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Amine Oxide: Synthesis and Analysis †

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Abstract: This paper presents the laboratory synthesis of zwitterionic surfactant—oleylamidopropyldimethylamine oxide (OADAO), as well as the selection of conditions for its implementation. The results of the analysis of synthesis products by potentiometric titration and IR-Fourier spectroscopy methods are presented, which prove the presence of the target product in the composition of the reaction mass at the end of synthesis. The positive results of the synthesis and the absence of light organochlorine compounds (LOC) in the end-products allow us to further consider OADAO, for example, as a gelling agent for aqueous fracturing fluids, by studying its technological characteristics.

Keywords: zwitterionic surfactant; amine oxide; light organochlorine compounds; Fourier transform infrared spectroscopy; potentiometric titration; fracturing fluids

1. Introduction

At the present stage of global development surfactants have found their application practically in the entire oil and gas industry. Surfactants are actively used in oil and gas production, transportation and storage of extracted resources as well as in a wide range of petrochemical and refining processes.

Zwitterionic surfactants are among the most promising for oil and gas applications. However, many of them are produced using organochlorine compounds, which has a negative impact on the refining processes [1]. Therefore, the development of organochlorine-free surfactant production processes is a challenge for oil and gas industry.

Light organochlorine compounds are absent in the entire amine oxides production process which makes these compounds promising zwitterionic surfactants. Furthermore, the initial raw materials for their synthesis are vegetable and animal fats, making them environmentally friendly.

2. Materials and Methods

The synthesis of OADAO was carried out in a four-necked flask using a mechanical stirrer, a backflow condenser, a heater, a temperature sensor and a drip funnel. The chemical reaction of obtaining the target product is shown in Figure 1.

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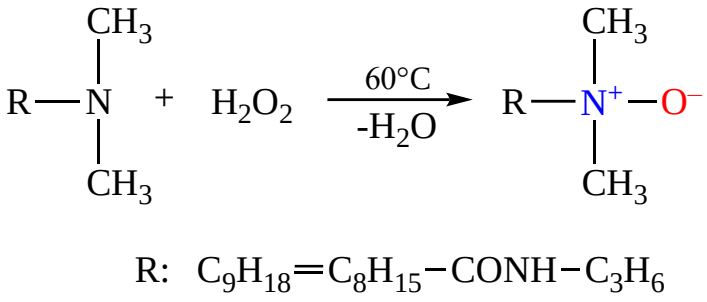

R: $C_9H_{18}=C_8H_{15}-CONH-C_3H_6$

Figure 1. The chemical reaction of obtaining OADAO.

The following steps were identified for the synthesis:

(a) 40.0 g of oleylamidopropyldimethylamine (amine number = 96 mg HCl/g) and 30.0 g of isopropanol are loaded into the reaction four-necked flask. 28.0 g of the prepared 28.7% hydrogen peroxide solution is placed in the drip funnel;

(b) The reaction flask is heated to a temperature of 55–60 °C. Then, without stopping stirring, hydrogen peroxide is gradually added from the drip funnel. At the same time, it is not allowed to increase the temperature of the reaction mixture above 65 °C;

(c) The solution is stirred for 5 h while maintaining the temperature of the reaction mixture at 60 °C. After the end of the process, the solution is cooled.

It should be noted that hydrogen peroxide in the end-product prevents its darkening during storage, so the neutralization of residual concentrations of hydrogen peroxide was not carried out, as it could lead to product's color problems in the future.

In the search for optimal synthesis conditions, particularary in selecting a solvent, three different approaches were tested. Low molecular weight alcohols are traditionally used as solvents in amine oxide production with isopropanol being the primary choice. Several patents suggest that using glycols, such as polyethylene glycol (PEG), can further enhance the performance characteristics of the resulting products [2,3]. Additionally, some literature sources propose that amine oxide can be synthesized without the use of a solvent [4].

