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TECHNOLOGY FOR THE PRODUCTION OF HIGHER FATTY ACIDS FROM SECONDARY PARAFFIN FEEDSTOCKS FOR ENHANCING THE LUBRICITY OF DIESEL FUELS

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Abstract. The objective of this study is to develop an efficient technology for the synthesis of higher fatty acids from secondary paraffinic feedstock and to evaluate their effect on the lubricating properties of diesel fuels. The scientific novelty lies in a comprehensive approach to the processing of paraffinic waste, involving oxidation, saponification, thermal treatment, and fractional distillation stages. The regularities in the formation of the composition of the obtained fatty acids and their influence on the operational characteristics of diesel fuel have been established. It has been shown that the use of the synthesized acids as additives significantly improves the lubricity of the fuel and reduces wear. The developed technology enables the effective utilization of secondary raw materials and the production of competitive additives for modern diesel fuels.

Keywords: higher fatty acids, oxidation of paraffins, saponification process, distillation, diesel fuel, additive, lubricity, anti-wear properties, secondary raw materials, EURO-6.

Аннотация. Целью данной работы является разработка эффективной технологии синтеза высших жирных кислот из вторичного парафинового сырья и оценка их влияния на смазывающие свойства дизельных топлив. Научная новизна заключается в комплексном подходе к переработке парафиновых отходов с использованием стадий окисления, омыления, термической обработки и фракционной дистилляции. Установлены закономерности формирования состава получаемых кислот и их влияние на эксплуатационные характеристики топлива. Показано, что применение синтезированных кислот в качестве присадок обеспечивает значительное улучшение смазывающей способности и снижение износа. Разработанная технология позволяет эффективно использовать вторичное сырьё и

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создавать конкурентоспособные добавки для современных дизельных топлив.

Ключевые слова: высшие жирные кислоты, окисление парафинов, процесс омыления, дистилляция, дизельное топливо, присадка, смазывающая способность, противоизносные свойства, вторичное сырье, EURO-6.

Annotatsiya. Ushbu tadqiqotning maqsadi ikkilamchi parafin xomashyosidan yuqori yog'li kislotalarni sintez qilishning samarali texnologiyasini ishlab chiqish va ularning dizel yoqilg'isining moylash xususiyatlariga ta'sirini baholashdan iborat. Ilmiy yangilik oksidlanish, sovunlash, termik ishlov berish va fraksiyonel distillash bosqichlarini o'z ichiga olgan parafin chiqindilarini qayta ishlashga kompleks yondashishdan iborat. Olingan yog 'kislotalarining tarkibini shakllantirish qonuniyatlari va ularning dizel yoqilg'isining ekspluatatsion xususiyatlariga ta'siri aniqlandi. Sintezlangan kislotalardan qo'shimchalar sifatida foydalanish yoqilg'ining moylash qobiliyatini sezilarli darajada yaxshilashi va eskirishni kamaytirishi isbotlangan. Ishlab chiqilgan texnologiya ikkilamchi xom ashyolardan samarali foydalanish va zamonaviy dizel yoqilg'ilari uchun raqobatbardosh qo'shimchalar ishlab chiqarish imkonini beradi.

Kalit so'zlar: yuqori yog'li kislotalar, parafinlarning oksidlanishi, sovunlanish jarayoni, distillash, dizel yoqilg'isi, qo'shimchalar, moylash, aşınmaya qarshi xususiyatlar, ikkilamchi xom ashyo, EURO-6.

INTRODUCTION.

At present, the global oil and gas processing and petrochemical industries are developing rapidly, with ongoing reforms aimed at introducing new technologies for the processing of hydrocarbon raw materials. In our country, significant progress has been achieved in developing various products based on local raw materials and secondary resources, improving the preparation and processing of hydrocarbon feedstocks, and implementing innovative technologies to enhance both the quality and volume of production. In this regard, scientific research focused on increasing the efficiency of utilizing paraffinic hydrocarbon mixtures of various compositions formed during the development of oil fields is of great importance.

The technology of hydrogenation of higher fatty acid esters has made it possible to produce fatty alcohols on an industrial scale, and this process has been implemented in many countries with sufficient raw material bases. Currently, the majority of such fatty acids are synthesized from petroleum paraffins, which significantly reduces the demand for natural oil and fat raw materials.

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MATERIALS AND METHODS.

