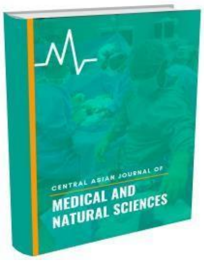

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Article

Synthesis, Characterization, and Surface Activity of Cottonseed Oil–Derived Diethanolamide Surfactants

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Abstract: Cottonseed oil was employed as a renewable raw material for the synthesis of nonionic surfactants via amidation of fatty acids with diethanolamine. The reaction was conducted under solvent-free conditions using alumina as a catalyst, yielding N,N-bis(hydroxyethyl)alkanamides with high efficiency (93–97%). The synthesized surfactants were characterized by gas chromatography, infrared spectroscopy, and nuclear magnetic resonance, confirming the presence of amide linkages and hydrocarbon chains. Physicochemical properties were evaluated through surface tension, cloud point, and critical micelle concentration (CMC) measurements. The surfactants demonstrated effective surface activity, reducing surface tension to 43.2 mN/m and forming micelles at relatively low concentrations (CMC_1 = 15.6 mg/L, CMC_2 = 31.2 mg/L). A cloud point of 57–59 °C indicated favorable solubility and thermal stability. These findings highlight the potential of cottonseed oil–derived diethanolamides as sustainable surfactants for applications in oil recovery, cosmetics, and related industries.

Keywords: Cottonseed Oil, Diethanolamide, Surfactant Synthesis, Critical Micelle Concentration (CMC), Cloud Point, Surface Tension, Renewable Feedstock, Sustainable Chemistry

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1. Introduction

Surfactants are amphiphilic compounds that reduce surface and interfacial tension, enabling their widespread use in detergents, cosmetics, pharmaceuticals, and enhanced oil recovery [1,2]. The growing demand for eco-friendly and biodegradable surfactants has stimulated interest in renewable feedstocks such as vegetable oils, which provide fatty acids suitable for surfactant synthesis [3].

Cottonseed oil is an abundant agricultural by-product, widely utilized in food and industrial applications due to its favorable fatty acid composition and availability [4,5]. It contains significant proportions of palmitic, oleic, and linoleic acids, making it a promising raw material for surfactant production [2]. Compared to other oils such as sunflower or coconut, cottonseed oil offers a balanced profile of saturated and unsaturated fatty acids, which influences the hydrophilic–lipophilic balance (HLB) and physicochemical behavior of derived surfactants [6].

Among surfactants, fatty acid diethanolamides are particularly attractive because they combine good emulsifying properties, low toxicity, and biodegradability [7]. These compounds are typically synthesized via amidation of fatty acids with diethanolamine, yielding nonionic surfactants with broad industrial relevance. Their performance is often evaluated through critical micelle concentration (CMC), cloud point (CP), and surface tension reduction, which are key parameters for assessing efficiency in aqueous systems [8].

Recent studies emphasize the importance of structural characterization (GC, IR, NMR) and molecular modeling to correlate fatty acid composition with surfactant properties [9,10]. Such analyses provide insights into bond lengths, charge distribution, and functional group presence, which govern reactivity and stability. Furthermore, understanding the relationship between fatty acid chain length, degree of saturation, and surfactant performance is essential for tailoring materials to specific industrial applications.

The present work focuses on the synthesis of diethanolamide surfactants from cottonseed oil fatty acids, followed by detailed physicochemical characterization. Special attention is given to their surface tension behavior, CMC determination, and cloud point analysis, which collectively establish their potential as effective, sustainable surfactants for use in oil recovery, cosmetics, and related industries.

2. Materials and Methods

2.1 Materials

A cottonseed oil was employed as raw materials for surfactant production. Diethanolamine (TU 2423-178-00203335-2007) served as the main reagent. Additional chemicals used included sodium hydroxide (NaOH, ≥99%), sulfuric acid (H_2SO_4, 95.5%, Sigma Aldrich), and potassium chloride (KCl, 99.9% purity, DIN ISO 9001).

2.2. Methods

2.2.1. Synthesis of surfactant

Fatty acids were obtained from cottonseed oil through alkaline hydrolysis. For this purpose, 95.1 g of NaOH was dissolved in 1000 ml of water and heated to 70 °C, after which 740 g of animal fat was added. The mixture was maintained under continuous stirring for 6 hours. Subsequently, a 20% sulfuric acid solution was prepared, and 560 g of this solution was introduced to convert the resulting soap into free fatty acids.

