DEVELOPMENT OF TECHNOLOGIES FOR OBTAINING COMPOSITE MATERIALS BASED ON THE USE OF OILSEED FLAX FIBERS

РОЗРОБКА ТЕХНОЛОГІЙ ОДЕРЖАННЯ КОМПОЗИЦІЙНИХ МАТЕРІАЛІВ НА ОСНОВІ ВИКОРИСТАННЯ ВОЛОКОН ЛЬОНУ ОЛІЙНОГО

Olga Gorach [1)], Olena Dombrovska [2)], Anastasiia Tikhosova [2)]
[1)] Kherson State Agrarian and Economic University, Kherson / Ukraine;
[2)] Kherson National Technical University, Kherson / Ukraine;
Tel: +38 (050) 537-98-42; E-mail: olga_gorach@ukr.net
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ABSTRACT

The article examines the ways to solve the problem of developing a scientific basis for obtaining composite materials of different functional purposes from oilseed flax fibers. The paper covers theoretical and experimental research in the area of processing flax raw materials. The purpose of the study is to provide scientific substantiation of developing the technologies for obtaining fillers to reinforce composite materials. In order to do it, modification of oilseed flax fiber was performed and a technology for processing oilseed flax straw with regulated technological and performance characteristics was developed. The article also presents the results of the research on determining causes of low wettability of oilseed flax bast. In order to find out the causes of low bast wettability, research on examining chemical composition and anatomy of straw stems was conducted. The formulation for preparing the fiber aimed to be used as a filler for reinforcement of composite materials is offered. The study suggests evaluation of the quality of composite materials produced based on using modified oilseed flax fibers.

РЕЗЮМЕ

Статтю присвячено вирішенню проблем розвитку наукових основ одержання композиційних матеріалів різного функціонального призначення з волокон льону олійного. Стаття містить теоретичні та експериментальні дослідження в галузі переробки лляної сировини. Завданням роботи є наукове обґрунтування створення технологій одержання наповнювачів для армування композиційних матеріалів. Для цього в результаті досліджень було проведено модифікацію волокна льону олійного та розроблено технології переробки соломи льону олійного з регульованими технологічними та експлуатаційними властивостями. У статті також наведено результати досліджень з визначення причин низької змочуваності лубу льону олійного. Для цього були проведені дослідження з вивчення хімічного складу та анатомічної будови стебел соломи. Подано рецептуру підготовки волокна з метою застосування його в якості наповнювачів для армування композиційних матеріалів. Надано оцінку якості одержаним композиційним матеріалом виготовлених на основі застосування модифікованого волокон льону олійного.

INTRODUCTION

Oilseed flax is a valuable industrial crop of versatile use. Its botanical name *Linum usitatissimum* means "early maturing". According to the FAO data, the sown areas of oilseed flax cover almost 3.5 million hectares over the globe *(FAO et al, 2000).* Flax is grown in many countries of the world. Oilseed flax takes more than 70% of sown areas in the world. Recently, oilseed flax production has been intensively developed in Canada and the USA.

Analysis of the global oilseed flax production shows that the leading producers of oilseed flax in the world are Canada, China, India, Argentina, the USA and Russia. The total gross seed yield in these countries makes 1.2 million tons. In Ukraine this crop has not been paid attention over many past years due to social and political processes occurring in our country in the course of centuries. Nowadays oilseed flax returns to Ukraine. A wide range of varieties, their diversity and high profitability contribute to rapid spreading and annual growth of sown areas for this crop cultivation.

In Ukraine flax seeds are processed to obtain oil in small volumes, and most of them are exported. The crop stems are rarely processed and largely burnt in the fields. The research conducted at Kherson National Technical University (Ukraine) showed that oilseed flax stems contain a sufficient amount of fiber similar by its structure to short fiber of linen flax *(Tikhosova H.A. et al, 2011; Zhivetin V.V. et al, 2000).*

