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CHANGES IN DENSITY AND VISCOSITY OF BIODIESEL FUEL AND ITS COMPOSITIONS DURING STORAGE

Research article

Abstract

The dependence of the physicochemical characteristics of biodiesel fuel synthesized from camelina oil and from linseed oil on the storage time in a metal tank and polyethylene tank was studied. Both the density and viscosity of biodiesel fuel increase during storage, especially in a metal tank. Azobenzene derivatives containing two hydroxyl groups in ortho positions to the azo group and electron-donating functional groups in aromatic rings were offered as antioxidant additives.

When storing compositions prepared from petroleum diesel fuel and camelina oil methyl esters, their delaminaton occurs, probably due to weak intermolecular interactions between petroleum hydrocarbons and biodiesel molecules. The most stable compositions are compositions with 50–70% of biodiesel fuel.

Keywords: biodiesel fuel, density, kinematic viscosity, fuel compositions, additives.

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ИЗМЕНЕНИЕ ПЛОТНОСТИ И ВЯЗКОСТИ БИОДИЗЕЛЬНОГО ТОПЛИВА И ЕГО КОМПОЗИЦИЙ ПРИ ХРАНЕНИИ

Научная статья

Аннотация

Изучена зависимость физико-химических характеристик биодизельного топлива, синтезированного из масла рыжика и масла льняного семени, от времени хранения в металлических и полиэтиленовых резервуарах. Плотность и вязкость биодизельного топлива в процессе хранения увеличиваются, особенно в металлическом резервуаре. Предложены производные азобензола, содержащие 2 гидроксогруппы в ортоположении к азогруппе и электронодонорные функциональные группы в ароматическом кольце как антиоксиданты.

При хранении композиций, полученных из нефтяного дизельного топлива и метиловых эфиров рыжикового масла, происходит их расслоение, предположительно из-за слабых межмолекулярных взаимодействий между нефтяными углеводородами и молекулами биодизеля. Наиболее стабильными являются композиции с 50–70% биодизеля.

Ключевые слова: биодизельное топливо, плотность, кинематическая вязкость, топливные композиции, присадки.

1. Introduction

Concern for our own food security, first of all, makes us think about increasing the volume of the agricultural production. At present, this is no longer possible with extensive methods. It is necessary to develop new intensive methods and technologies, to increase the level of the mechanization of the agricultural work, and, consequently, the volume of fuel used for internal combustion engines. An equally important aspect is the ecological purity of the grown products.

Hydrocarbon fuel is known to be one of the sources of the environmental pollution, which also affects the environmental characteristics of agricultural products. Widely advertised electric motors have so far only seen some use in passenger cars and are not used in agricultural machinery. However, the environmental friendliness of electric vehicles is currently a controversial issue.

Electric cars do not have exhaust gases containing harmful components. But these components are emitted into the atmosphere by thermal power plants that generate electricity for such cars. Battery disposal is a rather complex and environmentally hazardous process. The high mass of batteries leads to increased wear of the road surface and tires of cars. It increases the particulate matter emissions that are absorbed by a person when breathing. Also, it creates the prerequisites for asthmatic and cardiovascular diseases. If batteries are used in agricultural machinery, their large mass will lead to the soil compaction and reduced crop yields. A large mass of gas cylinders is, by the way, one of the obstacles to the spread of gas-powered engines in the agricultural sector.

At the present stage, it is impossible to abandon internal combustion engines. International and Russian standards and technical regulations that determine the performance characteristics of diesel fuel are increasingly tightening the requirements, for example, for the content of aromatic and sulfur-containing compounds. Therefore, Russian oil refineries are modernizing equipment and introducing new technologies for hydrodesulfurization and dearomatization [1], [2], [3]. The resulting diesel fuels meet increasingly stringent environmental requirements, but at the same time they have a low cetane number and unsatisfactory lubricating properties (primarily due to the removal of organic sulfur-containing compounds during hydrotreatment) [4]. And the improvement of the design of diesel engines increases the requirements for diesel fuels precisely for these indicators.

