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SYNTHESIS AND PHYSICOCHEMICAL CHARACTERIZATION OF FATTY ACID DIETHANOLAMIDES BASED ON VEGETABLE OILS: PROCESS KINETICS AND ANALYTICAL CONTROL

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diethanolamides, vegetable oils, amidation, gas chromatography, potentiometric titration, reaction kinetics, surfactants, activation energy, selectivity, synthesis

Abstract. *The paper presents the results of a comprehensive study of fatty acid diethanolamides (FADEA) synthesis from three types of vegetable raw materials: coconut, palm kernel, and sunflower oils of RBD grade. The fatty acid composition of the starting oils and the resulting amidation products were determined by gas chromatography on a Chromatek-Crystal 5000.2 analytical system. The coconut and palm kernel oils are characterized by a high content of lauric (47.8–50.8%) and myristic (15.8–18.0%) acids. It ensures the high foaming properties of the corresponding FADEA. Sunflower oil contains predominantly oleic and linoleic acids (88.4%), providing thickening and emulsifying characteristics. The synthesis of FADEA was conducted at a temperature of 100–125 °C under a nitrogen atmosphere using NaOH catalyst (0.1–0.5 wt.%). Kinetic studies showed 3–4 hours as an optimal reaction time. Further the concentration of free diethanolamine (DEA) stabilizes. We determine the free DEA content by two methods: potentiometric titration and gas-liquid chromatography (GC). The GC method for monitoring the DEA content is the most effective one. The obtained FADEA samples meet consumer requirements: free DEA content 4.6–5.5%, acid value 1.50–2.82 mg KOH/g, Gardner color scale 3–4. The values of the apparent activation energy of the amidation reactions for the studied oils (64–71 kJ/mol) and the selectivity of formation of the target diethanolamides (93.8–96.1% at 100 °C) were calculated.*

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Introduction

The modern chemical industry is transforming by tightening of the environmental standards [1] and growing consumer demand for biodegradable products [2, 3]. In the surfactant sector, there is a trend toward replacing traditional synthetic components with counterparts derived from renewable raw materials [4–6]. Fatty acid diethanolamides (FADEA) represent a promising class of nonionic surfactants obtained by the condensation of fatty acids (FA) of vegetable oils with diethanolamine [7–9].

These compounds possess a set of valuable properties: high foaming ability, thickening effect, emulsifying activity, and a mild effect on the skin [10–12]. FADEA demonstrate a high rate of biodegradation (≥ 90% over 28 days according to OECD 301B). Therefore, they are more effective ones in terms of the environmental safety [12, 13].

A considerable number of studies have been devoted to the synthesis of fatty acid amides of vegetable oils. Thus, Karpeeva I.E. et al. [14] studied the amidation of sunflower oil, demonstrating the possibility of obtaining products with high foaming characteristics at a temperature of 110–130 °C. Protopopov A.V. et al. [15] optimized the reaction conditions involving palm oil; however, the kinetic parameters of the process were not considered in their work. Lutfullina G.G. et al. [8, 10] studied the synthesis of nonionic surfactants based on corn oil, focusing on the rheological properties of the final compositions. Works [16–21] are devoted to the use of FADEA as corrosion inhibitors and antimicrobial additives. However, the issues of kinetic modeling and the influence of the temperature regime on the selectivity of the process are covered in them only fragmentarily.

Thus, optimization of the technology for producing FADEA to improve a product quality is an urgent task. Therefore, the aim of this work is to develop a procedure for the synthesis of FADEA from various vegetable oils, study the kinetics of the amidation process, and search for analytical methods for monitoring the free DEA content.

As a result, a comparative kinetic analysis of the synthesis of FADEA from three vegetable oils (coconut, palm kernel, and sunflower) for the first time was conducted under unified experimental conditions. Two analytical methods (potentiometry and GC) were proposed for monitoring free DEA, considering the influence of by-product amino esters on the analysis result. The optimal temperature regime was tested to minimize the formation of by-products without a significant decrease in the process rate.

