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STUDYING THE PRODUCTION OF NANOCELLULOSES

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Abstract. *Nanoscience is a broad field that includes nanophysics and nanochemistry. Nanotechnologies are aimed at obtaining nanoparticles with new properties and creating materials and devices based on them. This is one of the modern trends in the development of science in the 21st century.*

Based on the study of information about nano-sized materials, the mechanisms of their production and changes in their properties, we will consider the technology for producing nanocellulose from organic substances, i.e. from cellulose. The article describes a technology that examines devices for producing nanocellulose and the principles of their operation, as well as a combined method of purification and acid hydrolysis with high-pressure homogenization for the production of nanocellulose on an industrial scale. The types of nanocellulose and their microscopic images are also described. Our research work is aimed at obtaining nano-sized cellulose. Cellulose is one of the most common natural polymers that is part of an important part of the tissues of annual and perennial plants, algae, bacteria and fungi. Using this raw material, it is proposed to obtain nanocellulose, since cellulose is a linear homopolysaccharide, a macromolecule consisting of monomeric anhydride-β-D-glucopyranose units linearly linked to each other by 1→4-glucosidic bonds.

Keywords. *Nanocellulose, microfibrillated cellulose, nanocrystalline cellulose, bacterial nanocellulose, polymerization, crystallization, biodegradation.*

Annotatsiya. *Nanofan keng tarmoqli bo'lib u o'z ichiga keng qamrovli bo'lgan nanofizika va nanokimyo fanlarini oladi. Nanotexnologiyalar yangi xususiyatlarga ega nanozarrachalarni olishga va ular asosida materiallar va qurilmalar yaratishga qaratilgan. Bu XXI asrda ilm-fan rivojlanishining aktual yo'nalishlaridan biridir.*

Nanokimyo yo'nalishi bo'yicha nano o'lchamli materiallar, ularninng olinish mexanizmlari va ular asosida xossalarning o'zgarishi to'g'risida ma'lumotlarni o'rgangan holda organik moddalar asosida ya'ni sellyulozadan nanosellyuloza olish texnologiyasini ko'rib chiqamiz. Maqolada keltirilgan texnologiya asosida nanosellyuloza olish uchun qurilmalar va ularning ishlash prinsiplari ko'rsatilgan, hamda sanoat miqyosida nanotsellyuloza ishlab chiqarishning tozalash va yuqori bosimli gomogenizatsiya bilan kislotali gidrolizning kombinatsiyalangan usuli keltirilgan. Shuningdek nanosellyuloza turlari va ularning mikroskopiya orqali olingan tasvirlari izoxlangan. Ilmiy tadqiqot ishimiz nano o'lchamli sellyuloza olinishiga qaratilgan. Bir yillik va ko'p yillik o'simliklar, suv o'tlari, bakteriyalar va zamburug'lar to'qimalarining muhim qismini tashkil etuvchi eng keng tarqalgan tabiiy polimerlardan tsellyuloza. Aynan ushbu xom ashyolardan foydalangan holda nano sellyuloza olish ko'zda tutilgan, chunki sellyuloza chiziqli gomopolisaxarid bo'lib, bir-biriga 1→4-glyukozid bog'lari bilan chiziqli bog'langan monomer angidr-β-D-glyukopiranoza birliklaridan tuzilgan makromolekulalardir.

Tayanch so'zlar: *Nanotsellyuloza, mikrofibrillangan tsellyuloza, nanokristalli tsellyuloza, bakterial nanotsellyuloza, polimerizatsiya, kristallanish, biodegradatsiya.*

Аннотация. *Нанонаука — это широкая область, включающая нанофизику и нанохимию. Нанотехнологии направлены на получение наночастиц с новыми свойствами и создание на их основе материалов и устройств. Это одна из современных тенденций развития науки в XXI веке.*

На основе изучения информации о наноразмерных материалах, механизмах их получения и изменений их свойств мы рассмотрим технологию получения наноцеллюлозы из органических веществ, т.е. из целлюлозы. В статье приведена технология в котором, рассматривается устройства для получения наноцеллюлозы и принципы их работы, а также комбинированный

