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FIRST EFFORT OF RESEARCH IN THE PROCESS OF GARNETTING OF CELLULOSIC MATERIALS AND ANALYSIS OF EQUIPMENT OPERATION: FROM THE LABORATORY BENCH TO THE INDUSTRIAL INSTALLATION

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The article summarizes the first effort of research in creating and operating experimental and industrial installation for garnetting of cellulose-based materials. The experimental device was created on the basis of a household grain crusher, supplemented with a replaceable pegboard rotor, provided with an adjustable inlet and equipped with a suction pneumatic transport of the unfolded product. The device performs garnetting, mechanical grading, and pneumatic transportation of cellulose materials. The article provides examples of garnetting process studies of various materials, such as wood and cotton celluloses of various commodity forms and brands, waste paper, cellulose laboratory prototypes. Recommendations are formulated on the design parameters and garnetting process methods of pulp lap from sulfite wood cellulose from various manufacturers. Recommended specific capacity is 2.5 kg/(m^3·s). The specific energy intensity of the garnetting process is 75 kJ/kg. The power reserve coefficient of the electric motor is 1.5. The circumferential speed of the rotor's outer edge is at least 45 m/s. The moisture content of the initial pulp ranges from 5 to 10% (rel.). The size of the sieve cells is 5 mm. The specific productivity of mechanical classification of loosened pulp is 1.5 kg/(m^2·s). The bulk density of the unfolded product is from 30 to 120 kg/m^3. The experimental setup can be used for carrying out various experimental studies of the cellulose garnetting processes and development of the necessary experimental samples in the laboratory. The article provides an example of creating an industrial plant with a capacity of 500 kg/h for the production of technical sodium-carboxymethyl cellulose of various brands based on these recommendations, followed by an analysis of the experimental unit operation. The industrial plant is based on a hammer mill and is equipped with a suction pneumatic transport. The industrial plant is used for the cellulose folder garnetting for the needs of industrial cellulose derivatives production.

Introduction

Cellulose and lignocellulosic raw materials saw a rise in usage in recent years. Cellulose, a plant-based natural polymer, is widely used in many industries. Cellulose derivatives such as cellulose ethers are of particular interest. One of the most widespread and demanded is the ether of cellulose and glycolic acid – carboxymethyl cellulose (CMC). The industry usually uses sodium-CMC.

Technical sodium-CMC, along with the main substance – the sodium salt of CMC, also contains a fairly large (up to 50%) amount of by-products, mainly sodium chloride. The industry is also in demand for impurity-free products, such as polyanionic cellulose (PAC). PAC is mostly the same sodium-CMC, but with a higher content of the basic substance. The applied industrial technology for the production of PAC is implemented on the basis of suspension technology. The basic industrial technology for the production of sodium-CMC is the so-called solid-phase [1]. The implementation of the suspension technology [2] can significantly increase the content of the basic substance and eliminate the presence of by-product sodium chloride. In any production of cellulose or cellulose derivatives, it is required to prepare the cellulosic raw material or the cellulose itself for technological processing [3].

Cellulose is known to be a natural polymer of D-glucose. The starting element of cellulose as a polymer is a monomer unit. This link represents the remainder of the original glucose. Monomer units are combined into a macromolecule through β (1-4)-glycosidic bonds. The cellulose macromolecule has an almost linear structure. The length of the macromolecule (degree of polymerization) is determined by the origin of cellulose (the type of plant, tree or another living organism). The polymerization degree of the starting cellulose ranges from several hundred to several thousand.

