-27% for smokiness. At the same time, preliminary technical-economic calculations for this recommended option were also carried out and satisfactory results were obtained [13].
5. CDF with 95-90% purity C_1-C_4 alcohols and SAS3 (oxypropyl

esters of cottonseed oil acids) emulsifier 1:1 ratio was considered optimal and the compounds obtained in this ratio can be recommended for use. Stability periods are 33-22 days and 43-28 days in conventional and ultrasonic mixing. The reduction in the content of combustion products is 9-50% for CO, 8-36% and 9-21% for NO_x and SO_x and for smokiness it is 12.09-23.6% [2-3.5-6.9-17].

6. It was found that adding 0.5% mass of 1-acylate-propanol-2,3 mono ether (SAS4) to the compounds obtained in a ratio of 1:1 into CDF with 95-90% purity C_1-C_4 alcohols of the emulsifier and SAS3 emulsifier causes a 2-2.5 times increase in stability periods and is 70-64 and 74-68 days respectively [2-3.5-6.9-17].
7. 95-90% purity C_1-C_4 alcohols with 5% mass of CDF and 1:1 ratio of C_1-C_4 alcohols and SAS5 (petroleum acid oxypropyl esters) emulsifier are recommended for use, where stability times are 42-25 days and 50-29 days in conventional and ultrasonic mixing respectively. The reduction in the content of combustion products is 11-52% for CO, 9-38% and 10-22% for NO_x and SO_x and 25% for smoking [2-3,5-6,9-17].
8. It was found that adding 0.5% mass to the compounds obtained with 95-90% purity C_1-C_4 alcohols and CDF with SAS4 emulsifier and SAS5 emulsifier in a ratio of 1:1 causes a 2-2.2 times increase in the stability times of the obtained mixtures and, it is 112 -61 and 125-66 days respectively [8-9].
9. It was determined that emulsifiers SAS3 and SAS5 in CDF at 1-10% mass mixtures can be used as emulsifiers simultaneously with anhydrous and 5-15% mass water contained C_1-C_4 alcohols as well as resource-saving, anti-aging additives. Thus, when C_1-C_4 absolute alcohols are added to the CDF composition by 5%, the diameters of the corrosion spots of the obtained mixtures decrease by 10-29%. The addition of SAS3 and SAS3/SAS4 emulsifiers causes a decrease in the diameters of the corrosion spots by 21-42 and 34-58% [23,5-6,9-17].

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