https://doi.org/10.29013/AJT-22-3.4-76-81

Khamdamova Dilnoza,
Doctoral student of the department
"Pulp and Woodworking Technologies"
of the Tashkent Institute of Chemical Technology
Umarova Vasila,
Doctoral student of the department
"Pulp and Woodworking Technologies"
of the Tashkent Institute of Chemical Technology
Primkulov Mahmud,
Doctor of Technical Sciences Professor of the Department
of "Pulp and Woodworking Technologies"
of the Tashkent Chemical-Technological Institute

OBTAINING CELLULOSE FROM THE MEDICINAL PLANT MILK THISTLE

Abstract. Cellulose was extracted from the stem of the medicinal plant- milk thistle. The fiber length, sorption properties, physical characteristics of width were studied by the method of IR-spectrum.

Keywords: cellulose, milk thistle, the degree of polymerization, IR-spectrum, the size of cellulose fibers, hydrolysis.

Introduction. It is known that the Institute of the Chemistry of Plant Substances named after V.I. acad. S. Yu. Yunusov AS RUz is concerned with the extraction of pharmaceutical substances. The institute has developed a number of methods for extracting a variety of medicinal preparations from the flora in Uzbekistan. In particular, from the medicinal plant Saint-Mary-thistle – then milk thistle (Latin Silybum marianum, in Uzbek Ola o't) [1]. Milk thistle is an annual or biennial herb. Stem is upright, massive, branched, glabrous, slightly branchy, 100–150 cm tall. Its ripe fruits and seeds are raw materials for the production of medicines. After the extraction of a number of compounds, meal remains that is not used [2–4].

At the beginning, the stems were crushed to 5–-8 mm, then they were boiled in water to remove easily soluble substances. In this case, soluble substances are released from the stems, changing the optical density of the liquid. As soluble substances are released into the solution, the optical density of the solution increases, the degree of light transmission decreases (Fig. 1). The kinetics of changes in optical density was determined using a **KFK-2** photoelectric colorimeter. After 40–45 minutes, the optical density of the liquid does not change, which means that there is no soluble part in the stems.

Objects and research methods. The objects of research are milk thistle stems, cellulose and fiber. To study the sorption properties of the samples, the methods of swelling in water and the test method for characterizing cellulose fibers were used. And also the method of IR-spectroscopy was used.

The aim of this work – extracting cellulose from milk thistle stems, studying the features of its structure, determining the size and fractional composition of cellulose fibers.

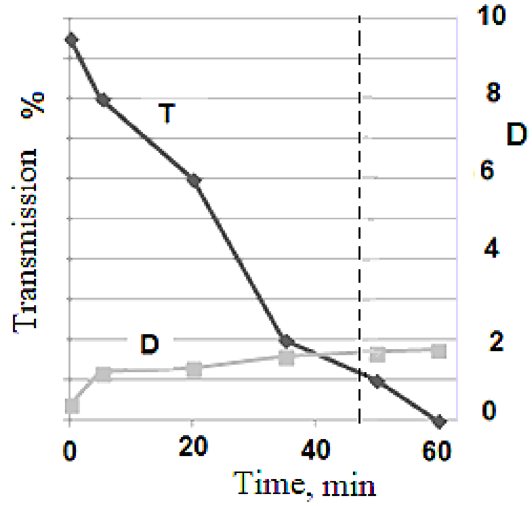


Figure 1. a) Kinetics of milk thistle stems extraction in water: T-light transmission; D-optical density of the liquid

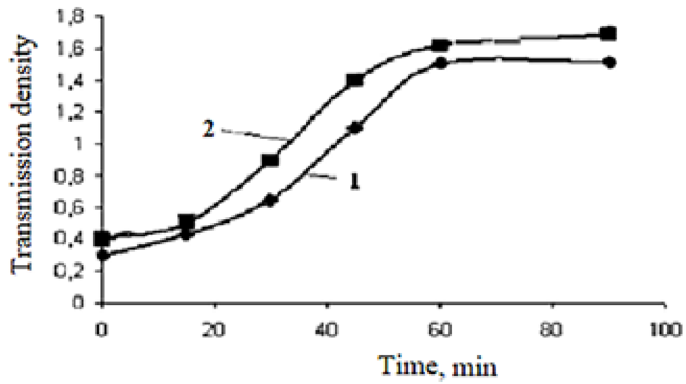


Figure 1. b) Kinetics of milk thistle stems extraction in alkali and peroxide: 1–7% NaOH; 2–10% H2O2

After separating the soluble part into water, the remaining pulp containing cellulose was cooked. Isolation of cellulose from the mass was carried out by cooking in a 7% alkali solution for 4–5 hours.

