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Research Article

Synthesis of a Novel Surface-Active Agent Based on Hexadecylamine and Ethylene Chlorohydrin for the Removal of Thin Oil Films from Water Surfaces

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Abstract

A novel surface-active salt based on hexadecylamine and ethylene chlorohydrin was successfully synthesized. The composition and molecular structure of the synthesized salt were characterized by Fourier-transform infrared (FTIR) and UV–Visible (UV–Vis) spectroscopy, while its principal physicochemical properties were determined. Tensiometric measurements demonstrated the high surface activity of the synthesized salt at the air–water interface. The specific electrical conductivity of its aqueous solutions was measured using an electroconductometric method, confirming that the synthesized salt exhibits electrolyte behavior. The aggregate sizes of hexadecylethanolammonium chloride (HDEAC) in aqueous solutions at different concentrations were determined by dynamic light scattering (DLS), and the corresponding particle size distribution histograms were obtained.

Keywords: catanionic salts, FTIR, UV–Vis spectroscopy, hexadecylethanolammonium chloride, oil spill remediation, oil collection

1. Introduction

The removal of petroleum-derived contaminants from water surfaces is of paramount importance for environmental protection. When crude oil and petroleum products enter aquatic environments, they spread spontaneously under the combined influence of surface tension, wind, and wave action, forming thin oil films on the water surface. This spontaneous spreading process is accompanied by a reduction in the system's free energy proportional to the increase in the contaminated surface area and is characteristic of dispersed water–oil emulsion systems discharged into the hydrosphere through industrial wastewater. Regardless of the mechanism of film formation, such oil films pose a serious environmental threat because the surface microlayer of aquatic ecosystems serves as a critical habitat for the most sensitive organisms and for the early developmental and reproductive stages of numerous aquatic species. Furthermore, the presence of an oil film adversely affects gas exchange, heat transfer, and water exchange between the hydrosphere and the atmosphere, thereby disrupting the physicochemical and biological processes occurring at the air–water interface [1], [2].

As is well established, relatively thick oil films are typically removed from water surfaces by mechanical methods. However, in the case of thin oil films, physicochemical techniques have proven to be considerably more effective. The removal and recovery of thin oil films (up to 0.5 mm in thickness) from aquatic environments is of great environmental and economic importance, since the recovered oil can be recycled and reused [3], [4].

In this regard, oil-collecting and oil-dispersing reagents play a crucial role. Owing to spontaneous emulsification, dispersants disrupt the continuous oil film, thereby restoring water, gas, and heat exchange between the hydrosphere and the atmosphere while promoting the natural biochemical oxidation of finely dispersed oil droplets suspended within the water column. Consequently, oil dispersion has become a widely accepted approach as an effective approach for mitigating oil-film contamination on water surfaces. Dispersing formulations are particularly suitable for the remediation of large-scale oil spills.

Alternatively, oil films can be removed from water surfaces using oil-collecting agents. In many cases, the dispersing and oil-collecting effects of these reagents overlap, as certain formulations exhibit both properties simultaneously. The principal active components of both oil-collecting and dispersing formulations are surfactants (surface-active agents) [5], [6], [7], [8]. To be effective as oil-collecting reagents, surfactants should exhibit rapid spontaneous spreading over the water surface with a spreading pressure exceeding 30 mN m^{-1}, possess low volatility, demonstrate adequate solubility in both water and oil, and maintain high chemical stability under operating conditions.

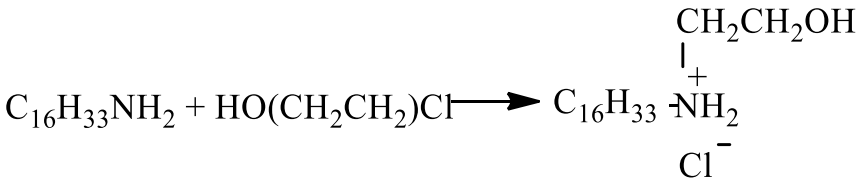
Oil-dispersing and oil-collecting reagents offer several advantages over conventional methods used to combat oil pollution. These include high efficiency, the ability to be applied regardless of the location, timing, or environmental conditions of an oil spill, and ease of application. Such reagents are particularly effective in the removal of ultrathin oil films, for which conventional mechanical and many physicochemical remediation techniques are often ineffective.