Furthermore, cellulose also has a complex, hierarchical supramolecular structure [4]. Cellulose macromolecules (in the amount of several dozen) are primarily combined with each other to form microfibrils. The basis of the combination is based on cellulose features. The monomer unit of cellulose contains three free hydroxyl groups. Thus, cellulose macromolecules contain a huge amount of hydroxyl groups. Hydroxyl groups of neighboring macromolecules form hydrogen bonds with each other. Hydrogen bonds are inherently weak in nature. However, due to the fact that they are being formed in huge quantities, they create a very strong bond between neighboring cellulose macromolecules. Cellulose microfibril is a very stable formation. Areas of microfibrils with a large number of hydrogen bonds have the properties of crystalline materials. This cellulose structure is very highly ordered. There are also amorphous regions with fewer or no hydrogen bonds.

In addition to cellulose, cellulose raw materials contain a number of other compounds. Hemicelluloses, also known as binding glycans, and lignins are present. Hemicelluloses and lignins are also natural polymers. Hemicelluloses are polymers of pentoses and hexoses. Lignins are polymers of phenylpropane derivatives. Pectins and some mineral components are also present.

Fibrils are formed in the plant tissue, which are a combination of microfibrils and binding components, such as binding glycans, lignins and, in some cases, pectins. Cellulose fibrils, along with other components, form the cell wall of plant tissues in the cellulose raw material. Thus, the cell wall is a natural composite material.

It is also worth noting that along with ordinary cells, plant tissues also contain special cells. These are the so-called plant fibers. They are elongated cells with a very long length. These cells are conventionally called fibers. Fiber cells play the role of conducting vessels. Also, fiber cells can act as power elements in plant tissue [4].

The essence of the pretreatment of both cellulosic raw materials and cellulose itself to prepare for technological exposure is to ensure the availability of cellulose macromolecules and other components to the effects of chemical reagents. Usually, the preparation of cellulose and cellulose raw materials consists comes down to grinding [6]. Grinding in this context does not refer to classical grinding, in which the size of the particles is ground is reduced, but rather, it refers to partial disordering of the supramolecular structure. In order to achieve this, a certain number of hydrogen bonds have to be broken. Under mechanical action, grinding will also occur, resulting in shortening of macromolecules in the composition of microfibrils. However, the role of this process is comparatively insignificant with a time-limited mechanical action.

Mechanical action also underlies the so-called mechanical activation of cellulose. Mechanical activation is characterized by intense and often prolonged mechanical action on the processed cellulosic or lignocellulosic raw materials [7-9]. To some extent, the garnetting of cellulosic materials can be considered the first stage of mechanical activation, the so-called pretreatment of cellulosic material. There are multiple stages of garnetting, firstly, a mechanical effect on the processed material with the supply of energy takes place. Secondly, the surface available for subsequent contact with reagents increases after the exposure. And, thirdly, with the garnetting of the commercial form of cellulose, the number of defects increases faster (aggregates of cellulosic material are disaggregated) than the disappearance of defects (aggregation of individual fibers of cellulose material, leading to the formation of lumps). Thus, the main signs of mechanical activation take place during garnetting.

Mechanical impact is coarse in nature, since the working bodies of grinding machines are incomparably large in comparison with the processed cellulose microfibrils. It is worth noting that a better result could be achieved if other mechanisms could be used, the scale of their effect being comparable to the size of cellulose microfibrils. For example, these could be turbulence microvortices arising under hydrodynamic action. Hydrodynamic grinding machines, such as colloidal mills and rotor-pulsating devices, are examples of this. There are successful results of using such machines for fine impact on cellulosic materials [10].

However, the hydrodynamic action involves "wet" processing of cellulose or cellulose raw materials [11]. The preparation of a relatively low-concentration water-fiber suspension is required. Based on the rheological characteristics of such a suspension, the concentration of the solid phase ranges from a few to twelve percent. That is, it will be necessary to process a huge amount of transport fluid, which reduces the efficiency of the process. In addition, not all processing technologies require the use of liquids in the preparation of cellulose or cellulosic raw materials. Therefore, the "dry" method of processing cellulose or cellulosic raw materials is often used [12, 13].