Experimental part

For the synthesis, vegetable oils of RBD grade (refined, bleached, and deodorized) from the manufacturer RusAgro (Russia) were used: coconut, palm kernel, and pressed (sunflower) oils; diethanolamine (DEA) of 'pure' grade with a main substance content of ≥ 99.0% (GOST 10626-76). The catalyst was sodium hydroxide (NaOH) (chemically pure) grade (GOST 4328-77). Gaseous nitrogen of special purity (GOST 9293-74) was used.

The fatty acid composition of the vegetable oils was determined by gas chromatography on a Chromatek-Crystal 5000.2 system with an HP-5MS capillary column (30 m × 0.25 mm). Conditions: injector temperature 270 °C; column program: 150 °C (2 min) → 10 °C/min →

270 °C (20 min); FID detector, 300 °C; carrier gas – helium, 1.0 mL/min. Samples of the oils and diethanolamides were preliminarily methylated with a methanolic HCl solution (5%) at 70 °C for 2 hours to obtain fatty acid methyl esters (FAME). Peak identification was conducted by the retention time of standard samples of individual fatty acid methyl esters.

Determination of free DEA in the synthesized diethanolamide samples. Potentiometric titration: a weighed portion of 1.5–2.0 g was dissolved in 30 cm^3 of isopropanol, 20 cm^3 of water indicators phenolphthalein and bromocresol green were added; the solution was titrated with 0.1 N HCl with potential recording on an EV-74 ion meter. The mass fraction of free DEA (X, %) was calculated using the formula:

$$X = \frac{V * k * 0.0105}{m} * 100\%$$

where V is the volume of titrant, cm3; ^{k} is the correction factor; m is the mass of the weighed portion, g.

The limits of relative error of the potentiometric titration method were ±3.5% (at a confidence level of 0.95).

Gas chromatographic analysis of the synthesized diethanolamides was performed on the same chromatograph using an internal standard (1,4-butanediol). The analysis was conducted for the content of free diethanolamine and glycerol in the synthesized product. Samples were dissolved in isopropanol (20 mg/mL), and 1 μL was injected. Calibration was performed using a series of standard solutions with a DEA concentration of 0.1–2.0 mg/cm^3.

The limits of relative error of the GC method were ±2.1% (at a confidence level of 0.95).

The acid value of the synthesized diethanolamides was determined by potentiometric titration with a 0.1 N KOH solution in an isopropanol/water mixture (3:1) in accordance with GOST 25794.1-83.

The apparent activation energy (E_a) was calculated using the Arrhenius equation:

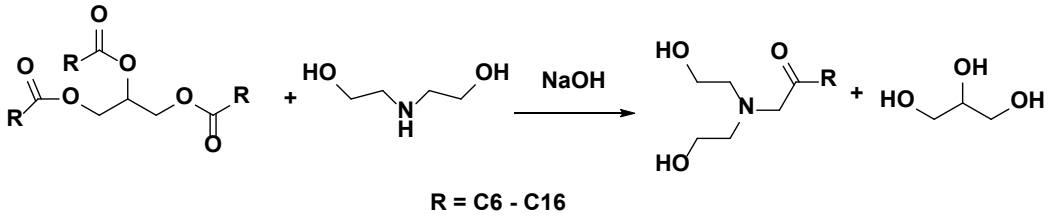
$$ln\frac{k_2}{k_1} = \frac{E_a}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right),$$

where k_1, k_2 are the rate constants at temperatures T_1 and T_2, R is the universal gas constant (8.314 J/mol·K).

