Современная наука и инновации.
2023. № 4 (44). С. 72-78.
Modern Science and Innovations.
2023;4 (44):72-78.

*ТЕХНОЛОГИЯ ПРОДОВОЛЬСТВЕННЫХ ПРОДУКТОВ /
TECHNOLOGY OF FOOD PRODUCTS*

Научная статья / Original article

УДК 664. 959.5:665.211
https://doi.org/10.37493/2307-910X.2023.4.8

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Переработка отходов производства рыбных жиров в смазочную добавку

The processing of fish oil production waste into a lubricating additive

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Аннотация. *Поиск и разработка рациональных направлений использования жировых отходов, образующихся при производстве рыбных жиров, является актуальной экологической задачей рыбной отрасли. В настоящее время эта проблема остается не решенной. Соапсток после рафинации рыбных жиров и жировая пенная масса после флотационной очистки производственных сточных вод производства рыбных жиров содержат в своем составе значительное количество полиненасыщенных жирных кислот и мыл. Указанные компоненты могут быть основой для получения различных технических продуктов. Установлено, что соапсток и модифицированная (омыленная) жировая пенная масса обладают анфрикционными и поверхностно-активными свойствами. Это позволяет определить направление их использования в качестве смазочной добавки к буровому раствору. Исследование экологической безопасности новой смазочной добавки на основе модифицированной жировой пенной массы позволило отнести её к малоопасным веществам, определить предельно допустимую концентрацию в воде рыбохозяйственных водных объектов и рекомендовать к использованию при разработке морских нефтегазовых месторождений.*

Ключевые слова: рыбный жир, соапсток, жировая пенная масса, антифрикционные свойства, поверхностно-активные свойства, предельно допустимая концентрация, смазочная добавка, буровой раствор

Для цитирования: *Петров Б. Ф. Переработка отходов производства рыбных жиров в смазочную добавку // Современная наука и инновации. 2023. № 4 (44). С. 72-78. https://doi.org/10.37493/2307-910X.2023.4.8*

Abstract. *The search and development of rational uses of fatty waste formed in the production of fish oils is an urgent ecological problem of the fishing industry. Currently, this problem remains unresolved. Soap stock after refining fish oils and fatty foam after flotation treatment of industrial waste water from fish oil production contain a significant amount of polyunsaturated fatty acids and soaps. These components can be a basis for obtaining various technical products. It was found that soap stock and modified (saponified) fatty foam mass have antifriction and surface-active properties. This allows to determine the field of their use as a drilling mud lubricant. The study of the environmental safety of a new lubricant additive based on modified fatty foam mass made it possible to classify it as a low-hazard substance, determine the maximum permissible concentration in the water of fishery water bodies, and recommend it for use in the offshore oil and gas fields development.*

Keywords: fish oil, soap stock, fatty foam mass, antifriction properties, surface-active properties, maximum permissible concentration, lubricant additive, drilling mud/drilling fluid

For citation: *Petrov BF. The processing of fish oil production waste into a lubricating additive. Modern Science and Innovations. 2023;4(44):72-78. (In Russ.). https://doi.org/10.37493/2307-910X.2023.4.8*

Introduction. Food waste is a serious environmental problem. Thus, in the process of refining fish oils, soap stocks are formed containing up to 7% neutral fat and up to 16% sodium salts of fatty acids. During flotation treatment of industrial wastewater from fish oil production, fatty foam masses are formed containing up to 70% lipids. To date, these wastes are not used rationally [1].

Currently, natural fats and their derivatives are increasingly being used in drilling fluids in the oil and gas industry. Fatty components are introduced into the drilling fluid to reduce friction and increase anti-wear properties. The use of natural fatty components as a lubricant additive increases the environmental safety of the drilling fluid [2].

It is necessary to study the possibility of using fatty waste generated during the production of fish oils as a lubricant additive to drilling fluid. The environmental safety of the proposed lubricant additive should also be assessed. This will make it possible to use it in the development of offshore oil and gas fields, incl. in the Arctic zone.

