Production of Esters Based on Waste Vegetable Oils

M. A. Yelubay, Orazbekuly Yerbolat, G. S. Aitkaliyeva, and S. R. Massakbayeva

Abstract—The paper presents the results of a study of the main characteristics of sunflower and waste sunflower oil. It is revealed that one of the main methods of their processing is esterification and transesterification with the formation of carboxylic acid esters. It is shown that the use of the two-stage method of esterification with ethyl alcohol leads to an increase in the yield of esters with improved physical properties.

It was revealed that when applying the two-stage method in the presence of $\rm H_2SO_4$ and KOH catalysts, the yield of esters from the initial sunflower oil is 94%, whereas for used oil this indicator reaches 93%, which indicates the feasibility of using the used oils as raw materials to obtain based on it higher fatty alcohols. It has been experimentally proven that the ethanol: oil ratio of 9: 1, respectively, is most optimal for producing an ester. The esterification reaction was also shown to occur at room temperature, however, with an increase in temperature to 50 $^\circ$ C, after 60 minutes the yield of ester reached more than 70%, and at a temperature of 75 $^\circ$ C it reached its maximum.

Index Terms—Waste oil, sunflower oil, esterification, fatty alcohols.

I. INTRODUCTION

The production of surfactants plays an important role for a wide range of industrial and consumer products, including detergents, paints, paper products, pharmaceuticals and cosmetics.

Currently, surfactants are divided into ionic and non-ionic. Ionogenic anionic and cationic surfactants dissociate in water to form charged ions. Anionic surfactants have a detergent effect at high pH values of the medium (with a strongly alkaline reaction); cationic surfactants are successfully washed in low-alkaline and weakly acidic environments. Amphoteric show their detergency depending on the pH environment [1].

The work related to the preparation and study of surfactants based on natural products: sugars, sterols and fatty acids, which is caused by their easy Biodegradability, is of great interest.

The use of natural raw materials for the synthesis of surface-active substances due to their renewability, consisting of continuous environmental cycles. They are constantly produced by nature and, therefore, are in principle available and cheap for commercial use with a small risk of shortage. It is worth noting the reduction of toxicity in their use, less environmental impact due to the degradation of

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surfactants into their natural, smaller components (that is, hydrophobic and hydrophilic), it is assumed that they participate in natural ecological cycles without toxicological effects.

The amount of natural products is large, but only a limited number of them are suitable for conversion into surfactants. Some connections are too expensive for general use.

The purpose of this work was the use of plant materials and their waste for the production of components of non-ionic surface-active substances. In contrast to ionic, nonionic surfactants do not produce charged ions and are derivatives of polyglycols, for example, polyethylene glycol, polypropylene glycol, and their derivatives. Therefore, to obtain cheap enough raw materials for the production of nonionic surfactants, it is necessary to use vegetable oil or their waste forms.

It is known [2], [3] that vegetable oils for 94-96% consist of mixtures of triglycerides of higher fatty acids, and the rest are substances close to fats, free fatty acids, and other components.

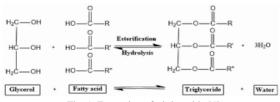


Fig. 1. Formation of triglyceride [4].

The production of fatty alcohols by direct hydrogenation of triglycerides is also possible, however, under the reaction conditions, glycerin is reduced to propylene glycol and propanol, which have no special commercial value, increasing the cost of the process due to the greater need for hydrogen and catalyst [4], [5]. Therefore, the direct hydrogenation of triglycerides does not find industrial application, and there is a need to modify vegetable oils to produce esters.

Fatty alcohols are surfactants widely used as emulsifiers, emollients and thickeners in alimentary and cosmetic industries [6]. Moreover, they can be substrates for the production of other surface-active materials, such as alkylamines and alkylsulfates.

Selective modification of these alcohols could allow to use them as a new resource for producing desired products that are valuable intermediates for the fine chemical, pharmaceutical [7] and agrochemical sectors. For instance, behenic acid ($C_{21}H_{43}COOH$) is used in cosmetics, hair conditioners and creams, due to its high wettability [8], and lignoceric acid ($C_{23}H_{47}COOH$) is used in pharmaceutical [9],]10] and health-care preparations [11] and as additives in foods [12].

Fatty alcohols are mainly produced through catalytic hydrogenation of fatty acids, methyl esters or wax esters.

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Oils and fats have important applications in the food and pharmaceutical industries [13]. They are composed of triglycerides of even numbered carbon fatty acids [14], which also are starting to be used as low cost renewable resources for the fatty alcohol production. Another promising source of higher alcohols are fatty acid esters obtained by the esterification of vegetable oils.