The quality of the fuel may deteriorate during storage due to the ongoing processes of oxidation, polymerization and coagulation, which increases the risk of breakdown of expensive equipment running on such fuel.

One of the obvious ways to solve the problem is the introduction of additives. The production of modern highly environmentally friendly fuels is impossible without their use. It is desirable that the additives used be polyfunctional, i.e., affect several characteristics of the fuel. The additive that increases the cetane number and lubricity of the hydrotreated fuel can be biodiesel produced from renewable plant resources. Biodiesel fuel has higher environmental characteristics. It practically does not contain sulfur and aromatic compounds, emits as much carbon dioxide during combustion as the plant absorbed during its growth, and promotes more complete combustion of fuel and reduces carbon monoxide and soot emissions. It is rather quickly decomposed by microorganisms during spills [5], [6]. But the molecules of this type of fuel contain radicals of unsaturated higher aliphatic acids. The presence of a reactive π-bond in such radicals increases the possibility of oxidation and polymerization reactions occurring during storage.

The change of the physicochemical characteristics of both biodiesel fuel and fuel compositions, including petroleum and biodiesel fuels, in the course of storage has not been fully studied to date.

The purpose of this work is to study the dependence of the physicochemical characteristics of biodiesel fuel and fuel compositions on the storage time.

2. Methods

Camelina and linseed oils were used as raw materials for the synthesis of biodiesel fuel. A transesterification reaction was carried out with methyl alcohol in the presence of potassium hydroxide as a homogeneous catalyst using methods of organic synthesis. The estimation of the physicochemical characteristics of biodiesel fuel and fuel compositions was carried out in accordance with the requirements of GOST R 52368-2005 and GOST 33131-2014. Apel AP101 photoelectric colorimeter was used to measure the optical density of the samples.

To carry out quantum-chemical calculations, the semiempirical method MNDO was used, which makes it possible to calculate the equilibrium geometry, energy and distribution of electron density in complex organic molecules.

3. Results

The physicochemical properties of any organic compounds depend on their composition. Biodiesel fuel is a mixture of esters of methanol alcohol and aliphatic acids, the molecules of which contain from 14 to 24 carbon atoms. Acid radicals can be saturated and unsaturated (containing up to four double bonds). If biodiesel is derived from vegetable oils, unsaturated radicals predominate. The biofuel under study, synthesized from camelina oil (FAME 1), contains mainly methyl esters of linoleic acid (C18:2) with 18 carbon atoms and two double bonds (32%), and erucic acid (C22:1) with 22 carbon atoms and one double bond (42%). The proportion of other acids with a long carbon chain (C22:0; C22:2; C24:0; C24:1) is also quite large - about 16% in total. There are relatively few acids with a number of carbon atoms from 14 to 16 (slightly less than 4%); the remaining 6% are stearic (C18:0) and oleic (C18:1) acids. The heavy molecular composition leads to high values of physicochemical characteristics. Viscosity at 40°C is 6,38 mm^2/s, and density at 15°C is 895 kg/m^3.

Biodiesel fuel synthesized from linseed oil (FAME 2) practically does not contain very long chain fatty acids: only 2% of arachidonic acid (C20:4). The largest share falls on linoleic (C18:2) and linolenic (C18:3) acids - 24 and 47%, respectively. Myristic and palmitic acids (C14:0) and (C16:0) account for a total of 16%, and the remaining 11% are for stearic (C18:0) and oleic (C18:1) acids. Differences in the molecular composition determine the difference in physicochemical characteristics: viscosity at 40°C is 4,44 mm^2/s, and density at 15°C is 890 kg/m^3.

It can be seen that biodiesel fuels contain many unsaturated compounds: 89% for methyl esters FAME 1 and 81% for FAME 2, while the proportion of polyenoic acids (containing from two to four double bonds in the radical) is also very high - 82 and 73%, respectively. Such acids easily enter into oxidation and polymerization reactions, which can lead to deterioration of the properties of biodiesel fuels during storage.