Materials and research methods. The purpose of the study was to develop a direction for the rational use of fatty waste from the production of fish oils as an anti-friction lubricant composition.

To achieve this goal, the following tasks were solved: studying the chemical composition and properties of the research objects; development of a method for producing a lubricant additive for drilling fluid based on them; study of the properties and environmental safety of the resulting lubricant additive.

The objects of the study were: soap stock formed as a result of the refining of fish oils; fatty foam formed during flotation treatment of industrial wastewater from the production of fish oils; model of drilling fluid in the form of an aqueous suspension of clay powder. Traditional lubricating additives used in the oil and gas industry were used as objects of comparison: synthetic fatty acids according to T U 38-50712-87 [3], a mixture of vegetable oils and liquid glass [4], a mixture of vegetable oils and boron compounds [5].

The chemical composition of the research objects was determined by standard methods in accordance with GOST 7636-85[1]. The content of sodium soaps was determined by titrating the sample with hydrochloric acid, first in the presence of phenolphthalein, then methyl red[2]. The fractional composition of lipids was determined using current layer chromatography [6]. The fatty acid composition of lipids was determined using gas chromatography in accordance with GOST 31663-2012[3]. The antifriction properties of the lubricant additive (friction coefficient, wear rate of steel grade U8) were studied using an MT -2 friction machine according to standard methodology in accordance with STO Gazprom 2-3.2-011-2005[4]. The main parameters of the model drilling fluid (density, viscosity, filtration index, shear stress, pH of the medium, electrical resistivity) were determined by standard methods[5]. The surface-active properties of the study objects were assessed by the decrease in surface tension (at the interface with air) and the decrease in interfacial tension (at the interface with heptane) [7]. The environmental safety of the lubricant additive was assessed using the “maximum permissible concentration” indicator, determined using standard methods[6].

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[1]GOST 7636-85. Fish, marine mammals, marine invertebrates and their products. Methods of analysis. Moscow, 2010.

[2]Guide to research methods, technical and chemical control and production accounting in the oil and fat industry / VNIIZH; under general ed. V. P. Rzhekhina, A. G. Sergeeva. T. 3. L. 1964. 494 p.

3GOST 31663-2012. Vegetable oils and animal fats. Determination by gas chromatography of the mass fraction of methyl esters of fatty acids. Moscow, 2019.

4STO Gazprom 2-3.2-011-2005. Drilling fluids. Methodology for measuring wear rate and friction coefficient on an MT-2 friction machine. Moscow, 2005.

5 GOST 33213-2014 (ISO 10414-1:2008). Monitoring the parameters of drilling fluids in field conditions. Moscow, 2015.

[6] Order of the Federal Agency for Fisheries dated 08/04/2009 No. 695 (as amended on 12/22/2016) “On approval of the Guidelines for the development of water quality standards for water bodies of fishery importance, including standards for maximum permissible concentrations of harmful substances in the waters of water bodies of fishery importance.”

Research results and their discussion. A study of the chemical composition of soap stock showed that it contains on average 80 % water, 7 % lipids, 12 % soap, 1 % minerals. Soapstock lipids contain on average 74 % polyunsaturated fatty acids.

The fatty foam mass contains on average 30 % water, 60 % lipids, 8 % soap, 0.08 % nitrogenous substances, 1.9 % minerals. Lipids in fatty foam contain on average 50 % triglycerides, 30 % free fatty acids, 10 % diglycerides, 10 % monoglycerides and hydroxy acids. The average fatty acid composition of lipids is: 38 % polyunsaturated fatty acids, 37 % monounsaturated fatty acids, 25 % saturated fatty acids.