The synthesis products were investigated by the method of IR-Fourier spectroscopy. Additionally, the initial synthesis reagent, oleylamidopropyldimethylamine, was analyzed using the same method. This was done to evaluate changes in various molecular fragments and functional groups compared to the synthesis product and to determine the approximate structure of the final product.. Before the analysis, isopropanol and water were removed from the synthesis products using a rotary evaporator.

3. Results and Discussion

Carrying out the synthesis without a solvent, or using PEG as a solvent, is challenging under laboratory conditions. The gradual addition of hydrogen peroxide to the reaction mass caused excessive foam formation, reducing the peroxide's contact with the entire mixture and hindering effective mixing. Therefore, isopropanol remains the most suitable solvent for laboratory synthesis of amine oxide, despite its potential to degrade the surfactant's performance characteristics later on.

The results of IR-Fourier spectroscopy are presented in the form of IR spectra in various wave number regions in Figures 2 and 3.

Based on the wave number range of 1800–650 cm^{-1} IR spectra shown in Figure 2, it can be seen that because of the synthesis of OADAO, the contribution of the C=O bond of the amide group decreased due to a decrease in the peak intensity of 1644–1650 cm^{-1}.

As for peaks 968 and 929 cm^{-1}, according to the literature data, they can be attributed to the N→O bond [5,6]. It is possible to assume the presence of more than one synthesis

product. The lower intensity of peaks 968 and 929 cm^{-1} compared to the literature data may indicate a low conversion or selectivity of the chemical reaction.

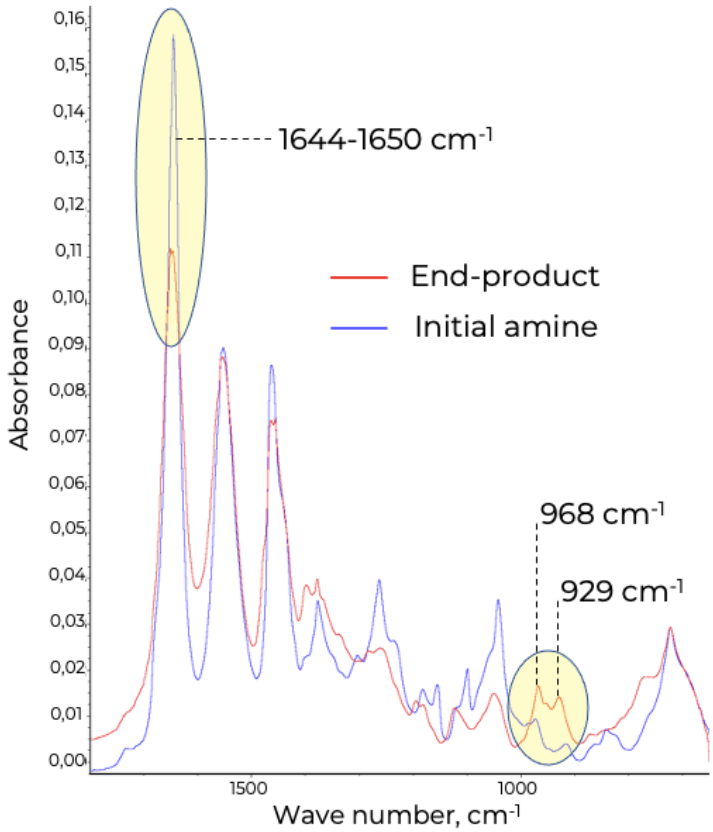

Figure 2. IR spectra (1800–650 cm^{-1}) of the initial reagent and the end-product (after distilling alcohol and water on a rotary evaporator).

