Hemp Flour as a Formaldehyde Scavenger for Melamine-Urea-Formaldehyde Adhesive in Plywood Production

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The possibility of using hemp flour as a filling material for melamine-urea-formaldehyde (MUF) adhesive was investigated for the process of manufacturing plywood. Adhesive mixtures were filled with various amounts of hemp flour and compared to rye flour as a reference mixture, and their properties were tested. The quantity of added filler had a significant effect on the resins viscosity. Moreover, the replacement of rye flour with the hemp flour resulted in acceleration of gel time and a slight increase in solid content. Due to the high proteins content, hemp flour led to a significant reduction of formaldehyde emission. Bonding quality of plywood made with hemp flour as a filling material for the adhesive mixture meet the requirements from the standard. Introducing it in an amount of 20 pbw and 25 pbw (parts by weight) allowed for the production of plywood characterized by equally good mechanical properties as that of reference plywood glued with an adhesive mixed with rye flour. In summary, the proper amounts of hemp flour can be applied as a filling material and formaldehyde scavenger for MUF adhesive without the deterioration of mechanical properties of manufactured plywood.

Keywords: Hemp flour; Melamine-urea-formaldehyde adhesive; Plywood; Filler

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INTRODUCTION

The widespread use of plywood, particularly as construction material, and its applications in many fields results from its optimal properties, such as its durability, transverse tensile properties, and the resistance to deformation. These unique mechanical properties result from its layer construction. In addition, plywood is also highly regarded for its high aesthetic values and creates opportunities for use as a finishing decorative material (Jóźwiak and Fojutowski 2018; Cao *et al.* 2019; Kawalerczyk *et al.* 2019a). Along with other wood-based panels such as particleboards and fiberboards, plywood panels are consistently gaining popularity. Their production on a global scale is still growing, requiring the increasing amounts of both wood and wood adhesives (Adhikari *et al.* 2016). Formaldehyde-based adhesives such as urea-formaldehyde (UF), phenol-formaldehyde (PF), and melamine-urea-formaldehyde (MUF) are still dominant in plywood production (Ong *et al.* 2018). The advantages of the amino resins are lack of color, high bonding strength, and relatively low cost (Dukarska 2013; Réh *et al.* 2019). MUF adhesives are increasingly replacing UF adhesives in plywood production due to a lower formaldehyde content and higher degree of crosslinking resulting from the ring structure of the melamine (Gao *et al.* 2012; Luo *et al.* 2015b). However, despite the fact that the participation of amino resins in the general range of binders produced for wood industry stands at 85%, both UF and MUF adhesives are still susceptible to long-term presence of water and the action of variable ambient conditions (Dziurka and Mirski 2010; Jóźwiak 2018).

The properties of wood-based materials are mainly determined by two factors, *i.e.*, the quality of wood used for the production and the nature of the adhesives (Kawalerczyk *et al.* 2019b). Their modification is a common way to improve the properties of wood-based panels by chemical modification of adhesives or the addition of various types of fillers (Pawlak and Boruszewski 2018). Fillers are defined as inorganic or lignocellulosic particles that are non-gluing, insoluble in the adhesives, and non-volatile. The purpose of modification is usually the improvement in mechanical behavior of the materials and the associated opportunity to reduce the amount of applied adhesive (Dziurka and Mirski 2014). Furthermore, changing the composition of adhesive mixture can also lead to increased water resistance, reactivity, and the reduction of formaldehyde content, which is very important especially in the case of amino resins. Although MUF resins have a lower amount of free formaldehyde compared to UF resins, they still generate harmful formaldehyde emissions, which should be reduced (Luo *et al.* 2015a; Demir *et al.* 2018). Formaldehyde (HCHO) is the simplest naturally occurring aldehyde, and it is categorized more distinctly than most other pollutants because of its potential carcinogenic effect on human health (Salem *et al.* 2013). Moreover, due to its toxicity, it may cause eye irritation, respiratory difficulties, sleepiness, and inability to concentrate (Colak *et al.* 2002; Milota 2000). The widespread use of products containing formaldehyde such as glues, insulating materials, wood-based materials and fabrics – a major anthropogenic source of this chemical – can seriously affect human indoor environment (Uchiyama *et al.* 2007).

