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SUNFLOWER STALKS AND LIGNOSULFONATE – ALTERNATIVE RAW MATERIALS FOR THE PRODUCTION OF ECO-FRIENDLY MEDIUM DENSITY FIBREBOARDS

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ABSTRACT

The main advantages of the technology for producing Medium Density Fibreboards (MDF) are the reduced requirements for wood raw material, the possibility of fabricating that type of product from sawmill residues and the potential of including other non-wood lignocellulosic raw materials in the composition of the panels.

Such lignocellulose raw materials are residues from agriculture. In our country and worldwide, there are many successful studies for at least partial replacement of wood raw material with agricultural residues. Sunflower plantations are the second largest in our country, which is why they would be a significant source of non-wood lignocellulosic raw material.

Another significant problem in MDF production is the presence of formaldehyde emissions from the panels. One of the main directions for solving this problem is replacing the traditional binders with alternative ones without formaldehyde emissions. These alternative binders should not significantly increase the cost of board production, maintain their performance, and, most importantly, not be hazardous to human health. This binder includes the so-called bio-based binders, of which lignin-based products are the most promising. A typical representative of such an alternative bio-based binder is lignosulfonate (methylated lignin).

That leads to the relevance of a study on the possibility of partially replacing wood raw material with sunflower stalks and reducing the number of synthetic binders used in MDF by adding lignosulfonate in the adhesive system.

As a result of the research on the influence of urea-formaldehyde resin content, lignosulfonate content, and pulp from sunflower stalks on the properties of thin MDF, the main conclusions were deduced, and recommendations were made on the practical use of these alternative raw materials.

Key words: eco-friendly MDF, sunflower stalks, lignosulfonate, urea-formaldehyde resin.

INTRODUCTION

Waste lignocellulosic raw materials can partially replace wood raw materials in producing fibreboard panels. It should be emphasized that the known research so far is focused mainly on wet-processed fibreboards. Research related to dry-processed fibreboards in Bulgaria is significantly more limited in quantity and scope of the studied factors. According to forecasts, in 2022, it is expected that 9.2 million decares of land with sunflowers will be sown, compared to 8.0–8.5 million decares in recent years. The interim operational data of the Ministry of Agriculture show more than four times the growth of the sown areas with sunflower in Bulgaria as of April 14, 2022, compared to the same period last year. (https://www.dnes.bg/) Therefore, residual products such as sunflower stalks are a significant source of lignocellulosic raw material in Bulgaria. Manufacturers of wood-based panels, particularly MDF, are constantly striving to reduce formaldehyde emissions from the panels. This reduction is currently being worked on worldwide in four main directions: 1) lowering the levels of free formaldehyde in the resins while preserving their adhesive properties as much as possible; 2) addition of acceptors to reduce the emission of formaldehyde from the plates; 3) surface treatment of the plates after hot pressing; 4) use of alternative binders that do not significantly increase the cost of board production, while maintaining their performance (Carvalho, L. et al. 2012).

Several known shortcomings are observed in all known studies. Enzyme lignin is difficult to obtain, and its price is high, so its use as a binder will significantly increase the cost of MDF production. This type of lignin is not water-soluble and is applied in the panels as a powder substance, which leads to significant difficulties from a technological point of view. Because of that, to activate the enzymatic lignins, it is necessary to increase the hot-pressing temperature and use extended press factors. These disadvantages can be partly overcome by using lignosulfonates as a binder. Lignosulfonates have the advantage over hydrolysis or enzymatic hydrolysis lignin that they are water-soluble and can be introduced in the form of solutions into the pulp. They are a waste product from cellulose production by the sulphite method. Currently, lignosulfonates are mainly used in the woodworking industry as a binder, giving additional mechanical stability in the production of pellets. In the production of lignosulfonate-based MDF, hot-pressing temperature plays a significant role. At temperatures below 190°C, the plates are dispersed, i.e. the connections built in them are not stable (Mihajlova, J. et al., 2017). No studies have been found at temperatures higher than 200° C. The problem with the concentration of lignosulfonates in the wood fibre mass is not fully clarified. On the one hand, this concentration should be such as to obtain an even distribution of the lignosulfonate in the volume of the compressible material and to form hydrogen bonds and their binding to the phenoxyl radicals of the lignosulfonate. On the other hand, as the concentration of the solutions decreases, the water content in the compressible material increases, which leads to a precondition for combustion of the panels and destruction of their structure as a result of the separation of the vapour-gas mixtures.

