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Preparation of diesel fuel blends and study of their physical properties

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Abstract: The aim of the present work was to synthesize biodiesel fuels from sunflower oil through a transesterification reaction in the presence of a novel catalytic system. Under the molar oil-to-methanol ratio of 1:3 and the temperature of 55 °C, the product yield was 83 %. Physical properties of the biodiesel and diesel fuels under study, as well as their blends containing 20 and 50 vol% of biodiesel fuel (B20 and B50), were investigated against ASTM standards. An increase in the proportion of biodiesel fuel increases both the density of the B20 and B50 blends, as well as their kinematic viscosity, which remains within the 2–5 mm²/s range at 40 °C thus meeting ASTM requirements. It is shown that the content of unsaturated compounds in the biodiesel blends altered within the range specified by ASTM. The flash temperature of the B20 and B50 biodiesel blends was shown to be higher than that of diesel fuel. This led to some deterioration in their flammability, at the same time as making the transportation and storage of these fuels safer. The sulphur content in the biodiesel blends under study decreased significantly with an increase in the biodiesel content: from 50 m.c. in diesel fuel up to 27 m.c. in the B50 blend. The use of such biodiesel blends reduces the content of sulphur oxides in exhaust gases, which has a beneficial effect on the environment and human health. The use of the proposed catalytic system reduces the amount of foam produced by biodiesel washing, thus simplifying its synthesis. Due to the absence of the neutralization stage, the described catalytic system can be reused after water removal through distillation.

Keywords: biodiesel, transesterification reaction, ASTM standards, new catalytic system, cetane number

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Приготовление дизельных смесей и исследование их физических свойств

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Резюме: Целью работы являлся синтез биодизельного топлива из подсолнечного масла по реакции переэтерификации в присутствии новой каталитической системы. При молярном соотношении масла к метанолу 1:3 и температуре 55 °C выход продукта составил 83 %. В соответствии со стандартами ASTM были исследованы важные физические свойства биодизельного и дизельного топлив, а также их смесей, содержащих 20 и 50 % (об.) биодизельного топлива (B20 и B50). Плотность смесей B20 и B50 растет с увеличением доли биодизельного топлива. Кинематическая вязкость смесей с ростом содержания биодизельного топлива незначительно увеличивается, оставаясь в пределах 2–5 мм²/с при 40 °C, что соответствует требованиям ASTM. Как показали результаты исследования, содержание ненасыщенных соединений в биодизельных смесях находилось в пределах, нормируемых ASTM. Температура вспышки для биодизельного топлива, смесей B20 и B50 выше, чем у дизельного топлива. Это приводит к некоторому ухудшению их воспламеняемости, но, с другой стороны, делает транспортировку и хранение этих видов топлива более безопасной. Содержание серы в биодизельных смесях значительно уменьшается с ростом содержания биодизельного топлива: от 50 м.д. в дизельном топливе до 27 м.д. в смеси B50. Использование таких биодизельных смесей

снижает содержание оксидов серы в выхлопных газах, что благоприятно сказывается на состоянии окружающей среды и здоровье человека. Использование новой каталитической системы позволяет снизить объем пены, образующейся при промывке биодизеля, что упрощает технологический процесс его синтеза. Благодаря отсутствию стадии нейтрализации применяемая каталитическая система может быть использована повторно после удаления промывной воды методом дистилляции.

Ключевые слова: биодизельное топливо, реакция переэтерификации, нормы ASTM, новая каталитическая система, цетановое число

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INTRODUCTION

Emissions from vehicles (carbon dioxide, sulphur oxide and other toxic gases resulting from fuel burning) are the main contributing factors to such environmental problems as the growing ozone hole, acid rains, accumulation of toxic compounds in the atmosphere and the greenhouse effect. Another serious problem is the depletion of existing fossil fuel resources [1–4].

Issues associated with reducing oil reserves and environmental pollution underpin the search for renewable and environmentally friendly alternative energy sources. Among promising fuels for diesel engines are biodiesel blends derived from vegetable oils and animal fats through the reaction of transesterification. Transesterification is the interaction of oil triglycerides with alcohols having a small molecular mass in the presence of various catalysts followed by the formation of a mixture of fatty acid alkyl esters and glycerine as a by-product.

