The role of catalysts in fat transesterification technology

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Abstract. Methods of mathematical and statistical treatment of experimental results in technology of interesterification of oils and fats have been used. The optimal technological modes and parameters that ensure reduction of amount of expensive oil and the cost of the ongoing technological stage have been established. New data in the direction of interesterification of oils and fats have been obtained. The main reasons providing quality improvement and ensuring safety of interesterified fats have been established.

1. Introduction

Lately, in experimental studies, much attention is paid to the use of methods of mathematical and statistical processing of results [1-3], which can significantly reduce the necessary costs for conducting experiments. The technology of interesterification of oils and fats includes many technological stages (figure 1), each of which requires its own experimental study. Therefore, the use of methods of mathematical and statistical processing in the technology of interesterification of oils and fats seems to be a topical issue. In this direction, the optimization of technological modes and parameters, which allow obtaining edible fats of improved quality and safety [4, 5] are of particular importance. The purpose is using methods of mathematical and statistical treatment of experimental results in the technology of interesterification of oils and fats for the production of target edible fats for margarine, bakery and confectionery products.

Objects of research are the technological modes of interesterification of oils and fats, indicators of the quality of the products obtained, factors affecting the technological and qualitative characteristics of edible fats, the norms and costs of parameters in the interesterification technology [6, 7].

2. Methods and materials

For mathematical and statistical treatment of experimental results of interesterification technology used modern methods [8,9], special attention is paid to the use of methods of us smaller squares [10,11] and algorithms of processes [12,13].

The structure chart of interesterification technology of fats and oils is shown in figure 1.

The essence of the main technological parameters of the process is as follows:

- Weight dosing and mixing of the original oils and fats in accordance with the recipe at 60-80 °C.
- Alkaline neutralization of a mixture of fats to acid number of not more than 0.20 mg KOH/g.
• Deep drying of mixture of fats at 135-140 °C and a residual pressure of no more than 4 kPa (30 mm Hg) to a moisture content of no more than 0.015%.
• Interesterification of a mixture of oils and fats at 80-90 °C in the presence of 0.10-0.15% catalyst.
• Deactivation of the catalyst with dilute saline solution (2-10% by weight of fat).
• Rinsing the interesterified fat with hot water (10% of the fat mass).
• Drying of interesterified fat at 90 °C and a residual pressure of more than 6.6 kPa (50 mm Hg).

Distinctive feature of the recommended technology is “deep” drying of the fat mixture in a continuous vacuum drying apparatus, as well as the supply of the interesterification catalyst in the form of a stabilized oil suspension.

The study and quality control of oils and fats was carried out by limitation and a small sample, that is, by a small number of observations or measurements, the number of which does not exceed 30, and often made 3-4 replicates.

The determination of the average measurement error was carried out according to the following sequence:

The arithmetic mean of the obtained experimental data (X) was determined by the formula:

$$\bar{X} = \frac{1}{n} \sum_{i=1}^{n} x_i$$  \hspace{1cm} (1)

Where $x_i$ is result of the 1st observation; $n$ is number of observations.

The standard deviation (S) of the actual data from their arithmetic mean was calculated using the formula:
\[ S = \sqrt{\frac{\sum d_i^2}{n-1}}, \quad n \ll 30 \]  
(2)

Where \( d_i \) is deviation of the actual value of the 1st observation from the arithmetic mean; \( n \) is the number of observations.

Standard deviation shows the quantitative variability of the investigated property relative to its arithmetic mean.

Coefficient of variation (\( V \)) or the degree of fluctuation of the studied property from its average value in percent was calculated using the formula:

\[ V = \frac{S}{\bar{x}} \cdot 100 \]  
(3)

Where \( S \) is standard deviation; \( \bar{x} \) is the arithmetic mean of the parameter under study.

Coefficient of variation characterizes the degree of homogeneity of the material under study.

Average error (\( m \)) of the measurements made was determined taking into account the normalized deviation from their average value according to the formula:

\[ m = \frac{t \cdot S}{\sqrt{n-1}} \]  
(4)

Where \( t \) is standardized deviation, depending on the value of the confidence level and the number of measurements; \( S \) is root mean square change; \( n \) is the number of observations.

Normalized deviation (\( t \)) represents the deviation of one or another measurement result (\( X \)) from their mean value \( \bar{x} \), referred to the value of the standard deviation \( S_x \):

\[ t = \frac{(x_1 - \bar{x})}{S_x} \]  
(5)

This indicator allows establishing how many \( S_x \) individual measurements deviate from their mean.

