EVALUATION OF THE QUALITY OF CELLULOSE SEMI-FINISHED PRODUCTS FROM TECHNICAL HEMP AND THE POSSIBILITY OF THEIR FURTHER USE

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ABSTRACT

The article discusses the issues of processing industrial hemp into goods for various functional purposes. Particular attention is paid to the processing of hemp trusts into cellulose-containing semi-finished products, as well as to the analysis of the properties of cellulose-containing fibrous materials obtained from industrial hemp. Based on the results of the study of physical, mechanical and organoleptic characteristics of the obtained cellulose-containing semi-finished products, their suitability for the pulp and paper industry and other sectors of the national economy has been established. The presented research is relevant for the purpose of creating an own raw material base for pulp and paper enterprises and light industry enterprises.

KEYWORDS

Technical hemp; Shale trust; Trust obtained by soaking; Husk layer; Fibrous semi-finished product; Hemp cellulose; organoleptic indicators.

INTRODUCTION

Technical hemp remains potentially the most profitable of the technical crops grown in Ukraine. Like no other agricultural crop, technical hemp has advanced technologies for in-depth processing into a large number of final products. Hemp stalks are a universal raw material in the textile industry, furniture production, in construction and composite materials, and it is also possible to obtain biofuel, cellulose and many other products from it [1-4]. Valuable medicinal, perfume and cosmetic preparations are obtained from hemp seeds, they are very useful as additives in many food products, and high-quality technical oils and paints are made from them.

The economic use of hemp has undergone a number of significant changes, reflecting the demand for fiber products depending on the level of technological progress and the changing needs of society. Due to the high exceptional properties of elementary fibers, fiber semi-finished products, cellulose and hemp fabrics are able to scatter and absorb sound waves and light rays. Chinese researchers claim that hemp clothing absorbs 95 % of UV rays, while others absorb 30 – 90 %. When heated to 370 °C, the color of the fabric does not change, and at a temperature of 1000 °C, when it becomes charred, its ignition does not occur [3, 4].

According to Lithuanian researchers, the hemp fibrous semi-finished product has the ability to resist the waves of mobile communication [5, 6]. In addition, it can be used for sewing military uniforms, including body armor. Considering the antistatic properties of hemp fiber and considering ancient medical research, linen and medical dressing materials made of hemp have a beneficial effect on the human body, prevent the development and reproduction of anaerobic bacteria.

Now, more than ever, the ecological danger to the environment from the use of synthetic materials, such as synthetic textile materials, packaging, chemical plastic, synthetic cellulose, is an urgent problem. Recently, the world has seen an increase in demand for pulp materials used to produce various types of paper and composite materials for various functional

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purposes. The growing shortage of wood raw materials and the high cost of imported pulp procurement make it possible to use industrial hemp to produce pulp-containing materials. In addition, given the properties of hemp to accumulate a huge biological mass and the ability to clean up contaminated areas, which is currently very important for the restoration of post-war Ukraine, the research presented here is relevant for the creation of a domestic raw material base for pulp and paper and light industry enterprises.

The aim of the presented studies is to evaluate the physical, mechanical and organoleptic properties of cellulose-containing semi-finished products obtained by the sodium method and neutral sulfite methods in order to determine the possibilities of their further use.

LITERATURE REVIEW

Recently, a number of publications have appeared in the literature on the processing of hemp stalks into cellulose-containing semi-finished products. Technical hemp is a very promising raw material. Due to its chemical composition and unique combination of properties such as hygiene, high strength, natural bactericidal properties, technical hemp has long been used for textile, paper, construction, fuel, cosmetology and other industries. Hemp fibers are used in the production of ropes, cables and twine. Hemp products are strong and durable. In addition, the cultivation of hemp does not require any chemical fertilizers, thanks to which such raw material is 100% environmentally friendly [7-9]. On the basis of cellulose from hemp pulp, a biopolymer can be made that decomposes in the environment, is recyclable and safely disposed of, and has high strength and flexibility. Such a polymer finds multifaceted application: from packaging for food products and industrial products to interior equipment of cars [10-12].

Cellulose is a valuable component because it gives fibers and fabrics made from them tensile strength, flexibility and elasticity, wearability, hygroscopicity, softness and shine. The content of cellulose in hemp fibers is 70 - 77 %, pectin substances - up to 3.3 %, lignin - 3.7 - 8 %, water - up to 10 %. Lignin adds stiffness and other negative properties to the fiber, so it is an undesirable component from a technological point of view [13-15].