Numerous investigations on the possible reduction of formaldehyde emission have been conducted. These studies include topics on changes in pressing parameters and modification of the adhesives composition by the addition of fillers. However, based on many studies, it can be concluded that often a decrease in the free formaldehyde content is accompanied by a decrease in strength properties of glue joints. The example of widely evaluated filler for plywood production is bark. In addition to the advantages consisting of the recycling of woodworking industry residues, it has also been shown that chestnut, fir, and beech bark powders can be used as formaldehyde scavengers without compromising mechanical properties of manufactured plywood. Moreover, the replacement of flour with bark led to a decrease in heat transfer during the pressing process (Aydin *et al.* 2017; Ružiak *et al.* 2017). Another example of the promising filler is a clay mineral named sepiolite. Studies have shown that the partial replacement of flour with sepiolite caused a decrease in the amount of emitting formaldehyde and the improvement in bonding quality of plywood (Li *et al.* 2015). In recent years, there have been many studies on an interesting concept of using fillers within the dimensional range of nanotechnology. For example, introducing nano-SiO_2 instead of flour to MUPF resin increased its reactivity and significantly reduced free formaldehyde content. Furthermore, the addition of nanosilica led to a decrease of the spread rate of applied resin (Dukarska and Bartkowiak 2016a; Dukarska and Czarnecki 2016b). Another study conducted by Zhang *et al.* (2011) investigated the effect of using cellulosic nanoparticles as a filler for UF resin. The addition of nanocellulose resulted in the significant improvement in shear strength and led to a decrease of formaldehyde emission. The replacement of rye flour with cellulose can also considerably reduce the VOCs emitted from the wood-based material (Ayrilmis *et al.* 2016).

In recent years, there has been an increasing social awareness of the need for the sustainable use of natural resources such as hemp (Mirski *et al.* 2018). The plant is characterized by many advantages. Hemp very easily adapts to various climate conditions and achieves maturity within 100 days (Mirski *et al.* 2017). It is a commonly used material in fabrics production and pharmacy. For years wheat and rye flours were the most popular flours in the food industry because of their high availability (Pourafshar *et al.* 2015). In recent years, alternative flours have gained great popularity because of many problems such as allergies, *etc*. Because of that, various kinds of flours including hemp flour have become more commercially available and financially reasonable. Thus, the aim of the study was to investigate the possibility of using hemp flour as a filler for MUF resin in plywood production.

EXPERIMENTAL

Materials

An industrial MUF resin was purchased from AkzoNobel (Września, Poland) with the following characteristics: viscosity of 1000 to 2500 mPa s, pH 9.5 to 10.7, solids content of 64 to 69%, density 1.27 g/m^3, and gel time at 100 °C of 63 s. Ammonium nitrate (20 wt%) was used as a hardener. Plywood was produced using rotary cut birch veneer sheets with dimensions of 320 × 320 mm, average thickness of 1.5 mm, and moisture content of 6% ± 1%. Two kinds of commercially available fillers were used in this study. Their chemical compositions were provided by the suppliers and they are summarized in Table 1.

Table 1. The Share of Main Components in Rye and Hemp Flour

Type of flour	Quantity (pbw* per 100 pbw of flour)			
	Fat	Carbohydrates	Proteins	Dietary fibers
Rye flour	2.8	51.2	5.9	11.4
Hemp flour	10	32	37	15

