The influence of technological parameters on the hygroscopic properties of textiles

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Abstract

In this paper the method of wet chemistry used for hydrophobization of coarse calico surface was described. It was found that hydrophobicity of coarse calico surface is achieved by the formation of aluminum oxide on the surface. The effect of soap solution temperature, aluminum chloride solution concentration, sample treatment time in aluminum chloride solution and heat treatment temperature on water absorption, contact angle, capillary absorption and calico moisture content was studied. The dependences of hygroscopic properties on the process parameters were obtained. Capillary absorption and water absorption of the test samples increased with increasing concentration of aluminum chloride solution. An increase in the processing time of coarse calico samples in an aluminum chloride solution of lead to a decrease in the hydrophobicity of the samples. The characters of the dependences of hygroscopic properties on the temperature of heat treatment and the temperature of the soap solution are similar.

Introduction

Textile is a promising type of materials has been widely used in various fields of science and technology. Depending on the main properties he found applications in microelectronics, aircraft manufacturing, light textile industry, filtration, construction, etc. (Lauterbach and Jung 2005; Alagirusamy and Das 2010; Horrocks and Anand 2015; Asadi Miankafshe et al. 2019; Shi et al. 2020)

A wide range of textile applications requires specific surface and bulk properties. Various modification methods are used to modify the surface properties of textile materials. Modification methods include deposition of layers of various substances, creating relief on the surface, changing chemical bonds, adding modifiers in the form of microcapsules, etc. (Wasti and Ali Awan 2016; Emam and Abdelhameed 2017; Nadi et al. 2018; Asadi Miankafshe et al. 2019; Yao et al. 2019; Belhaj Khalifa and Ladhari 2020; Jadoun et al. 2020; Nejman et al. 2020; Şimşek and Karaman 2020; Khan 2017)

Modification and control of the hygroscopic properties of textile materials such as contact angle of surface contact, moisture content, capillary absorption, water absorption, etc. is of particular interest (Slater 1977).

The problem modifying the hygroscopic properties of textile materials has attracted considerable interest for a long time, so many well-known modification methods have been developed (Gong et al. 2017; Wei et al. 2020). Sol-gel method (SiO_2 and TiO_2 sols) (Yang et al. 2018), plasma chemical treatment (treatment in fluorine-containing plasma) (Chaivan et al. 2005; Jelil 2015), treatment in solutions and emulsions (Bu et al. 2019; Kazaryan et al. 2019), as well as combinations of these methods can be considered as the most popular (Montarsolo et al. 2013; Zhou et al. 2019).

Surface treatments in liquid solutions and emulsions are very effective in achieving hydrophobicity in various textile materials. Such processing allows obtaining surfaces with a wetting angle of more than 150 degrees (Wei et al. 2020). It should be noted that the wet chemistry method does not require complex

expensive equipment, which makes this method even more attractive. Solutions containing ions of aluminum, fluorine, silicon, titanium, zinc, etc. are used as reagents.

Despite the simplicity of the method, there is little research aimed at exploring the capabilities of the method and various dependencies. Therefore, the processing of textile materials in liquid solutions of various compositions to obtain a hydrophobic surface is promising. The influence of technological parameters on the contact angle, water absorption, moisture content, capillary absorption, etc., as well as the reasons for the increase in hydrophobicity are of scientific interest.

Currently, one of the important tasks in any study is to plan and statistically process the result of the experiments (Janetzki 2016; Dormidontova and Filatova 2018). The result of any experiment is a combination of a large number of measurements. The numerical values of the measured physical characteristics are random in most experiments, therefore to quantify the degree of variation of experimental data, measurements are duplicated.

Taking into account a number of possible limitations related to the technical equipment of laboratories, the cost of materials, etc., as well as the desire to reduce the time spent on a large number of experiments, as a result, there is always a small amount of experimental data on the basis of which it is necessary to make conclusions regarding the properties and behavior of the object under study. The use of Taguchi experiment planning reduces the number of experiments, which means reducing financial, time and labor costs (Davis and John 2018; Taguchi and Yokoyama 1993)

Therefore, the purpose of the work is to identify and study the influence of technological parameters of treatment by the method of wet chemistry of textile materials on hygroscopic properties, such as contact angle, water absorption, capillary absorption, and moisture content, using the method of scientific experimental planning.

