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Fire performance and decay resistance of solid wood and plywood treated with quaternary ammonia compounds and common fire retardants

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Abstract In this study, the fire performance and decay resistance of solid wood and plywood treated with quaternary ammonia compounds (didecyl dimethyl ammonium chloride (DDAC) and didecyl dimethyl ammonium tetrafluoroborate (DBF)) were compared with the performance of untreated control specimens and specimens treated with common fire retardants ((monoammonium phosphate (MAP), diammonium phosphate (DAP) and ammonium sulphate (AS)). Test specimens were treated with 1% and 4% (% m/v) aqueous solutions of the chemicals. The fire performance tests were the fire tube test (ASTM E 69) which measures mass losses in the specimens and the cone calorimeter test (ASTM E 1354) which measures mass loss, heat release rate, time for sustained ignition, effective heat of combustion, and specific extinction area. The results from the cone calorimeter tests were used to estimate the flame spread index (FSI) in the Steiner tunnel test (ASTM E 84). Heat release rates of the specimens treated with MAP, DAP, and AS were lower than those of both DDAC and DBF-treated specimens and the untreated control specimens. Compared with the untreated specimens, higher heat release rates were observed for the specimens treated with the quaternary ammonia compounds, DDAC and DBF. The estimates for the FSI for DDAC and DBF were for values equal to or higher than for the untreated control specimens. At higher concentration levels, MAP, DAP and AS were effective in decreasing initial contribution of heat release to potential fire. Decay resistance tests were done according to AWPA E 10 standard method using one brown-rot fungus and one white rot-fungus. Decay resistance tests revealed that solid wood specimens treated with DDAC and DBF showed resistance against the fungi tested, however, MAP, DAP and AS did not provide complete protection. While DBF and DDAC increased resistance of plywood specimens, high mass losses in plywood specimens treated with MAP, DAP and AS were obtained.

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Brandverhalten und Fäuleresistenz von mit quaternären Ammoniumverbindungen und üblichen Feuerschutzmitteln imprägniertem Massivholz und Sperrholz

Zusammenfassung In dieser Studie wurden das Brandverhalten und die Fäuleresistenz von mit quaternären Ammoniumverbindungen (Didecyl-Dimethyl-Ammoniumchlorid (DDAC) und Didecyl-Imethyl-Ammonium-Tetrafluoroborat (DBF)) imprägniertem Massivholz und Sperrholz verglichen mit dem Verhalten von unbehandelten Kontrollproben sowie Proben, die mit üblichen Feuerschutzmitteln (Mono-

ammoniumphosphat (MAP), Diammoniumphosphat (DAP) und Ammoniumsulphat (AS)) imprägniert wurden. Die Prüfkörper wurden mit 1% und 4% wässrigen Lösungen der Chemikalien imprägniert. Durchgeführt wurden die Prüfung mit dem Brennrohr nach ASTM E 69, bei der die Masseverluste in den Prüfkörpern bestimmt werden, und die Cone-Calorimeter-Prüfung nach ASTM E 1354, bei der die Masseverluste, die Wärmefreisetzungsrate, der Entzündungszeitpunkt, die Gesamtwärmefreisetzung und die spezifische Rauchentwicklung bestimmt werden. Die Ergebnisse der Cone-Calorimeter-Prüfungen dienten zur Bestimmung des Flammenausbreitungsindex (FSI) im Steiner-Tunnel-Test nach ASTM E 84. Die Wärmefreisetzungsrate der mit MAP, DAP und AS imprägnierten Proben war niedriger als die der mit DDAC bzw. mit DBF imprägnierten Proben als auch der unbehandelten Kontrollproben. Die Wärmefreisetzungsraten der mit den quaternären Ammoniumverbindungen DDAC und DBF imprägnierten Proben waren höher als die der unbehandelten Proben. Der Flammenausbreitungsindex (FSI) der mit DDAC und DBF behandelten Proben war gleich oder höher dem der unbehandelten Kontrollproben. Durch Behandlung mit höheren Konzentrationen an MAP, DAP und AS konnte die Wärmefreisetzung verzögert werden. Fäuleresistenzversuche wurden gemäß der AWPA E 10 Methode unter Verwendung eines Braunfäule- und eines Weißfäulepilzes durchgeführt. Fäuleresistenzversuche zeigten, dass mit DDAC und DBF behandelte Massivholzproben gegen die beiden Pilze resistent waren, während MAP, DAP und AS keinen Komplettschutz boten. Während DBF und DDAC die Resistenz der Sperrholzproben erhöhten, zeigten sich bei den mit MAP, DAP und AS behandelten Sperrholzproben hohe Masseverluste.

1 Introduction

Development of new wood preservative systems has received much attention in recent years. Quaternary ammonia compounds (QAC) or alkylammonium compounds (AAC) are possible acceptable wood preservatives since they are used in disinfectants, fabric softeners and conditioners and have generally low mammalian toxicity (Hedley et al. 1982). Water soluble QACs have been studied extensively because QACs are surfactants, destroy bacteria and fungi, and show antielectrostatic and anticorrosive properties (Pernak et al. 2004). A wide range of QACs have been long evaluated for their efficacy against decay fungi and termites and their interactions with wood components (Butcher et al. 1977, Butcher and Drysdale 1978, Hedley et al. 1982, Preston and Nicholas 1982, Preston 1983, Tsunoda and Nishimoto 1983, Tsunoda and Nishimoto 1987, Jin and Preston 1991, Loubinoux et al. 1992, Loubinoux and Malek 1992). Didecyl dimethyl ammonium chloride (DDAC), as a QAC, is currently used as a component of ammoniacal copper quat (ACQ) wood preservative to improve its fungicidal properties (Lebow 1996, Wood Handbook 1999). Since cations and anions play a significant role in the formulation of wood preservatives determining their melting temperature, density, viscosity, and solubility in water and tetrafluoroborates are known to be stable in contact with water and air (Pernak et al. 2004), didecyl dimethyl ammonium tetrafluoroborate (DBF) containing tetrafluoroborate (BF_4^-) as a counter ion has recently been developed (Kartal et al. 2005, 2006a, b, Hwang et al. 2006, 2005).

