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Application Of Mathematical Processing Methods For Optimizing The Hydrogenating Properties Of Fat Hydrogenation Catalysts

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ABSTRACT

Dietary fats are an important food item. According to physiological norms, the recommended fat content in the human diet is 30.0-33.0 % of the total energy value of food, [1,2].

Fats are necessary not only as a reserve substance and a source of energy, but also as suppliers of physiologically active compounds - essential fatty acids, phospholipids, vitamins involved in the synthesis of cell membranes and other body tissues [3,4].

KEYWORDS

Randomized matrix , parallel , Calculated dispersion , catalyst,

INTRODUCTION

Solving the problem of the quality and safety of food fats and their processed products is one of the priority directions in the implementation of the concept of the state policy in the field of healthy nutrition of the population of the Republic,[5].

Improving the quality and ensuring food safety of high-solid fats can be carried out by changing the triglyceride composition of oils and fats in various ways to modify them. For the production of edible fats, methods of catalytic hydrogenation using various types of catalytic systems are used

Currently, many catalysts have been proposed in hydrogenation technology, [6,8].However, the problem of optimizing the composition of the catalysts used and their hydrogenating properties has not yet been solved. In this regard, the solution of the problems of optimizing the hydrogenating properties of stationary catalysts is of scientific, technical and practical interest.

The purpose of the work optimization of the hydrogenating properties of stationary nickel-copper-aluminum alloy catalysts promoted by some metals of the periodic system is performed.

The results of experimental studies we decided to find a highly efficient stationary alloy promoted nickel-copper-aluminum catalyst in order to recommend it for industrial testing and implementation for the production of food from cottonseed oil. Along with this, the task was to determine the degree of influence of the promoter on the hydrogenating properties (activity, selectivity, isomerizing ability) of a stationary catalyst, using modern methods of experimental planning [9, 10].

We preferred to choose the activity of the catalyst, the selectivity and the isomerizing ability of the process, which would ensure the production with sufficient productivity, the necessary melting point and hardness.

The response response functions or the set of factors that determine the activity (salaams iodine number), selectivity, and isomerizing ability of the catalyst were as follows:

X1 – palladium content in the alloy, % by weight;

X2 – germanium content in the alloy, % by weight

X3 – the content of rhodium in the alloy, % by weight;

X4 – tin content in the alloy, % by weight.

The levels of factors and the interval of their variation based on laboratory studies are presented in Table 1.

Table 1

Levels of factors and the interval of their variation

Factors	Main level x_0	Unit of variation	Lower level	Upper level
X_1	0,50	0,10	0,40	0,60
X_2	1,00	0,50	0,50	1,50
X_3	0,50	0,30	0,20	0,80
X_4	2,00	0,50	1,50	2,50

To plan the experiments in order to find the optimal ratios of the promoting metals in the alloy, 1/2 replica of the full factor experiment was used 2^{4-1}.

The studies were carried out according to a randomized matrix on two parallel samples of the catalyst. The procedure for the implementation of experiments for the preparation of alloys and the results of hydrogenation on the obtained catalysts according to the iodine number (activity) of hydrogenate, selectivity and isomerizing ability of hydrogenation are presented in Table 2.

Table 2.

Randomized matrix for the preparation of catalysts

№ variants of the composition of the catalyst	X_0	X_1	X_2	X_3	X_4	$X_1 \cdot X_2 = X_3 \cdot X_4$	$X_1 \cdot X_3 = X_2 \cdot X_4$	$X_2 \cdot X_3 = X_1 \cdot X_4$	Y – by the activity of the catalyst (Y. h.)				Y – by the selectivity of the catalyst,%				Y-by isomerizing ability,%			
									A_1	A_2	$A_{ср}$	$A_{выч}$	C_1	C_2	$C_{ср}$	$C_{выч}$	$И_1$	$И_2$	$\overline{И}_{ср}$	$И_{выч}$
1	+	+	+	-	-	+	-	-	47,3	47,4	47,3	47,32	76,5	77,3	76,9	76,85	36,1	35,3	35,7	35,86
2	+	-	-	-	-	+	+	+	44,2	44,3	44,3	44,32	89,7	89,5	89,6	89,61	29,3	29,6	29,4	29,70
3	+	+	-	-	+	-	-	+	44,9	45,0	44,9	44,90	78,6	78,9	78,7	78,73	44,1	44,4	44,2	43,86
4	+	-	+	-	+	-	+	-	57,0	57,2	57,1	57,10	91,1	92,0	91,5	91,53	40,5	40,2	40,3	40,10
5	+	+	+	+	+	+	+	+	61,4	61,7	61,5	61,52	92,8	92,5	92,6	92,65	47,6	47,9	47,7	47,98
6	+	-	-	+	+	+	-	-	58,7	58,5	58,6	58,60	92,3	92,0	92,1	92,04	45,9	46,2	46,0	46,14
7	+	+	-	+	-	-	+	-	60,1	60,0	60,0	60,02	93,0	93,3	93,2	93,23	37,3	37,5	37,4	37,18
8	+	-	+	+	-	-	-	+	59,8	59,7	59,8	59,82	92,7	92,2	92,4	92,41	30,6	30,8	30,7	30,38
V_A	54,2	-0,76	+2,24	+5,79	+1,33	-1,26	+1,54	-1,56				54,2								
V_C	88,4	-3,01	-0,02	+4,20	+0,35	-0,57	+3,35	-0,05							88,4					
$V_И$	38,9	+2,32	-0,32	+1,52	+5,62	+1,02	-0,16	-0,92											38,9	