The development of technologies for breaking oil emulsions began in the 1920s in the United States with the industrial use of soap solutions. Numerous studies were conducted in which chemical compounds were synthesized and their efficiency was evaluated. As a result, reagents based on unsaturated fatty acids were found to be the most effective demulsifiers [1]. The first industrial demulsifiers were obtained based on mixtures of fatty acids and inorganic salts. Later, they were replaced by petroleum sulfonic acids and their ammonium salts. At subsequent stages, additives such as castor oil, glycerin, and high-molecular-weight fatty acids were introduced into the composition of demulsifiers. These modifications significantly improved demulsification efficiency and demonstrated higher performance compared to previously used anionic surfactants, namely sulfonates and oils.

New nonionic demulsifiers exhibited high stability and efficiency due to their عدم reaction with water-soluble metal salts present in emulsions. Moreover, they were capable of effectively breaking complex emulsions even when used in amounts two times lower than anionic surfactants.

Scientific and technological advances in oil refining and petrochemical industries have enabled the comprehensive utilization of hydrocarbon raw materials and significantly reduced the use of food-based feedstocks for technical purposes. As a result, the use of grain and potatoes for the production of ethanol for technical applications has been completely eliminated. The development of technologies for producing higher fatty acids, alcohols, and surfactants based on petroleum feedstocks has contributed to substantial savings in animal and vegetable fats [2]. In the paint and coatings industry, the use of petroleum-polymer resins and low-molecular-weight rubbers represents an effective example of replacing food raw materials with chemical materials. The implementation of these technologies has led to annual savings of approximately 50 thousand tons of vegetable oils.

However, in current practice, only a portion of unsaponifiable substances is separated from the obtained fatty acids; the acids are not purified, not fractionated, and are used as mixtures of oxidation products. Such products do not fully replace natural fats, and this approach to paraffin utilization cannot be considered efficient. Further development of the higher fatty acid industry should be based on the максимально эффективное использование исходного сырья (paraffin) and the production of high-quality, well-fractionated distilled fatty acids. Although several plants producing distilled higher fatty acids exist in our country, their capacity is insufficient. Therefore, solving this problem is currently of great importance, especially considering that higher fatty acids and alcohols are still imported from foreign countries using foreign currency resources [3].

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At present, in industry, higher fatty acids are divided into the following fractions: low-molecular-weight water-soluble C_1–C_4 acids, which are removed вместе with water. The main raw material for their production is paraffin, from which C_{10}–C_{16} and C_{17}–C_{20} fractions are obtained [4].

A large body of industrial research data exists, demonstrating with sufficient accuracy how specific technological parameters affect the efficiency and quality of higher fatty acids obtained during the processing of solid petroleum paraffins. A detailed study of higher fatty acids shows that they have a complex composition. In addition to simple monocarboxylic acids, they contain branched-chain radicals, radicals with naphthenic rings, dicarboxylic acids of various structures, unsaturated, hydroxy-, and keto acids, as well as certain amounts of hydrocarbons and neutral oxygen-containing compounds. The content of individual components depends on the composition of the raw materials and the technological processes used for obtaining and separating fatty acids, which ultimately determines their fields of application [5].

Higher fatty alcohols (HFA) are among the main types of organic raw materials used to produce a wide range of products. Conventionally, HFA include monohydric alcohols containing more than five carbon atoms in their molecules. This group includes primary and secondary alcohols with both even and odd numbers of carbon atoms, as well as saturated and unsaturated compounds.

Such alcohols are produced by various synthesis methods based on petrochemical feedstocks, as well as by the reduction of acids obtained from natural fats and oils.

According to their consumer properties, higher fatty alcohols can be conditionally classified into the following groups: individual alcohols such as C_8, C_{10}, and C_{13}, as well as fractions consisting of homologues ranging from C_6 to C_{13}; and fractions composed of homologues with carbon numbers of C_{10} and higher (mainly C_{10}–C_{18}). Alcohols in the C_{10}–C_{18} range are widely used in the production of surfactants and related products, especially high-performance detergents. Surfactants based on higher fatty alcohols exhibit good biodegradability, low toxicity, and effective washing performance in hard water [6].

RESULTS AND DISCUSSIONS.

In industry, the oxidation of higher paraffins can be carried out in both batch and continuous modes.

The oxidation process is typically performed in vertical columns made of aluminum with a capacity of 8–12 m³, since low-molecular-weight acids cause severe corrosion. Oxidation occurs with the release of a significant amount of heat. For example, when oxidizing paraffin until the acid number of the oxidate reaches 70 mg KOH/g, approximately 2095 kJ of heat is released per 1 kg of oxidized

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paraffin. Heat is removed from the column through internal coils cooled by water. The process is conducted at atmospheric pressure, and the oxidation duration ranges from 20 to 24 hours. Industrial installations are equipped with multiple reactors to ensure the required production capacity. The oxidate formed during the oxidation process is treated with soda and alkali (this process is known as saponification). Fatty acids are rapidly neutralized and transferred into a soap solution [7].