The chemical composition of fat waste indicates that it can be a source of polyunsaturated free fatty acids and soaps. These compounds can form the basis of a lubricant additive to drilling fluid. The presence of fatty acid salts (soaps) ensures uniform distribution of the lubricant additive in the aqueous environment of the drilling fluid. However, with an increase in the amount of soap in the lubricant additive, its antifriction properties decrease. Therefore, it is necessary to search for the optimal ratio of fat phase and soap in the lubricant composition.

Soapstock contains a sufficient amount of soap, so there is no need to subject it to additional – saponification processing.

The soap content in the fatty foam mass is not enough to distribute it evenly in the drilling fluid. Therefore, the lipids of the fatty foam mass were additionally subjected to saponification 30 % alcohol solution of sodium aluminum methyl siloxanolate.

The main criteria for choosing a saponifying agent were: the presence of free alkali, widespread use in drilling wells, the presence of ethyl alcohol as a preservative and a substance that synergistically enhances the lubricating effect of fatty acids. The ratio of saponifying reagent: fatty foam mass was 1:10. The fatty foam mass after saponification had a pasty consistency, the color varied from white to light yellow. The physical and chemical characteristics of the product were: density 0.98…1.04 g/cm ^{3}, acid number 5…20 mgKOH/g, pH of 1% aqueous solution 8.0.

To evaluate the effectiveness of antifriction properties, soap stock and saponified fatty foam mass were introduced into a model drilling fluid in an amount of 1 % and studied the change in the friction coefficient and wear rate of steel. The friction coefficient characterizes the lubricity of the reagent under study. The reduction in the coefficient of friction occurs due to the formation of boundary layers with low shear resistance and high compression resistance on the interacting surfaces. The mechanism of formation of boundary layers and their properties are considered in [8]. As a result, the influence of friction and adhesion forces is reduced. The wear rate characterizes the strength of the boundary lubricating layer at high specific pressures.

Soapstock increases the antifriction properties of the model drilling fluid. Thus, when adding 0.5 to a clay suspension % soap stock, the friction coefficient value is reduced by 40 % And is 0.28, and the wear rate of steel is reduced by half and is 3.8 mm / h. The optimal soap stock content is 1.5…2.0 % by weight of drilling fluid. A further increase in its content in the composition does not increase the antifriction properties of the drilling fluid [9].

The introduction of soap stock into a clay suspension improves its filtration characteristics - the filtration index value decreases. This reduces the electrical resistivity of the suspension, which is an undesirable effect for the drilling fluid. For example, introducing soap stock into a model drilling fluid in an amount of 1.5 % reduces the value of the specific electrical resistance of the solution from 5.5 to 3.15 Ohm·m.

Saponified fatty foam also has a positive effect on the antifriction properties of the model drilling fluid. Thus, when introducing 1.5% saponified fatty foam mass into a clay suspension, the friction coefficient decreases nine times and is 0.1, and the wear rate of steel is reduced by four times and amounts to a specific load 20 MPa 1 mm / h

The introduction of the reagent stabilizes the technological parameters of the drilling fluid. In particular, the filtration index value decreases, the pH and electrical resistivity of the composition are stabilized.

A study of the shelf life of a lubricant additive based on saponified fat foam mass showed that during the year, separation of the dispersed system and separation of the aqueous phase from it were not observed. The reagent maintains stability and homogeneous structure.

Traditional lubricant additives used in the oil and gas industry were chosen as objects of comparison: synthetic fatty acids, mixtures of vegetable oils and liquid glass, mixtures of vegetable oils and boron compounds. These lubricant additives contain vegetable oils or their derivatives. Therefore, in terms of environmental safety, they are similar to lubricating additives based on fatty waste from the production of fish oils. The amount of traditional lubricant additives added to the model drilling fluid was 1.5 %.

Studies of the antifriction properties of traditional lubricant additives have shown that they are inferior to saponified fatty foam. The introduction of saponified fatty foam into the model drilling fluid reduces the wear rate of steel at a specific load of 20 MPa is four times, and the friction coefficient is two times compared with similar indicators of traditional lubricant additives.

