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SCIENTIFIC INVESTIGATION OF THE ENHANCED REACTIVITY OF COTTON CELLULOSE FOR CHEMICAL INDUSTRY PROCESSING

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Аннотация: Реакционная способность хлопковой целлюлозы во время химической переработке значительно ниже по сравнению с целлюлозами, полученных из других целлюлозосодержащих растений. Увеличение реакционной способности хлопковой целлюлозы улучшает качество получаемого продукта, а также повышает производительность труда.

Целью данной работы является повышения реакционной способности хлопковой целлюлозы для химической переработки, улучшение качества получаемого продукта и повышении реакции.

В данной работе использовались методы определения состава и структуры образцов хлопковой целлюлозы. Изучена реакционная способность полученных образцов к ацетилированию.

Повышение реакционной способности оценивали по уменьшению кристаллических областей на основе дифрактограмм, которые были записаны на управляемом компьютерном приборе ХРД-6100 (Шумадзу, Япония).

Предложен способ повышения реакционной способности хлопковой целлюлозы путем обработки ее электрическими зарядами высокого напряжения, при котором резко снижаются кристаллические участки, которые снижают реакционную активность.

Найдены следующие оптимальные параметры активации хлопковой целлюлозы напряжение – 11-13 кВ; количество импульсов – 22-24; требуемая емкость конденсатора – 0,6 мкФ

Благодаря использованию данного метода удалось достичь повышения реакционной способности хлопковой целлюлозы к химической переработки, в частности к ацетилированию. Это позволило к улучшению показателей качества ацетатов целлюлозы, повышению производительности готовой продукции засчет улучшения фильтрации ацетилцеллюлозы.

Ключевые слова:Хлопковой линт целлюлоза, электрический заряд, рентгеноструктурный анализ, реакционная способность, контроль.

Abstract: The reactivity of cotton pulp during chemical processing is significantly lower compared to celluloses obtained from other cellulose-containing plants. An increase in the reactivity of cotton cellulose improves the quality of the resulting product, and also increases labor productivity.

The aim of this work is to increase the reactivity of cotton cellulose for chemical processing, improve the quality of the resulting product and increase the reaction.

In this work, methods were used to determine the composition and structure of cotton cellulose samples. The reactivity of the obtained samples to acetylation was studied.

The increase in reactivity was evaluated by the reduction of crystalline regions based on diffractograms that were recorded on an XPD-6100 controlled computer instrument (Shumazu, Japan).

A method is proposed for increasing the reactivity of cotton cellulose by treating it with high-voltage electric charges, in which crystalline regions sharply decrease, which reduce the reactivity.

The following optimal activation parameters of cotton cellulose were found: voltage - 11-13 kV; the number of pulses - 22-24; required capacitance - 0.6 μF.

Thanks to the use of this method, it was possible to increase the reactivity of cotton cellulose to chemical processing, in particular to acetylation. This allowed us to improve the quality indicators of cellulose acetate, increase the productivity of the finished product due to improved filtration of cellulose acetate.

Key words: Cotton lint cellulose, electric charge, X-ray diffraction analysis, reactivity, control.

Introduction: Cotton fiber is crimped in nature, therefore, in dry and wet states, they quickly gather into lumps and nodules, forming flagella and ropes, are also enveloped with weed impurities and become difficult to clean [1-3].

Due to the above specifics, cotton fiber requires additional mechanical processing - chopping or chopping. For fiber grinding, rolls, conical and disk mills are mainly used [4-5].

A number of works provide descriptions of various methods of cleaning lint, boiling, etc. in order to obtain uniformly pure cotton cellulose, intended mainly for chemical processing such as acetates, nitrates and other cellulose ethers [6-7].

The reactivity of cotton cellulose during chemical processing is significantly lower than that of other types of cellulose, since its structure consists of crystalline and amorphous sites. During esterification, chemical reagents easily react with functional groups in the amorphous region, however, these reagents are difficult to penetrate into crystalline regions. As a result, part of the cellulose enters into chemical reactions, while the other does not. This leads to the shutdown of the production line due to the difficulties of passage through the filter obtained cellulose ether.

A study of the scientific and technical literature in the field of increasing the reactivity of cotton pulp revealed a number of works aimed at solving this problem. For example, a method has been proposed, the essence of which is as follows: cellulose swollen in water is frozen at a temperature of –15–20 °C followed by thawing, which ultimately leads to a certain decrease in crystalline regions in the structure. By treating cotton cellulose with nitrogen containing substances [8–16], as well as by partially esterifying cotton cellulose, a slight increase in the distance between cellulose macromolecules was achieved [17].

