

**СИНТЕЗ ПОВЕРХНОСТНО-АКТИВНОГО СРЕДСТВА ИЗ ПРИРОДНОГО СЫРЬЯ****Г.Г. Лутфуллина, Э.Э. Валеева**

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*Синтезировано поверхностно-активное средство на основе смеси пальмитиновой, олеиновой и стеариновой кислот, получаемых путем дистилляции и фракционирования масел и жиров, а также этилового спирта. Синтез проводили в 4 стадии, включающие образование смеси кислот, этерификацию, сульфирование и омыление. Предварительно хроматографическим методом изучен состав смеси жирных кислот. Выяснено, что наибольшее количество в смеси пальмитиновой кислоты (64,3%). Второе место занимает олеиновая кислота (29,1%). Исследованы строение и свойства поверхностно-активного средства с применением современных методов и приборов. Определен средний размер частиц средства: оно может быть отнесено к группе высокодисперсных систем с диаметром частиц от 10 нм до 1 мкм. Выявлено, что размер частиц поверхностно-активного средства увеличивается после достижения критической концентрации мицеллообразования. Оценка стабильности исследуемой системы показала, что  $\zeta$ -потенциал находится за пределами диапазона  $30 \text{ мВ} < \zeta < -30 \text{ мВ}$ . Проведенные исследования выявили наличие в полученном продукте поверхностно-активных свойств: моющих, эмульгирующих, смачивающих, пенообразующих. Согласно проведенным расчетам гидрофильно-липофильного баланса синтезированный продукт относится к моющим средствам. Наилучшие результаты по моющей способности по отношению к пигментно-масляному и белковому загрязнению проявляются при концентрации ПАВ  $4,0 \text{ г/дм}^3$  вне зависимости от вида ткани. Обнаружено, что эмульгирующая способность проявляется активно по отношению к индустриальному маслу. Анализ показателей поверхностного натяжения показал, что критическая концентрация мицеллообразования для исследуемого средства составляет  $4,0 \text{ г/дм}^3$ . Доказано, что оно обладает высокими смачивающими способностями вне зависимости от природы поверхности (гидрофильной или гидрофобной). При этом пенообразующие свойства удовлетворительные по сравнению с известными поверхностно-активными веществами из нефтехимического сырья, однако наблюдается стабильное пенообразование, прямо зависящее от концентрации полученного продукта.*

**Ключевые слова:** поверхностно-активные вещества, структура, свойства, поверхностное натяжение, угол смачивания, применение

**SYNTHESIS OF SURFACTANTS FROM NATURAL RAW MATERIALS****G.G. Lutfullina, E.E. Valeeva**

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*This article presents a surfactant synthesized from a mixture of palmitic, oleic, and stearic acids obtained by the distillation and fractionation of oils and fats, as well as ethanol. The synthesis was carried out in 4 stages, which involved the initial formation of an acid mixture, followed by esterification, sulfonation, and ultimately saponification. The composition of the fatty acid mixture was analyzed using chromatography, revealing that palmitic acid makes up the largest proportion at 64.3%, followed by oleic acid at 29.1%. The structure and properties of the surfactant were investigated using modern methods and instruments. The average particle size of the surfactant indicates that it can be classified as a highly dispersed system, with particle diameters ranging from 10 nm to 1  $\mu$ m. It was observed that the particle size of the surfactant increases once the critical concentration of micellization is reached. The stability of the system in the substance is indicated by a  $\zeta$ -potential that falls outside the range of  $30 \text{ mV} < \zeta < -30 \text{ mV}$ . The resulting product exhibits good surface-active properties such as detergency, emulsification, wetting, and foaming ability. According to hydrophilic-lipophilic balance calculations, the synthesized surfactant is classified as a detergent. It demonstrates a high ability to remove pigment-oil and protein contamination at a concentration of  $4.0 \text{ g/dm}^3$ , regardless of the fabric type. Additionally, it was found that the synthesized surfactant exhibits significant emulsifying ability towards industrial oils. The analysis of the surface tension of the surfactant showed that its critical concentration for micelle formation is  $4.0 \text{ g/dm}^3$ . The surfactant demonstrates high wetting abilities on both hydrophilic and hydrophobic surfaces. The foaming properties of the surfactant are satisfactory compared to known surfactants from petrochemical raw materials. Moreover, stable foaming is observed, which directly depends on the surfactant concentration.*

