
Increasing Reactivity Cotton Cellulose Intended For Acetylation

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ABSTRACT

The aim of this work is to study the possibility of replacing wood pulp imported from foreign countries with domestic cotton cellulose by increasing the reactivity of cotton cellulose for chemical processing, improving the quality of the resulting product and increasing the reaction rate.

Key words: Cotton cellulose, electric charge, X-ray diffraction analysis, reactivity, amorphous, crystalline areas.

Introduction: Cotton fiber is crimped in nature, therefore, in dry and wet conditions, they quickly gather into lumps and nodules, forming flagella and ropes, as well as enveloped with weed impurities and become difficult to clean.

Due to the above specifics, cotton fiber requires additional mechanical processing - chopping, chopping etc. For fiber grinding, rolls, conical and disk mills are mainly used [1].

A number of works [2-3] provide descriptions of various methods of cleaning lint, boiling etc. in order to obtain uniformly pure cotton cellulose intended for chemical processing and for acetates, nitrates and other cellulose ethers.

The reactivity of cotton cellulose during chemical processing is significantly lower than that of other types of cellulose, since the structure of cotton cellulose consists of crystalline and amorphous sites. 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 a shutdown of the processing line due to the difficulty of passage through the cellulose ether filters.

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 $-15-20^{\circ}\text{C}$, followed by thawing, which ultimately leads to a decrease in crystalline regions in the structure [4]. By treating cotton cellulose with nitrogen containing substances [5-7],

as well as by partially esterifying cotton cellulose, a slight increase in the distance between cellulose macromolecules was achieved [8].

In the process of cotton pulp obtained, along with the release of cotton pulp, 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 conditions of production.

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

The characteristics of acetylation of cotton cellulose were determined by the method proposed by the French company Speysen, 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 also studied.

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

A number 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), with wet cotton cellulose (2) and electrolyte treatment (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, 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 analysis data (Fig. 1-3), it is possible to assess the degree of crystallinity of the obtained samples in comparison with the original cellulose (samples 1-3).

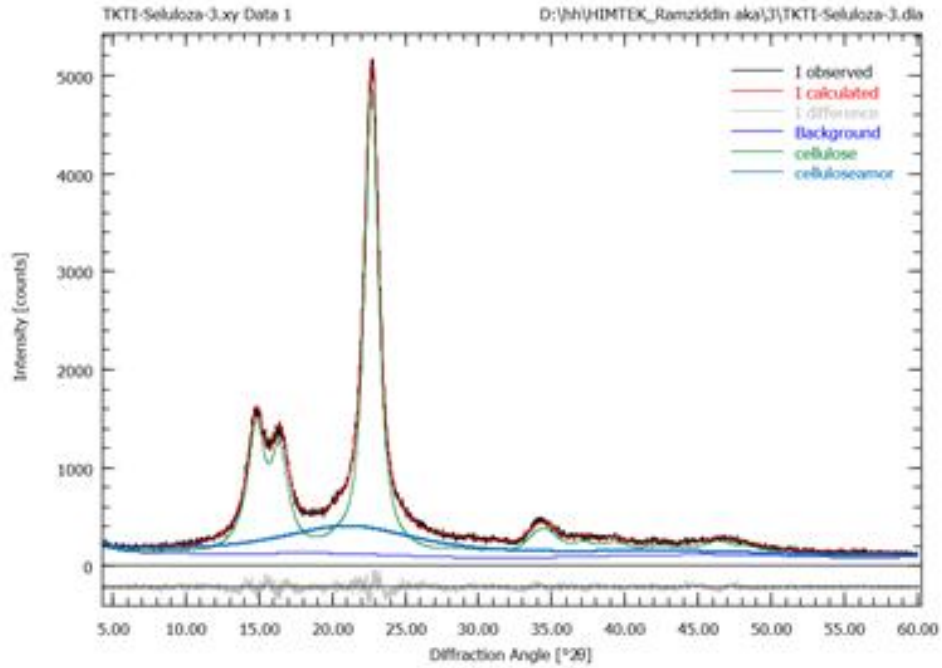


Fig. 1. X-ray diffraction pattern of sample 1.

