








# Synthesis and Production Technology of Horse Dea Surfactant by Amidation of Horse Fatty Acids with Diethanolamine

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## Abstract

One of the most significant classes of nonionic surfactants containing nitrogen is ethanalamides of horse fatty acids. The conditions for the formation of nanoscale and submicron emulsions and the factors influencing the phase inversion temperature (SIT) and phase inversion composition (SIS) are examined. These products are widely used as components of detergents, foaming agents, and stabilizers in cosmetic preparations, wetting agents and antistatic agents, dispersants, corrosion inhibitors, etc. Finding amides with nonionic foaming qualities in the presence of diethanolamines containing fatty acids extracted from horse fat is the aim of the study. In accordance with the reaction mechanism, amidation was performed using 400 grams of horse fatty acid (1.5 mol), 157.6 grams of diethanolamine (1.5 mol), and 3% of the Al<sub>2</sub>O<sub>3</sub> catalyst (*i.e.*, 12 grams) in relation to the total mass of fatty acid. The study's Horse DEA surfactant's HLB values were contrasted with those of other DEAs. As a local alternative to the popular Coconut DEA product for shampoo products, the Horse DEA developed in this scientific work has good solubility in oily environments with a DEA value of less than 10.

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## Keywords

Amide, Amine, Long Chain Fatty Acids, HLB Number, Foam Stability, Surfactant Model, Coconut DEA

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## 1. Introduction

Diethanolamides of equine fatty acids are one of the most important classes of nitrogen-containing nonionic surfactants. These products are widely used as components of detergents, foam stabilizers, wetting agents and antistatic agents in cosmetic preparations, dispersants, corrosion inhibitors, etc. [1]. Diethanolamides also serve as raw materials to produce other classes of surfactants, such as oxyethylated ethanolamides, their sulfoesters, and borates (salts and esters of boric acid) [2]. Interethanolamides of fatty acids containing 14 - 20 carbon atoms have been expanded [3]. Such products are fat-soluble, have low hydrophilic-lipophilic balance (HLB) values (2 - 3 units) and are therefore effective emulsifiers for water-in-oil emulsions, which are widely used, for example, in oil and gas production [4]. The main methods for preparing diethanolamides involve the aminolysis of fatty acid esters containing triacylglycerols (TAGs) and ethanolamines [5]. Currently, the first method is widely used due to the mild conditions of the aminolysis process, the simplicity of the apparatus design, and the availability of raw materials [6]. Aminolysis of equine fatty acid esters provides high conversion of raw materials and high product yield [7]. However, there is a need to use catalysts, which are usually represented by hydroxides and alkoxides of alkaline earth metals, which reduces the cost of this method [8]. In addition, the reaction contains glycerol, an aminolysis product of triacylglycerols (TAG), and in some cases requires additional purification [9]. However, when it comes to obtaining diethanolamides of vegetable fatty acids, which are widely used in detergents and cosmetics, this classic method has no competitors.

Direct amidation of equine horse fatty acids requires more severe synthesis conditions and special methods to eliminate the formation of water as a byproduct. However, this method is also of great practical importance, especially to produce technical-grade horse fatty acid diethanolamides. As a rule, this method does not require any catalyst and allows the use of inexpensive and cheap raw materials, such as high-quality oils and natural horse fatty acids. Another method, the acylation of diethanolamines with halogen anhydrides (replacement of a hydrogen atom), is practically not used, which is due to the high cost of the starting halogen anhydrides [10].

DEA is accompanied by numerous parallel and sequential reactions between directly equine horse fatty acids. However, the mechanism of amidation of horse fatty acids by DEA (diethanolamide) has not been fully studied. The data available in the literature do not fully describe the processes that occur and are often contradictory. Although the reactions of the first stage have been studied in detail, there are only

assumptions about the subsequent developments based on the nature of the products obtained and general ideas about the reactivity of diethanolamine derivatives. Let us consider in detail the main chemical reactions occurring in the HFA-DEA system, which is currently considered the most reliable mechanism [11].

Thus, the direct amidation of equine fatty acids with diethanolamines is one of the most important industrial methods to produce diethanolamides. Typically, the process is carried out in a cyclic mode by heating a mixture of equine fatty acids with diethanolamine at a temperature of 150°C - 180°C and continuously removing water from the reaction mass by evaporation. Water removal is improved by bubbling inert gas or by reducing the pressure using an azeotropic agent. In this case, the choice of synthesis time usually does not have a strict theoretical basis. The completeness of the reaction is often assessed by the residual acid value of the product or the amount of water removed from the reaction system, as well as when the water release stops [12]. This approach is reasonable for the synthesis of monoethanolamides of equine fatty acids, but it should be questioned for diethanolamides. The reason for this is that the reaction of equine fatty acids and monoethanolamine leads to the formation of an equilibrium mixture of amide, ester, and amido ester. All reactions occurring during this process are reversible. The composition of the final products depends largely on the ratio of reactants. Products containing more than 90% monoethanolamides are obtained in equimolar amounts of the starting materials, which indicates a sufficiently high selectivity of the reaction. Compared to monoethanolamines, diethanolamine amidation (DEA) is a more complex process [13]. The main difference is that the reaction of equine horse fatty acids with DEA does not stop at the stage of formation of monoacylated derivatives, namely diethanolamide and amino acid esters. As these products accumulate, secondary reactions begin to occur within the system, and then tertiary reactions begin. In addition, parallel processes exist. A complicating factor is the irreversibility of some of the side reactions. The chemical changes that occur in the horse fatty acid-DEA system are discussed in detail below. Here we only note that, as a rule, the content of diethanolamides in direct amidation products does not exceed 60% - 65%, and their composition depends not only on the ratio of reagents, but also on process conditions, such as temperature and, most importantly, synthesis time [14]. In this regard, a key question arises in the production of diethanolamides: how do process conditions and their duration affect the surface-active properties of the products obtained. Thus, this work aims to study the effect of the synthesis duration of horse fatty acid diethanolamides on the composition of the reaction products, their surfactant properties, and the stability of water-in-oil emulsions obtained on their basis. It should be noted that for each of the created surfactants, it is necessary to study their HLB numbers and emulsifying and demulsifying properties for the hydrophilic-lipophilic balance [15].