**Keywords:** surfactants, structure, properties, surface tension, contact angle, application

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## INTRODUCTION

Nowadays, surfactants are widely used in numerous industrial applications [1-20]. Surfactants have found wide industrial applications due to their ability to intensify technological processes at low concentrations and modify surfaces, improving the required properties. Most surfactants are produced from petrochemical feedstock. However, current problems in the global oil market may lead to higher prices for petrochemicals. Therefore, synthesizing surfactants from plant materials has become a solution to this problem. Additionally, surfactants synthesized from plant materials are environmentally friendly [21-32].

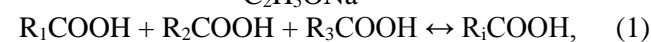
The research aims at synthesizing an anionic surfactant based on three fatty acids: palmitic acid, oleic acid, and stearic acid, along with ethanol. These acids are the constituents of palm oil.

Raw materials of plant and animal origin were used to produce this surfactant. Compared to common anionic surfactants, our product is monodisperse, with an increased detergent effect. It also exhibits high aggregative and sedimentation stability, as well as increased resistance to water hardness.

## EXPERIMENTAL PART

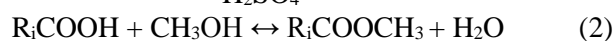
The anionic surfactant was synthesized as follows:

1) Transesterification reaction:

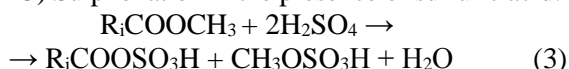


where  $\text{R}_1\text{COOH} = \text{C}_{16}\text{H}_{32}\text{O}_2$ ,  $\text{R}_2\text{COOH} = \text{C}_{18}\text{H}_{34}\text{O}_2$ ,  $\text{R}_3\text{COOH} = \text{C}_{18}\text{H}_{36}\text{O}_2$ ,  $\text{R}_1\text{COOH}$  is a mixture of three acids: palmitic acid; oleic acid; and stearic acid.

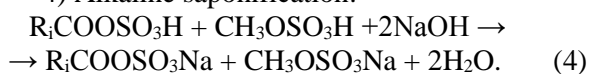
2) Esterification reaction:



3) Sulphonation in the presence of sulfuric acid:



4) Alkaline saponification:



The content of acids in the mixture used to synthesize a new anionic surfactant was determined chromatographically. The amount of palmitic and oleic acids in the mixture was 64.3% and 29.1%, respectively. Oleic and stearic acids are unstable at elevated temperatures and may transform into unsaturated linoleic acid. Additionally, partial degradation to palmitic acid may also occur. The results of the chromatographic analysis are presented in Table 1.

**Table 1**  
**Chromatographic results for the mixture of fatty acids**  
**Таблица 1. Результаты хроматографии смеси жирных кислот**

Acid	Content, %
C <sub>14</sub> – myristinic acid (C <sub>14</sub> H <sub>28</sub> O <sub>2</sub> )	0.5
C <sub>15</sub> – pentadecanoic acid (C <sub>15</sub> H <sub>30</sub> O <sub>2</sub> )	0.1
C <sub>16</sub> – palmitic acid (C <sub>16</sub> H <sub>32</sub> O <sub>2</sub> )	64.3
C <sub>18</sub> – stearic acid (C <sub>18</sub> H <sub>36</sub> O <sub>2</sub> )	0.7
C <sub>18:1</sub> – oleic acid (C <sub>18</sub> H <sub>34</sub> O <sub>2</sub> )	29.1
C <sub>18:2</sub> – linolic acid (C <sub>18</sub> H <sub>32</sub> O <sub>2</sub> )	4.8
C <sub>20</sub> – arachidic acid (C <sub>20</sub> H <sub>40</sub> O <sub>2</sub> )	0.1
C <sub>20:1</sub> – gondoinic acid (C <sub>20</sub> H <sub>38</sub> O <sub>2</sub> )	0.4

The degree of fat decomposition (acid number) for the synthesized anionic surfactant was equal to 8.11 mg KOH/g. The saponification number was 348.8 mg KOH/g.

