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USING OF EXPERIMENTAL AND STATISTICAL METHODS IN FOOD TECHNOLOGY RESEARCH PROCESSES

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ПРИМЕНЕНИЕ ЭКСПЕРИМЕНТАЛЬНО-СТАТИСТИЧЕСКИХ МЕТОДОВ ИССЛЕДОВАНИЙ ПРОЦЕССОВ ПИЩЕВЫХ ТЕХНОЛОГИЙ

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Abstract. On the basis of these studies there was confirmed that construction of the optimal model systems in addition to the use of formal mathematical and computational methods involves the use of various heuristic arguments, based on experience and deep understanding of content by engineers. This approach made it possible to develop a standard algorithm and produce the successive stages of mathematical modeling and experimental design and compromise multi-objective optimization of processing foods. The example of the optimal high pressure processing parameters of scrambled eggs with cheese its effectiveness.

Key words: experimental and statistical approach, optimization, food technology, high blood pressure, scrambled eggs with cheese.

Аннотация. На основании проведенных исследований подтверждено, что создание оптимальных модельных систем кроме использования формальных математических и вычислительных методов предусматривает применение различных эвристических соображений, основанных на опыте и глубоком понимании содержания предмета инженерами-технологами. Такой подход дал возможность разработать типовой алгоритм и представить последовательные этапы математического моделирования и планирования эксперимента и компромиссной многокритериальной оптимизации процесса обработки полуфабрикатов. На примере нахождения оптимальных параметров обработки высоким давлением омлета с сыром продемонстрировано его эффективность.

Ключевые слова: экспериментально-статистический подход, оптимизация, пищевые технологии, высокое давление, омлета с сыром.

Introduction.

The intensive development of applied biotechnology, information technologies, systems analysis and mathematical methods created the objective conditions for a new level of understanding of the physical nature of the analytical and numerical description of processes in the processing of raw materials of animal and vegetable origin. This allows the scientific validation for the possibility of obtaining new products with a given composition using alternative technologies, and the ability to process control at all stages of food production.

For the formal description of complex systems further development and improvement methodology a multivariate statistical models of linear parameters and, in general, nonlinear factors are necessary. Solving of real applications requires the development of sequential methods of experiment planning in arbitrary form, custom field factorial space, a formalized structure of multivariate statistical models, unknown to researcher, robust estimations of model coefficients in the original multicollinearity factors [1].

Mathematical apparatus used must meet the basic statistical properties of complex systems and processes [1]. Here are the main ones.

1. Results of the experiment are the result of cumulative exposure groups of managed, unmanaged and uncontrolled factors. Physical properties of the studied system and process can not be completely defined and contain uncertainty.

2. The experimental results are approximate numerical values without specifying any of their theoretical properties and forms (structures) of connection between the conditions of the experiment and its results.

3. The inductive method of knowledge is realized in the experiment – the result separate from its generalization to all hypothetical similar results.

4. It is assumed that the complex conditions of the experiment are typical for examined group of complex systems and processes, and therefore the result can be extended to other members of the class of systems studied.

Multivariate statistical modeling and optimization of multi-objective compromise are the most common applied research methods. All experts believe that regression analysis is one of the main methods of mathematical statistics. It is widely used in scientific and applied research in various spheres of human activity. However, solving applied problems "regression Analysis and interpretation of the results require deep knowledge and ability to properly assess the information received" [1].

Statistical methodology of regression analysis should provide stability, certainty is obtained by multivariate statistical models. You must use sustainable plans of multifactorial experiments, the choice of "true" structure models, stable models evaluating the coefficients in the original multicollinearity factors [1].

Experts in the field of statistical methods draw attention to the complexity of the problems of statistical methodology. Methodology for modeling of complex systems and processes inevitably involves heuristic recommendations and decisions that are difficult to formalize . These solutions are used in data analysis, experimental design theory, applied statistics and technological processes [1].