The process of obtaining cellulose consists in releasing it from other accompanying substances of plant tissue - lignin, hemicellulose, fats, etc. - by the method of chemical treatment. Since the main substance that must be removed as a result of processing is lignin, the process of obtaining cellulose is called delignification of the corresponding plant material. The product of delignification is called cellulose [16].

The quality of cellulose-containing semi-finished products is determined by a complex of physical-chemical and mechanical-strength properties. A large number of different indicators are used to characterize the physicochemical properties of fibrous materials: lignin content, yield, ash content, moisture and clogging. Mechanical strength indicators are breaking length, resistance to breaking, bending, tearing and crushing.

Depending on the conditions of processing cellulose-containing raw materials with various chemical reagents at elevated temperatures and pressure, as a result of cooking, technical cellulose is obtained with different quantitative yields. According to the amount of output from the mass of raw materials, technical pulps are divided into three main categories: semi-cellulose, high-yield cellulose and normal-yield cellulose. Production of semi-cellulose consists of two stages of processing - chemical and mechanical. In the process of chemical processing, lignin and hemicelluloses are released from non-wood raw materials, and during mechanical processing, it is separated into fibers under the action of hot grinding [17].

The possibilities of using hemp stems for the production of cellulose-containing semi-finished products have been investigated by the laboratory of the University of Toronto (Canada). The semi-finished product obtained by the sodium cooking method with a yield of 55% and a residual lignin content of 3% is suitable for the production of printing types of paper [16, 17]. The industrial use of hemp in the production of fibrous semi-finished products was studied by the Olympia College laboratory (Washington, USA). To obtain the semi-finished product, sulfate and sodium methods, cooking with organic solvents were used. As a result, a chemical-thermomechanical mass was obtained with a yield of cellulose-containing semi-finished products of more than 60% and satisfactory quality indicators [17].

In the literature, there was a report on the creation of a new method of environmentally friendly technology for obtaining cellulose from annual plants (straw, hemp, perennial plants, agricultural waste, etc.) called Natural Pulping. Formic acid and hydrogen peroxide are used as reagents for cooking and bleaching. The technology makes it possible to reduce the amount of harmful toxic emissions while improving the quality of the obtained semi-finished product in comparison with the sulfate method [18, 19].

RESEARCH METHODOLOGY

When processing non-woody types of plant raw materials, the choice of the processing method and regime should be related to the peculiarities of the morphological and anatomical structure, chemical composition, physical and mechanical properties of the raw material, as well as requirements for the quality of the semi-finished product and the main technical and economic indicators of production.

Table 1. Chemical composition and fiber length of hemp raw materials.

Chemical composition and fiber length	Stem		Fiber	
	fibrous part or lob	wooden part or bonfire	received from the trust obtained by soaking	obtained from the shale trust
Cellulose [%]	66.0 - 70.4	37.1 - 39.4	65.8 - 67.4	66.5 - 68.9
Lignin [%]	3.7 - 6.0	21.0 - 32.9	5.0 - 7.5	4.1 - 6.2
Hemicellulose [%]	7.0 - 7.4	18.9 - 21.2	3.5 - 4.5	4.1 - 6.8
Other components (pectin, tannins, nitrogenous substances) [%]	23.3 – 16.2	23.0 – 6.5	25.7 – 20.6	25.3 – 18.1
Length [mm]	5.0 - 55.0	0.60 - 0.75	5.0 - 55.0	5.0 - 55.0

A characteristic feature of the anatomical structure of technical hemp is the presence of bast fibrous and woody layers. The first layer is 30 – 40 % of the entire stem, and the second is 60 – 70 %, respectively. The most valuable cellulose fibers are located in the husk layer their content is 60 - 70%.

The chemical composition of hemp raw materials, potentially considered as raw materials for obtaining cellulose-containing semi-finished products, is given in Table. 1 [26].

The difference in the chemical composition of the fibers and their length makes it necessary to separate these two fractions before starting the cooking of hemp raw materials, as well as their separate use for the manufacture of cellulose semi-finished products for different purposes [23-25].

It is believed that the most acceptable alkaline methods for chemical processing of industrial hemp stems are sodium, sulfate, and neutral-sulfite. The sodium method of cellulose production allows processing any wood species and annual plants into cellulose and semi-cellulose. In the sodium method, a solution of caustic soda is used as a reagent.

In the sulphate method, the reagent is a mixture of caustic soda (NaOH) and sodium sulphide (Na_2S). Sulphite pulping is carried out using an aqueous solution of Na_2SO_3 and Na_2CO_3. Sulphate pulp is superior to sulphite pulp in terms of mechanical strength. In neutral-sulfite pulping, a mixture of caustic soda (NaOH) and sodium sulfite (Na_2SO_3) is used as a reagent [16,17,24].

