Improving the Three-Component Mixed Motor Fuel Manufacturing Process

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Abstract

It has been established that it is impossible to obtain motor fuel for diesel engines that meets the requirements of modern environmental standards without the use of additives. In the process of dearomatization and hydrodesulfurization of the diesel oil fraction, surfactants that are able to protect friction surfaces from wear are removed, which impairs the lubricity of diesel fuel. The advantages and disadvantages of using biodiesel are identified. It was revealed that the reason for the discrepancy of the physicochemical characteristics of biodiesel with the requirements of normative and technical documentation is the presence in it of only high molecular weight mainly unsaturated components (methyl esters of fatty acids). It is proved that direct hydrogenation of biodiesel components leads to a sharp increase in their boiling and melting temperatures, which makes reduced fuel unsuitable for practical use in modern diesel engines. The operational and environmental properties of modern diesel fuels can be improved by adding biodiesel and low molecular weight esters to them. A new composition of a three-component mixed motor fuel is proposed: the first component is mineral diesel fuel (40 vol. %); the second component – the heaviest – high molecular weight biodiesel fuel (50 vol. %); the third component – the lightest – low molecular weight limit esters (10 vol. %). It has been established that for the production of a three-component mixed motor fuel, the most promising is the mixing of mineral diesel fuel with high molecular weight biodiesel fuel and low molecular weight esters. The most optimal sources of plant materials for the preparation of a high molecular weight fuel component are determined. The initial compounds and parameters of the esterification reaction were selected to obtain a low molecular weight component. A technological process has been developed for processing raw materials of plant and organic origin to be added to mineral diesel fuel. For the first time, the physicochemical, operational, and environmental characteristics of a three-component mixed motor fuel were experimentally determined.

Keywords

Biodiesel; diesel; transesterification; reactor; technological process; three-component mixed motor fuel; esterification.

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Introduction

Numerous experimental studies of the use of vegetable oils as motor biofuels over the past 40 years have revealed their positive (a significant decrease in the emission of toxic exhaust components) and negative (after 200–240 hours of operation, problems such as coking occur) nozzle openings of atomizer nozzles, the formation of tarry deposits on the fuel filter, contamination of engine oil, varnish and carbon formation, up to the failure of the diesel engine) properties [1–5]. The attractiveness of using oils of plant and animal origin as biofuel is due to the fact that the chemical structure of their fatty acids is close to hydrocarbons of mineral diesel fuel. The main difference is that in the structure of fatty acid molecules, the hydrocarbon radical (CH3) is replaced by a carboxyl group (COOH), and for the structure of hydrocarbons and esters of fatty acids, the substitution of the CH3 radical by the COOCH3 group is typical [4, 6]. The noted regularities could not but affect the physicochemical properties of biofuel and mineral diesel fuel: bio-fuels have a heavier fractional composition, which leads to increased density and
viscosity [7, 8]. One of the promising directions for solving the identified problem is the use of three-component mixed motor fuels: the first component is mineral diesel fuel; the second component is biodiesel and the third component is the lightest (AI-80 gasoline was used in [9] to reduce the density and viscosity of biodiesel). It was established [9, 10] that the use of a three-component mixed motor fuel leads to a certain decrease in the calorific value (on average by 5–7% compared to diesel fuel). However, against the background of significant environmental benefits and taking into account the high efficiency of the diesel engine, this fact cannot be taken into account when operating internal combustion engines [9, 10].

At the same time, the use of petroleum fuel as a light component (AI-80 motor gasoline) leads to an additional overspending of light petroleum products (in [9], 10 vol. % Gasoline of the composition of a three-component mixed motor fuel was used) and causes serious economic and environmental difficulties in the operation of agricultural diesel engines.

