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Influence of the Fibrous Structure on the Physical and Mechanical Characteristics of Insulating Slabs from Flax Noils

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 $Abstract\,-\,$ The paper presents results of analysis of thermal insulation materials based on plant fibers. It is suggested to use secondary raw materials of flax processing plants – flax noils – as a fiberfill in production of insulating slabs.

The paper presents results of research on the microstructure of flax noils and flax fibers conducted using light microscopy and electron microscopy, respectively. A set of experiments aimed at determining physical and mechanical characteristics of thermal insulation materials has been conducted. Test results attest higher efficiency of the insulating slabs made from flax noils in comparison with flax fiber-based thermal insulation material. The main factors facilitating reduction of thermal conductivity coefficient of thermal insulation material made from flax noils have been determined.

Keywords – Density, flax fibers, flax noils, mechanical strength, microscopy, thermal conductivity coefficient, thermal insulation material.

I. INTRODUCTION

In the conditions of global energy crisis, development of manufacturing of efficient building materials and economy of fuel and energy resources, including minimization of heat loss through building, construction and technological equipment envelopes, remain one of the priority tasks. Improvement of thermal resistance of building envelopes with the help of thermal insulation materials is the main solution in reduction of heating energy costs.

In the European counties, in the field of thermal insulation material manufacturing, special attention is paid to the use of fibrous plant residues, which remain after harvesting and processing of crops [1]. Growing application of plant residues will not only ensure construction industry with additional raw materials and widen the range of locally sourced building materials, but will also promote preservation and rational use of natural resources [2]. Apart from significant saving of heating energy resources, plant-based insulating materials may help improve ecological conditions, including reduction of CO₂ emissions. Therefore, development of new efficient thermal insulation materials based on plant fibers meeting the abovementioned criteria is very topical in the field of insulation material manufacturing.

Insulating slabs *Ecoteplin* produced in Russia can be considered a successful development in this area. The slabs are produced from flax fiber and a binder – starch. Borates are used as fire and bioresistance agents [3], [4]. The slabs are used both in low-rise construction and for thermal and acoustic insulation of multi-apartment buildings. Material *Ecoteplin* has antiseptic

properties and is fully safe for human health. Density of thermal insulation slabs is from 32 kg/m^3 to 34 kg/m^3 , they ensure thermal conductivity of $0.037 \text{ W/(m^{\circ}\text{C})}$, water absorption capacity by volume -0.5 and vapor permeability $-0.4 \text{ mg/(m \cdot h \cdot Pa)}$, flammability class $\Gamma 1$. High cost and application limitations due to low density impede wide use of *Ecoteplin*. In addition, the insulating material is highly water permeable and gets wet quickly, therefore the insulated walls should be additionally insulated with a special membrane.

JSC AKOTERM FLAKS (Orekhovsk, Belarus) offers thermal insulation slabs containing flax fibers (85 %) as a fiberfill. Synthetic fibers are used as a binder (15 %) [5]. The material is used in low-rise construction for thermal insulation of walls and floor slabs. At the density of 32 kg/m³, thermal conductivity rate of AKOTERM FLAKS slabs is 0.038 W/(m°C), acoustic absorption coefficient – 0.4, vapor permeability coefficient – 0.4 mg/(m·h·Pa), flammability class Γ4. High fire hazard is the main drawback of this insulant. The material is characterized by low density, and that does not allow producing rigid compression resistant insulating slabs, which is another factor limiting application of AKOTERM FLAKS.

Production of thermal insulation materials on the basis of wood fibers is one of the most advanced thermal insulation production technologies. Thermal insulation wood fiberboards take a significant market share. These insulting materials have the following technical characteristics: density from 15 kg/m³ to 250 kg/m^3 , thermal conductivity $0.06 \text{ W/(m}^\circ\text{C})$, flexural strength from 0.5 MPa to 1.2 MPa [6].