Experimental procedure

For laboratory processing of both cellulosic raw materials and finished cellulose, a special machine for garnetting was used. The schematic illustration of the machine is shown in fig. 1. This machine is based on a modified household rotary grain crusher. The machine has two loading windows, a garnetting chamber, a drive and a sieve. A rotating rotor is used as a working body. This rotor is a steel plate. Pegs are installed on the plate in a staggered manner on both sides of the plate. The tuners are shaped like cones. To maintain the balance of the rotor, the mass of the working cone of the splitter is balanced by a counterweight. The rotor is removable.

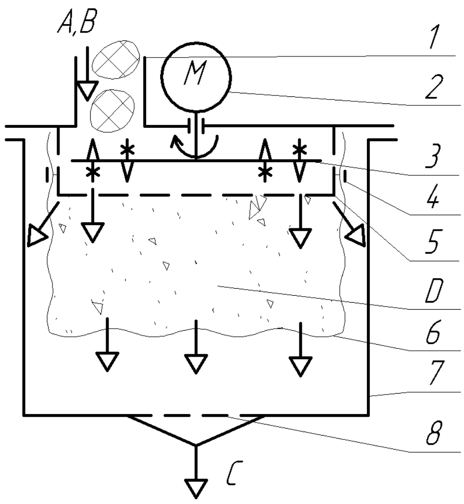

Fig. 1. Schematic illustration of a garnetting machine for cellulosic materials: 1 – loading window; 2 – electric motor; 3 – peg rotor; 4 – clamp; 5 – sieve; 6 – collecting vessel; 7 – suction chamber; 8 – filter; A – cellulosic flakes; B – air from the atmosphere; C – exhaust air; D – garnetted material

The sieve forms the end and side walls of the garnetting chamber. The sieve is also removable. The openings in the sieve are round and staggered. The size of the holes in the replaceable sieves is from five to ten millimeters. On one sieve, all holes are the same size. At some distance from the side surface of the sieve, an annular baffle deck is installed. This deck is used to exclude the waste of garnetting products.

One of the loading windows is a narrow slot and serves for feeding sheet materials, for example, a cellulose folder. Another feed port has a round shape and serves to supply other types of material, such as lumps or flakes of cellulose, stems of plant materials, etc.

The drive of the fiberizing machine is adjustable. The collector electric motor is powered by the direct current. The electric motor power circuit is shown in Fig. 2. A laboratory autotransformer (LATR) is used to change the supply voltage. The electric current after the LATR is rectified. An ammeter and a voltmeter are also installed in the power supply circuit of the electric motor to determine the current strength and the magnitude of the supply voltage.

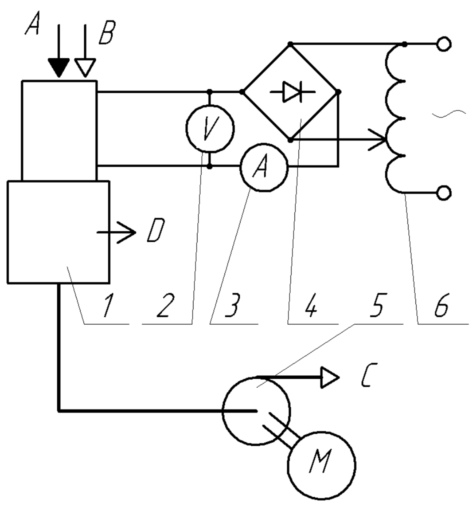

Fig. 2. Schematic illustration of an experimental setup for garnetting of cellulosic materials: 1 – garnetting machine; 2 – voltmeter; 3 – ammeter; 4 – rectifier; 5 – blowing machine; 6 – laboratory autotransformer; A – cellulosic flakes; B – air from the atmosphere; C – exhaust air; D – garnetted material

The experimental setup also includes a device for unloading the garnetted material. As noted above, hydrogen bonds between macromolecules are easily and quickly formed in cellulose. Therefore, cellulose easily compresses, crumples, aggregates and forms a cohesive medium. To prevent this, the loosened product must be quickly removed, so pneumatic transport was used. In order to avoid excessive dusting during loosening, a suction pneumatic transport is used. For this, a suction chamber is arranged after the sieve, which is then connected to the blower. A household vacuum cleaner was used as a blower. As a result of this solution, the blower has additional built-in filters for cleaning the exhaust air.