The book "Hydrophilic-Lipophilic Equilibrium and Phase Inversion" covers the following topics at a modern level: the structure and stabilization properties of emulsions, the theory and practice of hydrophilic-lipophilic equilibrium (HLB),

the principles of classifying surfactant emulsifiers on the HLB scale, methods for finding optimal emulsifiers of “essential oils” and “HLB” are important for obtaining direct and inverse emulsions [16]. The conditions for the formation of nanoscale and submicron emulsions and the factors affecting the phase inversion temperature (SIT) and phase inversion composition (SIS) are analyzed [17]. The characteristics of energy-saving methods of emulsification by the SIT and SIS methods are presented [18]. The purpose of the research is to obtain amides with nonionic foaming properties in the presence of diethanolamines with fatty acids extracted from horse fat.

## 2. Methods and Materials

According to the experiment, the amidation reaction mechanism was obtained from 400 grams of horse fatty acid (1.5 mol), 157.6 grams of diethanolamine (1.5 mol), and 3% of the  $\text{Al}_2\text{O}_3$  catalyst (*i.e.*, 12 grams) relative to the total mass of fatty acid. Pure horse fat acids for the reaction were prepared according to the methodology of the following article [1]. The process was initially carried out at 100°C in a flat 2-necked flask with a model faithful magnetic stirrer (directly at room temperature) operating (rotation frequency 40 - 50 rpm), rotating at a standard stirring torque, and the temperature was set to 152°C. To know whether the reaction will proceed to completion, we theoretically calculated that 27 grams of water would be produced when 1.5 mol of the reaction is calculated according to the reaction mechanism, and set a water level of 27 grams on the conical part of the decanter installed in the equipment cooler. In total, 9 - 11 hours should be observed. To ensure that room temperature does not affect the water separation in the reaction medium due to temperature compensation, that is, to ensure that the water separates well, condenses, and decants, the cooling section of the equipment was wrapped with a cotton fiber cloth. After 4 and a half hours, water was observed to be released from the process. When the temperature reached 160°C, no foaming occurred in our product at all. The internal temperature of our product rose to 97°C, while the temperature of our faithful device rose to 170°C. The extraction of our product lasted 7 hours, and we stopped the process when the water output line of 27 grams was reached in the decanter. The effective results were separated from the reaction mixture at the specified time intervals and sent for analysis. The total mass of our product was 530 grams.

Methodological analysis of the product was carried out using the following methods: The acid value of the amidation products was determined by titration with 0.1 normal KOH alcoholic solution using phenolphthalein indicator according to the AVAP. The amine value was determined by titration of the alcoholic solution of the test sample with 0.5 normal HCl solution in the presence of bromocresol green according to the AVAP. To determine the type of object, the sample was analyzed on a Spectrum Two IR Fourier spectrometer under the following analysis conditions. The HLB numbers of surfactants and fatty acids derived from oils were calculated using the Griffin Hydrophilic-lipophilicity method. Foaming

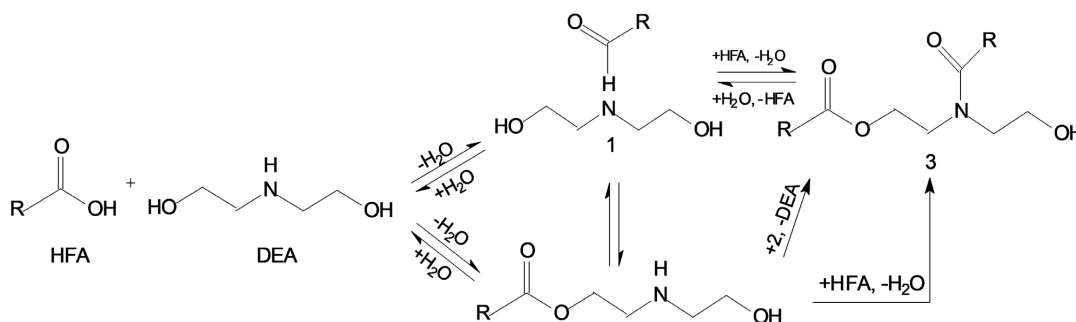
ability and its formation were carried out based on the following State standard 22567.5 - 93, State standard 33575 - 2015 (ISO 696:1975). Foam stability-determined based on the requirements of the State standard T 22567.6 - 93, State standard Ru 50551 - 93, State standard 31941 - 2012 (ISO 6889:1986). Demulsifier properties were determined based on the methodology of State standard 6370 - 83—“oil and petroleum products”.