*Note: pbw means parts by weight

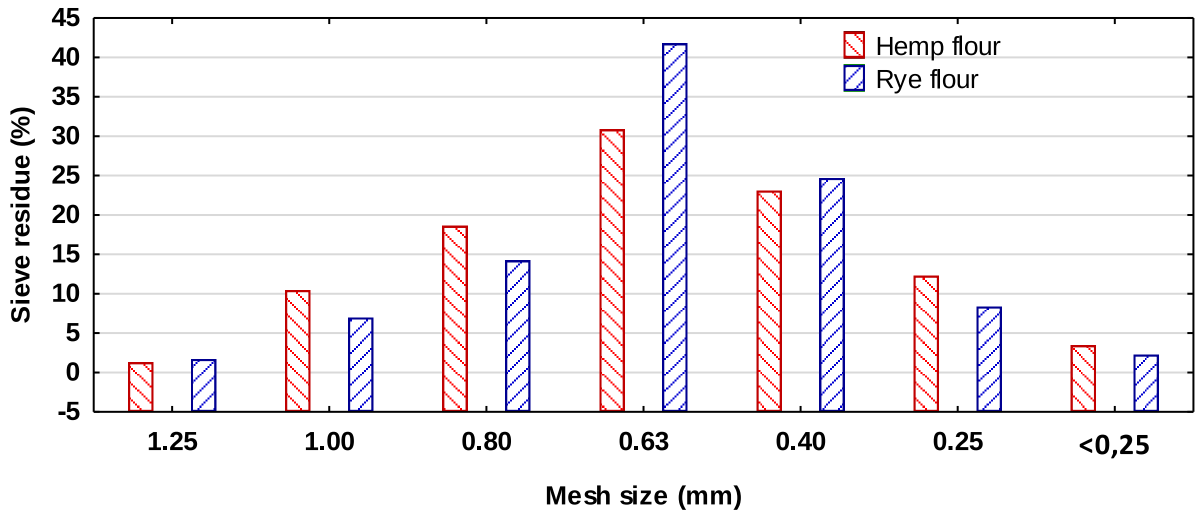


Fig. 1. Fractional composition of flours

In addition to the chemical composition, which may determine their hydrophilicity and reactivity, the size of fillers particle is also a very important factor. In order to evaluate the average dimensions of filler particles, flours were subjected to the sieve analysis using a mechanical sieve shaker (Multiserw-Morek, Marcyporeba, Poland) (Fig. 1).

On the basis of the fractional analysis it was found that both hemp and rye flour were similarly characterized by the significantly greater proportion of smaller fractions. A major share of small particles makes both flour suitable for use as a filler and allows a high level of homogenization of adhesive mixture. Moreover, a greater share of small dimensional fraction leads to an even distribution of the adhesive on the surface of the veneer. The dimensions of filling particles can determine mechanical properties, free formaldehyde content, and other key properties of manufactured wood-based materials (Ayrilmis *et al.* 2016).

Methods

Both rye and hemp flours were added directly to the adhesive in the amount depending on the variant (Table 2).

Table 2. Composition of Adhesive Mixtures

Variant Label	Type of Filler	Quantity (pbw per 100 pbw of solid resin)		
		Flour	H_2O	Hardener
REF	Rye flour	15	15	2
H15	Hemp flour	15	15	2
H20	Hemp flour	20	15	2
H25	Hemp flour	25	15	2
H30	Hemp flour	30	15	2

After the addition of all components, mixtures were subjected to the mixing process using a CAT-500 homogenizer (Ingenieurburo CAT, M. Zipperer GmbH, Ballrechten-Dottingen, Germany) at 1000 rpm for 3 min in order to achieve a proper level of homogenization. To determine the effect of hemp filler introduction on the properties of resin, the following tests were carried out: gel time at 100 °C according to Polish standard PN-C-89352-3 (1996), pH level according to EN 1242 (2011), solid content according to EN 827 (2005), and viscosity with its changes for 6 h using Brookfield DV-II + Pro viscometer (Middleboro, MA, USA). In order to manufacture three-layered plywood, the adhesives were applied to the surface of the veneer in the spread rate of 170 g/m^2. The pressing process was conducted at 140 °C for 4 min with the unit pressure of 1.3 MPa. Manufactured plywood samples were tested in terms of such properties as the following: free formaldehyde content using flask method according to EN 717-3 (1996); shear strength after 24 h of soaking in water and after ageing test including boiling in water for 6 h and cooling in water for 1 h according to EN 314-1 (2004); modulus of elasticity and bending strength according to EN 310 (1993). Results of investigations were subjected to the statistical analysis using Tukey test on the significance level of $\alpha = 0.05$ using Statistica 13.0 software (StatSoft Inc., Tulsa, OK, USA)

RESULTS AND DISCUSSION

Properties of the Adhesives

After the homogenization of adhesive, common industrial indicators such as pH, solid content, and gel time were used for quality control (Gonçalves *et al.* 2019). On the basis of values presented in Table 3, it was found that the replacement of rye flour with hemp flour had a beneficial effect on the properties of adhesive mixtures.

