Influence of Temperature on the Strength of Bonded Joints

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The objective of this study was to assess the influence of varying temperature on the strength characteristics of joints bonded using three types of one-component PVAc adhesives (*i.e.*, Rakoll Express D3, Titebond II Premium, and Rhenocoll 3W, 4B Plus) that belong to the group of thermoplastic wood adhesives intended for non-construction bonding applications. The measuring procedure was a transverse tensile test for estimation of joint strength. As documented by the test results, a higher joint strength achieved at a higher temperature was achieved again after a gradual increase in the joint temperature from 20 °C to 140 °C and subsequent cooling to the starting temperature. After cooling the joint to the starting temperature, all tested adhesives complied with the minimum strength, namely 10 MPa. The results obtained in this study indicated that the resistance and thus the strength of joints bonded using contemporary PVAc adhesives increases with time.

Keywords: Adhesive; Wood; Temperature; Strength of bond; PVAc

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INTRODUCTION

Maintaining the resistance as well as stability of bonded joints exposed to increased temperatures is essential for practical use. Bonded joints can be exposed to elevated temperatures in various situations, such as being exposed to solar radiation. In this case, joint temperature can exceed 60 °C at the exposed spots (Falkner and Teutsch 2006; Cidlina *et al*. 2014; Müller 2014). The effects of increased temperatures on bonded joints can also be seen in fires (Glos and Henrici 1991). In the wood structures sector, new adhesives have been developed in recent years, and, at the same time, the influence of increased temperature on bonded joints has been defined (George *et al.* 2003; Frangi *et al.* 2004; Richter *et al.* 2006).

Polyvinyl acetate dispersion adhesives (PVAc) belong to the group of thermoplastic polymers that are capable of being reversibly deformed within a specific temperature range (Eisner *et al*. 1983). Originally, these adhesives were designed for manufacturing plywood boards and, generally, for furniture manufacturing (Clauß *et al.* 2011; Gaff and Gáborik 2014). Their advantages consist of the ability to make high-strength joints, easy application, and short hardening time (Tout 2000; Bomba *et al.* 2014). The polyvinyl acetate adhesives do not contain additives, hardening agents, or stabilisers. Thermal stability was not considered in this sector of application.

As demonstrated in the studies conducted so far, non-modified polyvinyl acetate dispersion adhesives are characterized by increasing joint strength with temperatures rising from 0 to 30 °C (Fig. 1) (Örs *et al.* 2000; Dilik and Hiziroglu 2004). If the temperature rises above the glass transition temperature (around 30 to 40 °C), a reduction in joint strength is observed. At temperature exceeding 100 °C, there is a reduction in tensile strength below 1 MPa (Kaboorani and Riedl 2011b), a fact that has made the use of PVAc adhesives impossible for bonding teeth connection or load-bearing structural elements so far. The disadvantage of these adhesives consists in their chemical structure: PVAc is a linear amorphous polymer with weak polar couplings in its macromolecular chains, resulting in chemical bond degradation with increasing temperature (Eisner *et al*. 1983). This is why various research projects are focused on improving the properties of PVAc adhesives through modifying them.

Currently, there are several options for modifying PVAc adhesives in order to improve their resistance to higher temperatures, including co-polymerisation of vinyl acetate with several hydrophilic monomers and functional monomers (Chen 1996; Cai 1997) or mixing PVAc with other adhesives or hardeners (Lu 1996; Qiao *et al.* 2000; Huang *et al*. 2002; Kim and Kim 2005 a,b; Lopez-Suevos and Frazier 2006; Kaboorani and Riedl 2011a). Another option is PVAc adhesive modification with various types of nanoparticles (Kim *et al*. 2007; Kaboorani and Riedl 2012). This option is currently being tested. These modifications improve specific properties; however, at the same time they deteriorate other adhesive properties. Some additives are acidic; hence these modification agents can affect or reduce chemical bonds in wood joints. For instance, vinyl acetate and butyl acrylate or ethylene copolymers are capable of increasing the strength of bonded joints and their resistance to water; on the other hand, at higher temperatures they reduce the elasticity modulus as well as the adhesive stiffness (Qiao and Easteal 2001). Nevertheless, increased resistance to higher temperatures has been observed in some PVAc adhesives, with a resulting increase in their application in wood-based materials in residential buildings (Le Bras *et al.* 1996; Horold 1999; Kandola 2001; Kashiwagi *et al.* 2003; Zhang and Horrocks 2003; Bartholmai and Schartel 2004). Naturally, this has led to an increase in the price of these adhesives.

