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Content

SCIENTIFIC DEVELOPMENT OF INNOVATIVE TECHNOLOGIES OF OBTAINING COMPOSITE MATERIALS FROM OF OILSEED FLAX FIBERS

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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. Composite materials came to the forefront of production and demand for goods several decades ago, thanks to unsurpassed specific and mechanical properties as a result of growing consumer and industry demand for high-tech materials and structures. However, the combination of natural fibers with a polymeric material or matrix increases the difficulty of the process of forming composites and, as a rule, leads to problems in the physicochemical processes of interaction of the matrix and the filler. Composites with synthetic fillers have obvious advantages, but their disposal is difficult, requires the development of environmental processing technologies. The best way to save the environment when processing composite materials is to use non-toxic natural materials for their production, but this requires the development of innovative technologies for forming composite materials with natural fibrous fillers. 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, we performed modification of oilseed flax fiber and developed technologies for processing oilseed flax straw with regulated technological and performance characteristics. 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, we conducted research on examining chemical composition and anatomy of straw stems. The formulation for preparing the fiber aimed to be used as filler for reinforcement of composite materials is offered. The study suggests evaluation of the quality of composite materials produced on the basis of using modified oilseed flax fibers.*

Keywords: *oilseed flax, straw, bast, fiber, composite materials.*

1 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 [1, 2]. Flax is grown in many countries of the world (Figure 1). 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, 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.

Figure 1 Flax-growing countries (hatched)

Recently, Western Europe and other countries of the world have shown great interest to the use of oilseed oil in manufacturing different products of industrial purposes. The global tendency for expanding sown areas to grow this crop can be explained by the fact that it is a main source for production of industrial oil and is characterized by excellent biological and technical properties: high drought-resistance; lodging-resistance; fast maturation, it matures later than cereal crops. Moreover, oilseed flax is a good pre-crop for winter crops, high-protein feeds for animals, it has high seed yields (over 20 c/ha) and a high commodity price on the international market [1]. The article presents the results of theoretical and experimental research aimed at developing technology for the production of fillers for the reinforcement of composite materials.

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.

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.

The article introduces the results of theoretical and 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. Confirmation of this hypothesis will support scientific development of innovative technologies for obtaining composite materials of different functional purposes by using oilseed flax fiber.

1.1 Literature review

The leading world scientists L.A. Chusina, H.A. Tikhosova (Ukraine) and V.V. Zhivetin (Russia) prove that oilseed flax fiber is suitable for manufacturing industrial textile of different purposes. The scientists of many 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 chip boards. Zelenetskyi S. (Russia) succeeds in conducting research on modification of natural fibers to produce polymeric composite materials with natural fibers as fillers [2-14].

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. 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).

To conduct research and carry out experiments, we selected three varieties of oilseed flax – Evryka, Liryna and Aisberh, 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 in the List of Plant varieties of Ukraine since 2004; it was created by the Institute of Irrigated Agriculture of the NAAS of Ukraine by 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 being 3-4 mm, with branches in 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 28.8 c/ha. Seed oil content is 39.4%.

The variety Aisberh has been 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 20.8-21.8 c/ha.

The variety Liryna has been 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 25-29 c/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.

2 RESEARCH METHODOLOGY

The tasks set in the work were solved with the help of modern methods of theoretical and experimental research. Studies of chemical components were performed by traditional methods. Studies of anatomical structure were performed using microscopes. 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".

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.

After processing straw stems of oilseed flax using a modernized flax-scutching machine, we selected samples of the obtained bast and determined its physical-mechanical properties: a mass portion of shives and impurities, breaking load, bast output [15, 16].

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 do not meet the requirements of producers of industrial textile of different purposes. Therefore, in our 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, we obtained bast with the following physical-mechanical characteristics: 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, we carried out field trials 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 filler. Therefore, when carrying out the research, we determined the wettability of oilseed flax bast of the three varieties under study: Aisberh, Evryka and Liryna. The results of the experimental research showed that the average values of this index ranged from 5.0 to 6.3 g. Analysis of the obtained results indicate that bast fiber of oilseed flax has wettability that is 24 times less than cotton lint does. According to the requirements of the regulating documents, the 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 [17].

To find out the causes of such low wettability, the chemical composition of oilseed flax straw was studied and the anatomical structure of oilseed flax straw (Figure 2) was studied.