In the process of alkaline cooking of cotton cellulose, as well as with other cooking methods, along with the release of cotton cellulose, its structural changes also occur. When choosing the optimal regime, it is necessary to take into account changes in the macro- and microstructure of cellulose fiber depending on the production conditions [18–20].

Methods. In the work, physicochemical methods were used to determine the quality indicators of the obtained cotton pulp from various production conditions.

The characteristics of acetylation of cotton pulp were determined by the method proposed by the French company Speyshen, which is determined by the product of viscosity by filterability divided by 1000.

The obtained cellulose samples were processed into triacetates, where the kinetics of acetylation was studied.

The study of changes in the crystalline and amorphous regions of cotton cellulose after treatment with electric charges was carried out by identifying samples based on diffractograms that were recorded on a XRD-6100 apparatus (Shimadzu, Japan) controlled by a computer.

Results and Discussion. A series of studies have been conducted on the activation of cotton cellulose by electric charge, with the aim of reducing the crystalline regions that reduce the reactivity of cotton cellulose.

Samples of cotton cellulose without activation control (1), wet cotton cellulose (2) and cotton cellulose treated with electrolyte (3) were prepared for the study. A solution of ammonium carbonate was chosen as the electrolyte.

Using X-ray diffraction analysis, structural changes in cotton cellulose were studied before and after electrical treatment of the samples under study. Structural changes in cellulose samples, as well as determining the degree of crystallinity / SC / cellulose (SC) were studied by the most common X-ray method, which is based on a comparison of the scattering intensity of X-rays in the crystalline and amorphous regions.

According to the results of studies, it was found that the maximum SC of cotton cellulose is observed in the control sample. At the same time, when processing with an electric charge without an electrolyte and with an electrolyte, partial destruction of intermolecular hydrogen bonds is observed.

According to the diffraction pattern analysis (Fig. 1-3), it is possible to assess the degree of crystallinity of the obtained samples in comparison with the reference and initial microcrystalline cellulose (samples 1-3).

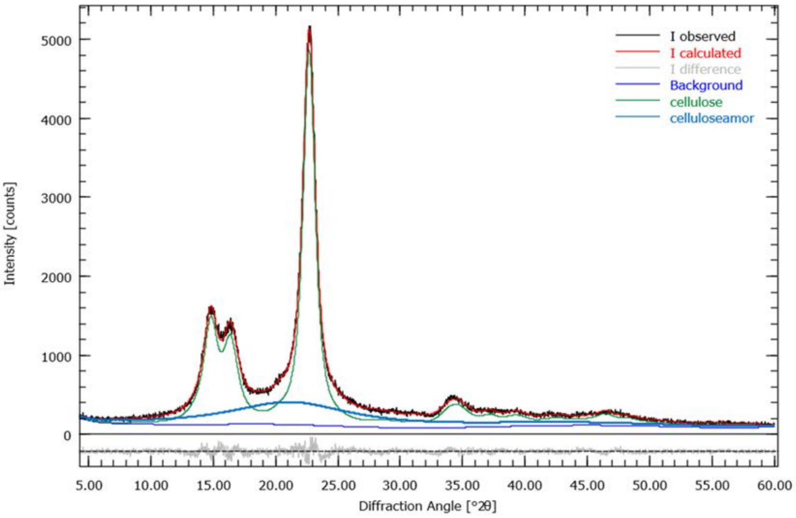


Fig. 1. X-ray diffraction pattern of sample 1.

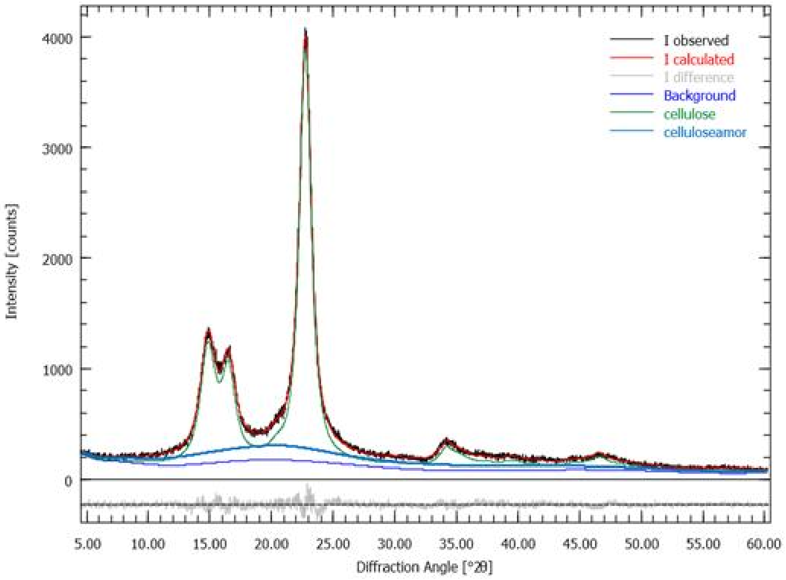


Fig. 2. X-ray diffraction pattern of sample 2.