The EN 204 (2001) standard specifies the strength requirements of joints bonded using thermoplastic adhesives intended for wood in non-construction applications. The main objective of this study is to determine the influence of temperature on the strength of bonded joints immediately after exposure to a higher temperature, and also after subsequent cooling to ambient temperature. Particular attention was paid to the permanent effects on joint strength after cooling.

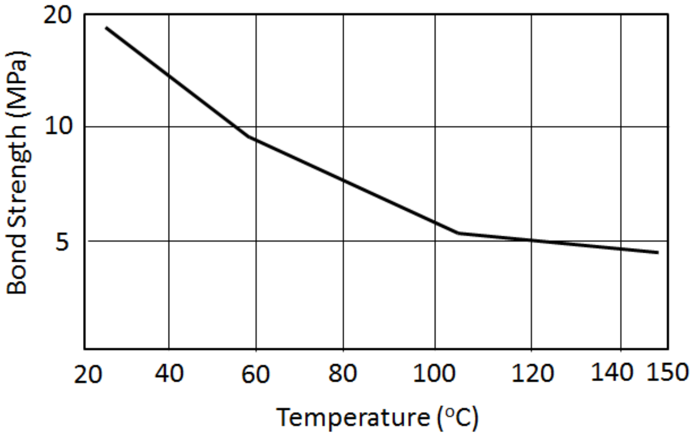


Fig. 1. Correlation between strength of the bonds made using PVAc Rhenocoll 3W, 4B Plus and temperature (Rhenocoll 3W 2004)

EXPERIMENTAL

Materials

The specimens for experimental measurement were made in accordance with the specification provided in EN 205 (2003). For each group, 12 test specimens were made. The specimens were made of strong, non-steamed, at first air dried and after that finally kiln dried beech (*Fagus sylvatica*) board with straight wood fibres of nominal density 700 ± 50 kg/m^3 and moisture content 12 ± 1%. The angle between annular rings and the board surface ranged from 30° to 90°. The specimens were subjected to 7-day standard conditioning (20 ± 2 °C and relative air humidity 65 ± 5%) to maintain wood humidity at 12 ± 1%.

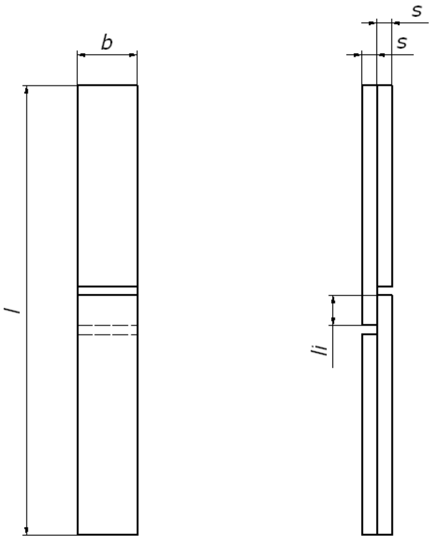
Methods

Measurements were conducted in accordance with EN 205 (2003) and EN 204 (2001) (Fig. 2). The lapping joints of the specimens were bonded using three PVAc adhesive products from various manufacturers, all products being of the water resistance class D3. The glued joints were constant loaded using a pressure of 1 MPa/mm^2 for 60 min. After that, the samples were conditioned for 7 days in standard environment 20 ± 2 °C and relative air humidity 65 ± 5% in a full cure (EN 205 (2003)). The bonding strength tests specimen were performed according EN 205 (2003) at crosshead constant speed of 5 mm/min. The testing velocity by lapping joints for PVAc adhesives of the D3 resistivity class is defined 5 mm/min. The first adhesive was one-component Rakoll Express D3 (H. B. Fuller, Nienburg, Germany). Rakoll Express D3 is a polyvinyl acetate adhesive in water dispersion. The properties of this adhesive are in compliance with EN 204 - D3 (2001).

