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SYNTHESIS AND TRIBOLOGICAL EVALUATION OF LUBRICITY ADDITIVES FOR EURO-5 DIESEL FUEL BASED ON LOCAL SECONDARY RAW MATERIALS

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Abstract. The reduction of sulfur content in modern diesel fuels significantly deteriorates their lubricity properties, leading to increased wear of fuel system components. Therefore, the development of effective lubricity-enhancing additives has become an important task for the production of environmentally friendly diesel fuels. In this study, a technology for obtaining lubricating additives based on higher fatty alcohols synthesized from locally available secondary raw materials was developed. Higher fatty alcohols (C_{12}–C_{17}) were produced through the oxidation of low-molecular-weight polyethylene and paraffinic hydrocarbons in the presence of boric acid at temperatures of 165–170 °C. The yield of the target products reached 35–40%. The obtained compounds were characterized using FTIR and GC–MS methods, confirming the formation of alcohol-containing structures. The synthesized additives were introduced into diesel fuel, and their lubricity performance was evaluated according to the ASTM D6079 (HFRR) method. The addition of 0.0163 wt.% of the additive reduced the wear scar diameter from 520 μm to 400–460 μm at 60 °C, meeting the requirements of Euro-5 diesel fuel standards. The proposed technology enables the utilization of local secondary resources and provides an effective alternative to imported commercial lubricity additives for the production of environmentally friendly low-sulfur diesel fuels.

Keywords: petroleum, diesel fuel, higher fatty alcohols (HFA), low-molecular polyethylene (LMPE), boric acid, lubricating additive, lubricity property, sulfur, fractional composition, viscosity.

Annotatsiya. Zamonaviy dizel yoqilg'ilaridagi oltingugurt miqdorini kamaytirish ularning moylash xususiyatlarini sezilarli darajada yomonlashtiradi, bu esa yonilg'i tizimi komponentlarining eskirishini oshiradi. Shuning uchun

moylash qobiliyatini oshiruvchi samarali qo'shimchalarni ishlab chiqish ekologik toza dizel yoqilg'ilarini ishlab chiqarish uchun muhim vazifaga aylandi. Ushbu tadqiqotda mahalliy ikkilamchi xom ashyolardan sintez qilingan yuqori yog‘li spirtlar asosida moylovchi qo‘shimchalar olish texnologiyasi ishlab chiqildi. Yuqori molekulyar yog'li spirtlar (C12–C17) past molekulyar og'irlikdagi polietilen va parafin uglevodorodlarni bor kislotasi ishtirokida 165–170 °C haroratda oksidlash orqali ishlab chiqarilgan. Maqsadli mahsulotlarning hosildorligi 35-40% ga yetdi. Olingan birikmalar FTIR va GC–MS usullari yordamida tavsiflanib, tarkibida alkogol bo'lgan tuzilmalar hosil bo'lishini tasdiqladi. Sintez qilingan qo'shimchalar dizel yoqilg'isiga kiritildi va ularning moylash xususiyatlari ASTM D6079 (HFRR) usuli bo'yicha baholandi. Qo'shimchaning 0,0163 og'irlik foizi qo'shilishi 60 °C haroratda eskirish chandig'i diametrini 520 mkm dan 400–460 mkm gacha qisqartirdi va bu Evro-5 dizel yoqilg'isi standartlari talablariga javob beradi. Taklif etilayotgan texnologiya mahalliy ikkilamchi resurslardan foydalanish imkonini beradi va ekologik toza kam oltingugurtli dizel yoqilg‘isi ishlab chiqarish uchun import qilinadigan tijorat moylash qo‘shimchalariga samarali muqobil bo‘ladi.

Kalit so'zlar: neft, dizel yoqilg'isi, yuqori yog'li spirtlar (HFA), past molekulyar polietilen (LMPE), bor kislotasi, moylash qo'shimchasi, moylash xususiyati, oltingugurt, fraksiyonel tarkibi, yopishqoqlik.

Аннотация. Снижение содержания серы в современных дизельных топливах значительно ухудшает их смазывающие свойства, что приводит к повышенному износу компонентов топливной системы. Поэтому разработка эффективных присадок, повышающих смазывающие свойства, стала важной задачей для производства экологически чистых дизельных топлив. В ходе данного исследования была разработана технология получения смазочных присадок на основе высших жирных спиртов, синтезированных из местного вторичного сырья. Высшие жирные спирты (C12–C17) были получены путем окисления низкомолекулярного полиэтилена и парафиновых углеводородов в присутствии борной кислоты при температурах 165-170°C. Выход целевых продуктов достигал 35-40%. Полученные соединения были охарактеризованы методами FTIR и GC–MS, подтверждающими образование спиртосодержащих структур. Синтезированные присадки были введены в дизельное топливо, и их смазывающие свойства были оценены в соответствии с методом ASTM D6079 (HFRR). Добавление 0,0163 мас.% присадки уменьшило диаметр следов износа с 520 мкм до 400-460 мкм при температуре 60°C, что соответствует требованиям стандартов на дизельное топливо Евро-5.

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Предлагаемая технология позволяет использовать местные вторичные ресурсы и является эффективной альтернативой импортным коммерческим смазочным присадкам для производства экологически чистых дизельных топлив с низким содержанием серы.

Ключевые слова: нефть, дизельное топливо, высшие жирные спирты (HFA), низкомолекулярный полиэтилен (LMPE), борная кислота, смазочная присадка, смазывающие свойства, сера, фракционный состав, вязкость.

INTRODUCTION.

At present, the production of diesel fuels with improved environmental characteristics and reduced contents of sulfur and aromatic hydrocarbons is of considerable importance.

This is because, during combustion, sulfur-containing and aromatic hydrocarbons generate toxic gases and, due to incomplete combustion, exert a greater negative impact on the environment. From an environmental perspective, research aimed at reducing such impacts is therefore highly relevant. However, decreasing the sulfur content in diesel fuel adversely affects its lubricity properties, which may result in non-compliance with standard requirements. In such cases, lubricity-improving additives are introduced into diesel fuel at low concentrations in order to bring its lubricity characteristics into conformity with the applicable standards.

Currently, in the Republic of Uzbekistan, diesel fuels are produced at oil refineries as well as at the Uzbekistan GTL plant. High-quality diesel fuel with reduced sulfur content is produced as a finished product by blending diesel fuel streams manufactured at oil refineries and the Uzbekistan GTL plant. During the production process, lubricity-improving additives imported from foreign countries and purchased in foreign currency are added to the fuel. The use of such additives inevitably affects the production cost of the fuel.