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метод очистки и кислотного гидролиза с гомогенизацией под высоким давлением для производства наноцеллюлозы в промышленных масштабах. Также описаны виды наноцеллюлозы и их микроскопические изображения. Наша научно-исследовательская работа направлена на получение наноразмерной целлюлозы. Целлюлоза — один из наиболее распространенных природных полимеров, входящих в состав важной части тканей однолетних и многолетних растений, водорослей, бактерий и грибов. С использованием этого сырья предполагается получение наноцеллюлозы, поскольку целлюлоза представляет собой линейный гомополисахарид, макромолекулы, состоящие из мономерных ангидрид-β-D-глюкопиранозных звеньев, линейно связанных друг с другом 1→4-глюкозидными связями.

Ключевые слова: *Наноцеллюлоза, микрофибриллированная целлюлоза, нанокристаллическая целлюлоза, бактериальная наноцеллюлоза, полимеризация, кристаллизация, биодеградация.*

Introduction

Nanocellulose is a material consisting of nano-sized cellulose fibers with a high aspect ratio of length to width and nanoparticles. The characteristic width of such a fiber is 5-20 nm, and the longitudinal size ranges from 10 nm to several microns. The material has the properties of pseudoplasticity, i.e. under normal conditions it is viscous, and through physical interactions it behaves like a liquid (in vibration, agitation, etc.). Its extraordinary properties make it possible to create ultra-light and ultra-strong materials based on it, for example, such as airgel [1-8].

There are mainly three types of thread-like, nano -sized crystals - nanocellulose:

1) microfibrillated cellulose (MFC) - thread-like particles with disordered sections and alternating crystals,

2) nanocrystalline cellulose (NCC) - represents thread-like single crystals without flaws,

3) bacterial nanocellulose (BNC) – an organic polymer material. The material has a number of unique properties that are not inherent in plant cellulose.

They have different sizes, functions and production methods (Figure 2).

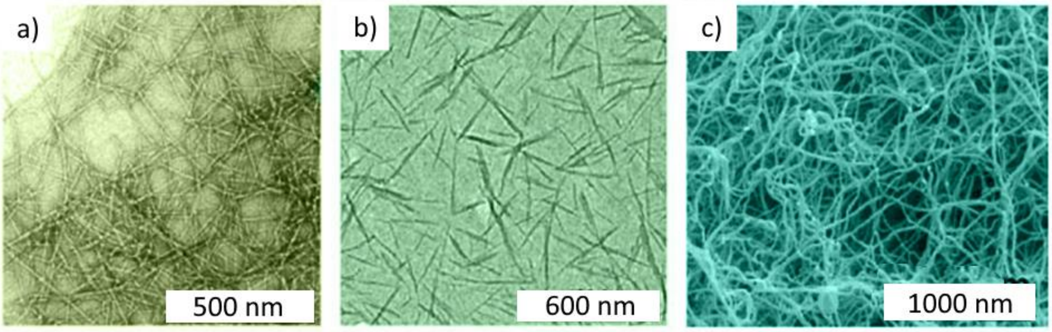

Fig.2. Microscope images of each type of nanocellulose: a) MFC, b) NCC, and c) BNC

Nanocellulose particles have many advantages over plant cellulose and partly over synthetic polymers [9-13]:

- high purity (does not contain lignin, hemicellulose, pectin);
- plastic;
- high degree of polymerization and crystallization;
- very large internal surface area of the nanofiber network;
- high mechanical stability;
- controlled biodegradation ability;
- increased bending strength and elasticity of synthetic polymers;
- tensile and tear strength;
- high hygroscopicity and ability to accumulate water.

Methods and materials. Figure 3 shows cellulose fibers and the small structures within them. The process of producing nanocellulose from cellulose is carried out in three main stages: the grinding process, the hydrolysis process and the homogenization process [14-20].

Experimental part

The grinding process of periodic and continuous grinding - rolls, conical mills, refiners, etc. is carried out in the presence of water at a fibrous mass concentration of 2-8% in grinding apparatus.