The second adhesive was one-component PVAc Titebond II Premium (Franklin International Inc. Columbus, Ohio, USA). Titebond II Premium is a synthetic-resin-based polyvinyl acetate adhesive in water dispersion. This high-strength adhesive meets the stringent hydrophobic specifications indicated in the US standard ANSI Type 2 (ANSI/HPMA 1994).

The third adhesive was one-component PVAc Rhenocoll 3W, 4B Plus (Rhenocoll-Werk e. K. Konken bei Kusel, Germany). Rhenocoll 3W, 4B Plus is a synthetic-resin-based polyvinyl acetate adhesive in water dispersion and contains components which prevent the penetration of water into transversal wood structures and inhibit moulds. This adhesive has been certified to be in accordance with DIN EN 14257 (2010) by the IFT Rosenheim (Window Technology Institute) and features excellent bonding properties at higher temperatures. For the parameters of the adhesives, please refer to Table 1.

The results value of bond strength according to adhesives were evaluated at a significance level of $\alpha = 95\%$ using an analysis of variance (ANOVA), and the difference between individual groups was determined using a post-hoc test (Tukey HSD test) (STATISTICA, version 12, StatSoft CR, Prague, Czech Republic). The results of the analysis of variance are shown in Figs. 3 and 4.



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

Fig. 2. Test specimens according to EN 205 (2003)

Table 1. Properties of Tested Adhesives (Rakol Express D3 2002; Titebond II Premium 2004; Rhenocoll 3W, 4B Plus 2004)

Adhesive	Rakoll Express D3	Titebond II Premium	Rhenocoll 3W, 4B Plus
Bond quality	D3	D3	D3
Application quantity (g/m^2) (adhesive applied on both adherents)	160 - 180	180 - 190	120 - 150
Viscosity at 20 °C (mPas)	13,000 mPa/s Brookf. HBT**	4,000 mPa/s Brookf. HBT	4,000 mPa/s Brookf. HBT
Minimum processing temperature (°C)	+ 5	+ 13	+ 3
Open time (min)	8 - 12	5	5 - 8
Wood moisture (%)	8 - 10	8 - 12	10 - 12
Pressure (MPa)	0.10 - 0.50	0.70 - 1.10	0.2 - 0.5
Density (g/cm^3)	–***	1.09	1.03
Pressing time at 20 °C joint bonding (min)	minimum 8*	minimum 10*	minimum 6*

*The pressing time of the test specimens was 3 h
**Type of viscosity measurement by Brookfield method
*** The manufacturer didn´t state a value

Test principle

Thirteen groups of test specimens (each including 12 units) were prepared for each adhesive, *i.e.*, 39 groups with 12 units each. The first group of each adhesive was subject to testing under normal conditions (*i.e.*, after 7 days conditioning). The remaining

12 groups were divided into sub-groups. Each sub-group was exposed to gradually increasing temperatures in the intervals 40, 60, 80, 100, 120, and 140 °C. The exposure time was set in such a manner as to allow the entire specimen cross-section to be heated. According to Standard ON 490651, heating of wood with a thickness of 1 cm takes 1 h. The steady-state of temperatures by the specimens were controlled by thermometer. After heating to an adequate temperature, each sub-group was taken from the heating chamber and divided into a further 2 sub-groups, one of them being immediately tested and the other stored under normal conditions for 7 days. This procedure was applied for each temperature stage and each type of adhesive. After the expiration of 7 days, the other group was also tested. A tensile shear strength test was conducted in compliance with the specification in EN 204 (2001), *i.e.*, the specimens were subjected to the application of force until damage, with a maximum applied force $F_{\max}$ being recorded in Newtons (N).