In the Department of Mechanical Wood Technology at the University of Forestry, satisfactory results were achieved for the physical and mechanical properties of MDF at 15% lignosulfonate content with a concentration of 30%, hot-pressing temperature 220ºC and press factor 1.5 $min.mm^{-1}$. These fibreboard panels were with formaldehyde emissions at wood levels. Still, the problems are the extended press factor and higher temperature of hot-pressing, as well as the significantly darker colour of the boards (Mihaylova, J et al., 2017). The present study aims to fabricate eco-friendly MDF panels with the participation of pulp from sunflower stems and lignosulfonate. This study also examined the effect of urea-formaldehyde (UF) resin, lignosulfonate (LS) and sunflower pulp on the properties of the panels.

MATERIALS AND METHODS

In laboratory conditions, three types of MDF were fabricated – with 10% UF resin, 5% UF resin and 5% lignosulfonate, and 10% lignosulfonate. All panels had a set thickness of 8 mm and a set density of 850 $kg.m^{-3}$. For each of the three types of plates, the mass content of sunflower stalks varies as follows: 0%, 10%, 20% and 30%. The experimental plan is presented in Table 1.

For the experiments, was used industrial pulp from the following wood species: beech – 57%, oak 35%, poplar – 8%. The pulp was dried to a moisture of 11%. Its bulk density was 29 kg.m^{-3}. The pulp from sunflower stalks was produced with laboratory disk refiners. To remove mineral inclusions and ash from the sunflower stems, they were soaked in water for 72 hours. The sunflower stalks were pre-cut. The slices thus prepared were boiled for 20 minutes and refined in a laboratory disk mil with additional circulating water. The resulting pulp was further processed in a second-stage laboratory refiner with continuous washing. Figures 1 and 2 show sunflower stalks pulp after primary and secondary refining.

Table 1: Experimental plan

№	MDF Density, kg.m^{-3}	Content of UF resin, %	Content of pulp from sunflower stalks, %	Content of lignosulfonate, %
1.	850	10	0	0
2.	850	10	10	0
3.	850	10	20	0
4.	850	10	30	0
5.	850	5	0	5
6.	850	5	10	5
7.	850	5	20	5
8.	850	5	30	5
9.	850	0	0	10
10.	850	0	10	10
11.	850	0	20	10
12.	850	0	30	10

Figure 1: Pulp from sunflower stalks, after first stage refining

Figure 2: Pulp from sunflower stalks, after second stage refining

The moisture content, after drying, of sunflower pulp was 11%, and the fractional composition of the pulp is presented in Table 2.

Table 2: Fractional composition of pulp from sunflower stalks

1st fraction	1÷ 2 mm	76.00%
2nd fraction	800 μm ÷1 mm	0.20%
3rd fraction	500 ÷ 800 μm	3.24%
4th fraction	315 ÷ 500 μm	6.81%
5th fraction	200 ÷ 315 μm	5.34%
6th fraction	100 ÷ 200 μm	4.80%
dust	-	3.61%

The calcium lignosulfonate used has the following characteristics: calcium – up to 6%; reduced sugars – 7%; ash content – 14%; dry content – 93%; acid factor in 10% solution – pH = 4.3 ± 0.8; bulk density – 550 kg.m^{-3}. The compositions of the panels are presented in Table 3.