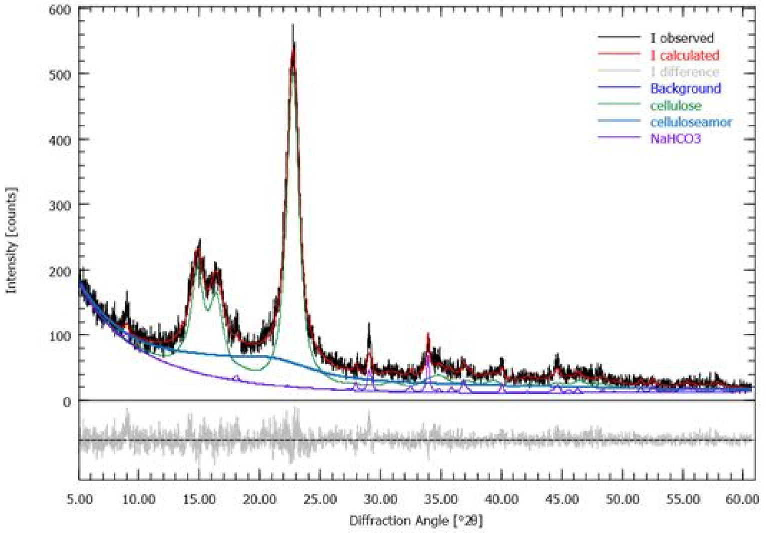


Fig. 3. X-ray diffraction pattern of sample 3.

Since amorphization or a decrease in crystallite size leads to expansion of the diffraction pattern peaks, integration of the most intense peaks of crystalline cellulose and summation of the integral peaks, taking into account the background and amorphous peaks, makes it possible to calculate the cellulose crystallinity index based on the data of X-ray diffractograms (Table 1).

Table 1
CRYSTALLINE CALCULATION DATA FOR CELLULOSE SAMPLES BASED ON X-RAY PHASE ANALYSIS

Samples	Integrals of 4 crystalline cellulose peaks	The sum of the integrals of crystalline peaks of cellulose	X-ray diffractogram integral (crystalline, amorphous peaks and background)	Cellulose crystallinity index (sum (integrals of crystalline peaks of cellulose / integral of x-ray diffraction patterns) * 100)
Sample-1	56.78942	282.47305	423.34631	66.72
Sample-2	54.52621	268.3713	410.37879	65.40
Sample-3	67.56028	303.29433	668.62411	45.36

In addition, the appearance of 3 peaks of Trisodium carbonate (bicarbonate) * 2H2O in the X-ray diffraction pattern of the sample as separate crystalline peaks with sufficient intensity for calculation allows us to conclude that a certain amount of bicarbonate impurity is present in the sample. For sample 3, an abnormal decrease in the crystallinity index is observed, despite the fact that the presence of about 5% impurity leads to the appearance of additional crystalline peaks.

