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RESEARCH OF INCREASING THE REACTIVITY COTTON CELLULOSE INTENDED FOR CHEMISTRY

R.S. Sayfutdinov, U.D. Mukhitdinov, N.M. Eshpulatov

Abstract: *The reactivity of cotton pulp during chemical processing is significantly lower compared to celluloses obtained from other cellulose-containing plants. Increasing the reactivity of cotton cellulose improves the quality of the products based on it, and also increases labor productivity.*

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.

In this work, we used methods for determining the composition and structure of cotton cellulose samples. The reactivity of the obtained samples to acetylation after treatment with electric charges was studied. An increase in reactivity was evaluated by a decrease in crystalline areas based on diffract grams that were recorded on a computer-controlled XRD-6100 apparatus (Shimadzu, Japan). A method is proposed for increasing the reactivity of cotton cellulose by treating it with high voltage electric charges. To reduce the crystalline areas of cotton cellulose, it was activated by an electric charge, which led to an increase in the reactivity of cotton cellulose. The optimal voltage limits are found, the number of pulses and the required capacitance of the capacitor, at which the maximum values of the reactive activity of cotton cellulose are achieved.

Key words:*Oxygen-alkaline cooking, cotton lint cellulose, electric charge treatment, X-ray diffraction analysis, reactivity, control.*

Introduction. Cotton fiber by its nature is crimped, therefore, in dry and wet conditions, they quickly assemble 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 or chopping. For grinding fibers, rolls, conical and disk mills are mainly used. [1-3].

A number of works [4-8] provide descriptions of various methods of cleaning lint, cooking and acidification in order to obtain uniformly pure cotton cellulose intended for chemical processing, in particular cellulose ethers.

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. 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 difficulties in passing through the filter (cellulose ethers).

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 decrease in crystalline regions in the structure [9]. By treating cotton cellulose with nitrogen containing

substances [8, 10–14], as well as by partially esterifying cotton cellulose, a slight increase in the distance between cellulose macromolecules was achieved [15].

In the process of oxygen-alkaline cooking, as well as with other cooking methods, along with the refinement of natural cellulose, its structural changes occur. Research on the changes in the macro- and microstructure of cellulose fiber depending on the cooking conditions is necessary when choosing the optimal mode.

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

The characteristics of acetylation of cotton pulp were determined by the method proposed by the French company Speyshen, which is determined by multiplying the viscosity by filterability and 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 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).

Results and discussion. We have conducted studies on the activation of cotton cellulose by electric charge in order to reduce the crystalline areas that contributed to the decrease in the reactivity of cotton cellulose.

For the study, samples of cotton pulp were prepared without activation control (1), with water treatment (2) and electrolyte treatment (3). As the electrolyte, a solution of ammonium carbonate salt was chosen.

Visual information about the structural changes in cotton cellulose after treatment with an electric charge in the samples under study is provided by X-ray diffraction analysis.

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 intensity of X-ray scattering in 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 by an electric charge without an electrolyte and with an electrolyte, partial destruction of intermolecular hydrogen bonds is observed.

According to x-ray phase 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).