Figures 1 and 2 show the results of changes of density (15°C, kg/m^3) and kinematic viscosity (40°C, mm^2/s) of biodiesel fuel synthesized from camelina and flax oils during storage for 15 months.

According to the data obtained, both the density and viscosity of biodiesel fuel increase during storage, especially in a metal tank. Iron cations, probably, play the role of a catalyst for oxidative processes.

The main processes leading to a change in the physicochemical characteristics of fuel during storage are oxidation processes. This is confirmed by the results of storing biodiesel fuel in conditions of almost complete absence of oxygen in

tightly closed bottles. During 12 years of storage, the viscosity of biodiesel fuel synthesized from linseed oil (with a high content of the linolenic acid radical) increased by only 1 unit. If oils with a lower content of polyunsaturated acid radicals were used for the synthesis of biodiesel fuel, the change in viscosity is even smaller: for fuel from corn oil it is 0,4 units, and for fuel from sunflower oil – only 0,2 units.

The viscosity of biodiesel fuel, normalized at 40°C, should be 3,5–5,0 mm^2/s. The viscosity of FAME 2 remains within the normalized limits for 7 months in a polyethylene tank and only for 4 months in a metal one. The viscosity of FAME 1 is initially higher than that required by GOST. Therefore, it can only be used in conjunction with petroleum diesel fuel as a component of a fuel composition with high environmental and performance characteristics.

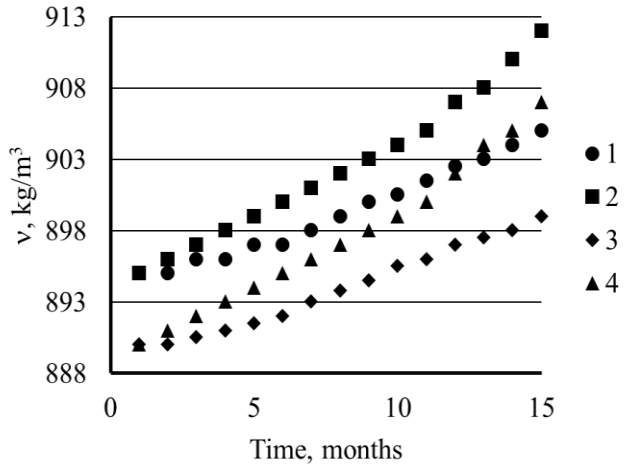


Fig. 1 – Kinematic viscosity, mm^2/s of biodiesel fuel:
1 – FAME 1 when stored in a polyethylene tank; *2* – FAME 1 when stored in a metal tank;
3 – FAME 2 when stored in a polyethylene tank; *4* – FAME 2 when stored in a metal tank

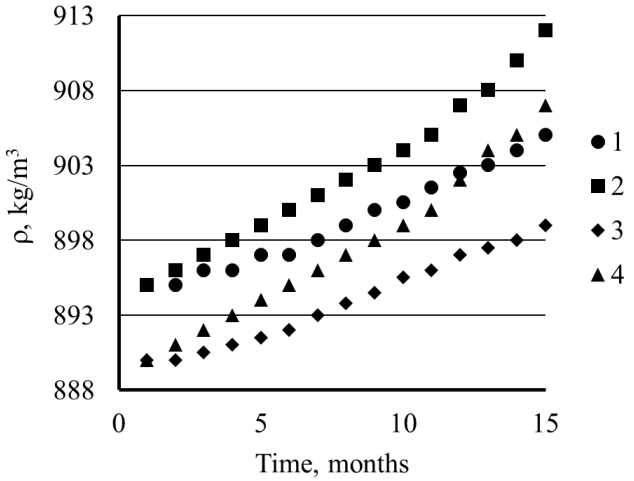


Fig. 2 – Density, kg/m^3 of biodiesel fuel:
1 – FAME 1 when stored in a polyethylene tank; *2* – FAME 1 when stored in a metal tank;
3 – FAME 2 when stored in a polyethylene tank; *4* – FAME 2 when stored in a metal tank

The density of biodiesel fuel should be in the range from 860 to 900 kg/m^3 in accordance with GOST. So, fuel containing heavy components can be stored in a polyethylene tank for 7–8 months, and in a metal tank – no more than 5 months.