The aim of this work – to demonstrate typical algorithm of this study to explore the effect of high pressure on processing parameters scrambled eggs with cheese. The author expresses his sincere gratitude to the Director of the Institute of Food Production DonNUET Mykhailo Tugan-Baranovsky Doctor of Technical Sciences, Professor. V.A. Sukmanov for experimental data on the properties of food masses under high pressure, which are executed under his supervision, for participation in the

discussion of planning and conducting experiments and developing methodology the problem of optimization of technological processes.

It was necessary for the experiment to evaluate the effectiveness of integrated action of several factors on the quality of the developed food, namely, cheese omelet. Dependent variables: y_1 - water activity (a_w); y_2- a comprehensive quality (K). Factors affecting these parameters: x_1 - pressure (P, MPa); x_2 - temperature ($t, ^\circ C$); x_3 - dovhotryvalist processing (τ, c); x_4 - weight of water per 100 g melange (g); x_5 - weight of milk powder per 100 g melange (g); x_6 - ksantovana gum (% of the total weight of the mixture); x_7 - weight cheese 100 g melange (g).

The experimental results indicate linear dependence of water activity on most factors, then this finding based on seven factors set we used fractional replica of marginal limiting factors for the number eight experiments.

Table 1

Plan an experiment to find the coefficients of the linear model

x_0	x_1	x_2	x_3	x_4	x_5	x_6	x_7	y_1
1	1	1	1	1	1	1	1	0,705
1	1	-1	-1	-1	-1	1	1	0,82
1	-1	-1	1	1	-1	-1	1	0,775
1	-1	1	-1	-1	1	-1	1	0,82
1	-1	-1	-1	1	1	1	-1	0,795
1	-1	1	1	-1	-1	1	-1	0,775
1	1	1	-1	1	-1	-1	-1	0,81
1	1	-1	1	-1	1	-1	-1	0,745

It is estimated coefficients eight following a busy plan (Table 2):

Table 2

The coefficients of the linear model

x_7	x_6	x_5	x_4	x_3	x_2	x_1	x_0
-0,00063	-0,00688	-0,0144	-0,00937	-0,03063	-0,00313	-0,01063	0,780625

To release the linear effects of the first order interactions used the method of "pass." In its application added a new cue, all the signs which are opposite the original remark. Got rates further and final linear model (Table. 3).

Table 3

The coefficients of additional and final linear model

	x_7	x_6	x_5	x_4	x_3	x_2	x_1	x_0
Additional LM	0,01125	-0,005	0,01	0,00125	-0,02625	0,005	-0,015	0,77625
Final LM	0,005312	-0,00594	-0,0022	-0,00406	-0,02844	0,000937	-0,01281	0,778438

After the addition, the model leaving only statistically significant factors:

$$y_1 = 0{,}778 - 0{,}013x_1 - 0{,}028x_3 \quad (1)$$

To determine the complex quality depending on the pressure, temperature and time we used a central composite uniforms, rotatabelnyy plan for three factors, the construction of which is related to conduct experiments in twenty double repeatability. Repeating provides increased accuracy assessments and promotes the release of weak signals over background noise. In addition, we used randomization - random order of realization of experiments designed to combat systematic error.

The first phase was conducted rotatabelne central compositional planning (TSKRP). The treatment results were typical method [2].

Parallel experiments in the center of the plan to determine that the regression equation of the first order received by PFE results may not provide a satisfactory mathematical description of model systems and planning must go to the second order to take into account evaluation factors and quadratic effects build adequate mathematical model.

Statistical analysis of experimental data contained calculating estimates of regression coefficients, checking their significance, evaluation of reproducibility of experiments and establishing the adequacy of the resulting regression equation. This used statistical criteria Cochran, Fisher and Student (for confidence level 95%).