To conduct production tests, we used hemp trust, obtained by the methods of cold-water impregnation (trust obtained by soaking) and dew impregnation with the use of chemical composite preparations (slaked trust).

Biological and chemical methods such as cold-water soaking, dew impregnation and steaming of stems are used to produce hemp trusses. Cold-water soaking and steaming are carried out in accordance with regulated technological regimes developed by the Scientific Research Institute of Institute of bast crops (Hlukhiv, Ukraine).

The production of hemp trusses by cold-water soaking and dew soaking with the use of chemical compositions was carried out in the laboratory at the Kherson National Technical University (Kherson, Ukraine). To obtain hemp trusses by cold-water soaking, stems are loaded into the chamber in bales with horizontal laying. The loading density is 80-100 kg/m^3. The initial water module (the ratio of dry matter to water) is from 1:10 to 1:12. The water level in the chamber should be 10-15 cm higher than the surface of the loaded straw. The first change of liquid is carried out 12-15 hours after the start of urination, draining 25 % of its previous volume and adding fresh water instead. Subsequent fluid changes are made depending on the acidity of the fluid. If more than 2.0 ml of 0.01 normal sodium hydroxide solution (molar concentration of NaOH in the solution, in this case, is 0.008 [mol/l] is used to titrate the urine fluid, 25 % of the fluid volume is changed in the chamber with the addition of fresh water. If the acidity of the liquid is lower, it is not changed until the end of the process. The final water module is in the range of 1:30 to 1:35.

Dew impregnation was carried out in the laboratory on a specially prepared artificial plot as follows: first, the stems were treated with chemical compositions based on surfactants, and then sprayed with water to maintain the level of humidity necessary for the development of pectin-degrading microflora. The duration of dew soaking was 20 days [15,20,21,26].

The difference between the samples in terms of color was visually observed. The sample that had a white-golden color was labeled as sample No. 1 (shale trust), and the sample that had a dark golden color with a greenish tint, respectively, as sample No. 2 (trust obtained by soaking).

The raw material – hemp pulp – was sorted on a laboratory sorter in order to remove rejected elements (small particles, mineral impurities), which must be removed at the preparation stage because their presence during the production of cellulose-containing semi-finished products will lead to disruption of the technological process. The crushed and sorted raw materials were placed in a desiccator for moisture averaging. After that, the moisture content of raw material samples was determined according to the standard method. According to the calculations, the average moisture content of sample No. 1 was 8.94%, and that of sample No. 2 was 9.08%.

The moisture content of the raw material significantly affects the process of its impregnation with the cooking solution, the concentration of active reagents

Table 2. Production of cellulose-containing semi-finished products by the sodium method.

Parameters of thermochemical treatment	Indexes	
	Sample №1	Sample №2
1. Weigth [g]	80	80
2. Consumption of NaOH [%]	15	15
3. Hydro module	1:4	1:4
4. Temperature [°C]	170	170
5. Duration [min.]	180	180
6. Coefficient of dryness	0.38	0.39
7. Yield after washing [%]	64.8	64.2
8. Degree of delignification [units. Kappa]	7.23	7.44

Table 3. Production of cellulose-containing semi-finished products by the neutral-sulfite method.

Parameters of thermochemical treatment	Indexes	
	Sample №1	Sample №2
1. Weigth [g]	80	80
2. Consumption of NaOH [%]	3	3
3. Consumption of Na_2SO_3 [%]	23	23
4. Hydro module	1:3	1:3
5. Temperature [°C]	170	170
6. Duration [min.]	180	180
7. Coefficient of dryness	0.33	0.41
8. Yield after washing [%]	66.4	71.89
9. Degree of delignification [units. Kappa]	11.6	14.98

Table 4. Quality indicators of the cellulose-containing semi-finished product obtained by the sodium method.

Physico-mechanical indicators of cellulose	Size	
	Sample №1	Sample №2
1. Density [g/cm^3]	0.67	0.63
2. Breaking length [m]	5070	5012
3. Absolute compression resistance [kPa]	250	248
4. Absolute tear resistance [mN]	1067	1003
5. Fracture [number of double inflections]	914	1048
6. Linen [%]	42.0	-

in the delignification process, and therefore the uniformity of cooking, the yield of cellulose and its quality. Therefore, the raw materials used for the production of cellulose-containing semi-finished products should have a uniform fractional composition and uniform moisture content, the fluctuations of these indicators should be insignificant.

After determining the moisture content, accurately weighed samples of crushed hemp pulp were loaded into laboratory reactors for thermochemical treatment. Cellulose was boiled by sodium and neutral sulfite methods in a laboratory autoclave at a temperature of 170 °C [17, 23, 25]. The process parameters are listed in Table 2 - 3.