IR spectroscopy was used to analyze the anionic surfactant structure. The compound structure has the following general formula:



Next, the properties of the synthesized surfactant were studied, and sodium alkyl sulfonate was used as a reference surfactant.

Surfactants are classified according to fractional composition of dispersed phase particles [33]. The dispersion degree of surfactant in solutions affects their properties and determines the appearance of an aqueous emulsion. Moreover, the shape and size of surfactant particles have a significant effect on diffusion process.

A 90Plus/BI-MAS automatic particle analyzer was used to measure the average size of surfactant particles. Fig. 1 illustrates that the surfactant can be considered as a highly dispersed system, with particle diameters ranging from 10 nm to 1 μm. At a surfactant

concentration of 2.0 g/dm<sup>3</sup>, the average particle size ranges from 192.8 nm to 193.0 nm. In comparison, the particle size of sodium alkyl sulfonate ranges from 97.8 nm to 148.3 nm at a surfactant concentration between 2.0-4.0 g/dm<sup>3</sup>.

Fig. 1 also demonstrates that once the critical micelle concentration (CMC) is reached, the particle radii start to rise. The particle size at CMC (4.0 g/dm<sup>3</sup>) is 242.5-242.8 nm. Sodium alkyl sulfonate (as a reference compound) exhibits an average particle size of 168.0-168.3 nm at CMC (4.0 g/dm<sup>3</sup>).

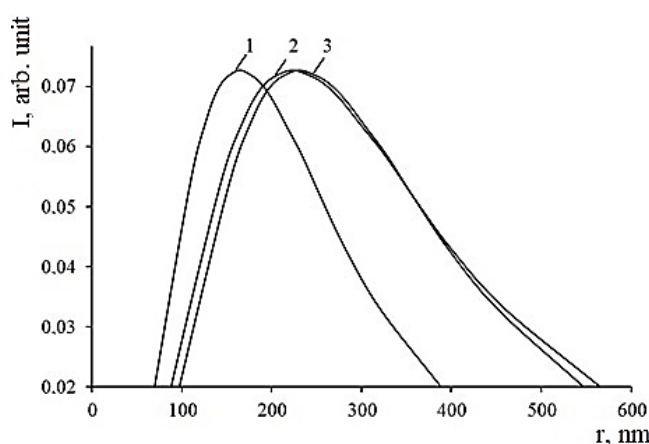


Fig. 1. Particle sizes of the synthesized surfactant, where 1 – surfactant concentration: 2.0 g/dm<sup>3</sup> (before CMC), 2 – surfactant concentration: 4.0 g/dm<sup>3</sup> (at CMC), 3 – surfactant concentration: 5.0 g/dm<sup>3</sup> (after CMC)

Рис. 1. Размер частиц синтезированного ПАВ: 1 – концентрация ПАВ 2,0 г/дм<sup>3</sup> (до ККМ); 2 – концентрация ПАВ 4,0 г/дм<sup>3</sup> (в точке ККМ); 3 – концентрация ПАВ 5,0 г/дм<sup>3</sup> (после ККМ)

The stability of dispersed systems and their aggregation ability depend on the zeta potential ( $\zeta$ -potential). The  $\zeta$ -potential is a criterion for electrostatic interactions (force of attraction or repulsion) between particles [33]. In this study, the  $\zeta$ -potential value was measured using a 90Plus/BI-MAS analyzer, which operates based on frequency and phase analysis of scattered light. This means that the  $\zeta$ -potential was measured using the electrophoretic light scattering method.