Also, low-density dry laid-up thermal-acoustic insulation slabs consisting from defibered boon particles with addition of up to 8 % tar and 1 % paraffin [7]. In production of thermal insulation materials, boon should not be preliminarily purified, grinded and graded. This is a very simple technology; it consists in defibering wetted flax boon on the defibrillator and preparation of homogeneous boon-glue mass. Such slabs are produced in Thailand, Great Britain and Canada. Studying the properties of slabs made from defibered flax boon at Kostroma State Technological University it was determined that at the density of 40 kg/m³ this thermal insulating material has thermal conductivity of 0.101 W/(m°C).

Research was conducted at Riga Technical University with an aim to develop thermal insulation materials based on hempshives, hydraulic lime and various additives. As a result, thermal insulation materials with the density of 312–337 kg/m³ were obtained, with thermal conductivity ranging from 0.101 W/(m°C)

to $0.087~W/(m^{\circ}C)$ and compressive strength from 0.179~MPa to 0.222~MPa [8]. This material can be used in low-rise construction.

II. MATERIALS AND METHODS

Flax noils and flax fibers No 10–12 were used in research, which were produced at Postavy Flax Mill (Belarus) and meeting the standard GOST (*FOCT*) P 53486 [9] and STB (*CTB*) 1195–2008 [10].

Sodium silicate produced by OJSC *Domanovsky Production* and *Trade Plant* was used as a binder in Water glass silicate is characterized by the following technical properties: silica modulus – 2.9, pH from 11 to 12, density from 1.45 to 1.47 g/cm³, viscosity – 0.0194 Pa·s [11].

Analysis of the microstructure of flax noils and the surface of flax fibers was conducted using light microscopy. Analysis of the microstructure on the optical microscope *Altami MET 5 C* allows obtaining images of the surface structure of the analyzed objects. The microscope has a special lamp installed at the lens that is based on light reflection. The obtained images were displayed on the computer screen and recorded on the hard disk. Application of *Altami Studio* software allows combining the obtained consequent images of sample fragments to enlarge the area of an image of the analyzed material.

Analysis of the morphological properties of flax fibers was performed with a scanning electron microscope *JSM-5610 LV*. Low-vacuum microscope mode ensures obtaining an image of a real surface without prior preparation of the samples. Microscope interface software that contains control program *INCA Energy 450* can be launched on a personal computer and is used for display, processing and storage of test results.

The main set of experiments aimed at determining physical and mechanical properties of the samples was conducted separately for the fiberfill made of flax noils and flax fibers, and also for the fiberfill made of the mixture of flax noils and flax fibers at the ratio of 80:20. Sodium silicate was used as the binder. Flax fibers up to 25 cm long and flax noils 5-10 cm long were used in analysis. A definite procedure was used in the preparation of the samples. First, component dosing was performed. Preparing flax noil samples, sodium silicate was added and mixed with the fiberfill. Insulating slabs based on flax fibers and the mixture of flax noils and fibers were prepared in sequence impregnating the layers of fiberfill by the binder. Slabs were formed by pressing under 0.002-0.007 MPa. The samples were cured in molds for 6 hours at temperature (20 ± 2) °C, then dried for 4 hours in the chamber drier at temperature from 45 °C to 55 °C. Average density and thermal conductivity were determined according to GOST (ΓOCT) 17177 [12] and STB (CTE) 1618 [13], respectively, on $250 \text{ mm} \times 250 \text{ mm} \times 30 \text{ mm}$ sample slabs. Compressive strength at 10 % deformation was tested on 100 mm × 100 mm × 100 mm sample cubes according to GOST (*FOCT*) 17177. Figures 1 and 2 demonstrate testing for thermal conductivity and compressive strength at 10 % deformation using a thermal conductivity meter $UT\Pi$ - $M\Gamma$ 4 and press ΠM - $2M\Gamma$ 4, respectively.