The authors previously evaluated DBF as a preservative to protect wood against mold and staining fungi in laboratory conditions in comparison with DDAC (Kartal et al. 2005). Previous laboratory studies on decay and termite resistance of wood treated with DBF showed that wood specimens treated with 0.5 and 1% DBF solutions at retention levels of 4 and 8 kg/m^3, respectively, inhibited *T. palustris* and *C. versicolor* attack even after a 10-day severe weathering process (Kartal et al. 2005, 2006a, b, Hwang et al. 2006, 2005). DBF treatment with 0.1% concentration at 0.8 kg/m^3 retention level was, however, effective against subterranean termites, *Coptotermes formosanus* (Kartal et al. 2006b). In another study by Kartal et al. (2005), the ability of DBF and DDAC to inhibit discolorations by selected mold and stain fungi was screened in laboratory conditions. Both DBF and DDAC at 1% concentration provided protection against the fungi tested. Soil bed tests showed that DBF-treated wood specimens treated with 1% DBF solution concentration (7.7 kg/m^3 retention level) showed better performance compared to 0.01 and 0.1% DBF treatments. The 1% solution level was also effective to protect the wood specimens against *Coniophora puteana* and *C. versicolor* in Basidiomycetes tests (Kartal et al. 2006a).

Kartal et al. (2007) showed that treatments of plywood specimens with two concentrations (3 and 11%) of MAP and DAP resulted in relatively higher mass losses compared to boric acid and borax treatments. As MAP and DAP retention levels in plywood specimens increased, mass losses decreased for both *T. palustris* and *C. versicolor*.

While DBF and DDAC was tested for leaching and fixation, decay and termite resistance in laboratory conditions and soil bed tests, no fire performance tests have been performed (Kartal et al. 2008, Hwang et al. 2007a, b). Considerable research has been carried out to evaluate fire performance of wood and wood based products treated with fire retardants. Fire-retardant formulations for wood products are generally prepared using compounds such as organo-phosphorus and aluminum and magnesium hydrates. The most common fire retardant chemicals used for wood are the inorganic salts, such as diammonium

phosphate (DAP), monoammonium phosphate (MAP), zinc chloride, and ammonium sulfate (AS) (LeVan and Winandy 1990). Boron containing fire-retardant chemicals are also effectively used as a smoke suppressant. Melamine containing nitrogen and its combination with phosphorus in its structure is another type of fire-retardant chemical that emerged in the 1990s (LeVan and Winandy 1990). Some fire retardant systems for wood are based on formation of a phosphate salt of an organic compound and are referred to as organic salts. However, all fire-retardant systems for wood rely on the elements of phosphorus, nitrogen, or boron and phosphorus is usually the central element (LeVan and Winandy 1990). Phosphoric acid esterifies the wood polysaccharides and water is released, which promotes the dehydration reactions of wood in case of phosphorus-based fire-retardant (Grexa et al. 1999) and the mechanism of acid dehydration reduces flame spread (Lebow and Winandy 1999a, b). Boron compounds can be also used to increase the resistance of composites and solid wood to fire and biodegradation (Barnes and Amburgey 1993, Murphy et al. 1993, Laks and Manning 1997, Tsunoda et al. 2002). The addition of borate-based buffers to fire-retardant chemicals was found to significantly mitigate thermal degradation (Winandy 1997). Boron compounds are also wood preservatives acting as both effective insecticide and fungicide with low toxicity (Thevenon et al. 1997). They show improved properties in hydrolytic stability, solubility in polar and non-polar solvents, boiling points, melting points, viscosity, and compatibility with a range of other chemical compounds (Vinden and Romero 1997). The fire performance of DDAC and ACQ in combination with known fire retardants were investigated in a study on new combination fire-preservative treatments for wood shingles (Sweet et al. 1996). Results for specimens treated with only the preservative were not reported.

In this study, both the decay resistance in laboratory conditions and the fire performance of solid sawn wood and plywood treated with a preservative (quaternary ammonia compounds, didecyl dimethyl ammonium chloride (DDAC) and didecyl dimethyl ammonium tetrafluoroborate (DBF)) or a fire retardant (monoammonium phosphate (MAP), diammonium phosphate (DAP) and ammonium sulphate (AS)) were evaluated. The intent was to determine whether the preservatives had fire retardancy or the fire retardants had decay resistance.

2 Materials and methods

2.1 Materials

The solid sawn wood specimens were cut from the sapwood portions of Scots pine (*Pinus sylvestris* L.) logs obtained from Karaoglulari Orman Urunleri Insaat Otomotiv Sanayi ve Ticaret A.S., Turkey.

Commercially manufactured rotary cut veneers of birch (*Betula* spp.) and larch (*Larix* spp.) wood obtained from Kurogullari Yapi Malzemeleri Ticaret ve Sanayi A.S., Turkey were used to manufacture structural plywood panels with eleven layers. Before the manufacture of the plywood panels, the veneers were equilibrated to a moisture content (MC) of 7%. The commercial phenol formaldehyde (PF) resin was applied to the veneers at a rate of 200 g/m^2 using a commercial glue spreader. The glue spread rate was kept constant by adjusting and using the same speed and glue thickness on the glue spreader. The veneers were weighed before and after spreading to determine the exact amount of adhesive actually applied. The 11-ply panel was assembled immediately after the veneers were spread with adhesive. The veneers were then laid-up into a plywood billet and hot pressed into 2200 mm by 1300 mm by 20 mm panels using a pressure of 1.2 N/mm^2 at 140°C for 12 min in a commercial press. The composition of plywood panels was in birch-larch-birch veneer order.

2.2 Treatments

Five chemicals were used in the treatments of solid wood and plywood specimens (Fig. 1). The chemicals included two quaternary ammonia compounds: (1) didecyl dimethyl ammonium chloride (DDAC), and (2) didecyl dimethyl ammonium tetrafluoroborate (DBF). The three common fire retardant chemicals tested for comparative purposes were: (1) monoammonium phosphate ($NH_4H_2PO_4$, MAP), (2) diammonium phosphate ($(NH_4)_2HPO_4$, DAP), and (3) ammonium sulfate ($(NH_4)_2SO_4$, AS).

The solid sawn wood and plywood specimens were treated with 1 and 4% (% m/v) aqueous solutions of the chemicals stated above. There were also untreated control specimens. Prior to treatment, the test materials were cut into the 100 mm (R) by 100 mm (T) by 20 mm (L) cone calorimeter specimens and the 9.5 mm (T) by 20 mm (R) by 1016 mm (L) fire tube specimens. The specimens were conditioned at 20°C and 65% relative humidity (RH) for three weeks before the treatments. The treatment cycle consisted of a 30 min vacuum at 53.3 kPa (400 mm Hg) and 30 min pressure at 196 kPa (2 kp/cm^2) in a treatment cylinder. After treatments, specimens were blotted dry and reweighed to determine the chemical retention.