N,N-bis(hydroxyethyl)alkanamides were synthesized by reacting fatty acids with diethanolamine at a 1:1 molar ratio. The reaction was conducted without solvent, using Al2O3 as a catalyst (1% relative to fatty acid mass), under a nitrogen atmosphere. The temperature was gradually increased from 70 °C to 140 °C while stirring at 200 rpm. After 3 hours, the reaction mixture was purified either by distillation or acetone washing, with progress monitored through acid number measurements. The final yield ranged from 93% to 97%.

The resulting surfactants were named according to the origin of its fatty acids: cottonseed oil surfactant - CSOS.

2.2.2. Characterization of samples

Gas chromatographic analysis was carried out using a capillary column with hydrogen as the carrier gas. For sample preparation, 0.1 g of the product was dissolved in 1 ml of toluene, followed by the addition of 1 ml of a methylating reagent (BF_3 in methanol). The mixture was treated in an ultrasonic bath at 60 °C for 10 minutes, cooled, and then mixed with 1 ml of bidistilled water. After phase separation for 10 minutes, 1 µl of the upper layer was injected for analysis. Infrared spectra were also recorded using liquid films placed between KBr plates over the 3800–700 cm^{-1} range.

2.2.3. Physicochemical Property Measurements

To determine the surface tension of surfactants using the Du Noüy method [11], an apparatus consisting of a ring, a dynamometer, and a beaker is employed. The procedure involves measuring the force required to detach the ring from the liquid surface for reference water and surfactant solutions of different concentrations. The resulting data are used to calculate the surface tension.

To determine the cloud point of the surfactants, a laboratory sample weighing 0.5 g (with an accuracy of ±0.01 g) is placed in a conical flask. Then, 100 mL of distilled water is added using a graduated cylinder and thoroughly mixed until the surfactant is completely dissolved. From this solution, 15 mL is transferred into a test tube, into which a thermometer is inserted. The test tube is placed in a beaker and heated on a heating device with constant stirring until cloudy ring-shaped zones appear. The cloud point temperature is recorded, ensuring that it does not exceed the specified value by more than 10 °C. Afterward, the test tube is slowly cooled with continued stirring, and the temperature at which the cloudy rings disappear is recorded. Sequential measurements of the cloud point are carried out by repeatedly diluting the initial solution.

Methods for determining the CMC are based on observing sharp changes in the physicochemical properties of surfactant solutions as their concentration varies. This behavior is associated with micelle formation in solution, which indicates the emergence of a new phase and leads to noticeable changes in system properties [12].

One such method involves measuring the surface tension of surfactant solutions at concentrations below and above the CMC. Plotting surface tension as a function of the logarithm of surfactant concentration allows the CMC to be determined as the intersection point of two distinct regions on the graph.

Ten surfactant solutions of different concentrations, including the expected CMC, are prepared in pre-weighed beakers. The mass of each solution is precisely adjusted to 50 g by diluting a stock surfactant solution with a mass concentration of 200 mg/dm^3.