Table 3: Compositions of the laboratory panels

№	MDF weight, kg	Wood fibres, kg	Fibres from sunflower stalks, kg	UF resin, kg	Hardener, kg	Lignosulfonate, kg	Wax emulsion, kg
1	1.088	1.328	0	0.2176	0.02176	0	0.018
2	1.088	1.195	0.133	0.2176	0.02176	0	0.018
3	1.088	1.062	0.266	0.2176	0.02176	0	0.018
4	1.088	0.930	0.398	0.2176	0.02176	0	0.018
5	1.088	1.328	0	0.1088	0.01088	0.1511	0.018
6	1.088	1.196	0.132	0.1088	0.01088	0.1511	0.018
7	1.088	1.062	0.265	0.1088	0.01088	0.1511	0.018
8	1.088	0.930	0.398	0.1088	0.01088	0.1511	0.018
9	1.088	1.328	0	0	0	0.302	0.018
10	1.088	1.195	0.132	0	0	0.302	0.018
11	1.088	1.063	0.265	0	0	0.302	0.018
12	1.088	0.930	0.398	0	0	0.302	0.018

The gluing of the pulp for the laboratory panels was performed in a high-speed laboratory blender with needle-shaped blades. A nozzle with a diameter of 1.5 mm was used to spray binders. For the hot-pressing was used a laboratory press PMC ST 100 Italy. The hot-pressing cycle was as follows: 1st stage (20% of the whole cycle) at a pressure of 3.0 MPa; 2nd stage (30% of the whole cycle) at a pressure of 1.2 MPa and 3rd stage (50% of the whole cycle) at a pressure of 0.6 MPa; the temperature hot-pressing was190°C, and the press factor was 30 s.mm^{-1}.

The physical and mechanical properties of the panels were determined according to the EN standards (EN 310:1999, EN 317:1999, EN 319:2002, EN 323:2001, EN 326-1). For each property were used 8 test samples and main statistics were determined.

RESULTS AND DISCUSSION

The summarized results for the physical and mechanical properties of MDF with the participation of UF resin from 0 to 10%, the participation of lignosulfonate from 0 to 10% and the participation of pulp from sunflower stalks from 0 to 30% are presented in Table 4.

The determined values for density, water absorption, thickness swelling, MOR, MOE and IB strength were processed by variation statistics. The results show that the values for the studied properties are statistically significant, the probability for all properties was below 5%, and the results can be used to analyze the effect of the studied factors. The densities of the MDF varied from 842 to 865 kg.m^{-3}, i.e. the difference in this primary classification and characteristic property is only 2.7%, which means that this difference would not affect the other physical and mechanical properties of the panels. That gives grounds to analyze the effect of the studied factors. The change in the water absorption of the panels is presented in Table. 4 and Figure. 3. Under the experiment's conditions, the laboratory's water absorption panels vary from 51 to 77%. From the data presented in Table. 4 and Figure 3, it becomes clear that deterioration in water absorption is observed in all types of MDF with increasing content of sunflower stem pulp. In the case of panels bonded only with UF resin, type A, the water absorption varies from 58.6 to 74.8%, i.e. with increasing content of sunflower stem pulp, there is a deterioration in the property by 16.2%. The decline is also observed with the inclusion of 10% sunflower stems, while the difference between the water absorption of the panels with 20 and 30% content of sunflower stems is relatively small.

Table 4: Physical and mechanical properties of MDF with the participation of pulp from sunflower stalks and lignosulfonate