Based on a review of the literature, the composition of lubricity-improving additives and the availability of domestic resources in the country were studied. A comparative analysis of studies conducted by researchers who synthesized additives for improving the lubricity of diesel fuels was carried out. Lubricity-enhancing additives for diesel fuels are generally synthesized through the catalytic hydrogenation of esters of higher fatty alcohols [1]. Higher fatty alcohols are also produced through hydroformylation of higher α-olefins, the use of aluminum-containing catalysts, and oxidation of alkanes with atmospheric oxygen.

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Studies on the synthesis of lubricity-enhancing additives have shown that not the fractional composition of the fuel but rather parameters such as the content of sulfur-containing compounds and the flow properties of the fuel play a decisive role. Only after establishing the significance of these factors did the development and synthesis of lubricity-improving additives begin to advance rapidly [2].

At present, diesel fuels produced at petroleum refineries commonly contain lubricity-improving additives based on polymers, oligomers, copolymers, and non-polymeric compounds. One class of additives that significantly influence the lubricating properties of diesel fuel includes polyolefins and their derivatives. Among them is low-molecular-weight branched polyethylene (LMWPE), a by-product formed during the production of high-pressure low-density polyethylene. Experimental studies have demonstrated that the addition of 0.1–1.0% LMWPE can reduce the wear scar diameter of diesel fuel to approximately 360 μm [3]. In the Republic of Uzbekistan, this low-molecular-weight polyethylene is generated as a secondary product at the Shurtan Gas Chemical Complex. This creates an opportunity for its targeted utilization as a raw material for the production of lubricity-improving additives.

In general, it can be concluded that the effectiveness of lubricity-enhancing additives increases with the length of the saturated linear hydrocarbon radical present in their structure, which significantly improves the lubricating properties of diesel fuels [4].

Based on the analysis of the lubricity-enhancing additives described above and the evaluation of their properties, it was considered appropriate to develop a synthesis method using locally available raw materials. To address this problem, scientific research was carried out focusing on the utilization of secondary raw material resources available in the country. Particular attention was given to the development and implementation of technologies for processing these resources and converting them into effective lubricating additives. The research aimed to obtain additives that are economically viable, environmentally safe, and capable of significantly improving the lubricating properties of diesel fuels. The results of the study demonstrate the feasibility of producing efficient and low-cost lubricity-enhancing additives from locally available feedstocks while simultaneously promoting sustainable utilization of secondary resources [5].

In this context, low-molecular-weight polyethylene (LMWPE), which is formed as a secondary industrial by-product during polyethylene production, can be considered a promising and economically attractive raw material for the synthesis of lubricity-improving additives. Through controlled oxidation in the presence of boric acid, LMWPE can be converted into higher fatty alcohols containing long hydrocarbon chains and polar hydroxyl groups. Such molecular

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structures are expected to adsorb effectively on metal surfaces and form a protective lubricating film, thereby reducing friction and wear in fuel injection systems. Therefore, the aim of this study is to synthesize higher fatty alcohol-based additives from LMWPE waste and to evaluate their effectiveness in improving the lubricity of Euro-5 diesel fuel using the ASTM D6079 HFRR test method.

LITERATURE REVIEW ON LUBRICITY-IMPROVING ADDITIVES

A literature review on additives that improve the lubricity properties of diesel fuel was conducted.

Additives based on ethylene-propylene (EP) copolymers and ethylene-propylene-diene (EPDM/EPDS) copolymers, as well as their derivatives, have also been widely applied in diesel fuels at concentrations of 1.0–5.0%. It has been observed that products obtained through thermal and thermo-oxidative degradation of dicyclopentadiene exhibit significantly higher lubricity-enhancing activity compared with conventional EP and EPDS additives.

The effect of the DAKS-D composite lubricity-enhancing additive for diesel fuel has also been investigated. This additive is synthesized through the production of alcohols from carboxylic acids followed by the formation of compositions with nitrogen-containing compounds. Research results indicate that pure carboxylic acids cannot be effectively used as additives for diesel fuels because they have little influence on the wear scar diameter across the fuel volume. However, composite additives containing higher fatty alcohols and nitrogen-containing compounds exhibit a synergistic effect, improving both lubricity and stability of petroleum products [6-8].

The influence of esters derived from triglycerides of higher fatty acids and vegetable oils on the lubricity and sedimentation stability of diesel fuel has also been investigated. Esters synthesized from polyoxyalkylene glycols containing C_{10}–C_{30} carbon atoms with molecular masses ranging from 200 to 2000 were obtained at temperatures of 200–340 °C and were shown to increase the viscosity and lubricity of diesel fuels. Additives synthesized from higher fatty acids and stearic acid improved the lubricating properties of diesel fuels when added in an amount of 0.1%, lowering the pour point by 2–5 °C.

To enhance fuel fluidity and lubricity, esters based on trimethylolpropane, trimethylolethane, and pentaerythritol have been employed. Studies on ester-based additives have shown that concentrations of 0.01–0.03% reduce the wear scar diameter to approximately 450 μm. Esters obtained through esterification of pentaerythritol with oxystearic acid at concentrations of 0.1–0.3% reduced the wear scar diameter of summer diesel fuel from –13 °C to –40 °C, while for dewaxed winter diesel fuel the wear scar diameter decreased from 570 μm to 360 μm [9].

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Compounds with molecular masses of 12 000–15 000, obtained from the reaction of aliphatic acid chlorides containing C_{12}–C_{18} carbon atoms with dextrin, have also been shown to improve both viscosity and lubricity of diesel fuel.

Condensation-type additives with lubricity-enhancing properties have been synthesized based on monoaliphatic alcohols containing C_{10}–C_{30} carbon atoms and malic acid, and their effectiveness in improving diesel fuel lubricity has been experimentally confirmed [10,11].

Esters obtained from higher fatty alcohols ($C_8H_{17}OH$, $C_{15}H_{31}OH$, $C_{16}H_{33}OH$) with molecular masses ranging from 300 to 1200, together with polyhydric alcohols such as glycerol, ethylene glycol, diethylene glycol, and triethylene glycol, and adipic acid, have also been investigated. When added in amounts of 0.1–1.0%, these esters reduce the wear scar diameter to 460 μm, meeting the requirements of the Euro-5 standard, while simultaneously improving the operational and environmental characteristics of diesel fuels.