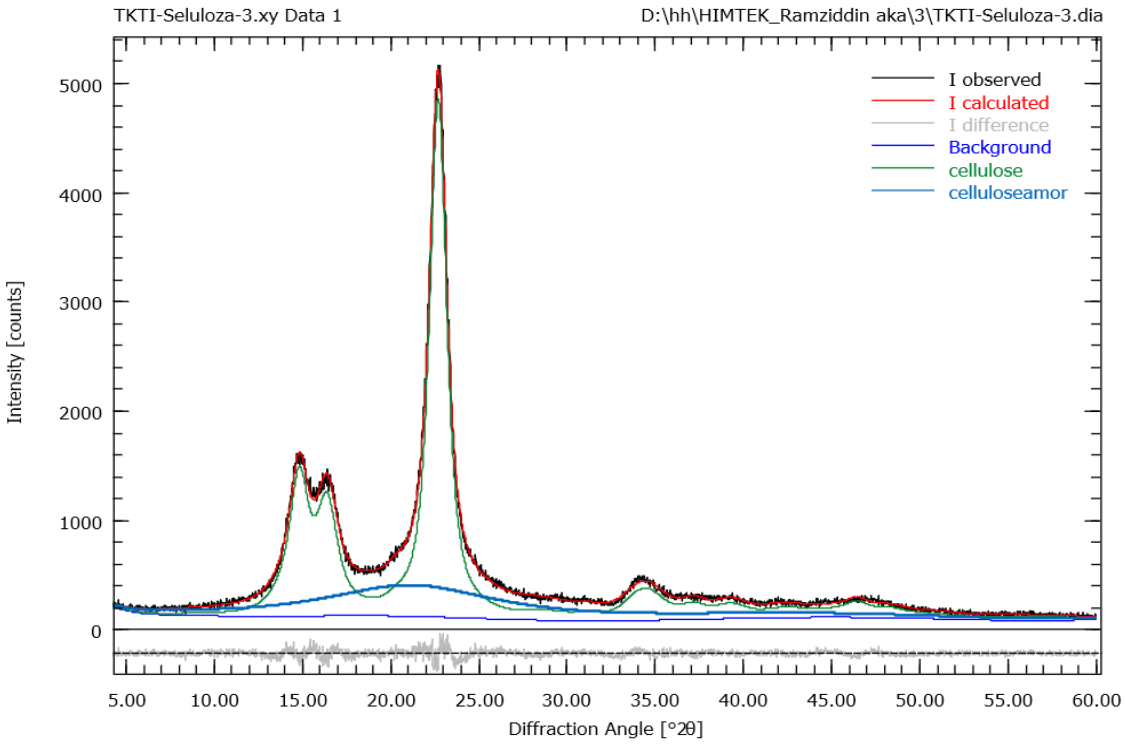


Fig. 1.X-ray diffraction pattern of sample 1

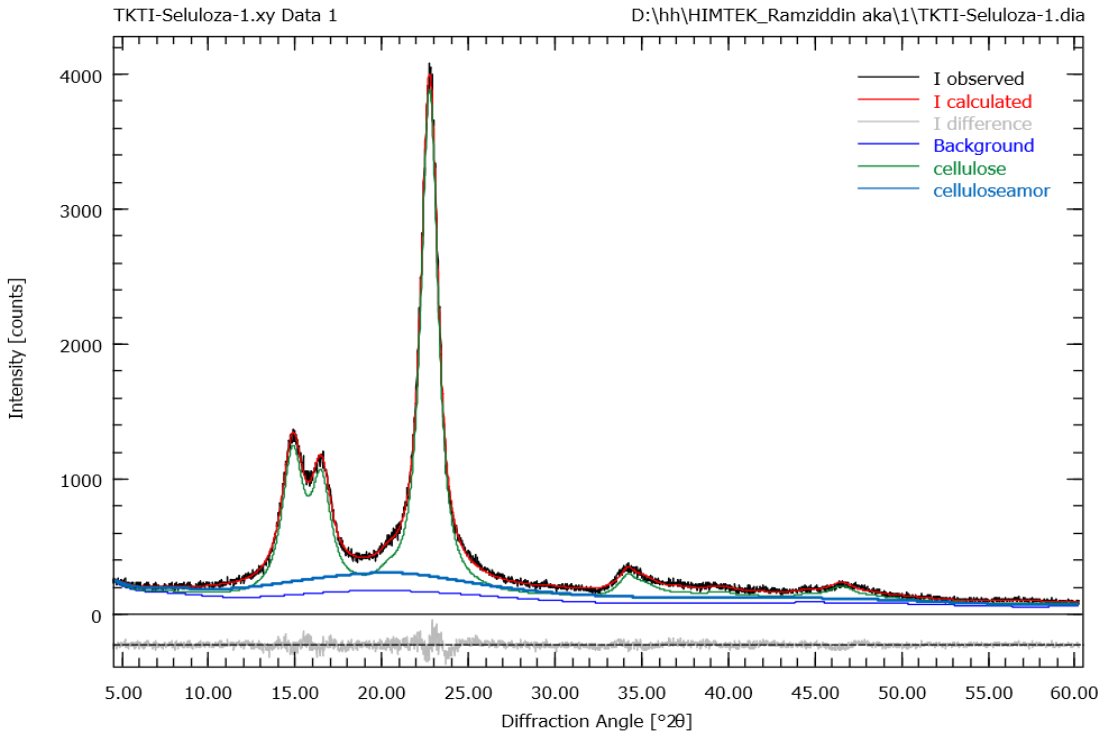


Fig. 2.X-ray diffraction pattern of sample 2

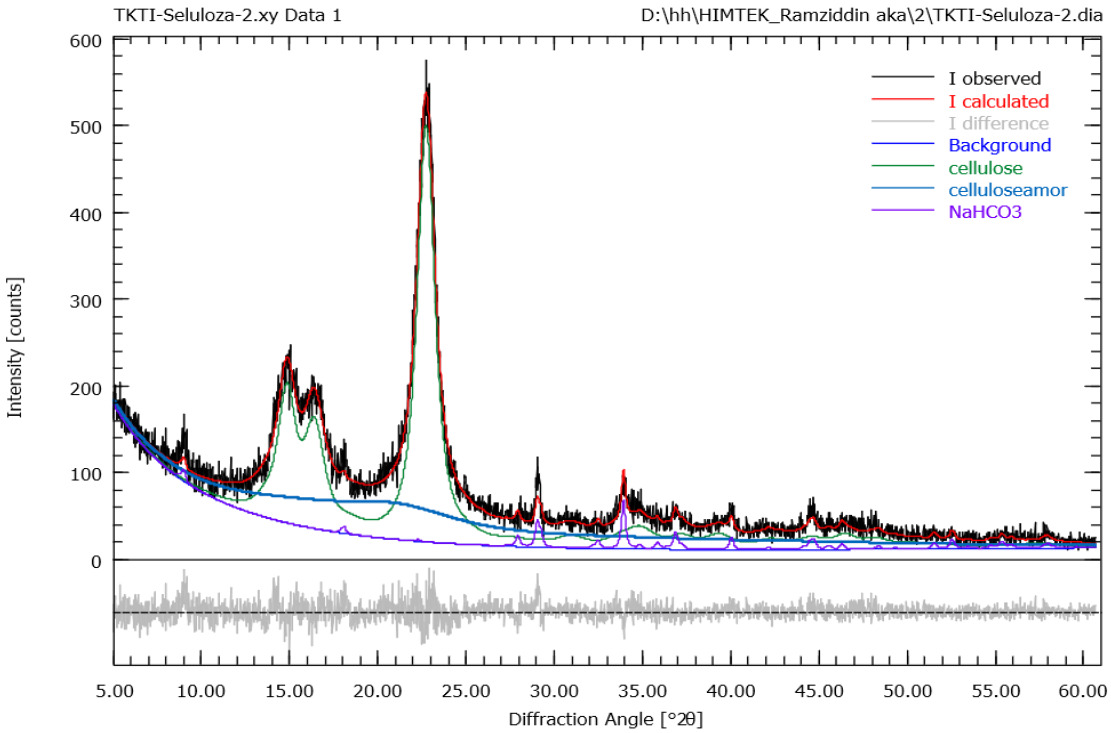


Fig. 3.X-ray diffraction pattern of sample 3

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 peak integrals, 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

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	67.56028	303.29433	668.62411	45.36
Sample-3	54.52621	268.3713	410.37879	65.40

In addition, the appearance in the X-ray diffraction pattern of the sample of 3 peaks of ammonium bicarbonate * $2H_2O$ in the form of separate crystalline peaks with sufficient intensity for calculation allows us to conclude that a certain amount of an admixture of ammonium bicarbonate is present