

Quality assessment of glued ash wood for construction engineering

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Received: 26 February 2015 / Published online: 30 October 2015
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Abstract Delamination resistance and tensile shear strength (TSS) are essential for load-bearing timber structures. Thus these two factors were investigated on industrially bonded ash wood (*Fraxinus excelsior* L.) to check for the suitability of adhesively bonded ash wood as a building material. Two melamine urea formaldehyde (MUF) resins, two polyurethanes (PUR), one emulsion polymer isocyanate and one phenol resorcinol formaldehyde resin were taken for bonding. Face milled and planed surface series were made to highlight potential differences. For PUR, an additional series with dimethylformamide primed surfaces was also made. The influence of the mixing ratio and the closed assembly time were analysed for one MUF system. The samples for the TSS were tested in dry and wet conditions. 80 % of the tested series met the standard requirements (EN 15425; EN 301) in dry condition, whereas only 30% passed in wet condition. None of the adhesives tested were able to pass the delamination test. No distinct influence of the different parameters studied is notable for most of the adhesive systems, only extended CATs and lower MRs seem to improve the bond quality of MUF. In addition, chemical analyses were performed to

find evidence for the poor bonding performance. It was found that acidic extractives, fatty acid content and pH of ash fell within the range of beech and spruce wood, with only formic acid being an exception with an amount four times higher than the other two wood species.

1 Introduction

The increase in hardwood harvest in Europe, particularly in Switzerland, is reasonable ground for devising further applications of these species in construction engineering (Krackler et al. 2011). The hardwood reserves in the forests are accumulating (Brändli 2010) and thus another economic use besides their energetic utilisation is desirable. The common hardwoods have better strength properties than the more utilised spruce wood and thus have good potential in the construction sector. However, wooden construction designs usually need the wood to be adhesively bonded, e.g. as glued laminated timber (glulam) or laminated veneer lumber (LVL), to achieve the required load bearing capacity. Technical standards define certain minimum requirements for such glue joints to guarantee safety and quality. Softwoods have been used in glued timber constructions now for more than 100 years. Thus their potential and applications are already well known. One consequence however is that adhesive systems are often designed for use with softwoods and consequently technical standards focus on their application. Hardwood applications in contrast are relatively young. Their feasibility has to be verified and their potential and problems have to be fathomed first.

The quality of glue joints is typically evaluated with tensile shear tests (EN 302-1 2013b) and delamination tests (EN 302-2 2013c). The second test in particular is a major

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hurdle for hardwood elements. It is basically a stress test for the joint by maxing out the swelling and shrinkage coefficients of the adherends, involving cycles of soaking in water with high pressure and vacuum, followed by drying in an air current at a raised temperature. Hardwoods have, in comparison with softwoods, higher swelling coefficients, higher Young's moduli and higher strengths, which altogether sum up to higher internal stresses when changing moisture. Thereby a failure at the joint is more likely in hard- than in softwood elements. Only recently Schmidt et al. (2010) were able to show that glulam elements made of beech wood can pass the delamination test according to EN 302-2 (2013c), following specific bonding parameters. Their findings however also show how difficult it is to pass this test with bonded hardwood. A similar study for ash wood glulam was done by Knorz et al. (2014), but they could not find any adhesive technology that would fulfil the minimum requirements of relevant standards (EN 301 2013a; EN 15425 2008).

In this study the influence of surface preparation, priming, mixing ratio (MR) and closed assembly time (CAT) on the glue joint performance of industrially bonded ash wood was investigated. Chemical analyses were conducted to determine the acidic wood extractives and fatty acids of beech, ash and spruce samples to identify potential causes for the different bonding performances. This research thus provides further information and contributes to realising a future solution for bonding hardwoods.

2 Materials and methods

2.1 Materials

The ash wood (*Fraxinus excelsior* L.) used for all experiments was sorted beforehand by density and ultrasonic measurement. The wood had a raw density of 650 ± 25 kg/m³ and a moisture content of 11.8 ± 0.7 %. Prior to bonding, the wood surfaces were either planed (P) or face milled (FM). Both wood sorting and surface preparation were conducted on the same machines for all experiments. Six adhesive systems of four different adhesive types were used for bonding, namely two melamine urea formaldehyde (MUF) resins, one phenol resorcinol formaldehyde (PRF) resin, one emulsion polymer isocyanate (EPI) and two polyurethanes (PUR). The adhesive systems MUF1, MUF2, PRF and EPI were provided by Dynea AS, the PUR1 and PUR2 by Jowat AG. All bonding parameters were strictly adopted from the manufacturers' guidelines. For the MUF1 system the bonding parameters CAT and MR were varied: CATs of 10, 20, and 30 min, and MRs with 100, 60 and 35 m% hardener admixture were

processed (later designated as e.g. 35 % MR). Both PUR systems were supplemented by a further surface treatment, where 40 g/m² of pure (99.8 %) dimethylformamide (DMF, CAS No. 68-12-2) was applied to the planed surfaces as primer. DMF was found by Kläusler et al. (2014) to be a suitable primer to improve the bond quality of PUR in combination with beech wood.