To prevent the fluffed cellulose from being carried away by the air flow into the blower, a collector is located between the sieve and the suction chamber. This collector is a cloth bag with frequent weave texture. The neck of the bag is equipped with an elastic cuff. The neck of the bag with the cuff is put on the baffle deck and is additionally secured with a removable clamp. To further prevent the loss of loose material, the outlet from the suction chamber is covered with a filter. The filter is used to capture fine particles of loose material.

The air path of the pneumatic transport includes one of the open loading windows, a garnetting chamber, a sieve, a collector, a suction chamber, a filter, an air duct of a blower and a blowing machine. There are three stages of air purification. The first stage is done through the permeable walls of the collector, the second is the filter at the bottom of the suction chamber, the third is the built-in filter of the blower.

The setup works as follows. The material is fed through one of the windows in a working garnetting machine to be garnetted. The unused loading window is pre-muffled to prevent the ejection of loosening products through it. The material in the garnetting chamber is subjected to intense action by the rotor with splits and is loosened. The air flow is sucked in by the blower, forming a vacuum in the chamber. The transit air flow captures garnetted products and carries them through the sieve into the collector. Then the air enters the suction chamber through the wall of the collector. The loosened particles settle on the collector wall. An additional layer is formed, which retains the remaining loose particles. This is similar to filtration with a layer of alluvial sediment. The resulting layer of fibrous cellulose material acts as an additional filtering partition. Exhaust air is discharged into the atmosphere. Small loose particles can accidentally slip through gaps in the wall of the collector or through leaks in the places where the collector is attached to the baffle deck. Such particles are retained by the filter at the bottom of the suction chamber.

The garnetting setup works at intervals. As the container fills up, the drive of the fiberizing machine stops, followed by the blower itself. The collector is removed from the baffle deck and emptied. To speed up the work, two collectors are used, and the filled collector is replaced with an empty one. The filter is also cleaned. If necessary, for example, when switching to another type of processed material or another prototype, the chamber is cleaned for fiber separation. For this, the sieve is dismantled and cleaned. Then the sieve is reassembled in its original place, an empty collector is installed, the suction chamber is connected, and the process is resumed. Cleaning of the built-in filters of the blower is required periodically.

Observation and evaluation

The described setup for garnetting cellulose materials has been used in many independent studies, both published [14-16] and unpublished. In the latter case, various assignments of industrial partners were carried out.

This setup was used for carrying out pre-treatment before the main technological processing of various fibrous cellulosic materials, such as wood pulp (bleached sulphite pulp and unbleached pulp of various grades) and cotton cellulose. Waste paper was also processed in order to obtain fluff pulp, then used to obtain cellulose ecowool. Various commercial forms of cellulose materials were processed, as well as cellulose semi-processed materials [17]. In particular, pulp flakes compressed into heaps were processed. This is a traditional form of packaging for cellulosic materials. A modern form of cellulosic raw materials – a cellulose folder [18, 19] – was processed as well. The cellulose folder is produced at pulp and paper mills using the technology of cardboard production and can have different surface density [20]. The basis weight (the mass per unit area of the cellulose folder) for the processed materials ranged from 600 to 1200 g/m^2. From cellulose semi-processed products, cellulose sections of various shapes and sizes were processed. The prototypes of cellulose obtained in laboratory conditions during various experimental studies were processed during the experiment as well.

Some additional preparation of cellulosic materials is required before the garnetting process can be started. The sheets of the cellulose folder are cut into narrow strips with a width of 25 to 40 mm. Cellulosic materials in heaps are manually torn into flakes with a diameter of no more than 30 mm. Such measures are a consequence of the small size of the experimental setup.