Method for determining the efficiency of demulsifiers for separating emulsions. Surface tension of surfactants-determined based on the standard requirements of State standard 20216 - 74, State standard 33445 - 2015 (ISO 304 - 1985), State standard 33629 - 2015 (ISO 1409:2006), and methods involving Du-Nui (RG11, RG10) rings. CMC-Critical micelle concentration was performed on a DataPhysics DCAT-15 Tensiometer. Interfacial tension, surface tension, and critical micelle concentration were determined by measuring the droplet volume formed at the interface of a distilled water-surfactant solution in the presence of octane at 19.5°C - 20°C using a DCAT-15 tensiometer. Distilled water with a conductivity not exceeding 5  $\mu\text{S cm}^{-1}$  was used. Validated by comparison with standard spectra from the DCAT-15 Tensiometer library.

### 3. Results and Discussions

The main property of surfactants is their foaming and their ability to dissociate or form ions in the presence of water, which is controlled by the concentration of the solution, depending on the pH of the medium. The main factors of a detergent determine its foam stability and washing ability. In this scientific work, a nonionic shampoo raw material was created that balances the drying and degreasing of the skin during washing the hair or scalp. This surfactant is explained by the process of amidation of fatty acids extracted from horse oil by amines.

The first step of amidation leads to the formation of the monoacylated products diethanolamide 1 and amino ester 2, according to the following reaction mechanism:



1—HFA is a compound of horse fatty acids.

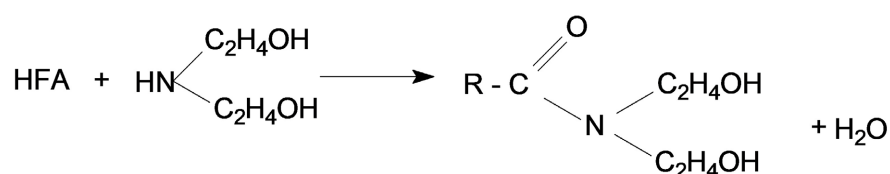
2—DEA-Diethanolamide.

The ratio of these products is mainly determined by the NH and OH reactive groups of DEA. Since the nucleophilic properties of the amino group are more pronounced than those of the hydroxyl group, the main product of the first step

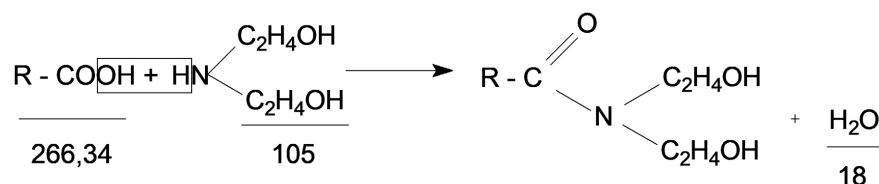
is diethanolamide 1. These reactions are reversible to form amide 1 and ester 2, and their equilibrium is determined by the amount of water introduced into the reaction system. In addition, there is an equilibrium conversion of amide 1 to ester 2 and vice versa, which can occur both in the reaction of these compounds with a free DEA molecule and as a result of intramolecular rearrangements. The reaction equilibrium  $1 \leftrightarrow 2$  is temperature dependent [19].

The latter is the case when the concentration of the ester increases, as the equilibrium shifts towards the aminoester 2. Let us consider the changes in the main characteristics of the reaction system during the synthesis. At the beginning of the process, the amine value decreases as quickly as the acid value, which indicates that the NH groups of DEA are consumed to form amide 1. After 2 - 3 hours of synthesis, when its smooth course begins to increase in temperature, the amine value reaches a minimum value corresponding to the acceleration of the substances in the reaction medium by approximately 35 mg of the catalyst  $\text{Al}_2\text{O}_3$ . The increase in the amine value at the present fatty acid values is undoubtedly due to the accumulation of products containing free amino acids in the reaction system. According to the previously proposed initial reaction system, it can be observed that such products are primarily formed as amino ester 2 and DEA. As mentioned above, a unique feature of the amidation of horse fatty acids with diethanolamine is that the process does not stop at the stage of formation of primary products. In parallel with the accumulation of the products of the first stage, secondary reactions occur in the reaction system.

For example, the reactions can occur individually as follows:



To carry out this reaction based on the formula of a single fatty acid, we take the arithmetic average molecular weight of the fatty acid molecules as 266.34. We take the molecular weight of diethanolamine as 105 and express the reaction mechanism as follows:



It is known from the reaction that when 1 mole of substances react, 18 grams of water is formed. If we consider that 1.5 moles of each substance participate, we observe that 400 grams of HFA (horse fat acids), 157.6 grams of diethanolamine,

and 27 grams of water are formed on the other side of the double-headed flask after cooling. These reactions are very diverse, but according to H. Nadeu. According to H. Kroll and several other researchers, the first reaction above may involve the disproportionation of two molecules of amino ester 2 to amino acids 3 and DEA. The amido ester 3, because of aminolysis of diethanolamine, returns two molecules of diethanolamine to diethanolamide 1. The rate of disproportionation of aminoester 2 is assumed to be higher than the rate of aminolysis of the resulting amidoester 3.

