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Bondingperformance ofheattreated wood with structural adhesives

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Abstract Bonding of untreated, intermediate (hydro-thermolysed) and heat treated wood with melamine-urea-formaldehyde (MUF), phenol-resorcinol-formaldehyde (PRF) and polyurethane (PUR) adhesives was studied. An industrial heat treatment process (Plato®) was used, which included two separate heat treatment stages and a drying stage inbetween. Laminated beams having four lamellas were prepared from untreated and treated timber for mechanical testing of the bond lines. The results of the tests showed that heat treatment affected the shear strength and the delamination of the laminated wood depending on the adhesive system used for bonding. The PUR and MUF adhesives performed in a rather similar way, and better than the PRF adhesive. The shear strength of laminated wood bonded

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with the waterborne MUF and PRF adhesives decreased for the specimens made of hydro-thermolysed timber and decreased further for the specimens made of fully heat treated timber. The difference in adhesive bond shear strength between untreated, intermediate and fully treated wood was less obvious in the case of the PUR adhesive. Delamination of the PRF bond line decreased drastically for all the specimens made of heat treated timber.

Verklebungsverhalten von warmebehandeltem Holz ¨

Zusammenfassung Die Verklebung von unbehandeltem, hydrothermisch behandeltem und wärmebehandeltem Holz mit Melamin-Harnstoff-Formaldehyd-Klebstoff (MUF), Phenol-Resorcin-Formaldehyd-Klebstoff (PRF) und Polyurethan-Klebstoff (PUR) wurde untersucht. Die Wärmebehandlung erfolgte nach dem Plato®-Verfahren, das aus zwei durch eine Trocknungsphase getrennte Wärmebehandlungszyklen besteht. Zur mechanischen Prüfung der Klebstofffugen wurde aus behandeltem und unbehandeltem Material Brettschichtholz mit vier Lamellen hergestellt. Die Ergebnisse zeigten, dass der Einfluss der Wärmebehandlung auf die Scherfestigkeit und die Delaminierung des Brettschichtholzes vom verwendeten Klebstoff abhängt. PUR und MUF ergaben ähnliche Werte, die besser waren als diejenigen von PRF. Die Scherfestigkeit von Brettschichtholz, das mit den Wasser basierten MUF- bzw. PRF-Klebstoffen verklebt worden war, nahm bei den hydrothermisch behandelten Proben und noch stärker bei den voll wärmebehandelten Proben ab. Bei PUR-Klebstoff war der Unterschied in der Scherfestigkeit zwischen unbehandelten, hydrothermisch behandelten und voll wärmebehandelten Proben weniger deutlich. Die Delaminierung der PRF-Klebstofffugen nahm bei allen wärmebehandelten Holzproben sehr stark zu.

1 Introduction

Heat treatment of wood is primarily used to increase the durability, reduce the hygroscopicity, and improve the dimensional stability of wood. Beside these desirable changes, heat treatment causes also some unfavourable effects such as diminished strength and toughness (Boonstra et al. 1998, Tjeerdsma et al. 1998a, Yildiz et al. 2006, Boonstra et al. 2007a). Changes in the properties of heat treated wood are associated with chemical and physical changes in the wood itself (Tjeerdsma et al. 1998b, Kamdem et al. 2002, Weiland and Guyonnet 2003, Tjeerdsma and Militz 2005, Boonstra and Tjeerdsma 2006, Nguila Inari et al. 2006, Nguila Inari et al. 2007, Paul et al. 2007). The degree and intensity of the reactions and/or modifications during heat treatment depend on the process conditions applied: the process type, the duration and the temperature of the heat treatment, and the nature of the wood itself (Nuopponen 2005).

Changes in the chemical, physical and structural properties of wood after heat treatment can affect the ability of adhesives to laminate the wood surface. The improved dimensional stability of heat treated wood generally improves the bonding performance, because the stresses due to shrinking or swelling on the cured adhesive bond are reduced. However, heat treatment can be expected to cause significant changes related to adhesion, which makes it necessary to adapt the bonding process. Strong adhesion between the adhesive and the wood is achieved by appropriate adhesive flow, penetration, wetting and curing. Heat treated wood is less hygroscopic (Boonstra et al. 1998, Paul et al. 2007), which can alter the distribution of the adhesive on the wood surface and the penetration of the adhesive into the porous wood structure. The intensity of water absorption from the waterborne adhesive could affect the hardening process of the adhesive and subsequently the quality of the adhesive bond. Several studies have shown that the wettability of wood with water is decreased after heat treatment (Pétrissans et al. 2003, Sernek et al. 2004, Follrich et al. 2006, Gérardin et al. 2007), mainly because the surface of the heat-treated wood is hydrophobic, less polar and significantly repellent to water. This might hinder waterborne adhesives from adequately wetting the surface.