Substituting commonly used rye flour with hemp flour caused a slight increase in solids content of resin. An increase in the amount of solid content was accompanied by the reduction in gelation time of the mixtures. Using hemp flour as a filling material can be beneficial due to the shortening of gel time. However, there was no clear correlation between the amount of flour and the gel time values. This is highly advantageous from the technological point of view due to the fact that the pressing cycle, because of its energy consumption, should be as short as possible. The reason for the differences in curing properties between the adhesives mixtures filled with different types of flours was likely due to their chemical composition, which determines their reactivity; the course of this chemical interaction will be investigated as a future work. Both types of filling material and the amount of introduced filler did not considerably affect the pH level of the adhesives.

Table 3. Properties of Adhesives

Variant Label	pH	Gel Time (s)	Solid Content (%)
REF	7.2	78 ± 1*	66.04 ± 0.06*
H15	6.9	67 ± 2	67.71 ± 0.02
H20	7.1	69 ± 1	68.44 ± 0.04
H25	6.8	64 ± 1	66.91 ± 0.07
H30	6.9	67 ± 2	67.69 ± 0.03

*standard deviation

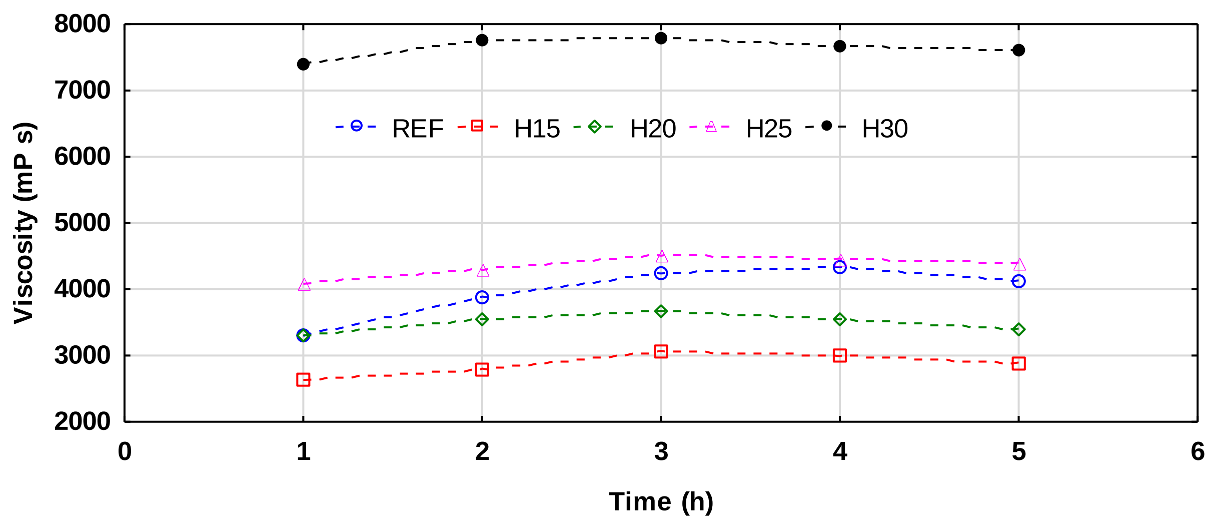


Fig. 2. Viscosity of adhesive mixtures

Figure 2 presents the results of the initial viscosity tested right after the homogenization of mixtures and its changes for 4 h. Viscosity is one of the most important parameters of the adhesive mixtures in plywood manufacturing. The highest dynamic of growth was observed for the first 2 h of measurements, and then it systematically slowed down. During the first hour of testing the viscosity of reference mixture increased faster, which was probably due to the high content of hydrophilic carbohydrates. There was a slight decrease in viscosity after 3 h of testing, which was probably due to the sedimentation of the filling