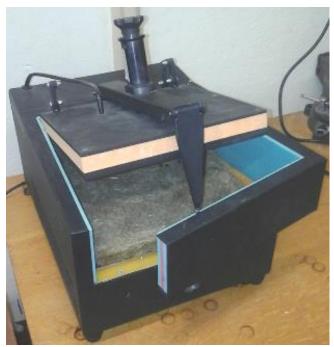


Fig. 1. Thermal conductivity test using $UT\Pi$ - $M\Gamma$ 4.



Fig. 2. Compressive strength test at 10 % deformation on $\Pi M\text{-}2M\Gamma 4$ press.



Fig. 3. Light microscopy of a flax fiber.

III. DETERMINING THERMAL CONDUCTIVITY OF PLANT FIBER-BASED INSULATION MATERIALS

At the initial stage of research, the following plant fibers were considered as potential raw materials for obtaining efficient fiberfill for thermal insulation slabs: nettle, reed, flax, flax noils, oil palm bark. Sodium silicate was used as binder. Thermal conductivity was determined for fiber fill of various origin in thermal insulation materials with density of 50 kg/m³. Binderfiberfill mass ratio for all samples was 1:5.

TABLE I
THERMAL CONDUCTIVITY OF PLANT FIBER-BASED THERMAL
INSULATION MATERIALS

No. of the sample	Fiberfill (country of origin)	Thermal conductivity, W/(m°C)	
1	Nettle (Belarus)	0.041	
2	Reed (Portugal)	0.055	
3	Flax (Belarus)	0.047	
4	Flax noils (Belarus)	0.041	
5	Fibers of oil palm bark (Malaysia)	0.054	

Among the obtained materials made from plant fibers, the lowest rate of thermal conductivity was demonstrated by the samples made from nettle and flax noils, it equals 0.041 W/(m°C), which is by 0.006–0.014 W/(m°C) lower than the rates of materials based on other types of fiberfill analyzed. Taking into consideration the fact that in the territory of Belarus the technology of harvesting, processing and obtaining nettle fibers is not operational on the industrial scale, application of flax noils and flax fibers is the optimal solution for obtaining efficient plant-based insulation material, which does not consist organic, synthetic binder with low flammability and is ecologically safe for human health. The microstructure of the flax noil and flax fiber was tested and analyzed to determine the parameters that allow ensuring good physical and mechanical characteristics of thermal insulation materials.

IV. APPLICATION OF LIGHT AND ELECTRONMICROSCOPY IN ANALYSIS OF MICROSTRUCTURE OF FLAX NOILS AND FLAX FIBERS

The images of a flax fiber (Fig. 3) and a flax noil (Fig. 4) were obtained with light microscopy combining the pictures of the consequently placed fragments of the analyzed sample. For example, Fig. 4 presents an image of a 6 cm long flax noil. The fragment highlighted by the frame in the figure is presented in the enlarged form in Fig. 4 b.

The obtained images attest that a flax fiber consists of a conglomeration of thinner fibers – bundles of elementary fibers firmly attached to each other through elementary fibers, as a result firm longitudinal connection of the fibrous system of a flax stem is ensured. At the same time, a flax noil consists from ragged bundles of elementary fibers (Fig. 4 a). Elementary fibers in a noil periodically interconnect with each other due to chaotic contact connections. As a result, mesh fiber frame is formed that ensures firm longitudinal connection of the entire structure of the flax noil. The noils get interconnected due to side branches in the form of elementary fibers forming spatial mesh fibrous system. Elementary fiber is a spindle shaped plant cell. In the microimage (Fig. 4b) in the reflected light it can be seen that elementary fibers have narrow inner channels from 4 µm to 6 µmin diameter. The length of elementary fibers varies from 10 mm to 40 mm at diameter from 8 μ m to 12 μ m.

Several concentrically arranged layers differing by various degree of refraction are distinguished in the structure of an elementary fiber [14]. The first region of the cover is relatively thin, it mainly consists of pectic substances that glue cells among each other. The primary wall consisting of cellulose with significant hemicellulose content, pectins and often lignin form the next region. The secondary wall is also formed from cellulose and is characterized by various rates of refraction due to smaller amount of additions of the above-mentioned substances. At the initial stage of development, elementary fibers are essentially round cells filled with plasma. As the respective zone grows, these cells elongate, their cover considerably thickens from the inside and reaches such degree of thickness that the inner cavity with plasma can be spotted only as a very narrow channel.