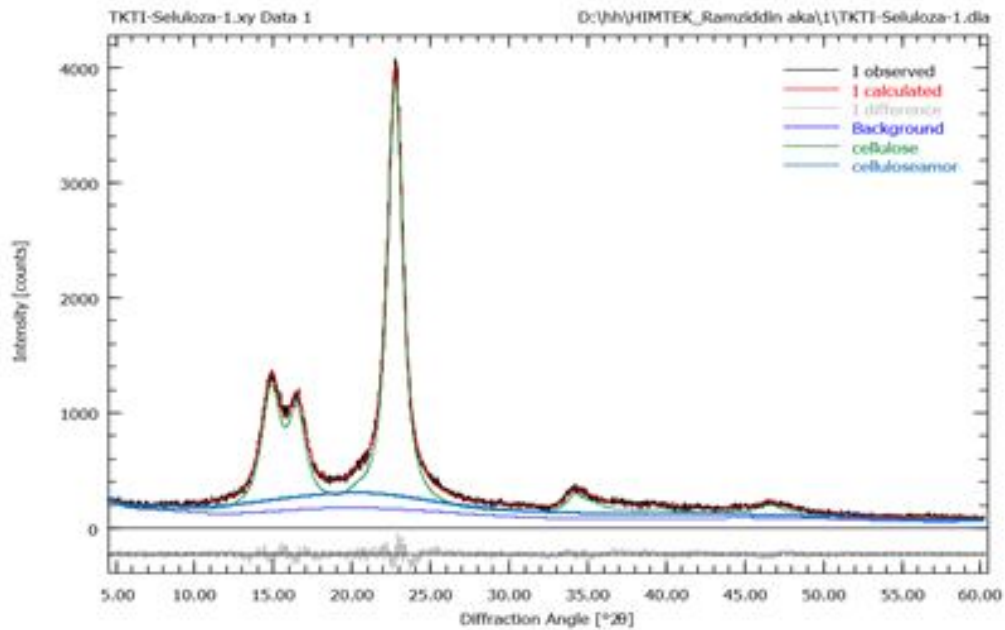


Fig. 2. X-ray diffraction pattern of sample 2.

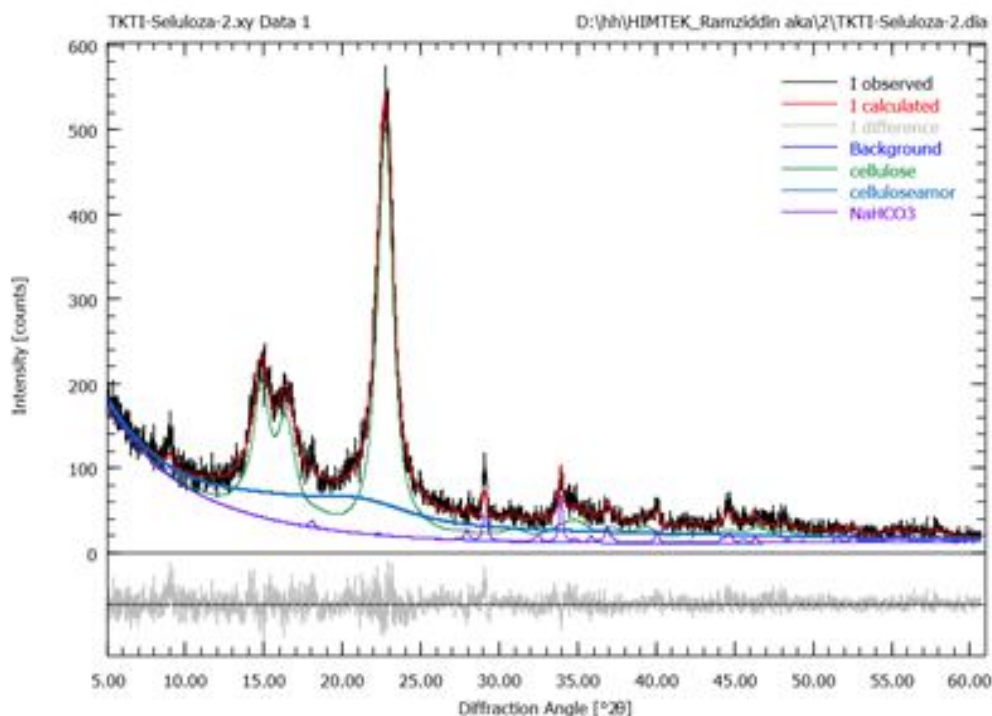


Fig. 3. X-ray diffraction pattern of sample 3.

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 diffraction patterns (Table 1).

