Integrated studies of the physical and mechanical properties of cotton fabric

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Abstract. The nature of the adhesive forces of the K-4 preparation is manifested in the occurrence of hydrogen bonds between molecules, as a substance with a sufficiently developed ability to form hydrogen bonds with cellulose. By the method of a one-factor experiment, the regularities of the influence of the concentration of the preparation alkaline-hydrolyzed PAN product on shrinkage, the total opening angle and weight gain of the fabric were revealed. It was found that the concentration of the preparation, equal to 75 g/l, provides the minimum shrinkage and rinsability of the sizing, as well as at the same time the largest value of TOA.

1 Introduction

At the moment, the predominant method of finishing fabrics for household use is treatment with compositions containing high molecular weight compounds. An important role in improving the consumer properties of finished fabrics is played by the properties of polymer preparations used in compositions for finishing [1-3].

Acrylic compounds and derivatives of water-soluble acrylic compounds are widely used in chemical finishing as finishing preparations [4]. Due to the presence of a double bond and due to substituted groups, derivatives of acrylic compounds are able to interact with functional groups of macromolecules of the fibrous material due to adhesive forces, as well as polymerize, forming flexible polymer films [5, 6]. Polyacrylonitrile latex in combination with cross-linking components is used as a finishing agent to give cotton fabrics low-shrink properties. The saponified polyacrylonitrile product is used as a sizing agent, thickener and a sizing component [7-10].

The purpose of this work is to establish the complex influence of the main factors on the physical and mechanical properties of cotton fabric in the process of final finishing [11].

Final finishing chemical operation based on the use of the K-4 preparation, which is an alkaline hydrolyzed PAN product. The nature of the adhesive forces of the K-4 preparation is manifested in the occurrence of hydrogen bonds between molecules, as a substance with a sufficiently developed ability to form hydrogen bonds with cellulose [12, 13].

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2 Materials and Methods

The authors studied the influence of the concentration of the preparation of the alkaline-hydrolyzed product PAN on the quality of the finish, expressed by shrinkage, by one-factor experiments. It was found that with an increase in the concentration of the finishing preparation of the alkaline-hydrolyzed product PAN, the tissue gain ranges from 5.6% to 11.4%. It should be noted that with the preparation concentration increase up to 75 g/l weight gain on fabric comprises 7.8% with minimum wash-off of the dressing, and at the same time the total opening angle of the finished fabric increases by 21% with a significant decrease in its shrinkage.

3 Results and Discussion

To establish the complex influence of various factors on the breaking load (Pp, N), elongation at break (lp, %) and conditional stiffness (in conditional, mcN·cm²), the mathematical method of experiment planning was applied [10], which is used to study multifactor systems. As a mathematical model, which is a polynomial, we take the form of the response function $y = f (x_1, x_2, ... x_n)$. For three factors, the polynomial of the first degree is expressed by the following equation (1):

$$y = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_{12} x_1 x_2 + b_{13} x_1 x_3 + b_{23} x_2 x_3 + b_{123} x_1 x_2 x_3 \quad (1)$$

As input factors, $X_1$ - K-4 (hydrolyzed PAN); $X_2$ - PVA (polyvinyl acetate); and $X_3$ - heat setting ($°\text{C}$) were taken. The levels and intervals of variation of the factors are presented in Table 1.

### Table 1. Levels and intervals of factors variation.

<table>
<thead>
<tr>
<th>#</th>
<th>Factors Code</th>
<th>Variation intervals</th>
<th>Levels of factors</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>K-4, g/l $X_1$</td>
<td>25</td>
<td>Upper +1 100 75 50</td>
</tr>
<tr>
<td>2</td>
<td>PVA, g/l $X_2$</td>
<td>10</td>
<td>Basic 0 45 35 25</td>
</tr>
<tr>
<td>3</td>
<td>Heat setting, $°\text{C}$ $X_3$</td>
<td>20</td>
<td>Lower -1 160 140 120</td>
</tr>
</tbody>
</table>

A full factorial experiment was used in the work, in which all possible combinations of N levels of factors (the number of experiments), determined by the following expression (2):

$$N = m^k \quad (2)$$

where, $m$ - is the number of levels of each factor and $k$ - is the number of factors.

For three factors, the full factorial experiment of type $2^3$ is represented by the matrix shown in Table 2. In order to eliminate errors, the experiments provided by the matrix are presented in a random sequence, i.e. according to the table of random numbers. The values of the coefficients of equation (1) for the breaking load Pp were found from the following dependencies:

1) on the warp of the fabric

   absolute term $b_0$: $b_0 = \frac{1}{N} \sum_{j=1}^{N} y_j = \frac{1804}{8} = 225.5$

   regression coefficient characterizing linear effects

   ...
The authors studied the influence of the concentration of the preparation of the alkaline-hydrolyzed product PAN on the quality of the finish, expressed by shrinkage, by one-factor experiments. It was found that with an increase in the concentration of the finishing preparation of the alkaline-hydrolyzed product PAN, the tissue gain ranges from 5.6% to 11.4%. It should be noted that with the preparation concentration increase up to 75 g/l weight gain on fabric comprises 7.8% with minimum wash-off of the dressing, and at the same time the total opening angle of the finished fabric increases by 21% with a significant decrease in its shrinkage.

