Influence of Moisture Content on the Bond Strength and Water Resistance of Bonded Wood Joints

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Bonding processes play a significant role in the wood and furniture industry. They allow for the creation of fixed joining of construction elements, creation of new materials and, last but not least, aesthetic appreciation of parts. However, the quality of bonded joints is affected by many factors, one of which is the moisture of the bonded material – wood. The main objective of this research was to determine the influence of wood moisture on the strength of bonded joints formed by polyvinyl acetate (PVAc) and polyurethane (PUR) adhesives. In current practice these adhesives are being increasingly used for their properties and zero formaldehyde content. The procedure for determining the bond strength (tensile shear strength of lap joints) corresponded to standard EN 205. It was ascertained that in addition to actual moisture of bonded wood, the quality of the joint is also affected by the environment to which the glued joint is subsequently subjected. In a normal environment, the strength of the tested joint PVAc adhesive decreases with increasing wood moisture, but it still meets the requirement of the standard. In a humid environment, the strength falls below the limit of the standardized value. In a normal environment the joint strength bonded with PUR adhesive is similar, but the decrease in strength is lower. In a humid environment it shows the highest strength at 20% wood moisture and meets the specified standard minimum strength (4 MPa). Graphs were created from the measured values that clearly show the influence of wood moisture on the final bond strength of a joint.

Keywords: Adhesive; Wood; Moisture; Water resistance; PVAc dispersion; PUR

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INTRODUCTION

Bonding technology has been developed over a long period of time and plays an important role both during the production of materials that eliminate defects in the structure of wood (*e.g.* agglomerated materials, structural timber and others), and in terms of replacing mechanical construction joints with bonded joints (*e.g.* woodworking, furniture components, and others) (Pizzi and Mittal 2003). Generally, bonded joints may provide a given material with better mechanical properties, such as greater joint strength over the strength limit of the actual bonded material, low weight in view of the structure of the created composite, the possibility to increase the resistance of the joint against the effects of biotic and abiotic agents (Richardson 1987), the possibility of simple joining of several dissimilar materials, and possible reduction of production costs (Custódio *et al.* 2009).

Adversely, bonded joints can also have a number of limiting properties. For example, the joints cannot be disassembled, wherein it is difficult to check the strength and lifespan of these joints, or to repair them. Another requirement is the careful preparation of the contact surfaces of bonded materials in relation to how they are processed (Dunky *et al.* 2002; Gaff and Gašparík 2013). In addition, some adhesives also have a more or less limited life span when affected by moisture, higher temperatures, or some chemical substances (Broughton and Hutchinson 2001). Use of formaldehyde-based adhesives began in order for the adhesive to have better resistance against the effects of water, but here there has been a problem with spontaneous unwanted release of formaldehyde into the surrounding environment (Pizzi 1993). For this reason, formaldehyde-based adhesives are often replaced with other types of adhesives with increased moisture resistance (Cremonini *et al.* 1997). These are most often PVAc and PUR adhesives, and large numbers of these joints can be found in available wood-based products. The aim is to find new ways to apply these formaldehyde-free adhesives to window, door, boards, and other joints. Each product is subject to different requirements for strength and resistance of joints in the relevant surrounding active conditions. The properties and quality requirements of joints must always be defined individually, and in order to achieve them, specific requirements must be defined during production (Örs *et al.* 1999; Hrázský and Král 2007). This also applies during the production of individual adhesives (chemical composition and process for their production), as well as in the area of technological process of the production of the joint itself (size of operating pressures and temperatures during pressing, open time during joint pressing and others) (Bomba *et al.* 2014).