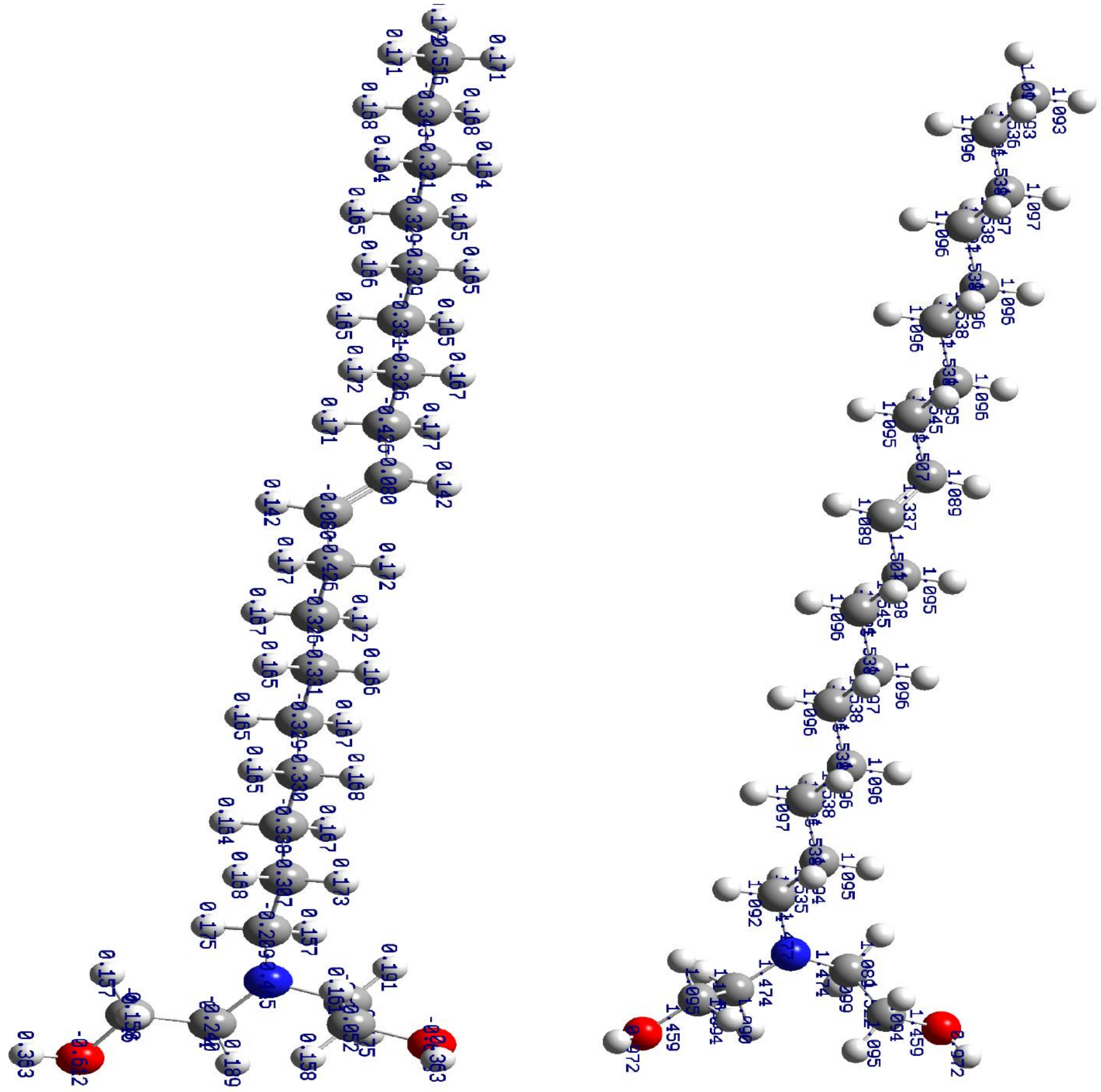
For approximate determination of the CMC region, the temperature of the water bath is maintained at (20 ± 1) °C or 5 °C above the Krafft point for anionic surfactants. For nonionic surfactants, measurements are conducted at (20 ± 1) °C. In each beaker containing the solution and covered with a watch glass, surface tension is measured in accordance with the tensiometer operating instructions.