№	UF resin, %	Pulp from sunflower stalks, %	Lignosulfonate, %	Density *ρ*, kg.m^{-3}	Water absorption *A*, %	Thickness swelling (24 h) *Gt*, %	Bending strength (MOR) *fm*, N.mm^{-2}	Modulus of elasticity (MOE) *Em*, N.mm^{-2}	Internal bond (IB) strength *ft*, N.mm^{-2}
1	10	0	0	854	58.58	15.28	33.38	4414	0.580
2	10	10	0	858	66.61	15.43	32.50	4205	0.539
3	10	20	0	859	71.01	19.33	33.40	4253	0.543
4	10	30	0	846	74.74	19.01	28.90	4054	0.520
5	5	0	5	862	51.38	13.77	37.04	4922	0.715
6	5	10	5	842	54.40	15.14	32.90	4582	0.702
7	5	20	5	856	56.91	18.55	33.38	4339	0.692
8	5	30	5	865	66.61	18.99	26.64	4237	0.674
9	0	0	10	853	57.07	15.16	34.25	4803	0.653
10	0	10	10	855	61.07	17.69	34.18	4650	0.593
11	0	20	10	846	64.70	20.41	30.39	4276	0.518
12	0	30	10	857	77.34	22.31	26.14	3806	0.507

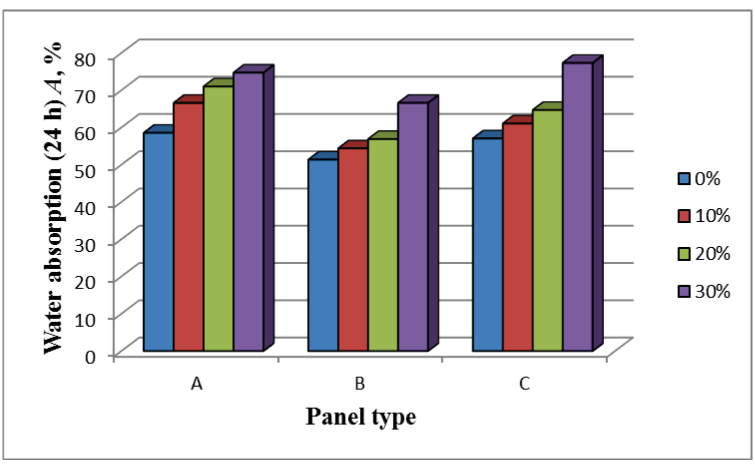

Figure 3: Variation in water absorption of MDF at different contents of pulp from sunflower stems (0, 10, 20 and 30%) depending on the content of UF resin and lignosulfonate: Panel type A – 10% UF resin; Panel type B – 5% UF resin and 5% lignosulfonate; Panel type C–10% lignosulfonate

The picture is similar for panels type B, which has a 5% content of UF resin and a 5% content of lignosulfonate. The variation in water absorption is from 51.3 to 66.6%. That is, there is a deterioration in the property by 15.3%. Here, however, the significant decline is when adding a 10% pulp from sunflower stalks and increasing the content of sunflower pulp from 20 to 30%. In the last type of panels, fabricated entirely with lignosulfonate – panel type C, the water absorption varies from 57 to 77% or deterioration of 20% is observed. That is, the most unsatisfactory results for this property are observed here. As with the previous type of panels, an increase in the values of the property is observed with the addition of a 10% pulp from sunflower stems. The following significant deterioration is when the content of non-wood lignocellulosic raw material increases from 20 to 30%. The best values for water absorption are at panels fabricated with 5% UF resin and 5% of lignosulfonate (type B, №6), Fig.3. The chosen hot-pressing regime can explain that. With only UF resin content at the hot pressing temperature, initial processes of binder degradation can be observed. When only lignosulfonate is used as a binder, the press factor, due to the relatively low lignosulfonate application concentration of 30%, is insufficient to separate the vapour-gas mixtures at safety speed.

The variation in the thickness swelling of MDF at different contents of sunflower stems, UF resin and lignosulfonate is presented in Fig.4.

As can be seen from the presented data, the thickness swelling of the laboratory panels varies from 22.31% to 13.77%, i.e. with 8.54%.

In MDF fabricated with 10% UF resin content, i.e. type A, without and with a 10% mass content of sunflower stalks meet the requirements of EN 622-5: 2010 for fibreboards of general-purpose and use in dry conditions, especially interior furnishing, including furniture. Those panels also meet the standard's load-bearing structures for use in dry conditions (17% thickness swelling). In that type of MDF, there is a property deterioration by 3.73% with increased sunflower stem content.