Bleaching was carried out in 10% hydrogen peroxide solution for 2 hours. Structural-dimensional characteristics of cellulose from milk thistle, the determination of the size of fibers and the fractional composition in length and width were carried out by an automatic analyzer L&W Fiber Tester, developed by the company "Yuman" [5]. It is known that the length of the fibers has a great influence on the physical properties: strength and elastoplasticity. The instrument allows for advanced analysis of the properties of cellulose fibers.

The analyzer determines the following characteristics of fibers:

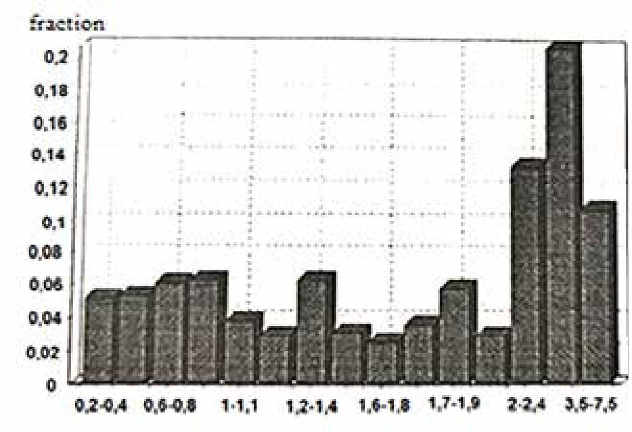
- average length of fibers in the sample, mm;
- average width of fibers in the sample, microns;
- average fiber shape factor in the sample;
- average bend angle;
- average number of all kinks per fiber;
- average length of one segment, mm;
- fraction of fines [6; 7].

The device was used to determine the average values of the length and width of the fibers, their fractional composition (distribution curves), as well as the shape factor (Fig. 2).

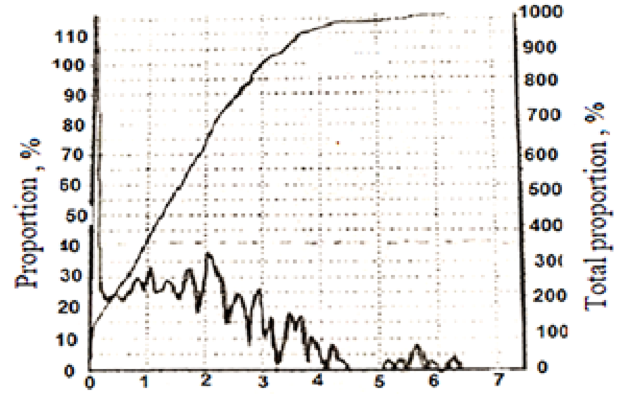
A

B

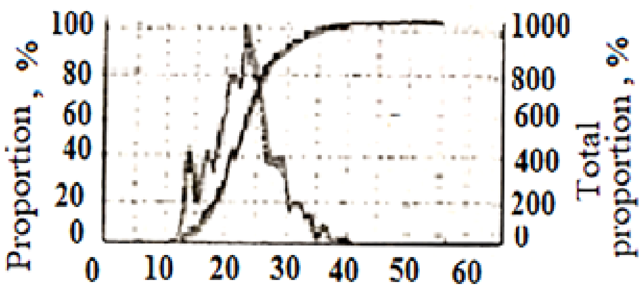
Figure 2. A – Automatic analyzer Fiber Tester; B – micrograph of milk thistle cellulose fibers



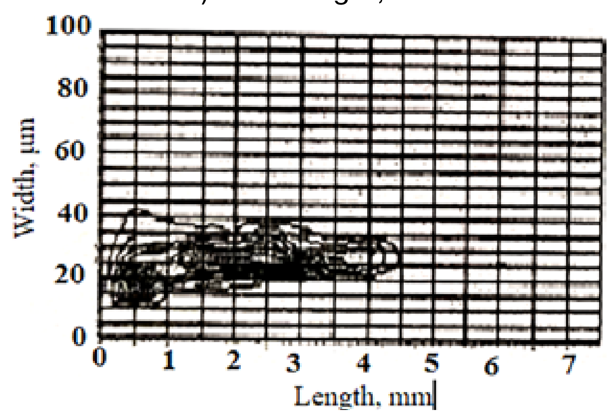
A) Fiber length, mm



B) Fiber length, mm



C) Fiber width, µm



D) Shape of the fiber

Figure 3. Fractional composition of milk thistle cellulose samples:
a,b – fiber length; c – the width of the fibers; d – the shape of the fibers

Before determining the fiber length distribution curves in the pulp, using a microscope the type of fibers in samples was established. To do this, a fiber sample was diluted with water and applied on the glass surface, after water evaporation, the sizes and shapes of the fibers were photographed. They are shown in the photo (Figure 2 b). The results of distribution curves averaged over the length of the fibers (integral curve 1) and percentage of the fraction (along the length of the differential curve; 2) are shown in (Figure 3). The results of determining the nature of the fibers are shown in (Table 1).