3. Results

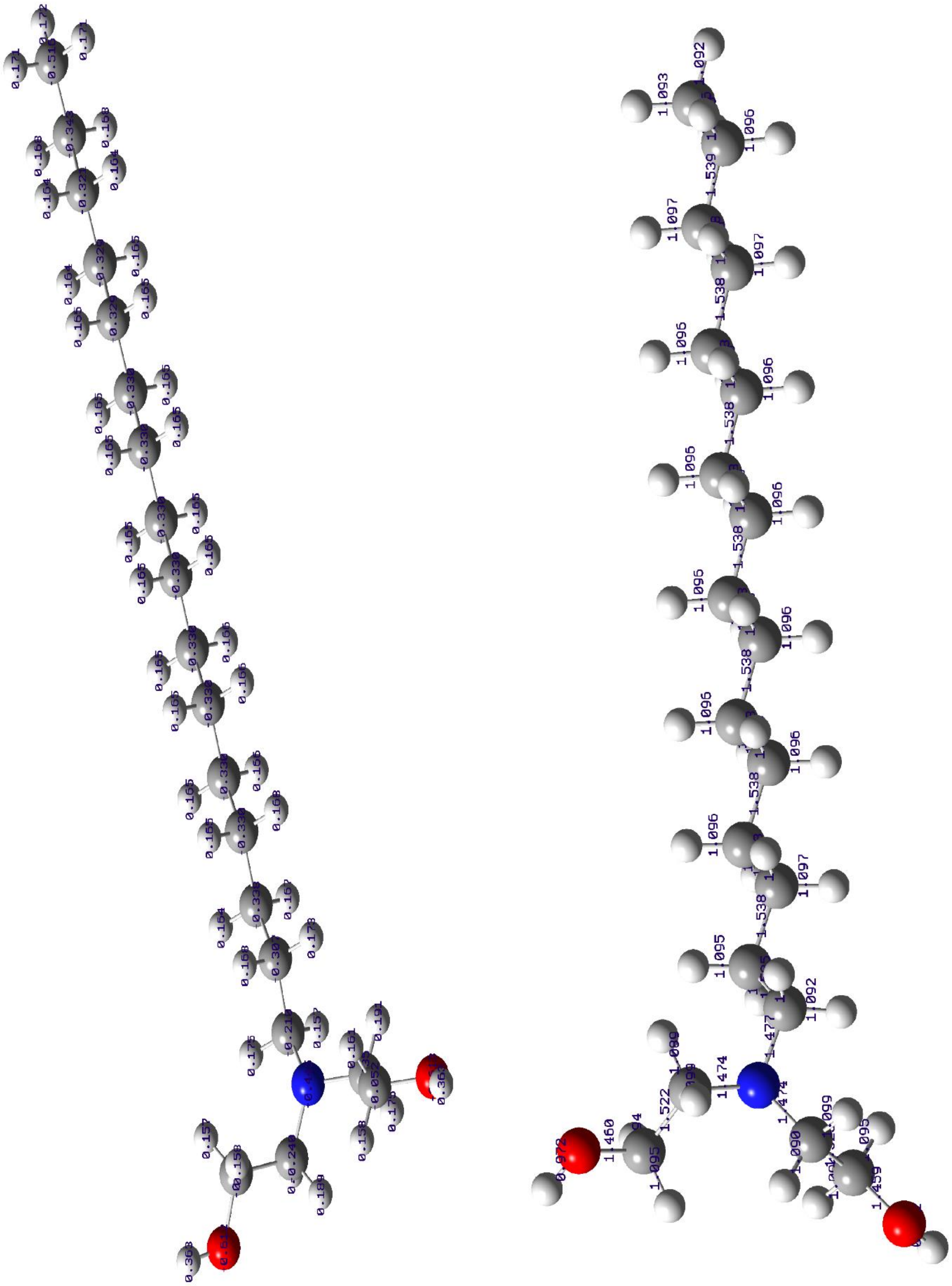
3.1. Molecular Modeling of Synthesized Surfactants

Bond length refers to the distance between the nuclei of two bonded atoms. It is one of the fundamental geometric characteristics of a molecule and plays an important role in understanding the strength and stability of chemical bonds, as well as the overall molecular geometry. Molecular charge represents the total electric charge of a molecule. The charge value depends on the distribution of electrons among the atoms and has a significant influence on the chemical and physical properties of the molecule.

Bond length and molecular charge are considered together to understand molecular chemical reactivity and stability.



a)



b)

Figure 1. Optimized bond lengths and charge values of the C16 and C18 hydrocarbon chains present in CSOS.

The molecular modeling results (Figure 1) clarify the structural characteristics of the dominant C16 and C18 hydrocarbon chains present in cottonseed oil–derived surfactants. The optimized geometries show stable, extended aliphatic conformations typical of long-chain fatty acid derivatives and favorable for surface activity.

The calculated bond lengths correspond to standard sp^3 and sp^2 carbon–carbon bonds, with minor variations in the C18 chains due to unsaturation. These structural differences influence chain flexibility and packing behavior, which are important factors in interfacial adsorption and micelle formation. Partial charge analysis indicates a largely nonpolar hydrocarbon backbone, with slight polarization near functional groups, contributing to intermolecular interactions in aqueous systems.

The coexistence of C16 and C18 chains provides a balance between structural stability and flexibility, supporting efficient emulsification and surface-active performance. Overall, the modeling results confirm the suitability of cottonseed oil as a renewable source for diethanolamide surfactants and provide a theoretical basis for their functional properties in aqueous applications.

3.2. Chemical Characterization

Chromatographic analysis plays a crucial role in determining the fatty acid composition of fatty acid mixtures derived from oils. It enables a more accurate identification and quantification of individual fatty acids in oils, which is important not only for fundamental scientific research but also critical for quality control in the food, cosmetic, and pharmaceutical industries.

Figure 2. shows the chromatogram of fatty acids extracted from cottonseed oil. As in the previous case, Table 1 summarizes the qualitative and quantitative results obtained by mass spectrometric analysis. The chromatogram similarly demonstrates that fatty acids began to be detected after the fifth minute of analysis, indicating comparable chromatographic behavior of fatty acids from sunflower and cottonseed oils.