The increase in the density and viscosity of biodiesel fuel is due to the fact that the ester molecules of biodiesel fuel are attacked during storage by reactive oxygen species: peroxide or hydroperoxide radicals are formed in the fuel. Probably, the radical attacks the carbon atom in the α-position to the carbon of a double bond. Such direction of attack makes it possible to obtain a new radical as a result, while the lone pair electron is conjugated with the π-electron density of the double bond. It stabilizes the resulting radical particle (Fig. 3).

Further, the oxidation process can proceed in two directions (Fig. 3, paths *a* and *b*) with the production of unstable hydroperoxides. In turn, hydroperoxides decompose with the formation of aldehydes. The aldehyde group readily undergoes further oxidation to form carboxylic acids. The end products may be monocarboxylic acids or dicarboxylic acid methyl esters with shorter carbon chains.

Metal cations present on the surface of walls of metal tanks contribute to the formation of radicals according to the equations 1–3 [7]:

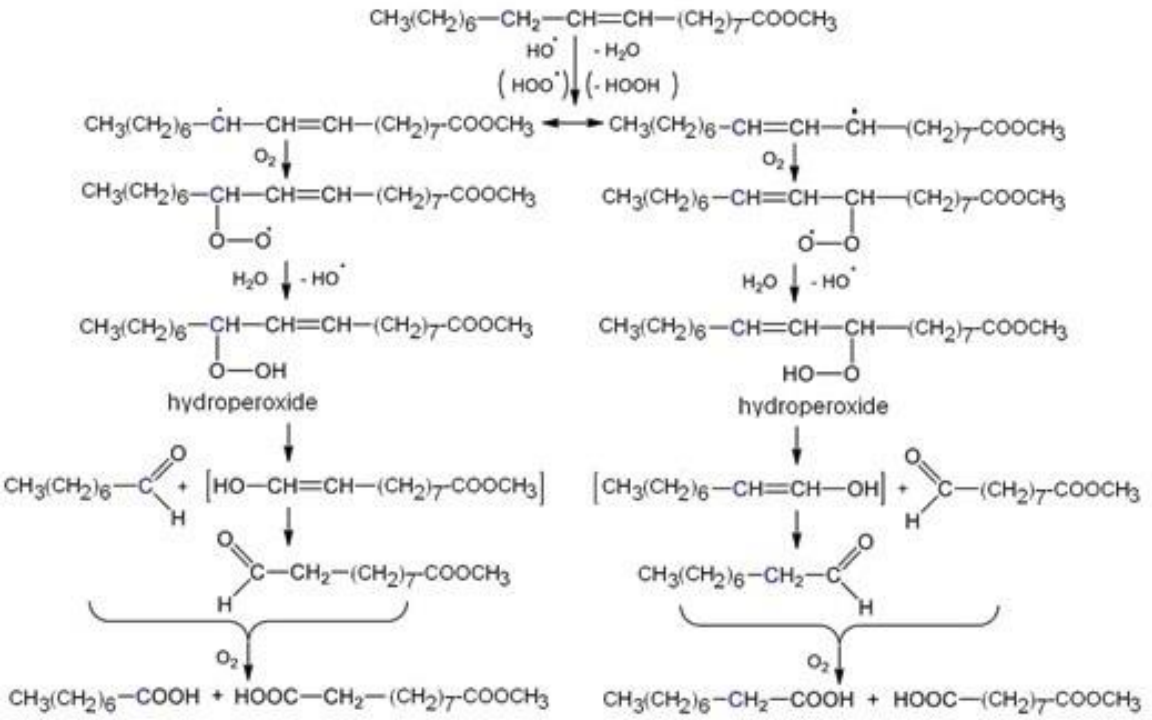
$$ROOH + Me^{n+} \longrightarrow RO^{\cdot} + OH^{-} + Me^{(n+1)+} \quad (1)$$

$$ROOH + Me^{(n+1)+} \longrightarrow RO_2^{\cdot} + H^+ + Me^{n+} \quad (2)$$

$$2ROOH \longrightarrow RO^{\cdot} + RO_2^{\cdot} + H_2O \quad (3)$$

In order to increase the stability of biodiesel fuel during storage in a metal tank, it is advisable to introduce additives that can stop radical reactions and, preferably, bind metal cations, because the fuel is stored mainly in metal tanks.