For Azerbaijan, with its numerous water bodies that have been extensively contaminated by crude oil and petroleum products, the development and investigation of novel, highly efficient oil-dispersing and oil-collecting reagents remain a matter of considerable environmental importance. Such reagents are typically based on amphiphilic chemical compounds containing both hydrophobic and hydrophilic structural fragments. This dual functionality provides a strong affinity for both the oil phase and the aqueous medium, thereby ensuring their high efficiency in the remediation of oil-contaminated water surfaces [9], [10], [11], [12], [13], [14].

2. Materials and Methods

The present study describes the synthesis and characterization of a novel catanionic surfactant with oil-collecting properties.

For this purpose, hexadecylethanolammonium chloride (HDEAC) was synthesized according to the procedure described previously [7]. The surfactant was prepared by reacting hexadecylamine with ethylene chlorohydrin in an equimolar ratio. The reaction was carried out at 21 °C for 4–5 h. The reaction scheme is presented below:



The synthesized hexadecylethanolammonium chloride (HDEAC) was obtained as a yellowish, viscous resin. The product was completely soluble in ethanol, whereas in water it formed a stable dispersion accompanied by the generation of abundant foam. The melting point of the synthesized salt was found to be above 200 °C.

The chemical composition and molecular structure of HDEAC were characterized by Fourier-transform infrared (FTIR) and UV–Visible (UV–Vis) spectroscopy. FTIR spectra (Figure 1) were recorded using an ALPHA FTIR spectrometer (Bruker, Germany) over the wavenumber range of 600–4000 cm^{-1}.

The surface tension (σ) of the synthesized catanionic surfactant, HDEAC, was measured at the air–water interface using a Du Noüy ring tensiometer (KSV Sigma 702, Finland). Aqueous surfactant solutions with

concentrations ranging from 0.00025 to 0.1 wt.% were prepared using deionized water. After equilibration for 24 h, their surface tension was measured at 19 °C.

The specific electrical conductivity (κ) of the aqueous HDEAC solutions was determined using an Anion-402 conductivity meter (Russia). The conductivity of aqueous solutions with concentrations ranging from 0.001 to 0.1 wt.% was measured 48 h after their preparation.

3. Results and Discussion

The chemical composition and molecular structure of hexadecylethanolammonium chloride (HDEAC) were characterized by Fourier-transform infrared (FTIR) and UV–Visible (UV–Vis) spectroscopy. The corresponding FTIR and UV–Vis spectra are presented in Figures 1 and 2, respectively.

The FTIR spectrum of the synthesized HDEAC is shown in Figure 1. A broad absorption band at 3263.4 cm^{-1} is attributed to the stretching vibrations of the hydroxyl (O–H) group. The absorption bands observed at 2921.7 and 2852.1 cm^{-1} correspond to the asymmetric and symmetric C–H stretching vibrations of the CH_3 and CH_2 groups, respectively. The absorption band at 1663.0 cm^{-1} is assigned to the stretching vibration of the carboxylate (C(O)O) group. The peak at 1577.8 cm^{-1} is attributed to the $–NH_2^+CH_2–$ group. The bands at 1254.2 and 1148.1 cm^{-1} are associated with the deformation vibrations of the C–H bonds in the methylene and methyl groups. The absorption peak at 834.1 cm^{-1} corresponds to the C–OH stretching vibration, whereas the band at 701.8 cm^{-1} is assigned to the deformation vibrations of the $(CH_2)_n$ alkyl chain.