Retention levels of the chemicals after treatment were much lower than those in commercial applications (Table 1). Commercial fire retardants are typically used at retention levels between 32 and 80 kg/m^3 (LeVan and Winandy 1990), however, in the study, the retention levels varied from 3.4 to 18.5 kg/m^3 and from 4.3 to 20.5 kg/m^3 for solid sawn wood specimens and plywood specimens,

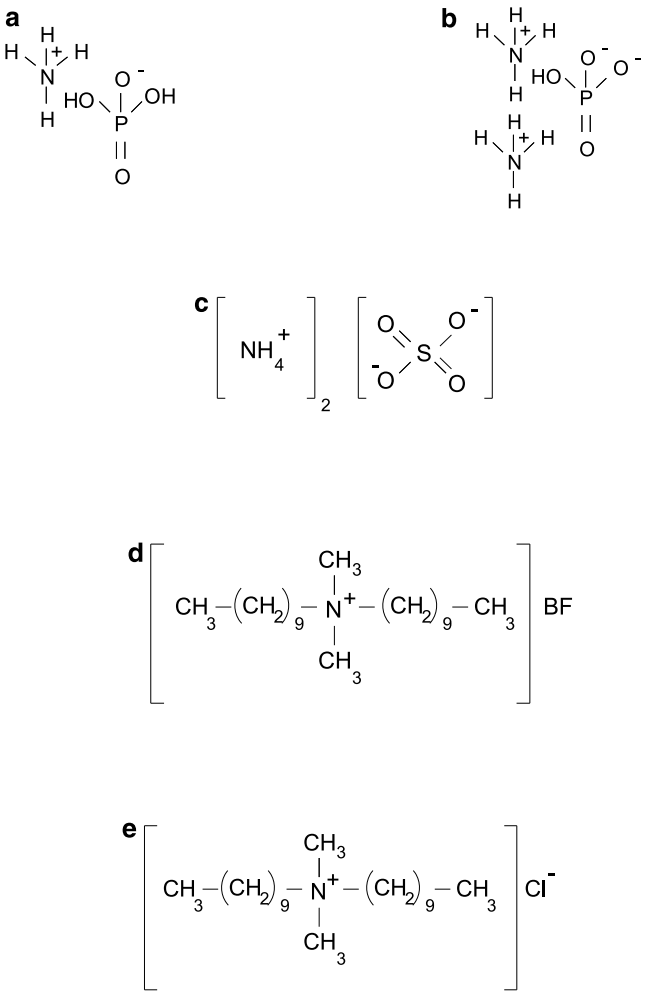

Fig. 1 Formulations of the chemicals used in the study **a** MAP, **b** DAP, **c** AS, **d** DBF, **e** DDAC
Abb. 1 Chemische Formeln der verwendeten Chemikalien **a** MAP, **b** DAP, **c** AS, **d** DBF, **e** DDAC

Table 1 Retention of chemicals in treated solid wood and plywood specimens
Tabelle 1 Eingebrachte Chemikalienmengen in die imprägnierten Massivholz- und Sperrholzproben

	Retention[a] (kg/m^3)			
	Cone calorimeter tests		Fire tube tests	
Chemicals	Solid wood	Plywood	Solid wood	Plywood
MAP 1%	3.5 (0.09)	4.6 (0.08)	3.9 (0.15)	5.5 (0.13)
MAP 4%	17.9 (0.56)	20.5 (0.18)	17.2 (0.66)	24.2 (0.76)
DAP 1%	3.5 (0.09)	4.3 (0.16)	4.1 (0.53)	4.6 (0.20)
DAP 4%	17.4 (1.63)	19.6 (0.17)	8.9 (0.67)	19.1 (3.40)
AS 1%	3.4 (0.04)	4.6 (0.29)	3.3 (0.54)	5.2 (0.07)
AS 4%	13.4 (0.60)	18.1 (0.52)	14.7 (0.87)	21.7 (0.58)
DDAC 1%	4.5 (0.06)	4.6 (0.29)	4.7 (0.07)	5.3 (0.28)
DDAC 4%	15.9 (0.70)	17.5 (0.39)	13.1 (0.19)	17.2 (0.38)
DBF 1%	4.7 (0.14)	5.1 (0.07)	4.9 (0.07)	5.4 (0.17)
DBF 4%	18.5 (0.69)	19.2 (1.18)	17.7 (0.62)	19.2 (0.53)

[a] Results are means (standard deviations) of 60 replicates.

respectively, in cone calorimeter tests, and from 3.3 to 17.7 kg/m^3 and from 4.6 to 24.2 kg/m^3, for solid sawn wood specimens and plywood specimens, respectively, in fire tube tests. In general, higher retention levels were obtained in the plywood specimens when compared to solid wood specimens in the study.

2.3 Fire performance – fire tube tests

The ASTM E 69 (ASTM 2002) test method known as the "fire tube test" was used to measure the percentage weight loss caused by combustion. Each 9.5 mm by 20 mm by 1016 mm specimen was placed in the fire tube apparatus, and the flame from a gas burner was placed directly beneath the vertically oriented specimen. Specimens were exposed to the gas burner for 3 min (Sweet et al. 1996). The duration of the exposure to the gas burner specified in ASTM E 69 is 4 min. Weight loss was recorded for an additional 7 min after the burner was removed. The percentage weight loss at the end of the 10 min was reported. Specimens were conditioned at 23°C, 30% RH prior to the fire tube testing. Three replicates of the specimens were tested.

2.4 Fire performance – cone calorimeter tests

The ASTM E 1354 (ASTM 2004) (also ISO standard 5660-1 (ISO 1993)) test method known as the "cone calorimeter test" was used to measure the mass loss, heat release rate, time for sustained ignition, effective heat of combustion, and specific extinction area. The 100 mm by 100 mm by 20 mm specimens were tested in the horizontal orientation with the conical radiant electric heater located above the specimen. The unexposed surfaces of the test specimen were wrapped in aluminum foil and the specimen placed on a piece of low density refractory fiber blanket within the holder. The external heat flux was 50 kW/m^2 and the retainer frame for the test specimen was used without the wire grid. The electric spark igniter was inserted above the test specimen until the time for sustained ignition of the test specimen was observed and recorded. The criterion for sustained ignition was 10 s. For the duration of the test, the heat release rate (HRR) due to combustion was determined using the oxygen consumption methodology. In addition, the mass loss of the specimen was recorded during the test. The effective heat of combustion (heat release per unit mass loss) was calculated from the heat release and the mass loss data. The amount of visible smoke produced by the burning specimen was evaluated by measuring the obscuration of a laser beam in the exhaust duct. The 57 mm orifice plate was used and the measured exhaust flow was 0.024 m^3/s. The scan rate for recording of the data was one reading per second. Specimens were conditioned at 23°C, 50% RH prior to the cone calorimeter testing. Three replicates of the specimens were tested.