The regression equation which adequately describes the dependence of response on factors which are managed, is:

$$y_2 = 0{,}965 - 0{,}022x_1 + 0{,}026x_2 + 0{,}008x_3 - 0{,}013x_1x_2 - 0{,}012x_1x_3 - 0{,}014x_2x_3 - 0{,}031x_1^2 - \\ - 0{,}006x_2^2 - 0{,}015x_3^2 \quad (2)$$

The coefficient of determination R^2 = 0,939508. The model is adequate for the Fisher criterion: $F_p = 4{,}67 < F_m = 5{,}05$ for significance level 0.05, 5 degrees of freedom.

In this case, one can see that a priori reasoning largely confirmed because not only were significant linear effects of factors, but the pair interaction and quadratic effects. Of the three linear effects of two distinguished: the effect factor - and the pressure factor - the duration of the experiment. Apparently quantify the factors influencing them stronger temperature of the experiment. Temperature variation in selected intervals not make a significant impact on this figure as a linear coefficient less. But the impact of this factor manifested equally with other factors in paired interactions. The content of the interaction effect is that the influence of one factor depends on what level is another factor.

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Graphical analysis of two-dimensional graphs and regression lines of a fixed value of one of the three factors shows that the maximum values of complex quality found in the experiment. This allows you to find the extremes of the classical methods of analysis. To find the optimal parameters x_1, x_2, x_3 first "compromise" optimization problem formulated in this way. Need to find the meaning of control factors that ensure the maximum $y_2 = f(x_1, x_2, x_3)$ at a given value $y_1 = \varphi_1(x_1, x_2, x_3)$. The values of the independent variables thus be kept in the experiment, which borders determined by the value factors "star" points. Analytical it can be written as an expression $\varphi_2(x_1, x_2, x_3) = x_1^2 + x_2^2 + x_3^2 = R^2$ of that factor space is a sphere of radius centered at the center of the experiment. Thus, we "compromise" optimization problem: maximize function

$$y_2 = 0{,}965 - 0{,}022x_1 + 0{,}026x_2 + 0{,}008x_3 - 0{,}013x_1x_2 - 0{,}012x_1x_3 - 0{,}014x_2x_3 - 0{,}031x_1^2 - \\ - 0{,}006x_2^2 - 0{,}015x_3^2 \quad (3)$$

under conditions $\begin{cases} y_1 = 0{,}778 - 0{,}013x_1 - 0{,}028x_3, \\ x_1^2 + x_2^2 + x_3^2 = R^2. \end{cases}$

For solving this problem applied Lagrange multiplier Lagrange. For this build target function $F_1(x_1, x_2, x_{2,}\lambda_1, \lambda_2)$, which is the sum equation and optimization of products φ_1, φ_2 on the relevant factors λ_1, λ_2:

$$F = 0{,}965 - 0{,}022x_1 + 0{,}026x_2 + 0{,}008x_3 - 0{,}013x_1x_2 - 0{,}012x_1x_3 - 0{,}014x_2x_3 - 0{,}031x_1^2 - \\ - 0{,}006x_2^2 - 0{,}015x_3^2 + \lambda_1(0{,}778 - 0{,}013x_1 - 0{,}028x_3 - y_1) + \lambda_2\left(x_1^2 + x_2^2 + x_3^2 - R^2\right) \quad (4)$$

In accordance with the method of computing algorithm built Lagrange system of equations containing partial derivatives of the objective function for all independent variable and uncertain Lagrange multipliers:

To solve the resulting system of equations using integrated suite of MAPLE 13. The value calculated by changing the radius of the sphere in the range of 1.628 to 0, and the lowest setting possible. Characteristic of the results of calculations are presented in Table 4.

$$\begin{cases} \dfrac{\partial F}{\partial x_1} = -0{,}022 - 0{,}013x_2 - 0{,}012x_3 - 0{,}062x_1 - 0{,}013\lambda_1 + 2\lambda_2 x_1; \\ \dfrac{\partial F}{\partial x_2} = 0{,}026 - 0{,}013x_1 - 0{,}014x_3 - 0{,}012x_2 + 2\lambda_2 x_2; \\ \dfrac{\partial F}{\partial x_3} = 0{,}008 - 0{,}012x_1 - 0{,}014x_2 - 0{,}030x_3 - 0{,}028\lambda_1 + 2\lambda_2 x_3; \\ \dfrac{\partial F}{\partial \lambda_1} = 0{,}778 - 0{,}013x_1 - 0{,}028x_3 - y_1; \\ \dfrac{\partial F}{\partial \lambda_2} = x_1 + x_2 + x_3 - R^2; \end{cases}$$