2.2 Tensile shear tests

The samples for measuring the tensile shear strength (TSS) were produced as required by the standard EN 302-1 (2013b). All the above-mentioned specimen types were investigated, and the MUF1 series were complemented with an additional CAT of 40 min. The final specimens were split into two groups and each of them subjected to either A1 or A4 treatment according to EN 302-1 (2013b). No further treatment was conducted on the A1 group besides storing at standard atmosphere (20 °C, 65 % relative humidity) until testing. However, the A4 group specimens were immersed in boiling water for 6 h, followed by immersion in cold water for 2 h and tested immediately afterwards, i.e. in wet condition.

All experiments were conducted on a Zwick/Roell Z010 universal testing machine with a 10 kN load cell mounted. The machine was operated displacement-controlled at constant 0.8 mm/min. This displacement rate was suitable for both the A1 and A4 series, since all specimens failed within 30–90 s, as specified in EN 302-1 (2013b). The percentage of wood failure (WF) was recorded directly after mechanical testing.

2.3 Delamination tests

The specimens for the delamination test were produced in an industrial glulam beam plant using the same materials and parameters mentioned above. Ash glulam elements consisting of 6 lamellae each 30 mm thick (mainly flat grain) and roughly 5 m in length were produced to cut out the final specimens according to EN 302-2 (2013c). The beams were bonded with MUF1, PUR1, PUR2 and EPI. PRF and MUF2 had to be omitted due to limitations of the glulam beam plant. The MUF1 with P surface were also not feasible, however the FM series with all variations of MR and CAT were possible.

The test procedure was adopted from EN (2013c) (adhesive type I), i.e. soaking under cycles of vacuum and high pressure, followed by drying at increased temperature (65 °C). The specimens underwent, in total, three such cycles of soaking and drying. These experiments were performed at two different laboratories to verify the reproducibility of the results. Both laboratories used therefore the same testing parameters.

2.4 Chemical analyses

Chemical analyses were carried out to identify potential causes for the variability in bonding quality between different wood species. Therefore, besides ash wood, also beech (*Fagus sylvatica* L.) and spruce (*Picea abies* Karst.) wood were taken into account to determine the three following properties:

- content of acidic extractives,
- content of fatty acids, and
- pH value.

Prior to the extraction the wood samples were milled using a Retsch mill equipped with a 1 mm sieve.

For the determination of the content of acidic extractives 10 ml of 0.025 m NaOH were added to 1 g of the milled wood sample, treated in an ultrasonic bath for 60 min, and subsequently shaken for approximately 12 h. The mixture was then filtered to obtain a clear extract. The determination of acetic acid and formic acid was carried out using an Ion chromatograph Metrohm 761 Compact IC with Metrohm 813 Compact Autosampler Programm: 761 PC Software 1.1, Metrosep Organic Acids—250 column, Eluent: sulphuric acid (0.5 mmol/l)/acetone (15 %), temperature: 23 ± 3 °C.

The fatty acid content was determined according to DIN 55957 (2000). This method focuses on the separation of fatty acids by the number of carbon atoms (preferably 14–18) and by the number of double bonds (up to three). The procedure includes addition of 70 ml of 0.5 m NaOH in methanol to 8 g of milled wood, 2 h treatment in an ultrasonic bath and shaking for approx. 12 h. After filtration, 20 ml of the extract were boiled under reflux for 1 min. Subsequently, 10 ml of 14 % boron trifluoride solution in methanol was added and the mixture was boiled for another 2 min whereupon 10 ml of heptane were admixed and the solution was boiled for 1 min. The solution was then cooled down and salted out by addition of saturated sodium chloride solution and shaking. Finally, the upper organic phase was separated and dried over sodium sulphate. The determination of the fatty acids' composition was determined by gas-chromatography using a RTX-65 30 m \times 0.25 mm \times 0.50 μ m column.