To enhance both fuel fluidity and lubricity, additives containing esters of linear carboxylic acids with N-hydroxyl groups (C_6–C_{12}) have also been used. Nitrogen-containing compounds such as triethanolamine, diethanolamine, methyldiethanolamine, ethyldiethanolamine, butyldiethanolamine, and diisopropanolamine, as well as derivatives of lauric, palmitic, and stearic acids, have been applied as components of such additives [12,13].

Low-molecular-weight compounds containing dialkylcyanamide groups with C_4–C_{16} carbon atoms have been synthesized by condensation of potassium cyanamide with haloalkyls in a dimethylformamide medium. When added at a concentration of 0.03%, these compounds demonstrated lubricity-enhancing properties in diesel fuel. Nitrogen-containing additives of this class effectively reduce the wear scar diameter.

EXPERIMENTAL RESEARCH.

At present, in production plants, the fractional composition of diesel fuels is lightened in order to improve their low-temperature properties. This leads to a reduction in viscosity. As viscosity decreases, the lubricity properties of diesel fuel also deteriorate [14].

To investigate the influence of fractional composition and viscosity on the lubricating properties of diesel fuels, low-sulfur diesel fuels containing up to 0.07 wt.% sulfur were selected for the study.

The experimental results showed that for diesel fuel samples 1 and 2, in which 50% and 96% distillation temperatures were within the ranges of 262–274 °C and 350–360 °C, respectively, the wear scar diameter was found to be 430–450 μm.

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A decrease in the distillation temperature of diesel fuel led to an increase in the wear scar diameter. As shown in Table 1. sample 4, characterized by a fractional composition with final boiling temperatures of 182–276 °C for the 50% and 96% fractions, exhibited a wear scar diameter of 610 μm, which corresponds to conditions close to dry friction.

This behavior is mainly attributed to the higher sulfur content in sample 4 (0.022 wt.%) compared with samples 1 and 2. For these fuels, the wear scar diameters were observed to be 870 μm and 835 μm, respectively. When the sulfur content of the fuel reached 0.03 wt.%, the wear scar diameter reached its minimum value of 430 μm.

The physicochemical properties of the investigated diesel fuels are presented in Table 1.

Table 1.

Main physicochemical properties of diesel fuels.

Fuel sample	Fractional composition, °C		Sulfur content, wt.%	Kinematic viscosity at 20 °C, mm²/s	Wear scar diameter, μm
	50%	96%			
1	193	322	0,010	1,84	870
2	222	296	0,015	2,19	835
3	255	351	0,020	3,74	520
4	182	276	0,022	1,53	610
5	**262**	**360**	**0,030**	**4,22**	**430**
6	274	350	0,040	5,49	450
7	204	262	0,070	1,88	520
8	269	334	0,080	4,94	545

Thus, the influence of fractional composition and sulfur content on the lubricating properties of diesel fuel can be clearly observed in samples 3 and 7, where both fuels exhibit the same wear scar diameter of 520 μm. In sample 7, the sulfur content is 0.07 wt.%, whereas in sample 3 it is 0.02 wt.%. Compared with sample 3, sample 7 is characterized by a lighter fractional composition and lower kinematic viscosity.

It can therefore be concluded that the lubricating properties of diesel fuels are not determined solely by their physicochemical and operational characteristics. Rather, they are also significantly influenced by factors such as the fractional composition of the fuel, its viscosity, and the sulfur content present in the fuel. At present, since diesel fuel fraction corresponding specifically to Sample 5 has been reprocessed at the Bukhara Oil Refinery, subsequent studies were carried out using a sample of this composition. [15].

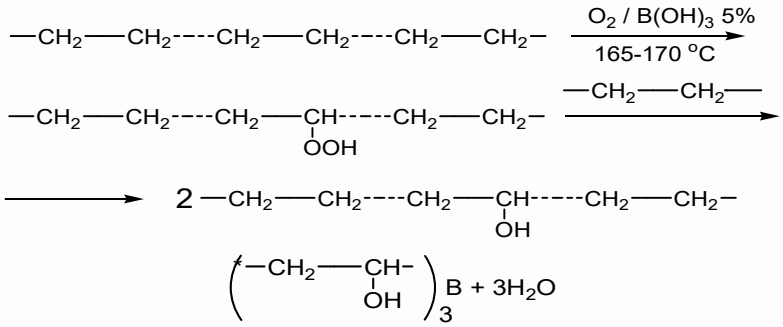
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As a constituent of diesel fuel, the petroleum hydrocarbon fraction boiling within the range of 262 °C to 360 °C is separated as an individual fraction, and its sulfur content is reduced by means of hydrotreating technology. In this fraction, the sulfur content generally amounts to approximately 0.03%. At present, it is brought to the finished-product stage by blending with diesel fuel produced at the Uzbekistan GTL plant, which contains virtually no sulfur. This, in turn, leads to a further deterioration of the lubricity properties of the fuel.

Therefore, when producing diesel fuel that meets the requirements of O‘z DSt 1134:2018, particularly Euro-5 and Euro-6 standards, the addition of lubricity-improving anti-wear additives is of particular importance.

Globally, such problems are addressed by adding additives based on higher fatty alcohols (HFA) to diesel fuel.

Considering the importance of higher fatty alcohols in various fields, it is appropriate to review and compare the raw materials and methods used for their synthesis. In this study, higher fatty alcohols were synthesized based on low-molecular polyethylene (LMPE), which is a secondary waste product of the Shurtan Gas Chemical Complex.



A total of 100 g of low-molecular polyethylene (LMPE) was charged into the reactor, after which a gas mixture of nitrogen and 3.0–4.5% oxygen was supplied from a cylinder, and 5 g of boric acid was added. The temperature was raised to 165–170 °C, and the reaction was carried out at this temperature for 2–3 hours. Oxygen supply was ensured using a sparger. The flow rates of air and nitrogen were monitored using a rheometer (Figure 1).

The flow rate of the oxidizing gas was maintained at 70 L/h per batch of LMPE. During the oxidation process, the gases leaving the reactor were vented to the atmosphere [23].