Table 1

Calculation data of crystallinity of cellulose samples based on x-ray phase analysis

Sam ples	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 of 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.

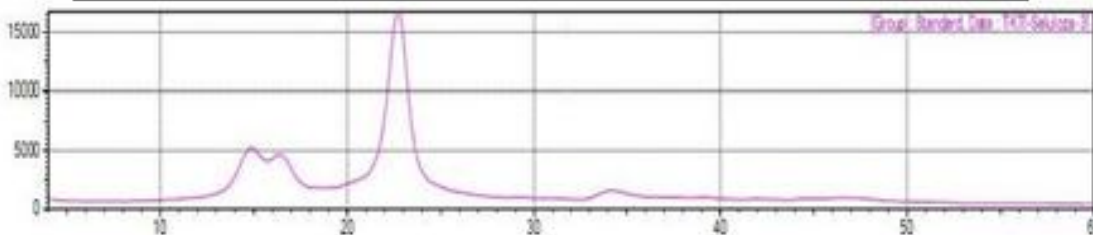
The use of the Rietveld method for analyzing the diffraction pattern of sample 2, using the least squares method to refine and approximate the theoretical line of the entire profile of the diffractogram to its experimental profile, allows us to analyze the crystal structure and obtain reliable results 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%).

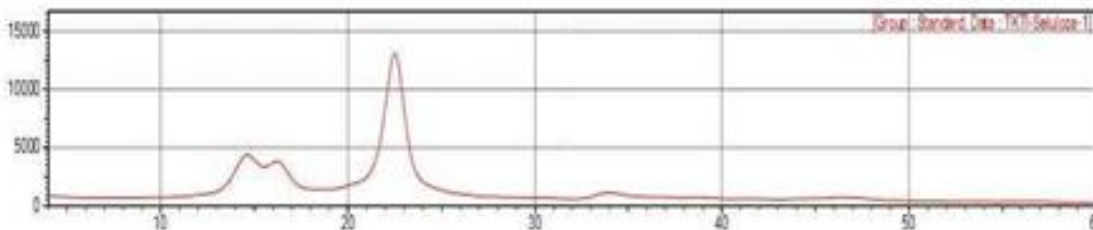
Table 2

Ritveld analysis data for cellulose samples

Sample C omponents	%	RNO (%)
Sample 1		
MK-celluloſe	67.1	9.1
Amorphous cellulose	32.9	9.1
Sample 2		
MK-celluloſe	48.5	2.4
Amorphous cellulose	46.7	2.4
Trisodium carbonate (bicarbonate) * 2H ₂ O	4.79	0.26
Sample 3		
MK-celluloſe	62.2	5.4
Amorphous cellulose	37.8	5.4



A)



B)

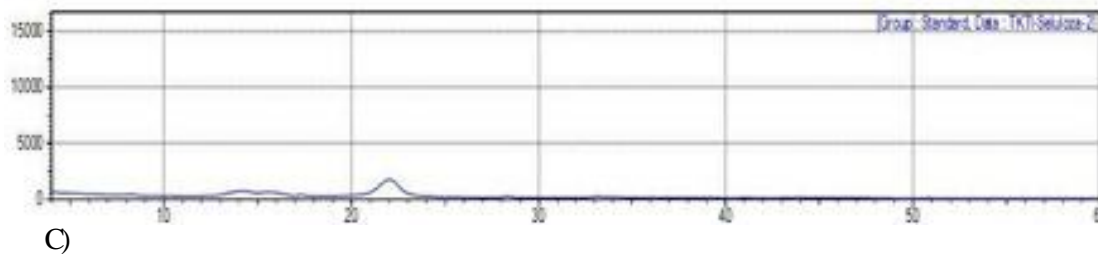


Fig. 4. Structures of cotton cellulose x-ray analysis.

- A) Control cellulose, B) Wet cotton cellulose,
C) 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. CuK α 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 processing 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.

Conclusion. A method is proposed for increasing the reactivity of cotton cellulose, by processing with an electric charge as a result of which the quality indicators of the resulting products are improved.

Determination of the dependence of cellulose reactivity on voltage, number of pulses and capacitor capacitance. The following optimal parameters were established experimentally: discharge voltage 11-13 kV, number of pulses 22-24 and capacitor capacitance 0.6 μ F, respectively.

According to research results, the highest quality index of acetate films and fibers was observed for cellulose triacetate, 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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