The pH-measurement was carried out after cold water extraction of milled wood with deionized water using a wood-to-water ratio of 1:10. The extraction procedure is similar to that of the determination of acidic extractives. Determination of the surface pH was carried out on wood board sections using a glass electrode with a flat tip. The pH value was measured at two sites per wood species after 2 min of wetting of the wood surface with 0.4 ml of deionized water for each site.

3 Results and discussion

3.1 Tensile shear tests

In Table 1 all results for the TSS under A1 conditions are given. The results for the A4 conditions are shown in Table 2. Besides the mean TSS, WF, and number of specimens n , the coefficient of variation (CV) and the confidence interval (CI) are also illustrated. For the CI a level of confidence of 95 % was used. The statistical evaluation of the gained data was done according to DIN 53804-1 (2002). The data are also illustrated in condensed form in Fig. 1 for a better overview. Note that the box plots show the mean value instead of the median, since the mean value is relevant for fulfilling standard requirements.

To pass the standards EN 301 (2013a) and EN 15425 2008, the TSS has to be, on average, above 10 MPa for A1 and 6 MPa for A4 testing. These thresholds are actually for beech wood, however, since beech and ash wood have similar strength properties, these thresholds were simply adopted here as quality measure. Approximately 20 % of the series failed to pass at A1, and 70 % failed at A4. Significant differences are present between the MUF1 variants, but no clear trend is notable. For the 100 % MR, the lowest TSSs are measured at 20 min CAT, but both longer and shorter CATs produce significantly higher TSSs. A similar observation can be made for 60 % MR, but not for 35 %.

The surface preparation shows no influence on MUF1. The TSSs for planed or face milled surfaces are statistically identical, with only two exceptions: the 60 and 35 % MR both show, in combination with a 30 min CAT and FM surface, a fatal drop in TSS below the 10 MPa hurdle, i.e. they do not fulfil the EN 301 (2013a) requirements, but at the same time still show 100 % WF. Additionally MUF1 with 100 % MR, 20 min CAT and FM does not fulfil these requirements (again with 100 % WF), but the same P variant does. The TSS FM is 9.86 MPa, the TSS P is 10.16 MPa, but with no statistical difference. The MUF2, PRF and EPI series show similar behaviour to MUF1, with TSS between 11 and 12 MPa.

The PUR specimens also show mainly wood failure in dry conditions, but not as much as the other adhesive systems. The surface preparations also have a stronger influence. Both PUR1 and PUR2 do not pass the 10 MPa in combination with planed and DMF primed surfaces. The PUR1 also fails the test without the primer, but the FM version does not, with a significantly higher TSS. For the PUR2 it is just the opposite, the P surface results in a significantly higher TSS than the FM surface; however, both are above 10 MPa. DMF as primer for PUR glue

Table 1 Results for TSS under A1 conditions

Adh.	MR (%)	CAT (min)	Surface	TSS (MPa)	CV (%)	CI (MPa)	n (–)	WF (%)
MUF1	100	10	P	12.33	15	0.86	20	98
			FM	11.58	8	0.44	20	100
		20	P	10.16	7	0.36	17	94
			FM	9.86	11	0.52	20	100
		30	P	11.43	14	0.76	19	94
			FM	10.08	8	0.44	20	100
	60	10	P	12.32	5	0.30	16	100
			FM	12.33	10	0.58	20	100
		20	P	10.75	12	0.59	20	98
			FM	11.23	12	0.63	20	100
		30	P	12.52	9	0.53	20	91
			FM	9.83	8	0.43	15	100
	35	10	P	11.51	10	0.54	20	100
			FM	12.51	13	0.89	15	99
		20	P	12.64	19	0.71	48	74
			FM	11.87	10	0.67	15	100
		30	P	11.80	11	0.36	49	81
			FM	9.66	10	0.55	15	100
40	P	12.35	18	0.83	30	54		
	FM	12.91	8	0.51	18	98		
MUF2	25	40	P	10.88	9	0.56	15	100
			FM	12.91	8	0.51	18	98
PUR1	–	–	P	9.85	17	0.79	20	94
			P + DMF	8.92	1	0.42	19	78
			FM	11.44	13	0.72	20	90
PUR2	–	–	P	12.65	6	0.35	19	97
			P + DMF	9.72	16	0.71	20	85
			FM	10.83	15	0.76	20	70
PRF	20	30	P	11.49	11	0.57	20	100
			FM	11.19	15	0.78	20	100
EPI	15	15	P	11.54	12	0.66	20	100
			FM	11.95	10	0.53	20	61

joints in ash wood thus seems not to have any advantage, unlike its application in beech wood joints (Kläusler et al. 2014). A recently released commercial primer might be an alternative (Luedtke et al. 2015), or also hydroxymethylated resorcinol (López-Suevos and Richter 2009; Vick et al. 1995) might increase the bond quality in ash wood. This, however, would have to be covered in further research.