Potential advantages of pure and blended biodiesel fuels include their biodegradability, higher cetane numbers, low emissions, as well as improved lubricating capacity leading to an increased service life of engines. Since biodiesel fuels contain no aromatic, nitrogen and sulphur compounds, their burning produces no toxic oxides responsible for serious environmental problems [5–17].

Biofuel production is steadily increasing. While the share of biofuels in the global production of fuel for road transport was 1 % in 2004, the International Energy Agency (IEA) has predicted its increase up to 7 % by 2030 [18].

This work presents a novel catalytic system for the transesterification reaction. Using the proposed catalytic system and sunflower oil, a biodiesel fuel was synthesized. The as-obtained product was subsequently mixed with oil diesel in the mass ratio of 20:80 (B20) and 50:50 (B50). The main physical characteristics and cetane number of the biodiesel fuel and biodiesel blends were determined.

EXPERIMENTAL

Samples of summer diesel fuel and sunflower oil were purchased at a gas station and markets in Baku, Azerbaijan. The B20 and B50 blends were prepared by mixing diesel and biodiesel. Biodiesel synthesized from sunflower oil and its blends were characterized in accordance with the American Standard of Testing and Materials (ASTM) methods.

Sunflower biodiesel (B100) was obtained by dissolving 0.25 g of potassium hydroxide (KOH) and 0.25 g of 4-bromophenacyl pyridinium hydroxide (BPPH) in 75 ml of methanol (CH₃OH) without heating (at room temperature). After complete dissolution, 100 grams of oil was added to this mixture. The reaction was carried out in a conical flask equipped with a reverse refrigerator and magnetic stirrer for 3 hours at 55 °C (rotation speed was maintained at 1000 rpm). After stirring, the reaction mass was aged for at least 12 hours in a dividing funnel. The reaction mass was divided into 2 layers using a dividing funnel: the upper layer contained biodiesel, the lower layer – glycerine. Untreated biodiesel was repeatedly washed with hot distilled water in order to remove catalysts. The conversion rate was 83% when using the molar ratio of oil to methanol 1:3.

4-bromophenacyl pyridinium hydroxide (BPPH) was obtained by boiling 10 mmol of phenacyl bromide and 10 mmol of pyridine in 50 ml of toluene in a flask with a reverse fridge. After the formation of a quaternary pyridinium bromide salt, 10 mmol of KOH was added for the synthesis of BPPH (Fig. 1).

NMR spectra of diesel and biodiesel fuel samples were recorded using a spectrometer AVANCE 300 (Bruker Corporation, Germany): 300,130 MHz for ¹H, 75,468 MHz for ¹³C, equipped with a temperature regulator – BVT 3200 sensor. The spectra were recorded in special vials 5 mm in diameter using the standard Bruker TopSpin 3.1.0 software. Chemical shifts ¹H and ¹³C were recorded relative to tetramethylsilane (TMS). The spectra for diesel and biodiesel fuels were recorded in CDCl₃ solutions.

Cetane numbers for the summer diesel, sunflower biodiesel and biodiesel blends under study were calculated according to the methods presented in¹ [19, 20].

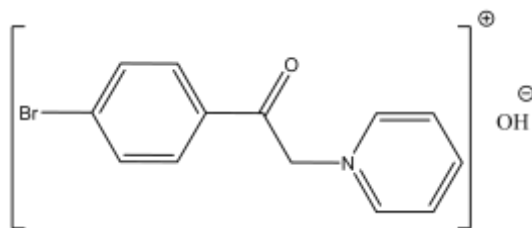


Fig. 1. Formula of 4-bromophenacyl pyridinium hydroxide (BPPH)

Рис. 1. Формула 4-бромфенацилпиридиниум гидроксид (БФПГ)

RESULTS AND DISCUSSION

The properties of biodiesel fuel depend on the structure of fatty acids contained in the triglycerides of vegetable oil, from which the fuel is synthesized. The fatty acid composition determines the physical and operational parameters of biodiesel, such as cetane number, viscosity, cloud temperature, etc. The physical and chemical properties of sunflower oil are presented in Table 1.

Table 2 describes physical properties of the summer diesel fuel, biodiesel fuel from sunflower oil (B100) and their blends (B20, B50) under study.

According to Table 2, the B20 and B50 blends demonstrated a higher level of density, since the density of biodiesel is higher than that of diesel. This can also be explained by the difference in the composition of these fuels: a mixture of hydrocarbons in diesel vs. a mixture of complex esters in biodiesel.