Oilseed flax stems, like linen flax stems, contain cellulose fiber in their bast. However, oilseed flax fiber has not been used in the global industry so far. After separating seeds, straw residues have been mostly burnt and mixed into soil, i.e. they were applied as fertilizer. Recently the issues of using oilseed flax have been paid much attention over the globe, but these studies are mainly aimed at processing seeds. Only few of them investigate the processing of oilseed flax stems to obtain fiber *(Gorach O. et al, 2018).* The article introduces the results of experimental studies on obtaining composite materials of different functional purposes from modified oilseed flax fibers. Examination and generalization of the results of the modern theoretical research in the area of production of composite materials allowed formulating the main hypothesis of the study: modification of oilseed flax fiber must result in the formation of its quality indexes, determined by physical-mechanical and chemical properties, parameters and modes of processing raw materials that can be considered as a complex criterion affecting final characteristics of composite materials of different functional purposes obtained from oilseed flax fiber *(Gorach O. et al, 2016; Chursina L. et al, 2017).* Confirmation of this hypothesis will support scientific development of innovative technologies for obtaining composite materials of different functional purposes by using oilseed flax fiber.

Application of oilseed flax straw will allow using annually renewable, environmentally friendly and safe raw materials, in particular, oilseed flax fiber to manufacture fillers for reinforcement of composite materials *(Gorach O.O., Hureieva S.S., Shot V.E. et al, 2019).* In order to determine suitability of the obtained bast for production of industrial textile, in particular, fillers for composite materials, production trials were carried out at the state enterprise “Plastmas” (Ukraine).

After reaching full maturity of oilseed flax straw stems and their mechanical processing by means of hackling, bast completely gets rid of its woody parts: phloem, xylem, parenchyma, and cuticle is left on the exterior of fibers, that adds hydrophobic properties to fibers. Cuticle is structureless transparent covering existing between fibers in the form of hairs. Cuticle consists of substances called cutins. As the study of Ordina N.A. proves, cutins are high molecular fatty acids, oxyacids, waxes and fats. They are resistant to the effects of strong chemical reagents such as concentrated acids and alkali. Cutins do not dissolve in sulfuric and chromic acids and even in copper-ammonia solution in which cellulose dissolves. It is availability of cutins on oilseed flax bast that causes its low wettability and a lack of adhesion to polymeric matrix *(Ordina N.A. et al, 1978).*

MATERIALS AND METHODS

Scientists from different countries of the world, namely, Langer E. (Germany), Kathleen V.D.V. (Belgium) carried out research on using natural fibers, in particular, oilseed flax fiber to obtain internal panels for cars. There are studies of Ton-That MT, Denault J. (Canada) on applying fibers to manufacture products for technical purposes. The papers of Mieleniak B., Bagley C., d'Anselme T., Guyader J. (USA) evidence that oilseed flax is traditionally grown on 700-800 thous. ha in the west of Canada. Annual yield of oilseed flax straw is nearly 1 million tons, and only 15-20% of this straw is used in production, mainly, to manufacture cigarette paper. Factories producing pulp and paper are located in the states North Carolina and New Jersey (USA). The studies of Pallesen (Denmark) highlight that fiber obtained by the technology of Rome research center (IPZS), after enzymatic treatment, extracting, active warm ventilation and carding process are used to produce composite materials, and shives – to produce chipboards. Zelenetskyi S. (Russia) succeeds in conducting research on modification of natural fibers to produce polymeric composite materials with natural fibers as fillers (*Mieleniak B. et al, 1985; Bagley C., d'Anselme T., Guyader J. et al, 1997; Guillay R., 1997; Joly C, Gauthier R., Chabert B. et al, 1996; Kathleen V.D.V. et al, 1998; Langer E. et al, 1998).*

Three varieties of oilseed flax – Evryka, Liryna and Aisberh were selected to conduct research and carry out experiments. They were significantly different from each other by technological characteristics. These varieties were grown in the climatic conditions of the South of Ukraine. Flax oilseed straw was obtained at the state enterprise “Research Farm “Askaniiske” of the National Academy of Agrarian Sciences of Ukraine.

The variety Evryka has been included in the List of Plant varieties of Ukraine since 2004. It was created by the Institute of Irrigated Agriculture of the NAAS of Ukraine using the method of hybridization with further individual-family selection. The variety is designed to obtain oil for food and industrial needs and protein meal to feed animals. The plant height is 57-62 cm. The stem is rounded. Its thickness equals 3-4 mm.