This corresponds to the accumulation of product 3 in the reaction mass. This assumption seems highly contradictory, since both reactions are based on the known amination reaction of amino esters. In addition, amide ester 3 can be formed by subsequent acylation of diethanolamide 1 or amino ester 2 by a molecule of equine fatty acids. In practice, the amidation process of equine fatty acids is carried out using at least a twofold excess of DEA to increase the rate of aminolysis of amide ester 3 and shift the equilibrium towards diethanolamide 1. However, even in this case, the products obtained contain 40% - 60% diethanolamides. It was observed that when we conducted a series of model experiments, the synthesis of diethanolamides of high-fatty acid fatty acids was more successful when we removed water from the continuous process at atmospheric pressure.

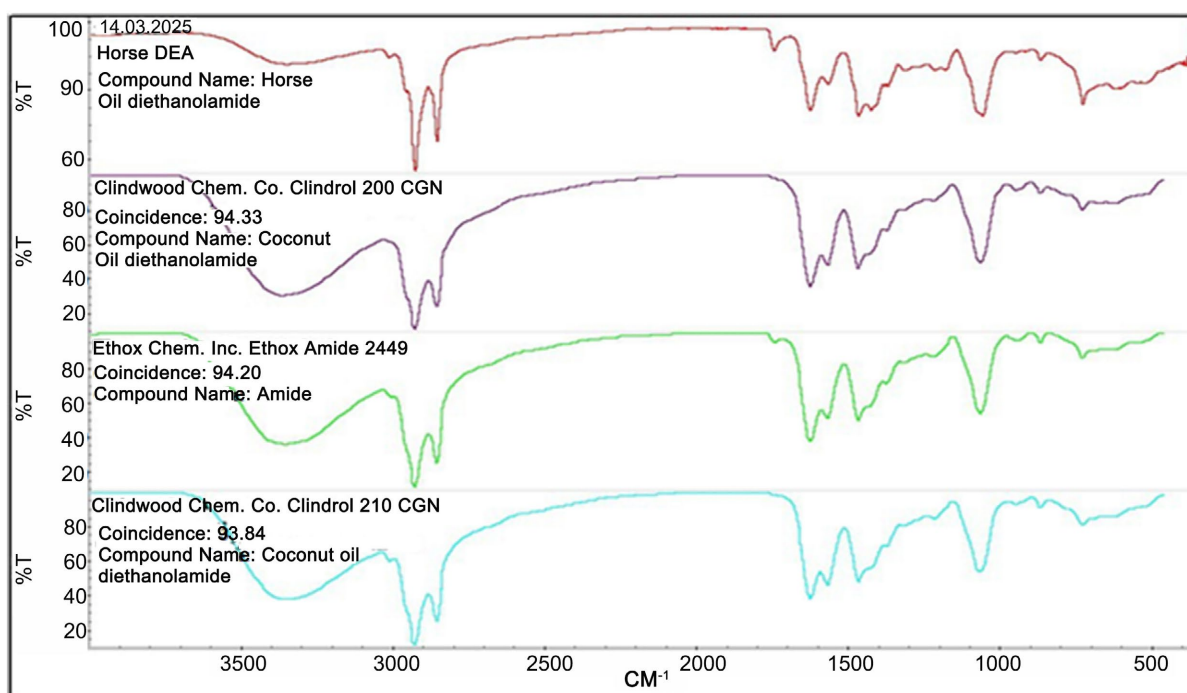
When using equimolar ratios of high-fat fatty acids and DEA, the synthesis temperature was  $170^{\circ}\text{C} \pm 3^{\circ}\text{C}$ , which is consistent with the most used methods for the preparation of amides, *i.e.*, classical methods. The process parameters were chosen to ensure the completeness of the first-stage reactions and the partial occurrence of the subsequent stages, making the reaction irreversible.

Although the mechanism proposed by H. Kroll and H. Nadeu fits most of the observed facts well, but it does not explain the fact that water is formed in the reaction in amounts that significantly exceed those predicted theoretically [20]. Thus, this mechanism needs to be revised or clarified. Analysis of the results obtained in this work allows us to propose a new mechanism for the amidation of carboxylic acids by DEA. The proposed mechanism is based on the following statements: 1) Although amido esters 3 may be formed, this is not one of the main steps in the process under consideration. 2) The reasons for the formation of excess water are cyclic reactions of DEA or DEA-HFA condensation products.

In general, the fact that amides have become a complete reaction product can be explained by the concept of their acid number as follows. When the acid value of the amidation products was determined by titration with 0.1 normal KOH alcohol solution using phenolphthalein indicator according to the AVAP, the result was 2.2 mg KOH. We will conduct a deeper analysis of the obtained products and conduct scientific research as follows.

When the product formed according to the proposed amidation mechanism was analyzed by IR spectroscopy, the composition of the products of the amidation of HFA (high fat acids) with diethanolamine was revealed to be a rather complex mixture of substances. IR—spectral analysis. To determine the type of

object, the sample was analyzed on a Spectrum Two IR Fourier spectrometer under the following analysis conditions: recording range  $4000 - 400 \text{ cm}^{-1}$ , number of scans—100, Horse DEA in **Figure 1**. In this case, a spectrum with characteristic transition bands of groups according to the characteristics of the object was determined—horse oil diethanolamide, which was also determined by comparison with standard spectra from the instrument library. The results are presented in the IR spectrum in **Figure 1**.