After thermochemical treatment, the resulting semi-finished product was crushed and loosened in a laboratory disintegrator, and then washed on a sieve. After washing, the mass was squeezed, the total yield of cellulose and the degree of its delignification were determined.

The preparation of the cellulose-containing semi-finished product for physical and mechanical tests consisted of grinding and making paper-like samples based on it. The following physical and mechanical parameters were evaluated experimentally: density, breaking length, absolute compression resistance, absolute tear resistance and fracture. The values of the resulting pulp-containing semi-finished product were determined using standard methods in accordance with the current international and national standards for wood pulp-based paper: STATE STANDARD 27015-86 "Paper and cardboard. Methods for determination of thickness, density and specific volume", STATE STANDARD 13525.1-79 "Fibre semi-finished products, paper and cardboard. Methods for determination of tensile strength and elongation at tension", STATE STANDARD 13525.8-86 "Semi-finished fibre products, paper and cardboard.

Table 5. Quality indicators of the cellulose-containing semi-finished product obtained by the neutral-sulfite method.

Physico-mechanical indicators of cellulose	Size	
	Sample №1	Sample №2
1. Density [g/cm^3]	0.7	0.82
2. Breaking length [m]	6230	6294
3. Absolute compression resistance [kPa]	349	393
4. Absolute tear resistance [mN]	2293	1280
5. Fracture [number of double inflections]	4000	4076
6. Linen [%]	55.7	53.0

Table 6. Organoleptic quality indicators of the cellulose-containing semi-finished product obtained from technical hemp.

Indicator	Cellulose-containing semi-finished product from technical hemp	
	received from the trust obtained by soaking	derived from a shale trust
Appearance and structure	fine fibrous loose mass	fine fibrous loose mass
Color	white with a grayish tint	light yellow
The presence of foreign impurities	contains impurities of single fibers from dark yellow to brown	does not contain non-cellulosic impurities

Method for determining the resistance to pressing", DSTU 3368-96 "Semi-finished fibre products and paper. Method for determination of tear resistance", DSTU 3476-96 (ISO 5626-97) (ISO 5626:1993) "Paper. Determination of fracture strength at multiple bends". The values of the quality indicators of the obtained samples are given in the Table 4 - 5.

It should be noted that the cellulose-containing semi-finished product obtained by both the sodium and neutral-sulfite methods, after dissolution in a disintegrator and washing, was well divided into bundles and individual fibers. A semi-finished product with a cellulose yield of 60% has a sufficiently low degree of delignification, which should not complicate the bleaching process in the future.

The obtained material has high quality indicators. Analyzing the physical and mechanical parameters of the obtained samples, it should be noted that the cellulose-containing semi-finished product obtained by the neutral-sulfite method with indicators of yield 70 %, whiteness 54-56 %, degree of delignification 12 - 10 units. Kappa, fracture resistance 4000 - 5000 number of double inflections and tear resistance of 1200-1500 mN, its quality exceeds cellulose obtained from wood.

Given the high strength indicators, the obtained cellulose-containing semi-finished product can be used in the composition for the production of high-strength, durable and special types of paper, non-woven materials, as well as for the production of technical textiles of various functional purposes.

The next stage of the research was the organoleptic evaluation of the samples of the cellulose-containing semi-finished product. The following indicators were chosen for evaluation and comparison: appearance and structure, color, presence of foreign impurities. The results of the research are presented in Table 6.

After analyzing the organoleptic evaluation of the studied samples, it was established that the samples of the hemp cellulose-containing semi-finished product have a loose, fine-fiber structure. The color of the semi-finished product obtained from slaked hemp trust is light yellow (Figure 1).

The color of the semi-finished product obtained from the received from the trust obtained by soaking is white with a grayish tint (Figure 2).

The difference in the color of the studied samples is explained by the conditions and regimes of primary processing of technical hemp with the aim of obtaining high-quality fibrous mass.

Having investigated the presence of foreign impurities, it was established that the cellulose semi-finished product obtained from shale trust does not contain foreign impurities in the form of trust, sand, pieces of rubber, metal inclusions and other impurities of a non-cellulosic nature.

Figure 1. A sample of cellulose-containing semi-finished product obtained from the trust obtained by soaking.

Figure 2. A sample of a cellulose-containing semi-finished product obtained from a shale trust.

Table 7. Physicochemical properties of cellulose-containing semi-finished products of various origins.