It has branches in its lower and upper parts. The length of the growing season is 81 days. The inflorescence is umbellate, 25-32 cm long. The fruit is a rounded capsule with 7-10 seeds. The seeds are black. The weight of 1000 seeds is 7-8 g. It is resistant to lodging, capsule cracking and seed shedding. The variety is stable in terms of yields. It is medium-resistant to pests and diseases, suitable for all growing zones. The seed productivity is 2880 kg/ha. Seed oil content is 39.4 %.

The variety Aisberh has been included in the List of plant varieties of Ukraine since 2001; it was created by the Institute of oilseed crops of the UAAS by the method of induced mutagenesis of Tsian variety seeds through irradiation with gamma-rays. The plant height is 54-57 cm, the duration of the growing season is 86-88 days. The variety is characterized by drought and lodging resistance. In the field experiments of the Institute of Agriculture of the Southern region of the UAAS (2004) its seed profitability was 2080-2180 kg/ha.

The variety Liryna has been included in the List of plant varieties of Ukraine since 2002; it was created by the German plant breeders of "Deutsche Saatveredelung AG". The variety is of an intensive type of usage. It generates high stable yields – 2500-2900 kg/ha. The growing season lasts 107-128 days. A large number of capsules with seeds ensure high yields even under low seeding density. The plant height is 58-78 cm. The weight of 1000 seeds is 5.6-7.2 g. Oil content is 44.3-46.1 %. Plants are characterized by uniform maturation. It is recommended for growing in forest-steppe and steppe zones.

In order to obtain fiber, the straw stems were mechanically processed using a modernized flax-scutching machine without a technological operation of flax straw preparation. The main reason for not preparing flax straw lies in the fact that agricultural enterprises are not interested in producing oilseed flax straw. It is related to additional financial, energy and labor costs, which agricultural producers cannot afford because of a lack of stable market for selling flax straw.

Having studied all the available methods for purifying bast to remove extraneous substances and waxes, the scientists of Kherson National Technical University developed a number of techniques to obtain cellulose from oilseed flax bast and fiber *(Gorach O.et al, 2016).* The most effective technique is acidification that ensures high cellulose output *(Gorach O.O., Hureieva S.S., Shot V.E. et al, 2019).*

This technique was used in boiling oilseed flax bast of the varieties under study obtained after double hackling.

The composition of boiling solutions and modes are given in Table 1.

Table 1

Modes and formulations for boiling oilseed flax bast to remove incrust and cutins from fiber

Technological operation	Composition of boiling solution, g/l	Mode of boiling
1. Oxidation boiling	Hydrogen peroxide (100%) – 4.0	1. Boiling at 100 ºC – 60-180 min.
	Sodium hydroxide – 10.0	2. Rinsing with cold water – 10 min.
	Calcined soda – 2.0	3. Acidification with sulfuric acid (2 g/l) – 10 min.
	Sodium silicate – 5.0	4. Rinsing with cold water – 20 min.
	sodium tripolyphosphate – 1.0	
	Wetting agent – 0.3	
2. Fiber pressing out and drying	-	Pressing out to moisture content of 60% and drying at 100 ºC

In order to find out the cause of such low wettability, the chemical composition of oilseed flax bast (Table 1) and the anatomy of oilseed flax stems were examined.

RESULTS

After processing straw stems of oilseed flax using a modernized flax-scutching machine, samples of the obtained bast were selected and its physical-mechanical properties were determined: a mass portion of shives and impurities, breaking load, bast output *(Kathleen V.D.V. et al, 1998; Langer E. et al, 1998).*

Analysis of the obtained results showed that bast, obtained from oilseed flax straw stems, has a high mass portion of shives – 27.2-30.6 % and low strength – 3.9-5.0 daN. However, bast output after mechanical processing by a modernized flax-scutching machine is sufficiently high: with the average stem length of 32.9-

36.0 cm, this index amounts to 32.4-39.4 %. Thus, we can draw a conclusion that bast-fiber raw materials with such high indexes of a mass portion of shives and impurities does not meet the requirements of producers of industrial textile for different purposes. Therefore, in the further research bast was additionally purified from shives by means of the operation of machine hackling.