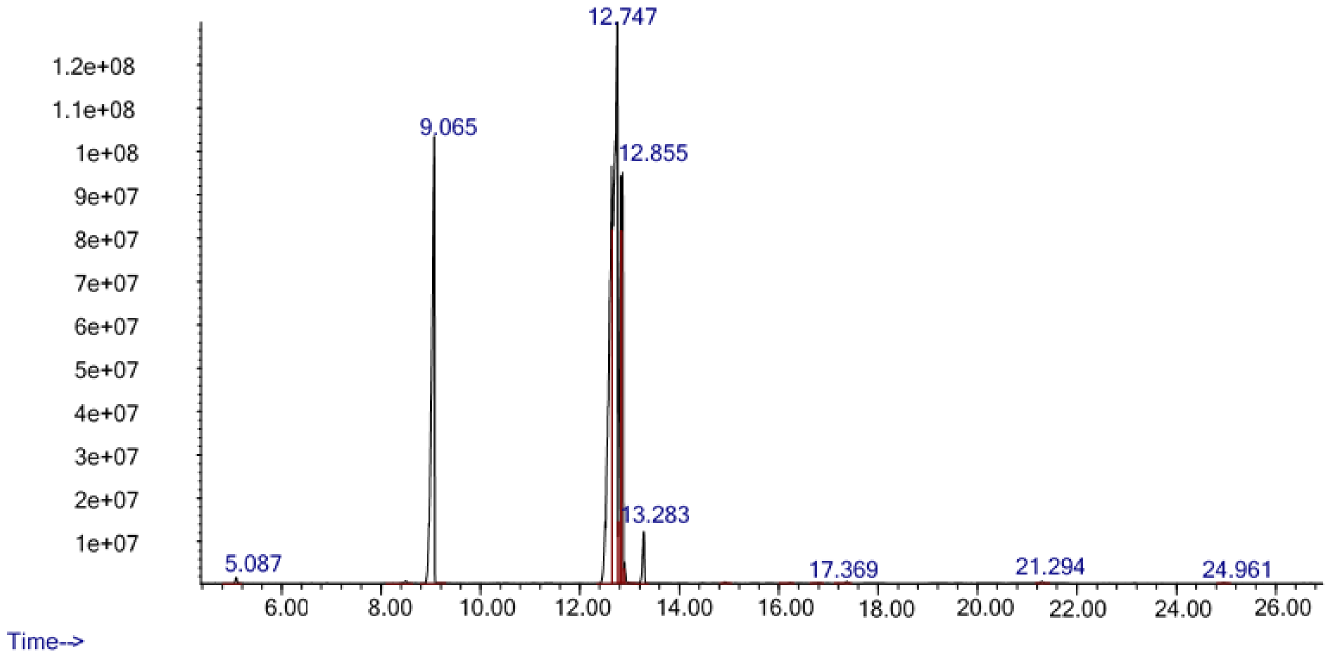


Figure 2. The chromatogram of fatty acids extracted from cottonseed oil.

Table 1. Qualitative and quantitative analysis of fatty acids in cottonseed oil.

No	Name of the acid	Quantities, %	Match with the base of the device
1.	Tetradecanoic acid	0,68	95
2.	Hexadecanoic acid	28,76	97
3.	9.12-Octadecadienoic acid	45,43	98
4.	9-Octadecanoic acid	21,42	96
5.	Octadecanoic acid	3,22	98
6.	Eicosanic acid	0,40	95
7.	Docosan acid	0,09	93

As shown in Table 1, seven major fatty acids were identified in cottonseed oil: myristic, palmitic, linoleic, oleic, stearic, arachidic, and behenic acids. This fatty acid profile is similar to that observed in the sunflower oil sample, indicating comparable chemical compositions of the two oils.

However, cottonseed oil contains a substantially higher proportion of saturated fatty acids (33.15%) compared to the sunflower oil sample (11.03%), and consequently a lower proportion of unsaturated fatty acids (66.85% versus 88.97%). Despite this difference, linoleic acid remains the dominant unsaturated fatty acid in cottonseed oil, accounting for 45.43% of the total fatty acid content, although its proportion is lower than that in sunflower oil (60.26%). Palmitic acid is the predominant saturated fatty acid in cottonseed oil (28.76%), whereas its content in the sunflower oil sample was significantly lower (6.72%).

Based on the obtained data, the average molecular weights of the fatty acid mixtures derived from the studied materials were calculated. The value was 272.55 g/mol for cottonseed oil fatty acids.