Materials And Methods

Materials

Square pieces (10x10 mm) and strips (10x150 mm) of coarse calico with a density of 142 g/m² were used as samples. The use of samples of various shapes for the study of hygroscopic properties is a requirement of the corresponding Government Standard. The warp and weft fibers are alternately interconnected in a 1:1 pattern.

The samples were immersed for 20 minutes in a soap solution with a concentration of 35 g/l and a temperature of 70 ° C to purify the surface of undesirable impurities. Soap with pH = 7 was used to make the solution. Resistive heating was used to provide the desired solution temperature. The temperature was controlled by a thermocouple type K. The concentration of AlCl₃ solution was 35 g/l. AlCl₃ solution was prepared from AlCl₃ powder of the “analytical grade” (GOST 3759-75) purity class. Samples were placed in a solution of aluminum chloride at room temperature for 25 minutes.

The textile samples were dried using three layers of filter paper without wringing. After the samples were treated in solutions, they were subjected to an ironing press at a temperature of about 120 ° C. To maintain constant humidity after treatment, the samples were placed in a desiccator, in which phosphoric anhydride was used as desiccant. We gave in the article (Endiiarova et al. 2021) a more detailed description of the used treatment method.

The measurement

The hygroscopic properties of coarse calico were investigated after processing. The contact angle was determined by the method of sitting drop. Water absorption and material humidity were measured according to GOST 3816-81 and capillary absorption according to GOST 29104.11-91 The method of X-ray photoelectron spectroscopy (SPECS HAS 3500) was used to study the surface composition of textile materials.

Taguchi method

The use of Taguchi experiment planning allows to reduce the number of experiments by using matrices to organize all parameters that affect the process and levels of parameter change. The difference between the Taguchi method and the multifactorial plan is that it tests pairs of combinations, which allows you to determine the factors that have the greatest degree of influence on the studied object with a minimum number of experiments (Taguchi and Yokoyama 1993; Roy 2010)

Taguchi matrices can be built manually using special algorithms, and also compiled using the software package MiniTab. Each Taguchi matrix has its own name, which can be determined using the matrix (Roy 2010) selector. Knowing the number of parameters and the number of levels of parameter change, you can select the required matrix. The name of the matrix in the selector is as follows: L4, L8, L36, etc. The number shows the number of experiments that need to be carried out.

The main advantage of the Taguchi matrix method is the reduction in the number of necessary experiments to determine the degree of influence of various process parameters on the technological characteristics. The algorithm for constructing matrices using the Taguchi method is as follows: the experiment is represented in the form of a geometric n-dimensional space, where n is the number of factors whose degree of influence is studied. For example, if two factors are investigated the matrix of experiments can be represented as a square, if the three factors are in the form of a cube and so on. To construct a matrix, it is necessary to select such experiments that, when projecting points (dots, in turn, indicated planned experiments) on any face of a n-dimensional figure (square, cube, etc.), there will be a uniform distribution of points over the entire area of the face. Fig. 1 shows an example of the selection of experiments to study the degree of influence of three parameters, with a level of variation equal to five. Since three parameters are examined, a cube model is used.

As you can see (Fig. 1 (a-c)), each face is uniformly filled with spheres of different colors (the color refers to the levels of the studied parameters, since their number is five, then there are also five colors). In Fig. 1

(d) shows the distribution of the spheres in the volume of the cube. The number of axes depends on the number of parameters studied, and the number of points on the axes corresponds to the number of levels of each parameter.

In order to rank the varied parameters according to the degree of their influence on the value of the output parameter of the process, it is necessary to calculate the «signal/noise» ratio (SN) for each experiment. In the tasks of minimizing the value of the output parameter of the process, this ratio is calculated by the form (Taguchi and Yokoyama 1993):

$$SN_i = -10 \lg \left(\sum_{u=1}^{N_i} \frac{y_u^2}{N} \right). \quad (1)$$

In the case of maximizing the output characteristic, the expression for calculating the SN ratio is (Taguchi and Yokoyama 1993):

$$SN_i = -10 \lg \left[\frac{1}{N} \sum_{u=1}^{N_i} \frac{1}{y_u^2} \right]. \quad (2)$$

In the formulas (1-2), y – the value of the output parameter of the process, i – the number of the experiment, u – the ordinal number of the measurement of the value y in the i -experiment, N_i – the number of measurements of the output parameter in the i -th experiment.