The kinematic viscosity of biodiesel is also higher than that of diesel. Therefore, the kinematic

viscosity of biodiesel blends increases with an increase in the proportion of biodiesel, although remaining within 2–5 mm²/s at 40 °C and meeting ASTM requirements. A significant increase in viscosity is undesirable for fuels intended for diesel engines due to such possible negative effects as impeded fuel pumping, appearance of drops, insufficiently thin spraying of fuel in the combustion chamber and incomplete combustion. However, the viscosity of the B20 and B50 blends almost equals that of diesel, which indicates their suitability for diesel engines.

Fuels with higher flash temperatures are slightly less flammable. This parameter has little effect on the engine performance; rather, it serves as an indicator of fire hazard during the storage and transportation of fuels. Higher flash temperatures make transportation and storage of fuels safer.

Temperatures of clouding and hardening of the biodiesel blends under study increased slightly with an increase in the proportion of biodiesel. The obtained results do not exceed the values specified for biodiesel fuels and do not affect the engine performance during warm weather months.

No unsaturated compounds were detected in the summer diesel fuel under study, which was confirmed by both the experimental determination of the iodine number and ¹H NMR (no signals related to olefin hydrocarbons were observed in the spectrum in the range of 4–6 m.c.). Biodiesel was synthesized from sunflower oil, in which the content of acids with unsaturated radicals can exceed 70 % (see Table 1). Such fuels always contain a significant amount of methyl esters of highly unsaturated acids. However, the degree of unsaturation in the biodiesel blends under study ranged within the limits indicated by ASTM, which was confirmed both by the iodine number (see Table 2) and NMR (Fig. 2a).

Table 1

Physical and chemical properties of refined sunflower oil

Таблица 1

Физико-химические свойства рафинированного подсолнечного масла

Parameter	Fatty acid (number of C atoms: number of double bonds)			
	16:0	18:0	18:1	18:2
Composition of fatty acids, wt%	3.5–7.6	1.3–6.5	14–43	44–74
Acid number, mg KOH/g	0.28±0.5			
Saponification number, mg KOH/g	193.3±0.5			
Iodine number, g I ₂ /100 g	121.4±0.5			
Viscosity, sP	34.1±0.5			
Flash temperature, °C	265			
Freezing temperature, °C	+12			
Density, g/cm ³	0.9186			

¹ Yanowitz J., Ratcliff M.A., McCormick R.L., Taylor J.D., Murphy M.J. Compendium of experimental cetane numbers. Technical report. 2017. <https://doi.org/10.2172/1345058>

Table 2
 Physical properties of diesel fuel, biodiesel synthesized from sunflower oil and their blends
 Таблица 2
 Физические свойства дизельного топлива, биодизельного топлива, синтезированного из подсолнечного масла, и его смесей

Parameter	ASTM methods	ASTM requirements		Experimental data			
		Diesel fuel	Bio-diesel	Diesel fuel	B20	B50	B100
Relative density	D1298	0.8–0.84	0.86–0.9	0.837	0.859	0.864	0.886
Viscosity at 40 °C, mm ² /s; min-max	D445	2–5	3.5–5.0	3.44	3.49	3.87	4.15
Flash temperature, °C, no less than	D93	65	>120	70	75	87	137
Cloud temperature, °C	D2500	-12	<20	7	9	10	11
Freezing temperature, °C	D2500	-15	<15	0	2	5	1
Iodine number, g (I ₂)/100 g	–	60–135	<120	1.58	47.69	88.97	113.86
Sulfur, mass content, no more than	D975-14	15	15	50	38	27	0
Water and sediment, vol. % No more than	D975-14	0.05	0.05	0	0	0	0
Copper corrosion, 3 hours at 50 °C, max	D975-14	No. 3	No. 3	No. 2	No. 1	No. 1	No. 1
Cetane number, min	D975-14	40	47	53	51.8	50.1	47.2