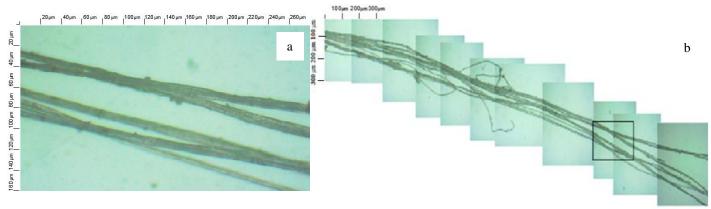


Fig. 4. Light microscopy of a flax noil: a – outer appearance of the noil, b – elementary fibers in the noil.

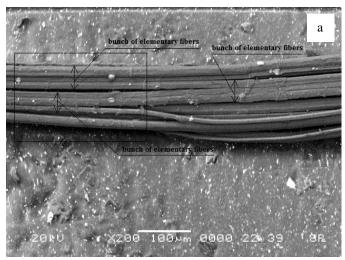
Thus, the obtained results of light microcopy aimed at the analysis of the fiber structure (Fig. 4 b) fully attest the presence of a void channel in an elementary fiber.

Application of a scanning electron microscope allowed visually attesting that the flax fiber consists of bundles of elementary fibers (Fig. 5a). In the image of the fiber, the frame highlights the fragment that is enlarged in Fig. 5 b. White formations marked by the arrow are micro fibrilles forming due to presence of non-cellulose polysaccharides and pectin [15]. The conducted electron microscopy analysis attests morphometric parameters of elementary fibers determined while studying flax noils with light microscopy, it also allows determining that the size of bundles is from 50 μm to 70 μm in diameter, given there are from 10 to 20 elementary fibers in the structure of the bundle. The data of microscopic analysis demonstrate that a less "coarse" and more efficient thermal insulation micro-mesh structure can be formed from flax noils in comparison with the materials based on flax fibers.

Heat transferrin fibrous materials is performed by means of heat transfer from one fiber to another, and also by means of convective transfer of inter-fiber air. Heat transfer decreases along with the thinning of fibers, because heat energy is spent in heat transfer from one fiber to another. The thinner is the fiber, the smaller is the contact area among the fibers, and that increases material resistance in heat transfer process. Although the number of contacts among the fibers grows in case of ultra-thin fibers, the total sum of areas of contact points is considerably smaller than the contact area in the flax fiber structure. Thus, the best thermal technical characteristics should be displayed by insulating materials based on flax noils, which are characterized by 2–5 times smaller length and 5–6 times smaller size cross section in comparison with flax fibers that consist of firm bundles of elementary fibers.

V. ANALYSIS OF THERMAL INSULATION MATERIALS BASED ON FLAX NOILS AND FLAX FIBERS

After preliminary batching of the raw mixture, physical and mechanical characteristics of thermal insulation slabs were determined separately for fiber fills made from flax noils or flax fibers, and also from the mixture of flax noils and fibers. Table II presents quantitative formulas and average density of thermal insulation materials.



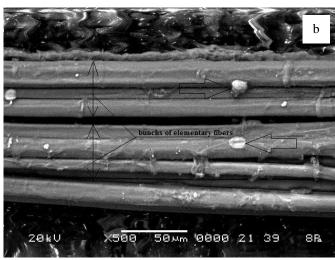


Fig. 5. Electron microscopy of a flax fiber: $a-200\ times\ enlargement,\ b-500\ times\ enlargement.$