Synthesis of FADEA. A 200 g portion of vegetable oil and the catalyst NaOH (0.1–0.5 wt.% based on the oil mass) were placed into a 500 mL three-necked flask equipped with a mechanical stirrer, a thermometer, and a reflux condenser. The flask was purged with nitrogen, the mixture was heated to a temperature of 100–125 °C, and a calculated amount of diethanolamine with an excess of 1–3% relative to the stoichiometry was added. The reaction time was counted from the moment the DEA addition was completed. Upon completion of the reaction, the mixture was cooled to 80 °C. The product was not isolated in individual form, since the commercial form is a liquid concentrate. The yield of the target product was 88–92% calculated on the oil.

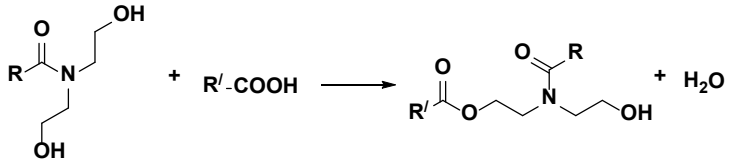
Results and discussion

The amidation reaction of oils can be represented by Scheme 1:



Scheme 1

However, side reactions may occur: the formation of amino esters (esterification) (Scheme 2) and thermal degradation (at T > 120 °C) of the unsaturated fragments of linoleic/oleic acids with the formation of colored polymerization products [12, 18].



Scheme 2

The substituents (R) vary depending on the type of oil. The fatty acid analysis of the starting raw materials was conducted by gas-liquid chromatography. The analysis revealed significant differences in the composition of the oils (Table 1).

Table 1. Fatty acid composition of coconut, palm kernel, and pressed oil

Component	Palm kernel oil, %	Coconut oil, %	Pressed oil, %
Caproic acid	0.204	0.514	-
Caprylic acid	4.101	7.820	-
Capric acid	3.808	6.243	0.065
Lauric acid	50.775	47.846	0.830
Myristic acid	15.753	18.006	0.077
Palmitic acid	8.818	9.498	7.145
Linoleic acid	0.210	-	88.355*
Oleic acid	14.566	7.575	
Stearic acid	1.766	2.499	3.528

*Total content of linoleic and stearic acids

Based on the obtained results, the following conclusions can be drawn:

- the fatty acid composition of palm kernel and coconut oils is similar; the main components are the saturated lauric and myristic acids. It might be a prerequisite for using these oils as raw materials for the production of highly foaming detergent additives;
- the fatty acid composition of pressed oil consists predominantly of C18 unsaturated acids (oleic and linoleic). It determines the use of FADEA from this oil as emulsifiers and thickeners.

The results of the analysis of the synthesized diethanolamides are presented in Table 2.

Table 2. Quality of the synthesized diethanolamides

Type of diethanolamide	Acid value, mg KOH/g	Diethanolamine content, %
Coconut diethanolamide	2.73	5.5
Pressed diethanolamide	1.50	4.6
Palm kernel diethanolamide	2.82	5.4

Chromatographic analysis of the fatty acid methyl esters obtained from the synthesized products showed the fatty acid profile corresponds to the starting raw material (Table 3).

Table 3. Fatty acid composition of the diethanolamides

Component	Palm kernel oil, %	Coconut oil, %	Pressed oil, %
Caproic acid	0.439	1.066	
Caprylic acid	4.622	10.389	
Capric acid	3.427	6.756	1.517
Lauric acid	45.715	47.572	
Myristic acid	14.235	16.390	0.263
Palmitic acid	8.077	8.304	6.035
Caproic acid	0.439	1.066	54.563*
Caprylic acid	4.622	10.389	
Capric acid	3.427	6.756	1.517
Lauric acid	45.715	47.572	
Myristic acid	14.235	16.390	0.263
Palmitic acid	8.077	8.304	6.035

*Total content of linoleic and stearic acids

To study the reaction kinetics, experimental syntheses of coconut diethanolamide were conducted at temperatures of 125 °C and 100 °C.

In the course of the experiment with the synthesis parameters (temperature 125 °C, DEA excess 3%, catalyst 0.3%), the change in DEA concentration in the reaction medium over time was studied. After 3–4 hours of reaction, the concentration of free DEA remains practically unchanged for coconut oil (Fig. 1).