After synthesis surfactant from this fatty acids mixture with diethanolamine, its different characterization studied.

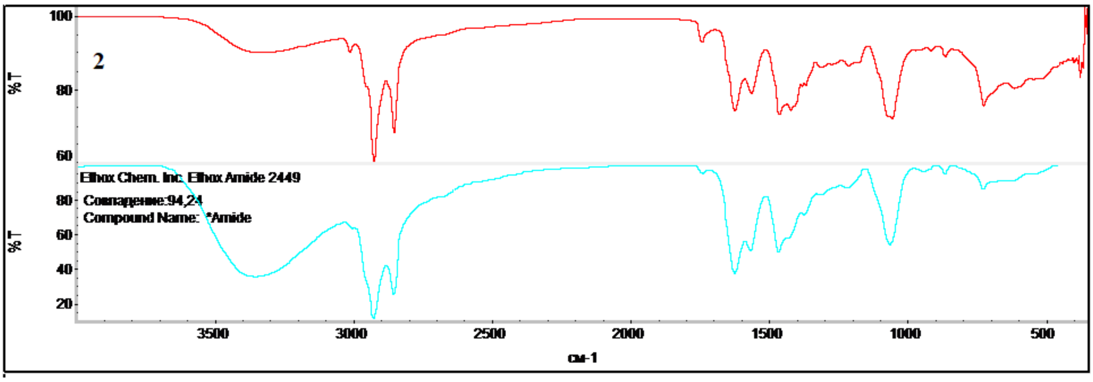


Figure 3. IR spectra of cottonseed CSOS.

It can be seen from Figure 3. that absorption bands observed at approximately 1739 cm^{-1} (1736–1739 cm^{-1}), indicate the presence of carbonyl (C=O) functional groups. Bands in the range of 1600–1500 cm^{-1} (notably at 1638.78 and 1536.78 cm^{-1}) are associated with amide bond vibrations, corresponding to the amide I and amide II bands, which are characteristic of compounds containing amide groups. Peaks detected between 1464.78 and 1051.19 cm^{-1} are attributed to C–H stretching vibrations in aliphatic chains and may also reflect the presence of C–O stretching vibrations [13].

Overall, all surfactant sample exhibit characteristic absorption bands associated with aliphatic hydrocarbon chains and amide linkages, which is typical for surfactants synthesized from fatty acids and diethanolamine.