The pH value of wood is another factor which could affect the bonding process, since the alkaline or acidic nature of the wood surface could interfere with the curing of the adhesive. Changes of the pH value of the wood surface might retard or accelerate the curing of adhesives, depending on the type of adhesive used for bonding. Heat treatment results in a decrease of pH (3.5–4) (Boonstra et al. 2007b), which probably affects the curing of adhesives. For instance, acetic and formic acids, present in wood after heat treatment, might neutralize the alkaline hardeners used for phenol-resorcinol-formaldehyde resins and hinder the adhesive hardening. On the other hand, a low pH of the wood surface could accelerate the chemical reactions of acid catalyzed amino resins: urea-formaldehyde and melamineformaldehyde (Pizzi 1983).

Several heat treatment processes are industrially available in Europe (Militz 2002). The Plato® process for thermal treatment of wood improves the dimensional stability and durability of wood while maintaining its mechanical properties. The Plato® technology involves five distinct process stages: (1) the pre-drying stage, (2) the hydrothermolysis stage, (3) the drying stage, (4) the curing stage, and (5) the conditioning stage (Boonstra et al. 2007a).

Heat treated wood, and Plato® Wood in particular, is mainly utilized in exterior applications, such as waterworks, garden furniture, fencing, claddings, window frames and doors. Heat treated wood might also have potential as a material for constructions, e.g. for use as structural elements in the building industry. For a number of construction products laminating is necessary, and exterior type wood adhesives, which can withstand long-term water soaking and drying, could be used to produce such products. The objective of this study was to evaluate the bonding performance of heat treated wood with three exterior structural adhesives. The adhesive bond strength and delamination were examined.

2 Materials and methods

2.1 Materials

Norway spruce (*Picea abies Karst*), Douglas fir (*Pseudotsuga menziessii* Franco), poplar (*Populus* species, I214), birch (*Betula pendula*) and alder (*Alnus glutinosa* Gaertn.) boards were used to study the influence of heat treatment on the bonding performance of small, lab-scale glued beams, which were bonded with three structural cold-setting adhesive systems (Table 1). The boards had a thickness of 25–40 mm and a width of 125–150 mm. The melamineurea-formaldehyde adhesive (MUF) was a honeymoon separate application type synthesized in the laboratory according to a procedure and formulation which has already been reported in detail (Properzi et al. 2001). Phenolresorcinol-formaldehyde (PRF) and polyurethane (PUR)

Table 1 Adhesive systems and mixing ratios Tabelle 1 Klebstoffsysteme und Mischungsverhältnisse

		Mixing ratio	
Label	Adhesive system	Resin	Hardener
MUF	Melamine-urea-formaldehyde	100	3
PRF	Phenol-resorcinol-formaldehyde	100	25
PUR	Polyurethane	100	

were commercial adhesives. Prior to heat treatment, the timber was kiln dried to a moisture content (MC) of 16% $(\pm 2\%)$ using a conventional drying process at 50–60 °C.

2.2 Heat treatment of wood (Plato® Wood)

Heat treatment was performed in two separate heat treatment stages, with a drying stage in-between. In the first stage of the heat treatment, the boards were treated in an aqueous environment at a super-atmospheric pressure (0.8–1.0 MPa). This is known as hydro-thermolysis treatment. The effective treatment temperature used was 165 ◦C for 30 minutes of effective treatment time. The timber was then dried to a MC of 8%–9% using a conventional drying process at 50–60 ◦C. After drying the timber was heat treated again in a special curing kiln for the second stage, now under dry and atmospheric conditions. This is known as "curing" treatment (at a temperature of 180 ◦C, with 5 hours of effective treatment time). During this stage, superheated steam was used as a sheltering gas to exclude oxygen.

2.3 Moisture content and pH determination

The native (untreated control) and heat treated boards (Table 2) were then cut into lamellas and conditioned in a standard climate with 65% relative humidity (RH) and at a temperature of 20 ◦C. The MC of the specimens was determined by the gravimetric method, whereas the pH value was evaluated by using extraction method -20 g of wood was ground into small particles and soaked in 160 g of distilled water for 24 hours. The extract was filtered and analyzed with a pH meter. The lamellas were planed to a thickness of 18 mm, cut to a rectangular shape (120 mm \times 500 mm), and hand sanded prior to bonding.

