Методом планування експерименту отримана математична модель, яка описує вплив процесу наповнення-пластифікації на технологічні властивості шкіри. Виконана максимінна згортка основних параметрів якості для переходу до однокритеріальної оптимізації. Розроблено обчислювальний модуль в середовищі Visual basic for Application для автоматизації розрахунку оптимального складу дисперсної системи методом Хука-Дживса. Отримані оптимальні значення концентрацій інгредієнтів наповнювально-пластифікуючої композиції і технологічних властивостей шкіряного матеріалу

Ключові слова: метод Хука-Дживса, оптимізація процесу наповнення-пластифікації, шкіряний матеріал, технологічні властивості

Методом планирования эксперимента получена математическая модель, описывающая влияние процесса наполнения-пластификации на технологические свойства кожи. Выполнена максиминная свертка основных параметров качества для перехода к однокритериальной оптимизации. Разработан вычислительный модуль в среде Visual basic for Application для автоматизации расчета оптимального состава дисперсной системы методом Хука-Дживса. Получены оптимальные значения концентраций ингредиентов наполнительно-пластифицирующей композиции и технологических свойств кожевенного материала

Ключевые слова: метод Хука-Дживса, оптимизация процесса наполнения-пластификации, кожевенный материал, технологические свойства

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OPTIMIZATION OF LEATHER MATERIAL FILLING-PLASTICIZING PROCESS USING DISPERSE SYSTEM

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1. Introduction

Development of innovative technologies of the leather and fur skin materials formation requires application of a wide range of new synthetic and natural reactants [1, 2] in the filling-plasticizing process. The efficient implementation of the process requires creating conditions for a deep diffusion of chemical particles in a microfibrillar collagen structure of the dermis. This can be performed due to the high dispersion of plasticizing reactants of hydrophilic and hydrophobic nature to improve features of elastic polyfunctional leather materials [3, 4]. Taking into account the wide range of interactions both between components of a filling-plasticizing system and the system component interaction with collagen of semi-finished structured material during the tanning process, it is necessary to create conditions for the diffusion of reactants. In this connection, during first interaction minimal interaction of the system components is provided.

Optimization of the chemical composition of the above-mentioned systems is an important aspect of the problem. Usage of natural materials such as natural fats of animal or vegetable origin, natural aluminosilicates that are subject to modification depending on the stage of the technological process, is a long-term research area [4]. This is an important point both in terms of obtaining aggregation-stable colloidal systems used in leather production technology and their diffusing capacity in the semi-finished leather.

The complex component composition of the filler-plasticizing dispersed system and multiple stages of the structure and properties of the leather material formation process requires the use of multi-criteria optimization approach. Such an approach promotes the rational use of leather raw materials and chemical reagents. In this case, minimax optimization criteria convolution of filling-plasticizing process has been used. This method allows improving the worst indicator among the technological properties of the material. After such a convolution of criteria, single-criteria optimization problem has been solved by the method of Hooke-Jeeves. This method consists of two main stages – the exploring search and pattern search (accelerating search). Acceleration search allows finding the global optimum of the function briefly.

2. Literature review and problem statement

The analysis of publications devoted to the development of innovative technologies of leather and fur skin materials formation showed that the authors mainly use the of experiment planning methods to obtain a mathematical description of the mentioned processes. In [5] it is shown that the simulation model and optimal area of the skin chrome tanning process were obtained by using Mathcad software where the noncomposite symmetric quasi-optimal plan was implemented. Mentioned software is not typically used at Ukrainian leather factories. Thus, such solution cannot be used to correct the optimum area of the process in operation mode under changing production conditions.

