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SYNTHESIS OF SURFACTANTS BASED ON FATTY ACIDS AND THEIR SPECTRAL ANALYSIS

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Abstract: This study investigates the synthesis of surfactants based on higher fatty acids and their physicochemical characterization. Natural and synthetic fatty acids with different hydrocarbon chain lengths were used as raw materials. The synthesized surfactants were characterized using infrared (IR) and ultraviolet (UV) spectroscopy to confirm their structure and identify functional groups. Spectral data analysis allowed determination of the structural features of the obtained compounds. The results confirm the successful synthesis and structural correspondence of the target products. The synthesized surfactants show potential for applications in enhanced oil recovery, detergent formulations, and various industrial processes.

Keywords: surfactants, fatty acids, synthesis, IR spectroscopy, UV spectroscopy, spectral analysis, micellization.

Introduction. Surfactants (surface-active agents) constitute a class of chemical compounds capable of significantly altering interfacial interactions due to the presence of hydrophilic and hydrophobic moieties within the molecule. They are widely applied in industry, including oil production, the manufacture of detergents, cosmetics, and pharmaceuticals. In recent years, increasing interest has been directed toward surfactants based on natural components, particularly fatty acids, owing to their biodegradability, low toxicity, and environmental safety. The synthesis of such compounds makes it possible to obtain substances with predetermined physicochemical properties, including micelle formation, surface tension, and compatibility with various media. Effective characterization of the synthesized surfactants requires the use of spectroscopic methods, such as infrared and ultraviolet spectroscopy, which allow the identification of functional groups and confirmation of the molecular structure. The present work is devoted to the synthesis of fatty acid–based surfactants and their spectral analysis in order to establish the structure and specific features of the chemical structure of the obtained compounds.

Methodology & empirical analysis. Fatty acids of natural origin, such as linoleic, oleic, and stearic acids, were used as starting materials for the preparation of biodegradable and environmentally safe surfactants. The synthesized compounds are

characterized by high efficiency and a broad range of applications in the chemical and oil industries.

Within the framework of this study, the results of surfactant synthesis and their spectral analysis are presented in order to determine their structural and functional characteristics. The application of spectroscopic methods makes it possible to investigate key parameters, such as the presence of functional groups, the degree of saturation, and molecular structure, which subsequently allows the evaluation of the efficiency and stability of the synthesized surfactants.

Results. To determine the required amount of diethanolamide, chromatographic analysis of the isolated fatty acids was carried out, on the basis of which the average molecular weight of the mixture was calculated. Based on the obtained data, the masses of reagents for the synthesis of fatty acid diethanolamide were determined, maintaining a 1:1 molar ratio. Since the reaction releases water, 0.5% aluminum oxide (Al_2O_3), relative to the mass of fatty acids, was added as a catalyst. The reaction was performed in a three-neck flask under an argon atmosphere: the mixture was heated to 80 °C for 20–30 minutes, then the temperature was increased to 150 °C while maintaining stirring at 400 rpm. The reaction was completed after 2 hours. The fatty diethanolamide was isolated by distillation at a pressure of 75 ± 1 mmHg, monitoring the progress of the process by measuring the acid number every 30 minutes until stabilization of the values. The product yield was 95–98%.

$$RCOOH + NH(C_2H_4OH)_2 > \quad RCON(C_2H_4OH)_2 + H_2O$$

The obtained surfactants synthesized on the basis of sunflower, cottonseed, and rapeseed oils were conventionally designated as DEASO, DEACO, and DEARO, respectively.

Quality indicators of the reaction between the fatty acid mixture and diethanolamide

FA	FA molecular weight, g/mol	DEA mass, g/100 g*	Surfactant molecular weight, g/mol	Yield, %	Surfactant designation
SO	278,95	38,4	365,95	97,2	DEASO
CO	273,71	39,1	360,71	94,9	DEACO
RO	259,22	42,3	346,22	96,8	DEARO

Analysis of the data on the reaction quality between the fatty acid mixture and diethanolamide shows that the yield and molecular weight of the final surfactant vary depending on the initial oil (sunflower, cottonseed, or rapeseed).

For sunflower oil, the molecular weight of the surfactant was 365.95 g/mol, and the yield reached 97.2%, indicating a high degree of conversion. In the case of cottonseed oil, the molecular weight of the surfactant was slightly lower, 360.71 g/mol, and the yield amounted to 94.9%, which is also high but somewhat lower than that obtained from sunflower oil. For rapeseed oil, the molecular weight of the surfactant was 346.22 g/mol, with a yield of 96.8%.