Taking into account all the above, it would be of much interest to find a stable and suitable raw materials for the production of fatty alcohols, as well as the best reaction conditions for high product yield.

II. MATERIALS AND METHODS

A. Study of the Main Characteristics of Vegetable Oils

In this paper, samples of sunflower oil and waste oil were studied by conventional methods to obtain a component of synthetic surfactants [15]-[19].

Density determination was carried out using an areometer according to GOST 3900. The essence of the method is to immerse the areometer in vegetable oil and take readings on the hydrometer scale.

Viscous studies were carried out according to GOST 33–2000 (ISO3104-94). The essence of the method consists in measuring, with a calibrated glass viscometer, the expiration time, in seconds, of a certain volume of the test liquid under the influence of gravity at a constant temperature. Kinematic viscosity is the product of the measured expiration time by a constant viscometer. Dynamic viscosity is equal to the product of kinematic viscosity and oil density.

Tests for determining the acid number of sunflower vegetable oil were carried out according to GOST R 52110-2003 «Vegetable oils. Methods for determining the acid number».

B. Modification of Vegetable Oils to Produce Carboxylic Acid Esters

One of the most popular in the literature methods of modification of vegetable oils to produce fatty alcohols is the reaction with the formation of new esters with improved physical properties - esterification and transesterification.

Basic catalysts (alkaline hydroxides, alcoholates, oxides, carbonates, anion exchange resins), acid catalysts (inorganic acids, p-toluene sulfonic acid, boron trifluoride, cation exchange resins) and enzymes (lipases) can be used as catalysts for transesterification. Currently, soluble catalysts are preferably used in the reaction mixture. They form a homogeneous mixture and provide high conversion rates and mild reaction conditions. The most used catalysts are sodium and potassium hydroxide, as well as sodium methylate, an alcoholic solution of which is mixed with vegetable oil. This method is known from the document AT-B 386 222 [20].

Vegetable oils and free fatty acids have good mutual solubility, but in the presence of free fatty acids, oil triglycerides cannot be processed into esters with an alkaline catalyst due to the formation of soaps [21], [22], and a significant disadvantage of the acid catalyst is the low reaction rate of transesterification of vegetable oils about 4000 times less [23], [24]. However, the esterification

reaction of free fatty acids with low molecular weight alcohols usually proceeds at a higher rate than the transesterification reaction [25]. Therefore, according to the works of the authors [26], a two – stage scheme was carried out: at the first stage, fatty acids were esterified with ethanol in acid catalysis (H_2SO_4) and at the second stage, oil with an alkaline catalyst (KOH) was transesterified according to the following scheme

The result of this reaction is a complex mixture consisting of ethyl esters - the main product of synthesis, glycerol, ethanol, and residues of unreacted catalyst.

The total reaction product was placed in a laboratory distillation apparatus. In this distillation apparatus, ethanol and water were first distilled from the reaction mixture at normal pressure.

Alkaline refining consisting of neutralization, settling, washing and drying of fat was used for purification of crude esters from unreacted fatty acids.

The chemistry of the process is to neutralize the free fatty acids of fat with aqueous solutions of sodium hydroxide (NaOH). Neutralization of acid with sodium alkali is carried out at a temperature of 50-80° C without stirring, after neutralization of acids, they are settled (for 30-60 minutes) in a sump. The settled ether precipitate is lowered into a separate receiver, dehydrated under vacuum for 10 hours.

III. RESULTS AND DISCUSSION

The experimental values of refractive index, density and dynamic viscosity, as well as the acid number of vegetable oils are presented in Table I.

TABLE I: PHYSICAL AND CHEMICAL PROPERTIES OF VEGETABLE OILS

| A sample of vegetable oil | • . | Dynamic viscosity, MPa*s | Refractive index | Acid number, mg Koh/g |
|---------------------------|-----|--------------------------|------------------|--------------------------|
| Source | 921 | MPa*s 56,8 | 1,471 | 0,4 |
| Waste | 925 | 59,5 | 1,484 | 1,1 |

According to the results of Table I, there is a change in the basic physical and chemical properties of sunflower oil as a result of its use: the density, dynamic viscosity and refractive index increases markedly.

For the studied sample of sunflower oil, the acid value of 0.4 is noted, which seriously increases as a result of its use to 1.1 mg KOH/g, which in turn is an indicator of the content in the oil of free fatty acids formed during cooking under the action of water vapor.

