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PREPARATION OF SURFACTANTS BASED ON FATTY ACIDS AND ETHANOLAMINES

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Abstract. The reactions of oleic acid and cottonseed oil with mono- and diethanolamine from alkanolamines were studied in order to obtain surfactants based on fatty acids and alkanolamines. The composition and structure of the resulting acid amides were analyzed using IR spectroscopy, the amount of formed amides was determined by the amount of free acids in the reaction mixture, and in the region of average intensity 1622,15 cm^{-1}, the carbonyl group of the acid C=O and deformation vibrations of amines in the form of salts are characteristic N-H, which is confirmed by the existence of $N^{+}OOC$ of amine salts in free acid.

Basic words and phrases: fatty acids, oleic acid, cottonseed oil, alkanolamines, surfactant, amidation, hydrophilic, hydrophobic, oil in water

Abstract. In order to obtain surfactants based on fatty acids and alkanolamines, the reactions of oleic acid and bellows oil with mono- and diethanolamine have been studied. The composition and structure of the resulting amides are analyzed by IR spectroscopy, the amount of amides formed is determined by the amount of free acid and the reaction mixture. It has been proved that at an average intensity of 1622,15 cm^{-1}, the region belongs to the deformation vibrations of the ammonium salt $N^{+}OOC$, formed by the carbonyl groups of the fatty acid C = O and the amino group of alkanoamines.

Key words and phrases: fatty acids, oleic acid, cottonseed oil, alkanolamines, surfactants, amidation, hydrophilicity, hydrophobicity, oil in water.

Annotation. The reactions of oleic acid and cottonseed oil with mono- and diethanolamine from alkanolamines were studied in order to obtain surfactants based on fatty acids and alkanolamines. A chromatographic analysis of fatty acids present in cottonseed oil has been carried out, it has been proved that oleic, linoleic and linolenic acids make up more than 80%, and the optimal conditions for the amidation processes have been determined. The composition and structure of the resulting acid amides were analyzed by IR spectroscopy, the amount of formed amides was determined from the amount of free acids in the reaction mixture. The ratios of hydrophilic and hydrophobic groups of the obtained substances have been studied, it has been shown that they correspond to the "oil in water" type, and are recommended for use in the production of detergents.

Key words and phrases: fatty acids, oleic acid, cottonseed oil, alkanolamines, surfactants, amidation, hydrophilicity, hydrophobicity, oil in water.

Today, with an increase in the population of the globe, the need for hygiene products is increasing, in particular, in laundry and perfume soaps based on solid technical, vegetable and animal oils. Especially important is the use of biological surfactants in the technology of soap making, to improve the technology for the production of hygienic products with enhanced detergent properties [1-3].

There is a lot of information about the production of detergents, the composition of which is enriched with surfactants, such as alkylbenzenesulfonic acid and sodium lauryl sulfate, with improved quality and physicochemical properties, which save material and technological costs [4–7]. For example, using new sources of raw materials, dyes, flavors and surfactants to create perfume soaps with improved detergent properties, the following new composition is widely used (table 1).

The table shows that the total amount of surfactants added to perfume soap from modern hygiene products is 1-3%. It is known that, according to its chemical composition and changes in solutions, it is subdivided into ionic and nonionic SFM [8–12]. Ionic SPMs, in turn, are subdivided into anionic, cationic, and ampholytes. Recently, sodium lauryl sulfonate (or sodium dodecyl sulfonate), which is widely used on an industrial scale, is produced in large quantities, and is added not only as a detergent and cleaning agent to shampoos, toothpastes, creams, but also as a diagnostic drug in medical and biological research, in the production of galvanic coatings, in the treatment of extreme temperatures, are used in the production of materials that are resistant to changes and mechanical stress.