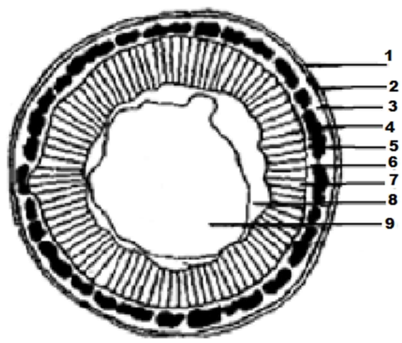

Figure 2 Anatomical structure of oilseed flax straw: 1 - cuticle; 2 - epidermis; 3 - parenchyma cow; 4 - fibrous bundles; 5 - floem; 6 - cambium; 7 - xylem; 8 - parenchyma; 9 - cavity

The study of anatomical structure of lounge oilseed flax showed that from the outside the stem is covered with a cuticle 1, under it there is an epidermis 2, behind it - a cow parenchyma 3, which surrounds fibrous bundles 4. From the inside of the fibers located floem 5, closer to the center there are cambiums 6 and wood 7, 8 (xylem and parenchyma), and the center of the stem is cavity 9.

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 [18]. They are resistant to the effects of strong chemical reagents such as concentrated acids and alkali. Cutins do not dissolve in sulphuric 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.

Thus, the results of the theoretical and 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 bust.

3 RESULTS

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.

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. The most effective technique is acidification that ensures high cellulose output. This technique was used in boiling oilseed flax bast of the varieties under study obtained after double hackling. The stages of chemical treatment of oilseed flax fiber are given in Table 1.

Table 2 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. Analysis of the data in Table 2 shows that boiling oilseed flax bast of the variety Aisberh according to the mode given in Table 1 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 we 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%.

Table 1 Stage of chemical treatment of oilseed flax fiber

Stage of treatment	Composition [g/l]	Mode
1. Oxidation boiling	Hydrogen peroxide (100%) – 4.0	1. Boiling at 100°C, 60-180 min.
	Sodium hydroxide – 10.0	2. Rinsing with cold water, 10 min.
	Calcined soda – 2.0	3. Sulfuric acid H_2SO_4 concentration 96% (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 drying	-	Pressing out to moisture content of 60% and drying at 100°C

Table 2 Physical-chemical indexes of fiber quality of the oilseed flax variety Aisberh

Number of experiments	Physical-chemical indexes					
	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

Table 3 Physical-mechanical quality indexes of the phenoplasts with oilseed flax and cotton fibers

№	Quality indexes	Type of filler					
		standard indexes	control variant	oilseed flax fibers of the variety Aisberh after boiling [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 samples without a cut,[kJ/m2], not less	8.8 (9.0)	16.43	9.5	11.3	8.2	12.38
5.	Bending stress under damage [MPa], 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	-

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” [19]. The obtained composite materials were compared by their color, appearance, fluidity, notch-toughness by Charpy on the samples without a cut and electrical robustness. Physical-mechanical quality indexes of the phenoplasts U1-301-07, reinforced with cotton fiber and oilseed flax fiber are given in Table 3.

Analysis of the quality indexes of the obtained composite materials given in Table 3 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 kgf/cm^2 after 1 hour of boiling and 562 kgf/cm^2 after 2 hours of boiling, i.e. a bit less than the standard index that must be not less than 600 kgf/cm^2 that is determined by low strength of oilseed flax fiber [20].

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%. We 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 perform chemical cleaning of cellulose to remove incrusts, i.e. lignin and pectin substances, and also high molecular fatty acids, wax substances and fats.

After performing theoretical analysis of advantages and disadvantages of different methods for obtaining cellulose, we chose a new acidification method for purifying bast to remove substances accompanying cellulose developed by the scientists of Kherson National Technical University. The research results showed that after boiling oilseed flax bast of the variety Aisberh for 3 hours we obtained samples of phenoplast that complied with the requirements of TU U 25.2-32512498-001-2004 by all the indexes except notch toughness being 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 experimental studies, we can conclude that the flaxseed fiber after boiling by the proposed method can be used as a reinforcing component for the manufacture of composite materials based on thermosetting resin alone or in combination with other types of cellulose fiber. Therefore, the scientific development of innovative technologies for obtaining composite materials from oil flax fibers for various functional purposes will allow the use of annually renewable, environmentally friendly and safe raw materials, which will promote resource-saving technologies and improve the environment and supply new products for various functional purposes.

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