The principle of fiber grinding is that the fibrous suspension flows in a continuous flow to the knives of the working part of the apparatus, regardless of the type of grinding apparatus, consisting of fixed knives (stator) and rotating knives, which are located on a drum, cone or disk (rotor). The gap passing between the rotor and stator knives can be adjusted, while the fibers are subjected to the cutting action of the edges of the knives and are shortened or split in the longitudinal direction, crushed by the end surfaces of the knives, combed and fibrillated.

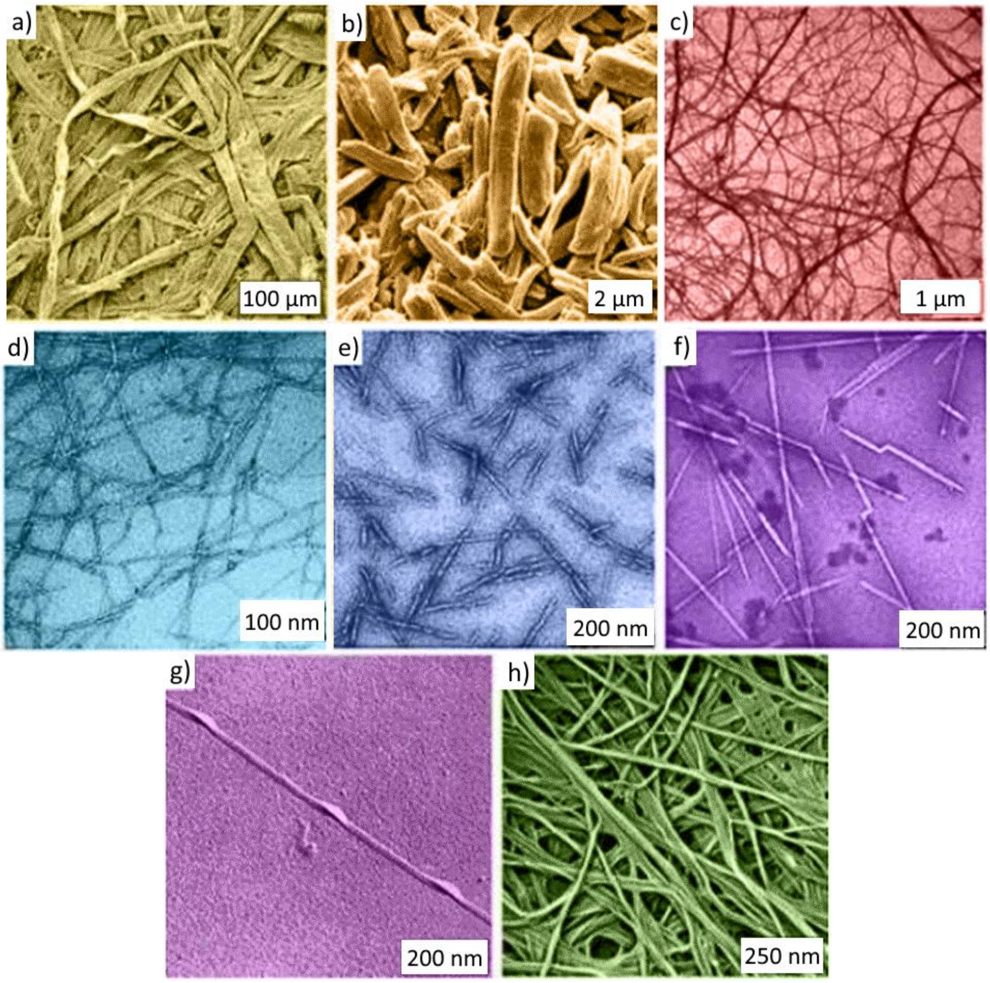

Fig.3. Cellulose fibers and small structures inside them: a) cellulose fiber, b) microcrystalline cellulose, c) cellulose microfibrils, d) cellulose nanofibrils , e) cellulose nanocrystal from wood pulp, f) cellulose microfibrils of animal (marine) origin, g), h) cellulose nanofibrils from other sources.