TABLE II

COMPOSITION AND AVERAGE DENSITY OF THERMAL INSULATION SLABS

sition	Component consumptionper 1 m³, mass fractions			age ity, n³
Composition No.	flax fibers	flax noils	sodium silicate	Average density, kg/m³
1	0.800	_	0.200	50
2	0.846	-	0.154	65
3	0.875	_	0.125	80
4	0.895	-	0.105	95
5	0.910	-	0.090	110
6	0.640	0.160	0.200	50
7	0.677	0.169	0.154	65
8	0.700	0.175	0.125	80
9	0.716	0.179	0.105	95
10	0.728	0.182	0.090	110
11	-	0.800	0.200	50
12	_	0.846	0.154	65
13	-	0.875	0.125	80
14	-	0.895	0.105	95
15	-	0.091	0.090	110

Analysis of the obtained correlations (Fig. 6) allows concluding that the increase of the amount of fiberfill to ensure insulant density of 80 kg/m³ irrespective of the amount of the binder and fiber type leads to decrease of thermal conductivity coefficient, further increase of density causes the increase of thermal conductivity. For example, at the density of 110 kg/m³, thermal conductivity of the insulating material based on flax fibers (composition 5) is $0.047 \text{ W/(m}^{\circ}\text{C})$. Decrease of the average density by 27 % (composition 3) leads to decrease of thermal conductivity coefficient to 0.042 W/(m°C). Further reduction of the average density of the insulation material to 50 kg/m³ leads to the increase of thermal conductivity by 12 % up to 0.047 W/(m°C). At the density of 50 kg/m³, thermal conductivity of the material made from the mixture of flax noils and flax fibers (composition 6) is 0.045 W/(m°C). Reduction of thermal conductivity coefficient to 0.039 W/(m°C) occurs at 1.6 times increase of density of thermal insulation material. Further increase of the average density leads to the increase of the coefficient of thermal conductivity to 0.044 W/(m°C). For compositions 11-15 minimal value of the coefficient of thermal conductivity 0.036W/(m°C) corresponds to the density of the material from flax noils equaling 80 kg/m³. It was determined that at the decrease of density (composition 11) thermal conductivity coefficient grows by 14 % to 0.041 W/(m°C). Increase of the analyzed parameter to 0.04 W/(m°C) occurs at the increase of the average density (composition 15). It was also determined that at equal densities, the lowest coefficient of thermal conductivity is displayed by composition 13based on flax noil fiberfill. For example, if fiber noils are used as a fiberfill at the density of material of 80 kg/m³ (composition 13), thermal conductivity equals $0.036~\rm W/(m^{\circ}\rm C)$. In case 80% of flax noils are substituted by flax fibers (composition 8), the coefficient of thermal conductivity increases to $0.039~\rm W/(m^{\circ}\rm C)$, at full substitution of flax noils by flax fibers (composition3), the analyzed parameter increases by 17 % and equals $0.042~\rm W/(m^{\circ}\rm C)$.

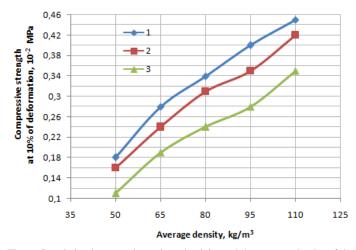


Fig. 6. Correlation between thermal conductivity and the average density of the insulation material: 1 – for compositions 1–5; 2 – for compositions 6–10; 3 – for compositions 11–15.

Based on research results, the best thermal conductivity indicators are displayed by flax noil-based materials (Fig. 7), which is determined by shorter length and smaller cross section of the flax oil compared to the flax fiber. The inner channel from 4 μm to 6 μm in diameter present in the elementary fibers is an additional factor that has a positive impact on the reduction of thermal conductivity. It reduces conductive heat transfer along the fiber body.



Fig. 7. Insulation materials based on flax noils.

Reduction of thermal conductivity is also promoted by spatial multi directionality of elementary fibers in the insulation material structure. Such distribution of fibers hinders convective air transfer due to reduction of the size of thin air spaces of irregular shape and their partial localization in the form of separate closed micro-voids.