Under wet conditions (A4) most series cannot pass the demanded 6 MPa hurdle. Again the CAT and surface preparation have no clear influence on the TSS of MUF1, but lower MRs seem to promote higher TSSs. With 100 % MR only one variant was able to pass; with 60 % MR two, and with 35 % four variants passed. However, none of the MUF2, EPI or PUR series passed the test. Only the PRF series achieved both TSSs above 6 MPa. Thus PRF is the only adhesive system that reliably fulfils the EN 301

(2013a) criteria regarding TSS, independent of surface preparation. Six of the 19 parametric setups for the MUF1 also fulfil these requirements, but no distinct trend is notable. Only lowering the MR for MUF1 seems to slightly improve the TSS for A1 and A4 conditions.

A correlation between WF and TSS can only be found for the PUR systems, i.e. more WF corresponds to higher TSS. The other adhesive systems do not exhibit a similar behaviour. The lowest WF percentage under dry conditions is, for example, found in one MUF1 series with TSS above 12 MPa, but other series with 100 % WF are below 10 MPa. Thus, WF may not be a suitable measure for the quality of glue joints, as already stated by many authors before (Kläusler et al. 2014; Knorz et al. 2014; Ohnesorge et al. 2010). However, under A4 conditions FM surfaces tend to produce more WF compared to P. This finding is concordant with Knorz et al. (2015).

Table 2 Results for TSS under A4 conditions

Adh.	MR (%)	CAT (min)	Surface	TSS (MPa)	CV (%)	CI (MPa)	n (–)	WF (%)
MUF1	100	10	P	4.73	37	0.82	20	37
			FM	5.51	6	0.15	19	6
		20	P	5.49	15	0.39	20	15
			FM	5.42	8	0.20	19	8
		30	P	5.70	15	0.59	11	15
			FM	6.01	7	0.19	20	7
	60	10	P	5.68	18	0.48	20	18
			FM	5.83	12	0.36	18	12
		20	P	6.15	6	0.18	19	6
			FM	5.36	5	0.15	17	5
		30	P	6.36	9	0.28	17	9
			FM	5.79	6	0.19	15	6
	35	10	P	5.64	17	0.45	20	17
			FM	7.11	12	0.47	15	12
		20	P	5.56	20	0.37	39	20
			FM	6.66	5	0.18	15	5
		30	P	6.36	16	0.33	40	16
			FM	6.60	14	0.51	15	14
40	P	5.29	19	0.39	29	19		
	FM	5.29	19	0.39	29	19		
MUF2	25	40	P	5.90	6	0.21	14	6
			FM	5.88	11	0.31	20	11
PUR1	–	–	P	4.24	23	0.45	20	23
			P + DMF	4.55	38	0.80	20	38
			FM	5.47	11	0.27	20	11
PUR2	–	–	P	5.01	13	0.31	20	13
			P + DMF	5.40	23	0.61	19	23
			FM	4.76	13	0.28	20	13
PRF	20	30	P	7.44	10	0.36	20	10
			FM	6.35	4	0.14	13	4
EPI	15	15	P	5.20	25	0.63	19	25
			FM	4.41	9	0.19	19	9

3.2 Delamination tests

Table 3 and Fig. 2 show the delamination test results. In Fig. 2 the results are shown separately for the two laboratories. The differences between the laboratories are generally small and, with regard to the requirements, come to the same conclusions.

The lowest average delamination was recorded at 28 % for the PUR2 with planed and primed surface. This value however is still far beyond the demanded 5 % (EN 15425 2008). Therefore, none of the adhesive systems tested was able to meet the requirements of the standard. Most series failed with more than 60 % delamination.

Besides these high values, two clear tendencies for MUF1 are remarkable. Firstly, the delamination decreases with decreasing MR and, secondly, the delamination decreases when extending the CAT. These tendencies are

also present for the TSS, but less distinctive than for the delamination.