Condensed droplets of the reaction mixture were returned to the reactor, while the aqueous condensate was collected in a water trap. The separated higher fatty alcohols were purified, and unreacted hydrocarbons were distilled off under vacuum.

As a result of oxidation, alcohols containing the same number of carbon atoms as the initial hydrocarbons were obtained. These products were mainly

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secondary alcohols, in which the hydroxyl group is attached at different positions along the carbon chain. The residue consisted mainly of boric acid esters, which formed a brown mass that solidified even at room temperature. The yield of the target product was 38–39%.

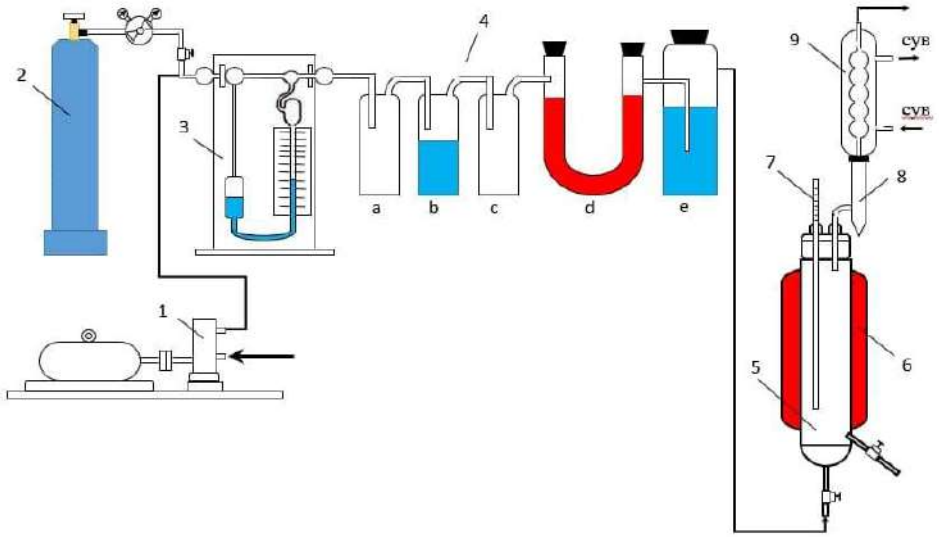


Figure 1. Schematic diagram of the laboratory setup for producing higher fatty alcohols by oxidation of LMPE: 1 – air sparger, 2 – nitrogen cylinder, 3 – rheometer, 4 – $CaCl_2$ dryer, 5 – reactor, 6 – heater, 7 – thermometer, 8 – Dean–Stark apparatus, 9 – reflux condenser.

The maximum oxidation efficiency of LMPE, i.e., the highest conversion, is achieved in the fourth hour of oxidation of low-molecular-weight polyethylene. By the fifth hour of synthesis, the amount of formed hydroxyl groups remains constant [24].

RESEARCH RESULTS.

The rate of formation of higher fatty alcohols depends on the temperature of the oxidation process of normal paraffinic hydrocarbons by molecular oxygen from air in the presence of boric acid. When paraffins obtained from local secondary raw materials are oxidized with atmospheric oxygen at 120–170 °C in the presence of boric acid and without a catalyst, the rate of increase in the number of hydroxyl groups decreases by 8–10 times (Figure 2).

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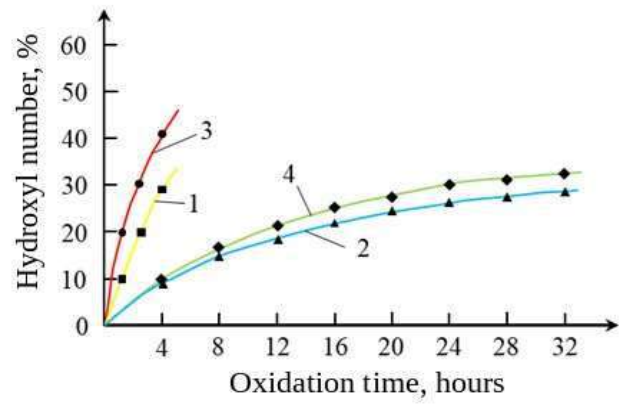


Figure 2. Time dependence of the formation of hydroxyl and ether groups during the catalyst-free oxidation of paraffinic hydrocarbons in the presence of boric acid at different temperatures: 1 – number of hydroxyl groups formed in paraffin oxidized at 170 °C; 2 – number of hydroxyl groups formed from paraffinic hydrocarbons oxidized at 120 °C; 3 – number of ether groups formed in paraffin oxidized at 170 °C; 4– number of ether groups formed in paraffin oxidized at 120 °C

If the temperature is reduced below 120 °C, the oxidation process slows down significantly; however, the ratio of alcohols to carbonyl compounds in the oxidation products, equal to 1:1.27, remains almost unchanged.

Therefore, in order to ensure the rapid accumulation of large amounts of primary oxidation products and maintain a high process rate, it is recommended to use a variable-temperature regime for paraffin oxidation. In this regime, the initial oxidation temperature should be 120–125 °C, after which, depending on the intensity of the process, the temperature should be decreased by 2–5 °C per hour to obtain higher fatty alcohols of the highest quality.

The oxidation of paraffinic hydrocarbons with oxygen from air is a heterogeneous process. Its rate is affected by temperature, the concentrations of hydrocarbons and oxygen, and the rate of oxygen diffusion into the liquid phase. Under the conditions of the oxidation process, the concentration of paraffin in the oxidized mixture does not have a significant effect on the oxidation rate. However, the concentration of dissolved oxygen affects both the degree of oxidation and the quality of the products formed. In the liquid-phase oxidation of paraffins to higher fatty alcohols, the rate of oxygen absorption is not constant throughout the process, and the maximum absorption rate varies depending on the operating regime.

During catalyst-free oxidation, the higher the temperature in the range of 120–160 °C, the greater the amount of absorbed oxygen. In this case, oxygen is consumed mainly for the formation of other oxygen-containing products rather than alcohols. When purified paraffin is oxidized on a large scale without a catalyst at 125–105 °C, oxygen absorption begins before the formation of the first carbonyl

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products and reaches a maximum corresponding to the highest rate of alcohol formation (Figure 4.2, curve 1).