Antioxidant additives for petroleum fuels are widely known. Hindered phenols or amines are most commonly used [8]. They react with radicals to form inert reaction products. The resulting radical conjugates with the developed aromatic π-electron system of phenol. The radical formed as a result of the reaction is stabilized to such an extent that it loses its reactivity.



a *b*

Fig. 3 – Scheme of peroxide breakdown of the biodiesel fuel component (oleic acid methyl ester): *a* – path A; *b* – path B

It is advisable to use compounds of a similar structure for biodiesel fuel. At present, not very many such additives are offered, for example, bisphenol, phenylenediamines, 2,6-di-tert-butylhydroxytoluene, and ionol are suggested for use [9], [10].

We offer compounds, which can also be attributed to sterically hindered phenols, as antioxidant additives. These are azobenzene derivatives containing two hydroxyl groups in ortho positions to the azo group and additional functional groups in aromatic rings. They have a developed π-electron system and can also form complexes with transition metal cations. The best results are shown by compounds containing electron-donating functional groups in aromatic structures, for example, trihydroxyazobenzene.

The action of the additive was evaluated by the method of accelerated oxidation in the presence of a copper plate at an elevated temperature. The copper cation is an even more effective catalyst for oxidative reactions than the iron cations. The introduction of the additive improves the chemical and physical stability of both petroleum and biodiesel fuels during storage.

So, the density of biodiesel fuel without additives during the experiment increased by 6 units, and in the presence of additives - only by one. The increase in the fuel viscosity without the additive was 19%, and in the presence of the additive it was 8%. No precipitate was formed in the biodiesel fuel in the presence of the additive. That is, it prevents not only oxidative, but also coagulation processes.

As has been shown, biodiesel fuel containing high-molecular-weight components with a high degree of unsaturation can be used as a part of a fuel composition.

Fuel compositions B10, B20, B50, and B70 were prepared as mixtures of petroleum diesel fuel and camelina oil methyl ester (FAME 1), with the content of esters, respectively, 10%, 20%, 50% and 70% (vol.). Glass cylinders with fuel compositions were kept at a room temperature in a dark room and in the light with access to oxygen. The fuel compositions were periodically examined, and the observed changes were recorded.

After 20 days, in composition B10, which was stored in the light, the initially homogeneous system began to separate. A layer of light-yellow liquid was formed in the lower part of the cylinder. It is probably a biodiesel fuel, the density of which is much higher (892 kg/m^3 at 25°C) than oil fuel (815 kg/m^3, respectively). The volume of exfoliated biodiesel fuel was approximately 15% (vol.) of the amount introduced into the B10 composition. All other compositions stored both in the light and in the dark, remained homogeneous.

After 60 days, the amount of biodiesel fuel exfoliated in the composition B10 increased to 20%. Delamination appeared in the composition B20, which amounted to 5% of the amount initially introduced into the mixture. The remaining samples have not undergone any visible changes by this time. By the end of the third month of storage in the light, the composition B10 already contained 30% of biodiesel fuel as a separate layer, and the composition B20 contained 10%. After 5 months of storage in the light, the amount of delaminated FAME 1 was 50% and 40% in the compositions B10 and B20, respectively. It increased to 70% and 50% by the end of the eighth month. The composition B50 turned out to be more stable; delamination became noticeable only after 4 months of storage. 4% of biodiesel fuel was separated by the end of the fifth month, and 6% - after eight months.