Plackett-Burman plan in the paper [6] is used to decrease the number of factors from 7 to 4. The survey of the full factorial experiment to study the process of chrome tanning with the use of modern polymer material – a derivative of maleic acid is shown in [7]. The adequate simulation model that describes the dependencies of semi-finished leather indicators on the consumption of basic materials and processing time is obtained. The rational conditions of tanning are identified. It should be noted that the Harrington's desirability function used for the formation of the generalized criterion of optimization is performed in the papers [6, 7]. With this approach, the formation of optimization criterion depends on the experience of the researcher [8]. The application of SWOT-analysis methods [9] in the marketing strategies area, connecting the factors of threats and opportunities, allows us to solve the problematic issues. However, it contains subjective evaluation of the company's management. It may adversely affect the activity of the enterprise in terms of dynamism and uncertainty.

The approach proposed in [5–8] to solve the optimization problem is associated with the complexity of the formalization and requires a significant amount of calculations. In papers [10, 11] it is shown that in some cases the wrong selection of driving directions to the optimum does not lead to the desired result. The choice of the optimization method and the optimality criterion formation method have a decisive impact on the quality of the obtained solutions. Using the modified method of Hooke-Jeeves [12] allows to solve constrained optimization problems as well as it allows to optimize the investigated processes with a higher degree and achieve the optimal area more rapidly.

3. Purpose and objectives of the study

The purpose of this paper is an improvement of basic technological properties of leather material formed by filling-plasticizing process optimization using the disperse system ingredients.

In accordance with the set goal, the following research tasks are identified:

– obtaining simulation models that adequately describe the effect of the filling-plasticizing process using dispersion system components on the technological properties of the leather material;

– development of software module allowing automation of the calculation of the required components concentration of the dispersed system;

– optimization of components concentration of the filling-plasticizing process using the developed software module.

4. Materials and research methods of dispersed system

In accordance with the set purpose, in the optimization of the leather material-filling-plasticizing process, semi-finished leather is used. It is obtained from cattle raw material using chrome tanning method and slicing the material to 1.5–1.6 mm thickness. The finely aluminosilicate (FA) modified by sodium format is used as a filler of semi-finished leather. The dispersing mixture of natural fats and oils: beef tallow, sunflower oil, and fish oil in a ratio of 1:1.75:2.25 by weight % [13] is used for plasticizing the semi-finished product. The nonionic surface active agent is used to emulsify the fats.

The process of filling-plasticizing of semi-finished leather is carried out in the experimental section of PJSC Chinbar (Kiev, Ukraine) at the ratio of water:semi-finished product 1:1 and temperature of 55–60 °C. The concentrations of the plasticizer, surfactant and aluminosilicate filler were varied, respectively, at a rate: 49.6–140.4; 4.4–31.6; 15.0–30.0 (g/l).

The effectiveness of the filling-plasticizing process is determined by the concentration of the following substances which influence the technological properties of the materials, g/l:

– plasticizer (X_1);

– modified filler BAC (X_2);

– nonionic surfactant (X_3).

The technological effect of the ingredients of filling-plasticizing dispersion system application on the leather material obtained by the control technology was evaluated by the following quality parameters:

– output area of the semi-finished product, % (y_1);

– the total content of fatty substances in the leather material, % (y_2);

– hardness of material, cH (y_3).

The output area of the leather material was calculated as the area ratio of the received prototypes of the finished material to the finished material obtained by the current technology. The total content of fatty substances in the finished leather was determined by the content of fat in the sample. The extraction in an organic solvent mixture after the hydrolytic destruction by alkali solution, followed by the extraction fatty acids with ether was used [14]. The hardness was measured by the PZU-12M equipment after drying and humidification processes. After that, the samples were cured in a desiccator at 20–22 °C and 16 % humidity for 24 hours.

The central rotatable second-order composite plan was designed to conduct the experiment. The plan is based on the full factorial experiment, complemented by experiments in the center of the plan and asterisk points [15, 16] which allow to evaluate the dispersion of reproducibility. The area of the experiment is described by the following model (1).

$$\hat{y} = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n-1} \sum_{\substack{j=i+1 \\ i \neq j}}^{n} b_{ij} x_i x_j + \sum_{i=1}^{n} b_{ii} x_i^2, \quad (1)$$

where x_i, x_j – factors, n – the total amount of factors b_i, b_{ij}, b_{ii} – coefficients.