3 Results and Discussion

To establish the complex influence of various factors on the breaking load \(P_p\), elongation at break (\(l_p\), %) and conditional stiffness (in conditional, \(m\)cN \(\cdot\) cm\(^2\)), the mathematical method of experiment planning was applied [10], which is used to study multifactor systems. As a mathematical model, which is a polynomial, we take the form of the response function

For three factors, the polynomial of the first degree is expressed by the following equation (1):

\[
b_i = \frac{1}{N} \sum_{j=1}^{N} x_{ij} y_j; \quad b_1 = 1.25; \quad b_2 = -22; \quad b_3 = 7.75;
\]

regression coefficients characterizing interaction effects

\[
b_{ij} = \frac{1}{N} \sum_{j=1}^{N} x_{ij} x_{ij} y_j; \quad b_{12} = -17.75; \quad b_{13} = -27.88; \quad b_{23} = 11.25; \quad b_{123} = -2;
\]

As a result of processing the experimental data, a regression equation with coded variables was obtained for the optimization parameter - breaking load \(P_p\) (3):

\[
y = 225.5 + 1.25 x_1 - 22 x_2 + 7.75 x_3 - 17.75 x_1 x_2 - 27.88 x_1 x_3 + 11.25 x_2 x_3 - 2 x_1 x_2 x_3 \quad (3)
\]

2) on the weft of the fabric:

\[
y = 188.25 + 1.25 x_1 + 13 x_2 - 2.75 x_3 + 11.5 x_1 x_2 + 27.75 x_1 x_3 - 41 x_2 x_3 + 11 x_1 x_2 x_3 \quad (4)
\]

To check the statistical significance of the coefficients of the regression equations (3) and (4), the absolute values of the coefficients /\(\Delta b_i/\), /\(\Delta b_{ij}/\) were compared with the confidence interval\(\Delta b_i\). Having previously calculated the variance of the regression coefficients using this equation (5)

\[
S^2 \{b_i\} = \frac{s^2}{N}, \quad (5)
\]

We find the confidence interval of the coefficients from the dependence:

\[
\Delta b_i = \pm tS\{b_i\} \quad (6)
\]

where, \(t\) is the table value of the Student's test, equal to 4.3 at a 5% significance level [6] and the number of degrees of freedom \(f = 2\).

The variance values \(S^2_y\) of the optimization parameter (breaking load \(P_p\)) for the investigated fabric on the warp and weft, respectively, are equal to: 16.5 and 9.5.

Taking into account dependencies (5) and (6) we get:

1) on the warp:

\[
S^2 \{b_i\} = \frac{s^2}{N} = \frac{16.5}{8} = 2.06 \quad \Delta b_i = \pm tS\{b_i\} = \pm 4.3 \cdot \sqrt{2.06} = \pm 6.18
\]

2) on the weft:

\[
S^2 \{b_i\} = 1.1875; \quad \Delta b_i = \pm 5.11
\]

The regression equations with statically significant coefficients are finally obtained (Table 2):

1) on the warp:

\[
y = 225.5 - 22 x_2 + 7.75 x_3 - 17.75 x_1 x_2 - 27.88 x_1 x_3 + 11.25 x_2 x_3 \quad (7)
\]

2) on the weft:

\[
y = 188.25 + 13 x_2 - 2.75 x_3 + 11.5 x_1 x_2 + 27.75 x_1 x_3 - 41 x_2 x_3 + 11 x_1 x_2 x_3 \quad (8)
\]
Table 2. Matrix of experiments’ planning and results.

<table>
<thead>
<tr>
<th>#</th>
<th>x₀</th>
<th>x₁</th>
<th>x₂</th>
<th>x₃</th>
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<th>x₁x₃</th>
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<table>
<thead>
<tr>
<th>Optimization parameters</th>
<th>Breaking load P₀</th>
<th>Elongation at breaking ñ₀, %</th>
<th>Conditional stiffness, micN·cm²</th>
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</thead>
<tbody>
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<td></td>
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<td>weft</td>
<td>warp</td>
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</tr>
<tr>
<td>8</td>
<td>198</td>
<td>219</td>
<td>32</td>
</tr>
</tbody>
</table>

To test the hypothesis of the adequacy of the model represented by equations (5) and (6), we find the variance of the adequacy below (9):

\[ S_{ad}^2 = \frac{\sum_{j=1}^{N}(y_j - \bar{y}_j)^2}{f} \]  

(9)

where, \( y_j \) - is the experimental value of the optimization parameter in the j-m experiment; \( \bar{y}_j \) - value of the optimization parameter in the j-m experiment, calculated by the equation (7) and (8); f is the number of degrees of freedom, \( f = N - (k + 1) \); and k-number of factors equal to 3.