2.5 Decay resistance tests

A soil-block test, AWPA E 10 test method (AWPA 2006), was used to measure the decay resistance. The 19 mm by 19 mm by 20 mm specimens for decay resistance test were cut from test specimens that had been treated for cone calorimeter tests. Nine replicate specimens of each treatment were dried to constant weight at 60°C and steam-sterilized at 100°C, weighed, and exposed to one brown rot fungus, *Tyromyces palustris* (Berkeley et Curtis) Murrill (FFPRI 0507) and one white rot fungus, *Corioulus versicolor* (MAD 697). After 12 weeks of incubation at 27°C and 70% RH, the surface fungus mycelium was removed, the specimens were dried at 60°C, and mass losses were determined as percentage of total specimen mass based on initial mass of the specimens before decay tests. The percentage mass loss in the test specimens provided a measure of decay resistance of the sampled material.

3 Results and discussion

3.1 Fire tube tests

The residual mass fraction after 10 min exposure in the fire tube apparatus of the solid wood and plywood specimens was the primary test result from the fire tube test (Fig. 2). Better fire performance is associated with greater residual mass fraction. More residual char reflects reductions in the production of combustible volatiles. In the reporting of these results and the statistical analyses, one replicate each of 4% AS treated plywood, 4% MAP solid sawn pine, and 4% DAP solid sawn pine were deleted as outliners from the rest of the data. The investigation of the statistical significance of differences in the means for different treatments was based on Duncan's Multiple Range Test with alpha of 0.05. Control specimens had a residual mass fraction of 0.18 in the solid sawn tests and 0.19 in the plywood tests (Fig. 2). In the tests, lower average residual mass fractions of 0.16 and 0.14 were found in the solid wood specimens treated with 1 and 4% DDAC, respectively, but the results for DDAC and DBF treated specimens were not significantly different than the results for the untreated specimens.

Except for the 1% AS, the residual mass fractions for the MAP, DAP, and AS treated specimens were significantly higher than the results for the untreated specimens. In the tests with MAP and DAP, considerably higher residual mass fractions occurred in the solid sawn specimens treated with 4% of the chemicals. The mean residual mass fractions for the 4% MAP, DAP and AS treatments of solid sawn wood were 0.603, 0.439 and 0.295, respectively (Fig. 2). The average retentions for these three treatments of solid sawn specimens were 17.9, 17.4, and 13.4 kg/m^3, respectively (Table 1). For DAP treated southern yellow pine solid sawn specimens (30% RH conditioning), literature results for final residual mass fractions in fire tube tests are 0.166, 0.297, 0.337, 0.363 and 0.837 for retentions of 0, 14.4, 16.0, 25.6, and 89.7 kg/m^3, respectively (Forest Products Laboratory 1953).

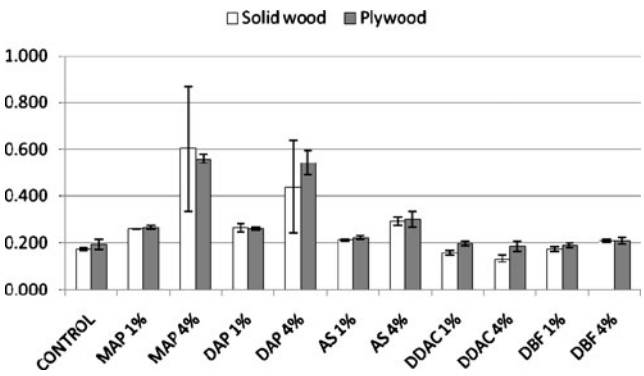

Fig. 2 Residual mass fraction in the specimens during fire tube tests (residual mass fraction = final mass/initial mass)
Abb. 2 Relativer Masseverlust (Endmasse/Anfangsmasse) in den Proben während des Brennrohrversuchs

3.2 Cone calorimeter tests

Heat release rate (HRR) is the main parameter in the cone calorimeter tests. The increasing uptake of a flame retardant has a strong influence on lowering the heat release rate (Grexa and Lübke 2001). A typical curve for wood is an ini-

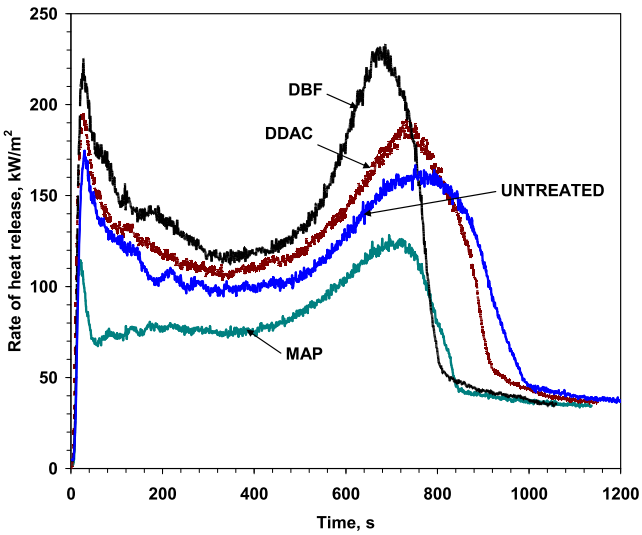

Fig. 3 Typical heat release rate curves for untreated solid sawn pine specimens and specimens treated with 4% solutions of the preservatives DBF and DDAC and the fire-retardant MAP
Abb. 3 Typische Wärmeentwicklungskurven für die unbehandelten Kiefernschnittholzproben und für die mit 4%-iger Lösung der Schutzmittel DBF und DDAC sowie mit dem Feuerschutzmittel MAP imprägnierten Proben

Table 2 Test results from cone calorimeter tests of solid sawn Scots pine
Tabelle 2 Versuchsergebnisse der Cone-Calorimeter-Prüfungen von Kiefernschnittholz