This model includes the first-order coefficients as well as pair and quadratic interaction effects. The results of the experiment are shown in Table 1.

Factors x_1, x_2, x_3 take the zero value at the concentration of (g/l): plasticizer – 90, modified filler – 18, nonionic surfactant – 22.5 respectively, and their variation interval – 30; 8.1 and 4.5.

The transition from the technological values X_i to the encoded x_i is carried out using the formula (2).

$$x_i = \frac{X_i - X_{i0}}{\Delta X_i}, \quad (2)$$

where X_{i0} – factor zero value, ΔX_i – variation interval.

Table 1

Central rotatable second-order composite plan with coded values of factors

№	x_0	x_1	x_2	x_3	$x_1 \cdot x_2$	$x_1 \cdot x_3$	$x_2 \cdot x_3$	x_1^2	x_2^2	x_3^2
1	1	1	1	1	1	1	1	1	1	1
2	1	−1	1	1	−1	−1	1	1	1	1
3	1	1	−1	1	−1	1	−1	1	1	1
4	1	−1	−1	1	1	−1	−1	1	1	1
5	1	1	1	−1	1	−1	−1	1	1	1
6	1	−1	1	−1	−1	1	−1	1	1	1
7	1	1	−1	−1	−1	−1	1	1	1	1
8	1	−1	−1	−1	1	1	1	1	1	1
9	1	1.68	0	0	0	0	0	2.8224	0	0
10	1	−1.68	0	0	0	0	0	2.8224	0	0
11	1	0	1.68	0	0	0	0	0	2.8224	0
12	1	0	−1.68	0	0	0	0	0	2.8224	0
13	1	0	0	1.68	0	0	0	0	0	2.8224
14	1	0	0	−1.68	0	0	0	0	0	2.8224
15	1	0	0	0	0	0	0	0	0	0
16	1	0	0	0	0	0	0	0	0	0
17	1	0	0	0	0	0	0	0	0	0
18	1	0	0	0	0	0	0	0	0	0
19	1	0	0	0	0	0	0	0	0	0
20	1	0	0	0	0	0	0	0	0	0

Note: the encoded values –1, 0, 1 correspond to the lowest, zero and top-levels of the plan; –1.68 1.68 – correspond to the asterisk points on the lowest and top levels of the plan

5. Simulation model of the filling-plasticizing process of semi-finished leather

According to the experimental plan, to study the efficiency of the effect of input variables on the technological properties of the leather material, 20 lots were collected. For this reason, the method of alternating halves [14] was used for semi-finished chrome-tanned leather, 3 pieces in each lot. The values obtained for the technological properties of semi-finished chrome-tanned leather after the implementation of the filling-plasticizing process are given in Table 2.

Table 2

Experiment Results

№	1	2	3	4	5	6	7	8	9	10
Y_1	105.6	98.7	103.8	96.5	101	95.1	98.4	93.2	104.7	93.7
Y_2	7.3	5.9	6.7	3.9	5.6	4.8	5.1	3.7	6.7	4.7
Y_3	24.5	28.5	23	29	22.5	24.5	32	37.5	25.5	31
Y_1	104.3	100.9	99.3	93.6	105.9	105.2	105.1	104.3	105.6	105.4
Y_2	7.8	5.7	4.3	2.8	7.5	6.4	7.2	6.1	7.4	6.9
Y_3	17	19	33	43	17.5	20	18	22	19	21

The coefficients of the simulation model (1) were calculated using the least squares method in a matrix form (3)

$$B = \left(X^T X\right)^{-1} \left(X^T Y\right). \qquad (3)$$

Reproducibility dispersion using the experimental points in the center of the plan is defined as follows (4)

$$S_0^2 = \frac{1}{N_0 - 1} \sum_{u=1}^{N_0} \left(y_{u0} - \bar{y}_0\right)^2. \qquad (4)$$