**Figure 1.** IR spectrum analysis of the horse DEA synthesis product.

According to this analysis in **Figure 1**, when we compare the parameters in the device's library memory with the currently leading surfactant coconut oil diethanolamide, the first product from the top, Horse DEA Clindrol 200 CGN, corresponds to 94.33% of Coconut Oil Diethanolamide (Coconut DEA), 94.20% of Elhux Amide 2449, and 94.84% of Coconut Oil Diethanolamide, Clindrol 210 CGN. Therefore, we can offer synthesized Horse DEA as a local raw material product, considering that foreign representatives have their own parameters. To clarify the physicochemical properties of Horse DEA, like other surfactants, we present the resulting data using the above methods.

To calculate the hydrophilic and lipophilic balance first, we weighed 47.5 ml of water and 47.5 ml of sunflower oil in 4 100 ml flasks on a DLAB XYSCALE analytical balance, poured 5 ml of the following surfactants: coconut DEA, olein DEA, sunflower DEA and horse DEA, added 0.3 gramm of dry NaOH in granules as a catalyst to each flask, slowly shaken 20 - 30 times, and placed it in a TC-1/80 CIY thermostat at  $25^\circ\text{C}$  for 1 day based on the technical conditions of TU 9452-002-00141798-97. When the reaction was complete, the citric active substances re-

maining in the products were removed from each flask, and their volume in a volumetric flask was determined to determine the hydrophilicity and lipophilicity values. **Table 1** below lists the volumes of substances involved in determining the hydrophilicity and lipophilicity values of surfactants, determined by taking 5 ml of each surfactant, and their calculated HLB values.

In the following formula, we calculate the HLB-hydrophilicity balances of surfactants based on their volumetric values.

$$HLB = \frac{\alpha_1 \times 20}{x_1};$$

$\alpha_1$ —amount of surfactant in the emulsion;

$x_1$ —total mixture volume.

**Table 1.** Volume-based values for hydrophilicity and lipophilicity of surfactants.

№ Flask	Surfactant type	The amount of surfactant in the emulsion (ml)	HLB numbers
1	Coconut DEA	52	10.4
2	Olein DEA	95	19.0
3	Sunflower DEA	98	19.6
4	Horse DEA	51	10.2

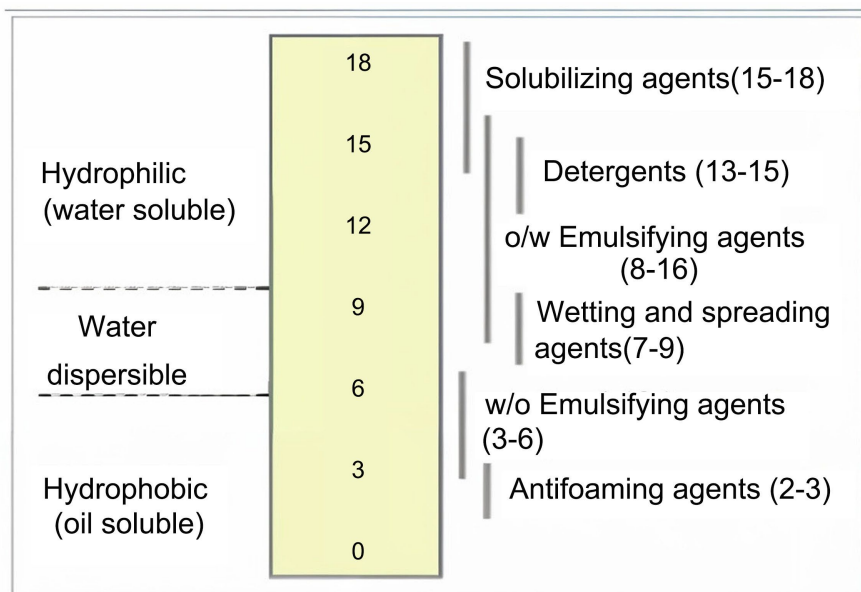
So,  $\alpha_1$  means the hydrophilicity of the surfactant molecule, while  $x_1$  is based on the number of molecules in the general environment, which is based on a scale of 0 to 20. That is, if the HLB number is 0, the Surfactant is considered completely lipophilic-hydrophilic, but if the HLB number is 20, it indicates the hydrophilic-lipophilicity of the molecule. If the HLB number is <10, it means that the surfactant is soluble in oil and insoluble in water. If the HLB number is >10, it means that the surfactant is soluble in water and insoluble in oil.

Based on the HLB value of the surfactant, we can choose the medium in which it will be active, its type, and the concentration of use. In conclusion, the Horse DEA created in this scientific work has good solubility in oily environments with a DEA value of less than 10, demonstrating its properties as a local substitute for the widely used Coconut DEA product for shampoo products. The determination of the HLB number above is presented below, based on the HLB values given in **Table 2**, which show the detergency properties of 0.1 gramm of dry NaOH in granules as a catalyst.

**Table 2.** Detergency of surfactants based on HLB value with hydrophilicity and lipophilicity values.

№ Flask	Surfactant type	The volume of surfactant in the emulsion (ml)	HLB numbers
1	Coconut DEA	68	13.6
2	Olein DEA	78	15.6
3	Sunflower DEA	78	15.6
4	Horse DEA	67	13.4

Therefore, we determine the properties of Horse DEA Surfactant based on the data presented on the scale of the diagram in **Figure 1**, which was developed based on the Griffin HLB numerical indicators of the detergency of the surfactant.