After calculating the SN ratio for each experiment, the average SN is calculated for each factor and level. After SN values have been calculated for each factor and level, the R range is calculated using the form (3):

$$R = SN_{max} - SN_{min}. \quad (3)$$

By the value of R, according to the principle, the greater the difference, the greater the influence of the variable parameter on the output characteristic of the process, the ranking of the factors is performed.

Results And Discussions

To determine the nature and degree of influence of process parameters, such as soap solution temperature (T), aluminum chloride solution concentration (C(AlCl₃)), sample treatment time in aluminum chloride solution (t(AlCl₃)) and heat treatment temperature (T_{tt}), on water absorption, contact angle, capillary absorption and calico moisture content, a Taguchi experiment plan was developed as shown in Table 1.

Table 1. Taguchi experiment plan

Nº	T, °C	C(AlCl ₃), g/l	t(AlCl ₃), min	T _{tt} , °C
1	40	15	15	70
2	40	20	20	90
3	40	25	25	110
4	40	30	30	130
5	40	35	35	150
6	50	15	20	110
7	50	20	25	130
8	50	25	30	150
9	50	30	35	70
10	50	35	15	90
11	60	15	25	150
12	60	20	30	70
13	60	25	35	90
14	60	30	15	110
15	60	35	20	130
16	70	15	30	90
17	70	20	35	110
18	70	25	15	130
19	70	30	20	150
20	70	35	25	70
21	80	15	35	130
22	80	20	15	150
23	80	25	20	70
24	80	30	25	90
25	80	35	30	110

Each experiment from the plan was carried out three times. The results of the experiments are shown in Table 2. To simplify the presentation of results, the table shows the average values of the measured

properties. In graphs where there is no standard average error, the values have not changed.

Table 2. The results of the experiments

Number of experiments	Moisture content, %	Water absorption, %	Contact angle, °	Capillary absorption, mm
1	0,1	16,86 ± 0,33	149 ± 1	0,1
2	0,7 ± 0,48	21,81 ± 0,50	147 ± 1	0,1
3	14,4 ± 0,59	48,96 ± 1,94	153 ± 2	0,1
4	0,1	26,88 ± 0,15	156	0,1
5	2,6	21,75 ± 0,14	156 ± 3	0,1
6	0,1	0,55 ± 0,01	142 ± 3	2 ± 1,5
7	0,1	32,58 ± 1,47	154 ± 2	0,1
8	3 ± 0,15	32,08 ± 1,10	151 ± 3	6 ± 2,5
9	0,1	22,87 ± 1,26	143 ± 1	0,1
10	0,3	30,69 ± 0,24	161 ± 1	0,1
11	22,12 ± 2,05	33,51 ± 1,75	156 ± 2	6 ± 2,8
12	1,06 ± 0,57	27,59 ± 1,84	156 ± 4	0,1
13	0,66 ± 0,02	37,41 ± 3,18	143 ± 3	8,3 ± 4,5
14	1,98 ± 0,01	24,55 ± 1,95	160 ± 3	0,1
15	0,1	41,52 ± 0,29	151 ± 2	11 ± 6,5
16	2,88 ± 0,02	26,27 ± 0,24	146 ± 3	0,1
17	0,65	44,95 ± 0,38	139 ± 1	3
18	0,1	30,38 ± 0,18	150 ± 1	7 ± 2,5
19	0,1	30,51 ± 0,30	154 ± 3	5 ± 2,5
20	0,1	41,47 ± 0,37	152 ± 1	7 ± 2,5
21	12,96 ± 0,08	58,63 ± 0,49	152 ± 2	12 ± 9
22	1,71	41,3 ± 0,37	157 ± 3	9,7
23	4,94 ± 0,02	48,04 ± 0,34	157 ± 1	17 ± 8
24	6,82 ± 0,04	43,49 ± 0,34	156 ± 3	11,6 ± 2,5
25	0,1	28,8 ± 0,16	147 ± 1	13 ± 5

The results of measuring the hygroscopic properties of untreated coarse calico are shown in Table 3.

Table 3. Hygroscopic properties of untreated coarse calico

Propety	Value
Moisture content, %	80,00
Water absorption, %	48,24
Contact angle, °	0
Capillary absorption, mm	122,0

By comparing the data presented in Tables 2 and 3, it can be concluded that the coarse calico treatment according to the procedure described results in significant changes in the hydroscopic properties such as contact angle, moisture content and capillary absorption. With regard to water absorption, the effect of the treatment process seems almost negligible, probably because this characteristic is mainly determined by the inner porous structure of coarse calico. Thus, the obtained results show that it is the modification of the surface of coarse calico that is responsible for the observed changes in hygroscopic properties.

