Substantiation of technological parameters for the production of flax pulp by alkaline cooking in the microwave field

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Abstract. Flax fiber is the most valuable, rapidly renewable source of cellulose. To isolate cellulose from the fiber, it is necessary to get rid of its non-cellulose components: lignin, pectin substances, waxes and hemicellulose. These substances can be removed by cooking flax fiber in an alkaline solution. To intensify the process of destruction of the non-cellulose complex, it is proposed to use the energy of the electromagnetic field of ultrahigh frequencies. The article presents the results of theoretical and experimental studies aimed at establishing the dielectric characteristics of the system components, which make it possible to prove the effectiveness of the effect of the microwave field on the process of destruction of the non-cellulose complex and to deduce the permissible limits of technological parameters. For the effective management of the technological process of alkaline cooking, the dielectric characteristics of the system components are established on the basis of theoretical and experimental studies. To solve this problem, the system was divided into components: fiber, solution, heterogeneous system. Further, the characteristics of each component are determined separately. The established characteristics showed the following values: the dielectric constant of the fiber in the solution reached a value of 24.8, the dielectric constant of the solution-73, the dielectric constant of the heterogeneous system is 64.55. These values allow you to conduct the technological process of delignification with high efficiency indicators.

1. Introduction
For many centuries, Russia has been the largest producer and exporter of flax. This is facilitated by favorable climatic conditions: a moderate climate, low-fat soil, a long daylight day.

According to the data of the expert-analytical center of agribusiness (Figure 1), the sown area of flax in Russia in farms of all categories was at the level of 50.6 thousand hectares. Over the past year, the area of crops increased by 13.1% (by 5.8 thousand hectares), over 5 years-by 0.2% (by 0.1 thousand hectares). In relation to the period of 2010, the acreage decreased by 1.2% (by 0.6 thousand hectares).
The most valuable product of the primary processing of flax is considered to be a long fiber. Expensive high-quality fabrics are produced from it, it brings a good profit to enterprises. Its produced volume is only 19-22% of the total volume of fiber produced. The majority of 78-81% is accounted for by low-grade short fiber. Short fiber is suitable for the production of building materials (tow, flax, rope) and for the manufacture of packaging fabrics, which, as a rule, have low profitability.

Therefore, to increase the profitability of production, it is necessary to expand the structure of products. The research of scientific organizations is aimed at the development of new technologies for processing short fiber. Which offer technologies for the production of polypropylene, highly hygienic nonwovens, medical dressings, medical cotton wool, cellulose esters and others [1].

One of the promising areas of processing short fiber into products that are in demand on our market is the production of cellulose. On the territory of Russia, cellulose is isolated from wood. This source of raw materials is very expensive, it is associated with high costs for the development of forest extraction capacities, and this resource is also long-renewable. The chemical composition of flax fiber indicates the content of a large amount of cellulose [1,2]. In addition, flax is a rapidly renewable resource, it takes 1 year to renew it. Thus, the meek fiber will be able to become a product that brings great profit to flax processing enterprises. It is possible to obtain cellulose on equipment for the production of cotton wool, in devices for liquid processing with a convective energy supply. The structure of flax fiber is different from cotton and requires a longer process of alkaline cooking. This leads to an increase in the cost of the process, a deterioration in the quality of the product and high energy costs.

The founders of the technology of flax pulp production are N. N. Osipova, A. I. Ryzhov, A. P. Maryganov, V. V. Zhivetin, N. S. Danyshева [3,4] and others, whose research is aimed at reducing the duration of cooking and improving the quality of the product.

The purpose of this study is to scientifically substantiate and develop a model of the technology of alkaline cooking of flax short fiber in the field of ultrahigh frequencies in order to isolate cellulose from it.

To achieve this goal, we will solve the following tasks:
* to investigate the dielectric characteristics of flax fiber;
* to investigate the dielectric characteristics of an alkaline solution;
* to investigate the dielectric characteristics of a two-component fiber-solution system.
2. Materials and methods
The results of the work were achieved using theoretical and experimental research methods. The fundamental provisions of the electromagnetic field theory were used to solve the tasks set.

Dielectric heating of materials in an electromagnetic ultrahigh-frequency field is characterized by the dielectric properties of the material and the parameters of the field itself.

The chemical composition of flax fiber has a significant effect on its dielectric properties.

The object of processing is an inhomogeneous structure consisting of two components: flax fiber and an aqueous solution of caustic soda alkali, which have different properties. At the same time, the entire system has unevenly distributed electrical properties.