Over the long term, water and damp air acting directly on the adhesive and inherent can reduce the bond strength of a joint, and this can lead to a complete collapse of the joint (Uysal 2005). There may be several reasons causing this phenomenon, and there are several hypotheses. The simplest explanation is provided by a mechanical theory according to which the adhesive effect is that the adhesive, in a liquid state, enters the pores and uneven areas of the bonded material (so-called adherent) and after hardening, it creates a large number of micro dowel joints. Based on this theory we can assume that due to the higher wood moisture (higher proportion of free and bound water), and consequently by the filling of the microscopic and sub-microscopic parts of wood, in this regard the adhesive will not have as much space to create so-called “micro dowel joints”, and for this reason the bonded joint is less resistant (Eisner *et al.* 1983). Over time, however, it has become apparent that the mechanical theory cannot satisfactorily explain the bonding of for example non-porous materials, or better adhesion of wood in a longitudinal section rather than in a transverse section, although the adhesive penetrates better and deeper into the wood, and fills a larger number of capillaries in a frontal cut. Bonding must therefore also depend on other factors, and not only on the penetration of the adhesive into the adherent. These factors should be sought in the physicochemical interaction between the adhesive and the adherent. Other theories have arisen as a result of this idea, which are included in the so-called specific theory of bonding, which was elaborated in further research into other theories, specifically the theory of mechanical interlocking (Browne and Brouse 1929), electronic or electrostatic theory (Gardner *et al.* 2005), adsorption (thermodynamic) or wetting theory (Gray 1962; Shi and Gardner 2001), diffusion theory (Gardner *et al.* 1994), chemical (covalent) bonding theory (Murrell *et al.* 1986), and the theory of weak boundary layers and interphases (Christiansen 1990).

All of the above theories agree that in order to achieve a high quality bonded joint, the substrate and adhesive molecules must get closer to each other so that there is sufficient adhesion between them. For this reason, at the moment of bonding it is necessary to have the adhesive in a liquid or at least plastic state. Another condition is that the adhesive must wet the surface of a solid substrate; therefore, the surface tension of the adhesive must be lower than the surface tension of the substrate (Liptáková and Sedliačik 1989). The adhesive must be kept for some time in a liquid state in a bonded joint in order to orient its molecules. Van der Waals forces or polarization forces may also act simultaneously, or there may be molecular diffusion due to Brownian motion. Chemical reactions with the substrate may occur at times. One can improve the adhesion via coatings of the substrate surface, through grinding or planing. Other ways of improving adhesion are increased pressure, temperature, and pressing time, as their effect is intensified by the orientation of the adhesive's molecules (Sedliačik 2005). However, at higher free and bound water content in bonded wood, an effect occurs where the molecules of the bonded substrate and the adhesive cannot sufficiently come closer together due to the presence of water molecules in the sub-microscopic and microscopic areas, which results in a less solid bonded joint (Mleziva 1993).

Several works are devoted to the effects of wood moisture on the joint strength: Niemz and Allenspach (2009); Gereke *et al.* (2011); Ren and Frazier (2012); Kägi *et al.* (2006). On the basis of the findings from Niemz *et al.* (2004), it was determined that slightly increased relative wood moisture (12 to 15%) has a relatively small impact on the speed of the hardening of the bonded joint where the PUR adhesive is used. Furthermore, Schirle *et al.* (2002) determined that the difference in the hardening time and the quality of a bonded joint under the effects of various moistures is also affected by various large proportions of tree rings of spring and summer wood. Radovic and Goth (1994) described the impact of relative humidity on the speed of hardening of polyurethane adhesives. When achieving a climatic temperature of 20 ^{o}C and a relative humidity of 65% (corresponds to 12% wood moisture), after the first 16 h they observed an increase in the strength of the polyurethane joint. Adversely, during a subsequent climate change to 15 ^{o}C and 90% relative humidity (corresponding to a wood moisture of approx. 30%), the strength of the joint decreases. The lifespan of a joint bonded with polyurethane adhesive at relative humidity was investigated by Vick and Okkonen (2000). It was found that during the hardening phase of the adhesives, the isocyanate groups (NCO) of the adhesive react with the hydroxyl groups (OH), which are mainly contained in hygroscopic water. The selection of a partner reactant to the OH group depends on the kinetic energy potential, in particular on the size of the attractive force. The isocyanate group NCO immediately reacts with a water molecule as soon as it is free. If this is not possible, the NCO will immediately join the OH group of the wood substance. If heat energy is simultaneously added, the hardening reaction will be further accelerated. Pizzi and Owens (1995) found that during heat hardening of a joint bonded with PUR a covalent joining occurs between the isocyanate and cellulose as soon as very high temperatures between 128 to 180 °C begin to act, and there is no free water available for reaction with the NCO. For this reason, covalent bonds of PUR adhesives are excluded from these areas of use of bonded joints.