Chemicals	Average heat release rate[a,b] ($kW\,M^{-2}$) 60 s	300 s	Average effective heat of combustion[a,c] ($MJ\,kg^{-1}$)	Average specific extinction area[a,c] ($m\,kg^{-1}$)	Average mass loss rate[a,d] ($g\,s^{-1}$-m^2)	Residual mass fraction[a,e]
Control	147 (1) C	116 (1) C	13.1 (0.1) A	98.8 (24.3) AB	9.9 (0.1) CDE	0.160 (0.010) F
MAP 1%	124 (7) E	92 (4) EF	11.7 (0.3) B	52.2 (17.1) DEF	9.4 (0.4) EF	0.212 (0.043) CD
MAP 4%	92 (10) F	73 (5) G	9.3 (0.6) D	9.1 (14.7) G	9.3 (0.3) F	0.296 (0.013) A
DAP 1%	132 (7) DE	98 (4) DE	11.8 (0.2) B	63.8 (24.4) CDE	9.6 (0.5) DEF	0.227 (0.026) C
DAP 4%	121 (7) E	90 (3) EF	10.5 (0.4) C	30.0 (6.2) FG	9.6 (0.2) DEF	0.266 (0.005) B
AS 1%	140 (15) CD	103 (8) D	12.9 (0.5) A	74.6 (15.3) BCD	9.2 (0.1) F	0.174 (0.020) EF
AS 4%	98 (7) F	86 (1) F	10.8 (0.5) C	40.7 (10.5) EF	10.0 (0.2) BCD	0.230 (0.004) C
DDAC 1%	163 (9) B	124 (5) BC	13.4 (0.4) A	85.8 (15.8) ABC	10.1 (0.1) CB	0.170 (0.012) EF
DDAC 4%	166 (2) B	126 (3) B	13.5 (0.1) A	109.8 (21.4) A	10.1 (0.3) BC	0.173 (0.008) EF
DBF 1%	165 (3) B	130 (6) B	13.5 (0.2) A	90.8 (20.3) ABC	10.5 (0.3) B	0.182 (0.005) EF
DBF 4%	182 (5) A	142 (6) A	13.4 (0.9) A	105.0 (15.6) AB	11.3 (0.3) A	0.199 (0.009) CDE

[a] Results are for the means, (standard deviation), and letter ranking per Duncan's multiple range test for significance level of 0.05 for three replicates. Differences between mean values with same letter are not significant. Only two replicates of AS 4%.
[b] Averaged for 60 or 300 s after the time for sustained ignition.
[c] Averaged over the entire duration of the test.
[d] Averaged over the time period from 10% of ultimate specimen mass loss to 90% of ultimate specimen mass loss.
[e] Calculated as final mass/initial mass.

Table 3 Test results from cone calorimeter tests of plywood
Tabelle 3 Versuchsergebnisse der Cone-Calorimeter-Prüfungen von Sperrholz

Chemicals	Average heat release rate[a,b] ($kW\,M^{-2}$) 60 s	300 s	Average effective heat of combustion[a,c] ($MJ\,kg^{-1}$)	Average specific extinction area[a,c] ($m\,kg^{-1}$)	Average mass loss rate[a,d] ($g\,s^{-1}$-m^2)	Residual mass fraction[a,e]
CONTROL	177 (4) AB	127 (4) A	11.5 (0.1) B	61.5 (9.6) A	12.2 (0.2) A	0.249 (0.002) BC
MAP 1%	173 (18) ABC	114 (4) B	11.0 (0.2) C	50.1 (30.0) AB	11.8 (0.1) B	0.238 (0.014) BC
MAP 4%	118 (11) D	90 (4) C	9.8 (0.2) D	24.3 (12.2) BC	9.8 (0.2) D	0.298 (0.015) A
DAP 1%	157 (5) BC	109 (2) B	11.0 (0.1) C	59.2 (5.7) A	11.9 (0.2) B	0.234 (0.010) C
DAP 4%	123 (10) D	91 (3) C	9.8 (0.2) D	12.8 (28.0) C	10.1 (0.2) C	0.281 (0.016) A
AS 1%	171 (9) ABC	114 (12) B	11.2 (0.04) C	76.7 (2.1) A	11.9 (0.1) B	0.240 (0.013) BC
AS 4%	151 (15) C	98 (1) C	11.1 (0.2) C	18.0 (13.6) C	9.9 (0.1) CD	0.258 (0.004) B
DDAC 1%	183 (10) A	130 (6) A	11.3 (0.2) BC	69.5 (5.4) A	12.5 (0.1) A	0.238 (0.018) BC
DDAC 4%	185 (18) A	137 (13) A	12.4 (0.1) A	74.0 (17.3) A	12.2 (0.1) A	0.240 (0.004) BC
DBF 1%	171 (6) ABC	126 (3) A	11.2 (0.1) BC	65.4 (10.1) A	12.3 (0.2) A	0.251 (0.006) BC
DBF 4%	188 (5) A	129 (4) A	12.2 (0.2) A	67.1 (10.1) A	11.8 (0.2) B	0.250 (0.007) BC

[a] Results are for the means, (standard deviation), and letter ranking per Duncan's multiple range test for significance level of 0.05 for three replicates. Differences between mean values with same letter are not significant. Only two replicates of AS 1%.
[b] Averaged for 60 or 300 s after the time for sustained ignition.
[c] Averaged over the entire duration of the test.
[d] Averaged over the time period from 10% of ultimate specimen mass loss to 90% of ultimate specimen mass loss.
[e] Calculated as final mass/initial mass.

tial increase to a peak HRR, then a drop to a steady-state HRR, which is followed by a second peak as the final portion of the specimen is consumed (Ayrilmis et al. 2007). For the solid wood (Fig. 3) and plywood specimens, the typical curves for wood products were observed in the tests. For reporting purposes, the peaks and averages derived from the curves are reported (Tables 2 to 5).

The average HRR's (Table 2) of the solid sawn specimens treated with the fire-retardant treatments MAP, DAP, and AS were significantly lower than for the untreated control specimens and the average HRR's of the DDAC and DBF-treated specimens were significantly higher than for the untreated control specimens. The reporting of the statistical significance of differences in the means for different treatments (Tables 2 to 5) is based on Duncan's Multiple Range Test with alpha of 0.05. Also for the solid sawn specimens, the initial peak heat release rate (PHRR) (Table 4), the 4% DDAC and the DBF specimens had significantly higher re-

Table 4 Model predictions derived from cone calorimeter solid sawn Scots pine
Tabelle 4 Abgeleitete Werte aus den Cone-Calorimeter-Prüfungen von Kiefernschnittholz