Table 1.– Structural-dimensional characteristics of cellulose

Variables	Weighted average value on the length	Weighted average value on the width
Average length	2.5 mm	1.038 mm
Average width	31.2 µm	22.2 µm
Shapes average factor	90.8%	80.6%

Cellulose fibers from milk thistle are characterized by a heterogeneous shape of various lengths (Fig. 3); the fraction of long fibers with a length of 3.5 to 7.5 mm reaches more than 0.2, when the fraction of the rest does not exceed 0.08. The width of the fibers is in the range of 12–35 µm, then it is probably due to the fact that the stem consists of two different morphological parts – a flat flexible upper layer (Fig. 4 a) and the middle of a loose white part (Fig. 4 b) in a ratio of approximately 90 : 10.

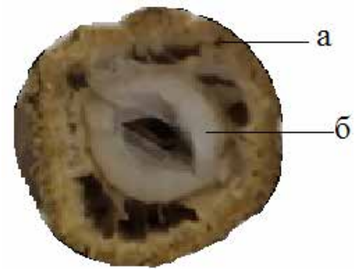


Figure 4. A cross section of the stem of a milk thistle

It is likely that small thin fibers are isolated from the middle part of the stem.

Sorption properties and IR-spectroscopy of milk thistle cellulose. After washing and drying, sorption properties of the resulting cellulose were determined, they are shown in (table 2).

From (table 2), it can be seen that the yield of cellulose is 26.0%, swelling in water is 187%, moisture sorption is about 11%, degree of polymerization is 870, ash content is 1.5% and whiteness is about 71%.

Study of IR-spectra of milk thistle cellulose. They are shown in (Figure 5). The spectra were obtained by pressing with KBr on an "IRAffinity-1" spectrophotometer [8–10].

Table 2 – Quality indicators of cellulose from milk thistle stems

yield of cellulose,%	Degree of swelling in water,%	* Moisture sorption,%	Degree of polymerization	Ash content,%	Whiteness,%
26.0	187	11.0	870	1.5	71.0

* *Relative humidity – 65%*

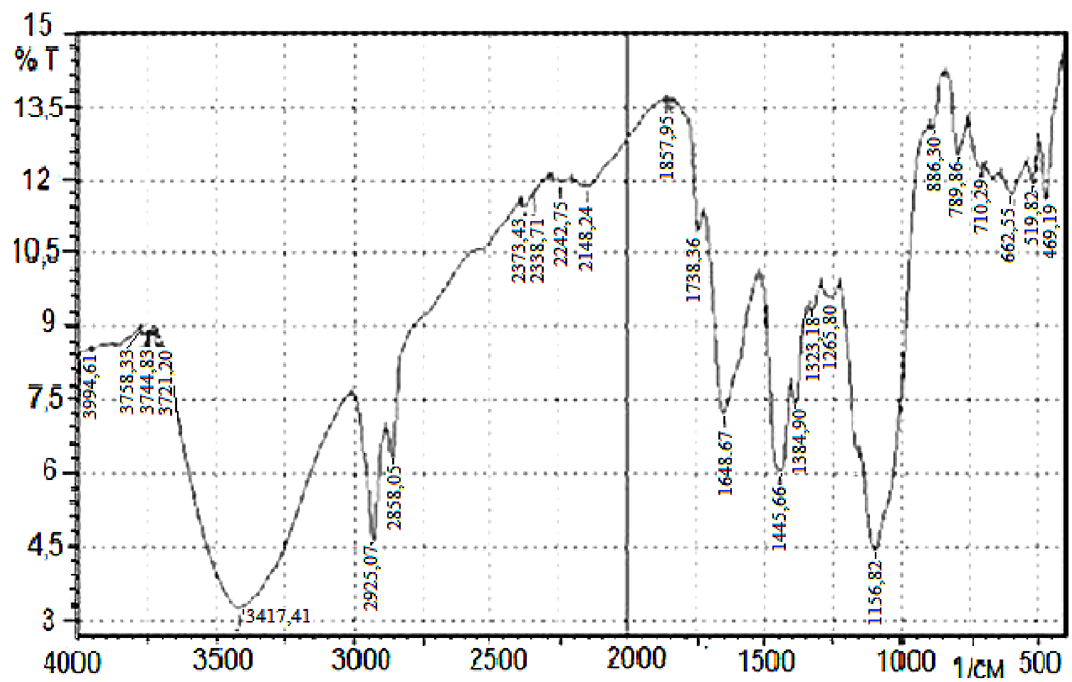


Figure 5. IR-spectra of milk thistle cellulose

Thus, the composition of milk thistle stems contains 26% cellulose with a degree of polymerization of 870, the average length and width of cellulose fibers is 2.5 mm and 22.2 μm, respectively. [11]

Analysis of the IR-spectra of cellulose shows that in the spectra in the range of 3250–3500 cm^{-1}, there is a broad blurry band with intense stretching vibrations caused by OH groups with a hydrogen bond.