Standard ČSN EN 204 defines the general conditions for industrial bonded joints in wood products. The effect of moisture is a key indicator affecting the quality of the bonded joint. The main goal of this work was to find the dependencies between changing wood moisture and the degree of bond strength, and the resistance of the joints to effects of moisture from the surrounding environment in joints bonded by PVAc and PUR adhesives. Furthermore, it will be determined whether or not the used adhesives fulfil the conditions for applicability according to the provisions in standard ČSN EN 204.

EXPERIMENTAL

Measurement Methodology

Measurements, preparation of test specimens, and evaluation of results were conducted in accordance with standards ČSN EN 204 and ČSN EN 205.

Test specimens were manufactured according to Fig. 1 from beech wood (*Fagus sylvatica* L.) with straight graining with a nominal density (700 ± 50 kg/m^3). The angle between the tree rings and the bonded surface was between 30° and 90° (radial or semi-radial surfaces).

The wood needed to produce specimens was divided into three groups and air conditioned under conditions such that the moisture within each group was stabilized at values of 8 ± 1%, 12 ± 1%, and 20 ± 1%. In order to air-condition the wood to the appropriate moisture, a WEISS TECHNIK type 12SD/15JU air-conditioning chamber with a measurement range from 0 to 40 °C, 5 to 100% relative humidity, and a steady flow of air at 0.15 m/s at the surface of specimens was used.

Once all three material moisture levels (8 ± 1%, 12 ± 1%, 20 ± 1%) were achieved, test specimens were manufactured. The surfaces intended for bonding were processed using planar milling, not earlier than 24 h before bonding. Two adhesives were used for bonding of specimens - PVAc dispersion adhesive Klebit 314.0 (Kleiberit) and PUR adhesive PUR Leim 501.0 (Kleiberit).

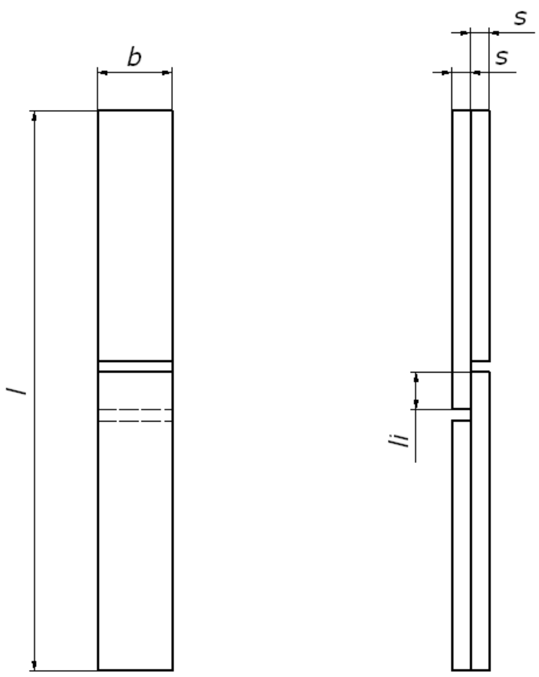
The conditions for bonding (deposition, open time, pressing pressure) were in accordance with the data specified by the manufacturer of the adhesives (Table 1). Bonding was carried out at a temperature of 18 ± 2 °C and such relative air moisture to avoid a change in the wood moisture. The moisture was measured using the weight method.

Batches of 120 test specimens were produced with wood moistures of 8 ± 1%, 12 ± 1%, and 20 ± 1%. Each group of specimens was bonded using two types of adhesives: 60 PVAc specimens and 60 PUR specimens.

