Improvement of flax husk production technology as raw material for cellulose nanomaterials

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Abstract. Experimental studies on ultrasonic dispersion of the organic complex of flax stem in the voiced medium on the installation of continuous operation IUSD-01 were carried out, flax fiber with high quality indicators was obtained.

1. Introduction
Flax is a unique culture that has been cultivated in Russia since ancient times. It was used to make clothes and oil, it was the subject of trade and craft. Currently, flax produces a huge range of products with exceptional properties, such as the most valuable linseed oil, short and long fibers, technical fabrics, gunpowder, medical materials, construction materials, raw materials for the textile and military industries [1]. Also, due to its special properties, it is possible to isolate the most valuable component from flax fiber – cellulose, which is a source for the production of a modern popular product – nanocellulose. To obtain this product, it is necessary to isolate a long fiber without damaging it. One of the main technological operations are operations for the production of flax straw. The quality of flax straw has a significant impact on the way it is prepared. The existing methods of preparing flax straw can be classified according to the method of resolving the bonds between cellulose and the pectin complex of the stem, into four types: biological, chemical, physical, physico-chemical [2]. Despite certain advantages of each of the methods, they have disadvantages that prevent obtaining a quality product. Therefore, it is necessary to introduce a new effect aimed at the destruction of non-cellulose complexes of flax stem. Modern, highly effective methods include ultrasound. Destructive, dispersing, emulsifying effect of ultrasound is used in many industries, including medicine and food processing industry. The use of ultrasound in the processing of flax straw in an aqueous environment, due to the course of physical and chemical processes, will allow to destroy the transverse chemical bonds between the fibrils of pectin substances and cellulose of plant tissue, as well as allow to detract proteins and disperse lignin, which will increase the efficiency of the process of liberation of fibers [3].

2. Literature review

2.1. Legal framework
The government of the Russian Federation adopted the "Federal scientific and technical program for the development of agriculture for 2017-2025", developed taking into account the Doctrine of food security of the Russian Federation, approved by presidential decree No. 120 of January 30, 2010 "on
approval of the Doctrine of food security of the Russian Federation”, and the Strategy of scientific and technological development of the Russian Federation, approved by presidential decree No. 642 of December 1, 2016” on the Strategy of scientific and technological development of the Russian Federation” [4].

2.2. Analysis of experimental and theoretical works on intensification of the process of accelerated maturation of flax straw

The founders of the theory of the destructive effect of ultrasound as an intensifying factor of the technological process were such scientists I. Elpiner, Ya. Zeldovich, E. Kikuchi, A. Agranat, F. Borodin et al [5-9, 14]. V. Kasatkin, N. Kasatkina, I. Badretdinova [10-13] proved and significantly developed the theory of destructive effects on biological objects. They theoretically substantiated the theory of the destructive effect of ultrasound in an aqueous medium on flax straw in different degrees of maturity: early, late and medium. The time of hydrolysis of straw of different degree of maturity is experimentally established. The results of the experiments are presented on figure 1.

Figure 1. Results of hydrolysis in the field of ultrasound in determining the duration of exposure.

Studies have indicated that hydration occurs faster in flax straw in the degree of late maturity.

Further, the kinetics of the process of soaking flax stems of late maturity in the voiced medium (figure 2) and the kinetics of the transition of the organic complex of the stem in the voiced medium were experimentally established.

Figure 2. Kinetics of the process of soaking flax stalks of late ripeness in the voiced medium.
As a result of processing of flax straw on laboratory ultrasonic installation samples with visualization of destruction of non cellulose components of a stalk of flax were received (figure 4).

The studies allowed to establish the optimal duration of the process, it was 95 minutes, the maximum loss of solids was 54%.

3. Materials and methods of research

3.1. Mathematical description of process stages

On the basis of theoretical and experimental studies of the kinetics of ultrasonic treatment of flax stem in an aqueous medium four stages of the destructive action of the organic complex in the cavitation medium are established and mathematically described:

- The first stage is the propagation of the ultrasonic wave in an aqueous medium, at this stage the energy expended on the propagation of the ultrasonic wave is determined

\[ E_{us} = 2\pi^2 Mf^2 \xi^2, \]  

where \( M \) - weight of the medium used for processing; \( \omega \) - cyclic frequency; \( \xi \) - amplitude of radiating surface displacement; \( f \) – oscillation frequency.

- The second stage is manifested with the origin of cavitation cavities in the zone of vacuum at the interface. At this stage energy is spent on the origin of the cavitation cavity \( E_p \) and on the heating of the medium \( E_h \):
\[ E_{us} = E_p + E_n, \]  
\[ E_n = cm\Delta t, \]  
\[ (2) \]
\[ (3) \]

where \( E_n \) – energy consumed to heat the medium, \( c \) - the heat capacity of the medium, \( m \) – weight of the medium used for processing, \( \Delta t \) – temperature variation.

- The third stage describes the process of pulsation of cavitation cavities formed on the surface and in the inner cavities of the stem. The energy at this stage spent on the formation of the cavitation cavity passes into the energy of the pulsating motion of the cavitation cavity \( E_{пуз} \):

\[ E_v \approx 2\pi\rho_0\dot{a}^2a^3, \]  
\[ (4) \]

where \( \rho_0 \) – density of medium, \( \dot{a} \) - the speed of oscillation of the bubble, \( a \) – current radius of the cavity in the medium.

