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OPTIMISATION OF THE PROCESS OF PREPARATION OF ACETYLSALICYLIC ACID COMPLEX WITH GLYCYRRHIZIC ACID

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Abstract
The process of preparation of acetylsalicylic acid complex with glycyrrhizic acid has been developed. Also, the optimal parameter conditions were found by the method of mathematical planning. The results of the quantitative assessment of the contribution of each of the factors selected as one of the tasks of process optimization by the method of mathematical planning of the experiment were analyzed.

Keywords Optimization, activated charcoal, sorbent,filtration, factors, regression equation, glycyrrhizicacid, licorice.

INTRODUCTION

Combinations of drugs, contribute to the prevention of toxic effects. However, an increase in the number of drugs taken by the patient can have an adverse effect on the liver, especially since NSAIDs themselves have a toxic effect on liver cells.In this regard, we have undertaken chemical and pharmacological studies of the complex of acetylsalicylic acid with glycyrrhizic acid, the aim of which was to create a new anti-inflammatory drug of prolonged action with high activity and fewer side effects on the basis of aspirin [1-3].

To determine the optimum conditions, as well as to quantify the influence of a number of factors on the process, to obtain its mathematical model, as well as to determine the conditions for obtaining the maximum yield of the product, we used the Box-

Wilson method [4-6].

Based on the analysis of the results of experiments and literature data, we selected the factors, established the main levels and intervals of variation for them.

Table 1

Factor levels and variation intervals

№	Levels of variation	X_1,hour	X_2, соот.	X_3, ^0C
1.	Lower level	2	2:10	30
2.	Main level	3	2:15	40
3.	Upper level	4	2:20	50
4.	Variation interval	1	0:5	10

X_1 – duration of reaction, hour.

X_2 – molar ratio of HA: ethanol, mol/mol

X_3 – reaction temeperature^{0}C.

Planning matrix

We conducted a full factorial experiment of type 23. The planning matrix and data of experiments are presented in Table 2.

In a three-neck flask, equipped with a stirrer, reflux condenser, water heating 164 glycyrrhizic acid are placed and 95% ethyl alcohol is poured. To the solution 36 g of acetylsalicylic acid is added and stirred under the conditions specified in the matrix.Then the solvent is evaporated in vacuum at 50 0C in a rotary evaporator. The used solvent is reapplied in the cycle. The residue is dried in vacuum for 2 hours at 50 0C and complex is obtained.

According to the experimental data, a mathematical model of the process was obtained, in which the effects of interaction of factors were also introduced:

Y = 86.31 - 0.57 X1 - 2.03 X2 + 5.61 X3

Table2

Planning matrix and experemental results

№ of experem, ent	X_0	X_1	X_2	X_3	$У_1$	$У_2$	$У_{ср.}$
1.	+	-	+	-	80.95	82,60	81,55
2.	+	+	+	-	80,82	83.42,0	82,12

3.	+	-	+	+	95.4	91,2	93,3
4.	+	+	+	+	79.3	81,0	80,15
5.	+	-	-	-	76.4	71.1	73,75
6.	+	+	-	-	86,7	84,0	85,35
7.	+	-	-	+	98,1	99,8	98,95
8.	+	+	-	+	98,49	92,17	95,33

According to the experimental data, we obtained the mathematical model of the process

$$Y = 86.31 - 0.57\,X1 - 2.03\,X2 + 5.61\,X3$$

Table 3

Statistical analysis

№ of experiment	$У_{ср}$	$\Delta У$	$\Delta У^2$	S_i^2	$\hat{Y}_{расч.}$	$\hat{Y}_{расч}-У_{ср}$	$(\hat{Y}-У_{ср})^2$
1.	81,55	1,05	1.10	2.20	79.24	2.31	5.34
2.	82,12	1.3	1.69	3.38	78.1	4.02	16.16
3.	93,30	2.1	4.41	8.82	90.46	2.84	8.07
4.	80,15	0.85	0.72	1.44	80.78	0.63	0.40
5.	73,75	2.65	7.02	14.04	73.3	0.45	0.20
6.	85,35	1,35	1.82	3.64	82.16	3.19	10.18
7.	98,95	0.85	0.72	1.44	95.52	3.43	11.77
8.	95,33	3.16	9.98	19.96	93.38	1.95	3.8
			27.46	54.92			55.92

The homogeneity of the variance was tested by the Cochran's criterion

$$G = \frac{S^2_{max}}{\sum^n_{i=1} Si^2} = \frac{19.96}{54.92} = 0.36$$

$G_{оп}$ = 0, 36< 0,6798= $G_{табл.}$ The variances are homogeneous.

We calculated: the mean variance of reproducibility

$$\sum^n_{i=1} Si^2 \qquad 54.92$$

$$S^2_{\{y\}} = \frac{}{N} = \frac{}{8} = 6.865$$

The adequacy variance is –

$$S^2_{ag} = \frac{2 \sum^n_i (\hat{y}-y_{cp})^2}{f} = \frac{2 * 55.92}{4} = 27.96$$

where f is the number of degrees of freedom:f= 4

The adequacy of the equation was checked by Fisher's criterion [6].

$$F = \frac{S^2_{ag}}{S^2_{\{y\}}} = \frac{27.96}{6.865} = 4.07$$

Ftable (4. 8) = 4, 8 Fop>Ftable The model is adequate.

We calculated the confidence interval using the formula$S^2_{\{y\}}$ 6.865

$$S^2_{\{B\}} = \frac{}{N} = \frac{}{8} = 0.858$$

$$S^2_{\{B\}} = \sqrt{0,858} = \pm 0.93$$

t table = 3.182

$$\pm B_i = t * S^2_{\{Bi\}} = 3,182 * 0,93 = 2.96$$

From comparing the confidence interval with the regression coefficients of the equation, it follows that the value of the optimisation parameter is mainly influenced by

X3 - reaction temperature

According to the obtained regression equation:

$У = 86,31 - 0,57 X_1 – 2,03 X_2 + 5,61 X_3$

In our case the coefficient X3 is significant (Table 4).

Table4
Significance of coefficients

Bi - values	Symbols	Bi - values	Results

86	>	2.96	Coefficient of significance
-0,57	<	2.96	Coefficient insignificant
-2,03	<	2.96	Coefficient insignificant
5,61	>	2.96	Coefficient of significance

One of the tasks of process optimisation by the method of mathematical planning of experiment is quantitative estimation of contribution of each of the selected factors.

As it can be seen from the table, the factor, X3 - reaction temperature turned out to be significant.

CONCLUSIONS

The conducted researches by the method of mathematical planning of experiment revealed the optimal conditions of the process.

- reaction temperature 45-50 0C,

- extraction time - 2 hours.

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