Soapstock's antifriction properties are somewhat inferior to traditional lubricating additives and saponified fatty foam mass. Introduction to Model Drilling Fluid 1.5 % soap stock reduces the wear rate of steel at a specific load of 20 MPa to 2.5 mm/h, 1.5 % of traditional lubricant additives up to 1.5 mm/h, 1.5 % saponified fat foam mass up to 1.0 mm/h The coefficient of friction when introducing the same amount of soap stock into a model drilling fluid is 0.25, traditional lubricating additives from 0.22 to 0.17, saponified fatty foam mass is 0.1. Apparently, this is due to the significant content of fatty acid salts (soaps) in soap stock, which reduces its effectiveness as a lubricant. The lubricating properties of soap stock can be improved by chemical modification, for example, with organometallic compounds.

Filtrates of a model drilling fluid with the addition of soap stock or saponified fat foam were examined for the presence of surface-active properties. The presence of these reagents in the filtrates in an amount of 1.5 % reduces the surface tension of the composition by 1.5...1.7 times and amounts to from 36 to 43 mN / m, and interfacial tension is 4.0…4.7 times and amounts to from 10 to 12 mN / m. A further increase in the content of reagents in the filtrates has little effect on their surface-active properties. The inclusion of soap stock or saponified fatty foam into the drilling fluid as a surfactant helps prevent clay particles from sticking to the drilling tool and to each other, increasing the drilling speed and improving the lubricity of the drilling fluid.

Due to its high anti-friction properties, the saponified fat foam mass was subsequently considered as a lubricant additive. The use of this reagent in the extraction of hydrocarbons in marine conditions requires an assessment of its effect on aquatic biological objects and on the hydrochemical regime of fishery reservoirs.

The toxicological experiment was carried out on unicellular algae, lower crustaceans, one-year-old rainbow trout and saprophytic microflora.

For unicellular algae, the boundary concentration of saponified fatty foam mass in water was 25.6 mg/ dm^3. At this concentration, biological objects showed a decrease in photosynthesis. This indicates the toxic effect of the reagent on them. The ineffective concentration of the reagent for unicellular algae was 12.8 mg/dm^3.

The lethal dose of the reagent for lower crustaceans was over 12.8 mg/ dm^3. At a reagent concentration of no more than 3.2 mg/$dm^{3,}$ no inhibitory effect on biological objects was observed.

For one-year-old rainbow trout, the inactive concentration of the reagent was no higher than 50 mg/ dm^3.

In terms of acute toxicity, the reagent under study belongs to low-hazard substances (fourth hazard class). The limiting hazard indicator is sanitary.

The effect of saponified fat foam on the chemical composition of water was also studied.

The experiment showed that when the concentration of the reagent in water is not higher than 1.0 mg/ dm^3 oxygen content on the third day of exposure remains at the level of the control experiment (without reagent) and is 8.7 mgO_2 / dm^3. When the concentration of the reagent in water is above 1.0 mg/dm^3, the content of dissolved oxygen decreases.

The concentration of the reagent in water above 10.0 mg/dm^3 shifts the pH of the medium to the alkaline region. However, on the thirtieth day of exposure, the pH of the environment returns to the neutral region.

Increasing the concentration of the reagent increases the oxidation of the environment. Thus, on the thirtieth day of exposure at a reagent concentration of 10 mg/dm^3, the oxidation of the medium was 5.18 mgO_2 /dm^3, and at a reagent concentration of 100 mg/dm^3, the oxidability was 6.54 mgO_2 / dm^3.

Reagent concentrations above 10 mg/dm^3 contribute to the accumulation of ammonium, nitrite and nitrate ions in water, as well as an increase in the “biological oxygen consumption” indicator.

An increase in the concentration of the reagent above 1.0 mg/dm^3 in the first three days of exposure promotes an increase in the number of saprophytic microorganisms and then inhibits their development.