2.4 Hygroscopicity determination

Untreated, hydro-thermolysis treated, and fully heat treated Norway spruce specimens (with a thickness of 30 mm, a width of 150 mm, and a length of 10 mm) were prepared and conditioned at 65% RH before hygroscopicity testing. The specimens were then conditioned at 90% RH (above a saturated hydrated zinc sulphate solution at 20° C), 95% RH (above a saturated potassium nitrate solution at 20° C) and at 98% RH (above water at 20° C) until equilibrium moisture content (EMC) was reached. The specimens were then oven dried (24 hours at 103 ± 2 °C). The specimens were weighed before and after hygroscopicity testing. Three boards and three specimens per board per variable were used for this test.

2.5 Bonding of the wood

Four lamellas were bonded together into a small beam (Fig. 1). The adhesive was applied with a brush at an ap-

Table 2 Treatments and properties of wood prior to bonding **Tabelle 2** Behandlung und Eigenschaften des Holzes vor dem Verkleben

Abb. 1 Abmessungen der kleinen Brettschichtholzträger und Jahrringlage der Lamellen (*oben*); Form und Abmessungen der Prüfkörper (unten)

plication rate of $220 g/m^2$. Pressing was carried out for 90 minutes in a hydraulic press at room temperature ($22 \pm 2 \degree C$) and at a pressure of 1.0 MPa. In total, 42 beams were bonded (7 groups of wood, 3 adhesive systems, 2 duplicates) (Table 2).

2.6 Test methods

The shear test of the bond line $(n = 10)$ was performed according to the standard EN 392 (1995) on specimens with a nominal size of 35 mm \times 43 mm \times 72 mm, which were cut out of the beams (Fig. 1). The specimens were tested "dry" (conditioned in a standard climate) and "wet" (immersion in boiling water for 6 hours, cooling in water for 2 hours). The shear test was carried out in a ZWICK/Z100 universal testing machine. Delamination test of the bond line $(n = 12)$ was conducted according to the standard EN 391 (2001) on specimens with a nominal size of $75 \text{ mm} \times 95 \text{ mm} \times 72 \text{ mm}$ (Fig. 1). The delamination test cycle used method B, subsection 6.4.3.

3 Results and discussion

of the MUF adhesive

Holzbruchanteil und Delaminierung der

3.1 Bonding performance of MUF adhesive

The shear strength, percentage of wood failure and delamination of the specimens bonded with the MUF adhesive are shown in Table 3. After the dry shear test, the percentage of wood failure was always 100%, except for heat treated Douglas fir (PDF) which was a bit lower (93%), but still very high. This indicated that the bonding performance of all specimens was good, as was expected for this particular type of MUF adhesive (Properzi et al. 2001). The differences in shear strength between the wood species tested are due to the inherent shear strength of each wood species and the effect of heat treatment which might differ for each wood species (Boonstra et al. 2007a). Among the Norway spruce specimens, the untreated specimens (NS) showed the highest shear strength (6.28 MPa), followed by the hydrothermolysed intermediate specimens (HTNS) with a shear strength of 5.70 MPa (−9%) and the fully heat treated specimens (PNS), where the shear strength dropped to 4.86 MPa (−23%). This is an indication that heat treatment decreases the shear strength of wood itself, especially because the percentage of wood failure was 100% after the dry test.

The shear strength of the specimens after the wet test (immersion in boiling water for 6 hours, cooling in water for 2 hours) dropped drastically compared to the dry specimens, whereas the percentage of wood failure of most of the (heat treated) wood species remained high (except for the heat treated Douglas fir and alder). This is an indication that the decrease in shear strength was mainly the result of weaker wood tissue due to water boiling/soaking, and not, or at least not entirely, due to degradation of the MUF adhesive after exposure to boiling water. The reduction in the shear strength of heat treated Douglas fir and alder could be due to weaker wood tissue, but the wood-adhesive interaction could also be involved since the wood failure percentage is rather low, especially for the heat treated alder. The decrease in the shear strength of hydro-thermolysed and fully treated Norway spruce (HTNS and PNS) after the wet

test, as compared to untreated Norway spruce (NS), is remarkable. A reduction of 22% and 58%, respectively, was observed, which was much higher than after the dry test. Since the wood failure percentage is quite high it must be the wood tissue which is weakened more in the case of heat treated than in the case of untreated Norway spruce after the wet test.