**Figure 2.** Determination of surfactant type based on the Griffin HLB number chart scale.

Since the volume of Horse DEA Surfactant in the emulsion is 67 ml and the Detergent Property is 13.4, which is in line with the requirement in the range of 13 - 15, as shown in **Figure 2**, we can see that Horse DEA Surfactant also exhibits detergency properties.

#### 4. Deemulgir Property

Based on the following scientific analysis of Horse DEA Surfactant, we describe its demulsibility as follows. Demulsibility is a property based on the separation of substances from an emulsion using certain effects: demulsifiers, heating, agitation, electric or electrostatic fields, pulses, centrifugation, and filtration processes. So, we need to check how much water is released from the Horse DEA surfactant. To calculate the demulsibility, we mathematically determine the amount of 1% Horse DEA surfactant and the amount of oil required.

Consequently, if 1 ton of oil requires 100 gramm of surfactant according to the State Standard 6370 - 83 methodology, then 0.2 gramm of 0.01 g of Horse DEA surfactant is taken from a 0.1% aqueous solution, 10 ml of 20% water-containing oil is added to a 10 ml flask, and 0.1 gram of Horse DEA surfactant is added, and the mixture is mixed well 10 - 15 times. When the ambient phase was left for 2 days, 2 ml of water should have separated from the sample. Still, when we observed it after 3 days, 0.5 ml of water began to separate from the sample during the day, so based on this property, we can be sure that the demulsification property exists.

## 5. Surfactant Foaming and Stability Properties

We have analyzed the foam stability and the method of its formation by analyzing the foam formation and stability of surfactants at different concentrations.

First, we will consider coconut surfactant as an object: Take 50 ml of coconut surfactant and prepare a 0.3% surfactant.

We calculate according to the following formula.

$$m_{(vva)} = \frac{50 \times 0.3}{100} = 0.15 \text{ gramm}$$

For example, 0.15 g of CocosDEA was weighed on a DLAB XYSCALE analytical balance, distilled water was added to it until it reached 50 gramm, and the mass was brought to 50 gramm, and This product was dissolved in a DLAB MS7-H550-Pro mixer for 20 - 25 minutes to dissolve it in water. A 0.2% solution was prepared from the prepared 0.3% solution by performing the above mathematical calculations. 0.1 gramm of this 0.3% solution was taken, 50 ml of distilled water was added to it and mixed well.

In the experiment, to prepare a 0.1% solution, 0.05 gramm of 0.3% solution was taken, 50 ml of distilled water was added to it and prepared, 20 ml of each of the above 0.3%, 0.2% and 0.1% solutions were taken and put into a 100 ml flask, the mouth was tightly closed, shaken 30 times and after 10 minutes, the foam stability was determined according to Coconut DEA and the results of the remaining surfactants were Horse DEA as the 2nd Object, Oleic DEA as the 3rd Object, and Sunflower DEA as the 4th Object are presented in **Table 3**.

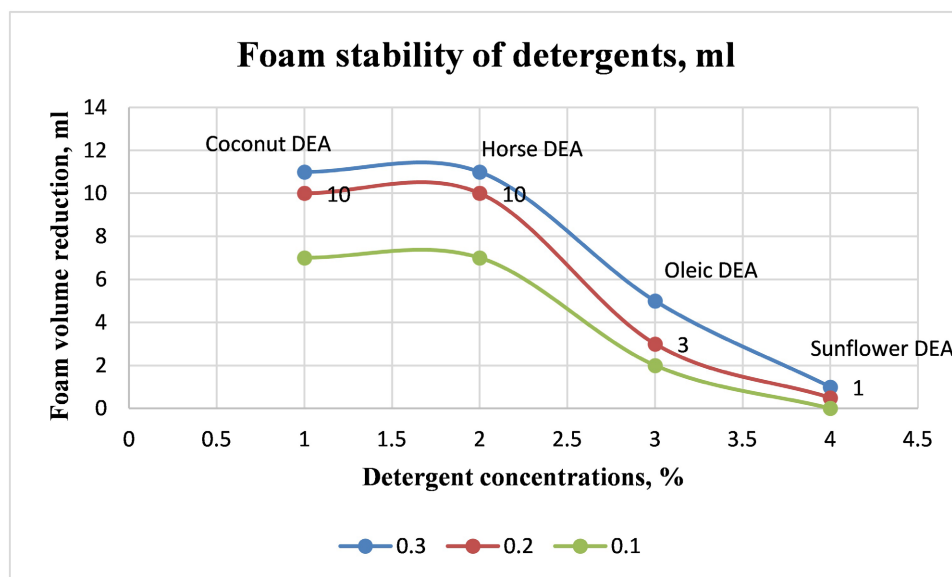
**Table 3.** Values of foam formation and time stability of surfactants prepared from Coconut DEA, Horse DEA, Oleic DEA, Sunflower DEA

Detergent naming	cons, %	Initial min. volume, ml	10 min. later volume, ml	cons, %	Initial min. volume, ml	10 min. later volume, ml	cons, %	Initial min. volume, ml	10 min. later volume, ml
Coconut DEA		49	38		37	27		19	12
Horse DEA	0.3	46	35	0.2	34	24	0.1	16	9
Oleic DEA		16	11		11	8		4	2
Sunflower DEA		5	4.5		3.5	2.5		2	2
Uz State Standard 25644-96			0.3 not less			0.3 not less			0.3 not less

**Figure 3** below shows the formation and volume reduction of foams prepared from Coconut DEA, Horse DEA, Oleic DEA, and Sunflower DEA surfactants, with a decrease in foam stability inversely proportional to the concentrations of surfactant solutions of 0.3%; 0.2%; 0.1%.