The value of the simulation model coefficients was tested by the Student's criteria and obtained by the formula (5)

$$t_{p_i} > t_T(f_0, q). \qquad (5)$$

The criterion impact was considered significant if its estimated value exceeded a tabulated point. After the elimination of non-significant coefficients, an adequacy of the model was determined by the Fisher criterion. For this purpose two dispersions were calculated: the residual dispersion and dispersion of adequacy by the formulas (6) and (7).

$$S_{res}^2 = \frac{\sum_{u=1}^{N} (\overline{y_u} - \hat{y}_u)^2}{N - 1}, \qquad (6)$$

$$S_{ad}^2 = \frac{S_{res} - S_0}{f_{ad}}. \qquad (7)$$

The Fisher criterion is calculated by the formula (8).

$$F_c = \frac{S_{ad}^2}{S_0^2}. \qquad (8)$$

The tabulated point of the Fisher criterion was taken according to the degree of freedom of dispersions of adequacy, dispersion and value level q, F_T (f_{ad}, f_0, q).

As the elimination of insignificant coefficients from the obtained model led to inadequate model $F_P > F_T$, the final model includes all the factors (Table 3).

In this case, the calculated value of the Fisher's criterion was less than the tabulated point $F_P < F_T$, for the specified degrees of freedom (Table 4). Thus, the resulting model is adequate.

Thus, the mathematical model adequately describes the process of filling-plasticizing of semi-finished leather. The following mathematical model (9) was used to optimize the composition of the dispersed system.

$$\begin{cases} y_1 = 105{,}2504 + 3{,}208549\, x_1 + 1{,}041569\, x_2 + \\ + 1{,}940374 x_3 - 2{,}146144 x_1^2 - 0{,}9414786\, x_2^2 - 3{,}120475 x_3^2; \\ y_2 = 6{,}888165 + 0{,}7152907 x_1 + 0{,}5663697 x_2 + \\ + 0{,}5218105 x_3 - 0{,}398291 x_1^2 - 1{,}160054 x_3^2; \\ y_3 = 19{,}61442 - 1{,}959721 x_1 - 1{,}821939 x_2 - 2{,}07405 x_3 + \\ + 2{,}9375 x_2 x_3 + 2{,}87359 x_1^2 - 0{,}7580667 x_2^2 + 6{,}328101 x_3^2. \end{cases} \qquad (9)$$

Table 3

Coefficients of the simulation model

b_{ii}	b_0	b_1	b_2	b_3	b_{23}	b_{11}	b_{22}	b_{33}
Y_1	105.2504	3.208548	1.041569	1.940373	–0.0625[1]	–2.14613	–0.94148	–3.12048
Y_2	6.911947	0.715291	0.56637	0.521811	0.125[1]	–0.40115	–0.0291[1]	–1.16292
Y_3	19.61442	–1.95972	–1.82194	–2.07405	2.9375	2.873593	-0.75807	6.328099

Note: the impact of factors on the technological properties of the material is insignificant

Table 4

Determination of the models adequacy

Model	Y_1	Y_2	Y_3
Dispersion			
– residual;	0.12902	0.03702	2.63670
– adequacy	0.22691	0.19585	4.15753
Fisher criterion			
– calculated;	0.4315	0.1165	0.8669
– tabulated	4.9503	4.7351	4.7351

6. Optimization of the filler-plasticizer dispersion system composition

Using the simulation model (9) as an objective function specifies the solution of the multiobjective constrained optimization problem. Firstly, we need to obtain a compromise solution to solve this problem. Namely, to find values of the factors where the technological properties parameters of the material take the same values in the given range of values. The methods of the main criterion, additive, multiplicative convolution of criteria, generalized Harrington's desirability function [12, 17] can be used. In addition, the papers that describe the alternative compromise optimizing modern methods [18] are known. However, the mentioned methods are not applicable to the solution of chemical engineering problems.