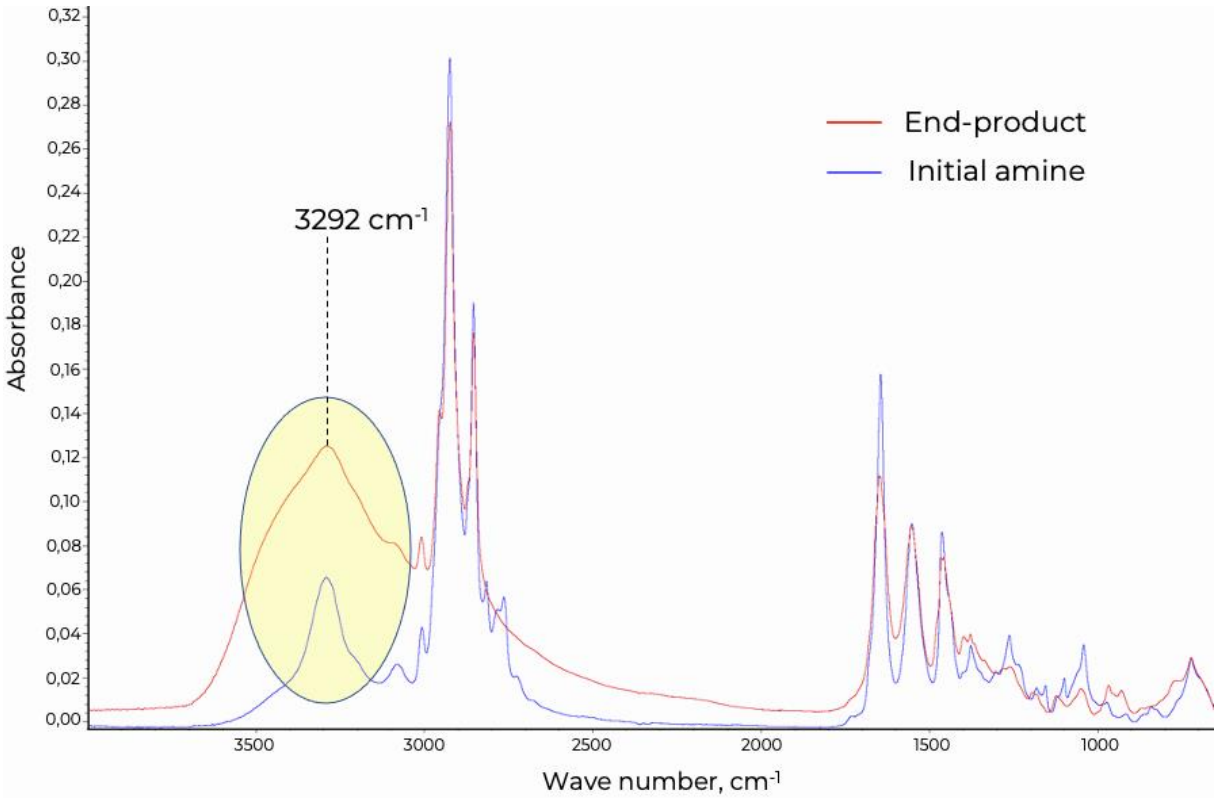

Figure 3. IR spectra (4000–650 cm^{-1}) of the initial reagent and the end-product (after distilling alcohol and water on a rotary evaporator).

The expansion of the wave number range of the obtained IR spectra to 4000 cm^{-1} allows us to conclude that the peak intensity of 3292 cm^{-1} has increased significantly in the synthesis products. This may be due to either an increase in the contribution of the N–H bond in amines, or the formation of a hydroxyl group with an O–H bond due to second order reactions.

Further analysis was carried out using the method of potentiometric titration in order to determine the quantitative content of OADAO in the end-products [7]. As a result of titration and calculation, it was determined that the concentration of OADAO is approximately 37% by weight, which is quite consistent with the literature data.

4. Conclusions

Based on the obtained titration results the conversion of the initial amine is approximately 90%. This is slightly lower than the conversion values presented in the literature, which, in turn, is consistent with the results of IR-Fourier spectroscopy.

Positive results were obtained in the synthesis of oleylamidopropyldimethylamine oxide. In the future, given optimal performance and favorable economic indicators, OADAO has the potential to become a highly successful viscoelastic surfactant in the oil and gas industry. For example, as a hydraulic fracturing fluid, which may be able to displace the polymer compositions of this technology.

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