From the above diagram, we can obtain the following results for surfactants: Coconut DEA and Horse DEA surfactants have the best foaming and stability, while Oleic DEA and Sunflower DEA surfactants have the lowest foaming and stability. We can also understand that Horse DEA surfactant is comparable to its

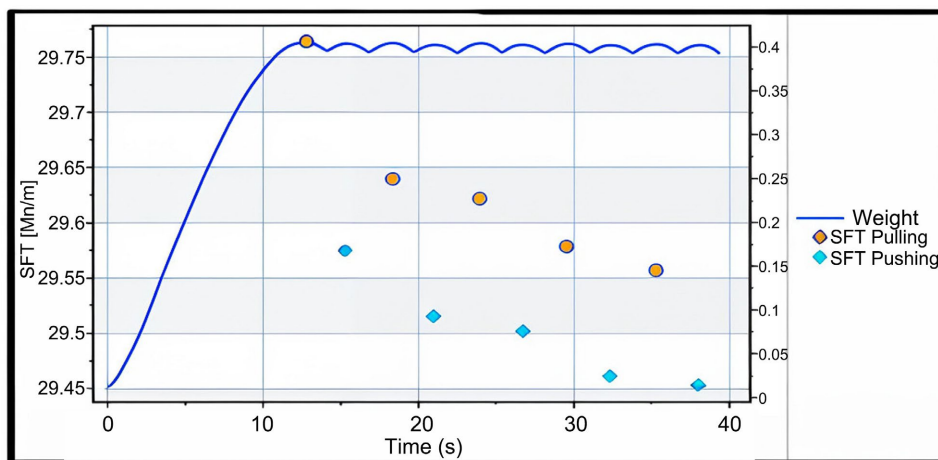
foreign counterpart, Coconut DEA surfactant. In general, we can also clearly see that when the concentration of detergents decreases, their foaming properties decrease.



**Figure 3.** Foam stability diagram of solutions with concentrations of 0.3%; 0.2%; 0.1% of surfactants: Coconut DEA, Horse DEA, Oleic DEA, Sunflower DEA.

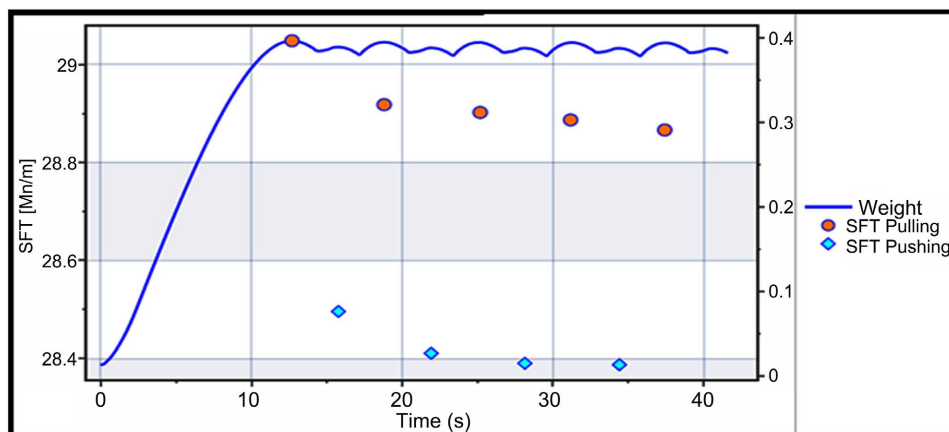
## 6. Surface Tension of Surfactants

Therefore, the surface tension of SFMs is measured in the DCAT-15 tensiometer based on their density, critical micelle concentration and indicators. The DCAT-15 model is a standard representative of the DCAT series of equipment, which embodies a wide range of information on the initial modules of substances. For example, in **Figure 4** below, we can see that the surface tension of the surfactant Coconut DEA reaches the values given in the diagram.



**Figure 4.** Time dependence diagram of the concentrations of 0.1%; 0.2%; 0.3%; solutions of coconut DEA, surfactants.

As the concentration of the surfactant Coconut DEA increased from 0.1 to 0.4 g/eq, its surface tension increased from 29.45 mN/m to 0.05 mN/m every 5 minutes, and it showed a surface tension of 29.65 at 10 and 20 minutes, 29.58 mN/m at 20 and 30 minutes, and 29.55 mN/m at 35 minutes, indicating that this substance is active in its detergent ability from the 10th to the 35th minute at constant temperature. As a test, to determine the initial detergency of Horse DEA surfactant, we can analyze the surface tension values of Horse DEA surfactant in the diagram shown in **Figure 5**.