The composition B70 proved to be the most stable; no delamination was observed during storage in the light and in the dark for 8 months. Delamination in samples stored in the dark became noticeable only after five months of storage. For the compositions B10 and B20, the amount of delaminated FAME 1 was 20% and 10%, respectively. By the end of the eighth month, it increased to 50 and 40%, respectively, and separation became noticeable in the B50 composition (about 3%).

Commercial petroleum diesel fuel acquired an intense yellow color during the fifth month of storage, and a layer of red-brown sediment formed at the bottom. When stored in the dark, no color change is observed during the entire storage time. The optical density of this fuel was 0,094 at 420 nm and 0,062 at 460 nm before the start of the experiment. After five months of storage it was 0,232 and 0,160 at 420 nm and at 460 nm, respectively. The density and viscosity of oil fuel changed significantly in 5 months. The density at 25 °C increased from 816 to 823 kg/m^3, and kinematic viscosity at 40 °C - from 2,36 to 2,71 mm^2/s. The deepening of the color and the increase in the density and viscosity are probably associated with the occurrence of oxidation and polymerization processes. The formation of a precipitate can have two main reasons - the processes of the coagulation of the oxidation products, and the precipitation of numerous additives. In [11], the authors observed the process of redistribution of the additive along the height of the tank; the lubricity of modern hydrotreated fuel taken from the top, from the middle, and from the bottom of the tank differed markedly after 60 days of storage (the average wear scar diameter was 294, 325, and 390 µm, respectively). Perhaps, in our case, we observed the combined effect of these factors. It should also be taken into account that petroleum fuel is a colloidal system, where the dispersion medium is saturated and unsaturated hydrocarbons, and the dispersed phase is natural high-molecular heteroatomic compounds, additives and water. Only weak intermolecular interactions are possible between nonpolar compounds.

Biodiesel fuels are also colloidal oleodispersed systems. Their molecules have a long non-polar hydrocarbon radical and a low-polarity ester group, i.e., belong to amphiphilic compounds.

The presence of only weak interactions in diesel and biodiesel fuels is confirmed by quantum-chemical calculations of the strength of bonds formed between fuel molecules. To do this, we calculated the total energy of a system consisting of two fuel molecules in two cases: when the distance between the molecules was comparable and incomparable with the length of the covalent bond. The difference between these energies was used to judge the strength of intermolecular interaction. The decane molecule was used to simulate petroleum fuel, and the methyl oleate molecule was used for biodiesel fuel. Figure 4 shows the ball-and-stick models of these complexes, and Table 1 shows the results of calculating the total energy of the system.