During the oxidation of a mixture of fresh and recycled paraffins at a ratio of 1:2, oxygen absorption begins immediately and proceeds intensively. It reaches its maximum 3–4 hours after the start of the reaction, which also corresponds to the period of the highest accumulation rate of alcoholic products (curve 2). However, the amount of absorbed oxygen does not exceed 20% of the oxygen supplied with air for oxidation.

When the same mixture is oxidized after the preliminary addition of 0.2% boric acid, oxygen uptake begins intensively from the very start of the process. The curve of the oxygen absorption rate passes through a sharp maximum and then rapidly decreases (curve 3). The maximum amount of oxygen absorbed from air is four times higher than that observed during catalyst-free oxidation and reaches 80%. During 20 hours, alcohol accumulation continues at a constant rate of 2.0–2.3% per hour.

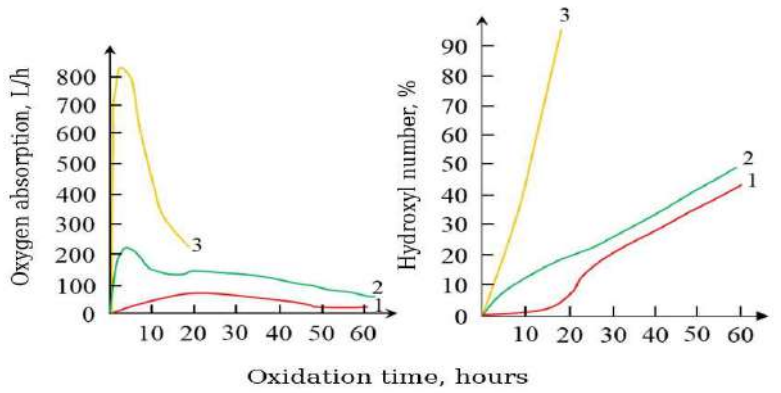


Figure 3. Changes in the amount of absorbed oxygen and the hydroxyl number of the oxidate during the oxidation of paraffins in the presence of various additives: 1 - initial paraffin; 2 - initial paraffin + recycled paraffin; 3 - initial paraffin + recycled paraffin + boric acid.

During catalyst-free oxidation, only approximately one-third of the absorbed oxygen is consumed for the formation of acidic products, while the remaining portion is used for the formation of other oxygen-containing compounds. In the presence of active soluble boric acid, the major part of the absorbed oxygen, up to 80.5%, is consumed for the formation of alcohols.

Therefore, during a rapid reaction in the presence of active boric acid, it is very important to ensure the supply of the required amount of oxygen to the oxidizing hydrocarbons.

In the production of synthetic fatty alcohols in oscillating columns, the supply of oxygen to paraffin molecules oxidized in the liquid phase is primarily determined by its diffusion rate. The amount of oxygen transferred by diffusion per

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unit reaction volume, **k**, is a function of the linear velocity of air in the column, **w**, and the average oxygen concentration in it, **c**:

$$k = f(w, s)$$

The average oxygen concentration in the gas phase depends on the height of the oxidizing product layer: the smaller the reactor, the higher this concentration becomes. Practical data show that variations in oxygen concentration depending on the size of the apparatus are not of major significance, since only 40–45% of oxygen is consumed during oxidation, and in some cases approximately 20%.

The linear velocity of air determines both the value of the oxygen transfer rate constant and the size of the interfacial contact area between the phases. Therefore, it is a decisive factor in creating optimal conditions for supplying oxygen to the oxidized paraffin molecules.

The relationship between the oxidation reaction rate and the air flow velocity in an oscillating column was demonstrated using paraffin oxidation as an example and was confirmed during the oxidation of soft paraffin to higher fatty alcohols (Table 4.1). In order to establish a rapidly proceeding liquid-phase oxidation process and scale it up to other production capacities, it is necessary to maintain the linear air velocity in oscillating columns at the level determined during the modeling process. The specific air consumption does not play a significant role [136, pp. 1056–1060].

Under laboratory conditions, when carrying out the liquid-phase oxidation process, the degree of air dispersion is of great importance, especially when comparing air supply through a simple tube and through a porous glass filter. However, in apparatuses where the height of the sparger zone is relatively small compared with the total volume, the degree of air dispersion plays only a minor role, since small bubbles coalesce into larger bubbles in the main volume of the oxidized mass.

Thus, during the liquid-phase oxidation of paraffin in the presence of active boric acid, it is necessary to ensure a sufficient supply of atmospheric oxygen in order to obtain high-quality higher fatty alcohols. This can be achieved by maintaining a linear air velocity of approximately 0.1 m/s in equipment of any size. With a significant increase in the linear air velocity, the ratio of the reaction products remains unchanged; however, the amount of exhaust gases leaving with the air increases sharply.

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Table 2.

Dependence of the oxidate characteristics on the linear air velocity during the oxidation of paraffinic hydrocarbons

Column / Scale	Loading, t	Linear air velocity, m/s	Specific air consumption, $m^3/(t \cdot h)$	Duration, h	Hydroxyl number, mg KOH/g		
					Hydroxyl number, mg KOH/g	Ester number, mg KOH/g	Carbonyl number, mg KOH/g
Industrial scale	30,2	0,095	59,0	8,5	68,6	56,5	4,9
Pilot scale	0,1	0,015	60,0	4,3	69,9	48,6	2,6
Pilot scale	0,1	0,094	382,0	8,3	69,2	54,6	4,1
Laboratory scale	$2 \cdot 10^{-4}$	0,093	60,0	0,0	23,1	35,2	1,0
Laboratory scale	$2 \cdot 10^{-4}$	0,094	1725,0	6,3	70,2	54,3	5,1

Increasing the oxygen content in air up to pure oxygen affects the reaction rate; however, it does not change the ratio of the components and does not affect the composition of the final products during the bubbling process. Improvement of oxygen diffusion conditions due to an increase in pressure does not significantly increase the degree of oxidation, although the solubility of atmospheric oxygen is almost proportional to pressure and may negatively affect product quality.