Table 3.– Characteristic oscillations in the frequency of functional groups of milk thistle celluloses

Wave number, sm^{-1}	Group	Oscillation types	Intensity
3750	O-H (H bond is not formed)	Valence	Very high
3400	O-H (H bond is formed)	Valence	Broad and intensive
2900	$-CH_2$	Valence	High
2260–2190	$-C \equiv N$	Valence	Average
1980	$-C = C = C -$	Asymm. valent	High
1465–1400	$-CH_2$	Асимм. деформ.	
1400–1350	$-CH_3$	Симм. деформ.	
1280–1100	– C-O-C-	Asymm. valent	Intensive
1075–1020	– C-O-C-	Asymm. valent	Intensive

Conclusion

A method has been developed for obtaining cellulose from the stems of medicinal plant – milk thistle. The optimal conditions, the average values of the length and width of the fibers, and their fractional composition have been determined. Its sorption properties, degree of polymerization, ash content and other indicators have been studied. The chemical composition of the structures of the formed cellulose has been established by IR-spectroscopy.

References:

1. Jahan S. M., Labooni A. P., Noori A., Quaiyyum M. A., Process For The Production Of Dissolving Pulp From Trema Orientalis (Nalita) By Prehydrolysis Kraft And Soda-Ethylenediamine (Eda) Process Jehat et al. (2008). "Dissoving pulp from Tremaorientalis" Bio Resources,– No. 3. 2008.– P. 816–828.
2. Serkov A. T., Serkova L. A. Opredelenie nabuhaniya volokna v vode. [Determination of fiber swelling in water]. Him. volokna,– No. 5. 1974.– P. 70–71.
3. GOST 12603–97. Bumaga I karton. Metod opredeleniya poverhnostnoy vpityivaemosti bumagi kapelnyim sposobom. [Paper and cardboard. Method for determination of surface absorption of paper by drip method]. – Moscow, Standart inform Publ., 2005.– 8 p.
4. Kocheva L. S., Brovarova O. V., Sekushin N. A., Karmanov A. P., Kuzmin D. V. Strukturno-himicheskaya harakteristika nedrevesnyih vidov tsellyulozyi. [Structural and chemical characteristics of non-wood pulp.]. Forest Journal,– No. 5. 2005.– P. 87–93.
5. Konerinskiy N. N. Kompleksnaya himicheskaya pererabotka drevesinyi. [Complex chemical processing of wood]. Uchebnik dlya vuzov. Izdatelstvo AGTU, 2002.– 347 p.
6. Autlov S. A., Bazarnova N. G., Kushnir E. Yu. Mikrokristallicheskaya tsellyuloza: struktura, svoystva, poluchenie i oblasti primeneniya. [Microcrystalline cellulose: structure, properties, production and applications]. Himiya rastitelnogo syirya,– No. 3. 2013.– P. 33–41.
7. Zhbankov R. G. Infrakrasnyie spektryii struktura uglevodov. [Infrared spectra and carbohydrate structure]. Science and technology, 1972.– 456 p.
8. Moryiganov A. P. Perspektivnyie polimernyie materialyi dlya himiko-tekstilnogo proizvodstva [Promising polymeric materials for chemical and textile production]. Respublikanskiy him. Zhurnalob-va. im. D. I. Mendeleeva,– No. 1. 2002.– P. 58–66.
9. Eshbo'taev A. G. IQ-practical application of the method spectroscopy.– Tashkent, 2014.– 34 p.

10. Jahan S. M., RahmanH, Rani S. P., Rahman M. Department of Chemistry, Eden Girls College, Dhaka. Ethylenediamine in alkaline cooking of jute stick for producing dissolving pulp Bangladesh. Res.,– No. 2. 2015.– P. 7–14.
11. Israel A. U., Obot I. B., Umoren S. A., Mkpenie Vand Asuquo J. E. Production of Cellulosic Polymers from Agricultural Wastes E-Journal of Chemistry,– Vol. 5.– No. 1. 2008.– P. 81–85.