Chemicals	Times for sustained ignition[a,b] (s)	Peak heat release rate[a,c] (kW M^{-2})	Total heat released[a] (MJ/kg)	Beta[a,c]	Estimated FSI[a,c]
Control	18 (3) A	175 (1) D	126 (2) AB	0.196 (0.008) E	82 (5) C
MAP %1	15 (2) ABC	177 (20) CD	103 (2) C	0.200 (0.026) DE	89 (20) BC
MAP %4	15 (1) ABC	131 (18) E	75 (6) E	0.110 (0.026) F	48 (7) E
DAP %1	16 (2) ABC	177 (7) CD	103 (1) C	0.195 (0.006) E	81 (4) C
DAP %4	16 (2) ABC	178 (15) CD	86 (1) D	0.188 (0.016) E	77 (10) CD
AS %1	15 (1) ABC	185 (8) BCD	121 (1) B	0.217 (0.010) CDE	104 (15) ABC
AS %4	14 (1) BC	130 (9) E	96 (5) C	0.126 (0.018) F	52 (5) DE
DDAC %1	16 (1) AB	193 (14) ABCD	128 (6) AB	0.230 (0.022) BCD	111 (29) AB
DDAC %4	13 (2) C	203 (8) AB	130 (4) A	0.255 (0.014) AB	EUL[d]
DBF %1	15 (1) ABC	199 (11) ABC	127 (4) AB	0.243 (0.017) ABC	119 (EUL[d]) A
DBF %4	14 (1) BC	213 (10) A	127 (8) AB	0.269 (0.019) A	EUL[d]

[a] Results are for the means, (standard deviation), and letter anking per Duncan's multiple range test for significance level of 0.05 for three replicates. Differences between mean values with same letter are not significant. Only two replicates of AS 4%.
[b] Observation of flaming ignition that was sustained for 10.
[c] Peak heat release rate, times for sustained ignition and total heat released are used for model predictions calculated using methodology described in Dietenberger and White (2001) and White and Dietenberger (2004).
[d] EUL – exceed upper limits of the logarithmic correlation between the ASTM E 84-07 flame spread index (FSI) and β that is used to obtain the estimate of the flame spread index for one or more tests.

Table 5 Model predictions derived from cone calorimeter of plywood
Tabelle 5 Abgeleitete Werte aus den Cone-Calorimeter-Prüfungen von Sperrholz

Chemicals	Times for sustained ignition[a] (s)	Peak heat release rate[a] (kW M^{-2})	Total heat released[a] (MJ/kg)	Beta[a,b]	Estimated FSI[a,b]
Control	24 (0.2) AB	223 (16) AB	142 (2) B	0.254 (0.022) ABC	143 (EUL[c]) A
MAP %1	25 (1) A	202 (8) BCD	136 (4) BC	0.214 (0.010) DE	100 (13) BC
MAP %4	22 (2) BC	160 (1) E	113 (2) D	0.146 (0.009) F	58 (3) D
DAP %1	22 (2) BC	186 (9) D	132 (3) C	0.198 (0.011) E	84 (8) C
DAP %4	20 (2) CD	160 (8) E	113 (4) D	0.154 (0.002) F	61 (1) D
AS %1	24 (1) AB	203 (3) BCD	137 (0.3) BC	0.220 (0.005) DE	107 (9) B
AS %4	18 (0.5) DE	198 (8) CD	136 (2) BC	0.229 (0.028) CD	97 (7) BC
DDAC %1	22 (1) BC	229 (4) A	141 (6) B	0.270 (0.004) AB	EUL[c]
DDAC %4	17 (2) E	220 (19) AB	156 (3) A	0.278 (0.025) A	EUL[c]
DBF %1	22 (2) BC	211 (8) ABC	141 (4) B	0.242 (0.008) BCD	142 (EUL[c]) A
DBF %4	17 (1) E	221 (6) AB	159 (4) A	0.279 (0.012) A	EUL[c]

[a] Results are for the means, (standard deviation), and letter anking per Duncan's multiple range test for significance level of 0.05 for three replicates. Differences between mean values with same letter are not significant. Only two replicates of AS 1%.
[b] Peak heat release rate, times for sustained ignition and total heat released are used for model predictions calculated using methodology described in Dietenberger and White (2001) and White and Dietenberger (2004).
[c] EUL – exceed upper limits of the logarithmic correlation between the ASTM E 84-07 flame spread index (FSI) and β that is used to obtain the estimate of the flame spread index for one or more tests.

sults than for the untreated control specimens and the results for 4% MAP and 4% AS were significantly lower. The total heat released (THR) (Table 4) of the DDAC and DBF solid sawn specimens were not significantly different than the results for the untreated specimens. The THR of the DAP, MAP and 4% AS specimens were significantly lower than the results for the untreated control specimens.

For the plywood specimens, the average HRR's for the DDAC and DBF-treated specimens were not significantly different than for the untreated control specimens (Table 3). Also for the plywood specimens, the PHRR were significantly lower for both 1 and 4% DAP, the 4% AS and the 4% MAP specimens compared with the untreated control specimens (Table 5). For THR, the results for the 4% DBF and 4% DDAC plywood specimens were significantly higher and the results for 4% MAP and 4% DAP were significantly lower than the results for the untreated control specimens (Table 5).

For the solid sawn specimens, the average effective heat of combustion (AEHOC) was calculated from the total heat release divided by the total mass loss for the duration of the test (Tables 2 and 4). The effective heat of combustion

(AEHOC) measured in the cone calorimeter corresponds mostly to the flame burning condition and thus to combustion of volatiles from material (Grexa and Lübke 2001). Lower AEHOC values reflect better fire performance characteristics. The AEHOC values for the 4% solution of DDAC and DBF were higher than the values for the untreated control specimens (Tables 2 and 3) but the differences were only statistically significant for the plywood specimens. Except for the 1% solution AS solid sawn specimens, the fire-retardant-treated (FRT) (MAP, DAP, AS) specimens had significantly lower AEHOC values than those for the untreated control specimens.

In addition to the effective heat of combustion, the heat release rates also reflect the mass loss rates. For the solid sawn specimens, the average mass loss rate (AMLR) results for DBF specimens were significantly higher than for the untreated control specimens (Table 2). The AMLR results for the 4% MAP and 1% AS were significantly lower than for the untreated control specimens. In the tests of the solid sawn specimens, the AMLR for the 1% DDAC and 4% DDAC specimens were not significantly different than the result for the untreated control specimens (Table 2). The AMLR for the other treated solid sawn specimens were significantly lower than the AMLR of the untreated control specimens.

The average specific extinction area (ASEA) is a measurement of the amount of visible smoke generated (Tables 2 and 3). Higher number reflects higher visible smoke being generated. The results for the DDAC and DBF specimens were not significantly different than those for the untreated control specimens. All treatments with MAP, DAP, AS at 4% concentration levels caused remarkable, and statistically significant, reductions in the average specific extinction area compared with the results for the untreated control solid wood and plywood specimens.