Thus, sunflower and rapeseed oils demonstrate the highest surfactant yields, whereas cottonseed oil shows a slightly lower value. These data may indicate differences in reaction efficiency depending on the molecular weight and structure of the fatty acids present in the various oils.

In this study, infrared (IR) spectra of the synthesized compounds were recorded under liquid film conditions applied between KBr plates. Spectral measurements were carried out using a SPECORD 75 IR instrument in the wavenumber range of 3800–700 cm^{-1}, which allowed detailed examination of the functional groups and molecular structure of the surfactants.

The study of the IR spectra of the surfactants obtained from different oils makes it possible to identify characteristic functional groups and to compare their molecular structures.

The study of the IR spectra of the surfactants obtained from different oils makes it possible to identify characteristic functional groups and to compare their molecular structures.

In the upper spectrum corresponding to the DEASO sample, several characteristic bands are observed. The band in the region of approximately 3369 cm^{-1} corresponds to N–H and O–H stretching vibrations, indicating the presence of an amide group as well as hydrogen bonding within the molecular structure. Intense bands in the regions of 2920 and 2850 cm^{-1} are attributed to C–H stretching vibrations of methyl and methylene groups, confirming the presence of a long hydrocarbon chain characteristic of the fatty acids from which the diethanolamide was synthesized.

Two significant peaks are also observed in the regions of 1650–1600 and 1550 cm^{-1}, which are associated with C=O (Amide I) and N–H (Amide II) vibrations in amide groups. These peaks are key to the identification of amide bonds and confirm the successful reaction between the fatty acids and diethanolamine.

The lower spectrum, corresponding to the standard sample of lauric acid diethanolamide, exhibits similar absorption bands. This spectrum also shows a broad band at 3339 cm^{-1}, indicating N–H and O–H vibrations characteristic of amide and hydroxyl groups. Peaks in the region of 2900–2800 cm^{-1} correspond to C–H vibrations in the long hydrocarbon chain. The spectrum also demonstrates distinct peaks at 1650 and 1550 cm^{-1}, attributed to C=O and N–H vibrations.

Comparison of the upper and lower spectra reveals similarity in the main absorption bands, indicating analogous functional groups and molecular structures in both samples. In particular, the coincidence of bands in the regions of 3400 cm^{-1} (N–H and O–H), 2900–2800 cm^{-1} (C–H), as well as 1650–1600 cm^{-1} and 1550 cm^{-1} (C=O and N–H of amide groups) confirms the presence of similar amide bonds and long hydrocarbon chains.