In terms of performance requirements for glued laminated timber (EN 386 2001), which takes into account only the dry test, the shear strength of each glue line must be at least 6.0 MPa. For coniferous wood and poplar, a shear strength of 4.0 MPa (for each glue line) is acceptable if the wood failure is 100%. This means that untreated Norway spruce, hydro-thermolysed Norway spruce, and heat treated poplar bonded with MUF adhesive fulfilled the requirements, whereas the others did not.

Heat treatment appears to affect delamination since heat treated Norway spruce showed 6.9% delamination after the test cycle, whereas no delamination was observed in the case of untreated Norway spruce. The high delamination percentage of heat treated Douglas fir is remarkable, and is again a confirmation of a low quality MUF adhesive bond. With respect to the requirements, untreated Norway spruce, hydro-thermolysed Norway spruce, and heat treated poplar showed a total delamination of less than 4%, which is stated as the upper limit (EN 386).

3.2 Bonding performance of PRF adhesive

In general, the bonding performance of heat treated wood with PRF adhesive was less satisfactory than that of similar wood with MUF adhesive (Table 4). The shear strength and percentage of wood failure of fully treated Norway spruce (dry and wet) bonded with PRF adhesive is significantly lower than the specimens bonded with MUF adhesive. This difference is not observed, or observed to a lesser extent, in the case of untreated and hydro-thermolysed Norway spruce. The heat-treated Douglas fir, poplar and alder specimens which were bonded with PRF adhesive showed a clear decrease in shear strength and wood failure compared to the specimens bonded with MUF adhesive (especially the heat treated Douglas fir specimens). A positive exception is the heat treated birch, which performed better in the case of the PRF bonded specimens, although the total delamination is higher.

Generally, PRF adhesives are structural wood adhesives with high performance for exterior use. The curing reaction of PRF adhesives usually proceeds in an alkaline environment and is susceptible to the low pH of the wood species (Pizzi 1983), like phenol-formaldehyde adhesives (Sernek et al. 2004). It can therefore be deduced that the low shear strength of specimens bonded with PRF adhesive was due to low pH values of the wood (Table 2), which slows down considerably the hardening reaction of PRF. For instance, the shear strength of the wet specimens bonded with PRF adhesive decreased with the decreasing pH values of untreated Norway spruce, hydro-thermolysed Norway spruce, and fully treated Norway spruce. The delamination of Douglas fir, which had the lowest pH value (3.6) among the studied wood species, was very high indeed (82.4%). Heat treatment reduces the pH of wood to 3.5–4 due to the production of acetic acid and formic

Table 4 Shear strength, amount of wood failure, and delamination of the PRF adhesive bond **Tabelle 4** Scherfestigkeit, Holzbruchanteil und Delaminierung der PRF-verklebten Proben

acid, whereas a pH of around 5.0 is common for untreated wood (Boonstra et al. 2007b). An alteration in the PRF composition in either the alkali content or pH might improve the bonding performance of heat treated wood. For instance, the addition of an alkaline buffer to the PRF adhesive can neutralize the acids present in the heat treated wood.

3.3 Bonding performance of PUR adhesive

Bonding with PUR adhesive resulted in high shear strength of the dry specimens, ranging from 5.51 MPa for fully treated Norway spruce to 7.77 MPa for heat treated birch (Table 5). Wood failure was 100% for all specimens, except for the heat treated birch, which was 90%. The shear strength of the specimens showed a decrease of about 50% after the wet test. The variation in shear strength between the different wood species was limited after the wet test, whereas the percentage of wood failure varied from 7% for heat treated birch to 100% for heat treated poplar. The total percentage of delamination was quite high for heat treated birch (41%), and significantly higher than for the other wood species.

All wood species bonded with PUR adhesives fulfil the minimum required value for shear strength of the adhesive bond, except heat treated birch and alder. In terms of delamination, untreated and fully treated Norway spruce, heat treated poplar, and heat treated alder fulfil the requirement, whereas hydro-thermolysed Norway spruce, heat treated Douglas fir and heat treated birch do not. Onecomponent PUR adhesives need moisture or water for their hardening processes. Since the MC of heat treated wood was rather low this could have an affect on the bonding process, and hence on the bonding performance.