To prevent additional consumption of gasoline, to eliminate its negative impact on the operation of a diesel engine during the long-term use of such fuel and to improve the environmental properties of modern diesel fuels, their cetane number and tribological characteristics, it was proposed [11, 12] to use two-component fuel: a mixture of biodiesel fuel (50 vol. %) and low molecular weight limit esters (50 vol. %). However, when using two-component biofuels as motor fuel, the initial engine adjustments may be violated, a number of performance indicators may deteriorate along with increased wear of engine parts and, as a result, a sharp decrease in the life of diesel engines [9]. Therefore, this work also proposes the use of three-component mixed motor fuels, but unlike [9], low molecular weight esters are used as the lightest component.

An analysis of the domestic and foreign researchers’ materials contained in the reviews [1–3, 13–16] showed that the bulk of the publications are devoted to the problems of choosing a raw material base for biofuel production. The physicochemical properties of the selected biomaterials were studied. Methods have been developed for the reactions of esterification and transesterification of fatty acids of plant and animal origin. The influence of nature, quality and quantity of the initial bio-raw material on the output of biofuel is revealed. The mechanisms of transesterification and esterification reactions depending on the type of catalyst used are considered. The necessity of adapting the operation of engines on biofuels is shown. Ecological advantages of using biofuel have been established. However, in all the works, the main material is devoted either to the direct use of vegetable oils as a motor fuel, or to the operation of a diesel engine on biofuel used after deep chemical processing of vegetable oils. Fundamental research [11, 12, 17–22] conducted at the “All-Russian Scientific Research Institute for the Use of Machinery and Products in Agriculture” with oils of vegetable and animal origin, including microalgae, revealed the peculiarities of using bio-raw materials as motor fuel. New features of the reaction mechanism of biofuel synthesis are revealed. It has been established that the physicochemical characteristics of biofuels depend on the ratio of medium- and long-chain radicals of fatty acids, as well as on their degree of unsaturation. It was shown that in order to improve the pour point and turbidity of biofuel in its composition, the fraction of unsaturated acid residues should be increased, but this can worsen its oxidative stability. It is proved that balance is required for the presence of non-saturated acid radicals with one double bond in the biofuel molecules (oleic and palmitooleic acids). The content of linoleic and linolenic acid residues (with two double bonds) should be minimal. It was found that the limiting radicals of short length increase oxidative stability and cetane number, but worsen the low-temperature properties of biofuels. It was shown that changing the fatty acid composition of biofuels by adding separately synthesized low molecular weight saturated esters to it requires an expansion of the raw material base for biodiesel production and a new hardware design for the esterification reaction.

The reviews [14–16] show the development evolution of the of biodiesel technology: from devices with mechanical mixing to rotary pulse-cavitation reactors and bioreactors with a vortex layer of ferromagnetic particles [23, 24], their advantages and significant disadvantages [22]. The work [25] shows promising areas of development for the creation of biofuels.

However, in all known publications, all technologies (mainly at the laboratory level) relate only to the biodiesel production. To date, in the scientific and technical literature there are practically no technologies for producing mixed motor (bio- and petroleum) fuels, and the problems of creating technologies for producing three-component mixed motor fuels have not only not been solved, but still have not been set despite them scientific interest and practical relevance.

Therefore, the aim of this work is to develop a technology for producing three-component mixed
Motor fuels based on a high-speed transesterification reactor under conditions of spontaneous interphase convection caused by the Marangoni effect through a moving phase boundary.

Main Part

The technological scheme for producing a three-component mixed motor fuel can be represented as a set of biodiesel producing units by transesterification reaction; purification of biodiesel from the catalyst and excess methanol; obtaining low molecular weight synthetic esters by the esterification reaction; mixing the obtained components with mineral diesel fuel.

Obtaining of a high molecular weight component

The duration of the transesterification reaction is due to significant differences in the physicochemical properties of vegetable and animal fats and low molecular weight alcohols, which are heterogeneous immiscible liquids forming two different liquid phases separated from each other by a moving phase boundary. At the initial stage, the reaction mass consists of two phases (alcohol and fat). The reaction does not occur in the entire volume of the system, but only in the volume of one of the phases as a result of the diffusion of a component from the other phase into it. The distribution of products between the fat and alcohol phases has not yet been established. Interaction requires the transport of at least one of the reagents through the phase contact surface.