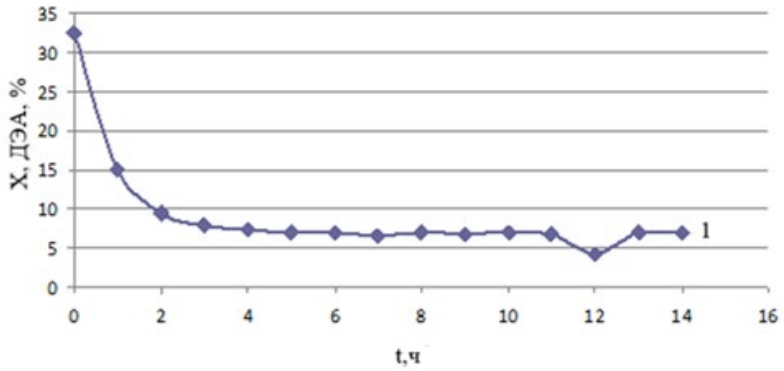


Fig. 1. Change in DEA concentration in the reaction mixture over time: 1 – the concentration of free DEA remains practically unchanged for coconut oil

The amidation reaction in the studied conversion range (0–80%) is described by a first-order kinetic equation with respect to the triglyceride concentration. The equation has the following form:

$$-\frac{d[TG]}{dt} = k_{\text{app}}[TG],$$

where $[TG]$ is the concentration of triglycerides.

Table 4 presents the calculated values of the apparent activation energy and the selectivity of diethanolamide formation for the studied oils. The selectivity was determined as the ratio of the amount of DEA that reacted to form an amide bond to the total amount of reacted DEA, calculating the fraction of side amino ester formation from the difference between the results of potentiometry and GC.

Table 4. Kinetic parameters and selectivity of FADEA synthesis

Oil	Selectivity for FADEA, %		E_a, kJ/mol*
	100 °C	125 °C	
Coconut	94.2	89.1	68
Palm cernel	93.8	88.5	71
Sunflower	96.1	91.3	64

*Calculated using the Arrhenius equation on the basis of the rate constants at two temperatures

A decrease in temperature from 125 °C to 100 °C decreases the rate constant by only a factor of 1.1–1.2. It corresponds to the calculated E_a values in the range of 64–71 kJ/mol. At the same time, the selectivity of formation of the target diethanolamides increases by 4–6%. It is explained by the suppression of the competing esterification reaction with the formation of amino esters.

In the analysis of coconut diethanolamide, the residual DEA content determined by GC is lower than that according to potentiometric titration data. The formation of side amino esters increases the titration results. At 125 °C, the fraction of these compounds increases. It raises the discrepancy between these methods, namely: the titration method shows the sum of all bases, while the GC method shows only free DEA. Therefore, the gas chromatographic method is the most preferable for the selective determination of specifically free DEA.

During the synthesis of FADEA from coconut oil at 100 °C, the reaction was studied and the results of potentiometric and GC analyses were compared (Fig. 2).

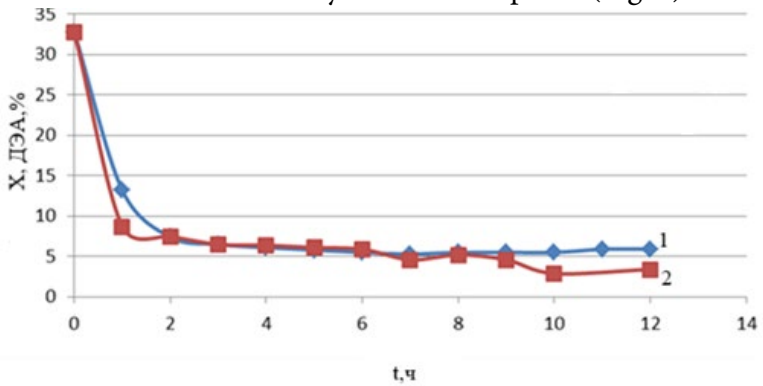


Fig. 2. Change in DEA concentration in the reaction mixture over time during the synthesis of coconut diethanolamide (100 °C, DEA excess 3%): 1 – potentiometric titration method; 2 – gas-liquid chromatography method.