To establish the dielectric characteristics of the entire object, it is necessary to study the dielectric properties of each component individually, and of the entire system as a whole.

The dielectric permittivity of the fiber was studied according to GOST 8.623-2006. The dielectric permittivity of the substance was determined by the resonator method, which is based on the registration of changes in the natural frequency of the resonator and its Q-factor [5].

The dielectric permittivity of an aqueous solution of caustic soda with a concentration of 2% was determined by measuring the physical properties of a liquid with dielectric losses – the method of A.M. Yershov [4,6]. The method is based on determining the Q-factor of wave resonators.

3. Results
To study the dielectric characteristics of the entire object, it is necessary to study the characteristics of its constituent components: flax fiber, liquid phase and heterogeneous system.

The study of the dielectric properties of flax fiber. The sample under study was a compressed short flax fiber in the form of a disk, with an average density of $\rho = 200 \, kg/m^3$.

As a volumetric resonator, a cylinder was used in which vibrations of the $H_{01p}$ type were excited. The determination of the relative permittivity $\varepsilon$ is based on the study of changes in the difference between the resonant length of the resonator $L_0$ and the resonant length of the sample insertion field at a constant resonant frequency $f_0 = f_0$.

The measurements were made at the stand in the mode of continuous generation of an ultrahigh frequency generator, the scheme of which is shown in Figure 2.

The permittivity was determined by the formula:

$$\varepsilon = \left( \frac{c}{2\pi f_0} \right)^2 \left[ \left( \frac{x}{b} \right)^2 - \left( \frac{v_{11}}{a} \right)^2 \right],$$

where $c$ – the speed of light in a vacuum, $m/m/s$;
$f_0$ – resonant frequency, Hz;
$b$ – sample thickness, mm;
$v_{11}$ – the first root of the Bessel function $J_1(z)$, $v_{11} = 3.831706$;
$a$ – resonator radius, mm;
$x$ – dimensionless value.
Figure 2. Block diagram of the measuring unit: 1-frequency meter; 2-microwave generator; 3,6-decoupling valve; 4-attenuator; 5-measuring resonator; 7-microwave detector; 8-resonance indicator with scan voltage output; 9-voltmeter.

The determination of the dielectric properties of a short fiber in the field of ultrahigh frequencies was carried out by the method of a volume resonator. The kinetics of the permittivity index of the fiber from its humidity is shown in Figure 3.

Figure 3. The kinetics of the index of the dielectric permittivity of the fiber from its humidity.

Studies have revealed the dielectric permittivity of the fiber at different humidity. The initial fiber has a humidity of $W_0 \leq 14\%$, this state will be considered conditionally dry, therefore, it is a dielectric with $\varepsilon_0 = 4.2$. Further, during cooking, the fiber is saturated with moisture in an aqueous solution of 2% caustic soda, reaching a humidity of $W_b \leq 100\%$. Consequently, it becomes an imperfect dielectric, due to its porous structure, with a permittivity $\varepsilon_b = 24.8$.

Study of the dielectric properties of the liquid phase of the system. The block diagram of the experiment to determine the dielectric properties of the liquid phase by the method of A.M. Ershov, based on the Q-factor of the resonators, is shown in Figure 4.
The kinetics of the value of the dielectric constant of a solution of caustic soda at a temperature of 22°C is shown in Figure 5.

The conducted studies show the dependence of the dielectric permittivity of an alkaline solution on the concentration of caustic soda. Since a solution with a concentration of 2% is used in the process of alkaline microwave cooking, the dielectric constant of the solution $\varepsilon_l = 73$ is of practical importance.

*Study of the dielectric properties of a heterogeneous system.* Since the delignification process is based on cooking flax fiber in an alkaline solution, it is necessary to evaluate the dielectric properties of the entire fiber-solution system. Let's consider this "fiber-solution" system as a complex heterogeneous medium.

Korobeynikov S. M. [7,8], argues that the dielectric constant of a heterogeneous medium is directly proportional to the dialectical permeability of the liquid part of the system. This statement can be confirmed by the Nielsen formula [9], for determining the permittivity of the fiber-solution system.
\[ \varepsilon = \varepsilon_s \frac{1 + 4 \cdot \omega_f \cdot \left(\frac{\varepsilon_f}{\varepsilon_l} - 1\right) / \left(\frac{\varepsilon_f}{\varepsilon_l} + 4\right)}{1 + \omega_f \cdot \left[\left(\frac{\varepsilon_f}{\varepsilon_l} - 1\right) / \left(\frac{\varepsilon_f}{\varepsilon_l} + 4\right)\right] \cdot \left[1 + \frac{1 - W_f}{W_f^2} \cdot \omega_f\right]} \]

where \( \varepsilon_s \) - the dielectric constant of the fiber;
\( \varepsilon_l \) – the permittivity of the solution;
\( \omega_f \) - the volume fraction of the fiber;
\( W_f \) - the maximum possible volume fraction of the fiber.