Table 1

The composition and constituent components of modern perfume soaps

Raw material name	Unit of measurement	A type of perfumed soap		
		Pinokkio (for kids)	Palmera (general)	Olivia (additional)
Palm oil (Palm Olein IV 65)	%	38,1	37,4	67,4
Palm stearin (RBD PALM STEARIN)	%	10	10	10
Coconut oil (RCHO)	%	20	20	20
Technical oil-oil mixture (salomas)	%	30	30	-
EDTA	%	0,05	0,05	0,05
Bleach	%	0,05	0,05	0,05
Aromatic substance	%	1	1	1
Paint	%	0,000025	0,000025	0,000025
Alkyl lactate	%	0,1-0,3	-	-
Allantoin	%	0,1-0,3	-	-
Stearic acid	%	-	1	1
Sodium benzoate	%	0,5	0,5	0,5

This sodium dodecylsulfanate SFM is produced according to TU 6-09-07-1563-86, it is obtained in the sequence of sulfation of dodecyl alcohol with chlorosulfonic acid in an acetic solvent, treatment of the sulfonated mass with amyl alcohol, naphthalization, drying, recrystallization of the final product in ethanol [13].

These methods are associated with a number of problems, in the first case, expensive reagents are used, a lot of ethanol is consumed, and in the second case, the purity of the SFA depends on the individual separation of fatty acids.

Purpose of work It is defined as the improvement of production technology by obtaining surfactants based on fatty acids and ethanolamines, including detergents enriched with biologically active additives.

Experimental part. Pure oleic acid, cottonseed and pistachio oils were chosen as acid reagents in the studies, from which a mixture of acids was isolated by saponification.

Alkanolamines (mono-, di- and triethanolamines), which are increasingly used in organic synthesis as nitriding reagents, have been selected; composition of the hydrocarbon chain.

At the beginning of the research, in order to determine the optimal conditions for the occurrence of amidation processes, syntheses were carried out with pure fatty acids, including oleic acid: oleic acid and monoethanolamine were placed in a three-necked flask equipped with a stirrer and a thermometer. and the Dean-Stark device in a molar ratio of 1:1.

Gradually stirring, the temperature rises to 160 - 170 °C. After heating and stirring for 4 hours, determine the acid number of the mixture in mg KOH/g. The reaction is considered complete when the acid number of the process is 6.0 mg KOH/g, the smell of fatty acids is almost gone and a transparent brown solid is formed.

Analysis of results. As already mentioned, fatty acids are first loaded into the reactor, and to prevent foaming during reaction with alkanolamines, it is heated to 150-160 °C and excess water is removed. Then the temperature in the reactor is reduced to 70 - 80 °C and alkanolamines are added in calculated amounts with vigorous stirring. In this case, the reaction mass thickens, the temperature itself rises to 100 ± 5 °C, and the formation of ammonium salts of fatty acids is observed. The reactions of primary fatty acids with amines in equimolecular amounts proceed according to the following scheme:

$$RCOOH + N(CH_2CH_2OH)_n \longrightarrow RCOON(CH_2CH_2OH)_n + H_2O$$

where: R is the fatty acid residues mentioned above;*n = 1 - 3*.

Prolonged heating at 170-180 °C (except for the case of monoethanolamine) leads to the decomposition of ammonium salts with the formation of acid amides and water molecules. When choosing monoethanolamine as the alkanolamine during the reaction (since the boiling point at atmospheric pressure is 171 °C), it is advisable to keep the reaction temperature at a slightly lower limit of 150-160 °C, thereby preventing the loss of monoethanolamine. The reaction is maintained at these temperatures for 3-4 hours, gradually increasing to 170-180 °C.

table 2

Important physico-chemical properties of the resulting products

Name of pointers	Based on oleic acid	Based on cottonseed oil	Based on pistachio oil
External state	Pale yellow powder	Brown sticky mass	orange mass
Mass fraction of acid amides, wt %, not less than	87,0	97,0	98,0
Mass fraction of fatty acids, % wt., not much	7,0	0.2	0.0054
Mass fraction of chlorides, wt %, not much	1,5	0,1	0,00035
Mass fraction of alkanols, wt%, max, wt%, max	2,5	0,007	0,036
Mass fraction of heavy metal ions (in terms of lead), % wt., not more than	0,001	0,005	0,0047
Mass fraction of phosphates, wt %, not much	-	0,001	0,0018
optical density of 10% solutions (d cuvettes - 1 cm)	2,9	0,3	0,15

The composition and structure of the resulting acid amides were analyzed by IR spectroscopy.