The experimental research resulted in obtaining bast with high quality indexes due to optimization of the modes and parameters of mechanical processing of oilseed flax straw stems. After the second hackling by the hackling machines with the rotational frequency of the main cylinder of 555 min^{-1} and the opening between it and the knife of 1.5 mm, bast with the following physical-mechanical characteristics was obtained: the staple length – 18.10 mm; the mass portion of shives and impurities – 0.01 %; linear density – 0.35 tex. In order to determine suitability of the obtained bast for manufacturing fillers for reinforcement of composite materials, production trials were carried out at the state enterprise "Plastmas" (Ukraine). As it is known, the main index of filler adhesion to phenol formaldehyde resins is wettability. Currently at the enterprise "Plastmas" cotton lint with the wettability of 120 g is used as a filler. Therefore, the wettability of oilseed flax bast of the three varieties (Aisberh, Evryka and Liryna) was determined. The results of the experimental research showed that the average values of this index ranged from 5.0 to 6.3. Analysis of the obtained results indicate that bast fiber of oilseed flax has wettability that is 24 times less than that of cotton lint. According to the requirements of the regulating documents, a filler for phenoplasts should have wettability of 116-120 g. Therefore, oilseed flax bast, obtained after mechanical processing of straw stems is unsuitable for producing fillers to reinforce composite materials on the basis of phenol formaldehyde resins because of its low wettability *(Gorach O.O., Hureieva S.S., Shot V.E. et al, 2019).*

After mechanical processing of straw stems using a modernized flax-scutching machine and hackling machines, cuticle remained on fiber. It is evidenced by micro-photos of the cross sections of oilseed flax blast of the varieties under study obtained as a result of mechanical processing (Fig. 1-3). Microphotographs were taken using microscopes.

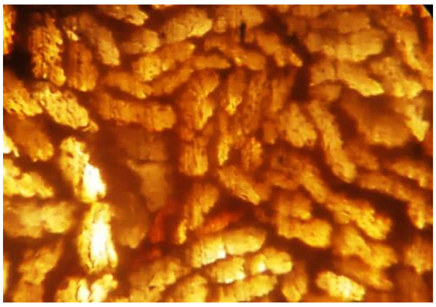

Fig. 1 - Micro-photo of the cross section of oilseed flax bast of the variety Aisberh

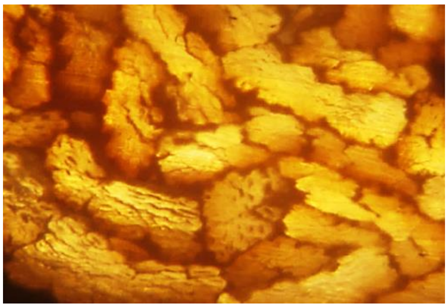

Fig. 2 - Micro-photo of the cross section of oilseed flax bast of the variety Evryka

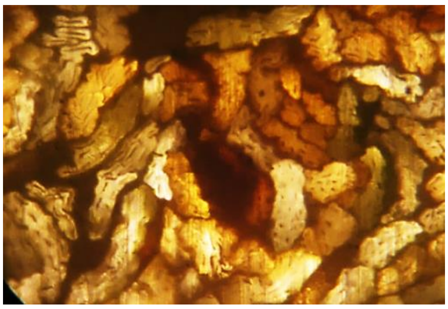

Fig. 3 - Micro-photo of the cross section of oilseed flax bast of the variety Liryna

Thus, the results of experimental research allowed establishing that after mechanical processing of straw stems of oilseed flax by means of a modernized flax-scutching machine and double hackling of the obtained bast using a hackling machine to remove incrusts and cutins, it is necessary to perform chemical processing (boiling) of bast.

Therefore, the second stage of developing the technology for obtaining fillers to reinforce composite materials from oilseed flax is chemical modification, i.e. purifying fibers to remove cellulose and substances accompanying cutin – high molecular fatty acids, oxyacids, waxes and fats. The chemical composition of oilseed flax bast of the varieties under study prior to boiling is given in Table 2.

Table 2

The chemical composition of oilseed flax bast of the varieties under study prior to boiling

Oilseed flax variety	Content of chemical components, %				
	cellulose	lignin	pectin substances	wax substances	mass proportion of hemicellulose
Aisberh	82.27	1.62	4.21	3.85	6.47
Evryka	81.37	1.31	4.03	3.74	6.77
Liryna	82.12	1.34	4.15	3.82	6.72

Table 1 shows that oilseed flax bast has a high content of lignin – 1.31-1.62 %, pectin substances – 4.03-4.21 %, wax substances – 3.74-3.85 % and hemicellulose – 6.47-6.77 %. Thus, oilseed flax bast is hardly suitable for using in production industries because of a high content of substances accompanying cellulose, therefore, in order to increase its wettability and adhesion, bast was boiled.