The best followed recognize data recorded in the second row of the table 9, when the value of complex quality becomes maximum, equal to 0.98. The optimal solution of encoded values and natural factors are presented in Table 5.

Table 4

The results of calculations for the optimal values of factors

R	*The coded values of factors*			*function review*	
	x_1	x_2	x_3	y_2	y_1
0,4	-0,229	0,285	-0,162	0,974	0,786
0,64	-0,1	0,6	-0,2	0,98	0,786
0,9	0,577	-0,449	-0,525	0,924	0,786
1,68	1,361	-0,443	-0,879	0,864	0,786

Table 5

The optimal solution

The coded values of factors			*Dietary factors mentioned*			*Function review*	
x_1	x_2	x_3	P, MPa	t, C°	τ, c	y_2	y_1
-0,1	0,6	-0,2	695	122	425	0,98	0,786

Graphic interpretation optimal solution below (Figure.1-3).

As the expert determination organoleptic qualities omelette that is integrated Quality can not be held simultaneously for all seven control factors (it is necessary to simultaneously carry out, for example, in the double frequency of 326 experiments TSKRP), we have decided to sequential replacement of the third factor (linear rate three times lower than the corresponding coefficients of the first and second factors) to other factors.

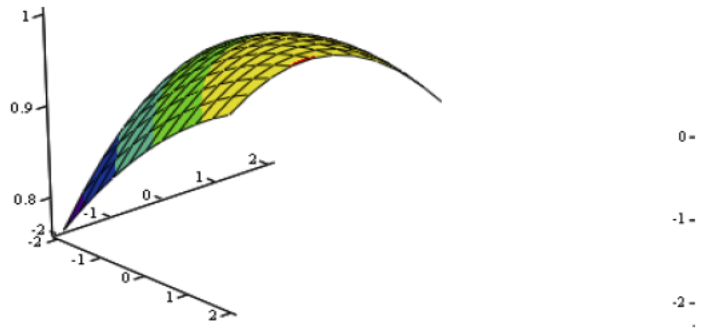

Figure.1. Surface Review and line-level regression at $x_1 = -0{,}1$.

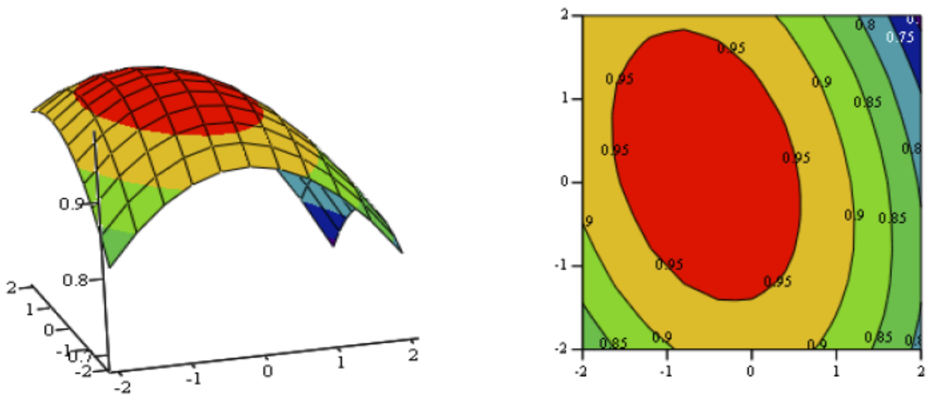

Figure.2. Surface Review and line-level regression at $x_2 = 0{,}6$.