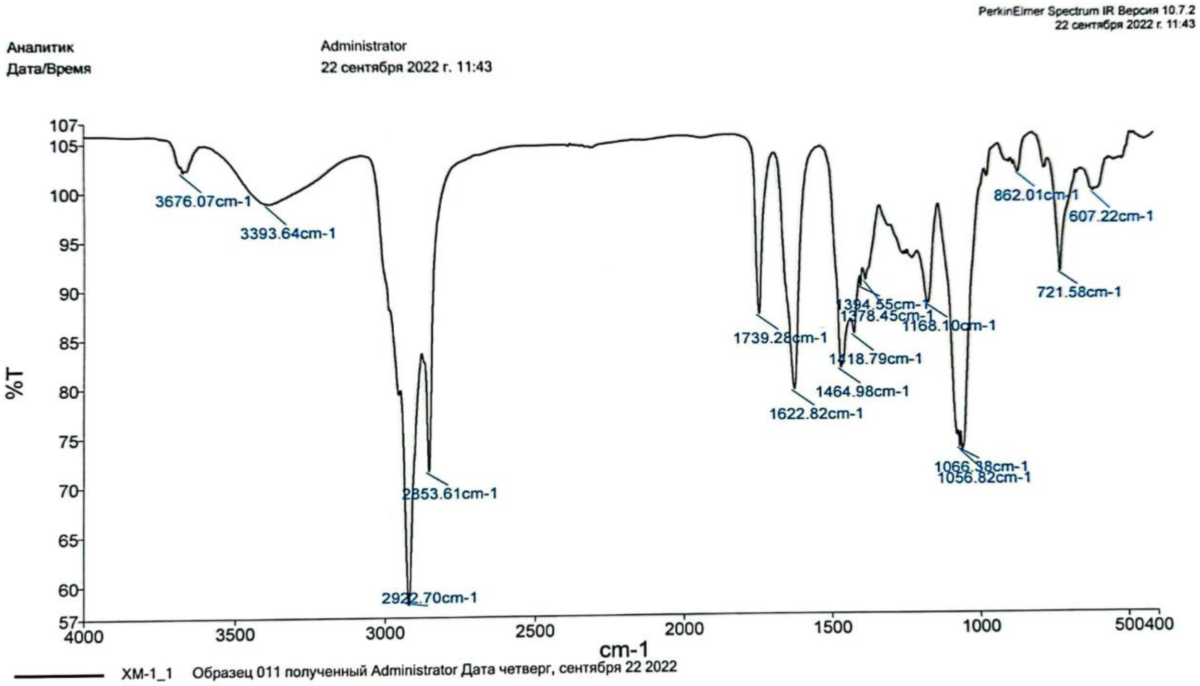

Figure 1. IR spectrum of the reaction product of oleic acid with diethanolamine

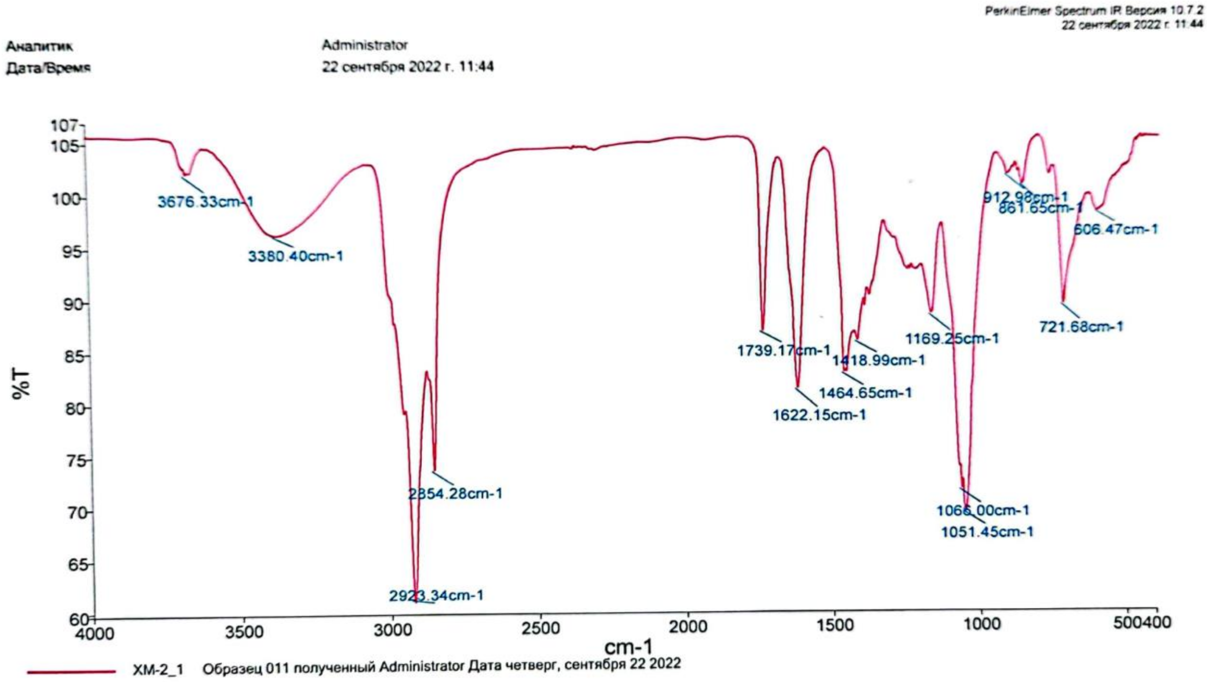

Figure 2. IR spectrum of the reaction product of cottonseed oil with diethanolamine

In the region of 861,65 cm^{-1} of the IR spectrum of the mixture of products based on cottonseed oil, the presence of bending vibrations in the double bond deviating from the plane characteristic of the C–H bond is seen, and at 721,68 cm^{-1} there is an area of intense vibrations characteristic of for group C–OH. In addition, in the region of 1065,00-1051,45 cm^{-1}, C–O bond stretching vibrations characteristic of alcohols were observed, which are considered to be the result of OH-deformation and C–O stretching vibrations. Absorptions in the region of 1169.25 and 1418,99-1464,65 cm^{-1} are characteristic of C–N stretching vibrations in amines, in the region of average intensity of 1622,15 cm^{-1} - of the carbonyl group of the acid C = O and bending vibrations of amines in salt forms N–H, $N^{+}OOC$ of amine salts in free acid prove its existence. Also visible in the IR spectrum are C=O stretching vibrations corresponding to carboxyl and esters at 1739,14 cm^{-1}, broad lines from the 2854,28–2923,34 cm^{-1} region to asymmetric and symmetrical stretching vibrations characteristic of methylene (-CH_2–) group and 3380,40. The presence of broad lines in the regions – 3676,33 cm^{-1} indicates the importance of hydroxyl (–OH) groups in alkanolamines.

The IR spectra of oleic acid and cottonseed oil show the presence of specific absorptions for functional groups of almost the same composition, differences are visible only in the regions of 3500-3300 cm^{-1} and 1078-1041 cm^{-1}.

Summary. The literature on the production of surfactants was analyzed, as well as data on the reactions of obtaining SPM based on sodium lauryl sulfonate, which is used in large quantities, and formulations of detergents with improved quality and physicochemical properties based on them were studied. The obtained IR spectrum was used to determine, and in the region of 1622,15 cm^{-1} of medium intensity, the carbonyl group of the acid C = O and the bending vibrations of amines in the form of N-H salts were proved by the presence of $N^{+}OOC^{-}$ of amine salts in the free acid.

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