In this paper, the maximin convolution of optimization criteria was used as it provides the best value of the worst criterion (10).

$$Y(x_1,x_2,x_3) = \max\min y_i(x_1,x_2,x_3). \qquad (10)$$

According to the formula (10), the minimum value of each parameter y_i is calculated, and the corresponding factors are determined. Optimum values of factors are defined as the maximum of the minimum value of the output variables. The method of Hooke-Jeeves, which is referred to the zero-order direct methods [12, 19], is used to solve this task. The Hooke-Jeeves method algorithm consists of two stages – the exploring search and the pattern search. The Hooke-Jeeves method specifies the starting point near the center of the test range. The starting point can be set as a random value. After that, the exploring search was conducted in the vicinity of the starting point, which allows to determine the direction of the function steepest ascent. Then, the accelerating search was carried out in the identified direction. If the function value ceased to increase, the search is conducted around the new point. If the exploring search failed in all directions and if the predetermined calculation accuracy is not reached, the step is narrowed down. The search is carried out until the optimum is found with a given accuracy [17, 18].

As the obtained model includes three optimization parameters and each of them contains all three factors, the software module in the Visual Basic for Application was developed to automate the calculation. It allows to specify the starting point, step size, and the factors boundaries. The main window of the module is shown in Fig. 1.

A user places initial data on the MS Excel sheet (Fig. 2), and then can run the calculation of the simulation model (9).

As a consequence of solving the multicriteria optimization task using the software module, the factors optimal values of the leather material filling-plasticizing process were obtained. The 115.267, 22.492, 23.883 (g/l) concentration of plasticizer, modified filler and nonionic surfactant should be set respectively.

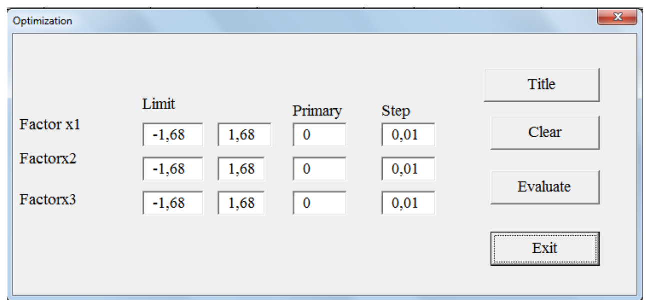

Fig. 1. Main window of software module

Coefficients	b0	b1	b2	b3	b12	b23	b13	b11	b22	b33
y1	105,2504	3,208548	1,041569	1,940373	0	0	0	-2,14613	-0,94148	-3,12048
y2	6,911947	0,715291	0,56637	0,521811	0	0	0	-0,40115	0	-1,16292
y3	19,61442	-1,95972	-1,82194	-2,07405	0	0	2,9375	2,873593	-0,75807	6,328099

Fig. 2. Coefficients of simulation model in MSExcel

Thus, the maximum leather material output area y_1=107.0393 %, the material hardness y_3=18.986 sN and the total content of fatty substances in the material y_2=7.565 % are provided.

7. Analysis of the semi-finished leather filling-plasticizing process simulation model

Presented graphical diagrams (Fig. 3, *a, b*) demonstrate the technological properties of leather materials and the composition of the dispersed system relationships. Namely, the increase in the plasticizer concentration in the system leads to increase in the material output area up to the saturation point 115.27 g/l. Further increase of the plasticizer content decreased the output area. The reason for this is that the mobility of the collagen dermis structure fibers increased up to the critical plasticizer concentration in the material during its deformation in different directions. After water removing, the deformation degree of semi-finished leather structure is stabilised.

Influence of the modified filler and a nonionic surfactant concentration on the leather material output area is symbatical as compared to the plasticizer influence. However, the nonionic surfactant provides the plasticizer and the filler particles diffusion to the product structure and their uniform distribution in the material by layers. Effective implementation of the process of filling- plasticizing of semi-finished leather can be carried out at the optimal concentration of surfactants in the disperse system. Insufficient amount of surfactants in the system does not provide effective plasticizer particles dispersion. However, the plasticizer overdosage increases the disperse system stability. In the former case, the plasticizer is deposited in the surface layer of the material. In the latter case, the plasticizer does not provide an active diffusion that leads to increased material shrinkage. Effect of filler on the material output area is due to its reduction resulted from thickness increase at a concentration above the critical value.