The findings here are in general concordant with Knorz et al. (2014). They also found that increasing the CAT can improve the delamination resistance of MUF glue joints. However, contrary to the investigations here, they found a positive trend when increasing the MR of MUF. This divergence however might be due to the different MUF systems used.

3.3 Chemical analyses

Table 4 shows the results of the determination of acidic extractives of the investigated wood samples. Beech wood contains the lowest concentration of acidic components. Consequently, its pH value is the highest compared to that of ash and spruce wood. The acid content, as well as the pH

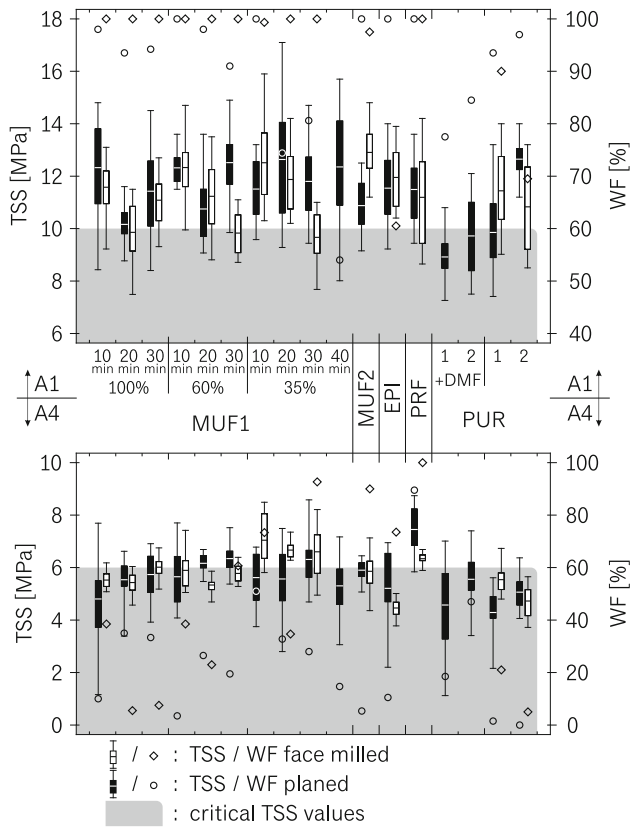


Fig. 1 Results from the TSS tests. *Top* A1, *bottom* A4 condition. Note that *box plots* show mean values instead of median

value of ash wood, fall within those of beech and spruce. However, a remarkable difference regarding the distribution of acetic and formic acid concentrations among the

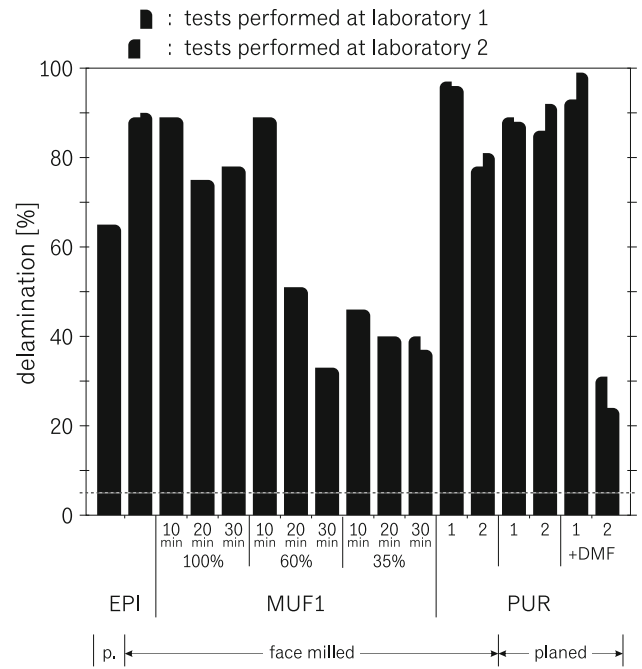


Fig. 2 Delamination test results measured at two different laboratories. Note that 5 % is the threshold

three wood species can be observed. Although spruce wood contains the highest amount of acidic components, ash wood contains the highest amount of formic acid. It exceeds the formic acid concentration of the other wood samples by a factor of 4. The results of the surface pH measurements correspond with those of the acidic extractives.