A – catalyst activity, Δ Y. h.;

C – selectivity of the catalyst, %;

I-isomerizing ability of the catalyst, % of trans-isomers of fatty acids.

The obtained results allowed us to find the regression equation for both the activity and selectivity and isomerizing ability of hydrogenation of catalysts:

$$У = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_{12}X_1X_2 +$$

$$+ B_{13}X_1X_3 + B_{14}X_1X_4 + B_{23}X_2X_3 + \dots \quad (1)$$

: $B_0, B_1, \dots B_4$ – regression coefficients that are calculated using the formula:

$$B_u = \frac{\sum_{u=1}^{N} Xju\bar{Y}u}{N} \quad (2)$$

where: u – experience (catalyst) number);

j– factor number;

N – number of catalysts

Y is the response function (in our case, the activity, selectivity, and isomerizing ability of the catalyst).

The coefficients of the equation were calculated from the average results of the preparation of eight catalysts, using the formula (2). For example, from the activity of the catalyst:

$$B_{A_I} = \frac{\sum_{u=1}^{N} X_{1u} A}{N} = \frac{47{,}3-44{,}3+44{,}9-57{,}1+61{,}5-58{,}6+60{,}0-59{,}8}{8} = -0{,}76 \quad (3)$$

Similarly, the remaining regression coefficients were calculated for both the activity and the selectivity and isomerizing ability of the catalyst. Substituting the values of "Vi" in the regression equation (64), we obtained the calculated values of the activity, selectivity, and isomerizing ability of the catalysts.

For statistical analysis of the significance of the coefficients of equation (64) and its adequacy, a line-by-line estimate of the reproducibility of a single measurement result in each experiment was determined by the formula:

$$S^2(Y_{ju}) = \frac{\sum_{u=1}^{N}(Y ju-\bar{Y}u)^{\ 2}}{n-I} \qquad (4)$$

where: $\bar{y}\ _u$ – the arithmetic mean value of the optimization parameter; n is the number of parallel experiments in the preparation of the catalyst (in our case, n = 2)

The calculated values of the dispersions for the obtained samples of catalysts are given in Table.3. The

Cochran criterion (δ) was calculated by the formula:

$$\delta = \frac{S^2(Y_{ju})\ _{max}}{\sum_{u=1}^{N} S^2(Y_{ju})} \qquad (5)$$

In this case, it is equal to:

by the activity of the catalyst

$$\delta_A = \frac{0{,}09}{0{,}17} = 0{,}5014 \qquad (6)$$

Table 3.

Calculated dispersion values for the obtained catalyst samples

№ the catalyst (u)	$S^2(y)$ – by the activity of the catalyst (Δ Й.ч.)			$S^2(y)$ – by the selectivity of the catalyst, %			$S^2(y_j)$ – according to the isomerizing ability of the catalyst,%		
	$A_{u1}-\bar{A}_u$	$(A_{u1}-\bar{A}_u)^2$	$S^2(A_{ju})$	$C_{u1}-\bar{c}_1$	$(C_{u1}-\bar{c}_1)^2$	$S^2(C_{ju})$	$И_{u1}-\bar{И}_1$	$(И_{u1}-\bar{И}_1)^2$	$S^2(И_{ju})$
1	0	0	0	0,4	0,16	0,16	0,4	0,16	0,16
2	0,1	0,01	0,01	0,1	0,01	0,01	0,1	0,01	0,01
3	0	0	0	0,1	0,01	0,01	0,1	0,01	0,01
4	0,1	0,01	0,01	0,4	0,16	0,16	0,2	0,04	0,04
5	0,3	0,09	0,09	0,2	0,04	0,04	0,1	0,01	0,01
6	0,2	0,04	0,04	0,2	0,04	0,04	0,1	0,01	0,01
7	0,1	0,01	0,01	0,2	0,04	0,04	0,1	0,01	0,01
8	0,1	0,01	0,01	0,3	0,09	0,09	0,1	0,01	0,01