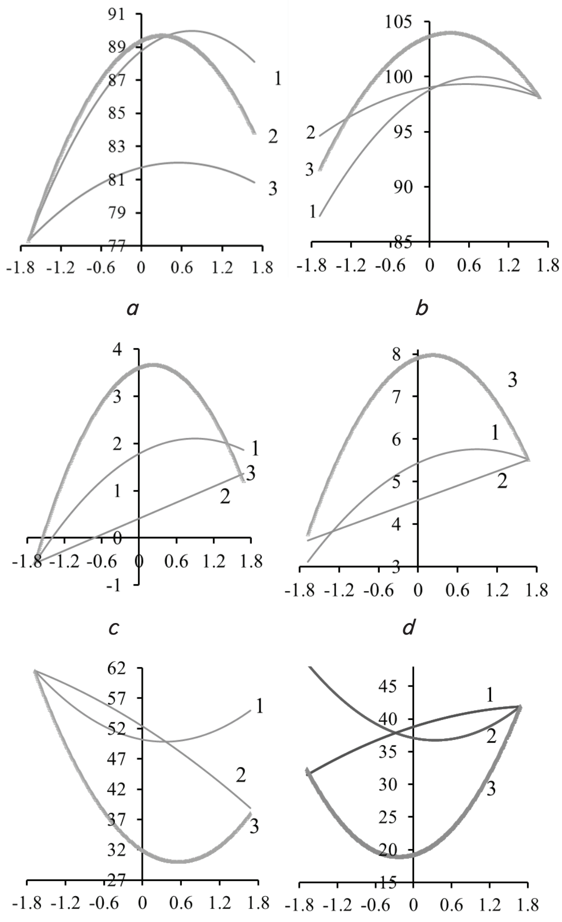

Fig. 3. Single-factor dependency for technological properties of leather material y_1 (*a, b*), y_2 (*c, d*), y_3 (*e, f*) in minimum and maximum concentration area, g/l: 1 – plasticizer, 2 – modified filler, 3 – nonionic surfactant: *a, c, e* – valley region; *b, d, f* – peak region

The concentration increase of the plasticizer from 49.6 to 119.05 g/l leads to an increase of the total content of fatty substances in the skin (Fig. 3, *c, d*). Further increase of fatty substances concentration in the system leads to a reduction of the total content of the plasticizer in the material. As a result, the degree of emulsification is reduced and the content in the disperse system is increased. An increase of the modified filler concentration due to the increased diffusion capacity of the hydrophilic filler leads to an increase of fatty substances content in the leather material.

The major impact on the leather hardness is caused by nonionic surfactant concentration (Fig. 3, *e, f*). The increase of concentration from 15 to 24.95 g/l leads to an increase of material elasticity that is caused by aggregate stability increase of disperse systems and reduction of diffusion of the plasticizer and filler particles in the leather structure. In this connection, the increase of surfactant concentration above the critical value leads to a decrease of the plasticizing effect of fattening substances in the material structure. At the same time, the hardness of the leather material is increased.

To evaluate the influence of each factor on the output area of leather material $W(x_i)$, the single-factor dependencies for x_1, x_2, x_3 respectively were obtained by the formula (11).

$$W_i = (b_i + b_{ij}x_j)x_i + b_{ii} \cdot x_i^2. \qquad (11)$$

For this purpose, each plot contains only analyzed factor index effects and the other factors are fixed at zero level. The diagrams of obtained dependencies (Fig. 4) have a similar nature.