Table 3 presents the results of the research on wettability and chemical composition of oilseed flax fiber of the variety Aisberh after boiling for 1, 2 and 3 hours. The results of the chemical analysis of oilseed flax fiber composition of the three varieties under study allowed establishing that different varieties of oilseed flax have similar content of chemical components. Therefore, we used mediated values of the experimental research of the oilseed flax variety Aisberh.

Table 3

Physical-chemical indexes of fiber quality of the oilseed flax variety Aisberh

Number of experi-ments	Physical-chemical indexes of oilseed flax fiber					
	Duration of boiling, hours					
	1		2		3	
	wettability, g	mass portion of α-cellulose, %	wettability, g	mass portion of α-cellulose, %	wettability, g	mass portion of α-cellulose, %
1	104.88	87.32	105.92	87.78	122.80	90.96
2	104.45	86.67	104.86	88.28	123.50	89.64
3	105.22	86.83	105.76	87.53	119.70	90.04
4	104.98	87.06	105.58	87.22	124.70	89.76
5	105.15	86.54	104.84	87.58	123.20	89.67
Average value	104.94	86.88	105.39	87.68	122.78	90.01

Analysis of the data in Table 3 shows that boiling oilseed flax bast of the variety Aisberh according to the mode given in Table 2 for 1, 2 and 3 hours allowed obtaining fiber which meets the requirements for wettability of fillers set by the enterprise "Plastmas". In the course of the previous research it was determined that this index for oilseed flax bast was only 5.0-6,3 g, and due to the technological operation of boiling the fiber, wettability increased to the necessary indexes – 104.94-122.78 g, a mass proportion of α-cellulose rose from 76.88 % in the control variant (without boiling) to 86.88-90.01 %.

The results of the research on bast chemical composition in the control variant and oilseed flax fiber of the variety Aisberh after boiling for 1, 2 and 3 hours are given in Table 4. According to the methods for conducting experiments, we considered the average mean of five parallel measurements of the content of cellulose, lignin, pectin and wax substances and the mass portion of hemicellulose to be the result of the trial.

Table 4

Chemical composition of oilseed flax fiber

Time of boiling, hrs	Content of chemical components, %				
	cellulose	lignin	pectin substances	wax substances	Mass portion of hemi-cellulose
control	82.27	1.62	4.21	3.85	6.47
1	91.27	0.31	-	0.04	3.77
2	93.22	0.64	-	0.02	3.72
3	94.06	0.68	-	0.01	3.05

Analysis of the data in Table 4 shows that boiling oilseed flax bast of the variety Aisberh according to the mode given in Table 2 for 1, 2 and 3 hours allowed obtaining fiber rich in cellulose and free of wax pectin substances. Cellulose content increased from 82.27 % to 91.27 % after boiling bast for 1 hour, to 93.22 % – after boiling for 2 hours and to 94.06 % – after boiling for 3 hours. The mass proportion of hemicellulose fell from 6.47% to 3.05%, and wax substances were almost removed. The index of their content in bast – 3.85%, and after chemical processing it dropped to 0.01-0.04%. However, by the relative breaking load and the relative breaking extension, flax fiber is significantly inferior to cotton fiber.

After examining physical-mechanical indexes of oilseed flax fiber and cotton fiber, comparative analysis of physical-mechanical properties of phenoplasts, reinforced with cotton and flax fibers, was performed. In order to do that, in the course of the experiment at the laboratory LLC "RDS IL" (Ukraine), were made experimental samples of the composite materials in which oilseed flax and cotton fibers were used as fillers.

The quality of the obtained phenoplasts, reinforced with oilseed flax and cotton fibers was evaluated according to the requirements of TU U 25.2-32512498-001-2004 with amendments 1, 2, 3, 4 "Pressing phenolic mass" *(TU U 25.2-32512498-001-2004 with amendments 1, 2, 3, 4. et al, 2004).* The obtained composite materials were compared by their color, appearance, fluidity, notch-toughness by Charpy impact test on the samples without a cut and electrical robustness. Physical-mechanical quality indexes of the phenoplasts У1-301-07, reinforced with cotton fiber and oilseed flax fiber are given in Table 5.