Test equipment

The test equipment used was a testing machine with a constant feed rate as described in ISO 5893 (2002).

Transverse tensile test according to EN 205 (2003)

Test specimens were tested in the tensile testing machine WPM ZDM5/91, VEB TIW RAUENSTEIN, Struppen, Germany. The specimens were fastened on both ends into the jaws of the machine at a length of 45 ± 5 mm. The test specimens were burdened by a tensile force until they were breached, and the highest exerted force $F_{\max}$ in Newtons (N) was recorded. The feed rate of the tensile testing machine was a constant 5 mm/min. Simultaneous measurements were also conducted on the surface of the bonded joints in each specimen.

Expression of results according to EN 204 (2001)

The bond strength of a bonded joint (τ) is expressed in MPa and calculated according to Eq. 1,

$$\tau = \frac{F_{\max}}{l_2 \times b} \tag{1}$$

where F_{max} is the ultimate 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

Measurements were carried out to determine the degree of strength at maximum load on the bonded joint under the effects of an external environment by specific degree of temperature. The measured values are specified in Tables 2 through 7.

Table 2. Overview of Measurement Readings for Rakoll Express D3

Temperature (°C)	20	40	60	80	100	120	140
X (MPa)	13.51	12.69	10.57	7.32	5.12	4.51	3.73
Max. (MPa)	19.82	22.61	12.29	10.31	6.79	5.82	5.92
Min. (MPa)	12.23	12.11	8.01	4.87	3.81	3.11	1.33
SD	2.22	3.52	1.51	1.71	1.10	0.83	1.11
v (%)	0.20	0.21	0.11	0.23	0.20	0.19	0.30

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

Table 3. Overview of Measurement Readings after Cooling to 20 °C for Rakoll Express D3

Temperature (°C)	40	60	80	100	120	140
X (MPa)	12.70	12.61	12.89	12.60	12.30	11.66
Max. (MPa)	21.91	20.50	22.14	13.71	13.17	13.11
Min. (MPa)	12.13	12.33	12.20	12.10	8.01	6.20
SD	4.30	2.30	3.53	0.51	1.44	1.82
v (%)	0.31	0.20	0.19	0.01	0.10	0.20

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

Table 4. Overview of Measurement Readings for Titebond II Premium

Temperature (°C)	20	40	60	80	100	120	140
X (MPa)	17.01	12.71	11.22	9.91	6.32	5.63	4.22
Max. (MPa)	22.54	21.12	12.23	10.93	7.71	6.11	5.21
Min. (MPa)	12.30	12.22	10.31	5.90	5.45	4.93	3.43
SD	3.62	3.21	0.88	1.91	0.61	0.42	0.60
v (%)	0.23	0.20	0.11	0.22	0.13	0.11	0.11

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

Table 5. Overview of Measurement Readings after Cooling to 20 °C for Titebond II Premium

Temperature (°C)	40	60	80	100	120	140
X (MPa)	13.01	19.10	19.64	13.01	12.22	12.13
Max. (MPa)	22.33	22.02	21.11	20.52	13.04	12.71
Min. (MPa)	11.72	12.43	12.44	12.21	11.63	11.24
SD	4.22	3.79	3.16	3.07	0.51	0.51
v (%)	1.11	4.27	3.82	0.71	0.29	1.61

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

Table 6. Overview of Measurement Readings for Rhenocoll 3W, 4B Plus

Temperature (°C)	20	40	60	80	100	120	140
X (MPa)	14.42	16.19	12.21	10.73	8.11	7.02	4.11
Max. (MPa)	22.22	20.91	13.13	11.81	10.12	8.52	5.02
Min. (MPa)	12.04	12.53	10.51	7.24	6.62	6.33	3.11
SD	3.61	3.52	0.72	1.63	1.21	0.62	0.52
v (%)	0.22	0.23	0.11	0.20	0.11	0.13	0.11