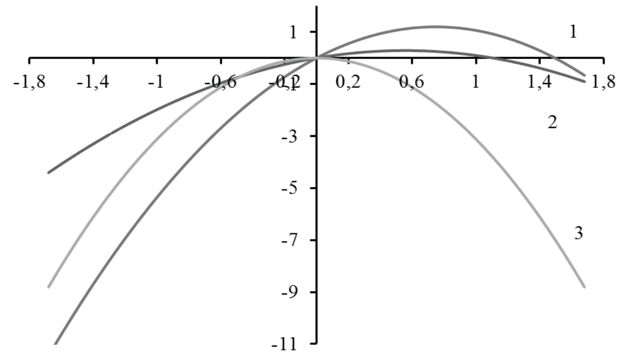

Fig. 4. Dependency for ingredients concentration output area, g/l: 1 – plasticizer, 2 – filler, 3 – nonionic surfactants

Thus, the leather material output area is mainly affected by the concentration of plasticizer and a nonionic surfactant. The stabilization of the filler concentration at zero level provides the optimal area of the process (Fig. 5).

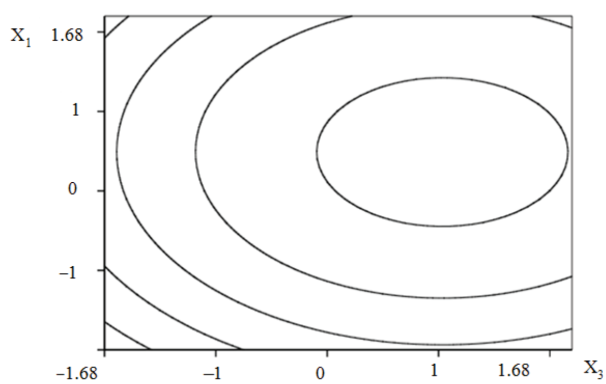

Fig. 5. Optimal area of semi-finished leather filling-plasticizing process

Fig. 5 shows that the optimal area of the filling-plasticizing process of semi-finished chrome-tanned leather produced from cattle raw material. The optimal area is located within the plasticizer and the nonionic surfactant concentration 84.2–123.1 and 21.8–26.6 g/l, respectively. The maximum output area of the leather material is achieved.

8. Discussion of the study results of the leather filling-plasticizing process

The obtained optimal area of the process of filling-plasticizing of the semi-finished leather allows to increase technological properties of the product, to decrease the stiffness of the leather material and to maximize the output area of the semi-finished product.

The output area of leather material with a minimum hardness of 19.0 sN is increased as compared to the control technology by 7.0 % using 115.3, 22.5, 23.9 concentrations in the studied solution (g/l) of plasticizer, modified filler and nonionic surfactant, respectively. Wherein fatty substances content in the finished material is 7.6 %, that complies with DSTU 3115-95 “Leather for Garments. General Specifications”.

The developed software module can be used to successfully optimize technological processes of forming various composite materials.

9. Conclusions

1. The second-order mathematical model is obtained on the basis of experimental data at the PJSC Chinbar (Ukraine). The model describes the semi-finished leather filling-plasticizing process adequately. The diagrams of single-factor dependencies for simulation models are obtained, and the influence of disperse system ingredients on basic technological properties of the leather material are analysed.

2. In order to convert multi-criteria task into single-criterion task using maximin convolution and Hooke-Jeeves method to solve the optimization task, a software module in the VBA environment (Visual Basic for Application) was developed. The developed module implements the considered computational schemes of a compromise solution. As a result, the optimum values of the factors that influence the semi-finished leather filling-plasticizing process conditions are found.

3. The optimum values of the concentration parameters of disperse system ingredients using multi-criteria constrained optimization of the filling- plasticizing process are found. The output area of leather material with a minimum hardness of 19.0 sN is increased as compared to the control technology by 7.0 % with 115.3, 22.5, 23.9 concentrations in solution (g/l) of plasticizer, modified filler and a nonionic surfactant, respectively. In addition, the content of fatty substances in the finished material is 7.6 % that meets the standard. The developed software module can successfully be applied for the optimization of technological processes of composite materials formation.

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