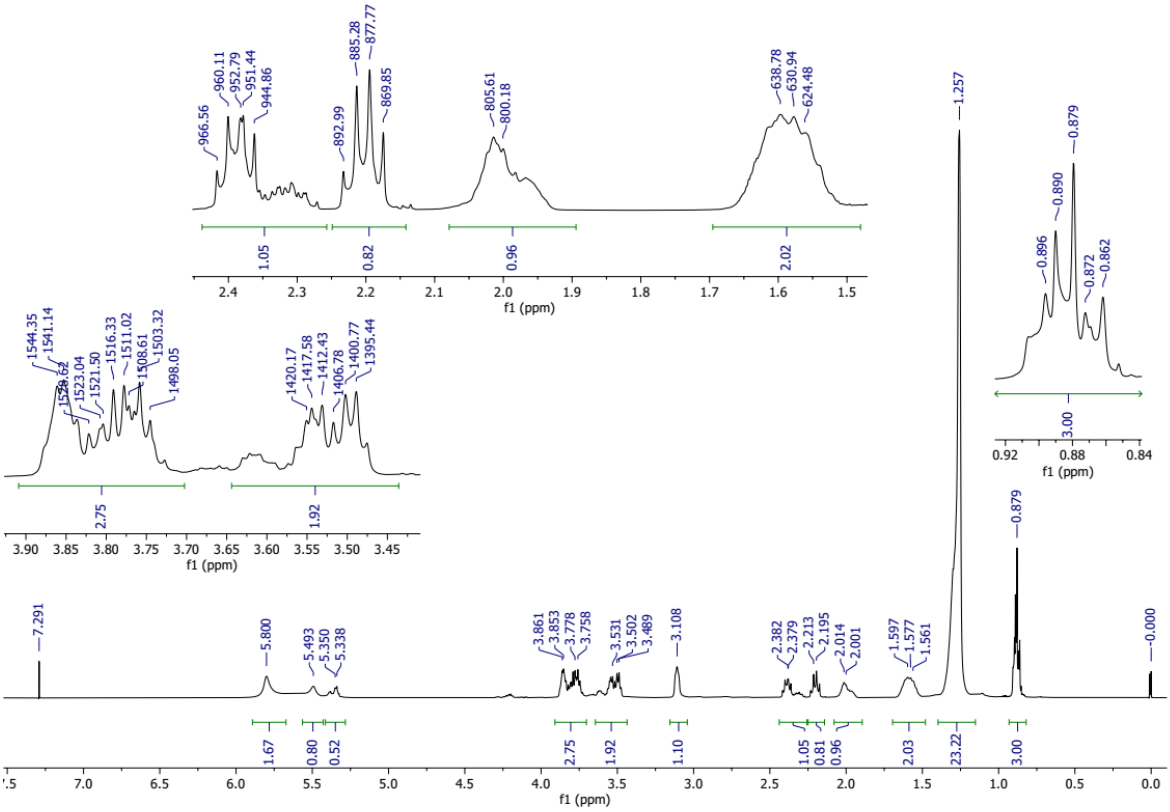


Figure 4. NMR spectrum of a surfactant derived from cottonseed oil.

A comparative analysis of the ^{1}H NMR spectra of diethanolamide derived from natural cottonseed oil revealed the presence of methylene group signals in the range of −0.5 to 3 ppm in sample. This observation indicates a similar chemical structure of diethanolamides obtained from various oils with respect to the main hydrocarbon backbone composed of methylene units, which is characteristic of fatty acid amides.

The presence and consistency of amide proton signals (approximately 7–8 ppm) and signals associated with ether groups (3.5–4 ppm) suggest that the amidation reactions proceed in a similar manner for different oils, despite variations in the initial fatty acid composition.

Distinct peaks observed in certain spectra may indicate the presence of specific functional groups or impurities, which could originate from the initial oils or fats or arise due to particular conditions of the amidation process. Variations in signal intensity and chemical shift positions among the samples highlight differences in fatty acid chain length and degree of saturation, which in turn influence the physicochemical properties of the resulting amides.

3.3. Surface Activity Studies

This sub-section discusses methods for determining the critical micelle concentration (CMC) of the synthesized surfactant and its solutions. Investigation of this parameter makes it possible to determine the concentration ranges at which these surfactants can effectively function as emulsifiers and stabilizers in various systems. The obtained data form an important basis for evaluating their applicability in the oil recovery, cosmetic, and other industrial sectors.

For result analysis, a graph of surface tension versus concentration is plotted, and the region containing the CMC is identified. Subsequently, six new surfactant solutions are prepared, and their surface tension is measured over a period of 3 hours at 15-minute intervals. Based on these data, a surface tension versus concentration curve is constructed, from which the CMC is determined (Fig.5).

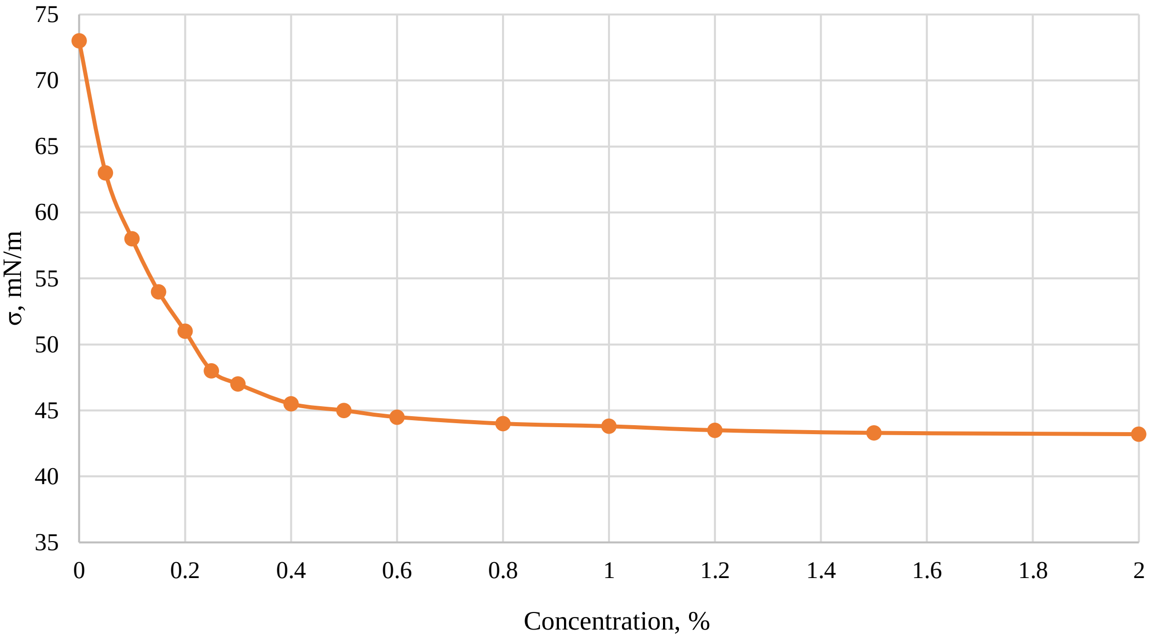


Figure 5. Variation of surface tension of surfactant solutions (25 °C).