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

Table 7. Overview of Measurement Readings after Cooling to 20 °C for Rhenocoll 3W, 4B Plus

Temperature (°C)	40	60	80	100	120	140
X (MPa)	12.72	18.74	13.42	12.52	12.51	11.63
Max. (MPa)	16.12	24.23	21.63	14.44	14.10	12.62
Min. (MPa)	12.11	12.52	12.38	12.14	11.62	7.41
SD	1.12	4.34	3.84	0.73	0.91	1.62
v (%)	0.13	0.24	0.21	0.11	0.12	0.11

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

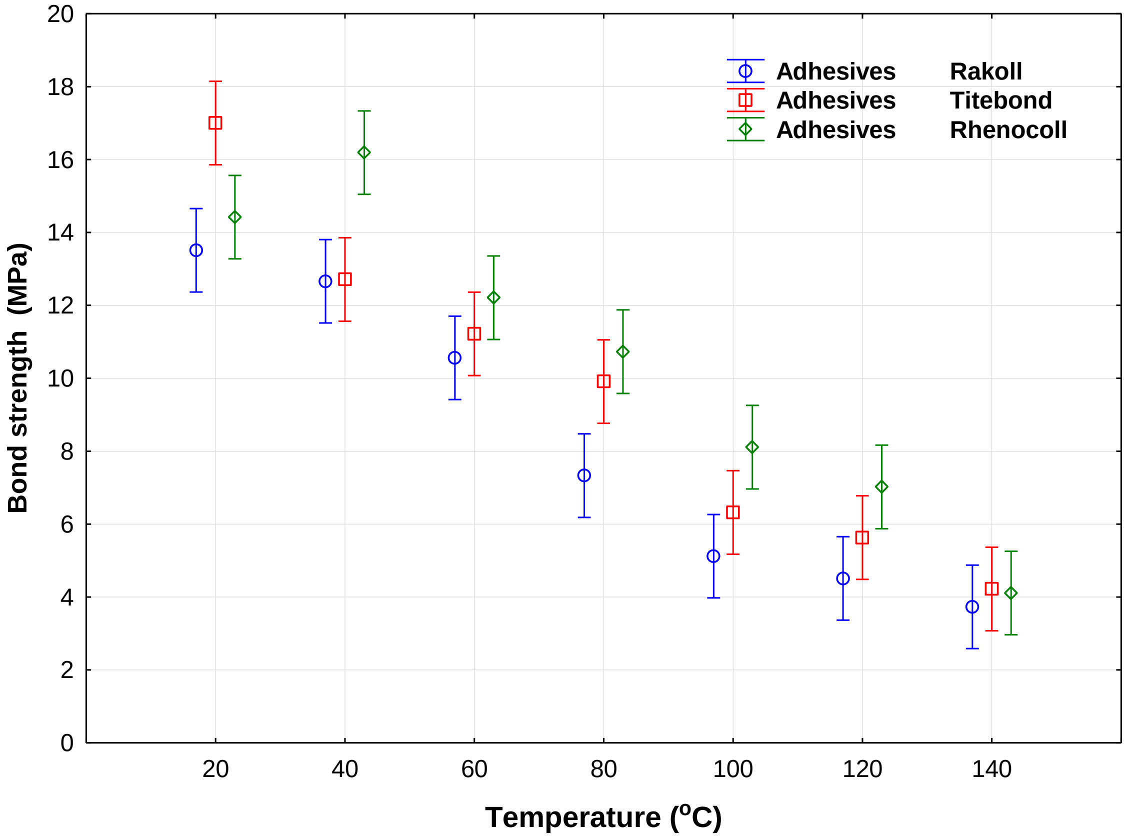


Fig. 3. Bond strength according to adhesives and specific wood temperature after 7 days and exposure to relevant temperature during 1 h. Data provided as the mean ± standard deviation