The Schopper-Rigler instrument is used to determine the ability of paper pulp to pass water through itself; The data obtained characterize the degree of development and grinding of fibers, as well as the degree of their hydration during grinding. Using the Schopper-Rigler device, you can determine the degree of grinding of the mass.

Acid hydrolysis with weak hydrochloric acid and alkaline hydrolysis with weak sodium hydroxide solution are two stages of hydrolysis during the processing of crushed cellulose pulp.

Hydrolysis of cellulose can be carried out using mineral acids. Polysaccharide macromolecules decompose into soluble fragments and dissolve the latter in acid at 20–40 °C by treating cellulose with concentrated sulfuric or superconcentrated (41%) hydrochloric acid.

The main apparatus for hydrolysis production is the hydrolyser. The crushed cellulose is loaded into the hydrolysis apparatus and diluted hydrochloric acid is immediately pumped in and the upper neck is tightly closed. Then the contents of the apparatus are gradually heated with live steam and blown off to remove air and volatile products. Within 30–40 minutes, the temperature is brought to 50°C and the pressure to 0.5–0.7 MPa. During this time, a significant part of the hemicelluloses is hydrolyzed and goes into solution. Then they begin continuous percolation, i.e., dilute hydrochloric acid heated to 70 ° C is continuously fed into the hydrolysate apparatus from above, and the hydrolyzate is continuously removed from below.

The temperature of the contents in the apparatus is brought to 80 - 90 ° C, the pressure is 0.9-1.2 MPa, in the process of continuous percolation. During this period, cellulose and the difficult-to-hydrolyze part of hemicelluloses are hydrolyzed. Macromolecules of polysaccharides are successively shortened during hydrolysis. Thus, hydrocellulose is first formed from cellulose, then cellodextrins (consisting of 10-60 glucose residues), oligosaccharides (3-10 glucose residues), cellobiose (disaccharide $C_{12}H_{22}O_{11}$) and glucose is formed. With continuous percolation, the monosaccharides formed from the reaction space are removed faster and their destruction is reduced.

According to a given program, steam, water, acid are supplied and hydrolyzate is selected, taking into account the fact that as hydrolysis occurs, the raw material shrinks and the content of polysaccharides in it decreases. The action of the process is controlled by the mass of substances contained in the apparatus; it is determined by the readings of a weight meter, on the scale of which zero corresponds to an empty apparatus.

The process ends when the amount of acid specified by the program is supplied to the hydrolysis apparatus, as well as water to wash the lignin remaining in the apparatus at the end of the process, and the specified amount of hydrolyzate is removed from it. Then the pressure is reduced to 0.6-0.7 MPa, the quick-acting valve is opened, and the hydrolyzed cellulose is blown into the cyclone in 0.5-1 minutes, from where it is discharged through the holes in the bottom of the cyclone using a rotating sump mechanism.

Homogenization is a mechanical process designed to reduce the fine particles present in a liquid so that they become uniformly small in size and distributed evenly. Decreasing the average particle diameter increases the number of individual particles. This leads to a decrease in the average distance between particles and increases their surface area. This process uses a high-pressure homogenizer microfluidizer M-110-EH (see Fig. 4). The retracting piston sucks the pulp down from the container into a small chamber, then the piston moves forward, pushing I (see Fig.5) are installed on the loop sequentially: the first, the so-called auxiliary chamber, is larger than the second, so the interaction chamber. The required pressure of approximately 1600-1700 bar is needed to push the material through the narrow chamber.

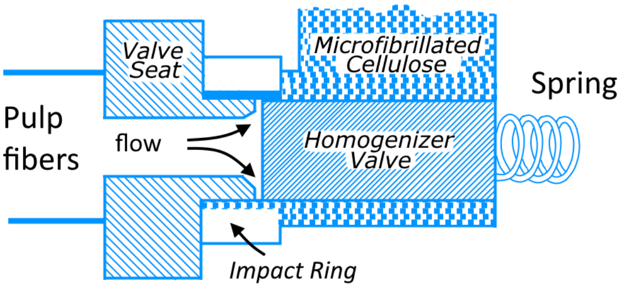

Fig.4. High pressure homogenization process diagram

As the product flows at high speed, the flow impacts on wear-resistant surfaces to bring the desired result inside the chamber, microchannels with a fixed geometry are specially designed through which the product flow will accelerate to high speeds, creating high shear rates and forces.