In a first approximation, based on our own and well-known literature data, we assume that in order to intensify the transesterification process, it is necessary, firstly, to create conditions for improving the solubility of phases, and, secondly, to increase the contact area of phases due to the intensity of mixing. An increase in the mixing speed of dissimilar immiscible liquids leads to emulsification. With an increase in the mixing speed, the diameter of the droplets of the emulsion decreases and, accordingly, the surface of the phase contact increases. In this case, the contact surface of the phases inversely depends on the average diameter of the droplets of the dispersed liquid.

The most promising is the use of reactors based on the vortex effect. In a vortex reactor, mixing intensification is achieved due to the interaction of two vortices, which move towards each other along the axis of the apparatus. The peripheral flow rotates according to the laws that describe the motion of a potential vortex. The paraxial flow rotates according to the laws describing the motion of a quasi-solid. Energy transfer from the axial layer to the peripheral layer is carried out mainly due to small-scale turbulent pulsations. It is possible that during this energy exchange a turbulent pulsating effect occurs, which activates the molecules of the initial reagents. This naturally leads to an increase in the rate of the transesterification process. Figure 1 shows the biodiesel synthesis unit.

Methanol from the storage tank E1 by means of a pump enters the mixer for the preparation of a solution of catalyst C1. A catalyst, potassium hydroxide, is introduced into the same apparatus using a screw dispenser D1. In the process of mixing, a methanolic solution of potassium hydroxide is obtained, which is then fed to a C2 mixer for mixing with vegetable oil heated to 60 °C. Cold vegetable oil from the storage tank E2 is fed to the reactor P1 with the help of a pump. The preheated vegetable oil enters the T1 plate heat exchanger, where it is heated by steam to 60 °C, after which it is fed to the C2 mixer, where it is mixed with the catalyst solution. The mixture then enters reactor P1, in which the methanolysis reaction takes place with the formation of a mixture of the target product – biodiesel fuel with a by-product – glycerol and unreacted methanol and catalyst. In addition, a small amount of mono- and triacylglycerols is present in the mixture. The mixture enters the separator C1, where it is divided into glycerol and ester phases.

The glycerin phase containing glycerin, methanol and alkali is collected in a container for crude glycerin E4. The ester phase is fed to a B1 evaporator to remove excess methanol. Methanol vapors from the upper part of the evaporator enter the refrigerator condenser X1 and then return to the methanol storage tank E1.

Figure 2 shows the biodiesel purification unit from the catalyst and excess methanol.

The ester phase is fed to a B1 evaporator to remove excess methanol. Methanol vapors from the upper part of the evaporator enter the refrigerator condenser X1 and then return to the storage tank for methanol E1 (Fig. 1). The ester phase from the bottom of the evaporator enters the apparatus for neutralizing C3, where a phosphoric acid solution is supplied from the mixer C4. The neutralized reaction mass enters the C5 washing apparatus, where the washing water is also dosed. The resulting mixture is fed to separator C2, from where water with the salts of phosphoric acid dissolved in it enter the storage tank E5. The solution can be used, for example, to produce phosphate fertilizers. The ester phase after the separator contains a certain amount of water and other impurities for purification; it is passed through a K5 column filled with a sorbent. Purified ester enters the storage tank E6.
Fig. 1. Technological scheme (a) and its 3D-model (b) for producing a high molecular weight component of the ester composition – biodiesel:

E1 – methanol storage tank; E2 – oil storage tank; E3 – glycerol phase storage tank; D1 – auger batcher for catalyst (alkali); C1 – mixer for preparing a catalyst solution; C2 – mixer for preparing a mixture of a catalyst solution and oil; P1 is a transesterification reactor; S1 is a separator for separating the glycerin phase; T1 is a heat exchanger for heating vegetable oil; Streams: 1 – methanol from the evaporator after condensation or from the tank; 2 – hard alkali; 3 – alkali solution in methanol; 4 – vegetable oil; 5 – water vapor for heating the oil; 6 – condensed water; 7 – reaction mass after the reactor; 8 – glycerol phase; 9 – ester phase (crude biodiesel)