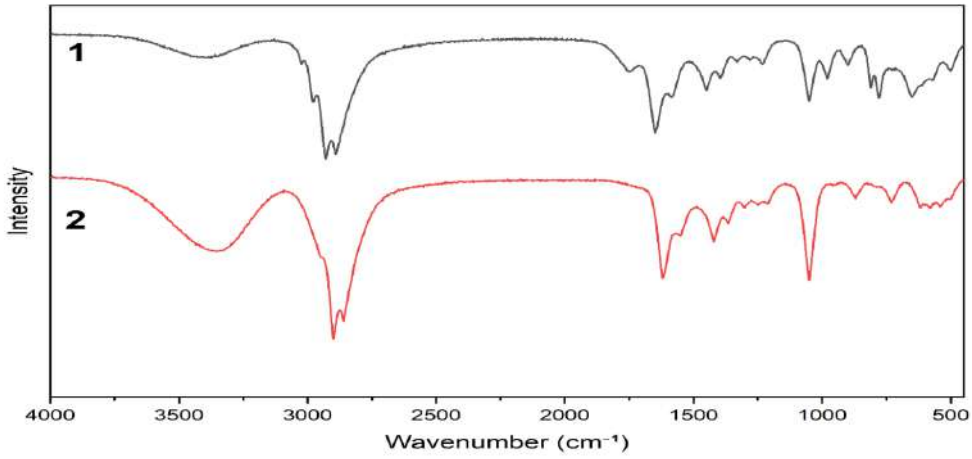

Figure 1. IR spectrum of the DEASO sample

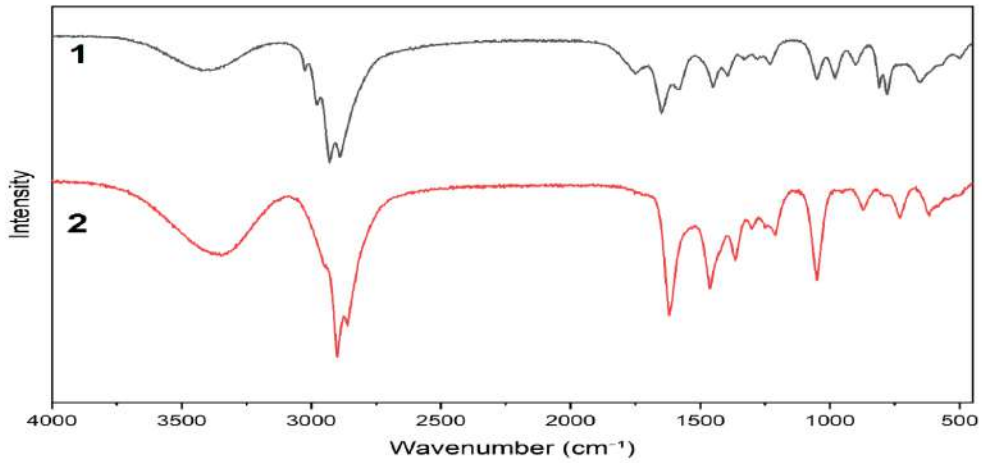

Figure 2. IR spectrum of the DEACO sample

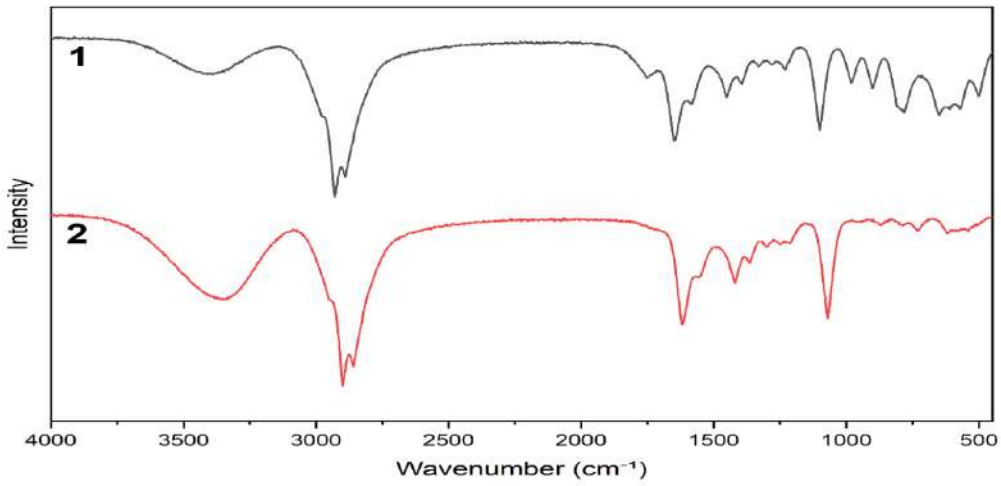

Figure 3. IR spectrum of the DEARO sample

A broad absorption band is observed, indicating the presence of N–H and O–H stretching vibrations associated with amide and hydroxyl groups. The peak around 1650

cm^{-1} corresponds to C=O stretching vibrations in amide groups, confirming the presence of an amide linkage in the DEACO structure.

Both spectra exhibit similar principal absorption bands, including regions corresponding to N–H, C=O, and C–H vibrations, indicating the presence of common functional groups (amide groups and hydrocarbon chains). However, due to differences in the fatty acid composition (typical of cottonseed and rapeseed oils), the intensities and exact positions of the peaks may differ slightly.

Thus, both spectra confirm the amide structure of the compounds synthesized from fatty acids of sunflower, cottonseed, and rapeseed oils, indicating the successful synthesis of diethanolamides with analogous functional groups and characteristic amide absorption bands.

Conclusions. As a result of the study, methods for the isolation of fatty acids were developed, the conditions for the synthesis of diethanolamides were optimized, and a comprehensive analysis of the synthesized surfactants was carried out to confirm their composition and structure.

Sunflower, cottonseed, and rapeseed oils were used as the objects of investigation, each characterized by a unique fatty acid profile. This approach made it possible to examine the influence of different compositional ratios of saturated and unsaturated fatty acids on the final properties of the synthesized surfactants.

Gas chromatography was employed for the identification and quantitative analysis of the fatty acid composition, revealing the content of saturated and unsaturated acids in each type of oil. The obtained data served as the basis for the subsequent synthesis of surfactants, since the fatty acid composition directly affects the efficiency and stability of the resulting surfactants under various operating conditions.

Particular attention was paid to the optimization of synthesis parameters, such as the molar ratio of reagents, reaction temperature, and synthesis time. Spectral analysis of the synthesized surfactants performed by infrared (IR) spectroscopy confirmed the presence of amide groups and characteristic hydrocarbon chains, indicating the successful formation of diethanolamides. Comparison of the IR spectra of the different surfactants demonstrated that the structure and composition of the diethanolamides are consistent with the original fatty acids isolated from each type of oil.

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