particles. The variants containing 20 and 25 pbw of hemp flour per 100 pbw of solid resin achieved acceptable values similar to the reference mixtures prepared in accordance with industrial formulations. Viscosity of the adhesive labeled as H15 was significantly lower in comparison to control mixture. Viscosity that is too low results in sinking into the porous surface of the veneer, and consequently the amount remaining on the surface is insufficient to ensure good shear strength of the glue joint. The mixture containing 30 pbw of hemp flour per 100 pbw of solid resin had a significantly higher viscosity, which was incomparable with control adhesives. Because of a very high viscosity, it would be impossible to use this variant in practice due to the difficulties during the resin application. For this reason, the authors decided to skip this variant in the further part of the research.

Properties of Manufactured Plywood

Free formaldehyde content was investigated using the flask method, and the results are presented in Fig. 3. The replacement of rye flour with hemp flour resulted in a significant decrease of free formaldehyde emission.

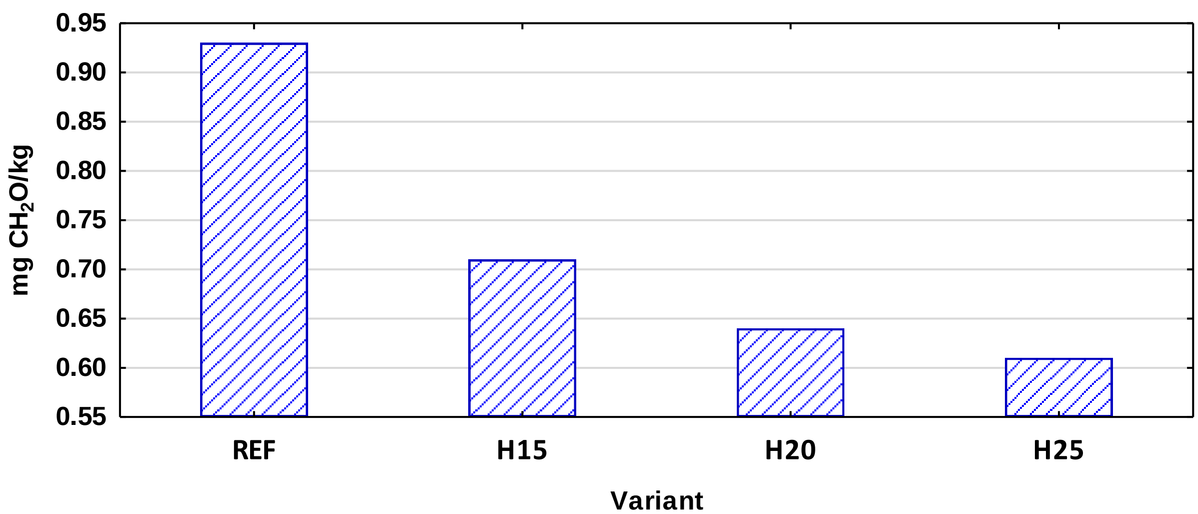


Fig. 3. Free formaldehyde content in plywood samples

The formaldehyde emission decreased from 23% to 34%, depending on the variant of experimental plywood. The more hemp flour was introduced into the resin, the less formaldehyde was emitted from manufactured panels. This progressive reduction occurs because of the increase in the share of substances absorbing formaldehyde. The reason for such a reduction was most likely the composition of new filler and more specifically the high content of proteins contained in hemp flour. Formaldehyde is the smallest aldehyde, and it is well known that an electrophilic molecule reacts with a variety of functional groups (*e.g.* amino, imino, amide, carboxyl, and sulfhydryl groups) (French and Edsall 1945;