Cellulose raw materials from various manufacturers were processed. We used wood pulp produced by the Syassky Pulp & Paper Mill (PPM), the Bratsk and Ust-Ilimsk PPM of the Ilim Group, the Arkhangelsk PPM and a number of others. We processed cotton lint produced by various manufacturers of the Republic of Uzbekistan, the Republic of Tajikistan, the Republic of Kazakhstan, as well as cotton cellulose from various manufacturers.

The resulting batches of garnetted cellulose were used for processing and obtaining various types of cellulose products. For example, various grades of sodium-CMC, refined cellulose for chemical processing and ecowool were obtained. An experimental setup was used to prepare a model environment for other experimental studies [15].

In addition to the production of the necessary batches of cellulose materials, the garnetting unit was used for independent experimental studies. For example, a study was carried out to determine the specific power consumption during the garnetting of various cellulose materials [14]. Another study determined the specific productivity of this unit for the garnetting of various cellulosic materials. The necessary working conditions were defined; in particular, the minimum working circumferential rotation speed of the rotor, and the results of processing cellulose materials of different moisture content. The practical significance of the results of such studies can hardly be overestimated. These studies provided important information about the necessary operating conditions for the garnetting unit.

Multiple designs of rotors were used, including flat knife rotors, and peg rotors, with pegs of various shapes, sizes and relative positions in relation to each other.

A number of studies have dealt with the assessment of quality parameters for the pretreatment of cellulosic materials. We used such physicomechanical and physicochemical parameters as bulk density, compaction density, specific surface area, dispersed composition, wettability index, and a number of others. However, these studies have not yet yielded definitive results [16]. Thus far, the most reliable indicator of the preprocessing quality of the cellulosic materials is to obtain a sample of the final product. For obvious reasons, this technique does not have the capabilities of express analysis, although it gives reliable information. Therefore, the question of choosing a parameter for the express analysis of the preprocessing quality of the cellulosic materials still remains open.

Recommended design parameters and equipment operating conditions were established, allowing to obtain a homogeneous and high-quality pre-processed product. These are displayed in Table 1.

Table 1. Recommended design parameters of equipment and process parameters of garnetting a cellulose folder made of wood pulp

Parameter	Unit of measure	Value
Specific output	$kg/(s{\cdot}m^3)$	2,5
Specific garnetting energy	kJ/kg	75
Power factor of the electric motor	–	1,5
Minimal peripheral speed of working bodies	m/s	45
Moisture content of cellulosic material	% (rel.)	5 to 10
Specific output of mechanical fluff cellulose breakdown	$kg/(s{\cdot}m^2)$	1,5
Bulk density of fluff cellulose	kg/m^3	30 to 120

One of the most significant research results is the creation of an industrial garnetting unit for one of the production partners. This unit was created using the results of various studies carried out on a laboratory model. An industrial installation for garnetting cellulosic materials consists of a hammer mill and an aspiration pneumatic transport serving its operation, as shown in Fig. 3.

According to safety requirements, the garnetting unit is isolated from the main production. During the operation process of the unit, dust is emitted, which is a combustible medium. The garnetting section is located in a room of category "B" (explosion and fire hazard).

The industrial unit was used for the preprocessing of cellulose in the production of various grades of sodium-CMC using solid-phase technology at JSC "Polyex" [3]. Various grades of cellulose, wood and cotton, provided by various manufacturers, were processed. All types of cellulose raw materials have been successfully processed and used for the production of both developmental and commercial batches of products, such as technical sodium-CMC and polyanionic cellulose.