Since the used adhesives had a D4 resistance class (interior with frequent, long-term effects of water or exterior exposed to weather), three tests were carried out for each adhesive with sequential numbers 1, 3, and 5 (prescribed in accordance with ČSN EN 204). The first test was carried out 7 days after bonding (test sequence number 1). For each test, the parameters of the environment in which the samples were conditioned for 7 days of 20 ± 2 °C and relative air moisture of 65 ± 5% in such a way moisture of wood does not change (hereinafter referred to as the normal environment).

The second test was carried out in such a way that after 7 days the samples were immersed for 4 days in water at 20 ± 5 °C, and then immediately tested (test sequence number 3). The third test was carried out so that after 7 days the samples were immersed for 6 h in boiling water and were then put in water at 20 ± 5 °C for 2 h, and then immediately tested (test sequence number 5). These 3 tests were performed for each adhesive and each degree of moisture, *i.e.* a total of 18 tests of 20 specimens.

The number of test specimens was chosen so that at least 10 valid values were measured for each test. Invalid results are considered those where there was a breach in the wood rather than in the adhesive layer, or where it was visually apparent that the adhesive was not properly applied.



l= 150 ± 5 mm
li= 10 ± 0.2 mm
b= 20 ± 0.2 mm
s= 5.0 ± 0.1 mm

Fig. 1. Test specimens according to ČSN EN 205

Table 1. Properties of Tested Adhesives (Datasheets)

Adhesive	PVAc Klebit 314.0	PUR Leim 501.0
Bond quality	D4	D4
Application quantity [g/m^2] (adhesive applied on both adherents)	150 - 200	100 - 200
Viscosity at 20 °C [mPas]	7.000 ± 1.000 according to RVT Brookfield	8.000 ± 1.000 according to Haake VT 180
Min. processing temperature [°C]	+ 9	+ 5
Open time [min]	4 - 7	20 - 25
Wood moisture [%]	8 - 14	8 - 12
Pressure [MPa]	0.7 - 1	min. 0.6
Density [g/cm^3]	1.15	1.13
Pressing time at 20 °C joint bonding [min]	min. 15*	min. 60*

*The pressing time of the test samples was 3 hours.

Test equipment

The test equipment used was a testing machine with a constant feed rate as described in ISO 5893.

Transverse tensile test according to ČSN EN 205

Test specimens were tested in the test machine. The specimens were fastened on both ends into the jaws of the machine at a length of 40 mm to 50 mm. The test specimens were burdened by a tensile force until they were breached, and the highest exerted force was recorded in $F_{\max}$ in Newtons (N). The feed rate of the tensile testing machine was a constant 50 mm/min. Simultaneous measurements were also conducted on the surface of the bonded joints in each specimen.

Expression of results according to ČSN EN 204

The bond strength of a bonded joint (τ) was expressed in MPa and calculated according to the following equation,

$$\tau = \frac{F_{\max}}{l_2 \times b} \qquad (1)$$

where F_{max} is greatest exerted force in Newtons (N), l_2 is the length of the bonded test surface in millimetres (mm), and b is the width of the bonded test surface in millimetres (mm).

RESULTS AND DISCUSSION

The measurements determined the degree of strength at maximum load on the bonded joint under the effects of bonded wood moisture (Test No. 1) and external environment moisture (Tests Nos. 3 and 5). The measured values are specified in Tables 2 and 3.