It is based on the dynamic light scattering method in the laser Doppler anemometer configuration, which is used to determine the velocities of liquid and gas flows.

The  $\zeta$ -potential is measured to control the processes of dispersion, aggregation, or flocculation, with the aim of optimizing the dispersion properties of colloidal solutions, suspensions, and emulsions during their preparation and production. Fig. 2 presents the dependence of the  $\zeta$ -potential on the concentration of surfactants.

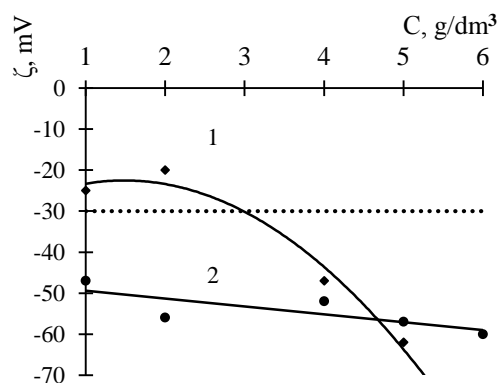


Fig. 2. Measurement results for  $\zeta$ -potential, where 1 – synthesized surfactant; 2 – sodium alkyl sulfonate

Рис. 2. Результаты измерения  $\zeta$ -потенциала: 1 – синтезированное ПАВ, 2 – алкилсульфонат натрия

Fig. 2 shows how the  $\zeta$ -potential decreases as the surfactant concentration increases. We explain it by the fact that an increase in the counterion concentration in the solution results in a decrease in the electrical double layer thickness.

It is known that to ensure system stability, the value of the  $\zeta$ -potential must be outside the range of  $30 \text{ mV} < \zeta < -30 \text{ mV}$ . In other words, the  $\zeta$ -potential should be greater than 30 mV or less than -30 mV.

Both investigated and reference surfactants meet these requirements. However, the synthesized surfactant is resistant to coagulation starting from a concentration of  $2.5\text{--}3.0 \text{ g/dm}^3$ , indicating a metastable state of the system. On the other hand, the reference surfactant is characterized by high system stability over the entire range of concentrations. During our measurements, we did not observe low  $\zeta$ -potential values ( $< \pm 10 \text{ mV}$ ), which indicated that the surfactant particles were not subject to rapid flocculation.

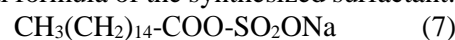
The measurement results indicate that the synthesized surfactant demonstrates high aggregative stability within the concentration range of  $2.5\text{--}4.0 \text{ g/dm}^3$  (CMC).

To characterize the detergent power of the synthesized anionic surfactant, we calculated the hydrophilic-lipophilic balance (HLB) value. HLB represents the ratio between the hydrophilic and hydrophobic portions of a surfactant [34]. Based on the chemical formula of the synthesized surfactant and its IR spectroscopy data, hydrophilic and hydrophobic groups were identified. A method was developed to calculate HLB, in which a group HLB value is assigned to each functional group of the molecule based on the coalescence rate of oil droplets in water and water droplets in oil [34]. For example, the group HLB value for  $-\text{SO}_3\text{Na}$  is 11.0, and for ether (free) it is 2.4. The hydrophobic properties of  $-\text{CH}_3$ - and  $-\text{CH}_2$ - are characterized by a group HLB value of 0.475.

Using these values we can calculate the HLB values for a surfactant:

$$\text{HLB} = \sum(\text{values of hydrophilic groups}) - \sum(\text{values of hydrophobic groups}) + 7 \quad (6)$$

Structural formula of the synthesized surfactant:



The content of palmitic acid in the surfactant is about 65%, while oleic acid accounts for about 30%. The calculation of the HLB took into account the content of these two acids.

Calculation of the HLB value for the synthesized surfactant:

$$(11 + 2.4) - (0.475 \times 16) + 7 = 12.8$$

The HLB value of the surfactant is determined to be 12.8 [34]. This value indicates that the synthesized anionic surfactant can be used as a detergent.