Table 5

Quality indexes of phenoplasts reinforced with oilseed flax and cotton fibers

№	Quality indexes	Type of a filler					
		standard indexes	control variant	oilseed flax fibers of the variety Aisberh after boiling for, hrs			cotton
				1	2	3	
1.	Color	from light-brown to dark-brown	complies	complies	complies	complies	complies
2.	Fluidity, mm	40-140	200	185-190	180-190	200	125
3.	Appearance of pressed samples	without cracks and blisters	does not comply	complies	complies	complies	complies
4.	Notch-toughness by Charpy on the samples without a cut, kJ/m^2 ($daN \cdot cm/cm^2$), not less	8.8 (9.0)	16.43	9.5	11.3	8.2	12.38

Table 5
(continuation)

№	Quality indexes	Type of a filler					
		standard indexes	control variant	oilseed flax fibers of the variety Aisberh after boiling for, hrs			cotton
				1	2	3	
5.	Bending stress under damage, MPa (daN·cm/cm^2), not less	58.8 (600)	632.55	571	562	706	692
6.	Specific volume electric resistivity, Ω ·cm	1 ·10^9	-	2.89·10^{12}	1.96·10^{13}	6.9·10^{13}	5.0 ·10^9
7.	Electric robustness, kW/mm	6.0	-	10.8	10.6	12.0	-

Analysis of the quality indexes of the obtained composite materials given in Table 7 evidences appropriateness of reinforcing phenoplasts with oilseed flax fibers on the basis of using thermosetting resin. Boiled for 1 and 2 hours, oilseed flax fiber of the variety Aisberh complies with the requirements of TU U 25.2-32512498-001-2004 by all the indexes under study except bending stress under damage. It equals to 571 daN/cm^2 after 1 hour of boiling and 562 daN/cm^2 after 2 hours of boiling, i.e. a bit less than the standard index that must be not less than 600 daN/cm^2, which is determined by low strength of oilseed flax fiber.

CONCLUSIONS

Oilseed flax fiber can be used as fillers to reinforce composite materials after mechanical processing and chemical modification. The results of the research on chemical composition of bast obtained after mechanical processing and analysis of the anatomy of oilseed flax stem structure allowed determining that high hydrophobic behavior and low wettability of bast are caused by the presence of cuticular layer on fiber. It consists of high molecular fatty acids, wax substances and fats, and their average content in bast equals to 3.85%. It was established that in order to use oilseed flax bast for reinforcing composite materials, in addition to mechanical purifying to remove extraneous substances and shives, it is necessary to perform chemical cleaning of cellulose to remove incrusts, i.e. lignin and pectin substances, and also high molecular fatty acids, wax substances and fats.

Experimental analysis of the advantages and disadvantages of different methods for obtaining cellulose was performed. A new acidification method was chosen for purifying bast to remove substances accompanying cellulose. The research results showed that after boiling oilseed flax bast of the variety Aisberh for 3 hours were obtained samples of phenoplast that complied with the requirements of TU U 25.2- 32512498-001-2004 by all the indexes except for notch toughness which was 8.2 kJ/m^2, whereas the standard index equals not less than 8.8 kJ/m^2. The samples of phenoplast obtained by means of oilseed flax bast without boiling, i.e. the control variant, complied with TU U 25.2-32512498-001-2004 by physical-mechanical properties, but the disks had numerous blisters and cracks, that did not make it possible to test these samples by electric indexes.

Thus, analyzing the results of the experimental research, can be drawn the conclusion that oilseed flax fiber after boiling according to the suggested method can be used as a reinforcing component to produce composite materials on the basis of thermosetting resin individually or in combination with other types of cellulose fiber. Therefore, scientific development of innovative technologies for obtaining composite materials from oilseed flax fibers of different functional purposes will allow using annually renewable, environmentally-friendly and safe raw materials that will contribute to the development of resource-saving technologies and will allow improving the environment and supplying the global market with new commodities of different functional purposes.

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