According to Figure 2, the reaction is conducted at 100 °C, the results of analysis by potentiometric titration and GC practically coincide. At 100 °C, the discrepancy between the methods does not exceed 0.5%. It confirms the suppression of the side esterification reaction.

In addition, titration of the product obtained at 100 °C gives a more pronounced pH jump and, as a consequence, a more accurate determination of the titration endpoint. Figure 3 presents Examples of titration curves.

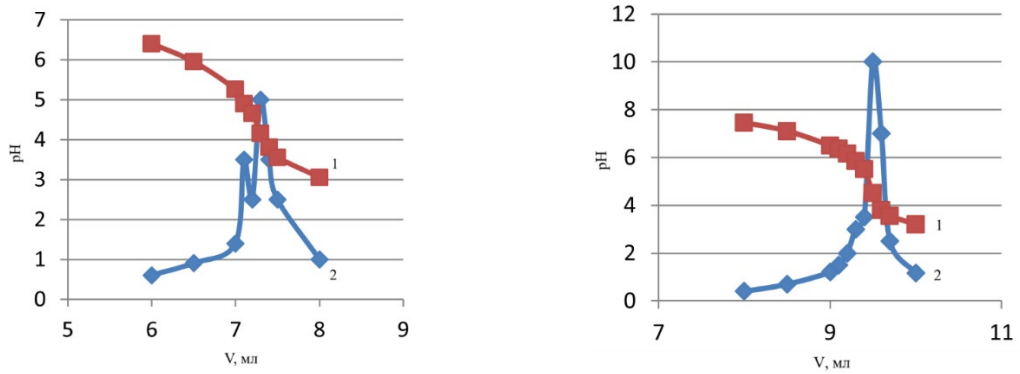


Fig. 3. Typical examples of titration curves of alkanolamide samples obtained at 125 °C (left) and 100 °C (right): 1 – titration curve (dependence of pH on titrant volume V); 2 – first derivative curve (dependence of ΔpH/ΔV on titrant volume V)

The difference in the initial pH values is due to the different content of basic components (free amine and amino esters) in the samples obtained at different temperatures. At 100 °C, the medium is more alkaline due to the absence of acidic by-products of the esterification reaction. Based on the obtained data, synthesis at a temperature of 100 °C with a process time of 4 h appears to be more preferable.

Conclusions

1. The procedure for the synthesis of fatty acid diethanolamides from coconut, palm kernel, and sunflower oils has been optimized. The yields of the target products are 88–92% at a temperature of 100 °C and a process duration is 3–4 h.

2. Based on the analysis of the fatty acid composition and comparison with the well-known structure–property relationships for alkanolamides [10–12], the functional properties of the obtained surfactants are determined by the fatty acid composition of the raw material: lauric and myristic acids (coconut and palm kernel oils) provide foaming. However, oleic and linoleic acids (sunflower oil) show thickening and emulsifying characteristics.

3. According to a kinetic analysis, an amidation reaction is described by a first-order equation with respect to the triglyceride concentration; a decrease in temperature from 125 °C to 100 °C reduces the process rate insignificantly (by a factor of 1.1–1.2) but increases the selectivity by 4–6% due to the suppression of amino ester formation.

4. Two methods of analytical control have been proposed: potentiometric titration and gas-liquid chromatography. For the quantitative determination of specifically free diethanolamine, the GC method is preferable. It excludes the influence of by-products on the result.

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Conflict of interest

The authors declare no conflict of interest in financial or any other sphere.

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