The curve showing the dependence of surface tension on concentration for solutions of the synthesized surfactants in the concentration range from 0 to 2 wt.% is presented in Figure 5.

The dependence of surface tension (σ) for PAVHS, as shown in Figure 3.1, demonstrates a linear decrease in surface tension with increasing surfactant concentration. This behavior is attributed to the progressive adsorption of PAVHS molecules at the air–water interface. Upon reaching a certain concentration, the surface tension attains a stable plateau value of 43.2 mN/m [14].

The observed reduction in surface tension confirms the effectiveness of PAVHS as a surface-active agent. This behavior is strongly influenced by the molecular structure of PAVHS, particularly the length of its hydrophobic alkyl chain and its hydrophilic–lipophilic balance (HLB). A suitable HLB value promotes the adsorption of amphiphilic molecules at the interface, thereby enhancing their ability to lower the surface tension of the aqueous system [15].

Determination of the critical micelle concentration (CMC) is a key parameter in evaluating the performance of PAVHS. The CMC represents the concentration at which PAVHS molecules begin to self-assemble into micelles, leading to noticeable changes in solution properties. This parameter is essential for assessing the efficiency and practical applicability of the surfactant in various technological processes.

The CMC values of PAVHS were determined from the dependence of surface tension on the logarithm of surfactant concentration. The obtained values are 15.6 mg/L (CMC_1) and 31.2 mg/L (CMC_2), indicating efficient micelle formation at relatively low concentrations.

In addition, the cloud point (CP) of PAVHS was found to be 57–59 °C, reflecting its thermal behavior and solubility characteristics in aqueous solutions. The combination of relatively low CMC values and a moderate cloud point suggests that PAVHS can effectively form micelles and maintain surface activity over a broad temperature range.

Overall, the physicochemical characteristics of PAVHS demonstrate its suitability for applications requiring efficient surface tension reduction and stable micelle formation, particularly under moderate temperature conditions

4. Discussion

This study demonstrates the feasibility of utilizing cottonseed oil as a renewable raw material for the synthesis of nonionic diethanolamide surfactants with favorable structural and functional characteristics. The combination of experimental synthesis and molecular modeling provides a comprehensive understanding of how fatty acid composition influences surfactant behavior.

The molecular modeling results confirm that the dominant C16 and C18 hydrocarbon chains adopt stable, energetically favorable conformations. Minor structural differences associated with chain length and unsaturation affect molecular flexibility and packing properties, which are critical parameters for interfacial activity and micelle formation. The largely nonpolar nature of the hydrocarbon backbone, together with localized charge polarization near functional groups, supports efficient intermolecular interactions in aqueous environments.

From a practical standpoint, the presence of both saturated and unsaturated chains contributes to balanced surface-active performance. C16 chains promote compact molecular packing and stability, while C18 chains enhance flexibility and adaptability at interfaces. This combination is advantageous for applications requiring effective emulsification, detergency, and stabilization.

Overall, the results support the potential of cottonseed oil–derived diethanolamides as environmentally friendly surfactants. The findings provide a theoretical basis for their observed properties and highlight the value of plant-based raw materials in the development of sustainable surfactant systems for industrial and biomedical use.

5. Conclusion

This study demonstrates the successful synthesis of diethanolamide surfactants from cottonseed oil fatty acids under solvent-free catalytic conditions. Structural characterization confirmed the formation of amide linkages and hydrocarbon backbones typical of fatty acid amides. The surfactants exhibited strong surface activity, with low CMC values and stable cloud points, indicating efficient micelle formation and favorable thermal behavior. Compared to conventional surfactants, the cottonseed oil–derived products combine high yield, biodegradability, and performance, underscoring their suitability for industrial applications such as enhanced oil recovery, cosmetics, and detergents. Overall, the work contributes to the development of eco-friendly surfactants from renewable agricultural by-products, supporting sustainable materials chemistry and circular economy principles.

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