Table 2. Overview of Measured Values for Adhesive PVAc

Wood moisture [%]	8			12			20		
Type of test	1	3	5	1	3	5	1	3	5
X [MPa]	16.96	2.16	2.06	14.91	2.52	2.52	10.75	1.38	1.34
Max. [MPa]	20.10	2.89	2.77	15.94	3.53	3.20	12.33	1.89	1.72
Min. [MPa]	14.71	1.49	1.46	13.46	1.53	2.08	9.22	0.98	1.08
SD	1.48	0.46	0.41	0.73	0.65	0.38	0.88	0.26	0.22
v [%]	8.74	21.53	19.74	4.90	25.85	15.24	8.24	18.79	16.73

X – average value; Max. – maximum measured value; Min. – minimum measured value;
SD – standard deviation; v – coefficient of variation

Table 3. Overview of Measured Values for Adhesive PUR

Wood moisture [%]	8			12			20		
Type of test	1	3	5	1	3	5	1	3	5
X [MPa]	14.20	5.82	4.07	13.53	6.12	4.69	11.62	6.44	5.22
Max. [MPa]	15.36	7.55	5.26	16.04	7.40	6.03	13.23	7.40	6.08
Min. [MPa]	11.65	4.61	3.07	11.35	4.81	3.85	10.15	5.28	4.45
SD	1.28	0.94	0.74	1.50	0.90	0.79	1.04	0.60	0.49
v [%]	9.04	16.09	18.13	11.08	14.74	16.89	8.93	9.35	9.44

X – average value; Max. – maximum measured value; Min. – minimum measured value;
SD – standard deviation; v – coefficient of variation

The results were evaluated at a significance level of α = 95% using an analysis of variance (ANOVA), and the difference of individual groups using a post-hoc test (Tukey HSD test). The results of the analysis of variance are shown in the graphs in Figs. 2, 3, and 4.

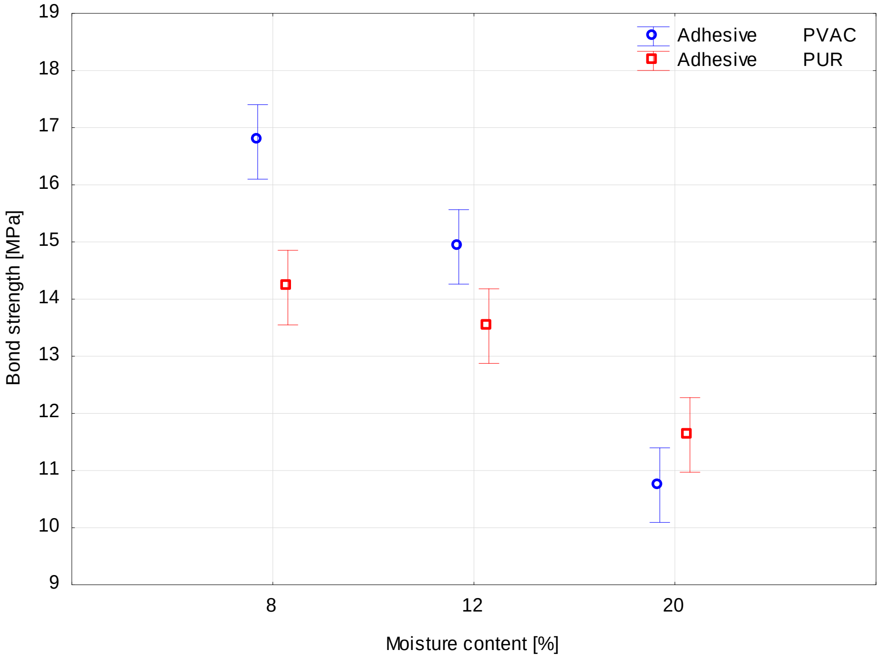


Fig. 2. Bond strength according to adhesives and wood moisture after 7 days in a normal environment (Test No. 1)