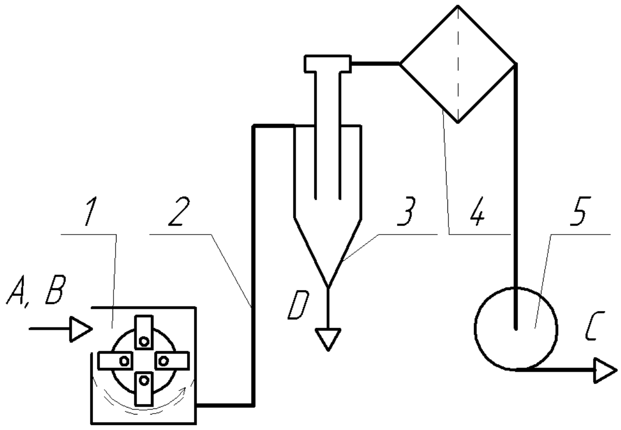

Fig. 3. Schematic illustration of an industrial garnetting unit: 1 – hammer mill; 2 – material line; 3 – cyclone unloader; 4 – filter; 5 – fan; A – cellulosic folder sheets; B – air from the atmosphere; C – exhaust air; D – garnetted material

Conclusion

The article summarizes the first effort of research in creating and operating experimental and industrial installations for garnetting of cellulose-based materials. The experimental device was created on the basis of a household grain crusher, supplemented with a replaceable pegboard rotor, provided with an adjustable inlet and equipped with a suction pneumatic transport of the unfolded product. The device performs garnetting, mechanical grading, and pneumatic transportation of cellulose materials. The article provides examples of garnetting process studies of various materials, such as wood and cotton celluloses of various commodity forms and brands, waste paper, cellulose laboratory prototypes. Recommendations are formulated on the design parameters and garnetting process methods of pulp lap from sulfite wood cellulose from various manufacturers. Recommended specific capacity is 2.5 kg/(m^3·s). The specific energy intensity of the garnetting process is 75 kJ/kg. The power reserve coefficient of the electric motor is 1.5. The circumferential speed of the rotor's outer edge is at least 45 m/s. The moisture content of the initial pulp ranges from 5 to 10% (rel.). The size of the sieve cells is 5 mm. The specific productivity of mechanical classification of loosened pulp is 1.5 kg/(m^2·s). The bulk density of the unfolded product is from 30 to 120 kg/m^3. The experimental setup can be used for carrying out various experimental studies of the cellulose garnetting processes and development of the necessary experimental samples in the laboratory. The article provides an example of creating an industrial plant with a capacity of 500 kg/h for the production of technical sodium-carboxymethyl cellulose of various brands based on these recommendations, followed by an analysis of the experimental installation operation. The industrial plant is based on a hammer mill and is equipped with a suction pneumatic transport. The industrial plant is used for the cellulose folder garnetting for the needs of industrial cellulose derivatives production.

An experimental laboratory unit for the purpose of garnetting cellulose materials was developed and created, based on a rotor grain crusher. The design uses household equipment as a base, and is low-budget in nature. This unit can be used for carrying out various experimental studies of the cellulose garnetting processes and development of the necessary garnetted cellulose material.

Recommended design parameters of equipment and process parameters of garnetting a cellulose material were established. Optimal specific output equals 2,5 kg/(s·m^3). Optimal specific garnetting energy during processing most dense material (cellulose folder) is up to 75 kJ/kg. Minimal peripheral speed of working bodies should not exceed 45 m/s. Specific output of mechanical fluff cellulose breakdown is 1,5 kg/(s·m^2). Bulk density of fluff cellulose, depending on the garnetting mode, ranges from 30 to 120 kg/m^3.

On the basis of the recommendations formulated, a technical specification was developed for an industrial garnetting installation with a capacity of 500 kg/h for the finished semi-processed product (garnetted cellulose). The equipment and the installed power of the mill drive motor were determined. An industrial garnetting unit has been created, tested and used for preliminary preparation of cellulose raw materials in the production of technical sodium-CMC. The industrial installation provides a stable quality of garnetted cellulose with a specified productivity.

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