The (\( t \)) values fluctuate within \( +3S \). The level of confidence (\( q \)) of the appearance of reliable measurement results depends on the value (\( t \)).

With a value of (\( t \)) equal to 1.0, 1.5, 2.0 and 2.5, the confidence probability (\( q \)) was, respectively 68.3, 86.6, 95.5 and 98.8 %.

Value of the mean error (\( m \)) shows in what range the arithmetic mean of the sample mean (\( X \)) fluctuates depending on unaccounted for random reasons. The true value of the general average (\( M_x \)) will be \( X \pm m \).

The reliability of the difference between the results of studies with a small number of observations was determined by the normalized deviation or the Student-Fisher criteria (\( t_p \)) according to the formula:

\[ t_p = \frac{\bar{x}_1 - \bar{x}_2}{\sqrt{m_1^2 + m_2^2}} \]  
(6)

Where \( x_1 \) and \( x_2 \) are arithmetic mean values of the received data, \( m_1 \) and \( m_2 \) are the average measurement errors.

If the difference in indicators is 2.5-3.0 or at least 2.0 times greater than the sum of their average errors, then with the probability determined according to table 1, it can be argued that the difference in the magnitude of indicators is not accidental but depends on some reason.

For the efficiency of the technology for obtaining interesterified fats from a mixture of vegetable oils and fats, it is necessary to use modern methods for optimizing technological processes based on the use of their mathematical models.
3. Results and discussion
Planning an experiment for low-temperature interesterification of a mixture of vegetable oils and fats allows identifying its optimal technological parameters by performing even minimal number of experiments.

Based on a full factorial experiment (FFE) – $2^{k-1}-1$ with a fractional (1/2) replica, studies were carried out to select the optimal parameters. The following variables are accepted as variable factors:

- $X_1$ is the temperature of the interesterification process, $^\circ$C.
- $X_2$ is the interesterification time, min.
- $X_3$ is the amount of fat additive, %.

Table 1. Variation levels of variable factors $X_1-X_3$.

| Variable parameter name         | Designation | Measurement unit | Variation levels |
|---------------------------------|-------------|------------------|------------------|
| Interesterification             | $X_1$       | °C               | 40 (-) 60 (+)    |
| Interesterification time        | $X_2$       | min              | 15 (-) 30 (+)    |
| Consumption of solid fat additive | $X_3$   | %                | 40 (-) 60 (+)    |

The following are taken as optimization criteria:

- $U_1$ is time of transparency of the interesterified fat at 0 °C, min.
- $U_2$ - iodine number of interesterified fat, % $J_2$.

In order to obtain informative data, information was accumulated during the normal operation of the oil and fat interesterification process line. Taking into account the specific features of this line and the duration of laboratory analyzes were the basis for choosing the interval between measurements, equal to 30 minutes. The processing of statistical data on a computer was carried out according to the algorithm shown in figure 2. The results are presented in table 2

![Figure 2. Algorithm for calculating mathematical models of the interesterification process of oils and fats.](image-url)
Table 2. Mathematical description of the process of interesterification of oils and fats and assessment of the adequacy.

| Technological line name | Mathematical description of the process | Calculated values of the criteria |
|-------------------------|----------------------------------------|----------------------------------|
|                         |                                        | Cochran | Student | Fischer |
| Interesterification of   | $Y_1 = 6.3 - 1.7 X_1 + 0.475 X_2 + 0.525 X_3$ | 0.221   | 0.434   | 0.717   |
| oils and fats            | $Y_2 = 113.49 - 2.94 X_1 + 0.99 X_2 + 1.36$   | 0.325   | 0.716   | 2.001   |
|                         | $X_3 = 1.19 X_1 X_2 X_3$                   |         |         |         |

Data in table 2 show that the mathematical descriptions of the output parameters $U_1$ and $U_2$, according to the values of the Cochran, Student and Fisher criteria, are adequate to the process under consideration.

Implementation of regression equations $U_1$ and $U_2$ allows predicting the state of the interesterification process of oils and fats of the resulting product and its quality. Moreover, the coefficients in the equations $U_1$ and $U_2$ allow determining the degree of influence of each factor and their interaction on the main indicators of this process.

4. Conclusion

Thereby, obtained regression mathematical models adequately describe the real process of interesterification of oils and fats and can be used to intensify its individual stages.

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