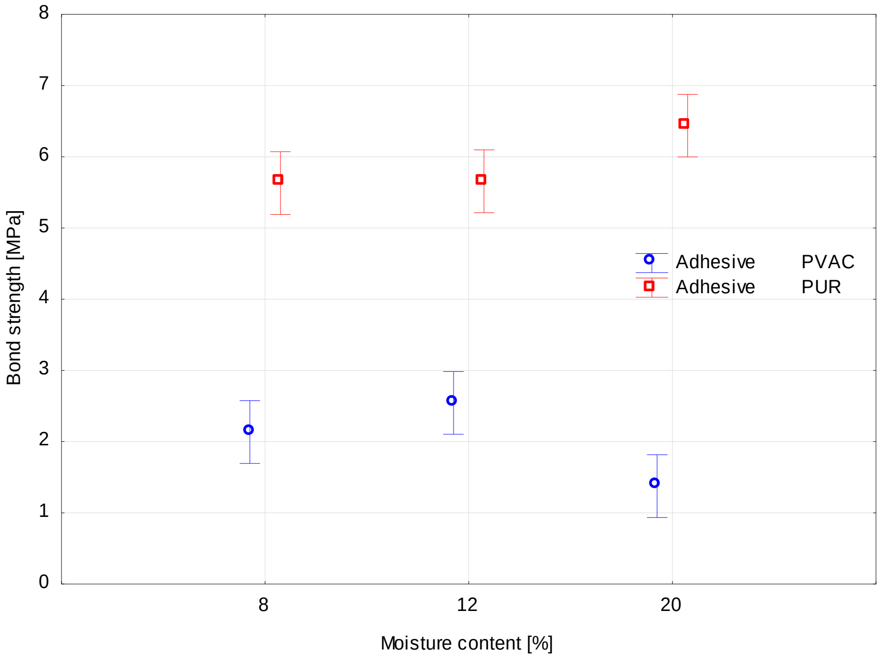


Fig. 3. Bond strength of a joint after 7 days in a normal environment, and then for four days in water at a temperature of (20 ± 2) °C (Test No. 3)

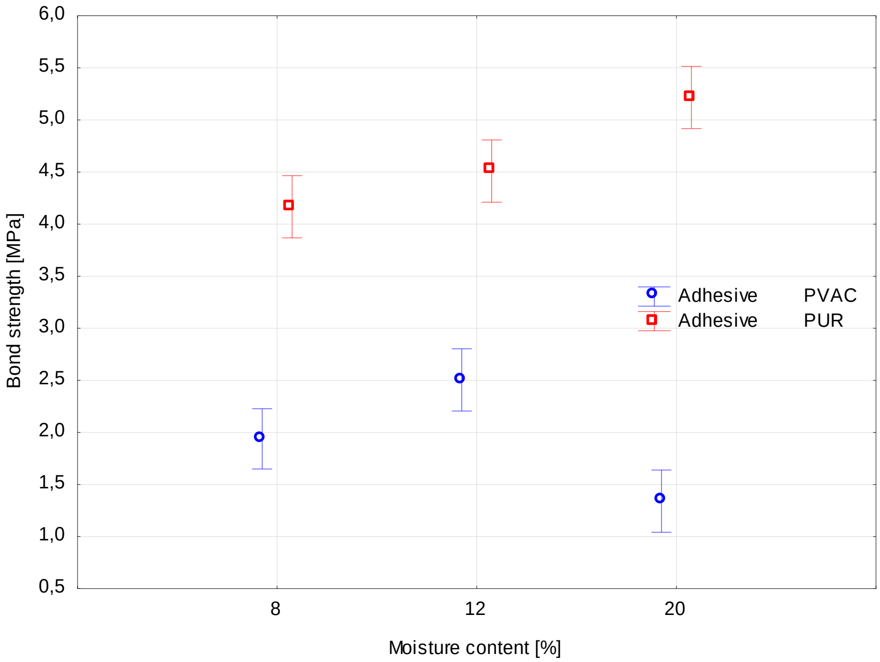


Fig. 4. Bond strength according to adhesives and moisture during their bonding, after 7 days in a normal environment, 6 h in boiling water and 2 h in water at a temperature of (20 ± 2) °C (Test No. 5)