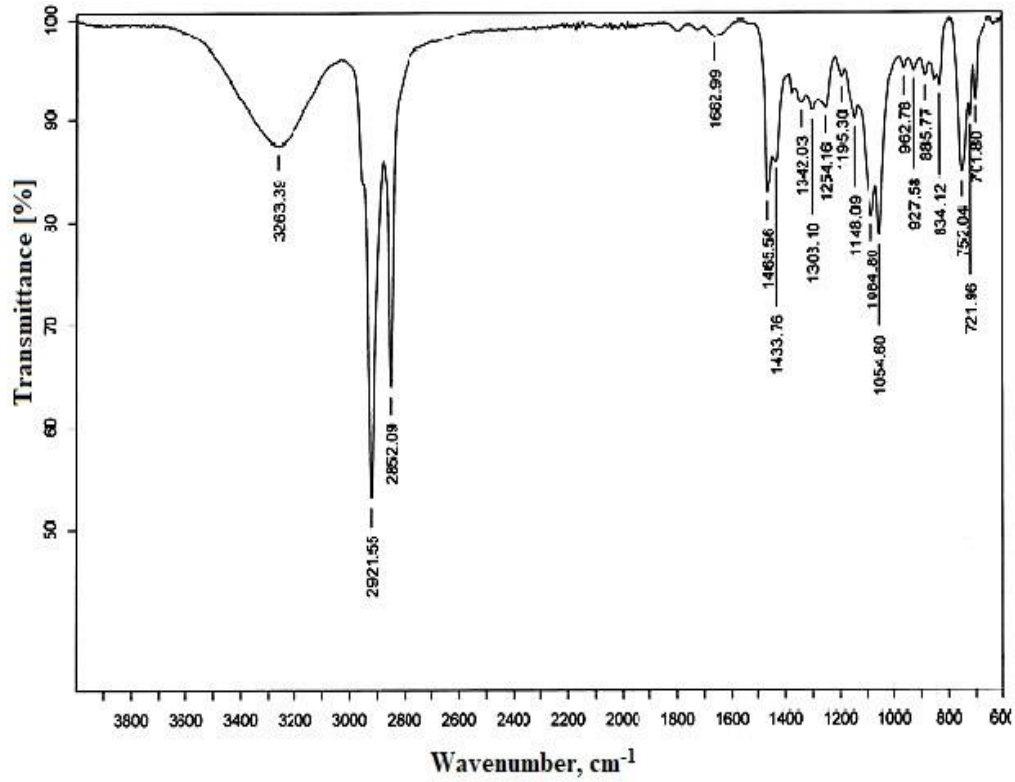


Figure 1. The FTIR spectrum of the HDEAC.

The absorption band at 211 nm is attributed to electronic transitions associated with the molecular structure of HDEAC (Figure 2).

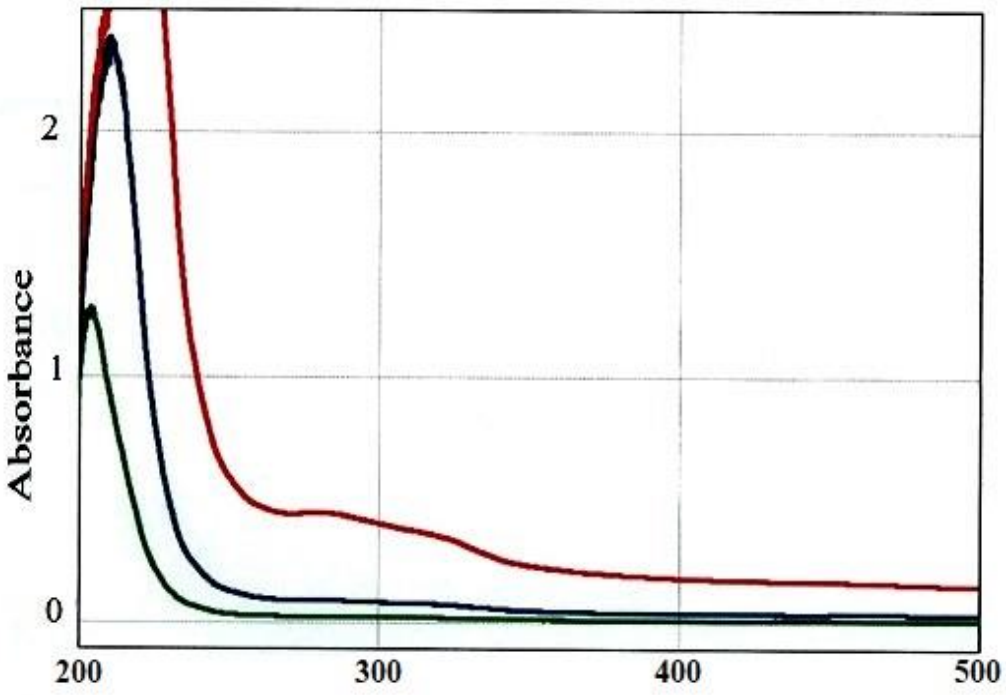


Figure 2. UV–Visible spectrum of HDEAC.