The IR spectrum of all vegetable oil samples (Fig. 2), which differs in its raw materials in the region of 700-3000 cm⁻¹, contains absorption bands at 2850 - 2900 cm⁻¹, characteristic of deformation oscillations of C-H bonds of methyl and methylene groups. Absorption bands in the range 1460 - 1370 cm⁻¹ due to CH₂ and CH₃ groups as a long

paraffin chains and alkyl substituents in the cycles.

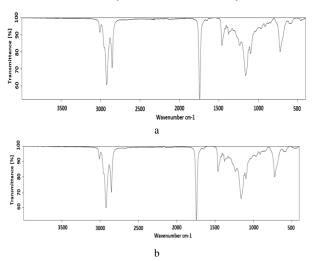


Fig. 2. IR spectra of initial (a) and spent sunflower (b) oil.

Intensive absorption bands in the region of 1000-1300 cm⁻¹ characterize the presence of unbranched paraffin chains in hydrocarbons. There is a peak of sufficiently high intensity in the region of 700 cm⁻¹, which is an analytical strip of vegetable oil [27].

With the presence of fluctuations of groups of carboxylic acids in the studied oils related absorption bands, caused by valent oscillations of C–O bonds (frequency 1750 cm $^{-1}$) with the deformation vibrations of OH - groups (frequency 2950–2800cm $^{-1}$), indicating a high content in the composition of oils fatty carboxylic acids.

Thus, the chemical composition, physical and physico-chemical parameters of the initial and spent sunflower vegetable oil were studied and the high prospects of using them with affordable and cheap raw materials for the production of fatty esters and alcohols were revealed.

Table II presents the results of the yield and main characteristics of carboxylic acid esters based on the initial and spent sunflower oil with ethanol using alkaline, acid catalysts and a two-stage method.

The results of Table II revealed that the same ratio of the components of the mixture in the case of a two-stage method achieved significantly better degree of transformation. There is also a high yield of carboxylic acid esters when used as a raw material of sunflower oil.

TABLE II: THE OUTPUT OF THE ESTER AND ITS PHYSICAL PROPERTIES

| Sunflower | Alcohol | Catalysts | Output | Physical pr | operties of |
|-----------|---------|--------------------------------|----------|-------------|-------------|
| oil | | | of | ether | |
| | | | ether, % | Viscosity, | Pour |
| | | | | cSt at | point, °C |
| | | | | 40 °C | |
| Source | Ethanol | H ₂ SO ₄ | 91 | 7,58 | -3 |
| Waste | Ethanol | H ₂ SO ₄ | 90 | 7,61 | -3 |
| | | | | | |
| source | Ethanol | КОН | 92 | 7,57 | -3 |
| Waste | Ethanol | КОН | 90 | 7,62 | -3 |
| source | Ethanol | two-stage | 94 | 7,51 | -3 |
| Waste | Ethanol | two-stage | 93 | 7,59 | -3 |

Table III presents the results of a study of the effect of the

quantitative ratio of components and reaction time on the yield of ester [28].

TABLE III: STUDY OF THE EFFECT OF THE RATIO OF COMPONENTS AND

REACTION TIME ON THE QUITBUT OF ESTERS

| REACTION TIME | BOILTING OCTION D | DILKO |
|-------------------------------|------------------------|----------------------------|
| The molar ratio of alcohol to | Reaction time, min | The yield of |
| oil | | ether,% |
| | | |
| 3:1 | 60 | 78 |
| | 120 | 79 |
| 6:1 | 60 | 84 |
| | 120 | 83 |
| 9:1 | 60 | 94 |
| | 120 | 91 |
| 12:1 | 60 | 88 |
| | 120 | 87 |
| The molar ratio of "spent | Reaction time, min | The yield of |
| alcohol - oil" | | ether,% |
| | | ctrici,70 |
| 3:1 | 60 | 74 |
| 3:1 | 60 120 | |
| 3:1 6:1 | | 74 |
| | 120 | 74 75 |
| | 120 60 | 74 75 81 |
| 6:1 | 120 60 120 | 74 75 81 81 |
| 6:1 | 120 60 120 60 | 74 75 81 81 93 |

According to the results of the table, it was found that with an increase in the amount of ethanol the yield of esters gradually increased, reaching a maximum at a ratio of 9: 1, and after reaching a ratio of 12: 1, a decrease in the yield of ester was observed. The optimal composition for producing the ester is a mixture of ethanol: oil in a ratio of 9: 1 and with a reaction time of 60 minutes. Similar results were obtained for spent sunflower oil.