The unsaponifiable fraction I, consisting of unoxidized paraffins containing oxygenated compounds, is recycled back to the oxidation stage. In addition to fatty acids, the oxidate also contains small amounts of hydroxy acids, esters, lactones, ketones, and alcohols, which are saponified only under severe conditions. These compounds dissolve in the soap solution along with small amounts of paraffin and exhibit surface-active properties.

However, their removal is necessary, as they ухудшают detergent and foaming properties of the soap and impart an unpleasant odor (especially hydroxy acids). For this purpose, the soap solution is subjected to heat treatment at 320 °C, resulting in a “refining” process: lactones and ketones undergo saponification, while hydroxy acids are dehydrated.

$$RCH_2 - CO - CH_2R + NaOH \rightarrow RCH_2 - COONa + CH_2R$$

$$RCH_2 - \underset{\underset{OH}{|}}{CH} - CH_2 - COOH + H_2O \rightarrow RCH = CH - CH_2 - COOH$$

During heat treatment, the unsaponifiable fraction II is stripped from the soap solution by superheated steam, which is either generated by the evaporation of water present in the soap or supplied externally. The separated unsaponifiable fraction II is recycled back to the oxidation stage [8]. The technological scheme for obtaining higher fatty acids is shown in Figure 1. The catalyst solution prepared in vessel 1 is mixed with paraffin in apparatus 2. A 15% solution of potassium permanganate (at a loading of 0.1–0.3%) is introduced into paraffin heated to 120 °C. Water evaporates, while $KMnO_4$ becomes dispersed in the paraffin. The paraffin–catalyst mixture is then fed into the oxidation column 3, where purified air is supplied.

The temperature in the column is maintained at 105–115 °C using heating and cooling coils. During the induction period (2–3 hours), heat is supplied to the column by feeding steam into the lower coil. The outgoing air carries water vapor, low-molecular-weight acids, and other volatile products, which are condensed in cooler 4. The condensate is separated in separator 5 and sent to collector 7. The cooled air is washed in column 6 and then directed to the furnace. The washing

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water from column 6 is also sent to collector 7, where the mixture separates into two layers: an upper (organic) layer and a lower (aqueous) layer. The aqueous phase is either collected or sent to washing column 9 for oxidate purification. This phase may contain C_1–C_4 acids [9]. The oxidate from the reactor is directed to sludge settler 8 and then to washing column 9 to remove water-soluble low-molecular-weight acids.

Clean water and aqueous condensate from collector 7 may be supplied to the column. The washed oxidate is fed into saponifier 10, where acids are neutralized with a 25% soda solution at 90–100 °C (first stage of saponification), and then transferred to apparatus 11, where sodium hydroxide solution is used (second stage). The saponified product is separated in settler 12 at 70 °C into unoxidized paraffin (unsaponifiable I) and soap solution. The paraffin is recycled to the oxidation stage, while the soap is fed under a pressure of 2 MPa into furnace 13, heated by flue gases. In the furnace, the temperature is gradually increased: first to 180 °C, then to 220 °C, and finally to 320 °C. After heating, the soap enters an evaporator, where at atmospheric pressure the unsaponifiable fraction II is stripped by superheated steam and transferred to absorption column 17 irrigated with cold water from settler 18. The condensate and water are collected in settler 18, from which the unsaponifiable fraction II is recycled to the oxidation stage. The dry soap is diluted in vessel 14 with an aqueous solution of sodium sulfate, after which the soap solution is decomposed in vessel 15 with 92% sulfuric acid at 100 °C under intensive mixing. The resulting mixture of higher fatty acids and sodium sulfate is washed with water in column 16. The washed acids are then sent for distillation (not shown in the scheme). Equipment used for treating soap solutions with sulfuric acid is coated to protect against corrosion [10]. The obtained acids are distilled in stills equipped with packed columns. The stills are made of acid-resistant cast iron, while the coils and columns are made of stainless steel [10]. As the molecular weight of the acids increases, the temperature in the stills is increased and the residual pressure is reduced.