We analyzed the detergent power of the anionic surfactant. The washing ability was evaluated on two different types of fabric (cotton and wool) with pigment-oil and protein contamination. At a concentration of  $3.0 \text{ g/dm}^3$  of the surfactant, the washing ability was found to be 51.5% and 60.0% for cotton fabric with pigment-oil and protein contamination, respectively. When the concentration of the surfactant was increased to  $4.0 \text{ g/dm}^3$ , the washing ability significantly improved, reaching 65.2% for pigment-oil contamination and 74.8% for protein contamination.

A similar pattern was observed when determining the washing ability of the surfactant in relation to woolen fabric. High washing ability was achieved with the use of the surfactant with a concentration of  $4.0 \text{ g/dm}^3$ . The washing index in the case of pigment-oil contamination was 63.1%, while for protein it was 73.4%.

One of the most important properties of surfactants is their ability to form adsorptive films on treated surfaces to prevent the re-adherence of contaminant particles. This is achieved due to the formation of adsorption films on the surface of contaminant particles, giving them high aggregative stability. This property of surfactants is characterized by emulsion stability and the ability to retain fat droplets and emulsified contaminants [35].

In this study, the emulsion stability of surfactants was defined at concentrations of  $0.25\text{--}5.0 \text{ g/dm}^3$  after a certain volume of a solution had separated. The results are shown in Table 2.

We applied the dispersion method, which is used to separate a coarsely dispersed system and create immiscible liquid layers [36]. Our mixture, containing motor, industrial oil and a surfactant, separated into three phases: the top layer was oil, the middle layer was a finely dispersed emulsion, and the bottom layer was

a surfactant solution. The system containing sunflower oil separated into two phases: the top layer was a homogeneous foam, and the bottom layer was a surfactant solution.

Table 2

**The influence of surfactant concentration on emulsion stability**

Таблица 2. Влияние концентрации ПАВ на стабильность эмульсии

C, g/dm <sup>3</sup>	Start of separation, min		
	Animal fat	Industrial oil	Motor oil
5.0	3	72	67
3.0	0.5	32	21
2.0	Unstable emulsion	27	Unstable emulsion
1.0	Unstable emulsion	5	Unstable emulsion
0.5	Unstable emulsion	4	Unstable emulsion
0.25	Unstable emulsion	2	Unstable emulsion

The experiments demonstrated that surfactant solutions began forming stable emulsions with animal fat at a concentration of 3.0 g/dm<sup>3</sup>. Furthermore, the emulsion stability increased with higher concentrations of the solutions. For motor oil, the emulsion also stabilized at a concentration of 3.0 g/dm<sup>3</sup>. The highest emulsion stability was observed with industrial oil, as even at a lower concentration of 2.0 g/dm<sup>3</sup>, the emulsion did not separate within 32 min.

In addition to emulsifying and washing ability of surfactants, their ability to reduce surface tension is of great importance. The high adsorption capacity of anionic surfactants makes them effective wetting agents that can easily penetrate into the dermis structure. We determined the surface tension using the Wilhelmy plate method.

The plate, made of platinum, was used for the measurements conducted at the interface between two phases: a surfactant solution and air. These measurements were carried out in a dynamic mode, allowing for no time to reach adsorption equilibrium after the surface had formed. Based on the results obtained, we plotted surface tension isotherm (Fig. 3).

The resulting product is a mixture of surfactants, because the surface tension of its solutions decreases with increasing concentration [34]. The solutions of the investigated surfactant exhibit a relatively low surface tension ( $\sigma = 36.5$  mN/m at 1.0 g/dm<sup>3</sup>).

A sharp difference in the physicochemical properties of the system in the CMC region should be taken into account as an important indicator for developing technological processes. Starting from a solution concentration of 4.0 g/dm<sup>3</sup>, the surface tension does not change within the experimental error. The CMC of the

tested surfactant is 4.0 g/dm<sup>3</sup>, while the CMC of sodium alkyl sulfonate is 2.5 g/dm<sup>3</sup>.