As with the fire tube test, the measurement of the residual mass fraction (RMF) is one of the evaluation methods used for fire-retardant chemicals. For all chemicals used, the mean RMF of the solid wood specimens at the end of the test were greater than the results for the untreated control specimens (Table 2). For the 4% solution DBF solid sawn specimens and all of the FRT specimens with the exception of the 1% AS specimens, the mean RMF was significantly higher than the value for the untreated control specimens. For the plywood specimens (Table 3), only the RMF results for the 4% solution MAP and DAP were significantly different and higher than the values for the untreated control specimens.

For the treated specimens, the ignition times ranged from 13 to 16 s for solid wood specimens and 17 to 25 s for plywood specimens (Tables 4 and 5). The untreated solid wood control specimens ignited at 18 s and plywood specimens at 24 s. For both the plywood and solid sawn specimens, the ignition times for the specimens treated with 4% solutions of DBF, AS and DDAC were significantly shorter than that for the untreated control specimens.

In the United States, the regulatory test for surface flammability of building products is the 7.32 m (25 ft) tunnel test according to ASTM E 84 standard test method (ASTM 2007). The Forest Products Laboratory (FPL) uses the cone calorimeter to predict the flame spread index (FSI) in the tunnel test (White and Dietenberger 2004). Dietenberger and White (2001) showed the development of the predictive model in which the surface fire growth propensity is represented by the initial PHRR and a bulk propensity parameter calculated from the total heat release, thickness and the inverse of the time for sustained ignition from the cone calorimeter test. In the model, a variable called β is calculated (Tables 4 and 5, Fig. 4) from the initial peak heat release rate and the bulk propensity parameter. Lower values for β reflect better fire performance. In the solid sawn tests, the β of the DBF and DDAC specimens were significantly higher than the β for the untreated control specimens. The values for 4% AS and 4% MAP were significantly lower than the value for the untreated control specimens (Table 4). For the plywood specimens, the results for DBF and DDAC were not significantly different than the results for the untreated control specimens (Table 5). The DAP, MAP and 1% AS results were significantly lower than the results for the untreated control specimens.

The calculated values for β are used to estimate the ASTM E 84 FSI and thus divide the model estimates into one of the three classes used in the U.S. building codes to classify materials. A β of 0.184 is used to differentiate

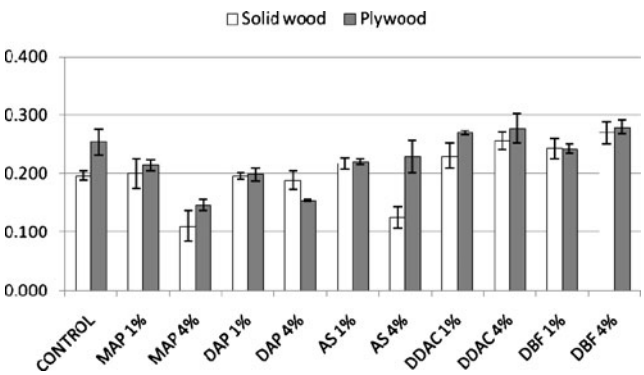

Fig. 4 The parameter "beta" that is obtained in the flame spread analysis of the cone calorimeter data. It depends on the initial peak heat release rate, the times for sustained ignition and the total heat release rate. The higher beta, the higher the expected flame spread index in the ASTM E 84-07 test for surface burning characteristics

Abb. 4 Parameter "Beta" ermittelt durch Flammenausbreitungsanalyse der Cone-Calorimeter-Daten. Beta hängt von der Wärmefreisetzungsrate zu Beginn, dem Entzündungszeitpunkt und der Gesamtwärmefreisetzungsrate ab. Je höher Beta, desto höher ist der zu erwartende Flammenausbreitungsindex nach ASTM E 84-07 Prüfung für die Verbrennungseigenschaften von Oberflächen

between Class C (FSI of 76 to 200) and Class B (FSI of 26 to 75) materials. The most restrictive class is Class A (FSI of 25 or less) which requires fire retardant treatment for U.S. domestic wood products. The model uses a β of zero to differentiate between Class B and Class A materials. In the U.S. building codes, the requirements for "fire-retardant-treated" (FRT) require the ASTM E 84 test to be conducted for a longer time period than specified in ASTM E 84. The model uses a β of -0.1 to identify materials that might qualify for such classification (Ayrilmis et al. 2007, White and Dietenberger 2004). Predictions for the ASTM E 84 FSI were calculated from a logarithmic correlation between β and the ASTM E 84-07 FSI (Dietenberger and White 2001) (Tables 4 and 5). Because of the logarithmic relationship between the β and the FSI, the equation used to estimate the FSI is not sensitive to variations in FSI greater than about 75 and does not produce a numerical estimate of the FSI for higher values of β (Tables 4 and 5) (Ayrilmis et al. 2007). In this study, no specimens were rated as Class A which is the most restrictive requiring fire retardant treatment of wood products. This likely reflected the relative low retention levels used in this study. For both the plywood and solid sawn MAP specimens, the FSI values increased from Class C to Class B as solution concentrations increased from 1 to 4%. This was also the case for AS treated solid sawn lumber and DAP treated plywood. In the specimens treated with the quaternary ammonia compounds, DDAC and DBF, the estimates for the FSI were for values equal to or higher than for the untreated control specimens. For one or more replicates of the DDAC and DBF specimens, estimates for FSI were not possible since the β value exceeded the limitation of the β–FSI correlation used to make the estimate (Tables 4 and 5).

Overall, MAP, DAP, and AS showed better fire performance as fire retardants, however, DBF and DDAC chemicals showed fire properties similar or slightly poorer than untreated wood based on both ASTM E 69 and ASTM 1354 standard tests. In this study, considerably lower retention levels of the chemicals were obtained when compared to commercial applications. It is likely that higher retention levels of the fire-retardant chemicals (MAP, DAP, and AS) would result in greater improved fire performance in both solid wood and plywood panels. Ayrilmis et al. (2007) showed that treatments of the face veneers in OSB panels using MAP and DAP were effective in reducing the initial contribution of heat release to potential fire growth and they reduced both the effective heat of combustion and the mass loss rate. It also delayed the times for sustained ignition. The OSB specimens coated with MAP and DAP-treated veneers had dramatically reduced initial PHRR and the MAP treatment was most effective in reducing this initial peak heat release rate. The DAP treatment reduced this second peak slightly higher than the MAP treatment but the differences were relatively small. Grexa et al. (1999) also evaluated fire performance of diammonium hydrogen phosphate-treated plywood by cone calorimeter tests. They found that the most efficient treatment was the application of diammonium hydrogen phosphate into wood mass and the magnesium hydroxide into the glue mixture. The time to ignition shortened for the sample with the flame retardant in the wood, samples with the flame retardant in the glue mixture only had the ignition time close to that for the untreated sample.