in the sample. Given the sensitivity of the X-ray phase analysis method and quantitatively analyzing the diffraction pattern of sample 3, we found in it the presence of 4.79% bicarbonate. In this case, an abnormal decrease in the crystallinity index is observed for

sample 3, despite the fact that the presence of a 5% impurity leads to the appearance of additional crystalline peaks.

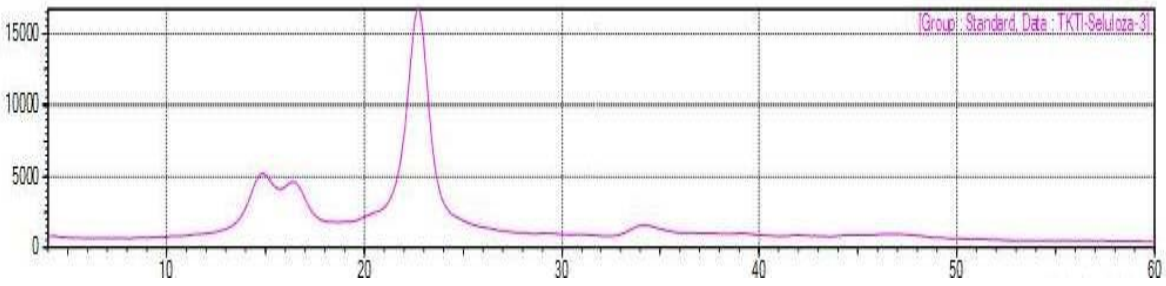
The use of the Rietveld method for analyzing the diffractogram of sample 3, using the least squares method to refine and approximate the theoretical line of the entire diffractogram profile to its experimental profile, allows us to analyze the crystal structure and obtain reliable results when the reflections from the crystalline phases of microcrystalline cellulose and bicarbonate overlap.