$\sum_{u=1}^{N} S^2(A_{ju}) = 0{,}17$ $\sum_{u=1}^{N} S^2(C_{ju}) = 0{,}55$ $\sum_{u=1}^{N} S^2(И_{ju}) = 0{,}26$

by the selectivity of the catalyst

$$\delta_C = \frac{0{,}16}{0{,}55} = 0{,}2909 \qquad (7)$$

according to the isomerizing ability of the catalyst

$$\delta_И \frac{0{,}16}{0{,}26} = 0{,}6153 \qquad (8)$$

Table values of the Cochran criterion for the significance level q= 0.05 in all cases is equal to 0,6798 [11,12], т.е. $\delta_{экс} < \delta_{табл.}$ therefore, the average of the variances can be taken as the variance of the experiment. (Table 3.)

To find the variance of the coefficients of the regression equation (2), we calculate –$S^2(Y_j)$; $S^2(\ddot{y})$; $S^2(B_u)$and S(B) by formulas:

by activity $S^2(Y_j) = \frac{\sum_{u=1}^{N} 2\,(A_{ju})}{N} = \frac{0{,}17}{8} = 0{,}021$ (9)

$$S^2(\bar{y}) \frac{S^2(A_{ju})}{n} = \frac{0{,}021}{2} = 0{,}010 \qquad (10)$$

$$S^2(B_u) = \frac{2(\bar{y})}{N} = \frac{0{,}010}{8} = 0{,}001 \qquad (11)$$

$$S(B_u) = \sqrt{S^2(B_u)} = 0{,}031 \quad (12)$$

by selectivity

$S^2(Y_j) = 0{,}069$ (13)

$S^2(\ddot{y}) = 0{,}034$ (14)

$S^2(B_u) = 0{,}004$ (15)

$S(B_u) = 0{,}063$ (16)

by isomerizing ability

$S^2(Y_j) = 0{,}032$ (17)

$S^2(\ddot{y}) = 0{,}016$ (18)

$S^2(B_u) = 0{,}002$ (19)

$S(B_u) = 0{,}044$ (20)

The significance of the regression coefficients is estimated by the Student-Fisher criterion [13, 14] at $\varphi = 8$ (i.e., for eight degrees of freedom) and the significance level $\alpha = 0.95$. Then:

$t(0{,}95; 8) = 2{,}31$ (21)

$\xi(B_u) = t(0{,}95; 8) \cdot S(B_u)$ (22)

The calculated values of ξ (Bu) are equal to:

by activity $\xi(B_u) = 2{,}31 \cdot 0{,}031 = 0{,}0716$ (23)

by selectivity $\xi(B_u) = 2{,}31 \cdot 0{,}063 = 0{,}1449$ 0716 (24)

by isomerizing ability $\xi(B_u) = 2{,}31 \cdot 0{,}044 = 0{,}1016$ 0716 (25)

Of all the coefficients, the most insignificant were:

by activity B_1

by selectivity B_1 и B_2; $B_{2,3} = B_{1,4}$

by isomerizing ability no

Equation (1), after excluding insignificant coefficients, will take the form:

by activity

$A = B_0 + B_2X_2 + B_3X_3 + B_4X_4 + B_{12}X_1X_2 + B_{14}X_1X_4 + B_{23}X_2X_3 + \ldots$ (26)

by selectivity

$C = B_0 + B_1X_1 + B_3X_3 + B_4X_4 + B_{12}X_1X_2 + B_{13}X_1X_3 + \ldots$ (27)

by isomerizing ability

$И = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_{12}X_1X_2 + B_{13}X_1X_3 + B_{14}X_1X_4 + B_{23}X_2X_3 + \ldots$ (28)

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Thus, in the regression equation, 6 factors were significant in terms of activity, 5 in terms of selectivity, and 7 in terms of isomerizing ability.