In the existing technologies of alkaline cooking of flax fiber, a fiber-to-solution ratio of 1:10 is used for the purpose of delignification [1.5]. This ratio is due to the large amount of alkali per kilogram of fiber, for a more active reaction with non-cellulose components. It also promotes the free release of decomposition products and prevents them from settling on the fiber.

In our research, guided by technological, environmental and economic considerations, we will take the ratio of fiber to solution 1:4. Reducing the amount of alkaline solution in the system will reduce the cost of water and chemical reagents consumption, as well as reduce the cost of wastewater regeneration [10-12].

Studies of the values of the dielectric permittivity of a heterogeneous system with a different volume of solution are shown in Figure 6.

The obtained kinetics shows the dielectric permittivity of the fiber-solution system at different ratios of its components. Thus, with a component ratio of 1:4, the dielectric constant of a heterogeneous system is 64.55, with a volume fraction of 0.8 solution. This value of the dielectric constant provides high dielectric losses [13-14].

**Figure 6.** The kinetics of the dielectric constant of a heterogeneous system on the moisture content, at a temperature of 22°C.

4. Discussions
The conducted studies show that the rate of thermal processes and the nature of chemical reactions affect the intensity of destruction of non-cellulose components in the process of alkaline fiber cooking. Classical technologies of thermal alkaline cooking can be represented in the form of the following cycles: the absorption of alkali by lignin molecules, the chemical reaction of alkali with lignin and the
hydrolysis of lignin. However, it is known that the reaction rate can be increased by activating an aqueous medium. Such an effect has a microwave heating. In addition to the actual temperature heating, the electromagnetic field has an effect that leads to a change in the intermolecular bond in the substance. When exposed to this kind, valence-unsaturated radicals with high electrochemical activity are generated, which destroy difficult-to-dissolve organic complexes, intensifying the processes of lignin destruction [14-15].

Also, the volume heating of the system will have a reduction in the speed of alkaline cooking in the microwave field. In the process of exposure to microwave, excessive pressure occurs in each fiber, which contributes to the intensive movement of moisture to the outer layers, while the fiber is destroyed from the inside.

The conducted studies make it possible to obtain cellulose by the method of alkaline cooking of flax fiber in the microwave field, under the modes presented in Table 1.

Table 1. Modes of alkaline cooking in the microwave field.

| Indicator              | Value | Brief justification                                           |
|------------------------|-------|--------------------------------------------------------------|
| Pressure               | $10^5$ Pa | Provides the necessary temperature conditions                |
| Temperature            | 100 °C | Supports the conditions of the chemical reaction of lignin decomposition |
| Concentration NaOH     | 2%    | Provides the most complete cleavage of non-cellulose components, and does not destroy cellulose fibers |
| Time                   | 97 min.| Maximum permitted exposure to the microwave field            |
| Fiber-to-solution ratio| 1:4   | It is determined by the dielectric properties of a heterogeneous system |

Certain modes provide complete mechanical unfolding of the fiber into elementary fibers, and thus intensify the process of alkali diffusion. As a result of compliance with the regimes, we get elementary fibers consisting of 97% cellulose.

5. Conclusion
The research allowed us to establish:

1. the chemical composition of the fiber, the concentration of the alkaline solution, the temperature and processing time, the radiation power, and the dialectical characteristics of the system components affect the efficiency of separating cellulose from flax fiber during alkaline cooking in an electromagnetic field above high frequencies;
2. dielectric characteristics of a multicomponent system-fiber, solution and fiber-solution system as a whole, allowing us to judge the possibility of the entire process;
3. theoretically, the maximum permissible duration of the process;
4. the main technological stages and parameters of the process of obtaining cellulose by the method of alkaline cooking in the microwave field;

The conducted studies have confirmed the hypothesis about the possibility of obtaining cellulose from flax fiber by the method of alkaline cooking in an electromagnetic field of ultrahigh frequencies. Allowing to significantly reduce the duration of the pulp extraction process and improve its quality.

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