The hypothesis of the adequacy of the model was tested using Fisher's F-test. For this, the calculated value of the criterion was found:

\[ F_p = \frac{S_{ad}^2}{S_j^2} = \frac{10}{16,5} = 0,606 \]

At a 5% significance level and the number of degrees of freedom for the numerator \( f_1 = 4 \) and for the denominator \( f_2 = 2 \), the tabular value of the criterion is \( F_r = 19.3 \). Since \( F_p < F_r \), the model represented by equation (7) is adequate. Let us check the adequacy of the model represented by equation (8):

\[ S_{ad}^2 = \frac{\sum_{j=1}^{g}(y_j - \bar{y}_l)^2}{N - (k + 1)} = \frac{23}{8 - (3 + 1)} = 5,75; \quad F_p = \frac{S_{ad}^2}{S_j^2} = \frac{5.75}{9,5} = 0,605 \]
Table 2. Matrix of experiments’ planning and results.

\[
\begin{array}{cccccc}
# & x_0 & x_1 & x_2 & x_3 & x_1 x_2 x_3 \\
1 & + & + & - & - & +
\end{array}
\]

Optimization parameters

- Breaking load $P_r$, Н
- Elongation at breaking $\ell p$, %
- Conditional stiffness $\sigma$, micN·cm²

<table>
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<tr>
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<th>warp</th>
<th>weft</th>
<th>warp</th>
<th>weft</th>
</tr>
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<td>21,5</td>
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<td>8</td>
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<td>219</td>
<td>32</td>
<td>22</td>
</tr>
</tbody>
</table>

To test the hypothesis of the adequacy of the model represented by equations (5) and (6), we find the variance of the adequacy below (9):

\[
(9)
\]

where,
- is the experimental value of the optimization parameter in the $j$-th experiment;
- value of the optimization parameter in the $j$-th experiment, calculated by the equation
  \[
  (7) \text{ and (8)};
  \]
- $f$ is the number of degrees of freedom, $f = N - (k + 1)$; and
- $k$-number of factors, equal to 3.

The hypothesis of the adequacy of the model was tested using Fisher's $F$-test. For this, the calculated value of the criterion was found:

At a 5% significance level and the number of degrees of freedom for the numerator $f_1 = 4$ and for the denominator $f_2 = 2$, the tabular value of the criterion is $F_\text{т} = 19.3$. Since $F_p < F_\text{т}$, the model represented by equation (7) is adequate. Let us check the adequacy of the model represented by equation (8):

Therefore, the model is also adequate ($F_p < F_\text{т}$). After processing the data of experimental studies for elongation at break (lp, %) in the same sequence, the following regression equations with coded variables were obtained (10, 11):

\[
y = 20,7 + 2,69 x_1 + 0,69 x_2 - 0,19 x_3 + 4,44 x_1 x_2 - 3,19 x_1 x_3 - 0,69 x_2 x_3 - 1,94 x_1 x_2 x_3 \\
\text{(warp) (10)}
\]

\[
y = 24,3 - 0,25 x_1 - x_2 - 2,63 x_3 + 1,19 x_1 x_2 - 0,5 x_1 x_3 - 0,88 x_2 x_3 + 1,17 x_1 x_2 x_3 \\
\text{(weft) (11)}
\]

Taking into account the values of the confidence interval of the coefficients for the warp and weft, respectively, equal to $\Delta b_1 = \pm 1.58$, $\Delta b_i = \pm 1.163$, we compose the regression equations with statically significant coefficients (12, 13):

\[
y = 20,7 + 2,69 x_1 + 4,44 x_1 x_2 - 3,19 x_1 x_3 - 1,94 x_1 x_2 x_3 \quad \text{(warp) (12)}
\]

\[
y = 24,3 + 2,63 x_3 + 1,19 x_1 x_2 + 1,13 x_1 x_2 x_3 \quad \text{(weft) (13)}
\]

The verification of the adequacy of the mathematical model described by equations (12) and (13) is confirmed by comparing the calculated and tabular values of Fisher's $F$-criterion, respectively, for the warp ($F_p < F_\text{т}$; 15.94 $< 19.3$) and the weft of the fabric ($F_p < F_\text{т}$; 14.12 $< 19.3$).