3.4 The influence of heat treatment on the bonding performance of Norway spruce

In the case of Norway spruce, it was possible to examine the effect of intermediate and full heat treatment on the bonding performance. The results showed that this effect depended on the adhesive system used (Fig. 2). For waterborne MUF and PRF adhesives, the shear strength of the Norway spruce specimens decreased after the hydrothermolysis stage (THNS), and then further after the curing stage (PNS). The effect was more pronounced in the case of wet specimens than in the case of dry specimens. Heat treatment did not significantly affect the shear strength of the specimens bonded with PUR adhesive, which is not a waterborne adhesive. These results indicated that the hydrophobic character of heat treated wood could diminish the ability of waterborne adhesives (MUF and PRF) to adequately wet the surface and establish physical adhesion. The surface of heat treated wood is less polar and thus repels water, resulting in a lower wettability than in the case of untreated wood (Gérardin et al. 2007). Additionally, a low MC of 6.9% in the case of the heat treated Norway spruce specimens (PNS) could decrease surface wettability. Differences in bonding performance between hydro-thermolysed and fully heat treated Norway spruce, as observed in this study, were expected since fully heat treated Norway spruce is more hydrophobic than the hydro-thermolysed intermedi-

Tabelle 5

Fig. 2 Influence of heat treatment on the bonding performance of Norway spruce (abbreviations see Table 2) **Abb. 2** Einfluss der Wärmebehandlung auf das Verklebungsverhalten von Fichtenholz (Abkürzungen siehe Tabelle 2)

Table 6 Differences in equilibrium moisture content (EMC) of untreated, hydro-thermolysed and fully heat treated Norway spruce **Tabelle 6** Gleichgewichtsfeuchte von unbehandelten, hydrothermisch behandelten und voll wärmebehandelten Fichtenproben

ate product (Boonstra et al. 2007a), which affects the bonding process especially of waterborne adhesives. Heat treated Norway spruce was also less hygroscopic (lower EMC) than hydro-thermolysed or untreated Norway spruce, respectively (Table 6). This could alter the distribution of the adhesive on the wood surface and the penetration of the adhesive into the porous wood structure. The intensity of water absorption from MUF and PRF adhesives could affect the hardening process and subsequently the quality of the adhesive bond.

Among the three structural adhesives tested, PRF performed the worst when used for bonding heat treated Norway spruce. This cannot be ascribed to the PRF adhesive. PRF adhesives are excellent for exterior use, as noticeable from the good results in bonding the untreated control specimens. The likely causes of the poor adhesion noticed in the heat treated Norway spruce joints bonded with the PRF adhesive are: (i) insufficient wetting hindering specific adhesion and (ii) the low pH of the substrate, which slows down the hardening.

4 Conclusions

This study revealed that the shear strength and delamination of bonded specimens of heat treated wood depended on the adhesive system used for bonding. In most cases, the PUR and MUF adhesives performed similarly and better than the PRF adhesive. In terms of the requirements for glued laminated timber (shear strength and delamination), PUR adhesive meets the requirements for bonding of Norway spruce (untreated and fully heat treated), and for heat treated poplar; MUF adhesive bonding was satisfactory in the case of untreated and hydro-thermolysis treated (intermediate) Norway spruce, and in the case of heat treated poplar; whereas only untreated and hydro-thermolysis treated Norway spruce specimens bonded with PRF adhesive fulfilled the requirements.

In the case of Norway spruce, the shear strength of the specimens of the waterborne MUF and PRF adhesives decreased with the degree of heat treatment (half or fully treated), but this did not affect the shear strength of the PUR adhesive bond. Delamination of the PRF bond line decreased drastically in the case of all the heat treated specimens, whereas in the case of the MUF and PUR adhesives delamination was very high for the PDF and PB specimens, respectively. The following concise conclusions can be drawn from the results of the study:

- Plato[®] heat treated wood can be bonded with structural adhesives.
- MUF and PUR adhesives perform better than PRF adhesive, although a definite conclusion regarding the latter can only be reached once its pH has changed.
- Untreated wood (Norway spruce) appears to perform better than heat treated wood, especially in the case of waterborne adhesives. The low pH (PRF) and the low wettability (PRF and MUF) of the heat treated wood are thought to be the main reason for this difference.
- More research needs to be performed regarding the potential improvement of the bonding performance of heat treated wood, e.g. alterations in the adhesive composition and/or the bonding processes.

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