For the evaporation of methanol, a film-type recuperative evaporation apparatus B1 is used. The film of the reaction mixture flows down on a heated surface, the temperature of which increases in the direction of travel from top to bottom. After evaporation through special channels in a heated surface, hot ester moves from bottom to top, transferring the acquired thermal energy to the evaporated solution (heat recovery).

Biodiesel is dried in a K1 column filled with a sorbent, for example, silica gel. To regenerate silica gel, it is calcined.

**Obtaining a low molecular weight component**

Figure 3 shows the synthesis block of a low molecular weight component. Acid and alcohol from the respective storage tanks 2 and 3 by means of
matering pumps enter the apparatus 1 filled with a solid catalyst. In the apparatus, the temperature reaches 105–110 °C. The water formed by the esterification reaction in the composition of the azeotropic mixture with alcohol evaporates, and then is sent for condensation to the apparatus 4. The separated water is sent to the steam production system, and the alcohol enters the storage tank 2. The synthesized ester is settled and cooled, and then passed through anion exchange purification apparatus 5 with anion exchange resin AB-17 or AB-16 in the OH form for neutralization and for separating unreacted acid and alcohol (after condensation, they are sent to storage tanks 2 and 3, and the final product, low molecular weight ester, enters the storage tank 6.

**Compounding unit for fuel components**

In the final stage of the technological process, compounding of a high molecular weight and low molecular weight component with mineral diesel fuel is carried out (Figure 4).

At the final stage of the preparation of a three-component mixed motor fuel, diesel fuel from the storage tank 1 and a low molecular weight component from the tank 3 enter the mixer 7. The resulting mixture simultaneously with a high molecular weight component is fed into a mixer 8, into which, if necessary, a polyfunctional additive 2,2',4'-trihydroxyazobenzene is added for compounding. For better mixing of the primers in a high molecular weight component, the latter is heated in a vortex apparatus 10. The required flow rates of the components are regulated using metering pumps 6. After mixing, the finished product through the issuing unit 11 enters the storage tank E11 (in Fig. 4b this capacity is not shown conditionally, since it is not part of the compounding unit) and then to the consumer.

The main difference between the proposed technological process and the known ones is the optimal composition of the three-component mixed motor fuel. Until now, only two-component fuel has...
Fig. 4. Technological scheme (a) and 3D-model (b) of the compounding block of the components of mixed motor fuel:

1 – The tank for diesel fuel; 2 – The reservoir for the macromolecular component; 3 – The reservoir for the low molecular weight component; 4 – Compressor; 5 – Control panel; 6 – pump unit; 7 – Block for pre-mixing mineral diesel fuel with a low molecular weight component; 8 – Block for the final compounding of a mixture of mineral diesel fuel and a low molecular weight component with a high molecular weight component; 9 – The reservoir for the multifunctional additive 2,2’,4’-trihydroxyazobenzene; 10 – Vortex apparatus for heating a high molecular weight component; 11 – Block issuing finished products; 12 – Frame

been used in research practice: either a mixture of vegetable oils with diesel fuel, or esters of these oils with mineral fuel. At the same time, the closest approximation of the properties of biofuels to the properties of petroleum diesel fuel can be achieved by using multicomponent biofuels. By optimizing the composition of these fuels, it is possible to provide the best indicators of fuel efficiency and toxicity of diesel exhaust gases. This is the main advantage of the proposed technological mode in comparison with the existing ones. But at the same time, it is necessary to take into account the real distribution of the modes of their operation under operating conditions. During operation, diesel engines can operate in various modes, each of which is characterized by the speed of the crankshaft and the load on the engine. Moreover, when agricultural machinery performs the main technological operations, the distribution of the operation modes of their engines differs significantly from the operating modes of diesel engines of vehicles operated in conditions of heavy urban traffic [9].