The obtained correlations (Fig. 8) attest that the increase of density of thermal insulation materials irrespective of the type of fibers at the constant consumption of the binder leads to the increase of the compressive strength at 10 % deformation. Thus, increase of density of the insulation material based on flax noils leads to the increase of compressive strength from $0.11 \cdot 10^{-2} \, \text{MPa}$ to $0.35 \cdot 10^{-2}$ MPa for composition 15in comparison with composition 11. At the density of 50 kg/m³ of the material made from the mixture of flax fibers and flax noils (composition 6), compressive strength at 10 % deformation is $0.16 \cdot 10^{-2}$ MPa. Further increase of density leads to increase of compressive strength by 162.5 % (composition 10). At the maximal density of 110 kg/m³ (composition 5), compressive strength equals $0.45 \cdot 10^{-2}$ MPa, that is, it increases 2.5 times in comparison with the rate of composition 1. It should be stated that thermal insulation materials based on flax fibers display the highest degree of durability. For example, at the density of 95 kg/m³, the insulating material based on flax fibers (composition 4) has compressive strength of $0.4 \cdot 10^{-2}$ MPa. Substitution of 20 % of flax fibers with flax noils (composition 9) leads to the decrease of the analyzed parameter by $0.5 \cdot 10^{-3} \, \text{MPa}$. If flax noils are used instead of flax fibers, durability of the insulation material at the density of 95 kg/m³ (composition 14) decreases to 0.28 · 10⁻² MPa in comparison with composition 4.

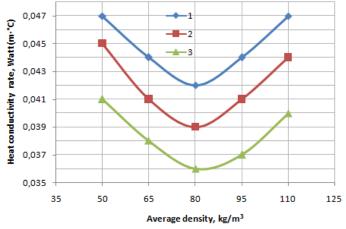


Fig. 8. Correlation between compressive strength at 10% deformation and the average density of the insulation materials: 1 – for compositions 1–5; 2 – for compositions 6–10; 3 – for compositions 11–15.

Decrease of compressive strength of the samples made from noils by 22–38 % in comparison with the insulation materials made from flax fiber, considering low values of the given parameter, cannot be the determining factor in selecting the most efficient fiberfill for the thermal insulation material. Thus, the insulation material based on flax noils is preferred as the most efficient thermal insulation material regarding its thermal insulation characteristics. In further research, it is planned to optimize flax noil compositions, which will allow improving physical and mechanical characteristics of the insulation material.

VI. CONCLUSIONS

Among the considered thermal insulation materials based on plant fibers, the insulation materials based on flax noils or nettle fibers display the best thermal conductivity indicators. However, absence of technologies of cultivation and obtaining of nettle fibers on the industrial scale in the territory of Belarus is a significant drawback that impedes using this raw material in production of insulation materials. At the same time, manufacturing of thermal insulation materials based on nettle fibers can be implemented in China, where nettle is cultivated to obtain fiber for fabric manufacturing.

The conducted light microscopy tests allowed determining that the microstructure of the flax noil is made from a conglomeration of chaotically interconnected elementary fibers, which conditions formation of the mesh fibrous frame of the flax noil. In the process of contact, flax noils form a spatial micro-mesh fiber system.

It has been determined that elementary fiber is a micro-tube from 8 μ m to 12 μ m in diameter, with void channel from 4 μ m to 6 μ m in diameter, which is compatible with the size of solid fibers of rock wool, ensuring formation of efficient isolating structure.

The obtained insulation materials made from flax noils have thermal conductivity of 0.036–0.041 W/(m°C) and compressive strength at 10 % deformation from $0.11\cdot 10^{-2}$ MPa to $0.33\cdot 10^{-2}\,\text{MPa}$ at the density from 50 kg/m³ to 110 kg/m³.

The factors that have a significant impact on the thermal conductivity rate of fiberfill have been determined: presence of the fibers less than 20 μm in diameter; presence of void channels in the fibers; chaotic directionality of fibers in space that ensures formation of mesh structural framework; reduction of the total fiber contact area; reduction of the size and localization of the micro-voids in the structure of thermal insulation material.

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