Hoffman *et al.* 2015). In the context of binding free formaldehyde, its reactivity relative to amino acid is an important advantage. Hemp flour is a source of protein and mainly consists of albumin and edestin, and it is rich in essential amino acids (Callaway 2004; Korus *et al.* 2017). Within the frame of this study, hemp flour served as a kind of formaldehyde scavenger. Reduced formaldehyde content in plywood samples may have resulted from the fact that formaldehyde reacted with the amino acids of the protein. This reaction likely occurred in the manner shown in Fig. 4. Extensive model studies have been conducted on formaldehyde reaction with different amino acids (Fraenkel-Conrat and Olcott 1948a; Fraenkel-Conrat and Olcott 1948b; Metz *et al.* 2004), and it was found that during the first stage, formaldehyde reacts with amino and thiol groups. As a result of this reaction, methylol derivatives are formed. The part of methylol adducts are dehydrated and Schiff-bases are formed which allows a further type of cross-links reactions with other amino acid residues (Metz *et al.* 2004; Hoffman *et al.* 2015).

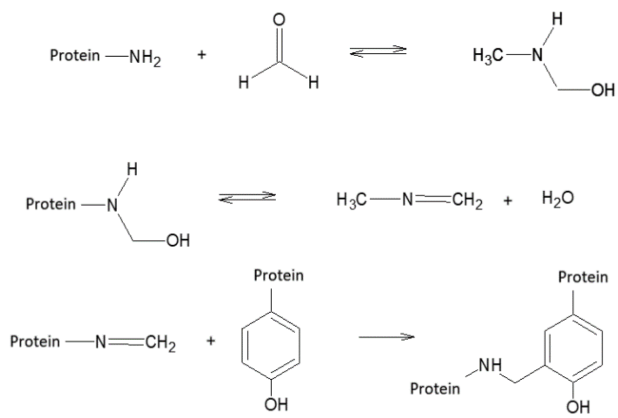


Fig. 4. The course of reaction of formaldehyde with proteins

Figure 5 presents the results of shear strength test, which is a commonly used indicator of adhesive behavior in manufactured plywood panels. Studies revealed that the effect of replacing rye flour with hemp flour on the bonding quality depended on the quantity of added filler (Fig. 5).