In the table. 2 shows the percentage of MK cellulose and amorphous cellulose based on the 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%).

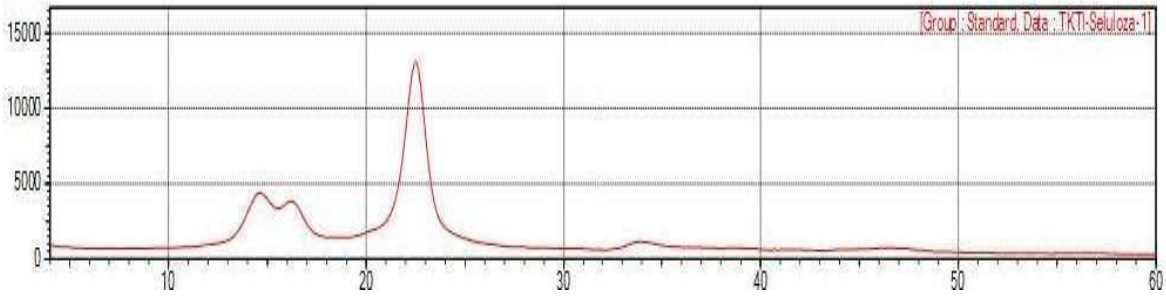
Table 2

Ritveld analysis data for cellulose samples

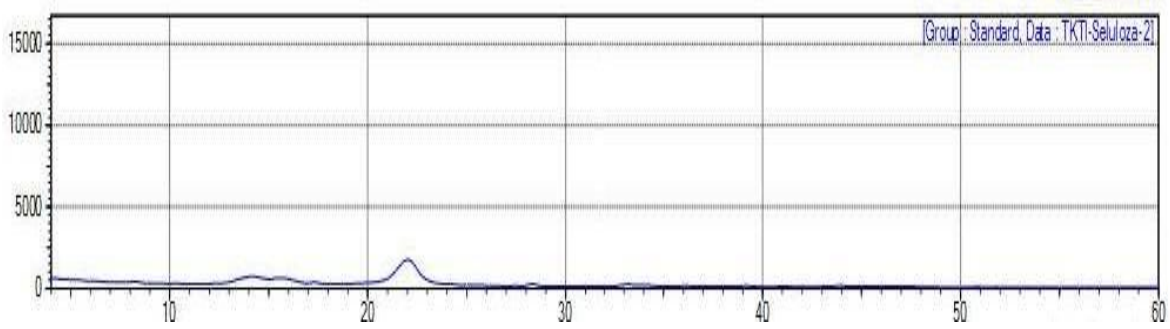
Sample Components	%	RNO (%)
Sample 1		
MK-cellulose	67.1	9.1
Amorphous cellulose	32.9	9.1
Sample 2		
MK-cellulose	62.2	5.4
Amorphous cellulose	37.8	5.4
Sample 3		
MK-cellulose	48.5	2.4
Amorphous cellulose	46.7	2.4
Ammonium carbonate (bicarbonate)*$2H_2O$	4.79	0.26



A)



B)



C)

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. CuKα radiation (β filter, Ni, 1.54178 current and tube voltage regimes 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 the cellulose practically does not change, i.e., the cellulose is not amorphized and is similar to the control sample (sample 1).

After chemical treatment with a bicarbonate salt followed by an electrical impulse, 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 to increase the reactivity of cotton cellulose, the quality indicators of the resulting cellulose ethers are improved by treating cotton cellulose with an electric charge.

The dependence of the reactivity of cellulose on the voltage, number of pulses and capacitance of the capacitor is determined. Optimum parameters were determined experimentally: Discharge voltage 11-13 kV, number of pulses 22-24 and capacitor capacitance 0.6 μF, respectively. According to the results of studies, 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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