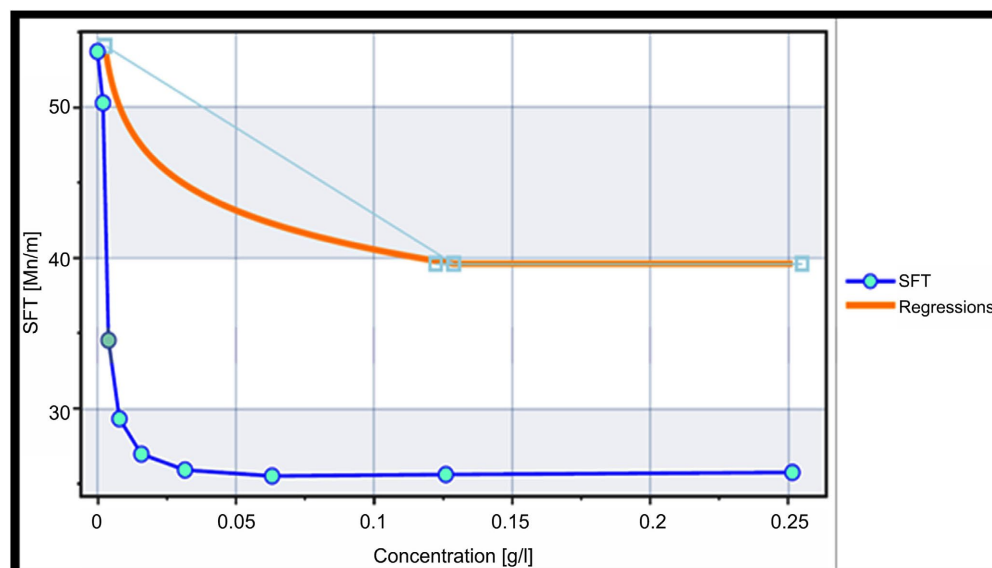


**Figure 5.** Time dependence diagram of the concentrations of surfactant solutions of Horse DEA, 0.1%; 0.2%; 0.3%.

As the concentration of the surfactant Horse DEA increases from 0.1 to 0.4 g/eq, its surface tension significantly differs from that of Coconut DEA, reaching a high surface tension of 28.4 mN/m within the first 10 minutes, decreasing by 0.2 mN/m every 5 minutes to 40 minutes, and showing a surface tension of 28.9 at 10 and 20 minutes, 28.85 mN/m at 20 and 30 minutes, 28.83 mN/m at 32 minutes, and 28.83 mN/m at 37 minutes. At constant temperature, this substance shows that its detergent ability is active from the 10th minute to 40 minutes, and we can see that it has a high stability and detergent ability over time compared to the Coconut DEA surfactant. If we consider these indicators of both surfactants to be almost equally capable, we will see that our Horse DEA surfactant will be a substitute for its foreign representative, Coconut DEA raw material, in the composition of detergents. In this study, the critical concentration of surfactants for CCM-micelles was also studied, and in the diagram in **Figure 6** below, we observe that the surfactant forms CCM at solution concentrations through its regression curve.

The critical concentration for micelle formation is determined by the point corresponding to the discontinuity or the steepest bend in the curve of the properties of solutions depending on concentration. It is assumed that in surfactant solutions at concentrations below the critical concentration, only molecules are present, and the dependence of any property on the anionic or cationic properties of the medium is clearly determined by the concentration of molecules, as seen from the regression curves. If, according to the diagram, we increase the concentration of

Surfactant from 0 to 0.05 g/l, we observe that the surface tension of Surfactant decreases, and the CCM index increases from 0.025 to 0.25 g/l, reaching a constant stability when compared to the regressive one, and we conclude that this surfactant shows the same washing ability regardless of the temperature.



**Figure 6.** CCM-critical concentration diagram of the surfactant horse DEA.

## 7. Conclusion

We studied the effect of the duration of direct amidation, the relationship between the fatty acid composition of the product and diethanolamine, the surfactant properties of the product, and its ability to stabilize water in the oil through its nonionic nature, using high-fatty acid and diethanolamine as starting materials. It was shown that the amount of diethanolamides in the reaction system reaches a maximum and then decreases due to the formation of secondary reaction products. Based on the data obtained, we proposed a new amidation mechanism involving some secondary transformation of the condensed products with horse fatty acids, which gives diethanolamine and its derivatives. We have presented the data considering that the best surfactant properties and the ability to stabilize water-in-oil emulsions were observed in amidation products with the maximum amount of diethanolamides. Exceeding the specified synthesis time may lead to a deterioration in the noted properties. The optimal synthesis time for direct amidation of horse fatty acids with DEA at 170 °C is 1.5 to 3 hours. The amount of acid formed during the synthesis and the amount of water released per 1 M in the reaction mechanism can be a reliable criterion for the completeness of the process.

## Credit Authorship Contribution Statement

**Rakhmanov Sherzod Yuldashevich:** Writing—original draft, Methodology, Investigation, Formal analysis, Data curation. **Rakhimov Dilshod Pulatovich:** Resources, innovator project, Writing—review & editing. **Xu Jing:** Project admin-

istration, Supervision. **Achilova Sanobar Sabirovna:** Writing—review & editing. **Khakimova Zulfiyakhon Azizovna:** Writing—review & editing. **Kenjaev Nuriddin Turaqobilovich:** Writing—review & editing. **Khamidova Madina Olimjonovna:** Writing—review & editing. **Asah-Asante Richard:** Writing—review & editing. **Khudoyberdiyev Fazlidin Isroilovich:** Writing—review & editing. **Salijanova Shakhnozakhon Dilmurodovna:** Writing—review & editing.

## Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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