As an example, confirming the efficiency and effectiveness of the proposed solution, we note the following experimental data. The optimal composition of the three-component mixed motor fuel has been established: the first component is mineral diesel fuel (40 vol. %); the second component is biodiesel (50 vol. %) and the third component is the lightest low molecular weight component (10 vol. %). When using this fuel, the specific mass emissions of the NOx nitrogen oxides in the 13-step test cycle decreased from 5.948 to 5.742 g/(kWh), i.e. by 3.5 %, of carbon monoxide CO – from 2.782 to 1.949 g/(kWh), or 29.9 %, of unburnt CH hydrocarbons – from 1.006 to 0.784 g/(kWh), or 22.1 %. At the same time, a slight decrease in the conditional effective engine efficiency \( \eta_{\text{conv}} \) by 1.7 %. In general, the conducted studies have confirmed the possibility of efficient use of three-component mixed motor fuel as fuel for domestic diesel engines.

**Experimental**

The synthesis of biodiesel is carried out by the reaction of methanolysis of vegetable oil in the presence of potassium hydroxide as a catalyst, followed by separation of the biodiesel phase and the glycerol phase. Biodiesel is further subjected to neutralization, washing, drying and filtering operations. The raw materials for the synthesis were the following types of vegetable oils: rapeseed, linseed, corn, sunflower, camelina and radish oil. Physico-chemical properties of biodiesel were determined in accordance with GOST R 52368-2005. “Dieselfuel EURO. Technical conditions”.

Low molecular weight esters are synthesized by an esterification reaction in the presence of sulfuric acid as a catalyst, followed by neutralization and washing with water. The synthesized esters are then subjected to neutralization, washing and distillation operations.

Physico-chemical properties of the esters were determined in accordance with GOST R 52368-2005. The vibrational spectra were recorded using an Infralum FT-801 IR Fourier spectrometer.
The fatty acid composition of biodiesel was determined using a Crystallux-4000M chromatograph according to GOST 30418-96 “Vegetable oils. Method for determining the fatty acid composition”, for processing the results of the chromatographic analysis, the software “Chromatek-Analyst” was used. The calculation was performed using the software product Excel (Microsoft).

The analysis of exhaust gas toxicity and fuel economy of a diesel engine was carried out on a D-245.12C type diesel engine (4 ЧН 11/12.5). Engine tests were carried out on the 13-speed cycle and external speed characteristics.

Obtaining a three-component mixed motor fuel consists in mixing mineral diesel fuel with biodiesel and low molecular weight ethers.

Results and Discussion

A quantum-chemical calculation of the triacylglycerol molecule was carried out, which suggested the direction of attack of the nucleophilic reagent. It is known that the preferred direction of nucleophile attack during hydrolysis of triacylglycerols in vivo in the presence of enzymes is the carbonyl carbon atom in the α-position. By analogy, they describe the mechanism of nucleophilic substitution in the non-enzymatic in vitro methanolysis reaction. But the features of the in vivo hydrolysis process are determined, first of all, by the structure of the active center of the enzyme molecule. In the absence of an enzyme, the reaction mechanism may be different. Calculations showed that the largest positive charge in the 1-oleyl-2-linoleyl-3-linolenylglycerol molecule is attributable to the carbonyl carbon atom of linoleic acid, which is in the position (0.318), it is lower for α-carbonyl carbon atoms (0.295). The calculation of the geometry of the molecule showed that the β-carbonyl atom is preferable for attack, and for spatial reasons. Double bonds in the radicals of the unsaturated acids of triacylglycerols of vegetable oils are in a less stable configuration 2. In the course of various chemical reactions in which triacylglycerols of vegetable oils can enter (for example, hydrogenation), a side reaction of isomerization can occur, in which the molecule transforms into a more stable transconfiguration. Clarification of the configuration of double bonds and the likelihood of an isomerization reaction can be done using vibrational spectroscopy. The presence of a double bond in radio channels in the transconfiguration can be confirmed by vibrations to which absorption bands in the range of 980–960 cm\(^{-1}\) correspond. The IR spectrum of rancid sunflower oil contains distinct intense bands at 3009 cm\(^{-1}\), which can be attributed to stretching vibrations of the carbon – hydrogen bond, and vibrations in the region of 721 cm\(^{-1}\) can be identified as non-planar deformation vibrations of the double bond in cis configuration. The biofuel spectrum contains the same bands characteristic of the cis configuration; there are no oscillations in the interval 980–960 cm\(^{-1}\). It can be concluded that in the process of methanolation, the mutual conversion of geometric isomers does not occur. An additive to biodiesel fuel – saturated esters of small molecular weight, was synthesized in the usual way by the esterification reaction:

\[ R_1\text{COOH} + R_2\text{OH} \leftrightarrow R_1\text{COOR}_2 + \text{H}_2\text{O}, \]

where \(R_1\) is a saturated acid radical; \(R_2\) is an alcohol radical.

Homogeneous catalysts can accelerate the reaction, the most common of which is sulfuric acid or heterogeneous catalysts, for example, ion-exchange resins. Starting alcohols and carboxylic acids for the esterification reaction can be obtained using products of petrochemical or various biotechnological industries. For example, aliphatic alcohols of small molecular weight can be isolated by distillation of fusel oils, acids with 4–6 carbon atoms can be synthesized as a result of fermentation or biological oxidation. A mixture of esters should be balanced in composition. To approximate the properties of biodiesel to the characteristics of petroleum diesel fuel, it is necessary first of all to reduce the temperature of the beginning of boiling and distillation of the initial fractions. Among saturated esters of small molecular weight with a suitable boiling point (160–190 °C), one should choose those with a density close to that normalized for diesel fuel (860 kg/m\(^3\)). For example, isoamyl butyrate (TKIP = 178 °C, δ = 865 kg/m\(^3\)) can be used to lower the boiling point of the fuel additive. To lower the distillation temperature of the initial fractions, octyl butyrate, octyl and heptylvalerates, heptylcaproate can be used. Their boiling points are in the range from 242 to 260 °C and the density (at 20 °C) is close to the density of diesel oil – 863–858 kg/m\(^3\). Biodiesel has a higher viscosity compared to petroleum. Therefore, at close boiling temperatures, it is preferable to use ester with a lower density. So, octyl butyrate and heptylvalerate differ only by 1 degree in boiling point, and the viscosity of butyric acid ester (1.39 mm\(^2\)/s) is almost 2 times lower than that of valerianic acid ester (2.75 mm\(^2\)/s), therefore its usage is preferable. The authors of this study obtained a number of saturated esters by the esterification reaction between such acids as butyric, valerianic, caproic, and saturated...
alcohols containing from 5 to 9 carbon atoms. The synthesized esters and biodiesel derived from non-edible sunflower oil were mixed in different ratios, obtaining mixtures of esters (SE) containing from 30 to 90% biodiesel and from 2 to 10% of various low molecular weight ethers.

**Conclusion**

A mixture of esters with diesel fuel, containing 10% of saturated molecular weight esters of low molecular weight and 50% of biodiesel, has physicochemical characteristics that meet the technical requirements for diesel fuels. Such a fuel improves the environmental properties of a diesel engine, its cetane number and lubricating properties. Using a quantum chemical calculation, it was determined that in the process of methanolation, the most likely direction of attack of a nucleophilic reagent on a triacylglycerol molecule is a β-carbonyl carbon atom. It was found that during the chemical reaction, the processes of cis-trans isomerization of double bonds do not occur in the radicals of unsaturated carboxylic acids, which are part of both the triacylglycerols of vegetable oil and the composition of biodiesel fuel molecules.

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