The most promising method for intensifying paraffin oxidation in the liquid phase is to create new and more favorable conditions for interaction with atmospheric oxygen by sharply increasing and continuously renewing the gas–liquid contact surface. This considerably increases the rate of oxygen absorption. For this purpose, foam-type apparatuses, hollow mixers, and similar devices can be used. In the foam regime, the nature of bubble motion is determined not by Archimedes' forces, as in conventional bubbling, but by the kinetic energy of the gas, which ensures instantaneous renewal of the bubbling surface. In practice, to create a foam regime, the gas velocity in the free cross-section of the apparatus should be within the range of 0.8–4.0 m/s. Despite the high gas velocity, foam apparatuses have low hydraulic resistance and a simple design; however, they require large operating volumes. The main factor determining the efficiency of an oxidation apparatus is its productivity per unit volume. Therefore, any increase in the volume of the foam apparatus compared with a conventional bubbling

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apparatus must be compensated by a corresponding increase in the overall rate of the oxidation reaction. Prolonged exposure of the oxidation products to high temperature and oxygen has a negative effect on their composition.

Thus, during oxidation, cleavage occurs at various positions of the LMPE molecules, resulting in a mixture of fatty acids containing different numbers of carbon atoms. The resulting mixture is then distilled through a column with a dephlegmator to separate it into narrow fractions. The initial fraction, based on its characteristics, resembles the acid obtained from coconut oil. The composition and properties of the first and second fractions are presented in Table 3.

Table 3.

Composition of the first fraction (C_{10}–C_{16})

Fractions	HFA content, wt %
C_{10} until	4,51–6,42
C_{10}–C_{11}	22,0–26,5
C_{12}–C_{13}	47,3–48,6
C_{14}–C_{16}	30,2–34,0
C_{16} above	17,6–21,5

The infrared (IR) spectrum of the higher fatty alcohols obtained by oxidation of low-molecular polyethylene (LMPE) was recorded using a Shimadzu IRAffinity-1S Fourier Transform Infrared (FTIR) spectrophotometer in the 300–4000 cm^{-1} range. The experiment was conducted under standard conditions: atmospheric pressure (762 mmHg), temperature of 25 °C, and relative humidity of 45%.

In the IR spectrum of the higher fatty alcohols obtained from oxidized LMPE:

- The absorption band at 1712.19 cm^{-1} corresponds to the C=O stretching of saturated aliphatic aldehydes.
- Absorption bands at 2848.86 cm^{-1}, 2914.44 cm^{-1}, and 2956.87 cm^{-1} are characteristic of C–H stretching vibrations in alkyl chains.
- The band at 1463.97 cm^{-1} corresponds to C–H bending vibrations.
- Bands at 1377.17 cm^{-1} and 1332.88 cm^{-1} are associated with the planar deformation vibrations of primary alcohol O–H bonds.
- Absorption bands at 1193.94 cm^{-1}, 1124.5 cm^{-1}, and 1074 cm^{-1} are characteristic of the O–H stretching vibrations of secondary alcohols.
- Bands at 719.45 cm^{-1} and 621.08 cm^{-1} indicate the presence of 14 and 12 $–CH_2$ groups, respectively.

This analysis confirms the presence of both primary and secondary hydroxyl groups in the synthesized higher fatty alcohols and provides detailed information about their molecular structure.

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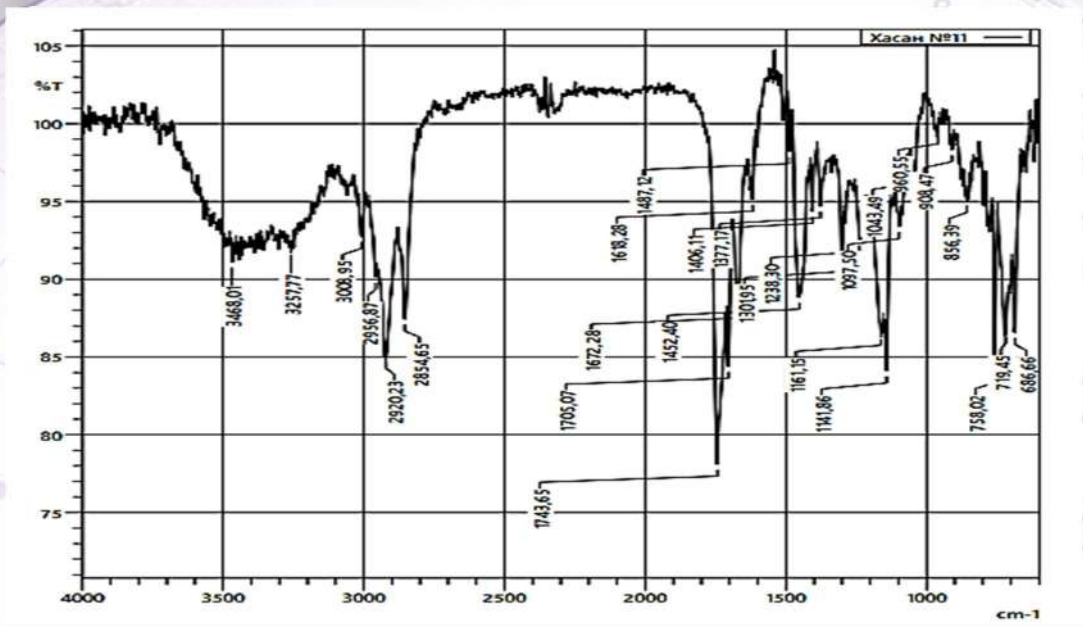


Figure 4. IR spectrum of higher fatty alcohols obtained by oxidation of low-molecular polyethylene (LMPE).

Higher fatty alcohols (extracts and ester oils) obtained from local secondary industrial raw materials via oxidation were analyzed using a gas chromatography–mass spectrometry (GC–MS) system Agilent 5975C inert MSD/7890A GC (Agilent Technologies) (Figure 6).