By quantitative contribution, the factors are arranged in the following order:

by activity

$X_3 > X_2 > (X_2X_3 = X_1X_4) > (X_1X_3 = X_2X_4) > X_4 > (X_1X_2 = X_3X_4)$ (29)

by selectivity

$X_3 > (X_1X_3 = X_2X_4)\ X_1 > (X_1X_2 = X_3X_4) > X_4$ (30)

by isomerizing ability

$X_4 > X_1 > X_2 > (X_1X_2 = X_3X_4) > (X_2X_3 = X_1X_4) > X_2 > (X_1X_3 = X_2X_4)$ (31)

The other factors are insignificant.

It should be noted that the greatest influence on the hydrogenating properties of the catalyst is exerted by the content of rhodium in the alloy.

To check the adequacy of the obtained regression equation for the true response surface, the Fisher criteria were calculated and compared with the table values.

To do this, we determine the residual dispersion – S_R.

$$S_R^2 = \frac{\sum_{u=1}^{N}(\hat{y}_u - \bar{y}_u)^2}{n-1} \quad (32)$$

The calculated values of the variances (SR) for the given example are shown in Table 4.

Table 4.

Calculated values of variances (*S_R*)

№ the catalyst (u)	S_R – by the activity of the catalyst		S_R – by the selectivity of the catalyst,%		S_R – according to the isomerizing ability of the catalyst	
	$A_u - \bar{A}_u$	$S^2(A_u)$	$c_u - \bar{c}_u$	$S^2(c_u)$	$И_u - \bar{И}_u$	$S^2(И_u)$
1	0,02	0,0004	0,005	0,0025	0,16	0,0256
2	0,02	0,0004	0,01	0,0001	0,30	0,0900
3	0	0	0,03	0,0009	0,34	0,1156
4	0	0	0,03	0,0009	0,20	0,0400
5	0,02	0,0004	0,05	0,0025	0,28	0,0784
6	0	0	0,06	0,0036	0,14	0,0196
7	0,02	0,0004	0,03	0,0009	0,22	0,0484
8	0,02	0,0004	0,01	0,0001	0,32	0,1024

$\sum_{u=1}^{N} S_R^2(A_u) = 0.0020$ $\sum_{u=1}^{N} S_R^2(C_u) = 0.0115$ $\sum_{u=1}^{N} S_R^2(И_u) = 0.5200$

Next, we calculate the variance of adequacy by the formula:

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$$S_{ад}^2 = \frac{S_R^2}{f_R} \quad (33)$$

where: f_R – the number of degrees of freedom, equal to 3.

$$f_R = N - K - 1 \quad (34)$$

where: N – number of catalysts (N = 8);

K – the number of factors (K = 4).

Then $S_{ад}^2$ equal to:

by activity

$$S_{ад}^2 = \frac{0{,}0020}{3} = 0{,}0006 \quad (35)$$

by selectivity

$$S_{ад}^2 = \frac{0{,}0115}{3} = 0{,}004 \quad (36)$$

by isomerizing ability

$$S_{ад}^2 = \frac{0{,}5200}{3} = 0{,}1733 \quad (37)$$

We calculate the Fisher criterion (F) in all cases:

$$F = \frac{S_{ад}^2}{S_{(\bar{y})}^2} \quad (38)$$

by activity, it is equal to

$$F = \frac{0{,}0006}{0{,}010} = 0{,}0600 \quad (39)$$

by selectivity

$$F = \frac{0{,}004}{0{,}034} = 1{,}1764 \quad (40)$$

by isomerizing ability

$$F = \frac{0{,}1733}{0{,}016} = 1{,}0830 \quad (41)$$

Table values of Fa = 0.95 = 4.1; i.e., Fr. << Ftable.

This indicates that the equation corresponds to the truth of the response surface. The best results of the hydrogenating properties of the catalyst were obtained on catalysts No. 3 and No. 5. Therefore, it was not necessary to conduct a steep ascent.

CONCLUSION

Thus, the promoter most affecting the hydrogenating properties of the nickel-copper-aluminum catalyst was rhodium and the palladium rhodium system at their next content in the alloy:

rhodium – 0.50 %

palladium (0.05 %) + rhodium (0.50 %).

The mathematical processing made it possible to significantly accelerate the identification of a stationary alloy catalyst for its introduction into the production of food hydrogenated fats.

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