Table 1

Distillation conditions and yield of higher fatty acids

Still №	Temperature, °C	Residual pressure, Pa	Acid fractions	Acid yield, % (wt. of feed)
1	160-180	266-400	C_5 - C_6	4-5
2	170-180	26,6	C_7 – C_9	12-13
3	185	20	C_{10} – C_{13}	-
4	200	20	C_{14} – C_{16}	65
5	280	20	C_{17} – C_{20}	-

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6	310	20	C_{20}	4-5

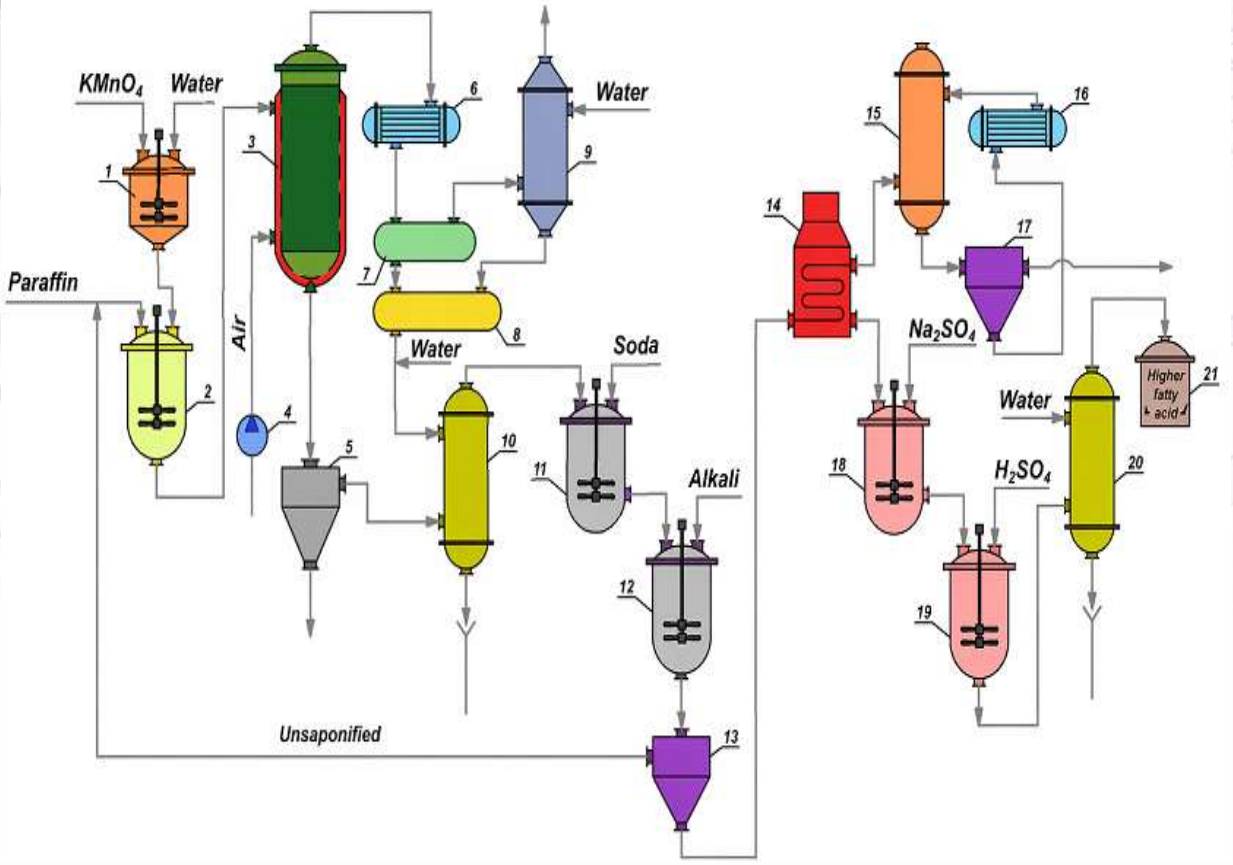


Figure 1. Technological scheme for obtaining higher fatty acids by oxidation of paraffin: *1, 18, 19 – mixing tanks; 2 – mixer; 3 – oxidation column; 4 – air compressor; 5 – sludge settling tank; 6, 16 – heat exchangers; 7 – separator; 8 – collection tank; 9 – washing scrubber; 10, 20 – washing columns; 11, 12 – saponification units; 13, 17 – settlers (clarifiers); 14 – heating furnace; 15 – absorption column; 21 – higher fatty acid tank.*

The use of unsaponifiable-II fractions for obtaining alcohols, as well as the separation of low-molecular acids from water condensate (scrubber) and from washing waters after oxidate washing, can improve the economic indicators of the process.

During the analysis, it was found that higher fatty acids synthesized from local secondary raw materials positively affect the operational properties of diesel fuels, improving their viscosity, lubricity, and anti-wear characteristics [12].

CONCLUSION

Thus, higher fatty acids obtained based on the proposed technology can compete with imported branded additives purchased from abroad. The synthesized higher fatty acids enable the production of environmentally clean diesel fuels at domestic oil refining plants with improved operational properties, enhanced lubricity and anti-wear characteristics, and a sulfur content that fully meets EURO-6 requirements.

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