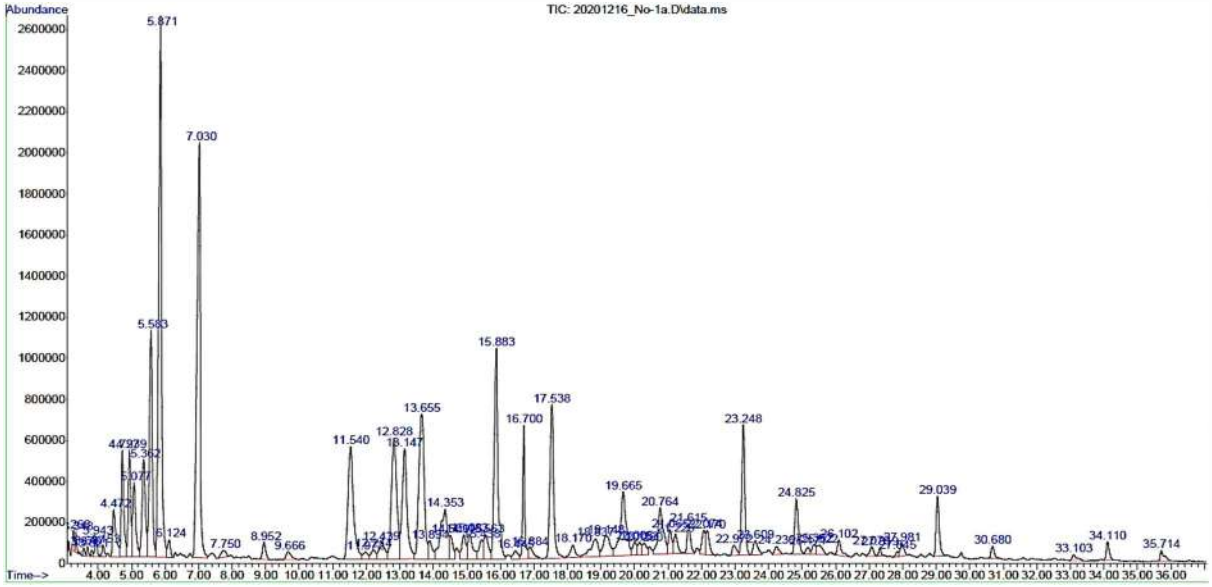


Figure 5. GC–MS analysis of higher fatty alcohols obtained by oxidation of low-molecular polyethylene (LMPE).

The analysis of the components present in the mixture was carried out using a quartz capillary column Agilent HP-INNOWax (30 m × 250 μm × 0.25 μm). The temperature program was set as follows: 60 °C (10 min), then increased at 4 °C/min to 220 °C (10 min), followed by heating at 1 °C/min to 240 °C (20 min).

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The injected sample volume was 0.2 mL (in hexane), while the flow rate of the mobile phase was 1.1 mL/min (H_2). The injector temperature was maintained at 250 °C.

The EI–MS spectra were recorded within the m/z range of 10–550 a.m.u. The identification of the components was performed by comparing the obtained mass spectra with data from the W9N11.L, W8N05ST.L, and NIST08 spectral libraries. The results of the chromatographic–mass spectrometric analysis are presented in Table 4.

Table 4.

Chromatographic–mass spectrometric analysis of compounds obtained from the oxidation of low-molecular-weight polyethylene.

№	Components	R_f, min	Content, %
1	n-Dodecane	**4,470**	**1,20**
2	Oxalic acid, hexyl L-menthyl ester	**4,725**	**2,00**
3	Oxalic acid, heptadecyl L-menthyl ester	**4,939**	**1,94**
4	1,4-Dimethyl-2-(2-methylpropyl)cyclohexane, (1alpha,2beta,5alpha)-	**5,075**	**1,70**
5	1-Ethyl-2,4-dimethylcyclohexane	**5,360**	**2,47**
6	(Z)-2-Dodecene	**5,585**	**4,87**
7	1,1,3-Trimethyl-3-(2-methylprop-2-enyl)cyclopentane	**5,870**	**11,49**
8	Hexylcyclohexane (1-cyclohexylhexane)	**7,028**	**10,20**
9	2-(Hydroxymethyl)-3-butyl-5-methylfuran	**11,539**	**4,50**
10	4-Butoxy-2,3-dicyanophenyl 4-butylcyclohexane-1-carboxylate	**12,827**	**4,81**
11	1,5-Diethyl-2,3-dimethylcyclohexane	**13,147**	**3,99**
12	Methyl 1-ethylcyclohexane-1-carboxylate	**13,658**	**5,79**
13	1,1,3-Trimethylcyclohexane	**14,352**	**2,60**
14	(E)-3-Tetradecene	**15,883**	**5,67**
15	2-Decanone (methyl octyl ketone)	**16,702**	**1,97**
16	Octylcyclohexane (1-cyclohexyloctane)	**17,539**	**4,24**
17	4-Butoxy-2,3-dicyanophenyl 4-hexylcyclohexane-1-carboxylate	**18,839**	**1,01**
18	Cyclotetradecane	**19,148**	**1,07**
19	1,2-Dodecanediol	**19,664**	**2,87**
20	2,3-Dicyano-4-(pentyloxy)phenyl 4-hexylcyclohexane-1-carboxylate	**20,762**	**1,86**
21	1-Propyltridecyl dichloroacetate	**23,249**	**2,96**

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22	Decylcyclohexane (1-cyclohexyldecane)	**24,828**	**1,48**
23	(E)-5-Octadecene	**29,036**	**1,35**

The chromatographic–mass spectrometric analysis of higher fatty alcohols obtained from the oxidation of low-molecular-weight polyethylene and paraffin is presented in Table 5. The results show that the yield of higher fatty alcoh,ols formed during the oxidation process was significant and efficient [25,26].

Since 2026, the requirements for producing Euro-5 grade diesel fuel at petroleum refineries have been strengthened. According to the GOST standards, the sulfur content in such fuels must not exceed 10 mg/kg, while the hydrocarbon composition must also comply with the established regulatory specifications. The use of this type of fuel provides several operational advantages. During engine operation, noise and vibration levels are reduced, engine starting becomes easier, and fuel consumption decreases. In addition, this fuel is designed to reduce the wear rate of engine components, thereby extending the service life of engine parts. Furthermore, Euro-5 diesel fuel is considered environmentally safer, as the lower concentrations of hydrocarbons and sulfur significantly reduce the emission of harmful combustion products into the atmosphere.

A lubricity-improving additive was synthesized based on higher fatty alcohols obtained by the oxidation of low-molecular-weight polyethylene. The effectiveness of this additive was evaluated in accordance with the ASTM D6079 standard. According to the results of the study, the addition of this additive at a concentration of 0.0163 wt.% to diesel fuel obtained by the compounding method at the Bukhara Oil Refinery reduced the wear scar diameter at 60 °C to a level not exceeding 460 μm, in compliance with the standard requirement. This indicates that the additive introduced at this concentration is sufficient to ensure the lubricity requirements established for Euro-5 diesel fuel.

Currently, the lubricity of diesel fuels worldwide is commonly evaluated using the HFRR (High Frequency Reciprocating Rig) method, which measures the high-frequency reciprocating motion of a ball against a stationary plate immersed in the fuel. The results obtained from this device are widely accepted as an international standard for assessing diesel fuel lubricity [29].