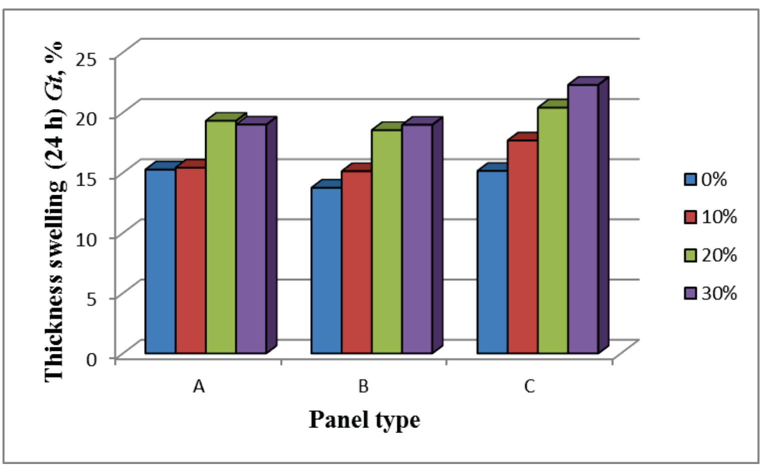

Figure 4: Variation in thickness swelling of MDF at different contents of pulp from sunflower stems (0, 10, 20 and 30%) depending on the content of UF resin and lignosulfonate: Panel type A – 10% UF resin; Panel type B – 5% UF resin and 5% lignosulfonate; Panel type C – 10% lignosulfonate.

For panels with 5% content of UF resin and 5% content of lignosulfonate without the participation of sunflower stems (type B – №5), the best (lowest) value of the property is – 13.77% (Table 3 and Fig.4). MDF with 5% content of UF resin, 5% content of lignosulfonate and 10% content of sunflower stems – type B – №6 also has satisfactory thickness swelling. These two MDF types also meet the requirements of EN 622-5: 2010 for general purpose and use in dry conditions, especially interior furnishing, including furniture and meet the requirements of the standard for load-bearing structures for use in dry conditions (thickness swelling below 17%). Thickness swelling worsens with increasing mass content of sunflower stems, as the highest value for swelling in thickness is reported for panels with 30% content of sunflower stems – 18.99%. From the MDF, which does not contain UF resin but lignosulfonate 10% – type C, only MDF without the participation of sunflower stalks meet the requirements for thickness swelling for general purpose panels and use in dry conditions, including furniture and the requirements of the standard for load-bearing structures for use in dry conditions (17% thickness swelling). As the content of sunflower stalks increases to 30%, the thickness swelling of the MDF increases and reaches 22.31%.

Figure 5. shows the variation of the bending strength of the MDF at different content of sunflower stalks, UF resin and lignosulfonate.

The requirement of EN 622-5:2010 for general-purpose MDF for use in dry conditions, especially interior fittings, including furniture in bending strength, is 23 $N.mm^{-2}$ and the standard for panels for load-bearing structures for use in dry conditions – respectively 29 $N.mm^{-2}$. From the data presented in Table 3 and Fig. 5, it is seen that, in general, the obtained laboratory plates have shown high values concerning this property as all types of panels meet the requirements for general-purpose boards for use in dry environments, especially interior furniture, including furniture. However, the decreasing trend is clear of the property with increasing content of sunflower stalks in the composition of the plates. As for the requirements of the standard for slabs for load-bearing structures for use in dry conditions to the bending strength, they are met in all MDF in which the share of sunflower stems is at most 20%, i.e. panels with a mass of 30% sunflower stalks do not meet the requirements for bending strength for load-bearing structures for use in dry conditions.