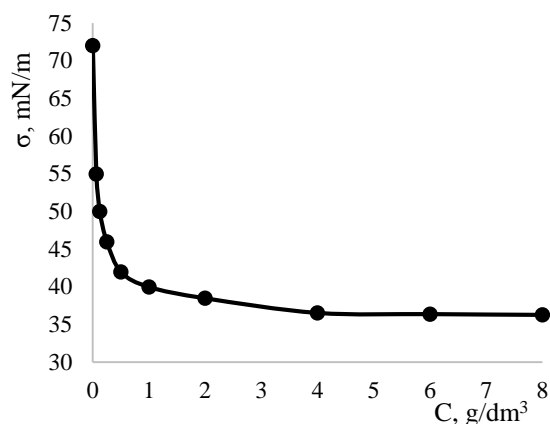


Fig. 3. Surface tension isotherm

Рис. 3. Изотерма поверхностного натяжения

After determining the surface tension, the wetting ability of surfactants was evaluated using the same tensiometer and a platinum plate. The ability to wet the surface is an essential criterion for surfactants used in the leather and fur industry, where raw materials are often heavily oiled and contaminated. Effective detergents are required for high-quality processing of these materials. From a physicochemical perspective, the mechanism of contaminant removal can be divided into the following stages: adsorption (accompanied by the wetting processes), emulsification, and solubilization.

Surfactants should demonstrate good wetting, aggregative, and sedimentation ability.

To analyze the wetting ability of surfactants, two surfaces were studied: hydrophilic (gelatin) and hydrophobic (paraffin). The hydrophobic surface was obtained by applying a thin layer of molten wax onto a glass surface.

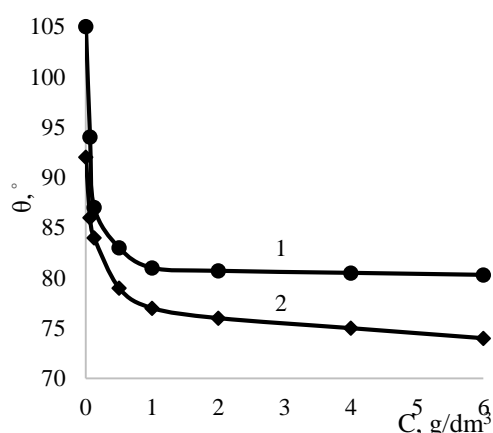


Fig. 4. Dependence of the contact angle of wetting on the surfactant concentration for hydrophobic (1) and hydrophilic (2) surfaces

Рис. 4. Зависимость краевого угла смачивания от концентрации ПАВ для гидрофобной (1) и гидрофильной (2) поверхностей

Fig. 4 shows that the investigated surfactant is a highly effective wetting agent. At a concentration of 2.0 g/dm<sup>3</sup>, the contact angle on gelatin and paraffin surfaces is 76.3° and 80.4°, respectively. As expected, the reference surfactant does not exhibit high wetting ability. Its contact angle on hydrophilic and hydrophobic surfaces is 85° and 90°, respectively.

The foaming properties of surfactants are of great importance for various industries. Excessive foaming is known to be undesirable for the processing of leather and fur in liquid solutions. Therefore, it is important to evaluate and control the foaming ability of surfactants. The foam-producing ability of surfactants is characterized by the foam expansion ratio. We evaluated the foam formation organoleptically and then defined the foam stability and foam expansion ratio (Table 3).

The obtained results confirm that the increase in the foam expansion ratio is proportional to the concentration of the foaming agent in the solution. It was observed that the foam produced by all tested anionic surfactants exhibited dispersion heterogeneity. Unstable coarsely dispersed foam was observed in the upper layer of a solution. Despite this, the foam stability was

quite high. It was stable for 10-30 min for all tested surfactants.

Compared to sodium alkyl sulfonate, the synthesized anionic surfactant based on three fatty acids demonstrates a weak dependence of the foam expansion ratio on the solution concentration. This indicates that its foaming process is stable. For sodium alkyl sulfonate, the foam expansion ratio increases from 0.6 to 2.5 with an increase in the surfactant concentration from 0.5 g/dm<sup>3</sup> to 8.0 g/dm<sup>3</sup>.