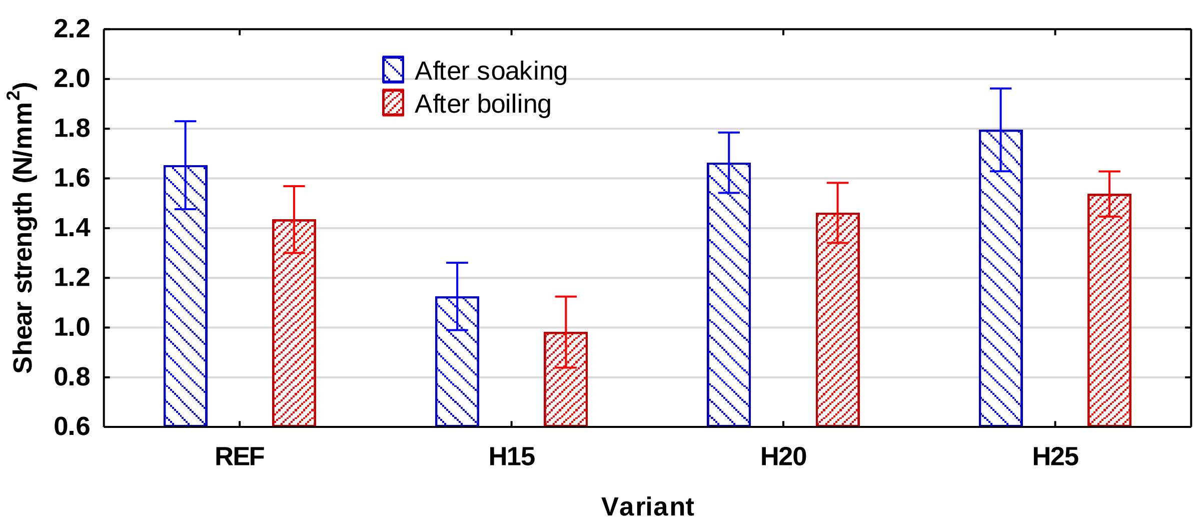
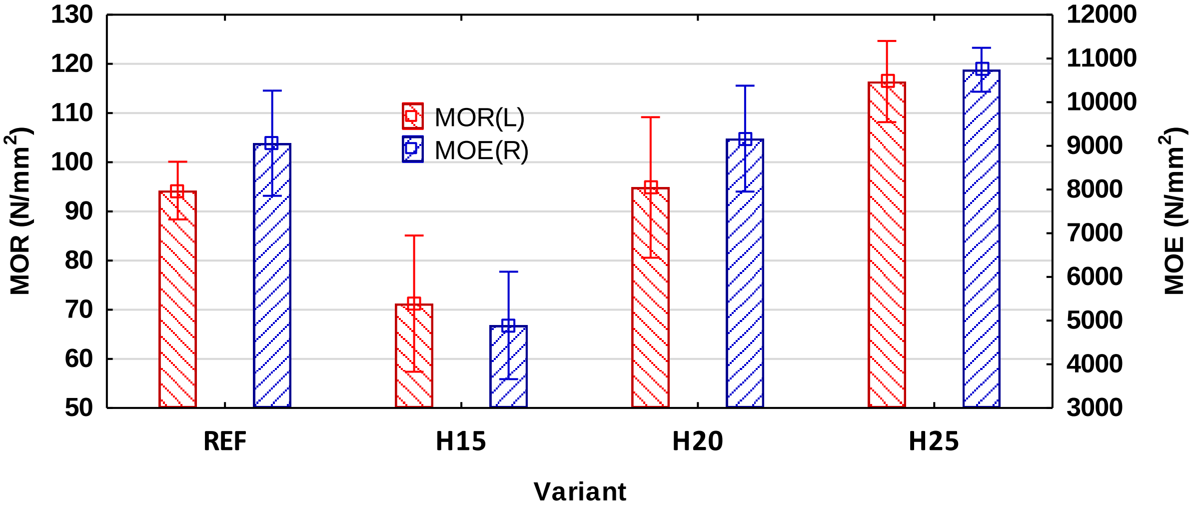
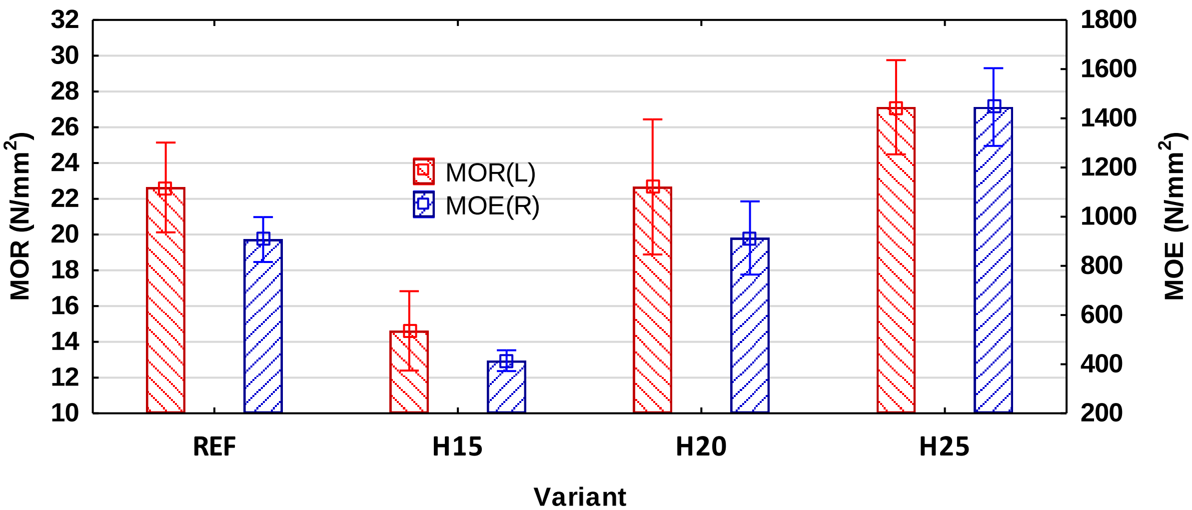