The pump completes its pressure stroke as an amplifier, this changes direction and attracts a new volume of product. At the end of the intake stroke, it reverses direction and again drives the product at constant pressure, repeating the process. After exiting the interaction chamber, the product flows through an on-board heat exchanger, which regulates the product to the desired temperature before exiting into the finished product pool.

There is a significant problem in modern industry - the development of an energy-efficient and environmentally friendly technology for the production of unique nanomaterials from available cheap, reproducible raw materials. Currently, research and development is underway in the use of cellulose-containing raw materials, the results of which are new types of nanomaterials. One such unique material is nanocellulose.

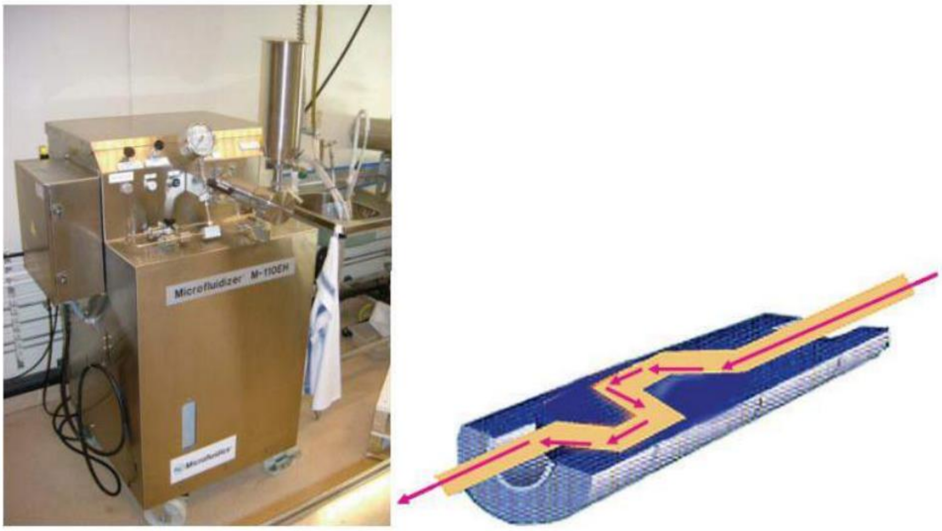

Fig.5. Microfluidizer M-110-EH and interaction chamber design diagram

The raw materials for the production of nanocellulose can be agricultural waste, algae, bacteria, but the most accessible raw material is cellulose from wood.

Nanocellulose production methods are divided into two stages: chemical and mechanical, but most often combined ones are used. One of the most effective methods for producing nanocellulose on an industrial scale is the combined method of acid hydrolysis with refining and high-pressure homogenization.

First place on the market among the most durable nanomaterials This nanocellulose is superior to Kevlar and carbon nanotubes in a number of respects. It is stronger than Kevlar, which, in turn, is 5 times stronger than steel, and nanocellulose can be thinner than paper. Nanocellulose has the unique properties of pseudoplasticity. Some of the most important structural and molecular properties of cellulose nanoparticles are geometric dimensions, average crystallinity and average degree of polymerization, which depend on the source of cellulose materials, and mechanical properties, which also depend on the cellulose-containing raw material. Nanocellulose suspensions are shear thinning and they have excellent water absorption capacity. These properties make it possible to create ultralight and ultrastrong materials from nanocellulose.

Conclusions

For vast and diverse applications, nanocellulose has the potential to become an important class of renewable nanomaterials due to its interesting physical, mechanical and chemical properties, abundance, light weight and biodegradability. It can be used in medicine and the food industry, as well as in construction and the automotive industry. Nanocellulose is effectively used in many areas of industry.

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