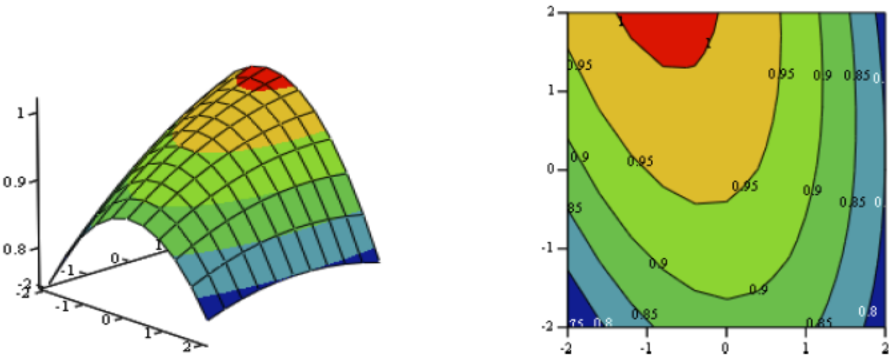

Figure.3. Surface Review and line-level regression at $x_3=-0,2$.

For all models we verified the significance of the regression coefficients. Since orthogonal plan, they determined the same variance. Next to the regression coefficients calculated confidence interval with a certain confidence probability. In this expression - criterion (Student test) has the same number of degrees of freedom, and the variance of reproducibility. Significant factor if its absolute value than the confidence interval.

Excluded non-significant regression coefficients and again inspected the adequacy of the model with significant coefficients.

If instead x_3 the plan will include the experiment x_4, the regression equation which adequately describes the dependence y_2 of the response factors now will look like:

$$y_2=0,953-0,007x_1+0,008x_2+0,011x_4+0,0025x_1x_2-0,001x_1^2-0,010x_2^2-0,005x_4^2 \quad (5)$$

The coefficient of determination R^2 = 0,949504. The model is adequate for the Fisher criterion: $F_p=0,08<F_m=5,05$ for significance level 0.05, 5 degrees of freedom.

The optimal solution of encoded values and natural factors are presented in Table 6 and graphical interpretation optimal solution is shown in Figure 4-6.

Table 6

The optimal solution

The coded values of factors			*Dietary factors mentioned*			*Function review*	
x_1	x_2	x_4	P, MPa	t, C°	x_4, g	y_2	y_1
-0,11	0,63	0,52	694,5	122,6	14,04	0,96	0,77