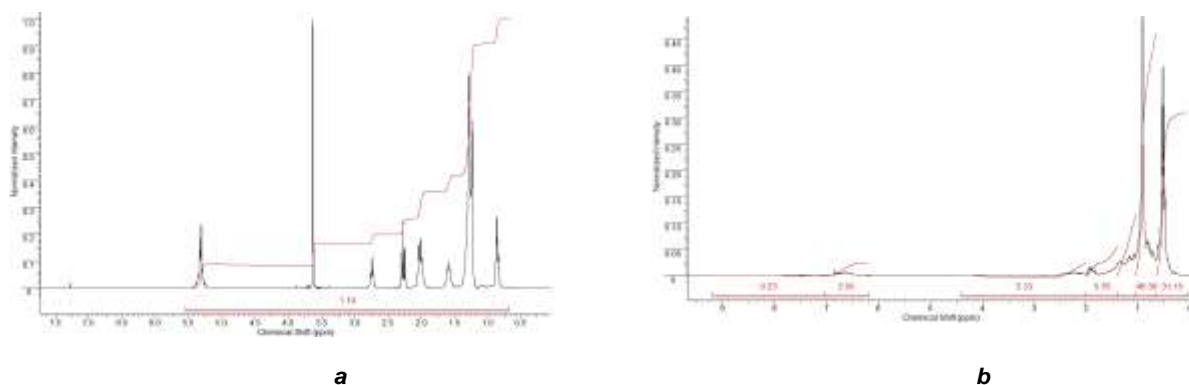


Fig. 2. ¹H NMR spectra for samples of biodiesel (a) and diesel fuel (b)

Рис. 2. ¹H ЯМР-спектры для образцов биодизельного (а) и дизельного топлива (b)

The sulphur content in the biodiesel blends decreased significantly with an increase in the biodiesel content: from 50 m.c. in the diesel fuel up to 27 m.c. in the B50 blend. The use of such biodiesel blends reduces the content of sulphur oxides in exhaust gases, which has a beneficial effect on the environment and human health.

The experimental results (see Table 2) revealed no water and mechanical impurities in the samples of the synthesized biodiesel, B20 and B50 blends. The determination of the corrosive aggressiveness of the samples showed only a slight or moderate tarnish of a copper strip (class 1 and 2). This confirms the high quality of the synthesized fuel and its blends.

The obtained ¹H NMR spectra clearly demonstrate the absence of water in the studied

samples: no signals in the spectrum related to water protons in the area of 4.7 m.c. (see Fig. 2a).

The cetane numbers for the B20 and B50 blends, although decreasing slightly, exceeded the level of 50, thus meeting ASTM requirements. This fact did not have a significant effect on the operational properties of the studied samples.

Alkaline or acid catalysts are frequently used in biodiesel production. The use of such catalysts should be restricted because of the complexity of their separation and recovery, as well as environmental pollution and equipment corrosion. In addition, the process of neutralizing and rinsing alkaline catalysts involves the formation of foam at a level of 2 % from the initial mass of oil used for synthesis. This makes it difficult to rinse and further separate layers of the reaction mass.

A significant advantage of the proposed catalytic system 0.25 % of BPPH – 0.25 % of KOH is the absence of foam formation. In addition, the proposed method omits the stage of catalyst neutralization; therefore, the catalytic system can be reused upon water removal. The biodiesel yield comprises 61 % and 38 % upon the second and third use of BPPH, respectively. Water removal is achieved by distillation.

Transesterification of sunflower oil with methanol followed by the formation of a biodiesel fuel is a typical reaction of nucleophilic substitution. It is known that OH⁻ ions contained in BPPH and alkali act as an active cite for biodiesel synthesis. In the first stage, methanol is activated by

OH⁻ ions. Upon activation, CH₃O⁻ ions attack the carbonyl group of triglycerides with the formation of fatty acid methyl ester. In the second and third stages, other carbonyl groups in triglycerides interact with CH₃O⁻ to form a mixture of fatty acid methyl esters (biodiesel).

CONCLUSIONS

The properties of summer diesel, biodiesel fuel derived from sunflower oil B100, as well as B20 and B50 blends, were investigated against ASTM standards. The proposed novel catalytic system for sunflower oil transesterification has a positive effect on the processes of separating biodiesel from glycerine.

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Contribution

Ibrahim G. Mamedov, Ophelia N. Javadova, Nargiz V. Asimova carried out the experimental work. The authors on the basis of the results summarized the material and wrote the manuscript. All authors have equal author's rights and bear equal responsibility for plagiarism.

Conflict interests

The authors declare no conflict of interests regarding the publication of this article.

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