Based on the data presented in Table 2, the degree of influence of each process parameter (contact angle, capillary absorption, water absorption and moisture content) on the hygroscopic properties of coarse calico was calculated (Figure 2).

As one can see from Fig.2 the treatment time in the aluminum chloride solution as well as the heat treatment temperature of the samples after the treatment have the greatest effect on the contact angle within studied ranges of process parameters variation. The concentration of aluminum chloride solution has the least effect, from which it can be concluded that this parameter can be fixed and its effect can be neglected to study changes in contact angle.

Dominant influence of soap solution temperature on such a characteristic as the capillary absorption should be noted, the degree of influence of the remaining process parameters on this characteristic is approximately the same. As to the water absorption and moisture content, approximately the same degree of influence of the process parameters on these characteristics was found.

Based on the data presented in Table 3, it can be concluded that the optimal parameters for obtaining hydrophobic surfaces on coarse calico textile materials are as follows: soap solution temperature 40 °C, AlCl_3 solution concentration 35 g/l, processing time 35 minutes, heat treatment temperature 150 °C.

To understand the reasons for the observed significant changes of the hygroscopic properties of coarse calico after processing, the resulting samples were analyzed by X-ray photoelectron spectroscopy. The oxygen peak in the typical XPS spectrum obtained is represented in Fig. 3.

The results of decomposition analysis of the oxygen peak of the treated samples allowed us to conclude that it is characterized by complicated structure and includes corresponding oxygen bonded with carbon ($\approx 530,50\text{-}531,51$ eV) and hydrogen ($\approx 530,98\text{-}532,00$ eV), as well as oxygen bonded with aluminum ($\approx 529,98\text{-}531,80$ eV). The bonds with carbon correspond to the composition of the textile material, and the bonds of oxygen with aluminum indicate the presence of aluminum oxide on the surface of the treated samples (Moulder et al. 1992). Thus, one can suggest that the hydrophobicity of the surface of the coarse calico after treatment in a solution of AlCl_3 is explained by the formation of aluminum oxide films. The chemical reactions occurring during the process are discussed in detail in our previous article (Endiiarova et al. 2021). The results of SEM of the treated and untreated surface of coarse calico presented in Fig. 4 confirm this suggestion.

As one can see from Fig. 4a the untreated pristine coarse calico has a clearly woven texture consisting of smooth and near round fibers with diameter of about 10 microns and the treatment process does not change its structure (Figures 4b and 4c). The surface of untreated fibers is rather smooth (Fig. 4d), however the SEM images of the surface of treated fibers are the evidence of formation of some precipitates with composition most likely corresponding to aluminum oxide (Fig. 4e, 4f). It is interesting to note that the deposits formed during 15 minutes of treatment in AlCl_3 solution had clear grain structure (Fig. 4e) however an increase in treatment time leads to formation of [continuous layers](#) (Fig. 4f). This fact allows to explain essential role of the treatment time in the aluminum chloride solution on the contact angle by decreasing of untreated surface area of the fibers with the treatment time. The strong influence of the heat treatment temperature on contact angle is probably connected with densification of the deposits with the annealing temperature. As to capillary absorption which is determined by the surface concentration of the available pores and capillaries the great effect of the fiber treatment is caused by closing the pores by depositing the oxide layer.

Therefore, it can be concluded that aluminum oxide deposits forming during processing on the surface fibers of the coarse calico do not influence the original structure of the textile material however cause significant improvement of its hydrophobic properties.

Conclusions

The results of the study show the effectiveness of the coarse calico treatment process in aqueous aluminum chloride solution to improve basic hygroscopic properties such as contact angle, moisture content and capillary absorption. It has been shown that the formation of alumina deposits on the surface of coarse calico fibers is responsible for changing basic hygroscopic properties

Declarations

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Availability of data and material. The information is not applicable.

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Figure 2

Histograms of the degree of influence of technological parameters on the hygroscopic properties of coarse calico

Figure 3

X-Ray photoelectron spectroscopy results - oxygen (O 1S) peak decomposition

Figure 4

SEM images of the coarse calico surface. a, d - before treatment; b, e – after treatment 15 min (experiment N° 10); c, f - after treatment 35 min (experiment N° 5)