A study of the influence of saponified fatty foam on the organoleptic properties of water showed that when the reagent concentration exceeds 1.0 mg/dm^3, the natural color and smell of the medium changes. Heating water up to 60 ^oC contributes to the appearance of a specific odor even at reagent concentrations above 0.5 mg/ dm^3.

Thus, according to the results of studies of hydrochemical parameters of water, the maximum permissible concentration of the test reagent should not exceed 0.5 mg/ dm^3. Taking into account the safety factor (2.5), the maximum permissible concentration of the reagent in the water of fishery water bodies is set to no more than 0.2 mg/ dm^3.

Introduction 1.5 % of saponified fatty foam mass in the drilling fluid provides it with good anti-friction properties and does not exceed the established maximum permissible concentration of the reagent in water.

The antifriction and surfactant properties of various fatty wastes were previously noted in the works of a number of authors. In particular, fatty waste from food production is proposed to be used in the composition of cutting fluids for metal processing, in anti-adhesive and anti-corrosion compositions, and as a flotation reagent in the enrichment of apatite nepheline ore [1, 10, 11, 12].

Conducted studies of the antifriction and surface-active properties of fatty waste from the production of fish oils indicate the possibility of their use as a lubricant additive to drilling fluid.

Recently, the possibility of using vegetable and animal fats in lubricant additives has been actively studied due to the tightening of environmental requirements for reagents used in drilling fluids [2, 13, 14]. Fatty substances of natural origin, compared to synthetic reagents, are easily biodegradable, which indicates their environmental safety. However, the use of such lubricating additives in the extraction of hydrocarbons in marine conditions requires an assessment of their environmental safety, as well as the establishment of maximum permissible concentrations in the water of fishery water bodies [15].

A study of the influence of a lubricant additive based on a modified fatty foam mass on aquatic biological objects and the hydrochemical regime of fishery reservoirs made it possible to establish its environmental safety and recommend it for use in the development of offshore oil and gas fields.

Conclusion. The chemical composition of fatty waste (soap stock and fatty foam) formed during the production of fish oils has been studied. The presence of polyunsaturated fatty acids and soaps in fatty waste allows us to determine the possible direction of their use as an anti-friction lubricant.

Soapstock's antifriction properties are inferior to lubricant additives traditionally used in the oil and gas industry. Apparently, the increased content of fatty acid salts (soaps) worsens its properties as a lubricant. It is advisable to investigate the possibility of improving the lubricating properties of soap stock by modifying it (for example, with organometallic compounds).

The fatty foam mass must be saponified to improve its functional properties. The resulting product has anti-friction properties superior to traditional lubricant additives used in the oil and gas industry. The high lubricity of the new reagent is ensured due to the optimal ratio of the fat component and saponifying agent.

Soapstock and saponified fat foam have good surface-active properties. The use of these reagents in the drilling fluid helps reduce the adhesion of clay particles to the drilling tool and to each other, increases the drilling speed and improves the lubricity of the drilling fluid.

The influence of saponified fatty foam on aquatic biological objects and the hydrochemical regime of fishery reservoirs has been studied. This made it possible to establish the maximum permissible concentration of the test reagent in the water of fishery water bodies of no more than 0.2 mg/dm ^{3}. The hazard class of the reagent has been determined - fourth, low-hazard substances. The limiting hazard indicator is sanitary.

The research results allow us to recommend a new lubricant additive for use in the development of offshore oil and gas fields.

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Конфликт интересов: автор заявляет об отсутствии конфликта интересов.
Conflict of interest: the author declares no conflicts of interests.

Статья поступила в редакцию: 28.09.2023;
одобрена после рецензирования: 02.11.2023;
принята к публикации: 06.12.2023.

The article was submitted: 28.09.2023;
approved after reviewing: 02.11.2023;
accepted for publication: 06.12.2023.