Indicator	Cellulose-containing semi-finished product					
	Cotton	from coniferous wood	viscose	linen	hemp from trust obtained by soaking	hemp from shale trust
Moisture [%]	8 – 10	8 - 12	6 – 10	7.5	6.22	5,6
Degree of swelling [%]	450 - 550	450 - 550	450 - 550	-	400	250
α-cellulose [%]	96 – 99	82-88	90 - 92.5	87 - 88	86.66	83.33
Lignin [%]	0.3 - 0.5	0.1 - 0.5	-	0.5	10	15
Ash content [%]	0.1 - 0.3	0.3 - 0.5	0.08 - 0.12	1.0 - 1.1	1.07	1.06

The semi-finished product obtained from the trust obtained by soaking contains impurities of single fibers from dark yellow to brown in color, but they are of cellulosic origin.

The results of research on the physical and chemical properties of cellulose-containing semi-finished products of various origins are given in Table. 7

Analyzing the given indicators, it should be noted that according to the humidity indicator, the tested samples of semi-finished products from hemp contain slightly less moisture than those from cotton and coniferous wood. Thus, the cellulose-containing semi-finished product from hemp contains 5.6 - 6.22 % moisture, cotton: 8 - 10%, and from coniferous wood: 8 - 12%.

The degree of swelling of the pulp was determined by the Schwalbe weight method, the essence of which is to determine the increase in pulp weight during its soaking in sodium hydroxide solutions. The degree of swelling (C_{sw}) was determined as a percentage by the formula:

$$C_{SW} = \frac{m_1}{m_2} 100 \, , \quad (1)$$

where m_1 is the mass of the test sample before swelling [g], m_2 is the mass of the test sample after swelling [g].

According to the degree of swelling, samples obtained from technical hemp have slightly lower values compared to other types. Thus, the semi-finished product obtained from shale trust - has a degree of swelling of 250 %, and obtained from urea trust - has a degree of swelling of 400 %. The difference in these indicators is due to the regimes and conditions of primary processing of industrial hemp with the aim of obtaining fiber, which subsequently affects the structure of hemp cellulose.

According to the content of α-cellulose, it should be noted that the largest amount of it is contained in the cotton cellulose-containing semi-finished product: 96 - 99%, slightly less in viscose: 90 - 92.5 % and linen: 87 - 88 %. It should be noted that the smallest amount of α-cellulose is contained in semi-finished softwood 82 - 88%. The examined samples of hemp semi-finished products contain 86.66 % and 83.33 % of α-cellulose, respectively.

Analyzing the lignin content, it was found that the studied samples of cellulose-containing semi-finished products from industrial hemp contain more lignin (10 - 15 %) compared to others (0.1 - 0.5 %). Lignin, namely its high content, is an undesirable component of the fibrous semi-finished product, which makes it impossible to obtain high-quality textile and cellulosic materials. Therefore, to obtain fibrous and cellulosic semi-finished products of better quality, it is necessary to use additional processing aimed at removing lignin.

Based on the results of research, it can be stated that the replacement of synthetic and traditional cellulose with cellulose from technical hemp will not lead to a decrease in the quality of products, because its physical and chemical indicators, including moisture content, ash content, α-cellulose content, are on a par with other types cellulose The main attention should be paid to the content of lignin, which is slightly overestimated in hemp cellulose, which is explained by the biological and morphological structure. At the same time, the lignin content can be corrected by additional processing of hemp pulp. Replacing flax and wood cellulose with hemp can improve the quality of fibrous materials, which is confirmed by the high content of α-cellulose as a valuable component for the production of fibrous semi-finished products, composite materials based on hemp cellulose, as well as packaging materials.

RESULTS

Thus, the conducted studies confirm the possibility of obtaining a fibrous semi-finished product from drug-free hemp. Based on the obtained results, it can be concluded that cellulose and fibrous semi-finished products obtained from drug-free hemp are characterized by a high level of physical and mechanical indicators, which are not inferior to the indicators of cellulose and fibrous semi-finished products obtained from other types of raw materials (cotton, wood, flax, synthetic fibers), and in some parameters even exceed them.

Given the unique properties of industrial hemp, namely the accumulation of a huge biological mass and the ability to clean up contaminated areas, hemp can be a unique alternative to replace imported raw materials and create its own raw material base for pulp and paper and light industry enterprises. Considering the unique properties of technical hemp and the results of the conducted research, it is possible to assert the possibility of using hemp products in various sectors of the economy: this includes the textile industry, pulp and paper industry, and the production of various types of composite materials. In addition, high physical and mechanical indicators (density, resistance to crushing and tearing) and sanitary and hygienic properties of hemp fibers confirm the possibility of using hemp raw materials not only for sewing clothes and underwear, but also for the manufacture of military clothing and protective body armor.

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