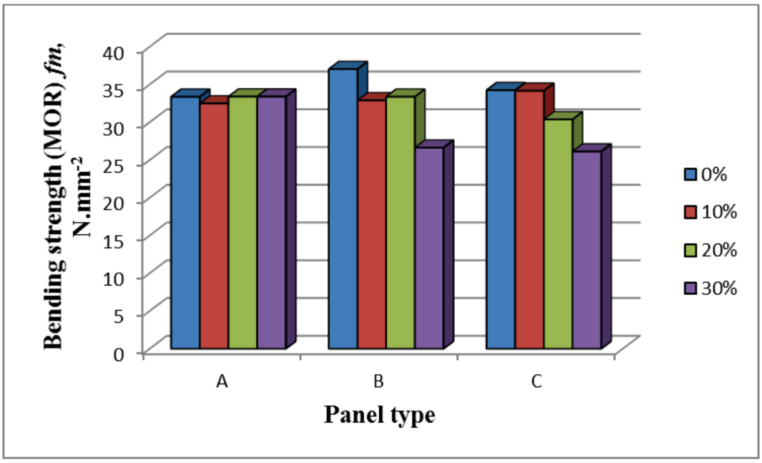

Figure 5: Variation in bending strength (MOR) of MDF at different contents of pulp from sunflower stems (0, 10, 20 and 30%) depending on the content of UF resin and lignosulfonate: Panel type A – 10% UF resin; Panel type B –5% UF resin and 5% lig nosulfonate; Panel type C – 10% lignosulfonate.

The variation in the modulus of elasticity (MOE) of the panels at different contents of sunflower stems, UF resin and lignosulfonate is presented graphically in Fig. 6.

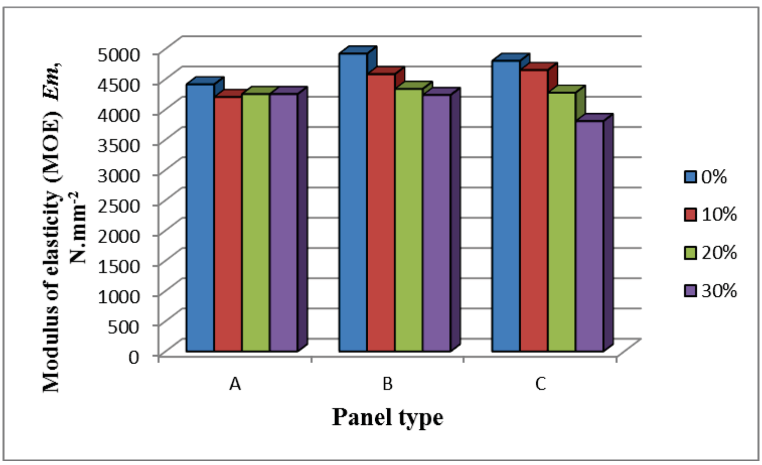

Figure 6: Variation in modulus of elasticity (MOE) of MDF at different contents of pulp from sunflower stems (0, 10, 20 and 30%) depending on the content of UF resin and lignosulfonate: Panel type A – 10% UF resin; Panel type B – 5% UF resin and 5% lignosulfonate; Panel type C – 10% lignosulfonate.

Given that the requirement of EN 622-5: 2010 for general purpose boards for use in dry environments, especially interior furniture, including furniture in terms of modulus of elasticity, is 2700 N.mm^{-2}, and the requirement of the standard to panels for load-bearing structures for use in dry environments, respectively 3000 N.mm-2, as well as the data presented in Table 3., and in Fig. 6 it is clear that all types of laboratory plates meet the requirements and have even shown significantly higher values ranging from 3806 to 4922 N.mm^{-2}. However, with this property, there is also a clear tendency to decrease the values with increasing content of sunflower stems in the composition of the MDF.

Figure 7 presents the variation in internal bond (IB) strength at different sunflower stalks, UF resin and lignosulfonate content.