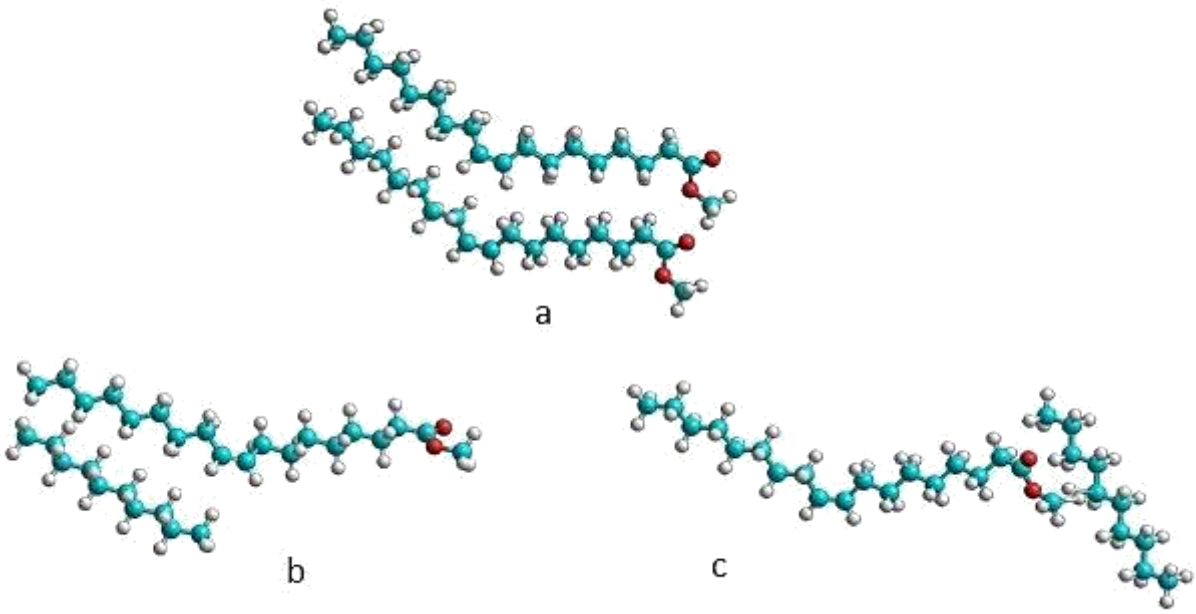


Fig. 4 – Ball-and-stick models of complexes:
a – methyl oleate – methyl oleate; *b* – and; *c* – methyl oleate – decane

Table 1 – The value of the total energy of the system of two fuel molecules at distances comparable (E1) and incomparable (E2) with the bond lengths

Fuel molecule	E_1, kcal/mol	E_2, kcal/mol	ΔE, kcal/mol
Decane - decane (petroleum diesel fuel)	– 73142,9	– 73141,5	1,4
Methyl oleate– methyl oleate (biodiesel)	– 164790,0	– 164788,0	2,0
Methyl oleate – decane (fuel composition)	– 118966,0	– 118965,0	1,0

The data obtained clearly show that the interaction between oil fuel molecules is very weak. Indeed, only one type of interaction is possible between non-polar hydrocarbon molecules. These are van der Waals forces. The orientational component of the van der Waals forces is proportional to the magnitude of the dipole moment to the fourth power, and the induction component is proportional to the second power. Oxidation products are polar compounds with a noticeable dipole moment; therefore, their formation significantly affects intermolecular interactions. Stronger bonds are possible between polar oxidation products, such as electrostatic and hydrogen bonds. Their formation leads to the destruction of the colloidal structure of the fuel and the sediment formation. Therefore, the formation of a precipitate during storage of petroleum fuel in the light is observed.

The interaction forces are slightly greater between the molecules of biodiesel fuel, because the main part of the molecule is also non-polar. The increase in the strength of interaction probably occurs due to the interaction of ester groups with a certain polarity.

The mixing of non-polar hydrocarbons of petroleum fuels and amphiphilic molecules of biodiesel esters when creating fuel compositions leads to the appearance of weak hydrophobic interactions due to non-polar radicals in the biodiesel molecule. There is probably no interaction between the ester group and the non-polar fuel oil molecule, so for the fuel composition the calculation gives the smallest interaction value. That is, fuel compositions are unstable colloidal systems. The occurrence of oxidative processes with the formation of more polar products worsens the intermolecular interaction even more, which leads to the separation of fuel compositions, primarily those where the content of biodiesel fuel is low.

4. Conclusion

The density and viscosity of biodiesel increase when it is stored in both polyethylene and metal tanks. These physico-chemical characteristics remain within the normalized limits for 7 months in a polyethylene tank, and only for 4–5 months in a metal tank.

The additives that belong to the series of sterically hindered phenols containing an azo group have been proposed for the stabilization. The density and viscosity increase in the presence of trihydroxyazobenzene during storage, but to a lesser extent than in fuel without additives. Additives also slow down the coagulation processes.

Biodiesel fuel, which contains high-molecular components with a high degree of unsaturation, has a high viscosity, so it is advisable to use it as part of a fuel composition. When storing fuel compositions, their delaminaton occurs. The compositions with a low content of biodiesel fuel (10–20%), stored in the light, are delaminated first. The compositions of 50–70% biodiesel are more stable.

The delamination may be due to weak intermolecular interactions between petroleum hydrocarbons and biodiesel molecules containing polar ester groups, as well as with the destruction of the colloidal structure of fuels.

Conflict of Interest

None declared.

Конфликт интересов

Не указан.

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