The results for all 3 adhesives tested were highly similar. A different characteristic was obtained with Rhenocoll 3W, where a moderate increase in strength could be seen between 20 and 40 °C. With regard to Rhenocoll, as soon as the temperature rose to the glass transition temperature (30 to 40 °C), the adhesive softened (its flexibility increased) and the joints lost some of their strength as the temperature increased (60, 80, 100, 120, and 140 °C). For Titebond II Premium, which had the highest joint strength under normal conditions, and for Rakoll Express D3, a continual reduction in strength was recorded across the entire temperature range (Fig. 3), and the differences in the values obtained for individual temperature stages were statistically relevant (p= 0.000046). Obviously, the best results in the temperature interval 40 to 120 °C were obtained with Rhenocoll 3W, 4B Plus, which meets the minimum required strength specification (10 MPa) even at 80 °C. The overall strength of bonded joints in all three adhesives decreased with increasing temperatures over the range 100 to 120 °C. Within this temperature range there was no disruption to the chemical bonds of the adhesives, merely softening. At temperatures above 120 °C, a change occurred to the internal structure of the adhesive, and the chemical bonds of the adhesive were destroyed. The strength of all three adhesives stabilized at approximately 4 MPa.

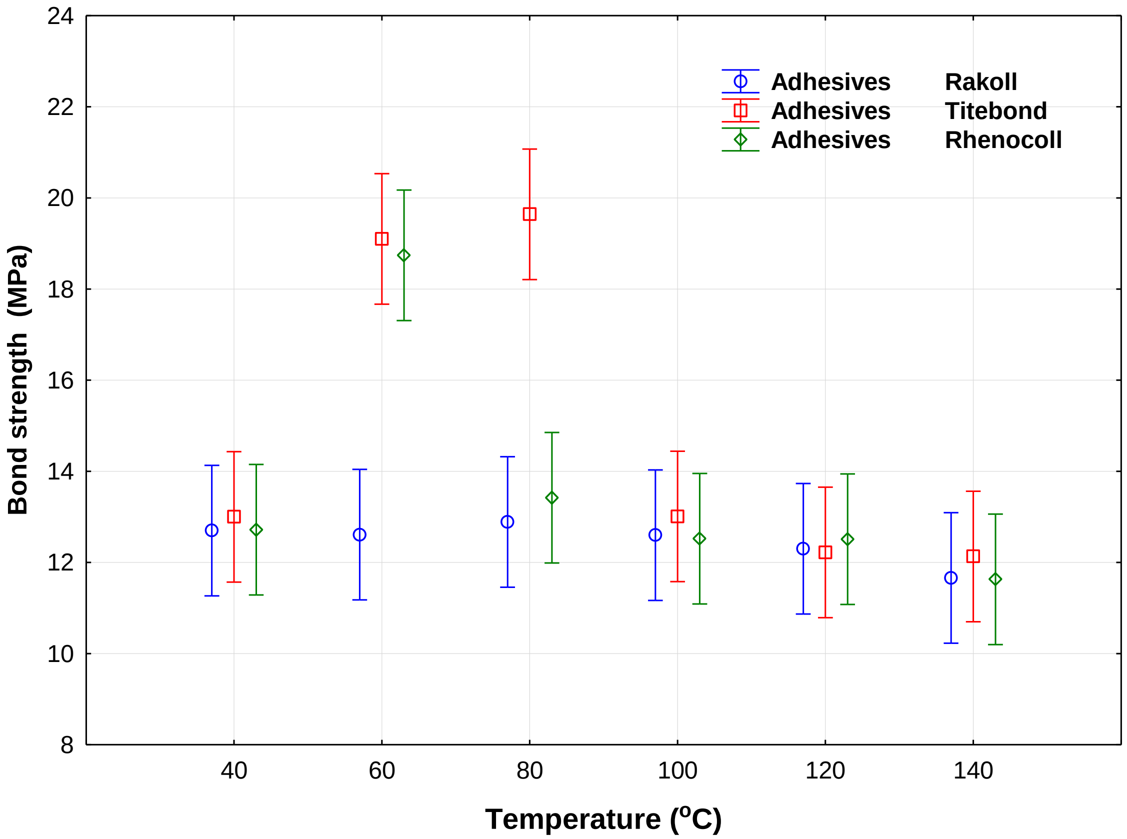


Fig. 4. Bond strength according to adhesives and specific wood temperature after 7 days, exposure to relevant temperature during 1 h and stored under normal conditions for 7 days. Data provided as the mean ± standard deviation

The results of the test, in which specimen temperature dropped to the ambient value, are shown in Fig. 4. In this case, an increase in joint strength (18.74 MPa) within the range 40 to 90 °C was obtained with Rhenocoll 3W, 4B Plus (18.74 MPa), and a significant increase ($p < 0.05$) in strength was obtained with Titebond II Premium up to 80 °C (19.64 MPa). When an adhesive-bonded joint is warmed, the macromolecules of the adhesive are able to better organize – they have more time to get out of the disordered condition – and after cooling, the joints may exhibit higher strength. In the course of further temperature increase above 100 °C, the corresponding reduction in joint strength was measured. After reaching 140 °C and above, the resulting strength value stabilized above 10 MPa. With Rakoll Express D3, the maximum strength (12.89 MPa) was obtained at 80 °C. Upon increasing the temperature to above 80 °C, a moderate decrease in strength occurred; however, this value is not statistically relevant. Up to 80 °C, the strength continued rising slightly; afterwards, it decreased slightly again as the temperature continued to increase.