The greatest bond strength values in a normal environment after 7 days were achieved by the test specimens bonded with the PVAc adhesive at wood moisture of 8% (approx. 17 MPa). With increasing bonded wood moisture (12 and 20%) the strength of this adhesive demonstrably decreased (14.91 MPa and 10.75 MPa), but at 20% moisture it still met the requirements of the standard for a minimum strength of the joint of 10 MPa. Joints bonded with PUR adhesive behaved similarly, but their reaction to moist wood was not as sensitive as that of the PVAc adhesives. The bond strength of a joint at 8% moisture was lower (14.2 MPa) than PVAc, but its decrease in wood with a moisture of 12% (13.53 MPa) was not demonstrable (p = 0.642533). It was only demonstrably lower during bonding of wood with a moisture of 20% (11.62 MPa); nevertheless, average values stabilized at higher values when compared to PVAc adhesives (Fig. 2). Here as well, all of the measurements met the minimum requirements of the standard. During these measurements, some of the specimens were breached in the wood, not in the bonding of the joint. Such values were excluded from the measurements.

For this test (No. 3) there was an evident significant difference between the results of both types of adhesives (p = 0.000124). While the bond strength for PUR adhesives was higher than the amount required by the standard (4 MPa, which is also confirmed by another study, *i.e.* Sterley *et al.* 2004), the joint strength of PVAc adhesives was significantly lower and did not reach the minimum requirements of the standard. During bonding of wood with moistures of 8% and 12%, there was essentially no difference in the PUR adhesives (5.82 MPa and 6.12 MPa). For wood with a moisture content of 20% there was a lower decrease in strength (6.44 MPa), but this difference was not statistically demonstrable. For PVAc adhesives there was a lower decrease in strength in samples with a moisture of 12% compared to 8% (2.52 MPa compared to 2.16 MPa), but this difference was also not demonstrable. However, there was a significant decrease in strength at a moisture of 20% (1.38 MPa) (p = 0.004599). The results are shown in Fig. 3.

The results for this test (No. 5) were very similar to results of the previous tests (Test No. 3). Joints bonded with the PUR adhesive again met the minimum strength of 4 MPa specified by the standard, but when testing samples with 8% moisture, some values were below this threshold. A lower strength decrease was observed in samples with 12% moisture, of although inconclusively. The highest strength in these tests was measured in samples with 20% moisture content. For samples with 8% moisture, joint strength in the PVAc adhesive was about 2 MPa. At 12% moisture the decrease in strength was lower and ranged around 2.5 MPa. The lowest strength was measured for samples with 20% moisture content (about 1.3 MPa) (Fig. 4).

CONCLUSIONS

1. In order to create a better quality joint using PVAc adhesive, it is necessary for the wood moisture to be around 8% to 12% during bonding, as specified by the manufacturer. Nevertheless, it was demonstrated that even higher moisture (20%) does not significantly decrease the quality of the joint when compared to lower moistures (8% and 12%).
2. Joints in wood bonded at increased moisture (20%) exposed to a normal environment also meet the standard threshold of 10 MPa.
3. The highest average bond strength values under the effects of water from the external environment are also achieved by joints bonded at 8% and 12% wood moisture. It should be noted that the tested adhesive, Klebit 314.0, did not achieve the required standard values for moisture class D4 in tests with sequential numbers 3 and 5.
4. The results of the measurements show that a joint created using a PUR adhesive meets the standard threshold value of 10 MPa or 4 MPa in all 3 environments at all 3 initial bonded wood moistures.
5. The results of average strength values for samples bonded with a PUR adhesive exposed to a normal environment achieve lower values than those for the same group bonded with PVAc adhesive. For the group bonded at 20% moisture, a statistical difference was not demonstrated, but the average strength values of the PUR adhesives are higher than the PVAc values.
6. The resistance of PUR adhesive Leim 501.0 against moisture is higher than the resistance of the PVAc adhesive. The technical sheet for the adhesive specifies optimal bonding moisture of 8 to 12%. In this wood moisture range, a difference was not demonstrated in the quality of the bonded joints – conversely, a statistically relevant difference was demonstrated for specimens bonded at 20% wood moisture. It was ascertained that it is better to bond products exposed to high moisture from the external environment using moist wood (20%).

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