The electroconductometric analysis revealed that the specific electrical conductivity (κ) of aqueous HDEAC solutions increased monotonically with increasing surfactant concentration. As the concentration was raised from 0.02 to 0.7 wt.% at 19 °C, the conductivity increased from 60.2 to 226.0 μS cm^{-1}, whereas the conductivity of deionized water under the same conditions was 5 μS cm^{-1}. These findings confirm that the synthesized hexadecylethanolammonium chloride exhibits the characteristic behavior of an electrolyte in aqueous solution.

The results of the surface tension measurements demonstrated that the surface tension of pure water at the air–water interface was 72.1 mN m^{-1} at 19 °C. The synthesized HDEAC exhibited pronounced surface activity, confirming its excellent surface-active properties. In the presence of HDEAC, the surface tension decreased substantially from 72.1 mN m^{-1} (pure water) to 29.5 mN m^{-1}, indicating its strong ability to adsorb at the air–water interface and significantly reduce the interfacial free energy.

The oil-collecting performance of the synthesized compounds was evaluated under laboratory conditions according to the procedure described in Ref. [4]. Briefly, 40 mL of water and 1 mL of crude oil were introduced into a Petri dish. After the formation of a uniform oil film on the water surface, 0.02 g of the reagent (applied as an aqueous solution) was added, and the area of the oil slick collected into a compact spot (S_0) was measured immediately.

The experiments were carried out using distilled water, freshwater, and seawater (Caspian Sea water). Crude oil obtained from the Pirallahi oil field possessed a density of $\rho_{20} = 0.924$ g cm^{-3}. In the laboratory tests, crude oil from the Pirallahi oil field was spread over the surfaces of the three types of water: distilled water, freshwater, and seawater (Caspian Sea water), forming a thin oil film with a thickness of approximately 0.17 mm. The efficiency of the synthesized reagents was assessed by monitoring the reduction in the surface area of the oil slick following treatment with 5 wt.% aqueous solutions of the synthesized salts. Their oil-collecting performance was evaluated using two parameters: the oil-collection coefficient (K), defined as the ratio of the initial oil-film area to the area of the oil spot after treatment, and the oil retention time (τ), representing the duration for which the collected oil remained compact. The experimental results are summarized in Table 1.

Table 1. Oil-Collecting and Oil-Dispersing Performance of Hexadecylethanolammonium Chloride (HDEAC).

Reagent status	Distilled water		Fresh water		Sea water	
	τ, time	K	τ, time	K	τ, time	K
HDEAC						
100% product	0–0.5 1.0–2.0 3.0–15.0 24.0–168.0	30.9 55.2 60.8 80.6	0–0.5 1.0–2.0 3.0–15.0 24.0–168.0	35.1 43.2 55.7 60.8	0–0.5 1.0–2.0 3.0–15.0 24.0–168.0	38.4 55.3 60.8 80.6
5% product	0–0.5 1.0–2.0 3.0–168.0	35.4 42.5 79.2	0–0.5 1.0–2.0 3.0–168.0	46.1 55.9 60.7	0–0.5 1.0–2.0 3.0–168.0	43.6 65.5 80.6

As shown in Table 1, the synthesized surfactant exhibited excellent oil-collecting performance. Both the undiluted product and its 5 wt.% aqueous dispersion demonstrated high oil-collecting and oil-dispersing efficiencies. The maximum oil-collection coefficient (K) reached 80.6, while the reagent maintained its effectiveness for more than 168 h, indicating excellent long-term stability and sustained performance during oil spill remediation.

4. Conclusion

Hexadecylethanolammonium chloride (HDEAC) was successfully synthesized by the reaction of hexadecylamine with ethylene chlorohydrin. FTIR and UV–Vis analyses confirmed the formation of the target compound, while conductivity and surface tension measurements demonstrated its electrolyte behavior and high surface activity. Laboratory tests showed excellent oil-collection performance in distilled, fresh, and seawater, indicating that HDEAC is a promising surfactant for the remediation of thin oil films from contaminated water surfaces.

Author Contributions

All authors contributed to the conception, investigation, analysis, writing, and revision of the manuscript. All authors have read and approved the final version of the manuscript.

Conflict of Interest

The authors declare no conflicts of interest.

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Abbreviations

Hexadecylethylammonium Chloride (HDEAC), Ultraviolet–Visible Spectrometer (UV–Vis spectrometer), Fourier-Transform Infrared (FTIR), Dynamic Light Scattering (DLS).

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