Table 3 Results from the delamination tests, showing the percentage of delaminated joints with CV

Adh.	MR (%)	CAT (min)	Surface	Delam. (%)	CV (%)	n (–)
MUF1	100	10	FM	89	8	6
		20	FM	75	15	6
		30	FM	78	8	6
	60	10	FM	89	7	6
		20	FM	51	29	6
		30	FM	33	29	6
	35	10	FM	46	23	6
		20	FM	40	9	12
		30	FM	38	25	12
PUR1	–	–	P	88	8	12
		–	P + DMF	96	4	12
		–	FM	97	3	12
PUR2	–	–	P	89	6	12
		–	P + DMF	28	19	12
		–	FM	79	7	12
EPI	15	<20	P	51	29	6
			FM	89	6	12

Table 4 Results of the acidic components' content as well as the pH values of the extractives and surfaces

	Ash	Beech	Spruce
Acetic acid (mg/kg)	227	114	423
Formic acid (mg/kg)	51	10	13
Σ (mg/kg)	277	124	436
Extractives pH (–)	5.18	5.39	4.74
Surface pH (–)	5.26	5.40	4.97

Table 5 Content of saturated and unsaturated fatty acids in the three wood species

	Ash	Beech	Spruce
Saturated fatty acids (mg/kg)	88	88	192
Unsaturated fatty acids (mg/kg)	517	81	499
Σ (mg/kg)	606	169	691
Oleic acid (mg/kg)	80	15	113
Trans-9-elaidic acid (mg/kg)	103	0	62
Linoleic acid (mg/kg)	303	66	235
Linolelaidic acid (mg/kg)	0	0	82
Linolenic acid (mg/kg)	23	0	0

The five most substantial unsaturated fatty acids are listed separately

During storage of solid wood, fatty acids can migrate from inner wood regions to the surface. They might decrease or obstruct the wood-adhesive interaction by forming thin non-polar films that interfere with the physical or chemical interaction of both components, e.g. formation of hydrogen bonds or, in the case of isocyanates, of polyurea or polyurethane bonds. It was therefore important to determine the content of fatty acids within the wood samples. The results are compiled in Table 5.

The results show that spruce wood contains the highest amount of fatty acids, whereas in beech wood far less of these substances were found. However, ash and spruce have comparable contents of unsaturated fatty acids. The most abundant among them are listed separately in Table 5.

Strong or medium strong acids may act as catalysts for the reaction of isocyanates with wood surfaces. Therefore, the distinctly higher concentration of acetic acids and the resulting lower pH value can serve as an explanation for the generally good adhesion performance of spruce wood. Fatty acids in concentrations found in this study obviously do not interfere with the formation of adhesive bonds.

Other structural prerequisites such as surface porosity or accessibility of OH groups of lignin or carbohydrates at the wood surface may affect the gluability of different wood species even more. Since isocyanate groups of the adhesive prepolymer require the presence of OH groups to form urethane or urea bonds, the wood surface has to provide

such groups. Recent investigations into the hydroxyl content of different wood species or their polymer constituents show that hardwood species such as beech or ash wood contain lower concentrations of OH group than softwood species. This is the result of a significantly higher content of partly acetylated xylan-based hemicelluloses (Gawron et al. 2014; Ucar and Ucar 2008) in hardwood with up to 70 % of C-2 and C-3 positions being acetylated, as well as the higher number of methoxyl groups in hardwood lignins due to a higher concentration of syringyl moieties (Lai and Guo 1991; Mansouri et al. 2011). Here, it was shown that the OH-content of lignins decreases with increasing methoxyl content.

Therefore, isocyanates may encounter less reactive groups on hardwood surfaces compared to softwood species. Surface treatment with wood swelling fluids, such as polar solvents, obviously improve accessibility of the OH groups by increasing the specific wood surface and subsequently improving the gluing quality.

4 Conclusion

The results obtained within this study allow drawing the following conclusions:

- None of the tested adhesive systems was able to meet the requirements of EN 15425 2008 nor EN 301 (2013a) respectively.
- The PRF was the only adhesive system that reliably achieved the stipulated TSS.
- The bonding performance of MUF1 profits from low MRs and long CATs.
- WF and TSS correlate only for PUR adhesives.
- No explicit trends were identifiable for PUR, EPI, PRF or MUF2 regarding TSS or delamination.
- In the conducted chemical analyses, values for ash wood mostly lie within the range of beech and spruce wood values. The only exception is the formic acid content, being four times higher than for the other species.

Acknowledgments This research was carried out at ETH Zurich and funded through the National Research Programme NRP 66 of the Swiss National Science Foundation and the forest and timber research fund of the Swiss Federal Office for the Environment FOEN.

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