The pH-value of the synthesized and reference surfactant solutions was equal to 7–8. Table 4 presents the properties that were studied for both the synthesized and reference surfactants.

Table 3

## Calculated foam expansion ratio for surfactants

Таблица 3. Рассчитанная кратность пены синтезированного ПАВ

Concentration, g/dm <sup>3</sup>	Foam expansion ratio
0.5	1.1
1.0	1.3
2.0	1.4
4.0	1.5
8.0	1.6

Table 4

## Surface active properties

Таблица 4. Поверхностно-активные свойства

Property	Sodium alkyl sulfonate	Synthesized surfactant
1	2	3
Appearance	homogeneous translucent liquid	viscous pasty substance of milky-cream color
Mass fraction of the main substance, %	30	-
CMC, g/dm <sup>3</sup>	5	4
HLB	14	12.8
Foam expansion ratio at 1.0 g/dm <sup>3</sup>	2.3	1.3
Surface tension of an aqueous solution at 1.0 g/dm <sup>3</sup> , σ, mN/m	44	36.5
Wetting angle of an aqueous solution at 1.0 g/dm <sup>3</sup> on hydrophilic surface	87.5	76.9
Wetting angle of an aqueous solution at 1.0 g/dm <sup>3</sup> on hydrophobic surface	89.3	81.8
pH of aqueous solution	7-8	7-8

## RESULTS AND DISCUSSIONS

The obtained anionic surfactant is a viscous pasty substance of a milky-cream color at room temperature.

The presence of specific functional groups (hydroxyl, carboxyl, ester, and sulfo groups) in the synthesized compounds was confirmed.

Particle size analysis showed that the synthesized surfactant occupies an intermediate position between nano- and microparticles, and its fractional composition closely resembles the type of monodisperse systems.

On the other hand, sodium alkyl sulfonate is a polydisperse system with a wide range of mean values. The dispersion degree of surfactants affects properties such as detergency and surface tension.

The experiments conducted to determine the ζ-potential of the synthesized surfactant demonstrated high aggregative stability within the concentration range of 2.5 g/dm<sup>3</sup> to the CMC at 4.0 g/dm<sup>3</sup>.

The hydrophilic-lipophilic balance of the surfactant was measured to be 13.275, classifying it as a detergent.

The experiments on the study of detergent properties revealed that the synthesized surfactant can effectively remove the mentioned pollutants from cotton and woolen fabric.

The emulsifying properties of the synthesized surfactant practically did not differ from the emulsifying ability of alkyl sulfonate. This similarity arises from the fact that both surfactants contain the sulfo group in their composition, which provides good resistance to water hardness and ensures the dissolution of protein fibers. However, neither surfactant is capable of preventing the re-adhesion of contaminants. The problem can be solved by formulating compositions containing nonionic surfactants [17, 18].

Solutions of the investigated surfactant exhibited a relatively low surface tension ( $\sigma = 36.5$  mN/m at 1.0 g/dm<sup>3</sup>). The surfactant CMC was 4.0 g/dm<sup>3</sup>. Further increasing the surfactant concentration in the solution did not result in a decrease in surface tension or an increase in detergency.

We defined that the synthesized surfactant is a highly effective wetting agent at concentrations higher than 2.0 g/dm<sup>3</sup>.

There was a slight dependence of the foam expansion ratio on the solution concentration, indicating that surfactants provide stable foaming.

#### CONCLUSIONS

A new surfactant, based on three acids: palmitic, oleic, and stearic acid, was synthesized. Its structure and properties were studied. The synthesized surfactant exhibited good surface-active properties including detergency, emulsification, wetting, and foaming ability. Moreover, the experimental results confirmed that the synthesized compound can be recommended for use in various industries, particularly, in the leather and fur industry.

*The authors declare the absence a conflict of interest warranting disclosure in this article.*

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