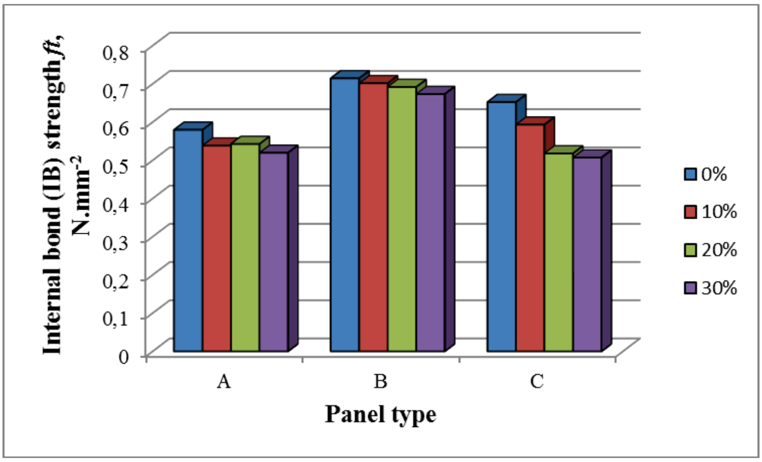

Figure 7: Variation in internal bond (IB) strength of MDF at different contents of pulp from sunflower stems (0, 10, 20 and 30%) depending on the content of UF resin and lignosulfonate: Panel type A – 10% UF resin; Panel type B – 5% UF resin and 5% lignosulfonate; Panel type C – 10% lignosulfonate.

For the test conditions, the IB strength varies from 0.5 to 0.7 N.mm^{-2}. The requirement of EN 622-5: 2010 for general purpose panels for use in dry conditions, especially interior furnishings, including furniture, in terms of IB strength is 0.65 N.mm^{-2}, and the requirement of the standard for MDF for load-bearing structures for use in dry conditions, respectively 0.70 N.mm^{-2}. From the data presented in Table 3 and Fig. 7, it is seen that in the case of panels type A, i.e. with 10% UF resin and 0% lignosulfonate, no type of MDF meets the requirements of the standard. From panels type B – with 5% UF resin and 5% lignosulfonate, all types of panels meet the requirements for general-purpose tiles for use in dry conditions, especially interior furniture, including furniture, and tiles with 0% and 10% sunflower stems (type B – №5 and №6), also meet the requirements for slabs for load-bearing structures for use in dry environments. With an increasing percentage of sunflower stems, the values of the property decrease – from 0.715 to 0.674 N.mm^{-2}. In the case of panels type C – with 10% lignosulfonate, only the MDF without the participation of a mass of sunflower stems meet the requirements for general purpose and use in dry conditions, especially interior furnishing, including furniture. The IB strength decreases with the increasing content of sunflower stems from 0 to 30%, respectively, from 0.653 to 0.507 N.mm^{-2}.

After summarizing the results, it is clear that panels type B – №5 and №6 fully meet the requirements of EN 622-5: 2010 for general purpose and use in dry conditions, especially interior furnishing, including furniture, as well as requirements for r load-bearing structures for use in dry conditions.

CONCLUSIONS

The production of eco-friendly MDF with the participation of natural binders and, in particular, lignin products is a promising direction for developing technology in this production.

On the other hand, in Bulgaria and worldwide, there is a significant potential of residual lignocellulosic agricultural raw materials, particularly sunflower plantations, which can replace part of the scarce wood raw material.

In the implementation of the present study, some serious difficulties emerged, namely:

- The pulp obtained from sunflower stalks has a relatively short length of fibres. Because of that, when it is used in the composition of MDF, its amount should not exceed 10%;
- When using lignosulfonates as binders, difficulties are observed in the application of shortened hot-pressing cycles;
- To reduce the press factor and the content of synthetic binders, and hence the formaldehyde emissions, the use of a combination of resins and lignosulfonate is recommended;
- The introduction of lignosulfonates can be done in a dry state, which can be easily implemented in industrial conditions – at the beginning of pulp dryers, immediately after the introduction of urea-formaldehyde resin.

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