The majority of cracks (defects) (about 95%) were cohesion fractures. The defects occurred in the adhesive material itself (mass), and traces of the adhesive on both surfaces of the substrate could be seen. The minority of cracks (defects) (about 5%) were adhesion fractures on both surfaces of the substrates. The defect occurred in the joints between the adhesive and the substrate. The adhesive was partially separated from both fastener substrate surfaces.

CONCLUSIONS

1. The results of the first bond-strength test according to adhesives and specific wood temperature after 7 days, and then exposure to the relevant temperature for 1 h, showed strength curves that are different from Fig. 1. The continuing curve of Rhenocoll 3W, 4B Plus was exponential, like that in Fig. 1. The curve obtained with Titebond II Premium corresponded to that in Fig. 3. A similar curve was obtained with Rakoll Express D3.
2. For Rhenocoll 3W, 4B Plus, the resulting joint strength values were similar to those shown in Fig. 3 at the start and end of the temperature range; however, 10 MPa, *i.e.*, the minimum value as specified in the relevant standard, was obtained at a significantly ($p < 0.05$) higher temperature (90 °C) compared with Fig. 1 (55 °C). The threshold value was exceeded at 78 °C for Titebond II Premium, compared with 60 °C for Rakoll Express D3.
3. Different strength curves were obtained for adhesives supplied by different manufactures. Contemporary adhesives obviously performed significantly ($p < 0.05$) better at higher temperatures. In general, the best results in this test were obtained with Rhenocoll 3W, 4B Plus; however, Titebond II Premium had higher strength at 20 °C and 140 °C. After exceeding 140 °C, the joint strength achieved with these two adhesives was 4 MPa, which is deemed a very good result.
4. The greatest differences were documented for Titebond II Premium, where joint strength decreased to 11.11 and 9.91 MPa at 60 to 80 °C, respectively, with stabilization at 19.10 and 19.64 MPa after cooling to ambient temperature. Generally, the best results after cooling to ambient temperature were obtained with Titebond II Premium. After cooling to ambient temperature, the minimum strength, *i.e.*, 10 MPa, was obtained with all tested adhesives.

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