3.3 Decay resistance tests

The fungi used in the study caused mass losses of around 30% in untreated control solid wood and plywood specimens (Figs. 5 and 6). Natural durability of solid wood specimens and wood species used in the plywood manufacturing is an important factor in obtaining high mass losses in the untreated control specimens exposed to decay fungi. Scots pine sapwood (the solid wood material used in the study) and larch wood (one of the wood species used for plywood manufacturing in the study) are gener-

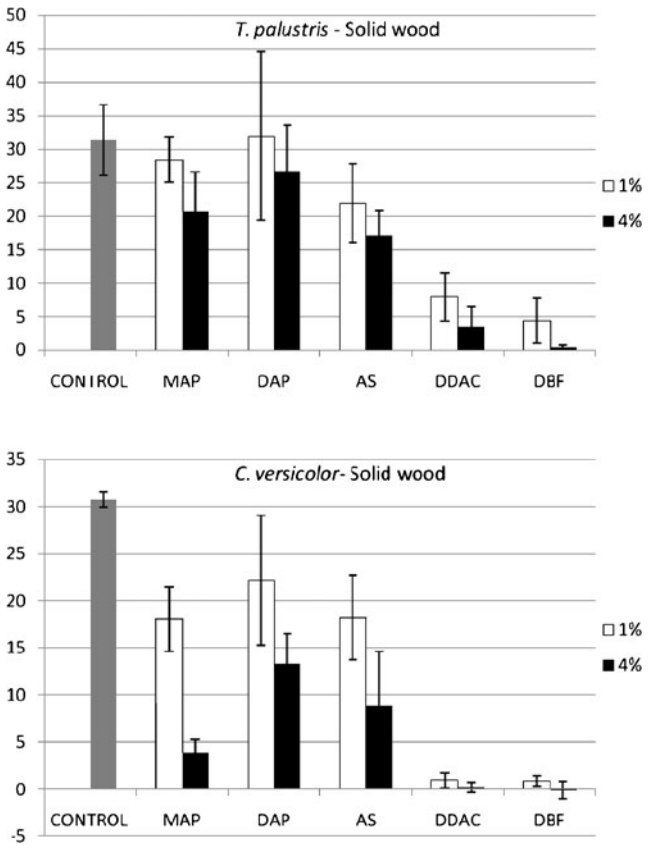

Fig. 5 Mass losses in the solid wood specimens exposed to *T. palustris* and *C. versicolor*
Abb. 5 Masseverluste in den Massivholzproben nach einem Befall mit *T. palustris* und *C. versicolor*

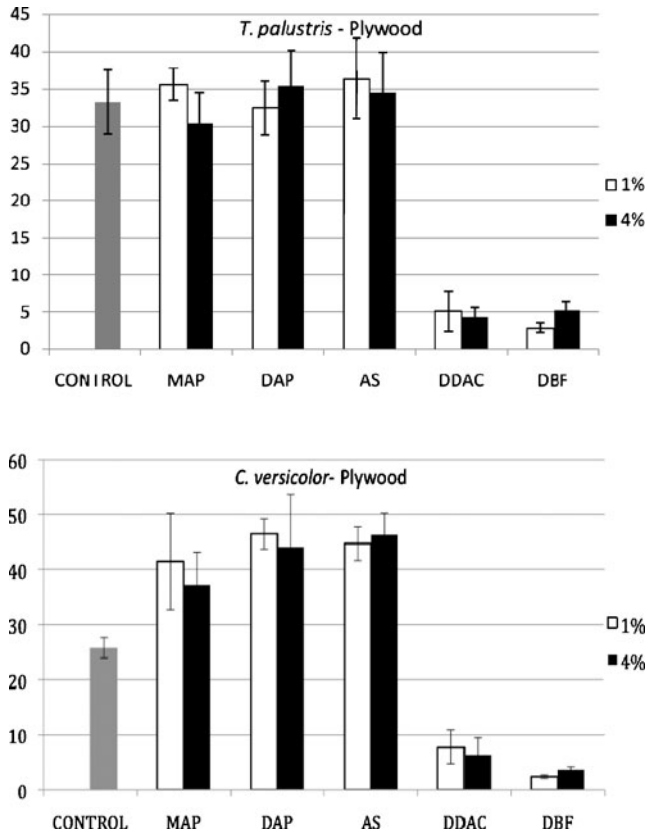

Fig. 6 Mass losses in the plywood specimens exposed to *T. palustris* and *C. versicolor*
Abb. 6 Masseverluste in den Sperrholzproben nach einem Befall mit *T. palustris* und *C. versicolor*

ally non-resistant and moderately resistant, respectively; however, birch wood (the other wood species used in the plywood panels) is non-resistant against fungal degradation (Panshin and De Zeeuw 1980, Scheffer and Morrell 1998). Treatments with MAP and AS at 1% concentration level resulted in slight decreases in mass loss, however, increased concentrations of these chemicals resulted in lower mass losses when compared to untreated control specimens. On the other hand, DDAC and DBF treatments resulted in considerably lower mass losses suggesting that DDAC and DBF provided protection against the fungi tested. Higher mass losses occurred in the plywood specimens treated with MAP, DAP and AS and control specimens exposed to *T. palustris* when compared to solid wood specimens (Fig. 6). Mass losses in the plywood specimens treated with DDAC and DBF were lower than 5% in the tests with *T. palustris*. Interestingly higher mass losses were obtained in the specimens treated with MAP, DAP, and AS with *C. versicolor*. DDAC and DBF, however, caused much lower mass losses in the specimens exposed to *C. versicolor*. In this study, the leach-resistance of the treatments was not investigated. In many practical applications, preservative treatments must have a high degree of leach resistance.

4 Conclusion

As expected, the fire-retardant chemicals tested in the study showed better performance than wood preservative chemicals, DDAC and DBF based on both ASTM E 69 and ASTM 1354 standard tests. Heat release rates of the specimens treated with MAP, DAP, and AS were lower than those of both DDAC and DBF-treated specimens and untreated control specimens. Higher heat release rates were observed for the specimens treated with the quaternary ammonia compounds, DDAC and DBF. The estimates for the FSI for DDAC and DBF were for values equal to or higher than for the untreated control specimens. At higher concentration levels, MAP, DAP and AS were effective in decreasing initial contribution of heat release to potential fire. DBF and DDAC, on the other hand, provided protection against fungal degradation in both solid and plywood specimens. However the fire-retardant chemicals used in the study were not able to protect wood from decay fungi. Combinations of the fire-retardants and wood preservatives might be beneficial in getting better performance against both fire and fungal decay. Practical treatments will also have to be leach-resistant.

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