The processing of the results of experimental studies for the optimization parameter conditional stiffness $\sigma$, micN·cm² according to the above algorithm gave the following regression equations with coded variables (14, 15):

\[
y = 9210 - 1622 x_1 + 707 x_2 - 1055 x_3 + 875 x_1 x_2 + 999 x_1 x_3 + 1913 x_2 x_3 - 584 x_1 x_2 x_3 \\
\text{(warp) (14)}
\]

\[
y = 433 - 2141 x_1 + 827 x_2 + 1232 x_3 - 996 x_1 x_2 + 1280 x_1 x_3 - 546 x_1 x_2 x_3 \\
\text{(weft) (15)}
\]

which adequately describe the model according to the $F$-criterion of Fisher. Thus, for equation (15), the following relation was obtained:

\[
F_p = 15.2 < F_\text{т}, \quad F_\text{т} = 19.3
\]

Thus, we obtained regression equations (7), (8), (12), (13), (14) and (15) with coded variables, which allow us to estimate the degree and nature of the influence of input factors and their paired interactions on optimization parameters.

Verification and confirmation of the hypothesis of the adequacy of mathematical models in the form of the obtained regression equations allow us to switch to the method of steep ascent according to Box-Wilson [8] to achieve the optimum region of the considered response functions. They choose for one factor, and for the rest $i$ calculate it using this equation (16):

\[
\Delta_i = \Delta_i^{\frac{b_i \ell_i}{b_i \ell_i}}, \quad (16)
\]
where, $\Delta_1$ is the selected step of movement for the factor 1; $\Delta_i$ - movement step for the i-th factor; $b_{i1}$; $b_{i}$ - regression coefficients of the i-th and l-th factors; and $\varepsilon_{i1}$; $\varepsilon_{i}$ - intervals of variation of the i-th and l-th factors.

The movement along the gradient starts from the zero point (the main level of the factor). Having calculated the step of movement for each factor, the conditions of “mental” experiments are found. Some thought experiments are carried out to test the results of a steep ascent. A steep ascent is terminated if the optimization conditions are found, and also if the constraint on the factor makes further movement along the gradient unreasonable.

Let us make a steep ascent (Table 3) along the response surface for the optimization parameter - breaking load (on the warp). A steep ascent begins on condition $x_1 = x_2 = x_3 = 0$, which corresponds to the value of the input factors: 75; 35 and 140. Let us take the step of movement for the input factor $x_3$ equal to $\Delta_3 = 5^\circ C$. According to Equation 16, the step of movement was calculated for the factors $x_1$ and $x_2$ (Table 3):

$$\Delta_1 = \Delta_3 \frac{b_{1}\varepsilon_1}{b_{3}\varepsilon_3} = 5 \frac{(-1622) + 25}{1232 + 20} = -8.2285;$$

$$\Delta_2 = \Delta_3 \frac{b_{2}\varepsilon_2}{b_{3}\varepsilon_3} = 5 \frac{70 + 10}{1232 + 20} = 1.4347$$

<table>
<thead>
<tr>
<th>Name</th>
<th>$x_1$</th>
<th>$x_2$</th>
<th>$x_3$</th>
<th>$y$</th>
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<td>Basic level</td>
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<tr>
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<td>707</td>
<td>-1.055</td>
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</tr>
<tr>
<td>Variation interval $\varepsilon_{i1}$</td>
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<td>20</td>
<td>-</td>
</tr>
<tr>
<td>$b_{i}\varepsilon_{i1}$</td>
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<td>Experiment 9 realized</td>
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<td>Experiment 10 realized</td>
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The maximum breaking load value (on the warp) was obtained in experiment No. 10, which was $P_P = 311N$. Thus, the optimum region for breaking load was reached with the following values of the input factors: Preparation K-4 - 58.5 g/l and PVA- 37.8 g/l; Heat setting -150 $^\circ C$.

Thus, 11 tests were required in order to determine the optimal conditions for the final finishing of the cotton fabric, ensuring the maximum breaking load.

### 4 Conclusions

By the method of a one-factor experiment, the regularities of the influence of the concentration of the preparation of the alkaline-hydrolyzed product PAN on the shrinkage, the total opening angle and the weight gain of the finished tissue were revealed. It was found that the concentration of the drug, equal to 75 g/l, provides the minimum shrinkage and wash-off of the sizing, as well as at the same time the highest value of the total opening angle (TOA).

Using the method of multifactorial planning of the experiment, regression equations were obtained for various optimization parameters - breaking load, breaking elongation of the stiffness conditional depending on the input parameters. The steep ascent along the
response surface provided the best experimental conditions for optimizing the breaking force of the tissue.

References

5. P. Hwan, Ch. Hyung-Min, O. K. Wha, Cellulose 4, 3107-3119 (2014)