Fig. 5. Shear strength of plywood

Samples were tested both after soaking in water for 24 h and after a previously-described ageing test named "boiling" in Fig. 5. Variants of plywood glued with the adhesive mixture containing 20 and 25 pbw of hemp flour per 100 pbw of solid resin achieved satisfactory results, which were very similar to reference samples tested both after soaking and after boiling. However, there was a major deterioration of bonding quality in the case of plywood glued with H15 mixture. Values of shear strength decreased by 31% and 32% after soaking and after boiling, respectively. Further statistical analysis confirmed that the changes were significant in comparison to control samples. The reason for such distinct reductions in perpendicular tensile strength was probably due to low viscosity of the resin.



(a)



(b)

Fig. 6. Modulus of elasticity and bending strength of plywood tested: **(a)** parallel and **(b)** perpendicular to the grains of face layer

The adhesive mixture containing small amounts of fillers in the process of manufacturing plywood penetrated the surface of the veneers during the application and the pressing processes. Moreover, the application of resin was hard and it was almost impossible to evenly spread it on the surface because it flowed down into the cavities of the wavy veneer. Thus, the amount of adhesive remaining on the surface was not enough to ensure good bond properties. Due to the fact that the shear strength of plywood labeled as H15 tested after boiling fell below 1 MPa, the standard requires the investigation of the percentage of wood failure (WF), and it was 70%. This value meets the requirements of the standardized relation between average percentage of wood failure and average values of shear strength. Consequently, all plywood samples achieved bonding quality required by the EN 314-2 standard (1993). In order to evaluate the effect of replacing rye flour with hemp flour on the mechanical properties the experimental plywood were subjected to the three points bending test to assess modulus of elasticity (MOE) and bending strength (MOR) (Fig. 6).

The results of bending strength and modulus of elasticity corresponded with the shear strength values described in the previous section. According to the results of the statistical analysis, the adhesive mixture labeled as H20 can be used to manufacture plywood with the equally good mechanical properties as the reference sample because there were no significant differences. Moreover, further addition of filling hemp flour resulted in achieving even better results in comparison to the control samples glued with resin with rye flour and the improvement was significant. However, the addition of 15 pbw per 100 pbw of solid resin caused a major decrease in mechanical properties. The reason for this deterioration, as in the case of perpendicular tensile strength, was probably uneven distribution of the particles and penetration of the adhesive into the veneer due to its low viscosity. Studies have shown that the key to achieve an optimum reinforcement effect was the suitable properties of the liquid adhesives. In addition, their properties are significantly affecting the properties of manufactured wood-based materials.

CONCLUSIONS

1. Introducing the hemp flour adhesive instead of commonly used rye flour as a filler into the melamine-urea-formaldehyde (MUF) resin had a significant effect on the curing and rheological properties of the adhesives. The addition of hemp flour resulted in acceleration of gel time and the increase of solid content. The optimum level of viscosity suitable for plywood production was achieved with the addition of 20 and 25 pbw of hemp flour per 100 pbw of solid resin.
2. Due to the increasingly restrictive regulations on the emission of very harmful formaldehyde, it is very beneficial to reduce its content in wood-based materials manufactured with the use of amino resins. The free formaldehyde emission was significantly lowered in all variants containing hemp flour. The maximum reduction was 34% compared to control samples.
3. The amount of added filler had a significant effect on the mechanical properties such as bending strength, modulus of elasticity, and bonding quality of manufactured plywood. Introducing 20 and 25 pbw per 100 pbw of solid resin resulted in obtaining favorable results, which were equally good as the control samples. However, introducing too small a quantity of the filler caused a major deterioration of plywood mechanical properties.

ACKNOWLEDGMENTS

The paper was partially financed within the framework of Ministry of Science and Higher Education programme 'Regional Initiative of Excellence' in years 2019-2022, Project No. 005/RID/2018/19

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Article submitted: January 7, 2020; Peer review completed: March 21, 2020; Revised version received and accepted: April 1, 2020; Published: April 10, 2020.
DOI: 10.15376/biores.15.2.4052-4064