Table 5

Results of BNKIZ tests of lubricating additives for diesel fuels

Sample designation	Additive content, %	Lubricity by ASTM D6079, wear scar diameter at 60 °C, μm (should not exceed 460 μm according to the standard)

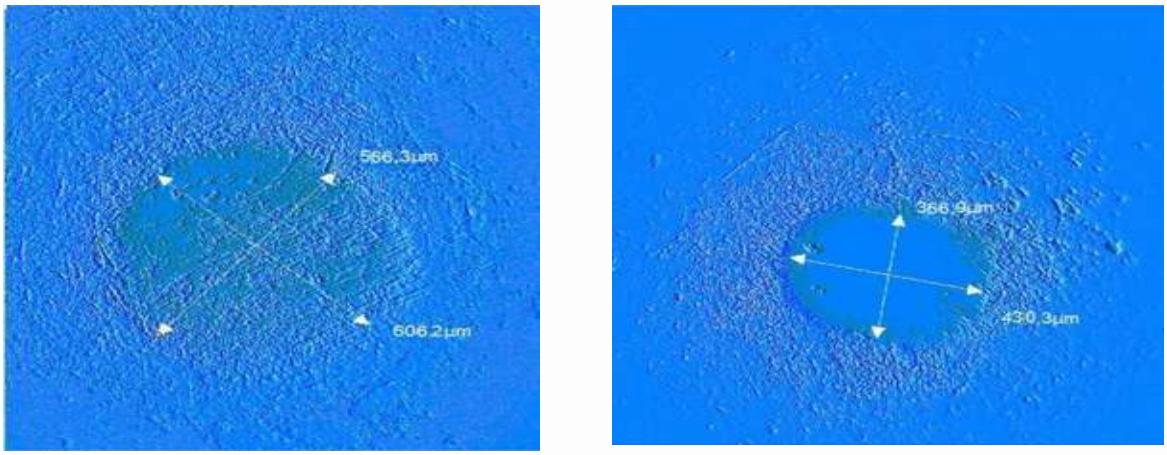
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Euro-5 diesel fuel without additive	-	520
Euro-5 diesel fuel with additive	0.0163	460

Research on the effect of the synthesized higher fatty alcohols on the lubricity properties of diesel fuel was continued. In this study, the optimal concentration of the additive was determined based on the lubricity requirements for Euro-5 diesel fuel according to the ASTM D6079 method. If the wear scar diameter exceeds 460 μm, the lubricating performance of the fuel is considered non-compliant with the standard requirements.

Currently, worldwide, the lubricity of diesel fuels is most often assessed using the HFRR method (High-Frequency Reciprocating Rig), in which a ball slides against a plate in the fuel at high frequency. The HFRR test is recognized by professionals as the standard method for evaluating the lubricating properties of diesel fuels [30].

Images of the wear scar diameter on the steel balls, obtained using a digital microscope (ToupCam model), are presented in Figure 5. The images compare samples with and without the synthesized lubricity additive developed in this study.



a) *b*)

Figure 6. Wear scar marks on the steel ball obtained in pure diesel fuel without additives (a) and in diesel fuel containing 0.0163 vol.% additive (b).

The physicochemical properties of diesel fuel containing 0.0163 wt.% of a lubricity additive synthesized from higher fatty alcohols (C_{12}–C_{17}) were comprehensively analyzed (Table 6).

Table 6.

Main characteristics of diesel fuel complying with Euro-5 requirements

Parameter	**Value**
Cetane number, not less than	51
For Class 1	49

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Cetane index, not less than	46
Density at 15 °C, kg/m³	820–845
Density at 15 °C for Class 1, kg/m³	800–845
Polycyclic aromatic hydrocarbons, wt.%, not more than	11
Sulfur content in fuel, mg/kg, not more than	10
Flash point (closed cup), °C, not lower than	55
Carbon residue on 10% distillation residue, wt.%, not more than	0.30
Ash content, wt.%, not more than	0.01
Water content, mg/kg, not more than	200
Total contamination, mg/kg, not more than	24
Copper strip corrosion (3 h at 50 °C), class	Class 1
Oxidation stability, g/m³, not more than	25
Lubricity: wear scar diameter at 60 °C, μm, not more than	**460**
Kinematic viscosity at 40 °C, mm²/s	2.0–4.5
Kinematic viscosity at 40 °C for Class 1, mm²/s	1.5–4.0

Thus, the synthetic higher fatty alcohols proposed in this study demonstrated superior lubricating performance compared with existing anti-wear additives, which makes it possible to obtain low-sulfur, environmentally friendly diesel fuels [31].

CONCLUSIONS.

In this study, a technology for obtaining lubricity-enhancing additives for diesel fuels based on locally available secondary raw materials was developed. Higher fatty alcohols (C_{12}–C_{17}) were synthesized through the oxidation of low-molecular-weight polyethylene (LMPE) and paraffinic hydrocarbons in the presence of boric acid at temperatures of 165–170 °C. Under the selected reaction conditions, the yield of the target products reached 35–40%, confirming the effectiveness of the proposed synthesis method.

Chromatographic–mass spectrometric (GC–MS) analysis demonstrated that the obtained products consist of a mixture of higher fatty alcohols and oxygen-containing compounds, confirming the successful oxidation of the initial hydrocarbons. The synthesized higher fatty alcohols were subsequently applied as lubricity additives for diesel fuel.

Experimental results showed that the addition of 0.0163 wt.% of the synthesized additive to diesel fuel significantly improved its lubricating properties. According to the ASTM D6079 method, the wear scar diameter at 60 °C decreased from 520 μm for untreated diesel fuel to approximately 400–460 μm after the addition of the additive, which fully satisfies the requirements of Euro-5 standards.

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The obtained results demonstrate that the synthesized additives based on higher fatty alcohols exhibit higher lubricity efficiency compared with several commercial anti-wear additives. In addition, the proposed technology utilizes local secondary raw materials, including industrial polymer waste, which contributes to resource efficiency, cost reduction, and environmental sustainability. Therefore, the developed lubricity-enhancing additive can be considered a promising alternative to imported commercial additives and may be successfully applied in domestic oil refineries for the production of environmentally friendly low-sulfur diesel fuels with improved operational and tribological properties.

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