In order to determine the optimal temperature for carrying out the transesterification reaction, the yield of the ester was studied as a function of temperatures from 25 to 75 $^{\circ}$ C and a reaction time of 60 to 120 minutes (Fig. 3, 4).

Thus, it was shown that the transesterification reaction also proceeds at room temperature, however, with an increase in temperature to $50\,^\circ$ C after 60 minutes, the yield of ester reaches more than 70%, and at a temperature of $75\,^\circ$ C it reaches its maximum. A similar situation was observed in the case of the use of used sunflower oil with a slight correction, which under similar conditions the degree of transesterification was lower.

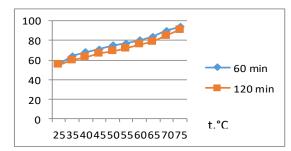


Fig. 3. Effect of temperature on the yield of ester based on the starting oil at a synthesis time of 60 and 120 minutes.

The IR spectrum of all vegetable oil samples (Fig. 2), which differs in its raw materials in the region of 700-3000 cm⁻¹, contains absorption bands at 2850 - 2900 cm⁻¹, characteristic of deformation oscillations of C-H bonds of methyl and methylene groups. Absorption bands in the range

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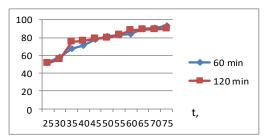


Fig. 4. Effect of temperature on the yield of ester based on waste oil at a synthesis time of 60 and 120 minutes.

For further synthesis of fatty alcohols on the basis of the esters obtained by us, a hydrogenation process of our ester of fatty acids was carried out in a column in the presence of hydrogen, obtained in a TRH-300E hydrogen generator. To obtain fatty alcohols, a hydrogenation reaction was carried out using a catalyst containing Cu-Cr oxide compounds.

The results for determining the optimal conditions for the release of fatty alcohols using various raw materials are presented in Table IV.

TABLE IV: THE OUTPUT OF FATTY ALCOHOLS DEPENDING ON THE TIME AND TEMPERATURE OF THE REACTION WITH OXIDE COMPOUNDS CU-CR AS

| | | A CATALYST | 0/ | |
|-----------------------|------|-------------------|-----------------------|--|
| Experience conditions | | Yield, % | | |
| Time, min | T,°C | ester based on SO | ester based on USO | |
| 60 | 150 | 25,8 | 23,9 | |
| 120 | 150 | 36,4 | 34,8 | |
| 180 | 150 | 58,1 | 56,6 | |
| 240 | 150 | 70,1 | 64,7 | |
| 60 | 180 | 32,3 | 31,1 | |
| 120 | 180 | 45,6 | 44,4 | |
| 180 | 180 | 67,8 | 65,1 | |
| 240 | 180 | 76,4 | 74,6 | |
| 60 | 200 | 47,7 | 45,6 | |
| 120 | 200 | 59,1 | 56,7 | |
| 180 | 200 | 69,4 | 68,1 | |
| 240 | 200 | 79,3 | 76,3 | |

According to the results of Table IV, it was revealed that the yield of fatty alcohols increases with increasing contact time and temperature.

The highest yield of fatty alcohols is observed when sunflower oil esters are used as raw materials at a temperature of 200 °C (contact time is 240 minutes) and reaches 79.3%.

Work to determine the best conditions for the hydrogenation reaction of carboxylic acid esters continues [29]-[31], but on the basis of the data obtained, it can be concluded that the spent oil can also serve as a raw material for the synthesis of fatty alcohols.

IV. CONCLUSION

As a result of the study, it was found that the presence of free fatty acids in the composition of the used oils determines the possibility of obtaining carboxylic acid esters based on them. The yield of fatty acid ethyl esters is increased in a two-stage scheme using acid and alkaline catalysis in the second stage in the first stage. The data on the influence of the reaction temperature, its duration, and the ratio of reaction

components on the yield of ester are presented. It was revealed that the most optimal results were obtained with a ratio of alcohol: oil components of 9: 1 at a temperature of 75 °C and an exposure time of 60 minutes. The production of fatty alcohol based on the obtained esters was implemented on a platinum catalyst, further studies on the selection of the most effective and cheap catalyst are ongoing.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

M. A. Yelubay and Orazbekuly Yerbolat conducted the research of Vegetable Oils' modification to Carboxylic Acid Esters; G. S. Aitkaliyeva analyzed the main characteristics of Vegetable Oils; S. R. Massakbayeva wrote the paper; all authors had approved the final version.

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