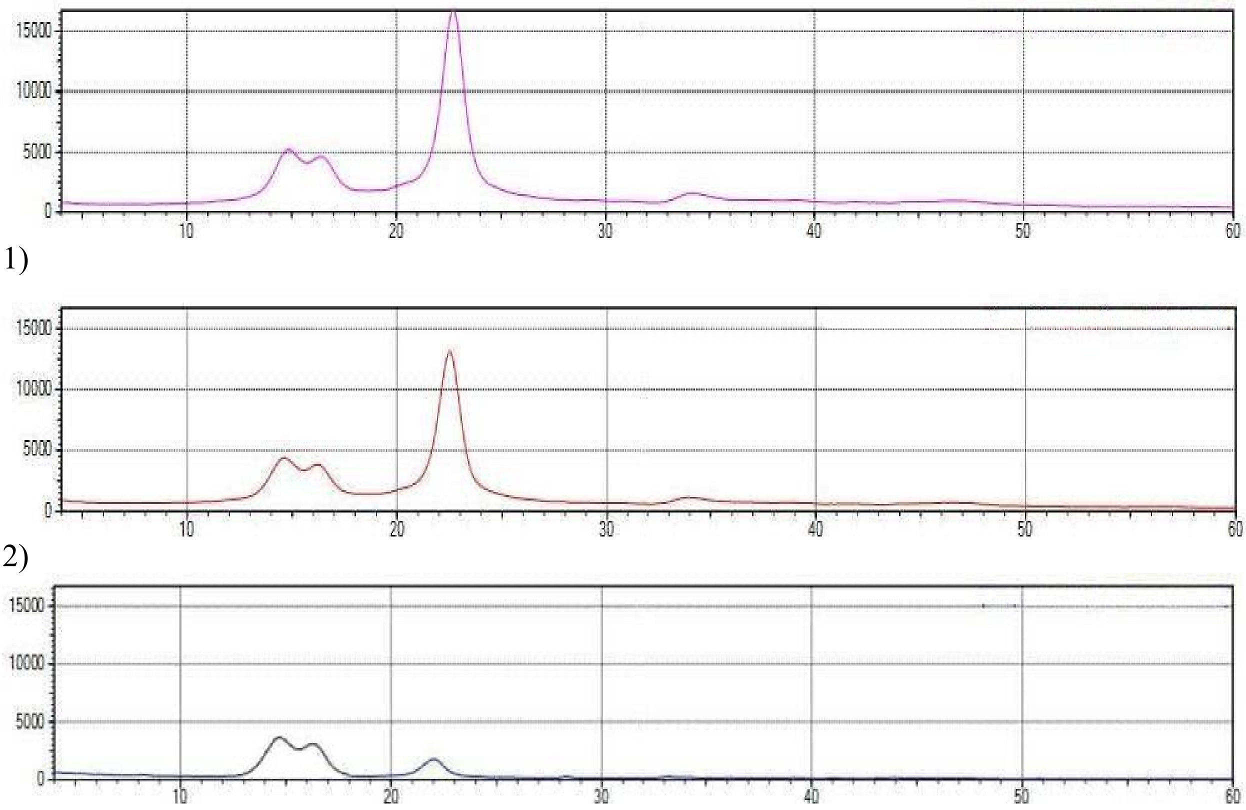
The use of the Rietveld method for analyzing the diffraction pattern of sample 2, using the least squares method to refine and approximate the theoretical line of the entire profile of the diffractogram to its experimental profile, allows us to analyze the crystal structure and obtain reliable results when overlapping reflections from the crystalline phases of microcrystalline cellulose (MK) and bicarbonate.

In the table. Figure 2 shows the percentage of MK cellulose and amorphous cellulose based on Rietveld analysis. Based on the data on the relative standard deviation of RNO (%), which does not exceed 5-9%, it can be concluded with a high degree of certainty that sample 3 has a more amorphous structure and a smaller crystallite size compared to the initial and reference MK cellulose, t. e. there is a decrease in crystallinity (MK cellulose content) from 62-67% to 49% (about 20%).

Table 2

RITVELD ANALYSIS DATA FOR CELLULOSE SAMPLES

Sample Components	%	RNO (%)
Sample 1		
MK-cellulose	67.1	9.1
Amorphous cellulose	32.9	9.1
Sample 2		
MK-cellulose	62.2	5.4
Amorphous cellulose	37.8	5.4
Sample 3		
MK-cellulose	48.5	2.4
Amorphous cellulose	46.7	2.4
Ammonium carbonate (bicarbonate) *$2H_2O$	4.79	0.26



1)

2)

3)

Fig. 4. The structure of cotton cellulose according to x-ray analysis.
Sample 1. Control cotton cellulose.
Sample 2. Wet cotton cellulose.
Sample 3. Cotton cellulose moistened in an electrolyte.

Identification of the samples was carried out on the basis of diffraction patterns, which were recorded on an XRD-6100 apparatus (Shimadzu, Japan), controlled by a computer. CuKα radiation (β filter, Ni, 1.54178 current and tube voltage modes of 30 mA, 30 kV) and a constant detector rotation speed of 4 deg / min in increments of 0.02 deg were used. (ω / 2θ coupling), and the scanning angle varied from 4 to 80 ° (Fig. 4).

Thus, we can conclude that during the treatment of cellulose with an electric pulse, sample-2, the structure of cellulose practically does not change, i.e. cellulose is not amorphized and is similar to control sample-1.

After chemical treatment with a bicarbonate salt followed by an electric pulse, the cellulose is amorphized and the peak in region 220 disappears, which indicates the complete disappearance of the crystalline sections of cellulose sample-3.

Conclusions. A method is proposed for increasing the reactivity of cotton cellulose to etherification by treatment with an electric charge, as a result of which the crystalline sections of cotton cellulose are sharply reduced, due to which the quality indicators of cellulose ethers are much improved, and productivity is also increased several times.

The dependence of the reactivity of cotton cellulose on the voltage, number of pulses and capacitance of the capacitor is determined. The following optimal parameters were determined experimentally:

the discharge voltage is 11-13 kV, the number of pulses is 22-24 and the capacitance of the capacitor is 0.6 μF, respectively.

According to the research results, the highest quality index of acetate films and fibers was observed for cellulose acetate, obtained on the basis of cotton cellulose, moistened with an electrolyte and treated with an electric charge in the optimal mode.

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