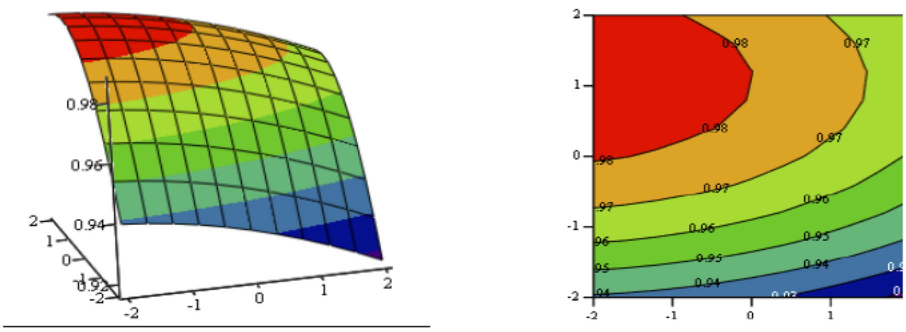

Figure.4. Surface Review and line-level regression at $x_2=0,63$

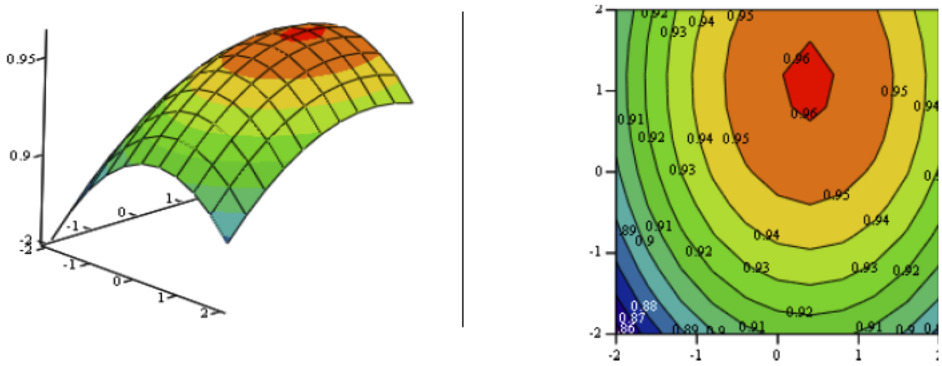

Figure.5. Surface Review and line-level regression at $x_1 = -0{,}11$

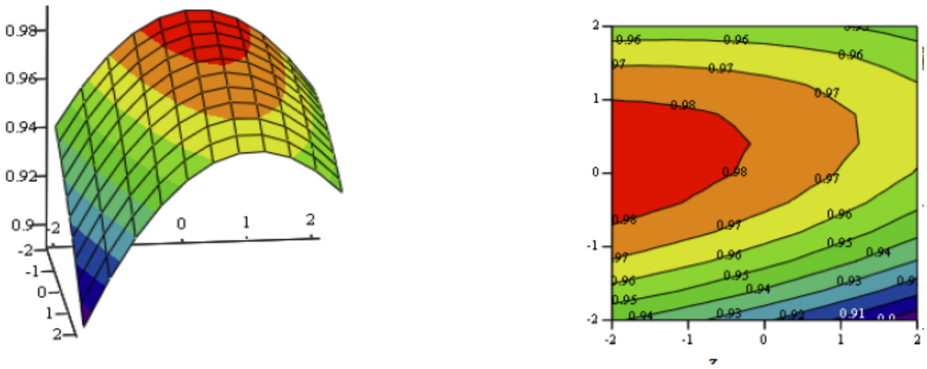

Figure.6. Surface Review and line-level regression at $x_4 = 0{,}52$.

In the same way investigations are carried out when replacing in terms of experiment variable X_3 sequence variable X_5, X_6 and X_7.

Statistical analysis of the complete model interpretation in terms of the object of study. His do so. We provide experimental verification found the optimal parameters and assessment of the accuracy and the reliability parameters optimization. Under these optimal values of ten factors was conducted parallel experiments for all seven factors results and statistical analysis are shown in Table 7.

Table 7

Results optimization

parameter optimization	The value		Dispersion	Criterion	Error	Confidence interval
	y_i^p	y_i^e	S^2	t_p	δ	
y_1	0,786	0,748	0,0045	1,79	0,048	0,738-0,834
y_2	0,98	0,941	0,005	1,74	0,051	0,929-1,0

Here t_p - the estimated value of Student's t test, δ - the forecast error parameter optimization.

The results of calculations for the optimization of given parameters, presented in the form of confidence intervals indicate that their experimental values do not exceed the respective ranges and hence proving the reliability of the results.

The results have practical use and they have been accepted as the basis for selecting rational parameters of cooking omelette with these additives, processed high pressure.

Summary and Conclusions.

Experimental values for optimization parameters do not extend beyond the suitable confidence intervals obtained by calculation method, indicating the accuracy and reliability of the results.

Thus, we can conclude that in addition to the formal mathematical and computational methods, various heuristic arguments, based on experience and deep understanding of content engineers, play an important role on creating of the optimal model systems.

Thus, we developed a structural parametric model for prediction of a quality of the finished product at optimum settings of the parameters and characteristics of biological materials process high pressure processing foods.

The algorithm of mathematical modeling and multi-objective optimization compromise the processing of foods is elaborated.

The proposed research methodology can be applied to find the optimal parameters of other processes food technology. Further improvement of methodology using fuzzy sets theory, the concept underdetermined models and interval analysis are planned.

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