- The fourth stage describes the process of bursting a pulsating bubble and the formation of a powerful shock wave. There is a transition of energy into the energy of the explosion. It is described by the formula:

\[ E_e = \frac{Ggl_o}{4}, \]  
\[ (5) \]

where \( G \)- mass of the destroyed substance, \( g \) – acceleration of gravity, \( l_o \) – distance from the center of the pulsating bubble to the surface of the stem.

By the value of \( G \) it is possible to judge the efficiency of ultrasonic destruction of the non cellulose complex of the flax stem.

Considering the whole process as a whole, we obtain a General regularity describing the efficiency of the process of destruction of the pectin complex of the flax stem:

\[ G = \frac{4(2\pi^2Mf^2\xi^2 - cm\Delta t - 2\pi\rho_0\dot{a}^2a^3)}{g\xi^2}. \]  
\[ (6) \]

To implement these processes, a prototype of a continuous ultrasonic flax soaking unit was developed and manufactured (installation ultrasonic dielectric) IUSD-01, productivity of 250 kg/h was developed and made.

3.2. Description of the installation IUSD-01

Figure 5 shows a schematic diagram of the installation IUSD-01.

![Figure 5. Schematic diagram of the installation IUSD-01](image-url)
The unit is designed for continuous destruction of organic flax complex in the field of ultrasound immersed in an aqueous medium. The body of the unit is made in the form of a bath (2), protected by a special coating that prevents the bath from ultrasonic forging and subsequent destruction. The transportation system (1) is used for the continuous flow of straw to the tub, its design provides a uniform filling of the bathtub with a straw and prevents it from emerging at the initial stages, ultrasonic transducers (3.5) is installed in a position fulfilling the condition of maximum energy absorption, i.e. emission of ultrasound is aimed at meeting product movement. The system is equipped with a level dispenser (6) as well as a spin system (7).

3.3. Plant operation

Prepare the unit for operation: connect the disposal and supply channels, fill the ultrasonic bath with water to a predetermined level, apply the supply voltage, transfer all control sub-units to the "manual" mode in the "start" state.

From the control panel transfers the unit in the "MANUAL" mode to the "START" state. The control unit sends a command to the control system of heating systems to output the electric water heater to the operating mode. When the temperature reaches 20°C the control system of the heating systems gives an intermediate signal of readiness – «G1». The control unit sends a command to start the voice control system. When a ready signal is received, the control unit issues a test run command. The control unit gives the command to start the control system of the heating system and the control system of the sound system. Next, the control unit waits for the exit to the "OFF" mode from the provision of thermal heating. The control unit uses the first level systems to supply water to a certain level. The process continues until the linen straw no longer appears in the voiced bath. The control unit informs the operator three times with a sound signal about the end of the operation.

4. The results of experimental studies

The fiber obtained by the technology of primary processing on the basis of ultrasonic soaking surpassed the quality of the fiber obtained by steaming and the fiber obtained by soaking. The study on the separability of the fiber from the organic complex was carried out by the fiber obtained by steaming at LLC "Izhlen" in Izhevsk UR and the fiber obtained by spreading at LLC "Sharkansky flax plant" UR, humidity 20…21%.

Fiber separability indicators are shown in figure 6.

![Figure 6. Kinetics of fiber separability depending on flax straw preparation methods and processing time.](image)

Laboratory studies of fiber obtained from flax straw obtained at the installation IUSD-01 showed the following dynamics of loss of non-cellulose complex of flax stem (figure 7).
Below is a comparative description of the methods of cooking flax straw with ultrasonic method (table 1).

**Table 1. Comparative characteristics of methods of production of flax straw.**

| №  | Parameter                              | Spreading | Thermal lobe | Steaming | IUSD-01 |
|----|----------------------------------------|-----------|--------------|----------|---------|
| 1  | Duration of operation, h               | 270…485  | 37…49       | 4        | 1,5     |
| 2  | Specific power consumption, kW / h     | 2,9       | 3,3          | 1,6      | 0,75    |
| 3  | Treatment temperature, °C              | 6…19      | 24…0        | 95…100  | 18…22  |
| 4  | Amount of fiber (the output), %        | 20…27     | 20…28       | 17…21   | 25…30  |
| 5  | The strength of the fiber, N           | 190…220   | 180…220     | 130…180 | 190…220 |

The result of the analysis of methods indicate a reduction in price, reducing labor intensity and eliminates the influence of weather and climatic factors on the production process of flax straw. Compliance of laboratory studies and mathematical model was 93.5%. With this in mind the technical requirements of the continuous ultrasonic dispersion unit have been developed IUSD-02.

**5. Conclusion**

The possibility of using ultrasonic soaking in the technology of primary processing of flax is proved. The stages of the operation of ultrasonic soaking of flax straw in an aqueous medium at the stage of producing flax straw are theoretically described. An analytical description of the process of ultrasonic soaking of straw in an aqueous medium in order to obtain flax straw. The study of laboratory samples of trust, revealed an undoubted advantage over the methods of steaming and soaking. Developed and tested laboratory sample installation IUSD-01, allowing the flax straw to get the best quality, and significantly reduce the time to 95 minutes.

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